Visco-plasticity and damage modeling of single crystal superalloys at high temperatures: a tensorial microstructure-sensitive approach
Adriana Mattiello

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Visco-plasticity and damage modeling of single crystal superalloys at high temperatures: a tensorial microstructure-sensitive approach

Thèse de doctorat de l'Université Paris-Saclay préparée à l'Ecole Normale Supérieure Paris-Saclay

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Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
Introduction

In turboshaft and turboprop engines the high pressure turbine is located just after the combustion chamber and used as an energy converter (Figure 1). The burned gas expansion generates an aerodynamic force on blades which produces the rotating motion of the turbine. The energy absorbed from the rotation is then transmitted to the other rotating parts of the engine through the crankshaft. Blades are therefore subjected to high temperatures, to the aggressive environment of the reacting burned gas and to strong centrifugal solicitations. The energy of the hot flow is only partially absorbed by the high pressure turbine. Another part is directly used on power wheels to extract a mechanical work onto the output shaft used to power, e.g., an helicopter main rotor. The whole power produced then depends on the energy gained from the combustion process. Since a more energetic combustion produces hotter flows, gaining more energy requires the blades to work in more severe conditions. High pressure turbine blades are then the most critical components of aero-engines and, moreover, their thermomechanical performances are the most limiting factors in power production. For these two reasons, blades are cast using the most temperature resistant materials: Ni-based Single Crystal Superalloys. Thanks to their chemical composition, to their unique well-ordered $\gamma/\gamma'$ microstructure and to the absence of grain boundaries, these alloys provide excellent mechanical properties and oxidation resistance at high temperatures.

Figure 1 – Location of the High Pressure Turbine in a turboshaft engine for helicopter.

However, disposing of very high temperature resistant materials is not the only aim of Original Engine Makers (OEM). The design of these mechanical components and the engine maintenance scheduling require accurate estimations of blades lifetimes and elongations, both at the design stage and for in-service conditions. In order to get these estimations, OEM need constitutive equations which model well the material’s response.

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Unfortunately, developing a mechanical model that provides quite precise estimations is a challenging task involving many issues:

– The operative temperature out of the combustion chamber overcomes 750°C, then the design loading is mainly creep. For a high power production an internal cooling system may be used (Figure 2). In this case the design loading is mainly thermomechanical fatigue (Reed, 2006, Moverare et al, 2009). Design loadings then cause the material to work in its visco-plastic regime, meaning that constitutive equations have to account for the loading rate and the history dependence of the material response. Furthermore, beside design loadings, the Certification Specifications demand to test the engine under more complex and severe thermomechanical loading paths than during service. Such testing cycles include fast and repeated overheatings simulating the One Engine Inoperative (OEI) events. The maneuver prescribed to twin-engine helicopters pilots in the case of one engine failure stage (Figure 3) is called “OEI”. It is made up of three phases. The first one is called “OEI 30s” and consists in pushing the only one working engine to its maximum operative temperature and speed, in order to supply for the total power needed to maintain the helicopter airborne and to re-gain altitude. This is the most critical phase of the maneuver because temperature gets closer to the material melting temperature with a heating rate up to 60°C for second. During the other two phases, the “OEI 2min” and the “OEI continuous”, temperature and speed are lower than the during the OEI 30s phase but they are still higher than during normal on-service conditions. From a modeling point of view, this means that the material response under very complex thermomechanical cycles –including thermal shocks and accelerations– has also to be reproduced.

Figure 2 – High Pressure Turbine Blades: (a) uncooled blades, (b) cooled blades.

– Despite the excellent hardening properties, the $\gamma/\gamma'$ microstructure involves also drawbacks. First, the elasto-(visco-)plastic response presents a strong dependency on the crystalline orientation. The material presents in fact a cubic symmetry at the micro-, meso- and macro-scale. Secondly, at high temperatures ($T > 900^\circ C$), the microstructure evolves. This evolution involves a change of the $\gamma/\gamma'$ morphology and a consequent degradation of the material strength. In addition to temperature,
the applied stress also contributes to microstructural changes: different morphology changes happen depending on stress sign and on the crystal direction (Caron et al, 1988, Khan and Caron, 1991). The various morphologies differently affect the material strength and the microstructural evolutions. This is a stress (intensity and orientation) dependent phenomenon which contributes to the anisotropy of the mechanical response. Thus, to summarize, the material response is anisotropic and, in addition, this is strongly dependent on microstructural changes. Many literature studies, as for example the works of Gaubert (2009), Tinga et al (2009b), Fedelich et al (2012b), Cormier et al (2015), le Graverend et al (2017), have evidenced that constitutive equations have to account for these evolutions in order to correctly reproduce the history and rate dependency of the material visco-plastic response.

There already exist thermo-mechanical models taking into account microstructural information. Examples are the Polystar model (Cormier et al, 2010, Ghighi, 2013, Giraud, 2013a, le Graverend et al, 2014a), the phase-field based models (Gaubert et al, 2015) and the models enriched with micromechanics elements (Svoboda and Lukas, 2000, Fedelich, 2002, Tinga, 2009a). Several of these models are mostly aimed to reproduce the response along the $<001>$ crystal direction because this one coincides with the centrifugally stressed axis of blades. However, because of the casting processing limitations, a misalignment of $\sim 15^\circ$ can be tolerated (Figure 4). Moreover, the geometry of blades is highly complex and the internal stress state is multiaxial. Others crystallographic directions are, then, unavoidably loaded. Hence, the anisotropy of the material cannot be neglected and the problem cannot be treated as unidimensional. Unfortunately, the most detailed models (as for example the one proposed in Tinga et al (2009b), Fedelich et al (2012b), Gaubert et al (2015)) still present accuracy limitations or, in some cases, these are too much CPU consuming, especially for Finite Elements computation on real geometries. At the same time, standard Norton’s visco-plastic modeling affects the precision of the estimations and computing the mechanical response on simplified
geometries with such models does not account for the real stress state in engine components.

![Figure 4 – Maximum misalignment tolerated between the material principal directions and the centrifugal axe of blades.](image)

It can be concluded that a suitable mechanical model for Single Crystal Superalloys should be a **3D model, history and rate sensitive, accounting for the anisotropy of the material –including the modeling of the microstructural evolutions– and allowing for efficient Finite Elements computations.** It is to address these issues that the present Ph.D. study was born. Three partners have taken part in the thesis project: SAFRAN Helicopter Engines, the PPrime Institute and the LMT Cachan. The developed modeling is therefore a synthesis of industrial, experimental, metallurgical, constitutive modeling and computational competences. The correct description of the visco-plastic properties of the material is essential for predicting the response under any type of load, so the prevision of creep and monotonic tension behaviors are the first aims of the modeling. For this purpose a new phenomenological model based on the Local State Method has been developed (Halphen and Neguyen, 1975, Germain et al, 1983). This choice has been done for two main reasons. First of all, the internal variables represent the state of the material, either mechanical and micro-structural. They naturally allow to account for the history dependency of the material response by rate form evolution laws. In addition to this, phenomenological models with internal variables have usually lower computational costs compared to micromechanics and phase-field based approaches.

The work has been carried out as follows. A literature review on the deformation mechanisms acting during high temperature creep of single crystal superalloys has been first carried out. Thanks to this preliminary study the first guidelines of the modeling have been drawn and the missing information about the material response have been pointed out. Special attention has been paid to the studies dealing with the CMSX-4 alloy, which is the material of interest in the present study. After the literature review an experimental campaign has been carried out. In order to investigate on the temperature and stress magnitude impact on the anisotropy of the mechanical response, creep and monotonic tensile tests have been carried out at high temperature ($\geq 900^\circ$C), low temperature ($< 900^\circ$C), at different stress level and along different crystal orientations. The two temperatures cho-
The tests have been mainly carried out in order to provide a consistent database for the model identification, complementary to initial database built during the Ph.D. thesis of R. Giraud (Giraud, 2013a). The temperature of 1050°C in fact corresponds to the average metal temperature of the blade section during take-off mainly undergoing creep damage. This temperature then will serve as basis for the mechanical modeling. As opposite, tests at 850°C have been carried out not only to enrich the experimental database for the model identification, but also to investigate on the low temperature/high stress creep response of the CMSX-4. The literature study has in fact highlighted some opened questions about the deformation mechanisms underlying the anisotropy of the mechanical response in the low temperature creep regime. Beside creep and monotonic tensile tests, further tests have been carried out in order to enrich the database required for the present modeling and also for further future developments. Dissolution tests have been realized in order to identify the equation governing the microstructural evolution and cyclic tests have been carried out to investigate the deformation and damage mechanisms activated by non-isothermal cyclic loads. Based on literature studies and on the results of the experimental campaign, the mechanical model has been developed (and identified). Since 1050°C is the most interesting temperature for both industrial and scientific purposes, the assumptions underlying the modeling have been formulated based on the creep and monotonic tension response of the CMSX-4 observed at this temperature. Confirming what was already observed in literature, microstructural evolutions have been found to govern the mechanical response at this temperature, so in the new model proposed a tensorial internal variable has been introduced to account for γ/γ′ microstructural evolution. As it will be shown in the following chapters, microstructural evolutions and their load dependency can be efficiently modeled at the mesoscale by exploiting the cubic symmetry of the material. For this reason it has been chosen to formulate the microstructural evolution laws at the Representative Volume Element (RVE). Visco-plasticity is also described at the mesoscale but, in order to account for the real mechanisms which generate plasticity in Face Centered Cubic crystals (FCC), two criterion functions and two flow equations have been formulated (chapter 3). Similarly to crystal plasticity (Mandel, 1973, Hill and Rice, 1972, Rice, 1975, Asaro and Rice, 1977, Asaro, 1983, Peirce et al, 1983), one criterion function and one flow equation describe the deformation produced by slips on octahedral systems, while the other criterion and flow equations account for the plasticity produced by cross-slips and shearing along the {001} planes. The degradation produced by the plastic damage, the topological inversion, the shearing of the hardening phase and the oxydation have finally been modeled via the scalar damage variable D. The equation describing the evolution of this variable, D = ..., has also been formulated at the mesoscale by means of a modified Lemaitre law. Modifications to the original Lemaitre damage law have been made in order to introduce the strain rate dependency of ductility. Thanks to the support provided by SAFRAN, the constitutive equations have been implemented in the Z-set language and structural computation on Finite Element (FE) model of real geometries have been performed by using the Z-set FE solver.

The structure of the present thesis reflects the above mentioned work steps. The first
chapter contains a synthesis of the preliminary literature review, as well as the first conclusions withdrawn about the modeling and the missing experimental information for considered CMSX-4. The second chapter describe the motivation, the strategy and the results of the experimental study carried out in the present Ph.D. In this chapter only the elements required to discuss the results are given. Further details are presented in the Appendices A, B and C. In the third chapter the constitutive equations formulated to describe microstructural evolutions and visco-plasticity are presented. The model proposed is identified on the 1050°C experimental results presented in the second chapter. At the end, several partial conclusions and perspective are given. The fourth chapter contains the description and the testing of an original damage law/threshold proposed in the present Ph.D. A brief literature review on the damage mechanism observed in single crystal superalloys is done as corresponding chapter introduction. Global conclusions and perspectives are presented at the end of the thesis. Appendices from D to M contain complementary elements of the modeling. In particular, the thermodynamic framework of the model presented in the chapter 3 is presented in the Appendix I and its application to complex loadings is presented in the Appendix M. Appendices from N to O present the parameters of the full model (visco-plasticity coupled to microstructural evolution and damage). Appendices Q and R present the code written in Z-set programming languages and the appendix S presents the input files for the Z-set solver. The appendix T presents the thermodynamic framework of the encoded equations. There exists a differences between the equations presented in the chapters 3 and 4 and the equations encoded in Z-set. Two exponential isotropic hardening laws (one for each criterion function) have been introduced in the code. It has been mainly done in order to facilitate further developments. The parameters governing the isotropic hardening are set to zero (Z-set material input file is contained in the Appendix S), thus, finally, the encoded model (presented in the Appendix Q and R) corresponds to the one presented in the chapters 3 and 4.
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— the Pprime institute of ENSMA in Poitiers where I have been supervised by Jonathan Cormier
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new and enriching collaborations.

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Chapter 1

Material, microstructural evolutions and mechanical response

“Welcome to the jungle”
Guns and Roses
Introduction

This chapter is a literature review on the deformation mechanisms, on the mechanical response of CMSX-4 and of Nickel based single crystal superalloys is proposed. The material microstructure, chemical composition, deformation and hardening mechanisms are presented first. Then, microstructural evolutions are described and the impact that they have on the material response during creep, monotonic tension and thermal cycling along the \(< 001>\) crystal direction (the most important crystal orientation from a technological point of view) is analyzed in detail. A point on the anisotropy of the creep response is done as well. At the end of the chapter, several conclusions are drawn. First modeling guidelines are deducted and the already missing information concerning the material response are pointed out.

1.1 CMSX-4: a structured material

CMSX-4 is a Ni-based single crystal superalloy produced by the Cannon Muskegon corporation. The chemical composition and the microstructure of this class of material are conceived to provide the best mechanical response at high temperatures.

1.1.1 Microstructure

A hardening phase is precipitated in a Nickel solid solution in order to strengthen the alloy. Hence, the material consists of two phases: a hardening and a ductile phase. The hardening phase is an ordered phase, called “\(\gamma'\)” or “precipitate” phase. At the micro-scale, this has an FCC crystal structure. This is shown in figure 1.1(b). Ni atoms are substituted by the hardening elements, Al and Ti or Ta, at the cube vertexes. The ordering relationship is of LI\(_2\), the stoichiometry is Ni\(_3\)(Al,Ti,Ta). The lattice parameter \(a_{\gamma'}\) corresponds to the Al-Al distance. At the meso-scale, precipitates have a cubic shape, as shown in figure 1.2.

The Nickel solid solution is called “matrix” or “\(\gamma\)’ phase. This is the disordered ductile phase containing the ductile and the refractory elements, i.e. Tungsten, Molybdenum, Cobalt, Chromium, Ruthenium and Rhenium. At the micro-scale, this presents a FCC structure, shown in figure 1.1(a). The lattice parameter \(a_{\gamma}\) is in this case equal to the Ni-Ni distance. At the meso-scale, the matrix is a continuous phase containing the cubic precipitates (figure 1.2(a)). Each precipitate is surrounded by matrix channels parallel to the principal directions of the crystal frame. These are shown in figure 1.2(b) \((w_1, w_2\) and \(w_3)\).

The coherency relationships between crystal cells are such that cube faces are parallel (Eq. (1.1)).

\[
\{100\}_\gamma // \{100\}_{\gamma'}, \quad \langle 010 \rangle_\gamma // \langle 010 \rangle_{\gamma'} \quad (1.1)
\]
Figure 1.1 – Schematic crystal structure and lattice parameters of: (a) the precipitate phase or $\gamma'$ phase (b) matrix phase or $\gamma$ phase.

Figure 1.2 – Example of the initial cubic microstructure of Single Crystal Superalloys: (a) SEM observation of a crystal oriented along $<001>$ (Cormier (2006), MC2 alloy) and (b) schematic representation of the microstructure.

Since the lattice parameters of the two crystals are slightly different, a non-zero mismatch ($\delta_u$, Equation (1.2)) exists. At ambient temperature and zero load the CMSX-4 natural misfit is positive but, as shown in Heckl et al (2011b), it decreases with increasing temperature. The same has been observed for many other Ni-based superalloys, as for example in Nathan et al (1985), Kuhn et al (1991), Royer et al (1995), Link et al (2000), Siebörger et al (2001), Diologent et al (2003), Zhang et al (2005), Mughrabi (2009), Heckl et al (2011a,b), Dirand et al (2013)

$$\delta_u = \frac{1}{2} \frac{a_{\gamma'} - a_{\gamma}}{a_{\gamma'} + a_{\gamma}}$$

At the micro-scale, elastic coherency strains accommodate the lattice misfit. For this reason a non-zero stresses state exists (figure 1.3 and 1.4). Precipitates are then subjected to a bi-axial tension state (along the directions parallel to the $\gamma$ channels) while the horizontal matrix channels result subjected to compression (along $\gamma'$ surfaces).
**1.1.2 Deformation mechanisms**

Plastic deformation occurs in crystals when dislocation slips are produced along the most closely packed directions lying in the most densely packed planes.

In FCC crystals there are four \{111\} closed packed planes. Each of these planes contains three closely packed \langle 011 \rangle directions. 12 slip systems can be activated (figure 1.5, Equation 1.3), depending on the crystal orientation solicited and on the stress magnitude. In Continuum Mechanics these slips are called “octahedral slips”.

\[
\frac{a}{2} \langle 011 \rangle \{111\} \tag{1.3}
\]

where \(a\) is the lattice parameter of the FCC cell.

The \(\gamma\) and the \(\gamma'\) phases have both FCC-like cells (disordered FCC structure for the \(\gamma\) phase and an ordered LI\(_2\) structure for \(\gamma'\) phase) thus potentially deform themselves by octahedral slips. There exist however several differences in \(\gamma\) and \(\gamma'\) deformation mechanisms. They mainly reside in the differences in their lattice parameters. Further details
are given in the following sections.

From a mechanical point of view, other slip systems can be activated in addition to the octahedral ones. These will be introduced in the following subsections. Generally speaking, a certain amount of energy has to be provided to produce a dislocation slip. According to the crystal plasticity theory (Mandel, 1973, Hill and Rice, 1972, Rice, 1975, Asaro and Rice, 1977, Asaro, 1983, Peirce et al, 1983), this means that a critical value of the shear stress has to be overcome on each slip system. The Schmid criterion (Schmid and Boas, 1950), provides an easy way to compute the actual shear stress on slip systems because it simply leans on geometrical considerations. However, from a micro-mechanical point of view, slips do not simply happen by dislocation motion on slip systems. They are rather the result of more complicated displacements and dislocation reactions. However, in many of the studies presented in section 1.5, experimental results are discussed based on Schmid criterion and crystal plasticity. These are then also cited in the following subsections. Further details are presented and discussed in the subsection 3.9.1.

\(\gamma\) deformation mechanisms – The shortest lattice vector on \{111\} closed packed planes is \(\frac{a}{2} < 011 >\). Since it is contained in \{111\} planes, the crystal deforms by \(\frac{a}{2} < 011 >\) \{111\} dislocations. The matrix then deforms by octahedral slips. During these slips atoms move to identical sites, therefore a perfect crystal structure and stacking sequence are left. \(\frac{a}{2} < 011 >\) \{111\} dislocations are then called “perfect dislocations” in the \(\gamma\) phase. According to Frank’s rule, the propagation of a single \(\frac{a}{2} < 011 >\) \{111\} is less favored than the propagation of two Shockley partials. The following dissociation is then possible

\[
\frac{a}{2} < 011 > \{111\} \rightarrow \frac{a}{6} < 211 > \{\overline{1}11\} + \frac{a}{6} < 12\overline{1} > \{\overline{1}11\} \tag{1.4}
\]

The passage of the first partial generates a fault in the ABCABCABC sequence of
staked \{111\} planes, extrinsic if it adds a plane (ABCACBCABCABC...), intrinsic if it remove a plane (ABCACABCABC...). This fault is removed by the passage of the second so that a perfect packing order is recovered. Thus, when the Shockley pair propagates, a perfect crystal structure is observed upstream and downstream while a stacking fault ribbon is observed between the propagating pairs (figure 1.6).

\[\gamma^\prime\] deformation mechanism – In the \(\gamma^\prime\) phase the shortest lattice vector is \(a < 001 >\) but it does not lie on \{111\} closed packed planes. Slips then happen along \(< 011 >\) directions. But for an Al atom to slip to another Al atomic site of the LI\(_2\) structure, twice the distance than in the matrix FCC cell has to be covered. This means that twice the energy is also required for the slip activation. What actually happens is that a coupled pair of \(\frac{a}{2} < 011 > \{111\}\) matrix dislocations enters the \(\gamma^\prime\) phase or that the \(a < 011 > \{111\}\) dissociates in slips requiring less energy to be activated. In both cases planar defects are produced.

The first case is observed at low temperatures. A pair of strongly coupled \(\frac{a}{2} < 0\bar{1}1 > \{111\}\) matrix dislocations enters the \(\gamma^\prime\) phase. The two perfect \(\frac{a}{2} < 0\bar{1}1 > \{111\}\) matrix dislocations are not perfect in \(\gamma^\prime\) because the Burger vector magnitude is half the length to displace an Al to another Al atomic site. Matrix perfect dislocations are then called “superpartial” when propagating in precipitates. A single superpartial causes a fault in the stacking sequence of \{111\} of the LI\(_2\) structure, thus the superpartial pair transports an Anti-Phase boundary. The \(a < 011 >\) dislocation is called “superdislocation”.

In the second case different types of dissociation take place depending on the amount of available energy, therefore on the temperature and on the applied stress (Pope, 1984). Some of these dissociation mechanisms involve slips on octahedral planes but along the \(< 112 >\) lattice vectors.

These slips are commonly called “secondary octahedral slips” (figure 1.7). According to the Schmid criterion, 12 secondary octahedral slips can be activated depending on the crystal direction solicited. Their activation has many consequences.
Figure 1.7 – Thompson tetrahedron representation of $<011\{111\}$ and $<122\{111\}$ slips in FCC crystals. (After Knowles and Gunturi (2002)).

First, $\frac{a}{6}<112\{111\}$ are involved in the Kear-Wilsdorf lock (Kear and Wilsdorf, 1962) mechanism which is responsible of the anomalous yielding stress peak at intermediate temperature (Sun and Hazzledine, 1988, Condat and D´escamps, 1987, Wang et al, 2009).

Figure 1.8 – After MacLachlan et al (2002): variation of the yield stress with temperature observed for the CMSX-10, CMSX-4, SRR99 and RR2000 alloys (tensile tests performed at $10^{-4}$s$^{-1}$).

Secondly, when a $\gamma'$ particle is sheared by superdislocations, the superpartials return to be perfect ones in the matrix once crossed the precipitates. On the contrary, $<112>$ slips shear the $\gamma'$ particles and the matrix as well, thus a deformation front spreads in the material causing higher deformation than the one produced by superpartials.

Finally, according to Kear and Giamei (1968) and Kear et al (1969), $\frac{a}{6}<\overline{1}12>$ displacements cause Al atoms to lie above Ni atoms and this alters the stacking sequence.

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
of \{111\} planes. Then a Complex Stacking Faults (CSF) forms. Moreover, \(\frac{a}{3} < 211 >\) displacements involve the formation of Superlattice Intrisic and Extrinsic Stacking Faults (SISF/SESF). While the anti-phase boundary is removed after the passage of the second superpartial, CSF, SISF and SESF are not removed. This is the reason why several authors consider the shearing of \(\gamma'\) particles by \(< 112 >\) slips as a damage source (MacLachlan and Knowles, 2000, MacLachlan et al, 2002).


As shown in figure 1.9, dislocations loops expand in matrix channels and deposit 60°segments at the matrix/precipitates interfaces. An interfacial dislocation network then forms, driven by the actual stress in the channels (see previous comment on the internal stress state resulting from the \(\gamma/\gamma'\) coherency).

![Figure 1.9 – Schematic representation of dislocation propagation in matrix channels: (a) propagation of a perfect dislocation loop (Fedelich, 2002) and (b) deposition of dislocation segments at the \(\gamma/\gamma'\) interface (Probst-Hein et al, 2001).](image)

Evidences of another type of slip system have been observed testing \(< 111 >\)-oriented crystals, the “cubic slips” (Equation 1.5 and figure 1.10) (Shah and Duhl, 1984, Pan et al, 1995, Kolbe et al, 1997). This is in agreement to the Schmid criterion which states that in crystals loaded along the \(< 111 >\) direction cubic slips are more likely than octahedrals. These slips were believed to be responsible for the tension/compression asymmetry of the response of Ni base Single Crystal Superalloys (Pan et al, 1995).

\[
\frac{a}{2} < 011 > \{001\} 
\]  
\quad (1.5)

However, TEM analysis performed by Völkl et al (1994), Bettge and Österle (1999) have revealed that no dislocation moves along cubic systems, neither in the matrix nor at the \(\gamma/\gamma'\) interfaces and that cubic slip is the result of the cross slip of the screw component.
of $\frac{4}{2} <011> \{111\}$ dislocations from a $\{111\}$ plane to another. The cross slip is produced when a screw segment is blocked by a $(100) \gamma/\gamma'$ interface. The constriction provides in fact the necessary amount of energy to cross slip on another $\{111\}$ plane. Macroscopic slip traces on $\{001\}$ planes are produced by the propagation of the repeated cross slips 1.11(b). The two trailing lines of the dislocation loop glide along $<011>$, shearing the material in parallel with $\{001\}$ planes.

Thus, properly cubic slips do not occur when mechanical loading is applied along the $<111>$ direction. Traces of this type of slip are the result of the cross-slip on octahedral planes. For this reason they are referred as “pseudo-cubic slips” in this study.

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
1.1.3 Structural hardening

Structural hardening is the result of the strengthening effect obtained by the presence of two phases material. A first hardening source is the difference between the elastic constants of the two phases, which produces the local coherency stresses. Coherency stresses contribute to increase the stress threshold to be overcome for a dislocation to move in the matrix. Other hardening effects are produced by the difference of the lattice parameters of two phases and by the ordering effect (Nathan et al., 1985, Pettinari et al., 2001, Zhang et al., 2004). In section 1.1.2, it has already been explained that shearing of \( \gamma' \) particles is much more energetically expensive than shearing the matrix. It was also pointed out that each type of dislocation decomposition and reaction involves the formation of high energy defects because \( \gamma' \) is an ordered phase. For these two reasons, unless very specific conditions occur, plastic deformation remains confined in the matrix and the mean free path of dislocations is limited by the matrix channels width. The precipitate shape, size and volume fraction also impact the alloy strength. Concerning the \( \gamma' \) shape, the cubic shape has been found to oppose the highest resistance to shearing (Lisiecki, 1992). As shown in Caron et al. (1986, 1988), Khan and Caron (1991), the onset of specific deformation mechanisms along each crystal orientation depends on the \( \gamma' \) size. For example for uniaxial loading along the \(<001>\) direction plasticity develops by matrix slips. Since having larger hardening particles also implies to have narrower matrix channels, more creep strength is generated. On the contrary, smaller \( \gamma' \) particles and wider \( \gamma \) channels promote the cooperative shearing of the \( \gamma/\gamma' \) microstructure and then the propagation of planar lattice faults. Moreover, as suggested by Mori and Tokushige (1977), dislocation climb on small particles is less energetically expensive.

Finally, single crystal superalloys have been developed so as to achieve an high \( \gamma' \) volume fraction. In this type of alloys the main hardening source consists in the existence of an additional stress (beside the critical shear stress required to move dislocations on slip systems) which has to be overcome for a dislocation loop to propagate in the matrix (Orowan, 1948). This stress is called Orowan stress, denoted \( \tau_{ORO} \) when expressed in terms of resolved shear stress, and depends on the matrix channels width:

\[
\tau_{ORO} \propto \frac{GB}{w}.
\]

In the equation (1.6), \( B \) is the Burgers vector magnitude of octahedral slips and \( w \) is the \( \gamma \) channels width. Since the Orowan stress depends on \( w \), the amount of Orowan hardening also depends on \( \gamma' \) morphology, volume fraction and size (Lapin et al., 2009).

An adapted aging heat treatment is performed in order to obtain the best hardening according to applications (Khan and Caron, 1991). High Pressure Turbine casting is aimed not to produce a misalignment higher than 15°between the \(<001>\) crystal orientation and the centrifugal axis of blades. The \(<001>\) orientation is chosen because this has the lower Young modulus and hence the best fatigue strength. Then, the microstructural parameters and the aging treatment are chosen to improve the creep strength along this direction. As shown by Caron and Khan (1983) the best \(<001>\) mechanical response is obtained with a medium precipitate size of 350 – 500 nm and, as shown by Murakumo
et al (2004), with a precipitate volume fraction of 60 – 70% at ambient temperature. The heat treatment sequence recommended for CMSX-4 by its producers is (Caron and Khan, 1983)

\[1140^\circ C/6h/AQ + 871^\circ C/20h/AQ\]

where AQ = Air Quench.

A slight microstructural inhomogeneity is observed between the dendrites and the inter-dendrites areas. They are a direct consequence of the solidification process. Since the inhomogeneity also concerns the chemistry more details are given hereafter.

### 1.1.4 Chemical composition

According to their alloying elements, single crystal superalloys are divided in generations. A quasi-exhaustive list of the existing superalloys is contained in Reed (2006). CMSX-4 belongs to the second generation of superalloys because it contains the 3 weight % of Rhenium (Harris et al, 1983). This alloy has been developed by the Cannon Muskegon corporation. Its nominal chemical composition is given in table 1.2. Example of alloys of other generations are given in the tables 1.1, 1.3 and 1.4.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Cr</th>
<th>Co</th>
<th>Mo</th>
<th>W</th>
<th>Al</th>
<th>Ti</th>
<th>Ta</th>
<th>Nb</th>
<th>Re</th>
<th>Ru</th>
<th>Hf</th>
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<td>10.0</td>
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<td>2.6</td>
<td></td>
<td>1.5</td>
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<td>C, B, Zr</td>
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Table 1.1 – Chemical composition (in weight%) of some common cast first generation Ni base Single Crystal Superalloys (Reed, 2006).

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Cr</th>
<th>Co</th>
<th>Mo</th>
<th>W</th>
<th>Al</th>
<th>Ti</th>
<th>Ta</th>
<th>Nb</th>
<th>Re</th>
<th>Ru</th>
<th>Hf</th>
<th>al&lt;0.2</th>
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<tr>
<td>CMSX-4</td>
<td>6.5</td>
<td>9.6</td>
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<td>6.4</td>
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<td>1.0</td>
<td>6.5</td>
<td>3.0</td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>Rene N5</td>
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<td>6.2</td>
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<td>3.0</td>
<td>0.25</td>
<td></td>
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</tbody>
</table>

Table 1.2 – Chemical composition (in weight%) of some common cast second generation Ni base Single Crystal Superalloys (Reed, 2006).

What has been tried to be obtained from one generation to another is mainly to increase creep resistance and then, from fourth generation alloys, to reduce microstructural instabilities.

For example:
- Cobalt has been added in order to reinforce the matrix;

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<table>
<thead>
<tr>
<th>Alloy</th>
<th>Cr</th>
<th>Co</th>
<th>Mo</th>
<th>W</th>
<th>Al</th>
<th>Ti</th>
<th>Ta</th>
<th>Nb</th>
<th>Re</th>
<th>Ru</th>
<th>Hf</th>
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<td>CMSX–10</td>
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<td></td>
<td></td>
<td></td>
<td>C, B</td>
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Table 1.3 – Chemical composition (in weight%) of some common cast third generation Ni base Single Crystal Superalloys (Reed, 2006).

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<th>Alloy</th>
<th>Cr</th>
<th>Co</th>
<th>Mo</th>
<th>W</th>
<th>Al</th>
<th>Ti</th>
<th>Ta</th>
<th>Nb</th>
<th>Re</th>
<th>Ru</th>
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<td>2.0</td>
<td>Hf</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 1.4 – Chemical composition (in weight%) of some common cast fourth generation Ni base Single Crystal Superalloys (Reed, 2006).

- Chromium has been added to harden the matrix and protect the alloy from hot corrosion;
- Tungsten and Molybdenum have been added to enforce the $\gamma/\gamma'$ solid solution and to improve the high temperature resistance;
- Ruthenium has been added in third generation superalloys to stabilize the chemical bounds with refractory elements preventing hence the precipitation of Topologically Closed Packed (TCP) phases (Pyczak et al, 2000, Rae et al, 2000b).

Rhenium has been added to second generation superalloys in order to reinforce the $\gamma/\gamma'$ interfaces, to algebraically decrease the negative mismatch between the two phases (its absolute value increases but it remains negative) and, above all, to slow down the kinematics of $\gamma/\gamma'$ microstructural degradation. Rhenium has in fact the lowest diffusion rate among all the alloy elements (Karunarate et al, 2000) and this helps to decelerate the diffusive phenomenon leading to the instability of the $\gamma/\gamma'$ microstructure and to the loss of the hardening properties described in the section 1.1.3. Microstructural instabilities are described in the section 1.2. The addition of elements causes several differences in the mechanical response of alloys belonging to different generations. The Rhenium concentration makes the CMSX-4 mechanical response different from the one of other generation alloys. In the following section this difference is highlighted when necessary. Comparisons between CMSX-4 and its Rhenium-free first generation version CMSX-2 can be found in (Blavette et al, 1988, Frazier et al, 1990).

A chemical inhomogeneity is usually observed between the dendrite and the inter-dendrites spacings (the dendrite/inter-dendrite structure of CMSX-4 is shown in figure 1.12). This is mainly due to the physics of the solidification process (Gell et al, 1980). During solidification dendrites form first. The chemical species required to form the $\gamma/\gamma'$ microstructure are then primary employed in the dendrite areas. Several matrix constitutive elements remains segregated in the inter-dendrites areas, as for example Al, Ti and Ta (Volek et al, 2005, Heckl et al, 2011a, Schulze and Feller-Kniepmeier, 2000), while W, Ma and Re remains segregated in the dendrites spacings. This also causes the microstructural inhomogeneity introduced in the previous section and, moreover, a difference of the
lattice misfit in the two areas (measurements performed on CMSX-4 are presented in Völkl et al (1998)).

Figure 1.12 – Dendritic structure of the CMSX-4 alloy observed by SEM after creep at 850°C and 400 MPa (test realized during the present Ph.D. study and presented in the section 2.4).

A solution heat treatment is performed on the as-cast material in order to dissolve all the γ′ particles and eutectic pools (Khan and Caron, 1983, Caron and Khan, 1999). The main aim of this solution treatment is to reduce as much as possible chemical heterogeneities across the dendritic structure without reaching incipient melting. Table 1.5 contains the description of the treatment suggested by Cannon Muskegon Corporation for the CMSX-4. However in second, third and fourth generation alloys, differences in content of Rhenium and Rheuthenium still exists between dendrite and interdendrite before the aging (Caron and Khan, 1999, Schulze and Feller-Kniepmeier, 2000). Moreover, as pointed out by Grosdidier et al (1998), Steuer et al (2014), attention has to be paid to the final cooling rate of the heat solution treatment, because this can also impacts the precipitates size.

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Duration</th>
</tr>
</thead>
<tbody>
<tr>
<td>1277 °C</td>
<td>2 h</td>
</tr>
<tr>
<td>+ 1288 °C</td>
<td>2 h</td>
</tr>
<tr>
<td>+ 1296 °C</td>
<td>3 h</td>
</tr>
<tr>
<td>+ 1304 °C</td>
<td>3 h</td>
</tr>
<tr>
<td>+ 1313 °C</td>
<td>2 h</td>
</tr>
<tr>
<td>+ 1316 °C</td>
<td>2 h</td>
</tr>
<tr>
<td>+ 1318 °C</td>
<td>2 h</td>
</tr>
<tr>
<td>+ 1321 °C</td>
<td>2 h/GFC</td>
</tr>
</tbody>
</table>

Table 1.5 – Steps of the solution heat treatment suggested by the Cannon Muskegon Corporation for CMSX-4 alloy. GFC = for Gas Furnace Cooling.

The dendrites and inter-dendrites difference in the content of certain chemical species has several consequences on the microstructural stability and on the material response. A
specific study on the fourth generation superalloy MC-NG has been carried out by Arnoux (2009). The above-mentioned differences concerning the present study will be mentioned in the sections 1.2.1 and 1.2.3.

1.2 Microstructural evolutions

Despite the hardening properties, a two-phase microstructure, rich in different alloy elements presents also a drawback. At high temperatures the hardening cubic microstructure evolves and it has several consequences on the mechanical response of the material at high temperatures. There are three main reasons why the microstructure evolves and consequently three main types of microstructural evolution: the $\gamma^\prime$-rafting, the $\gamma^\prime$-coarsening and $\gamma^\prime$ dissolution/re-precipitation. They are described in the following subsections.

1.2.1 $\gamma^\prime$-rafting

The $\gamma^\prime$-rafting is the consequence of a diffusive phenomenon which causes a redistribution of the chemical species. A non-zero system of internal stresses exists at the matrix/precipitates scale (section 1.1.1). When the equilibrium of the internal stresses is perturbed by the external loads, the potential of the chemical species changes. A stress controlled diffusive process initiates and the distribution of the alloy elements changes (whose global orientation depends on the sign of the effective (constrained) misfit $\delta_u$ (Carroll et al, 2008, Dirand et al, 2013)). The hardening species migrate from the most stressed channels to the less stressed until saturating them. Thus precipitates grow along the $<001>$ directions until becoming plate-shaped. As a consequence, the most stressed $\gamma$ channels close and the less stressed widen, meaning that the morphology of the initial hardening $\gamma-\gamma^\prime$ microstructure changes. A schematic representation of rafting in a $[001]$ crystal is shown in figure 1.13. An example for a CMSX-4 $[001]$ crystal (having a negative misfit) is given in figure 1.14.

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{rafting_schematic.png}
\caption{From Pollock and Argon (1994): schematic representation of the chemical species diffusion bringing to microstructural evolution during tensile creep along $<001>$ (case of $\delta_u < 0$).}
\end{figure}
Figure 1.14 – N-rafting along a [001] crystal of the CMSX-4 alloy. (a) In-plane crystal frame. SEM observation and schematization of (b) the cubic initial microstructure and (c) of the rafted microstructure after 101h of creep at 1050°C and 140 MPa. Test realized during the present Ph.D. study and presented in the chapter 2

Rafting has been observed many times on blades after service (Draper et al, 1989, Epishin et al, 2008, Biermann et al, 1996), in many single crystal superalloys during creep (Tien and Copley, 1971, Tien and Gamble, 1972, Mackay and Ebert, 1985, Caron et al, 1988, Khan and Caron, 1991, Henderson et al, 1998, Pollock and Argon, 1994, Reed et al, 1999, Gaubert, 2009, Ghighi, 2013) as well as in polycrystalline aggregates having an high volume fraction of precipitates (Altincekic and Balikci, 2014). For example, it has been observed in both the polycristalline and the single crystal version of Udimet 700 (Sullivan et al, 1968, Tien and Copley, 1971, Tien and Gamble, 1972), in the first case only in several grains, in the second in the whole volume of the material. In the following paragraphs the factor affecting the γ′-rafting kinetics will be detailed.

Temperature— The temperature is a fundamental element in the rafting onset and kinetics, because diffusion is thermally activated. For this reason γ′-rafting is generally observed at temperatures in excess of 900°C (Caron et al, 1988, Sass et al, 1996, Henderson et al, 1998, Matan et al, 1999a). With the increase of temperature diffusion develops faster and a more rapid γ′-rafting completion is observed (Matan et al, 1999a).

The alloy chemistry— As shown by Giamei and Anton (1985), Pyczak et al (2005), Reed et al (2008), Kuznetsov et al (2015), the alloy chemistry also affects the rafting kinetics. Rhenium and Ruthenium promote more microstructural stability, since they decrease the lattice mismatch in algebraic value. Moreover, since Rhenium has a low diffusion rate, it retards the γ′-rafting completion. An example is given in (Reed et al, 2007a, Arnoux, 2009), where a lower rafting kinetics is observed in the dendritic spacing.

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The reson is that, as mentioned in the section 1.1.4, Rhenium increases the absolute value of the (negative) misfit and this increase involves a slower $\gamma'$ rafting kinetics.

**The stress state at the matrix/precipitates scale**— The final morphology of the microstructure depends on the mass transport direction. This therefore depends on the actual stress state at the matrix/precipitates scale. For example, as observed by Serin et al. (2004), after a double shear creep test a rafted microstructure inclined of $45^\circ$ within the loading axe is observed, while after a tensile creep test the rafted particles are perpendicular to the loading direction. The stress state at the matrix/precipitates scale is mainly the result of the superposition of external loads and coherency stresses. For this reason the morphological evolutions also depend on many other factors, listed in the following paragraphs.

**Different elastic constants between the two phases**— As shown by Pineau (1976), since the coherency stresses are the result of the accommodation of the elastic strain of the two phases, the difference between the elastic constants of the two phases influences the rafting kinetics.

**Stress sign**— Different final microstructures have been observed after tensile and compression creep tests (Tien and Copley, 1971, Miyazaki et al., 1979). Most of commercial Ni base Single Crystal Superalloys used for gas turbine applications have a negative $\gamma - \gamma'$ lattice mismatch at high temperatures (as explained in section 1.1.1). This implies that, at zero external load, precipitates are under biaxial tension while matrix channels are in compression.

During tensile creep tests along the $[001]$ crystallographic direction the stresses in the vertical channels relax while the stresses in the horizontal channels become more severe. The $\gamma'$ elements migrate from the horizontal to the vertical channels until these saturate (Pollock and Argon, 1994, Kamaraj et al., 1998). $\gamma'$ particles grow along the $[010]$ and $[100]$ directions, thus $\gamma'$-plates develops perpendicularly to the load direction.

On the contrary, during compression tests along the $[001]$ crystallographic direction, the mass transport follows the opposite direction. Horizontal channels close and verticals widen. As a result, $\gamma'$ particles coalesce parallel to the loading direction.

When rafting develops normally to the loading direction it is referred as “N-rafting” (Normal rafting, figure 1.15) while it is referred as “P-rafting” (Parallel rafting, figure 1.15) in the compression case (Fredholm and Strudel, 1984).

The stress sign influences the microstructural evolution also under stress reversal. As shown by Xingfu et al. (2009), Ott and Mughrabi (1999), Mughrabi and Tetzlaff (2000), Giraud et al. (2012) if first a compressive and then a tensile creep load are applied along a $[001]$ direction, first a P-type rafting develops and then, during the stress reversal, the former P-type rafting breaks and progressively a N-type rafted microstructure is formed.

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Lattice mismatch – Several alloys have a positive $\gamma/\gamma'$ lattice mismatch. This is for example the case of Cobalt based superalloys, as for example 9W-2Ta-0.0813 (wt %) (Bauer et al, 2012, Pyczak et al, 2013), therefore the internal stress state at the precipitates/matrix scale is the opposite of the one of Ni base superalloys: matrix channels are under tension while precipitates are subjected to biaxial compression. Tensile and compression loads thus have the opposite effect compared to Ni base superalloys in this class of LI$_2$ strengthen Co-base alloys. P-rafting is observed during tensile creep along $<001>$ while N-rafting is observed during compression creep along $<001>$ (Pollock and Argon, 1994).

Crystal orientation – The influence of crystal orientation (compared to loading direction) relies on the different ways in which the equilibrium of internal stresses is perturbed and on the resulting plasticity development in the matrix channels.

During creep tests along the $<111>$ crystallographic direction, the three matrix channels are symmetrically loaded. For this reason diffusion does not have a preferential direction. The $\gamma'$ precipitates and the matrix channels grow symmetrically along the three $<001>$ directions thus precipitates initially remain almost cubic (Peng et al, 1996, Sass et al, 1996). At the end of creep, the evolved 3D $\gamma/\gamma'$ microstructure appears roof-shaped (figure 1.16(a)) (Golubovskii et al, 1987, Khan and Caron, 1991). Since this process involves a homothetic growth of cubic precipitates and of the $\gamma$ channels, in the present work this evolution is called “mechanical coarsening”. Similarly, [011] crystal direction is an in-plane crystal symmetry direction. During tensile creep along the [011] crystallographic direction the two matrix channels parallel to [010] and [001] are equally stressed. Even in this case, the final microstructure after tensile creep test along the $<011>$ direction, ap-
pears roof-shaped, but only in \{100\}-type planes (figure 1.16(b)) (Agudo Jácome, 2013, Ghighi, 2013).

![Figure 1.16](image1.png)

Figure 1.16 – From (Khan and Caron, 1991): SEM observation of the AM3 alloy after creep at 1050°C and 120 MPa 1.15(a) along \[011\] and 1.15(b) along \[111\].

Misalignements with respect to the perfect \(<111>\) and \(<011>\) orientations cause a loss of load symmetry. In this case, there exists a preferential \(<001>\) growing direction. The rafted \(\gamma'\) phase results plate-shaped and inclined within the load direction (figure 1.17) (Peng et al, 1996, Han et al, 2010, Chatterjee et al, 2010, Ghighi, 2013, Yu et al, 2013). Similar rafted microstructures have been observed after creep along \(<102>\) and \(<112>\) by Ghighi (2013), Yu et al (2013).

![Figure 1.17](image2.png)

Figure 1.17 – Microstructure of the SRR99 alloy observed along the \[011\] crystallographic direction after tensile creep at 1040°C and 150 MPa (by Han et al (2010)).

**Plasticity** – Segments of dislocations gliding in the matrix channels are deposited at \(\gamma/\gamma'\) interfaces (Carry et al, 1981). The interfacial dislocation network relaxes the coherency stresses between the two phases in different ways in the horizontal and vertical channel, contributing to the stress difference between the channels. As shown by Matan
et al (1999a), the rafting process can then continue even at zero stress if enough plastic deformation at high temperature (i.e. T > 900°C) has been produced.

### 1.2.2 $\gamma'$ load free coarsening

The load free coarsening of $\gamma'$ particles consists of the homothetic growing of precipitates along $<001>$ directions because of a spontaneous Oswald-ripening phenomenon (Ostwald, 1897). The material is in fact a heterogeneous Nickel solid solution, where $\gamma/\gamma'$ interfaces have an energetic cost. In addition, even if the material is subjected to specific aging treatments to homogenize and optimize the size of precipitates (see section 1.1.3), in the final microstructure slight differences in $\gamma'$ sizes still exist. As the existence of larger particles is more energetically favored than the existence of smaller ones, a spontaneous diffusion process following the LSW theory (Lifshitz and Slyozov, 1961, Wagner, 1961) takes place and larger particles grow at the expense of the smaller ones. At constant volume the homothetic growth of $\gamma'$ particles causes a homothetic growth of $\gamma$ channels too. This is a low, spontaneous phenomenon, independent from mechanical loads and enhanced by temperature. At higher temperature the LSW kinetics is accelerated but precipitates size variation remain limited with respect to the one caused by the $\gamma'$-rafting.

### 1.2.3 $\gamma'$-dissolution

The third type of evolution depends on the tendency of the precipitate phase to dissolve into the matrix. This type of dissolution causes a decrease of the volume fraction of precipitates (Nathan et al, 1985, Diologent et al, 2002, Cormier, 2006, Reed et al, 2007a).

The dissolution is a thermally activated phenomenon and its kinetics is also governed by temperature and by the prior thermo-mechanical history (Giraud et al, 2013b).

Under constant temperature exposure, for a sufficiently long time, the $\gamma'$ phase reaches the thermodynamics equilibrium, meaning that the $\gamma'$ volume fraction does not evolve anymore. The value of the precipitates volume fraction at the thermodynamics equilibrium (denoted $f'_{\gamma'\text{eq}}$) varies depending on the alloy chemistry, as it can be seen for example by comparing the value measured for the AM1 and MCNG by Diologent et al (2002), for the MC2 by Cormier (2006) and for the CMSX-4 by Reed et al (2007a).

As for the $\gamma'$-rafting, chemical inhomogeneities between dendrites and inter-dendrites involve differences in the $\gamma'$ dissolution kinetics between these genes (figure 1.18) (Burgel et al, 2000).

The studies contained in the works of Cormier (2006), Cormier et al (2007a,b), le Graverend et al (2011) have highlighted that the kinetics of $\gamma'$-dissolution is highly sensitive to temperature variation and thermal rate. Both accelerate the dissolution process, especially when the constant temperature phases are short and when the temperature is larger than 1000°C. In detail, during fast thermal variation, $\gamma'$-dissolution does not reach any thermodynamics equilibrium state. Then, if the overheating is followed by a fast cooling, the dissolved hardening elements recombine and a second smaller population of
precipitates phase appears (Cormier et al, 2007b). These hyper-fine precipitates are often called tertiary precipitates ($\gamma''_t$). Figure 1.19 shows the microstructure observed after an overheating of 30s at 1200 °C. $\gamma''_t$ particles have a cubic form because, as the secondary $\gamma'$ phase, they have to be coherent with the matrix (Xiang et al, 2016).

As shown by Giraud et al (2013b), plasticity also plays a major role in the dissolution kinetics. This is related to the density of dislocation at the $\gamma/\gamma'$ interfaces, which promotes a “pipe diffusion phenomenon” (Legros et al, 2008, Kontis et al, 2018).

The stress magnitude and the crystal orientation do not impact the $\gamma'$ dissolution kinetics.

### 1.2.4 Topological inversion

The topological inversion consists in a destabilization of the rafted microstructure. The $\gamma'$ rafted precipitates progressively evolve toward a situation where they completely surround $\gamma$ phase particles. The $\gamma'$ phase becomes then the connected phase. This further microstructural evolution has been observed during creep of CMSX-4 (Mughrabi, 1996, Epishin and Link, 2004), of many other alloys (Fredholm and Strudel, 1987, Caron et al, 2000, Epishin et al, 2001, Diologent et al, 2002, Epishin and Link, 2004) and in a wide temperature/stress range. Studies carried out by Fredholm and Strudel (1984), Caron et al (2008) have shown that the topological inversion occurrence is closely linked to the volume fraction of precipitates $f_{\gamma''_{eq}}$ at a given temperature. As explained in the subsection 1.2.3, the tendency for $\gamma/\gamma'$ inversion decreases with temperature. Caron et al (2008) have observed that the necessary condition for the inversion onset is that $f_{\gamma''_{eq}} \approx 50\%$. Then, the higher the fraction of $\gamma'$ phase is, the earlier the inversion occurs during the creep life. In the same study the relation between the topological inversion and the volume fraction of precipitates has been explained considering that the reduction of the interfacial energy.
acts as the inversion driving force. Similarly to what happens during coarsening, as the precipitates grow, the interfaces tend to migrate in order to minimize the elastic energy. A further detailed study on the inversion kinetics can be found in Epishin et al (2001). This microstructure evolution has been observed to lead to a creep strain rate increase (Epishin et al, 2001, Matan et al, 1999a), as such, after considered as a damage mechanism in creep. Same attempts have been made to account for its consequence on the creep rate, as for example in Milhet et al (2012), Matan et al (1999a). It will also be taken into account in this work by means of a damage law. Details are presented in the chapter 4.

1.3 High temperature response along $<001>$

At temperatures higher than 900 °C, microstructural evolution takes place, causing the progressive widening of $\gamma$ channels and therefore a loss of the hardening properties described in the section 1.1.3. The impact of microstructural evolution on the creep and monotonic tension responses is described below.

1.3.1 Creep

During high temperature exposure and at constant mechanical load in $<001>$ direction, the $\gamma'$ dissolution and $\gamma'$ rafting take place at the same time. In the primary creep stage the density of matrix dislocations increases. Plasticity develops by dislocation glide in the matrix channels, following the mechanisms described in the section 1.1.2. At the same time, the $\gamma'$-rafting and the dissolution process starts. As pointed out by Carry and Strudel (1977), Benyoucef et al (1995), Pollock and Argon (1994), the widening of $\gamma$-channel causes a decrease of the Orowan stress and a progressive softening of the visco-plastic response. Meanwhile, strain hardening is produced by two phenomena:
— the progressive formation of a stable dislocation networks at the $\gamma/\gamma'$ interfaces which relaxes the coherency stresses (Probst-Hein et al, 1999, Carry et al, 1979, Field et al, 1992, Tian et al, 2000, Zhu et al, 2012, Zhang et al, 2002);
— the closure of the vertical channels, which blocks the dislocation climb around precipitates (Reed et al, 1999).

The first hours of a creep test— in vertical $<001>$ direction— are then characterized by a competition among:

- the plasticity produced by the increase of mobile dislocation density;
- the softening due to the widening of $\gamma$ channels;
- the hardening produced by the formation of stable dislocation networks at the $\gamma/\gamma'$ interfaces;
- the hardening produced by the closure of vertical $\gamma$ channels.

According to Giraud et al (2013b), dissolution is accelerated during primary creep because of the increasing density of interface dislocations. When interfaces saturate, the dependence on plasticity disappears and the precipitates volume fraction continues to evolve only because of temperature. Once the thermodynamic equilibrium is reached, the volume fraction does not evolve anymore. Further $\gamma$ channels widening only depends on $\gamma'$-rafting. A study on the decrease of the volume fraction of precipitates during the first stage of isotherm creep can be found (Cormier et al, 2008). Figure 1.20 shows that at 1200°C the $\gamma'$ volume fraction decreases linearly during the firsts 5 minutes on of creep, with a slope which increases (its absolute value increases) with the stress.

![Figure 1.20 - After Cormier et al (2008): $\gamma'$ dissolution during the first 5 minutes of isotherm creep at 1200°C as a function of time and of the applied stress.](image)

As pointed out by Mackay and Ebert (1985), Nathan et al (1985), Diologent et al (2002), $\gamma'$-rafting completion has also been generally observed at the end of primary creep. Since $\gamma'$-coarsening kinetics is lower compared to $\gamma'$-rafting, the coarsening kinetics is negligible before the $\gamma'$-rafting completion.

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Figure 1.21 – From (Diologent et al, 2002): microstructural evolution observed during creep along $<001>$ of the AM1 alloy at 1050 °C and 150 MPa.

Depending on the temperature, on the load magnitude and on the crystal orientation, the plasticity development in the matrix channels is more or less rapid and so the rafting process is. There also exist situations during which the classical primary creep stage is not observed. Further details are given in the section 1.5.1 (and in next chapters). An initial negative creep deformation is sometimes observed as well. This depends on the contraction generated by vertical interfaces rearrangement in the first stage of precipitate evolution (Louchet, 1995).

Secondary creep is characterized by a steady strain rate. Since $\gamma'$-coarsening is a slow phenomenon, the microstructure does not evolve significantly. Plasticity is produced by a combination of dislocation glide in the matrix channels and climb around precipitates (Carry and Strudel, 1978, Pollock and Argon, 1994, Svoboda and Lukas, 1997, Epishin and Link, 2004, Zhu et al, 2012).

Tertiary creep consists in a strong strain acceleration. Recently, le Graverend et al (2017) have shown that the onset of tertiary creep does not depend on the plastic damage but rather on the softening produced by microstructural evolutions. This study has shown that no correlation between the growth of the pores volume fraction and the plastic strain acceleration exists until the final phase of tertiary creep and that, therefore, the first acceleration is just caused by the $\gamma$ channels widening and destabilization of the $\gamma/\gamma'$ microstructure (i.e. the $\gamma/\gamma'$ topological inversion). An example is given in figure 1.22.

Then, during tertiary creep, the growth of $\gamma$ channels continues slowly. An additional acceleration of the plastic strain is produced by the shearing of $\gamma'$ particles (Sugui et al, 2000, Link et al, 2005, Sarosi et al, 2007), by plastic damage (Link et al, 2006, Reed et al, 2007b) and, at the higher temperatures, by oxydation. At this point, if the $\gamma'$ volume fraction at the testing temperature is higher than 50%, the microstructure becomes topologically inverted (see section 1.2.4). As explained in Caron et al (2008), topological
Figure 1.22 – Comparison between the plastic strain rate evolution (blue rectangular-shape markers) and the volume fraction of pores (red diamond-shape markers) as a function of time during creep at 1100°C and 180 MPa. Measurements performed by ex-situ X-ray computed tomography and presented in (le Graverend et al, 2017). Inversion strongly contributes to the total strain acceleration. The matrix plastic deformation leads to an increase of the internal stresses but, since these cannot be anymore relaxed by the glide and climb around precipitates, the $\gamma/\gamma'$ interface dislocations promote the shearing of the $\gamma'$ particles.

### 1.3.2 Monotonic tension

Diologent et al (2002), Gabrisch et al (1994), Cormier (2006), Tinga (2009a), Giraud (2013a), Lapin et al (2013) have shown that at temperatures higher than 900°C, the $\gamma'$-rafting has a detrimental effect on the monotonic tension response at low strain rates (i.e. $\dot{\varepsilon} \leq 10^{-5} \text{s}^{-1}$). The reason is that in these conditions the tension tests are long enough to induce microstructural evolutions. A gradual softening is observed even before the onset of plastic damage (figure 1.23 give true (Cauchy) stress vs true (logarithmic) strain curves). The latter –enhanced by necking– contributes to the material degradation only in the last stage of the test.

As the strain rate increases, i.e. $\dot{\varepsilon} > 10^{-5}\text{s}^{-1}$ for instance, the test duration decreases. The high temperature exposure is then not long enough for the onset of the microstructural evolutions. In this case samples deform more, with a softening that only depends on necking and plastic damage.

In the works of Gabrisch et al (1994), Giraud (2013a), a higher ductility has been observed during high strain rate tests compared to low strain rate tests (figure 1.23(a) and 1.23(b)). This relies on the fact, that at the higher strain rates, the microstructure does not evolve thus softening is just produced by damage.

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A similar macroscopic behavior has been also observed by Cormier (2006) in MC2 alloy, by Steckmeyer (2012) on ferritic steel, by Dade (2015) in nano-enforced alloys of fourth generation and by Praud et al. (2012) in a Fe-14%Cr ODS alloy.

1.4 Non-isothermal loading conditions

Pioneering works focused to analyze the mechanical response under non-isotherm loading were performed by Rowe and Freeman (1956, 1957, 1960), Weiss et al (1957a, 1975b) on many polycrystalline superalloys and by Cailletaud (1979) on IN100. Further
Figure 1.24 – Microstructure of the SC16 alloy observed by Gabrisch et al (1994) after a tensile test (a) at $\dot{\varepsilon} = 10^{-6}$ and (b) at $\dot{\varepsilon} = 10^{-3}$.

Systematic studies have been more recently carried out for Ni-based superalloys:

In each case microstructural evolutions have been found to govern the mechanical response. Coarsening does not play a major role during thermal cycling because, as it is shown in the following paragraphs, precipitates rafting and dissolution are dominating.

**Non isothermal creep** – During non isothermal creep tests, the impact of an overheating simulating the One Engine Inoperative condition has been investigated. $\gamma'$-dissolution and re-precipitation have been found to be the cause of the rapid increase of the plastic deformation during overheating and of the hardening observed after. According to Kakhei (1999), the appearance of the $\gamma''_I$ modifies the driving deformation mechanism during creep. As shown by Cormier (2006), the $\gamma''_I$ harden the alloy’s response by providing additional obstacles to the dislocation motion in the $\gamma$ channels. At the same time, when the thermal shock is followed by isothermal creep, Oswald-ripening promotes the dissolution of the finer precipitation and hardening slowly saturates. As a result, a new primary creep phase is observed after the thermal shock (see figure 1.26(b)). At the same time, during the high temperature peak, a dynamic recovering mechanisms reduces the interface dislocation density (figure 1.25). This recovering is responsible of the “beneficial effect” that longer overheating have on the creep lifetime of MC2 alloy, (figure 1.26(b)) but it is also of the higher rupture elongations. The study performed by Cormier et al (2010) has evidenced that micro-rafted $\gamma''_I$ particles can form in the matrix.

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channels of a rafted microstructure during high temperature excursion. As shown in figure 1.27, different plastic strain are produced depending on the $\gamma''_{II}$ morphology. As for the $\gamma'$ phase, cubic $\gamma''_{II}$ precipitate interfere with dislocation motion more than the $\gamma''_{II}$-rated particles.

Figure 1.25 – From Cormier et al (2007b): schematic representation of the $\gamma'$ dissolution and re-precipitation produced by performing an overheating on a rafted microstructure. A) initial dislocation network in the matrix channels, B) widening of matrix channel caused by the dissolution during the overheating and C) precipitation of the finer $\gamma''$ phase.

Thermal cycling with low temperature thermal excursion – Thermal cycling with minimal temperature of 400 °C and maximum temperature level of 1150 °C have been performed by Raffaitin et al (2007), Goti et al (2012) in order to simulate the temperature variation on the upper part of uncooled blade profiles. According to what observed in the previously described study, the $\gamma'$ dissolution on the rafted microstructure has been found to be responsible of the strain rate increase observed after each thermal transitory phase, while the $\gamma''_{II}$ precipitation and re-dissolution has been found to characterize the hardening at the beginning of the stationary temperature stages. Figure 1.28(a) shows the temperature cycles used to test the MC2 response by Goti et al (2012), the resulting deformation curves (figure 1.28(b)) and the microstructure observed after the cycle b (1.28(c)). Moreover, since the coupling of $\gamma'$-rafting and dissolution accelerates the plastic deformation after each transient stage, the temperature cycle frequency has also been found to have a detrimental effect on the mechanical response (figures 1.28(a) and 1.28(b)).

Thermal cycling with high temperature thermal excursion – Thermal cycling including 1 minute stages at 1100 °C and at 1150 °C have been performed by Graverend et al (2010), Cormier et al (2010), Giraud et al (2012), Ghighi (2013), Cormier (2006). In any cases $\gamma'$-rafting and the $\gamma''_{II}$ precipitates have been observed on post-mortem SEM images of the final microstructure. Rupture has not revealed the presence of any specific damage mechanism, therefore microstructural evolutions have been found to be responsible of the high creep strain rate observed. In a consistent manner with the already cited studies a plastic deformation jump with increasing amplitude has been observed after each high temperature change (Ghighi (2013), figure 1.29).

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Strain jumps have also been observed by Giraud (2013a) on CMSX-4. In this case the increase of the strain jumps amplitude has been found to be less pronounced than the ones observed by Ghiggi on MC2, probably because of the additional microstructural stability provided by Rhenium and by the higher volume fraction of precipitates contained in the CMSX-4 compared to MC2 in the investigated temperature range. Recently, similar tests have been performed by Cormier (2016) to highlight the chemistry influences on the alloy mechanical response and to provide guidelines for new alloys development. It was confirmed that, since the $\gamma'$ dissolution and the rafting are influenced by the alloy composition, alloys of different generations perform differently during thermal cycling.

An experiment reproducing a 150h certification engine test of helicopter engines has also been carried out in Mauget (2012), Ghiggi (2013). This test has been performed on the MAATRE burner rig (see Appendix A for more details on experimental rigs). A serie of repeated thermal shocks simulating the One Engine Inoperative condition have been included in the thermal path. As shown in figure 1.30, a high strain rate acceleration has been observed in this stage of the test, suggesting that thermal shocks promote
Figure 1.27 – Plastic strain jumps observed because of 1 minute of dwell excursions at 1150°C and 1100°C applied during non-isotherm creep of MC2 alloy at 1050°C and 120 MPa (a): comparison between measurements performed on a specimen having an initial rafted microstructure (b) and on a specimen having an initial cubic microstructure. A directional coalescence of $\gamma''$ particles is observed in the first case.

the transition from secondary to tertiary creep. Evidences of the finer precipitation in the $\gamma$ channels of the rafted microstructure have also been found on post-mortem SEM observations, suggesting that both $\gamma'$-rafting and dissolution take part in the strain rate acceleration. Anyway, further investigations would be necessary to quantify the respective roles of microstructural evolutions and of plastic damage during repeated thermal shocks.
Figure 1.28 – Thermal cycling test conducted at constant stress on the MC2 alloy by Goti et al. (2012): (a) schematic representation of thermal cycles, (b) deformation curves and (c) appearance of the finer precipitate phase $\gamma''$ during the cooling phases of a thermal cycling test at 1150 °C (SEM observation carried out by Viguier et al. (2011)).

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Figure 1.29 – Thermal cycling test performed by Ghighi (2013) on the MC2 at constant stress: (a) schematic representation of the thermal cycle and (b) the measured plastic deformation.
Figure 1.30 – After Mauget (2012): (a) schematic representation of the thermal load and (b) measured plastic deformation during a 150h engine type test performed on MC2 alloy.
1.5 Anisotropy of the creep response

Considering the stress-temperature couples at whom a significant inelastic creep deformation is observed in single crystal superalloys (figure 1.31), the typical stress-temperature solicitation of blades profiles and the deformation mechanism governing the creep response, two domains can be distinguished:

- **High temperatures and low/intermediate stresses**, \( T \geq 900 \degree C \) and \( \sigma \leq 300 \text{ MPa} \);
- **Low temperatures and high stresses**, \( 750 \leq T \leq 850 \degree C \) and \( \sigma \geq 600 \text{ MPa} \).

![Figure 1.31](image)

Figure 1.31 – From (Reed, 2006): typical evolution of the yield stress defined at the 0.5% of plastic strain as a function of temperature. The dashed area correspond to the possible evolution of the yield stress decreasing the strain rate.

Details on the temperature, stress magnitude and crystal orientation dependency of the creep response are given in the following section for each domain.

1.5.1 High temperatures and low/intermediate stresses

Several studies have shown that at high temperatures and low stresses, the anisotropy of the creep response of single crystal superalloys tends to decrease (Caron et al, 1988, Sass et al, 1996, Matan et al, 1999b, Liu et al, 2008, Han et al, 2010, Segersäll et al, 2014) until disappearing at \( T > 1100 \degree C \) (Hana et al, 2010, Mataveli Suave et al., 2016).

This isotropisation of the visco-plastic properties is due to the fact that along all the crystal orientations and at all the temperature/stress couples the creep response is mainly influenced by the decrease of the volume fraction of precipitates and by multiple slips in the matrix in the whole high temperature domain. Thus, for this reason, creep curves often present the same shape at a given temperature-stress condition (Ghighi, 2013) (figure 1.32).

Slight differences in creep lifetimes, rupture elongations and steady state strain rates are however observed according to the temperature, the stress and the crystal orientation tested.
Temperature accelerates the microstructural evolution by accelerating diffusion and dissolution rates (as it has been highlighted in the section 1.2.1 and 1.2.3). Thus the $\gamma'$-rafting completion and the dissolution thermodynamic equilibrium are attained faster at the highest temperatures.

The stress magnitude mainly affects the primary and secondary creep phases. There exists cases where no classical primary and secondary creep stages are observed. Examples are shown in figures 1.32 and 1.33(a). An “incubation” phase is observed in the first hours of creep (Khan and Caron, 1991, Reed et al, 1999). The microstructure stays cubic until $\gamma'$-rafting takes place. Then, $\gamma'$-rafting starts. The rafting kinetics results accelerated when high stresses are applied, involving a rapid strain rate acceleration and the absence of a steady-state rate stage. At lower stresses no incubation phase is observed. Strain hardening prevails on the $\gamma'$-rafting kinetics and a primary creep phase is observed (Reed et al, 1999). An example is given in figure 1.33(b) for the CMSX-4 alloy. In this case the end of the primary creep corresponds to the $\gamma'$-rafting completion (as also evidenced by Diologent et al (2002) for the AM1 alloy) and it is followed by a classical secondary creep stage.

The type of activated slips, the mechanisms of slip propagation in the matrix and the morphology of the final microstructure all depend on the crystal orientation.

For instance, as observed by Pollock and Argon (1994), Kamaraj et al (1998), Reed et al (1999), during creep along crystals close to the $<001>$ theoretical orientation multiples octahedral slips (see section 1.1.2) propagate in the matrix channels. Hardening is mainly produced by the mechanism described in the section 1.3. At the same time $\gamma'$ dissolution and N-rafting cause a widening of the matrix channels and reduce Orowan hardening.

Crystals having high misalignments within the $<001>$ direction deform by a combination of octahedral and pseudo-cubic slips (Bettge and Österle (1999), sections 1.1.2 and 1.1.2), with a predominance of pseudo-cubic slips along $<111>$-like crystals.

Concerning the microstructural evolution, crystals oriented close to the $<111>$ theoretical direction present a growth of the cubic particles 1.2.1. Since the widening of the
Figure 1.33 – Creep response of the CMSX-4 alloy observed by Reed et al (1999) at (a) 950°C 185MPa and (a) 1150°C 100 MPa.

γ-channels produced by the growth of cubic particles is lower than the one produced by an N-rafted microstructure and since more strain hardening is produced because of the existence of multiples γ/γ’ interfaces, lower strain rates and longer lifetimes are observed along these crystals (Golubovskii et al, 1987). Contrarily, when the misalignment is such that plate-shaped particles inclined of angle 45° within the loading direction form, more softening and less strain hardening are produced. Faster strain rates and shorter lifetimes are then observed.

As described in the section 1.2.1, the effect of misalignments within the theoretical <011> direction on the microstructural morphology are similar to the ones observed during creep along <111> misoriented specimens (Chatterjee et al, 2010). The main difference between <011> and <111> behaviors is that, in the first case, the roof structure grows along two <001> directions, while in second case the it grows along three <001> directions. In addition to this, when loading an <011>-like orientation, different octahedral and matrix slips are activated, also depending on the misalignment within the theoretical <011> (Ghosh et al, 1990). Furthermore, the crystal lattice is unstable when loading along <011>. Thus, the response along this crystallographic orientation is also impacted by the crystal lattice rotation.

The creep response along quasi <112> and <102> crystals also strongly depends on the misalignment within the theoretical <112> and <102> orientations. Different misalignements involve in fact the activation of different slip number and types in addition to different morphology of the evolved microstructure. Further details about the creep response along these two direction can be found in Ghighi (2013) and Yu et al (2013).

Concerning misaligned crystals, another factor impacts the creep response of the material: the crystal lattice rotation. Crystals misaligned within the <001> and <111>
theoretical directions present an unstable crystal lattice which tends to rotate toward the
direction of the Burger vector of the activated prevalent slip (Taylon, 1934a, Taylor,
1934b). At high temperatures, multiple matrix slips govern the deformation, thus the
lattice rotation is observed almost at the end of creep tests and close to stress concentra-
tion and to high stress triaxiality zones (Staroselsky et al, 2010). In this case the rotation
results in a change of the activated matrix slips and in a different orientation of the rafted
particles close to the rupture (Ghighi, 2013, le Graverend, 2013). These local changes
can produce localized softening or hardening zones that play an important role in the final
creep deformation and lifetime.

1.5.2 Low temperatures and high stresses

Many studies have shown that the creep response of single crystal superalloys at low
temperatures and high stresses is very sensitive to:
— the crystal orientation (Leverant and Kear, 1970, Leverant et al, 1973, Mackay
et al, 1980, Mackay and Maier, 1982, Caron et al, 1988, Ardakani et al, 1998,
Kakhei, 1999, Gunturi et al, 2000, Rae et al, 2000a, Rae and Reed, 2007, Barba et
al, 2017);
— the stress magnitude (Sass et al, 1994, 1996, Matan et al, 1999b, Rae and Reed,
2007);
— the alloy chemistry (Giamei, 1979, Caron et al, 1988, Rae et al, 2003).
Further details are presented in the next paragraphs.

Crystal orientation

The crystal orientation dependency of the creep behavior of Ni-based single crystal
superalloys has started to be investigated for MAR−M200 by Leverant and Kear (Lever-
Mackay and Maier (1982). Primary creep strains and secondary creep rates were found
to increase in the following order:
  – tensile axe misaligned within to the [001] − [0T1] boundary, especially close to the
theoretical [0T1] orientation;
  – tensile axe lying between the [001] − [0T1] and the [001] − [1T1] boundaries;
  – tensile axe lying close to the [001] − [1T1].
The creep lifetimes increase in reverse order of creep rates.

Secondary octahedral slips and stacking fault propagation (figure 1.34(d)) have been
found to be responsible for the high primary deformations and for the short lifetimes.
The observed anisotropy has been then rationalized on the base of the Schmid criterion
(section 3.9.1) (Mackay and Maier, 1982) (figure 1.36 and 1.35). The shortest lifetimes
and the most extended primary creep stages have been observed during creep for crystals
favorably oriented for single slip. Along these crystals poor strain hardening is produced,
because few slips are activated. Hence, hardening have been related to the amount of
rotation required to observe intersecting slips.

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature:
a tensorial microstructure-sensitive approach
An incubation period was observed during several creep tests, as for example during the one reported in figure 1.34(c). This has been attributed to the absence of grain boundaries (acting as dislocation source in polycrystals) and to the time needed to reach the critical resolved shear stress required for dislocation mobility.

Similar results have been found by Gunturi et al (2000), who performed tests at 750°C on CMSX—4 crystals misaligned within the [001] – [111] boundary. Parallel slip bands and a planar failure on a specific crystallographic plane have been observed for crystals having higher misalignments and deforming by single slip (figure 1.37(b)). Two perpendicular planes have rather been observed after the creep rupture of the specimen lying on the boundary (figure 1.37(c)).

Many aspects of low temperature creep along crystals close to < 001 > have been
clarified in Rae et al (2000a), Rae and Reed (2007). In these works tests have been conducted on CMSX-4 crystals at 750°C and 850°C. Rae et al (2000a) have observed that the creep response at low temperature cannot be totally rationalized on the base of the Schmid criterion and lattice rotation as proposed by Mackay and Maier (1982). In fact, even if crystals close to the [102] theoretical orientation have an high Schmid factor on the secondary octahedral systems, they deform by $a/2 < 011 > \{111\}$ slips and hardening is produced by the multiple slip intersection.

In Rae and Reed (2007), octahedral slip reactions producing secondary octahedral of $< 112 > \{111\}$ slips have been finally identified. The crystal orientation dependency has been found to rely on the activation of matrix perfect dislocations suitable for combining according on these reactions. The initial incubation period, already observed by Leverant and Kear (1970), has been found to depend on the nucleation of these dislocations in the matrix channels.

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature:  
a tensorial microstructure-sensitive approach
Anisotropy of creep and stress relaxation of MD2 has recently been investigated by Segersäll et al (2014) at 750°C and 950°C along $<001>$, $<111>$ and $<011>$. From conventional creep tests the $<011>$ direction has been found to have the poorest creep ductility and a crystallographic fracture along a $\{111\}$ type plane. Single slip bands a crystalline rupture surface have been observed after creep along $<011>$, in agreement with Gunturi et al (2000). The observation of the $<001>$ and $<111>$ samples have rather revealed multiple slips traces (figure 1.38).

Stress dependence

The stress dependence of the low temperature creep response has been investigated by Sass et al in references (Sass et al, 1994) and (Sass et al, 1996) on crystals of CMSX—4 alloy at 800°C and 850°C and for stresses higher than 500 MPa. In these studies, creep rate-vs-creep strain plots have shown that the orientations deforming by secondary octahedral slips have a sigmoidal shape during primary creep. This sigmoidal primary creep
has been observed along the direction [001] at 650 MPa but not at 500 MPa, while it has been observed at all stress levels during creep along [011], suggesting that the activation of $<112>\{111\}$ slips depended on a stress threshold overcoming along each crystalline orientation. The existence of this stress threshold has been definitely proved in Matan et al (1999b). Its value has been measured by Rae and Reed (2007) for CMSX-4 specimens quite well aligned within the perfect $<001>$ direction; it has been found temperature dependent. At 750°C a stress higher than 400–450 MPa has to be overcome in order to observe primary creep, while at 850°C the stress threshold increases to 500–550 MPa (figure 1.40).

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: 
a tensorial microstructure-sensitive approach
Anisotropy of the creep response

Figure 1.40 – Stress dependency of the CMSX-4 creep response observed by Rae and Reed (2007). Creep curves at 750°C (a) and at 850 °C (b). Primary creep strain amplitude as a function of the applied stress at 750°C and 850°C (c).

Alloy chemistry

The first studies on the impact of chemical composition on the low temperature creep behavior have been conducted by Giamei (1979) and Caron et al (1988), who first perceived that the Rhenium addition in second generation superalloys has the consequence to promote planar fault propagation and the cooperative shearing of the γ/γ’ microstructure. Further investigations have been performed by Rae et al (2003). They have shown that
second and third generation superalloys are more apt to be deformed by \(< 112 \{111}\) slips than the ones belonging to the first generation, because the Rhenium addiction decreases the energy required to produce stacking faults. Thus, if at intermediate and high temperatures the creep response is improved, this is obtained at the expense of a higher anisotropy at low temperature and above all, at the expense of higher strains and shorter lifetimes along several crystal orientations at low temperatures/high applied stresses.

**Twinning**

Ardakani et al (1998), Kakhei (1999), Barba et al (2017) have observed that a different deformation mechanism takes place during creep along \(< 011 \) crystals. This is the phenomenon of mechanical twinning, which is quite unusual and rarely observed in Ni-based single crystal superalloys. While Ardakani and Kakehi have just observed the phenomenon (in SRR99 and CMSX-4 alloys respectively), Barba et al have investigated its occurrence and stress/temperature dependence using MD2 alloy. The results of these investigations suggest the existence of a well defined stress/temperature range where twinning bands develop in the material. In fact, at 800°C the twinning bands volume density has been found to increase with stress variation from 625 to 675 MPa, while at 825°C twinning bands have been observed only at 625 MPa (figure 1.41(a)). Moreover, this study has highlighted that the rupture surfaces of twinned specimens lye along twinning propagation planes (figure 1.41(b)).
Figure 1.41 – From Barba et al (2017): (a) volume density of twinning bands as a function of stress and (b) rupture surface of twinned specimens.
1.6 Conclusion

Conclusions are presented in term of modeling guidelines and experimental perspectives.

1.6.1 Modeling guidelines

CMSX-4 is a single crystal superalloy. Because of the absence of grain boundaries, the cubic symmetry is transposed from the micro- to the meso- and to the macro-scale. From a modeling point of view, this means that the model should reproduce the cubic symmetry of the elastic and plastic response.

At high temperatures the mechanical response is governed by microstructural evolutions and matrix visco-plasticity. Visco-plasticity develops in the matrix by the dislocations motion on octahedral or pseudo-cubic slip systems according to the crystal direction loaded. When loading along the $<001>$ crystal direction, inelastic deformation develops by slips on octahedral systems while, when loading along $<111>$, inelastic deformation develops mainly by pseudo-cubic slips. Finally, when loading along crystal directions misaligned within $<001>$ and $<111>$, the material deform by a combination of these two slips modes. This means that both octahedral and pseudo-cubic slips have to be included in the modeling for a proper description of anisotropy. Microstructural evolutions condition both the isothermal and the non-isothermal mechanical responses. They produce the strain acceleration at the beginning of tertiary creep (le Graverend et al, 2017), the gradual softening during monotonic tension at low strain rates (Gabrisch et al, 1994) and the strain acceleration during thermal cycling (Cormier et al, 2007a,b, le Graverend et al, 2011, Giraud, 2013a, Ghighi et al, 2012, Cormier et al, 2015). $\gamma'$-rafting, coarsening and dissolution have then to be modeled. This experimental evidence, which is the basis of Institut PPrime and Ecole des Mines Polystar constitutive equations (Cormier and Cailletaud, 2010b), is reinforced by the results of many modeling studies which have shown that these characteristics of the mechanical response cannot be reproduced without including the description of microstructural evolutions (Tinga, 2009a, Fedelich et al, 2012b, Ghighi et al, 2012, Cormier et al, 2015, Graverend et al, 2013, Giraud, 2013a).

Similarly to matrix slips, different morphological evolutions develop, according to the crystal direction solicited. Rafting develops during creep along $<001>$ crystals, mechanical coarsening develops during creep along $<111>$ crystals and both $\gamma'$ growth tendencies are present when soliciting others crystal directions. Rafting and mechanical coarsening have then both to be modeled, as well as their strain rate dependence observed during monotonic tensile tests. And having in mind Finite Element computations the full model has to be 3D.

Lattice rotation also impacts the creep response. During high temperature creep the rotation of crystal lattice is localized and limited to the areas having high stress triaxiality. Instead, during low temperature creep along unstable crystal lattice directions, the whole crystal lattice rotates. Rotation changes the activated matrix slips and changes the stress state at the $\gamma/\gamma'$ scale. This also conditions the microstructural morphological evolutions.
Crystal lattice rotation should then also be included in the modeling in order to correct describe the creep response, especially along unstable crystals. Focussing in present thesis on (stable) $<001>$ and $<111>$ orientations, we will nevertheless leave the modeling of the consequences of lattice rotations on the mechanical behavior to further works.

1.6.2 On experimental aspects

The $\gamma'$ dissolution kinetics is highly sensitive to the alloy chemistry. Data on the variation of the volume fraction of precipitates with temperature can be found in (Hemmersmeier and Feller-Kniepmeier, 1998, Link et al, 2000, Roebuck et al, 2001, Serin et al, 2004, Reed et al, 2007a, Link et al, 2011) and in (Giraud, 2013a) for the CMSX-4. However, the chemistry of the alloy tested in all these studies is slightly different from the alloy tested in this study, except of the one tested by Giraud (2013a). At the same time, the dissolution tests performed by Giraud have been aimed at studying the impact of plastic deformation on the $\gamma'$ dissolution kinetics at 1200°C and 1250°C, thus they do not provide information at the others (lower) temperatures. For this reason, further dissolution tests should be performed in order to accurately characterize the variation of the $\gamma'$ volume fraction with temperature.

The material response at high temperatures is governed by $\gamma'$-rafting phenomenon. Many studies on the creep response have been performed on CMSX-4 at temperatures higher than 900°C. $<001>$-like crystals have been tested by Caron et al (1988), Peng et al (1996), Matan et al (1999a), Reed et al (1999), Henderson et al (1998), Epishin et al (2000), Epishin and Link (2004), Serin et al (2004), Epishin et al (2008), Giraud (2013a) while tests on crystals oriented along $<011>$ and $<111>$-like directions can be found in the works of Caron et al (1988), Peng et al (1996), Sass et al (1996). Additional datas are also contained in several modeling studies, as for example those of Svoboda and Lukas (1997), MacLachlan and Knowles (2000), Fedelich et al (2009), Tinga (2009a), Fedelich et al (2012b). However the whole set of literature experiments does not provide enough datas for identifying a complex model such as the one proposed in chapter 3. Hence, creep tensile tests should be carried out at the main temperatures of interest in order to derive the constitute equations and to identify and validate the model. Apart from creep, Gabrisch et al (1994), Diologent et al (2002), Cormier (2006), Giraud (2013a) have shown that the monotonic tensile response and ductility dependency on the strain rate is also related to the onset and kinetics of the $\gamma'$ morphological evolutions. Datas concerning the CMSX-4 response are only available in Giraud (2013a) and Tinga (2009a) but in Giraud (2013a) these only concern the $<001>$ crystals while in Tinga (2009a) no information about ductility are provided. Then, further monotonic tensile tests should be performed in order to prepare to the modeling.

Literature studies have shown that the non-isothermal response of single crystal superalloys highly depends on $\gamma'$-dissolution and $\gamma'$-rafting kinetics. For this reason, the behavior under non-isothermal loading depends strongly on the alloy’s chemistry. Creep tests and thermal cycling tests have already been carried out on CMSX-4 by Giraud (2013a) but certification-engine conditions have been tested only on MC2 by Ghighi (2013).
Operative-like conditions should be tested also on CMSX-4. *Further investigations on the deformation/damaging mechanisms introduced by the repeated overheatings should also be carried out.*

Almost all the aspects of the high temperature creep anisotropy have already been clarified in (Caron et al, 1988, Peng et al, 1996, Sass et al, 1996, Matan et al, 1999b, Ghighi, 2013) and in the other studies cited in the section 1.5.1. Concerning CMSX-4, a systematic study on the stress magnitude impact on the creep response at high temperature has been been carried out in Matan et al (1999b), but just focusing on crystals slightly misaligned within the $<001>$ theoretical direction. Then misoriented creep tests could also be used to complete the pannel about CMSX-4.

At low temperatures, several studies on the anisotropy of creep response have already been carried out on CMSX-4, but they have been performed mostly along $<001>$-like crystals (Matan et al, 1999b, Rae et al, 2000a, Rae and Reed, 2007). Crystals having higher misalignments have been tested only at few stress levels (Caron et al, 1988, Sass et al, 1996, Ardakani et al, 1998, Kakhei, 1999). As for creep at high temperatures, several datas are available in many modeling studies, as for example those of MacLachlan et al (2002), Svoboda and Lukas (2000), Tinga (2009a), but these data do not provide a complete database for the model elaboration. Further tests have been performed on other alloys (Leverant and Kear, 1970, Leverant et al, 1973, Mackay et al, 1980, Mackay and Maier, 1982, Segersäll et al, 2014), but since the creep response at low temperature strongly depends on the alloy chemistry (Rae et al, 2003), the CMSX-4 response is expected to be different. Finally, if all the above-mentioned studies agree on the fact that the creep anisotropy at low temperatures is generated by $<112>\{111\}$, just in few of these studies it has been found that local conditions in the inner microstructure can bring to the formation of mechanical micro-twinning bands (Ardakani et al, 1998, Kakhei, 1999, Barba et al, 2017). Thus, in order to provide both a database for the model identification and a further investigation on the anisotropy of the creep response of CMSX-4, *further tests should be carried out on highly misaligned samples of CMSX-4 at low temperatures and high stresses.*
Chapter 2

Mechanical response and microstructural evolution of CMSX-4 during creep, load-free thermal exposure and non-isothermal loading.

“Blowing in the wind”
Bob Dylan
2.1 Motivations and strategy

In this section, the crystal orientations tested are listed in reference to the theoretical directions of the standard stereographic triangle. In the next sections, results are analyzed and discussed as a function of the real orientations. These results are presented in terms of $\theta$ and $\rho$, the angles used for the stereographic projection (figure 2.1).

![Standard stereographic triangle](image)

Figure 2.1 – Standard stereographic triangle.

Experimental tests are grouped in four categories:
- high temperature tests;
- non-isothermal tests;
- dissolution tests;
- low temperature tests.

The strategy adopted in choosing the testing conditions and the motivations behind are described in the paragraphs below. Details about the CMSX-4 tested in this study, the experimental rigs and the observation techniques can be found in the Appendix A. The techniques used for data and image post-processing are presented in the Appendix B. Tables summarizing all experimental results are presented in the Appendix C. The testing conditions have been chosen based on the average temperatures variations along the blades profiles during the helicopter cruising phase.

**High temperature tests** – The description of the high temperature response of the material is the primary objective of the modeling part of this work, in particular at 1050 °C. An experimental database is then required to identify and to test the model at this temperature. Several data can be found in the work of R. Giraud Giraud (2013a), who has performed experimental creep and monotonic tensile tests on the same material studied in this work. In particular, the most interesting Giraud’s tests for the present study are:
- isothermal creep tests along crystallographic directions close to the theoretical $<111>$, $<011>$, $<112>$ and $<102>$ at 1050°C and 140 MPa;
- the monotonic tensile tests along the $<001>$ crystallographic orientation performed at 1050°C and presented in the section 1.3.2.
Complementary tests have been carried out during the present Ph.D. work to supply for the missing data required for the modeling. Creep tests have been primarily carried out along the $<001>$ and $<111>$ crystal directions. The reason is that, as seen in the sections 1.1.2 and 1.5.1, for loadings along $<001>$ and $<111>$, different matrix deformation mechanisms and microstructural evolutions take place. In addition, the response of CMSX-4 along the other crystal directions results from a combination of these phenomena. Then, equations describing these phenomena are introduced in next modeling and so, appropriate tests are required to identify the corresponding material parameters. Further creep experiments are performed at 1050°C along $<102>$, $<011>$, and $<112>$ directions under different several stress levels. Details on the modeling are contained in chapter 3. In the present chapter, the results of the tests performed are presented and discussed in order to provide —altogether with existing tests— a complete picture of the anisotropy of CMSX-4. In fact, if on one hand creep experiments allow to identify the model, on the other hand they also constitute a base for the systematic investigation of the impact of the crystal orientation and the stress magnitude on the high temperature/low stress creep response. Similarly, monotonic tensile tests are performed along quasi-$<111>$ crystals. These tests allow to both identify the model and investigate on the strain rate effect on the flow stress.

**Non-isothermal tests** — The prevision of the material response under close to life conditions is the second objective of the modeling. Two cycling tests are performed in order to test the model suitability in reproducing non-isothermal creep conditions. The thermo-mechanical loading path is similar to the one of the tests performed in Mauget (2012), section 1.4. During these tests, the material is subjected to repeated thermal shocks reproducing the three phases of the One Engine Inoperative condition (described in the general introduction, page 2). These tests also allow to investigate on the material degradation under emergency and in-service conditions.

**Dissolution tests** — As pointed out several times in the previous chapter, $\gamma'$-dissolution plays a fundamental role in the mechanical response during isothermal and non-isothermal conditions and its kinetics is very sensitive to the chemistry of the alloy. Then, specific tests are performed in order to quantify the volume fraction of precipitates at the thermodynamic equilibrium ($f_{\gamma'_{eq}}$) in a wide temperature range, going from 950°C to 1300°C.

**Low temperatures tests** — Several questions are still open about creep at low temperatures/high stresses of CMSX-4. For example, micro-twinning has been observed in (Ardakani et al, 1998, Kakhei, 1999) while it has never been observed during the studies carried out by Rae et al (Rae et al, 2000a, 2001, Rae and Reed, 2007). In addition, few tests have been carried out along highly misoriented CMSX-4 specimens. Since the low temperature response has been proved to be very sensitive to Rhenium addition (as shown by Rae et al (2003)), results found in Leverant and Kear (1970), Leverant et al (1973),
Mackay et al (1980), Mackay and Maier (1982) may not be transposed to CMSX-4. For these reasons, many creep tests are performed in the low temperatures/high stresses range during this Ph.D. The temperature chosen for these tests is 850°C because the material response at 700-750°C has already been documented in literature (Kakhei, 1999, Matan et al, 1999b, Rae et al, 2000a, Rae and Reed, 2007).

2.2 1050°C tests

2.2.1 Creep tests

At 1050°C, creep tests are performed at 140, 180 and 200 MPa. Primary and secondary orientation are given in table 2.1 (Giraud results also included). In figure 2.2, crystal orientations are represented in the standard stereographic triangle.

Table 2.1 – Primary and secondary misorientation (respectively $\theta$ and $\rho$) of the CMSX-4 samples tested at 1050°C in this study and during the Ph.D. of R. Giraud.

<table>
<thead>
<tr>
<th>Bar</th>
<th>$\theta$</th>
<th>$\rho$</th>
<th>$\sigma$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1.3</td>
<td>3.3</td>
<td>140,180,200</td>
</tr>
<tr>
<td>B</td>
<td>23.7</td>
<td>2.3</td>
<td>180,200</td>
</tr>
<tr>
<td>C</td>
<td>45.1</td>
<td>6.3</td>
<td>180,200</td>
</tr>
<tr>
<td>D</td>
<td>52.9</td>
<td>44.9</td>
<td>180,200</td>
</tr>
<tr>
<td>E</td>
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<td>38.9</td>
<td>180,200</td>
</tr>
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<td>21.4</td>
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</tr>
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<tr>
<td>D'</td>
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<td>41.9</td>
<td>140</td>
</tr>
</tbody>
</table>

Table 2.1 – Primary and secondary misorientation (respectively $\theta$ and $\rho$) of the CMSX-4 samples tested at 1050°C in this study and during the Ph.D. of R. Giraud.

Figure 2.2 – Location of the creep specimens tested at 1050°C within the standard stereographic triangle.

Creep curves and rate vs strain curves of the specimens cast from the bars A, B and B’ are presented in the figure 2.3, while figure 2.4 presents the same curves for the specimens cast from the bars C,D,D’ and E. Figures 2.5, 2.6 and 2.7 presents the measured primary creep strain, minimum creep rate and lifetimes.
Crystal orientation impact – Creep and rate curves have almost the same shape along all the crystallographic orientations tested (figure 2.3 and 2.4). No steady state stage is observed. The primary creep stage is short and leads to low final deformations lower than 0.4%. Thus, an initial quasi-zero rate phase is followed by a strain rate acceleration. The measured minimum creep rates are very close (figure 2.6), as well as the observed lifetimes 2.7). The maximum deviation among the lifetimes is observed at 140 MPa, where the D'-oriented specimen takes twice the time of the specimen oriented along A (close to the <001> crystal direction) to fail. D and D' specimens (i.e. close to the <111> orientation) present the longest lives and the lowest final ductility at each stress. On the contrary, C-oriented specimens (i.e. close to the <011> direction) exhibit the shortest lifetimes and the largest final strains. A specimens have an intermediate behavior between C and D/D' ones, while the specimen oriented along E (i.e. close to the <112> crystal direction) has an intermediate behavior between A and D/D’. Finally, B specimens (i.e. oriented close to the <102> crystal direction) have creep ductility and lifetimes similar to the ones observed along A. At the end of creep experiments a transformed γ/γ’ microstructure has been systematically observed. Figure 2.8 contains the SEM post-mortem observations of the sample A tested at 140 MPa and of the samples C, D and E tested at 180 MPa. N-rafting is observed after creep along the <001> direction and mechanical coarsening is observed along a close-to-<111> direction. Rafted particles inclined with respect to the loading direction are observed during creep along E. The absence of a primary creep phase and the rapid strain rate acceleration can be explained by the rapid onset of microstructural evolutions. At the beginning, the creep flow is partially blocked by the vertical channels closure process. Once the γ’-rafting is completed, the plastic rate rapidly accelerates because of the higher dislocations mobility in horizontal channels. As observed by le Graverend et al (2017), plastic damage contributes to the strain rate acceleration only when a certain amount of plastic deformation has already been attained. The lowest final creep strains and longest lifetimes observed along D and D’ orientations can be related to the higher difficulty for dislocations to propagate into roof-shaped channel. The intermediate behavior of E/E’ between A and D/D’ can also be rationalized by microstructure morphology changes. E/E’ samples show mostly plate-shaped particles. However several perpendicular channels can be still recognized (figure 2.8(e)). The latter are the traces of the competition between the oriented coalescence along a unique {001} plane and the mechanical coarsening. B and B’ specimens have not been observed. SEM observations after creep along B/B’ crystals of MC2 alloy at 1050°C can be found in the Ph.D. thesis of J. Ghighi (2013). γ’-rafted particles inclined with respect to the load direction have been observed, confirming that even after creep along directions close to the theoretical <102> γ’ particles grow along the <001> planes. Moreover, as in the case of the <112> direction, several perpendicular channels can be still recognized. It has to be remarked that most of the micrographies contained in this section show matrix particles surrounded by the γ’ phase (figure 2.8). Connectivity number determination have been carried out by performing an image analysis on SEM post-mortem observations of almost all the tested specimens (according to Caron et al (2008)). Since topological inversion is not the focus of this study, these measures are
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not presented in this chapter but rather reported in Appendix B. The reason is that it has been chosen to roughly include the topological inversion effects in the phenomenological damage modeling of chapter 4.
Figure 2.3 – Results of the creep tests performed at 1050°C along the specimens casted from the bars A, B and B’ (table 2.1): (a) creep curves, (b) rate vs strain curves.
The fracture morphology also shows a dependency on the loading direction. During creep along the $<001>$ direction at 140 MPa, cracks initiate and propagate perpendicularly to the loading direction, as evidenced in figure 2.9. During creep along C (i.e. close to the $<011>$ crystallographic direction) at 180 MPa, cracks and fractures propagate along the same planes where rafted particles grow (see figure 2.10). Along D (close to the $<111>$ crystallographic orientation) at 180 MPa, the rupture is indeed almost crystalline (as shown in figure 2.11(a)). Figures 2.9(c), 2.10(a) and 2.11(d), show that the microstructure close to the fracture surfaces is more degraded than what is observed far from the fracture (figure 2.8). The extremities of the $\gamma'$-rafted particles are rotated. This additional degradation is caused by the high stress triaxiality and stress concentration associated to macro-fracture formation and propagation (Reed et al, 2007a, Caccuri et al, 2017).

Finally, even if the morphology of the $\gamma/\gamma'$ microstructure and the fracture surface orientation depend on the crystallographic direction solicited, no anisotropy of the visco-plastic properties has been observed. Along all the crystallographic orientation tested, the absence of a classical primary creep stage and the continuous strain acceleration are a consequence of a fast $\gamma'$-rafting completion (as described in the subsection 1.3.1). Then, it can be concluded that the crystal orientation does not fundamentally affects the mechanisms characterizing the creep response in this temperature/stress domain. This is in agreement with what has been observed in literature, as for example by Khan and Caron (1991), Matan et al (1999b) (section 1.5.1).
Figure 2.4 – Results of the creep tests performed at 1050°C along the specimens casted from the bars C, D, D’ and E (table 2.1): (a) creep curves, (b) rate vs strain curves.

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Figure 2.5 – Primary creep strains observed during the creep tests performed at 1050°C.

Figure 2.6 – Minimum creep rates observed during the creep tests performed at 1050°C.
Figure 2.7 – Creep lifetimes observed after the creep tests performed at 1050°C.

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Figure 2.8 – SEM post-mortem observations after creep at 1050°C: (a) along A at 140 MPa, (b) along C at 180 MPa, (c) along D at 180 MPa and (d) along E at 180 MPa. Images are taken in the bulks of the gauge length far from failure to avoid triaxiality effects on the γ/γ′ microstructural evolutions. The location of the specimens in the standard stereographic triangle are shown in figure (e). The arrows in the figures (a),(b),(c) and (d) indicate the direction of the applied stress.
Figure 2.9 – SEM post-mortem observations after creep at 1050°C and 140 MPa along the crystallographic direction A (i.e. close to the < 001 > theoretical direction): (a) fracture surface; (b) crack initiation from a pore; (c) microstructure at 1mm from the fracture; (d) location of the specimen in the standard stereographic triangle. The arrows in the figures (a), (b) and (c) indicate the direction of the applied stress.
Figure 2.10 – SEM post-mortem observations after creep at 1050°C and 140 MPa along the crystallographic direction C (i.e. close to the <011> theoretical direction): (a) fracture surface; (b) crack initiation close to a pore; (c) microstructure at 1mm from the fracture; (d) location of the specimen in the standard stereographic triangle. The arrows in the figures (a),(b) and (c) indicate the direction of the applied stress.
Figure 2.11 – SEM post-mortem observations after creep at 1050°C and 140 MPa along the crystallographic direction D (i.e. close to the <111> theoretical direction): (a) fracture surface; (b) crack initiation close to a pore; (c) microstructure at 1mm from the fracture; (d) location of the specimen in the standard stereographic triangle are shown in. The arrows in the figures (a),(b) and (c) indicate the direction of the applied stress.
Stress magnitude impact – At all the loading conditions tested creep and rate curves have almost the same shape (figure 2.3 and 2.4). The highest ductility are observed at the highest stress along $<001>$ (A samples), $<011>$ (C samples) and $<112>$ (E samples) while, on the contrary, they are observed at the lowest stress along $<111>$ and $<102>$ (respectively samples D’ and B’). The minimum creep rates are slightly different from one stress to the other but values are, however, very close (figure 2.6). The longest lifetimes are observed at the lowest stresses. The longest creep test is the one performed at 140 MPa along the $<111>$ crystal direction. In this condition the sample takes almost 622h to fail, while at 200 MPa the creep lifetimes is reduced to 66.9h. A similar dispersion is observed along all the crystallographic orientation tested. Figures from 2.3 to 2.7 show that the slope of the creep curve is not impacted by the stress magnitude. SEM images, presented in figure 2.12, show that the morphology of the evolved microstructure is not impacted by the stress either, except that during creep along the $<011>$ orientation. In this case the $\gamma'$-rafted particles result inclined within the loading direction after creep at 180 MPa, while they are perpendicular to the applied stress after creep at 200 MPa. This difference has probably to be attributed to the crystal lattice instability during non-symmetrical loading. Under $<011>$-like loading, the crystal lattice probably rotates and the $\gamma'$-particles growing directions rotates too. EBSD measurements would be necessary to characterize the lattice rotation but the final morphology suggests that the crystal has rotated toward $<001>$. This should also explain the similarity of the creep and rate curve observed between the A and C at this stress (figure 2.13).

No change of the morphology of the $\gamma/\gamma'$ microstructure is observed after creep along directions close to the theoretical $<001>$ and $<111>$ (A, D and D’ respectively) according with the stress.

The crystal lattice rotation also controls the fracture mode. Cracks do not initiate and propagate anymore along a crystalline plane but perpendicularly to the load.

Similar conclusions can be established for the specimen creep deformed at 200 MPa along the $<111>$ crystal orientation (sample D). At 180 MPa fracture initiation and propagation are almost crystalline. At 200 MPa the sample exhibits a N-rafted and roughly roof-shaped/inclined rafted particles in several zones close to the fracture surface, meaning that probably the crystal lattice has locally rotated toward a $<001>$-like behavior. This could explain why the fracture surface is partially oriented perpendicularly to the loading direction and partially lies along crystalline plane (figure 2.15 (a)).
Figure 2.12 – SEM post mortem observations after creep at 1050°C and 200 MPa: (a) along A, (b) along C, (c) along D and (d) location of the tested specimens in the standard stereographic triangle. The arrows in the figures (a),(b) and (c) indicate the direction of the applied stress.
Figure 2.13 – Comparision between the creep responses at 1050°C along A and B crystals at 200 MPa: (a) creep strain vs time and (b) creep strain rate versus strain curves.
Figure 2.14 – SEM post-mortem observations of the C crystal tested at 1050°C and 200 MPa: (a) fracture surface, (b) microstructure at 1mm from the fracture surface and (c) location of the specimens in the standard stereographic triangle. The arrows in the figures (a) and (b) indicate the direction of the applied stress.
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Figure 2.15 – SEM post-mortem observation of the C crystal tested at 1050°C and 200 MPa: (a) fracture surface, (b) microstructure at 5mm from the fracture surface and (c) microstructure at 1mm from the fracture surface. The arrows in the figures (a),(b) and (c) indicate the direction of the applied stress.
2.2.2 Monotonic tensile tests

Monotonic tensile tests have been performed at 1050°C using specimens machined from bars oriented along a quasi-<111> crystallographic direction. The orientation of the specimens as well as the testing condition are summarized in the table 2.2. Two tests have been carried out at constant rate at $10^{-5}s^{-1}$ and $10^{-3}s^{-1}$. One test has been carried out by varying the strain rate in the range of $10^{-5}s^{-1}$-$10^{-2}s^{-1}$.

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<tr>
<td>D</td>
<td>52.9</td>
<td>44.9</td>
<td>variable in $10^{-5}s^{-1}$-$10^{-2}s^{-1}$</td>
</tr>
</tbody>
</table>

Table 2.2 – Monotonic tensile test along a <111>-like crystal orientation: orientation of the samples and imposed rate.

Test results are shown in the figure 2.16. These are presented in terms of logarithmic stress and strain.

The test realized at the lowest strain rate does not show any sign of the softening induced by microstructural evolutions (figure 2.16(a)) until a quite high strain level (see section 1.3.2). At the same time, SEM observations prove that microstructural evolutions have already initiated (figure 2.17(a)). Hardening then acts simultaneously with mechanical coarsening and is probably produced by the high dislocation density in the $\gamma$ channel and at the the $\gamma/\gamma'$ interfaces (figure 2.17(a)). Comparing the first and the second stages at $10^{-5}s^{-1}$ and $10^{-3}s^{-1}$, one can notice that hardening is also produced during the $10^{-2}s^{-1}$ stage (figures 2.16(b) and 2.17(c)). At this strain rate, the shearing of $\gamma'$ particles could also play an important role. TEM analysis should be performed in order to confirm it.

No sign of microstructural evolution are observed after the test realized at $10^{-3}s^{-1}$, as well as after the complex test (figures 2.17(b)). Still in agreement with literature (section 1.3.2), the strain rate effect on the microstructure evolution has been found to depend on the test duration. The $10^{-3}s^{-1}$ test lasted for 0.1 h and the complex test lasted for 1.41 h. The $10^{-5}s^{-1}$ test lasts 7.96 h. Then, while in this last case enough time is left for mechanical coarsening development, the other two tests finished too early to observe an evolution of the $\gamma/\gamma'$ microstructure.

The results of monotonic tensile tests performed at constant rate (figure 2.16(a)) show that the ductility strongly depends on the strain rate (details on the final elongation and the variation of cross section measures after tensile tests are given in the Appendix B). SEM observations do not reveal a large amount of pores, meaning that plastic damage just sets at the end of the tests. The sample tested at $10^{-5}s^{-1}$ does not even show high necking (figure 2.18(a)). This means that at this strain rate, the main degradation source is mechanical coarsening. At $10^{-3}s^{-1}$ no degradation is produced until necking enhances the plastic damage (figure 2.19). This sample then has a higher elongation at failure. Similar considerations can be extended to the complex test 2.20. Deformation/damage mechanisms similar to the ones observed at $10^{-3}s^{-1}$ occur during the high rate phases (strain rate $> \simeq 10^{-3}s^{-1}$). At the opposite, deformation/damage mechanisms similar to
Figure 2.16 – Monotonic tensile response along $<111>$ crystallographic orientation (sample D) at 1050°C: (a) at a constant strain rate (b) at a variable strain rate.

the ones observed at $10^{-5}$s$^{-1}$ occur, of course, during the low rate phases (strain rate $< \approx 10^{-4}$s$^{-1}$). At $10^{-5}$s$^{-1}$ a fracture in mode 1 is observed (figure 2.18). At $10^{-3}$s$^{-1}$ the presence of multiple cracks initiation on the surface suggests that in this case fracture propagates from the surface to the volume. At this strain rate, as well as after the complex test, the fracture surface is less regular. During the complex test, crack initiation zones rotate, suggesting that rotations also occur during fracture propagation. This explains the mixed form of the fracture surface. This is probably due to the shearing of $\gamma'$-particles which happen at this high strain rate.

Thus, finally, it can be concluded that the deformation/damage mechanisms characterizing the monotonic tensile response along the $<111>$ crystallographic direction at
$1050^\circ C$ tests

\[
\begin{align*}
(a) \quad \sigma & \equiv D, \ 10^{-5}s^{-1} \\
(b) \quad \sigma & \equiv D, \ 10^{-3}s^{-1} \\
(c) \quad \sigma & \equiv D \text{ variable strain rate} \\
(d) \quad \sigma & \equiv D \text{ variable strain rate}
\end{align*}
\]

Figure 2.17 – SEM post-mortem observations after the tensile tests along $\approx <111>$ oriented specimens: (a) microstructural morphology observed in the cross section of the specimen tested at $10^{-5}s^{-1}$, (b) microstructural morphology observed in the longitudinal section of the specimen tested at $10^{-3}s^{-1}$, (c) microstructural morphology observed in the cross section of the specimen tested at variable strain rate and (b) location of the tested specimens in the standard stereographic triangle. “· $\sigma$” means normal to foil.

$1050^\circ C$ are similar to the one observed by Giraud (2013a) along $<001>$. At low strain rates microstructural evolutions cause the degradation of the mechanical response and favor the plastic damage development. At higher strain rates ($\approx 10^{-3}s^{-1}$) plasticity governs the material response. No microstructural evolution set and plastic damage occurs during the last stage of the test, when it is enhanced by necking. Two differences between $<001>$ and $<111>$ have however been observed. The first is that $\gamma'$-mechanical coarsening happen when loading along $<111>$ while $\gamma'$-rafting occurs when loading along $<001>$. Furthermore, hardening is produced when loading along $<111>$. For this reason, no softening (due to microstructural evolution) is observed when testing the $<111>$ oriented samples while it is observed when testing $<001>$ samples (compare figure 2.16(a) to 1.23(b).

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Figure 2.18 – SEM post-mortem observations after the tensile test realized along $\sim<111>$ crystallographic direction at 1050°C and $10^{-5}s^{-1}$: (a) close to failure surface, (b) zoom on the pores contained in the red box area of left figure and (c) location of the tested specimen in the standard stereographic triangle. The arrows in the figures (a) and (b) indicate the direction of the applied stress.
Figure 2.19 – SEM post-mortem observations after the tensile test realized along $\approx<111>$ crystallographic direction at 1050°C and $10^{-3}\text{s}^{-1}$: (a) close to failure surface, (b) zoom on the crack initiation contained in the red box of the previous figure, (c) zoom on the pore close to the crack initiation and (d) location of the tested specimen in the standard stereographic triangle. The arrows in the figures (a), (b) and (c) indicate the direction of the applied stress.
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Figure 2.20 – SEM post-mortem observations after the tensile test realized along ≃<111> crystallographic direction at 1050°C by varying the strain rate from $10^{-2}\text{s}^{-1}$ to $10^{-5}\text{s}^{-1}$: (a) close to failure surface; (c) zoom on a pore showing rotating cracks initiation zones; (d) location of the tested specimen in the standard stereographic triangle. The arrows in the figures (a),(b) and (c) indicate the direction of the applied stress.
2.3 Non-isothermal MAATRE tests

Two endurance engine like tests have been performed using the MAATRE burning rig (described in the Appendix A). The applied thermomechanical loading paths are presented in figures 2.21.

![Diagram of thermomechanical loads imposed during non-isothermal experiments: (a) test-01 and (b) test-02.](image)

The first test, here referred to as the “Test-01”, consists of three stages (figure 2.21(a)). Stage I and Stage II are repeated 15 and 10 times respectively. Both contain a 1 h long phase at ≃ 1150°C. This is included to reproduce the last part of the One Engine Inoperative event (OEI regimes are described at page 2). Similarly, the High Temperature Stage contains many overheating reproducing all the three phases of this event, including the 30 sec OEI, which is the most severe one. The second test, referred to as “Test-02” is presented in figure 2.21(b). This is composed by the same cycles as for the first test, ex-
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Except for the addition of the OEI Stage. In this additional stage the three steps of the One Engine Inoperative events are repeated in a rapid sequence.

**Test-01** – During non-isothermal creep tests carried out using the MAATRE burner, temperature is controlled with a spot welded S-type thermocouple while total and creep strain are followed by video-extensometry (see Appendix A). Unsmoothed curves are shown in figure 2.22(a). Mechanical strain is computed by subtracting the thermal strain to the total strain. However, the thermocouple is welded at the gauge length center while the extensometer measures the displacement between two points located at ±2 mm from the center. The heating/cooling rates at the gauge points where the thermocouple is spot welded and where the strain is measured are different during thermal shocks. This is the reason why two peaks appear at each thermal transient phase when subtracting the thermal deformation (figure 2.22(b)). However, they are just artifacts produced by data post-processing. Moreover, peaks are very localized and confined. Thus, they do not affect the overall measurements and post-processed data.

The mechanical strain has a creep-shaped curve. Figure 2.23(a) shows the smoothed plastic strain (details on data post-processing and smoothing techniques are given in the Appendix A). According to what was observed by Mauget (2012), the High Temperature Stage causes a rapid strain rate acceleration. As shown in (Cormier, 2006, Giraud, 2013a), this acceleration depends on the evolution of the $\gamma/\gamma'$ microstructure. The high amount of plastic deformation produced in the $\gamma'$ rafted microstructure accelerates the $\gamma'$ dissolution kinetics. For this reason plasticity is enhanced by each thermal shock. As a consequence, plastic strain jumps of increasing amplitude are observed after each phase of the cycle. Figure 2.23(b) shows the mechanical strain variation during the thermal-shock-cycle. Plastic strain steps, observed in the same cycle but during the Test-02, are shown in figure 2.29(b).

Tested specimens have been observed by scanning electron microscopy. Micrographs have shown that oxydation contribute to a local degradation of the material’s microstructure (see 2.24). As a consequence of the final cooling, the finer $\gamma''$ precipitation is observed in the matrix channel of the $\gamma'$-rafted microstructure. Close to the $\gamma'$-depleted layer, the decrease in Al and Ti contents causes a variation of the lattice mismatch. Because of the lower $\gamma'$ volume fraction just below the $\gamma'$-depleted layer, circular $\gamma''$ particles are observed in these zones (figure 2.25(a)). Cubic particles are instead observed moving toward the bulk of the specimen which is unaffected by oxydation. The overall results and observation are in agreement with the studies carried out by (Bensch et al, 2013, Srivastava et al, 2012).

The last isothermal phase at $\simeq 1200^\circ$C is too short for the Oswald ripening to occur (figure 2.26(a)). Thus, the $\gamma''$ phase produced after the last thermal shock is not re-absorbed by the $\gamma'$-rafted particles. An additional precipitate phase ($\gamma'''$) is observed at the end of the test 2.26(b). This phase precipitates during the very final cooling after the iso-temperature stage at $\simeq 500^\circ$C.

Finally, SEM observations did not reveal the onset of specific damaging processes. Microstructural evolution and oxidation are the main reasons of the material degradation.

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Figure 2.22 – Test-01: (a) unsmoothed curves of the total, thermal and mechanical strain, (b) post-processing artifact on the mechanical strain curve during temperature transient phases.

while, similarly to creep, plastic damage occurs only at the end of the test.
Figure 2.23 – Test-01: (a) plastic strain and (b) mechanical strain steps during the High Temperature Stage.
Figure 2.24 – $\gamma'$-depleted layer observed (a) at the leading edge and (b) at the trailing edge after the Test-01. Location of the tested specimen in the standard stereographic triangle (c). The arrows in the figures (a) and (b) indicate the direction of the applied stress.
Figure 2.25 – Shape change of the finer $\gamma''_II$ phase observed after the Test-01: (a) circular $\gamma''_II$ precipitates observed close to the depletion layer because of the lower Al and Ti content and (b) cubic $\gamma''_II$ precipitates observed in gauge length center. Location of the tested specimen in the standard stereographic triangle (c). The arrows in the figures (a) and (b) indicate the direction of the applied stress.
Figure 2.26 – Temperature conditions at the end of the Test-01 (a) and a SEM observation at the end of the test revealing a i-population of $\gamma_{\text{III}}'$ hyper-fine particles (b). Location of the tested specimen in the standard stereographic triangle (c). The arrow in the figures (b) indicates the direction of the applied stress.
Test-02 – The second non-isothermal test is interrupted after the third OEI Stage in order to investigate damaging processes. As shown in figure 2.29(a), at this point of the test the specimen is already in the tertiary creep stage. However, no mechanical damaging process in the form of crack or massive capitation development is observed. Again, microstructural evolutions and oxidation are found to be the main cause of the material degradation (figures 2.27 and 2.28). Since no a large amount of pores is observed, it is hypothesized that plastic damage occurs only at the very end of the test (figure 2.27(b)).

Figure 2.27 – SEM observation after the interruption of the Test-02 during the tertiary creep phase: (a) microstructure and (b) pore. (c) Location of the tested specimen in the standard stereographic triangle. The arrows in the figures (a) and (b) indicate the direction of the applied stress.

Regarding the global mechanical response, an initial quasi-zero-creep-rate phase is observed during the first three repetitions of the Stage I. Then, as already discussed for the Test-01, plastic strain ramps of increasing amplitude are observed after each OEI (figure 2.29(a)). For this reason wide plastic strain jumps are observed after each OEI Stage (figure 2.29(b)). After the second OEI Stage the jump amplitude is already higher that the one observed after the first OEI Stage. This is probably related to a most degraded state of the microstructure.
Figure 2.28 – γ′-depleted layer observed at the leading edge (a) and at the trailing edge (b) after the non-isothermal Test-02. Location of the tested specimen in the standard stereographic triangle (c). The arrows in the figures (a) and (b) indicate the direction of the applied stress.
Figure 2.29 – Plastic strain evolution during the non-isothermal Test-02: (a) during the first OEI Stage and (b) over the whole test.
2.4 850°C tests

Creep tests have been carried out at 850°C and 500, 600 and 650 MPa in order to investigate on the stress and crystal orientation dependency of the creep response. The orientation of the tested crystals are represented in the standard stereographic triangle in figure 2.30. Two further tests are performed to highlight the stress magnitude impact on the creep deformation mechanisms: a first one along C’ at 400 MPa and a second one along D at 350 MPa. Additional creep tests are carried out to investigate on the deformation modes and kinetics:

— one test along the orientation A at 750 MPa;
— one interrupted test at 650 MPa along the orientation D;
— four interrupted tests at 600 MPa along the orientation C’;
— one in-vacuum test at 400 MPa along the orientation C’.

A synthesis of misorientations and testing conditions is given in the table 2.3.

![Figure 2.30 – Location of creep specimens tested at 850°C within the standard stereographic triangle.](image)

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Table 2.3 – Test conditions and misorientations of the samples tested at 850°C.

In agreement with literature (see section 1.5.2), at 850°C, the creep response is highly sensitive to the crystal orientation. As shown in figure 2.31, primary creep strain amplitude, minimum creep rates and creep lifetimes are very different from one orientation to the other.
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The highest primary creep strains are observed for creep tests performed along the \(\simeq<011>\) and \(\simeq<111>\) orientations (figure 2.31(a), C' and D respectively). However, according to literature (as for example with (Caron et al, 1988, Sass et al, 1996, Ardakani et al, 1998, Gunturi et al, 2000)), deformation happens following different mechanisms along these crystal directions. Figure 2.33(c) shows that a sigmoidal creep behavior is observed along the \(\simeq<011>\) crystal orientation at all the stresses applied except at 400 MPa, while along \(\simeq<111>\) a classical primary creep stage is observed (figure 2.33(b)). Along \(\simeq<011>\), the sigmoidal primary creep stage is followed by high steady state rate and leads to the shortest lifetimes. A similar behavior is observed along E, i.e. along the \(\simeq<112>\) crystal direction, which also has a sigmoidal creep but which attains very different final strains. This difference can be related to the different amount of lattice rotation occurring during the test. Both crystals mainly deform themselves by single slip but C' is a very unstable lattice direction (Mackay and Maier, 1982, Shollock et al, 1997, Ardakani et al, 1998, MacLachlan et al, 2001, Ghighi, 2013, Stinville et al, 2015). Thus, during creep along the direction \(\simeq<011>\), the crystal lattice rotates toward the direction of the Burger vector of the activated slip. At the same time, in agreement with what has observed by Rae et al (2000a), the lattice rotation causes a rapid ovalization and necking of the cross section (figure 2.34). Since creep tests are carried out at constant load, necking causes a true-stress increase. As a result, the direction \(\simeq<011>\) deforms more and faster. Deformation is so fast that the samples tested at 650 MPa and 600 MPa reach the maximum passable displacement after less than an hour without reaching failure (due to a creep rig extension limitation). On the contrary, E is less unstable. It then undergoes lower rotations, lower necking and thus lower strains. This is confirmed by the ellipticity (e) and cross section area reduction measurements shown in figure 2.35. The definitions of e, \(S_{\text{max}}\%\) and \(S_{\text{min}}\%\) are given in the Appendix C. The longest lifetimes and a classical –not sigmoidal– creep response is observed along the \(\simeq<102>\) and \(\simeq<111>\) crystal directions (as shown in figures 2.31(b) and 2.32(b)). The \(\simeq<111>\) crystal directions also performs the lowest steady state rates and primary creep strains, figures 2.31(c) and 2.31(a)). These results are still in agreement with literature, as for example with what observed by (Mackay et al, 1980) (see subsection 1.5.2 for more details).

Since our observation are in agreement with literature results, one can suspect that the samples oriented along the \(\simeq<011>\) and \(\simeq<112>\) (C' and E) are mainly deformed by \(<112>\ \{111\}\)-type slips and stacking fault propagation, while the behavior along the \(\simeq<111>\) and \(\simeq<102>\) directions (D and B) is controlled mainly by \(<110>\ \{111\}\) slips. Additional evidences are found by observing the specimens.

Single slips traces are observed at the surfaces of the \(\simeq<011>\) specimens (C’ in figures 2.34(a), 2.34(b), 2.36(a) and 2.36(b)). A fracture surface lying on a crystalline plane is observed after creep at 500 and 400 MPa (figure 2.36). The same is observed after creep along the \(\simeq<112>\) orientation. The specimens oriented along the \(\simeq<111>\) crystal orientation show instead no trace of single slip but a roughly crystalline fracture surfaces after each test (D in figure 2.37). The \(\simeq<102>\) direction is not analyzed here in detail because everything about low temperatures/high stresses creep of CMSX-4 along almost \(<102>\)-aligned crystals has already been clarified in Rae et al (2000a).

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Regarding the impact of the stress magnitude, this does not seem to change the creep deformation mechanisms and fracture mode in the 500-650 MPa range. On the contrary, along the $\simeq<011>$ at 400 MPa the creep rate vs creep strain curve does not show any sigmoidal creep and creep test along D at 350 MPa shows a very low minimum rate and small strains. As for high temperature creep, the stress impact on the creep response along the $\simeq<111>$ crystal orientation is essentially linked to the test duration. Longer creep lifetimes are observed at lower stresses. As a consequence, microstructural evolutions have more time to develop. An example is given in the figure 2.38. This figure presents the SEM post-mortem observations of the $\simeq<111>$ oriented sample tested at 350 MPa. A $\gamma/\gamma'$ microstructure at the beginning of mechanical coarsening is observed.
Figure 2.31 – Results of the creep tests performed at 850°C. Primary creep strain (a), Minimum creep rate (b) and creep lifetimes (c) as a function of the initial applied stress.
Figure 2.32 – Creep curves obtained after the creep test performed at 850°C (a) and zoom at the 20% of strain and 20h (b).
Mechanical response and microstructural evolution of CMSX-4 during creep, load-free thermal exposure and non-isothermal loading.

After creep along the $\approx<011>$ direction at 400 MPa, the deformation mechanism depicted by the creep curve is not in agreement with what is observed at the specimen surface: even if there is no sign of sigmoidal creep, single slip bands and a crystalline rupture are observed (figure 2.36(b)). Similarly, the planar rupture observed after creep testing along the $\approx<111>$ crystallographic direction suggests that another deformation mechanism acts during creep along this crystal direction and that probably this does not concern primary creep. SEM observations and EBSD measurements have show in fact that mechanical twinning plays a major role in the creep response along the $\approx<011>$, the $\approx<111>$ and the $\approx<112>$ directions. Further details about the deformation mechanism are given in the next sub-section.
Figure 2.33 – Rate versus strain plots outcoming from the creep tests performed at 850°C: the whole set of curves (a), (b) zoom along B and D at the 30% of strain and (b) zoom along C and E at the 10% of strain.
Figure 2.34 – Observation of the gauge length and measure of the cross section diameter of the samples \( \simeq 011 \) (C’) tested at 850°C and: (a) 650 MPa, (b) 600 MPa. nominal diameter before test \( D_i \simeq 4\text{mm} \), Appendix A). Location of the specimens in the standard stereographic triangle (c). The arrows in the figures (a) and (b) indicate the direction of the applied stress.
Figure 2.35 – Cross section variations of the samples C' and E crept at 850°C : (a) ellipticity ($e$), (c) cross section area reduction computed using the minimum measured diameter ($S_{\text{min}}\%$) and (b) cross section area reduction computed using the maximum measured diameter ($S_{\text{max}}\%$).
Figure 2.36 – Observation of the gauge after creep at 850°C along the \( \simeq <011> \) crystal direction (C'): (a) at 500 MPa and (b) at 600 MPa. Location of the specimens in the standard stereographic triangle (c). The arrows in the figures (a) and (b) indicate the direction of the applied stress.

Figure 2.37 – SEM observations close to the fracture surfaces observed by SEM after creep along the \( \simeq <111> \) crystal orientation at 850°C: (a) at 650 MPa, (b) at 600 MPa and (c) at 500 MPa. Location of the specimens in the standard stereographic triangle (b). The arrows in the figures (a), (b) and (c) indicate the direction of the applied stress.
Figure 2.38 – Microstructure observed after 1001 h of creep along the \(\approx<111>\) (sample D) at 850°C and 350 MPa (test interrupted at 0.36 % of strain). Location of the specimen in the standard stereographic triangle (b). The arrow in the figure (a) indicates the direction of the applied stress.
2.4.1 Twinning

In the present study, twinning bands are observed after creep in the volume of the samples aligned close to the \( <011 > \) and \( <112 > \) crystallographic directions (figures 2.39, 2.40(a), 2.40(b)) and along the sample aligned close to the \( <111 > \) crystallographic direction (figures 2.40(c)). EBSD measurements are presented in the figures 2.41 and 2.43.

![Figure 2.39 – Twinning bands observed after creep along the \( \approx<011 > \) (sample C') at 850°C and 650 MPa (a). Location of the specimen in the standard stereographic triangle (c). The arrow in the figure (a) indicates the direction of the applied stress.](image)

The table 2.4 presents a summary of the tests after which twinning is observed.

<table>
<thead>
<tr>
<th>crystallographic orientation</th>
<th>T (°C)</th>
<th>( \sigma ) (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \approx&lt;011 &gt; )</td>
<td>850</td>
<td>650</td>
</tr>
<tr>
<td>( \approx&lt;011 &gt; )</td>
<td>850</td>
<td>600</td>
</tr>
<tr>
<td>( \approx&lt;011 &gt; )</td>
<td>850</td>
<td>500</td>
</tr>
<tr>
<td>( \approx&lt;011 &gt; )</td>
<td>850</td>
<td>400</td>
</tr>
<tr>
<td>( \approx&lt;111 &gt; )</td>
<td>850</td>
<td>650</td>
</tr>
<tr>
<td>( \approx&lt;111 &gt; )</td>
<td>850</td>
<td>600</td>
</tr>
<tr>
<td>( \approx&lt;112 &gt; )</td>
<td>850</td>
<td>650</td>
</tr>
<tr>
<td>( \approx&lt;112 &gt; )</td>
<td>850</td>
<td>650</td>
</tr>
</tbody>
</table>

Table 2.4 – Summary of the testing conditions after which twinning bands are observed.

The overall results suggest that a relation exist between twinning development and
— the crystallographic direction;
— the applied stress;
— stress concentration and triaxiality;

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Figure 2.40 – Post-mortem SEM observations after creep at 850°C and 650 MPa along: (a) \( \approx <011> \) direction, (b) \( \approx <112> \) direction and (c) \( \approx <111> \) direction. Location of the specimens in the standard stereographic triangle (d). The arrows in the figures (a),(b) and (c) indicate the direction of the applied stress.
— crack nucleation and propagation.
This results are presented thereafter.

SEM post-mortem observations suggest that there exist crystallographic orientations more suitable for twinning development than others. An high bands density is observed after creep in the volume of the sample aligned close to the $<011>$ crystallographic orientation (sample C', figure 2.40(a)), while few bands are observed in the volume of the specimen aligned close to $<112>$ (figure 2.40(b)). The sample aligned close to the $\simeq<111>$ is the one presenting the low density of bands. In this case bands are in fact confined in the surrounding areas of pores (figures 2.40(c) and 2.42).

A dependency of twinning on the stress magnitude has been also observed. The density of the bands and their thickness is found to decrease with the applied stress. At the lowest stresses, the twinning bands are confined next to microstructural irregularities and close to the fracture surface also after creep along the $\simeq<011>$ and $\simeq<112>$ directions. This suggests that the development of the bands is not only linked to the stress magnitude but probably also to the stress multiality and concentration.

As shown in figures 2.42 and 2.43, twinning bands are parallel to fracture surfaces. EBSD measurements (see figure 2.43) confirm that fracture develops along one of the $\{111\}$ planes, which are also the planes where twinning bands propagate. Then, twinning also influences the fracture surfaces morphology. This is in agreement with what already observed by Barba et al (2017). The link between twinning and rupture also rationalizes the discrepancies between the fracture morphology and the deformation mechanism along $\simeq<011>$ and $\simeq<111>$ previously highlighted. Figure 2.43(a) shows the examples of the $\simeq<111>$ crystal tested at 600 MPa and of the $\simeq<011>$ crystal tested at 500 MPa.

Cracks and twinning bands are also found to co-exist and to be parallel at the edges of the pores. This is shown in figure 2.44. Re-crystallization is also observed within the bands.
(a) \( \leftrightarrow \sigma \equiv E, 650 \text{ MPa} \)

(b) \( \uparrow \sigma \equiv C', 650 \text{ MPa} \)

Figure 2.41 – EBSD measurements performed after creep at 850°C along (a) \( \sim <112> \) (sample E) at 650 MPa and (b) along \( \sim <011> \) (sample C’) at 500 MPa. Location of the specimens in the standard stereographic triangle (c). The arrows in the figures (a) and (b) indicate the direction of the applied stress.
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Figure 2.42 – Twinning bands close to the fracture surface of the $\simeq<011>$ specimen (C') tested at 850°C and 400 MPa: (a) global vision and (b) zoom on the bands. Location of the specimen in the standard stereographic triangle (c). The arrows in the figures (a) and (b) indicate the direction of the applied stress.

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Figure 2.43 – EBSD measurements compared with the fracture surface planes after creep at 850°C along (a) D at 600 MPa and (b) at 500 MPa. Location of the specimens in the standard stereographic triangle (c). The arrows in the figures (a) and (b) indicate the direction of the applied stress.
Figure 2.44 – SEM post-mortem observations after creep at 850°C along $\approx<011>$ at 500 MPa: (a) twinning bands close to crack propagation (b) recrystallization in a twinning band.
Twinning nucleation – The presented results suggest that twinning mainly originates from pores. Then, more generally, a relation is assumed to exist between twinning genesis and high stress triaxiality and concentration. This is in agreement with several already existing studies, as for example with those of Fu et al (2013), Lv et al (2014), Fu et al (2014), Graverend et al (2017). In details, the studies of Fu et al and of Lv et al have been conducted on the TMS-82, TMS82+ and TMS-196 alloys subjected to out-of-phase thermomechanical fatigue. In this case, twinning bands have been found to be generated during the low temperatures phases and close to pores. In the work of Graverend et al (2017), twinning bands have been observed also at high temperatures, in the notches of a $<001>$ oriented specimen due to the local high stress triaxiality.

In the present study, a succession of planar slips and twinning bands is observed in many cases. An example is given in the figure 2.45. In agreement with what has been observed by Kakhei (Kakhei, 1999), a relation between these two phenomena exists: twinning bands form to accommodate the difference in slip between adjacent shear bands. In this sense, twinning is then favored when high stresses are applied along crystals deforming by single slip or close to stress concentration zones. This is why this is observed close to pores and crack tip in Thermo-Mechanical Fatigue (TMF) (Fu et al, 2013, Lv et al, 2014, Fu et al, 2014).

For example, a creep test along $<001>$ at 850°C and 650 MPa has been carried out by Giraud (2013a). In this case the microstructural observations have not revealed any trace of twinning but, on the contrary, at the end of the test (creep lifetimes = 185h) a $\gamma'$-rafting started is observed. In present study a further test along the $<001>$ direction (sample A) has been carried out at 750 MPa and post-mortem observations reveal no sign of shear or twinning.

In (Rae et al, 2003) the Rhenium addition has been shown to decrease the stacking fault energy of the alloy, promoting slips by $<112>$ $\{111\}$ systems and shear bands propagation. Hence, the alloy chemistry also plays an important role in the onset of twinning deformation mechanism.

In the case of the $\simeq<111>$ samples, the crystal lattice rotation also seems to play a role in twinning nucleation. In fact, even if the sample exhibits only a small rotation toward the theoretical $<111>$ (figure 2.47(c)), twinning bands are confined to high stress triaxiality zones, i.e. close to pores. Thus, locally, the crystal lattice could have performed larger rotations.

The measured crystal rotations shown in figure 2.47 are in agreement with what proposed by Mackay and Maier (1982), confirming that $<112>$ $\{111\}$ slips are activated. These measurements also confirm what already deduced about the differences observed between the behavior of the sample aligned close to the $<011>$ direction and the sample aligned close to the $<112>$ direction. The $\simeq<011>$ sample experiences a higher rotation than the $\simeq<112>$ sample (figures 2.47(b) and 2.47(c)), this this sample show an higher ovalization at the end of the creep test (see figure 2.35).

Four creep tests have been also carried out along the $\simeq<011>$ direction and interrupted at different strain levels (reported in Table 2.3). These tests are performed to further investigate the onset of twinning and its interaction with dislocations by Trans-
Mechanical response and microstructural evolution of CMSX-4 during creep, load-free thermal exposure and non-isothermal loading.

mission Electron Microscopy (TEM). At this day, TEM analysis are realized only on the sample deformed until a 5% of creep. The creep curve of this test is shown in figure 2.48. As shown in figure 2.49(a), these analysis reveal that at 5% of creep deformation twinning bands have already propagated.

Observations contained in the figures 2.49(b) and 2.49(c) point out that interactions exist between twinning bands, matrix dislocations and shear bands.

Twinning consequences on plasticity and damage – Twinning causes a 60° rotation of the lattice along \{111\} planes. Well defined zones having different crystal orientations act like grain boundaries. The single crystal character of the material is, then, locally lost. This loss involves a change in the plasticity/damage onset mechanisms and further development. From a damage point of view, the presence of grain boundaries enhances the degradation promoted by the interaction with the environment, hence oxidation contribution cloud be accelerated. In addition, since grain boundaries constitute preferential ways for the diffusion and coalescence of cavities, twinning also enhances the plastic damage kinetics. The creep lifetime reduction due to environmental interaction has been highlighted by performing a test along the \( \sphericalangle <011> \) orientation at 850°C and 400 MPa in high vacuum. Figure 2.50 shows the comparison between the creep curves in high vacuum and in the laboratory air (the non in-vacuum test has already presented in the figure 2.32). Table 2.5 presents the comparison of creep lifetimes, final strains, cross section variations and ellipticity.

<table>
<thead>
<tr>
<th></th>
<th>( t_{\text{rupt}} ) (h)</th>
<th>( \varepsilon_{\text{rupt}} )</th>
<th>( S_{\text{min}} ) %</th>
<th>( S_{\text{max}} ) %</th>
<th>( e )</th>
</tr>
</thead>
<tbody>
<tr>
<td>AL</td>
<td>514.9</td>
<td>4.38</td>
<td>−14.5</td>
<td>−3.05</td>
<td>0.06</td>
</tr>
<tr>
<td>HV</td>
<td>480</td>
<td>2.21</td>
<td>−6.88</td>
<td>−1.99</td>
<td>0.03</td>
</tr>
</tbody>
</table>

Table 2.5 – Comparison between an in-vacuum creep test performed at 850°C and 400 MPa along a \( \sphericalangle <011> \) (sample C’) with the same test realized in air laboratory. AL = Air Laboratory, HV = High-vacuum.

These results show that the material has a lightly higher creep strength when the interaction with the environment is removed, showing that oxidation is accelerated because of grain boundaries formation. However the differences in creep lifetimes is not dramatic. The reason is that, as shown in figure 2.42, at this stress there are just few and thin twinning bands close to the fracture surface. From figure 2.50, one can note that the final strain observed after testing in high vacuum is lower than the one observed after the test exposed to the air. This does not depend on any deformation mechanism change but just on the testing boundary conditions. In fact, even if twinning acts in both cases, the test in high vacuum has been performed with a rig that does not allow any rotation of the specimen’s heads, so the rotation of the crystal lattice does not develop in the same way.

Regarding plasticity, it is very well known that more complex dislocation propagation mechanisms are observed in polycrystalline aggregates. In this sense, TEM observations of the other interrupted tests will probably provide more information. A last creep test
along D orientation at 650 MPa is realized and interrupted at 5% of strain. EBSD measurements reveal that very thin bands have already formed close to pores (figure 2.51(b)). As highlighted in figure 2.51(a), the 5% of strain corresponds to the very beginning of the strain rate acceleration. This suggests that the onset of twinning is the main cause of the creep acceleration and of the high final strain.
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Figure 2.45 – Development of twinning between two slip bands observed after creep at 850°C and 650 MPa along the $\approx<112>$ direction (sample E).

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Figure 2.46 – Microstructure observed after creep along $\simeq<001>$ at 850°C and: (a) 650 MPa (from Giraud (2013a)), (b) 750 MPa. Location of the specimens in the standard stereographic triangle (c). The arrows in the figures (a) and (b) indicate the direction of the applied stress.
Mechanical response and microstructural evolution of CMSX-4 during creep, load-free thermal exposure and non-isothermal loading.

Figure 2.47 – Lattice rotations measured after creep at 850°C: (a) along C’ at 500 MPa, (b) along E at 650 MPa and (c) along D at 600 MPa.
Figure 2.48 – Creep curves of the interrupted creep tests along $\approx<011>$ at 850°C and 600 MPa.
Figure 2.49 – Bright field TEM measurements performed after creep along $\approx<011>$ at 850°C and 600 MPa (test interrupted at the 5% of strain). Location of the specimen in the standard stereographic triangle (c).
Figure 2.50 – Comparision of the creep performances at 850°C and 400 MPa in air (AL) and in high vacuum (HV) along a $\simeq<011>$ direction: (a) creep strain and (b) creep rate vs creep strain curves. Location of the specimens in the standard stereographic triangle (c).

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Mechanical response and microstructural evolution of CMSX-4 during creep, load-free thermal exposure and non-isothermal loading.

Figure 2.51 – Creep response along $\langle 111 \rangle$ at 850°C and 650 MPa: (a) creep rate vs creep strain plot of the test interrupted at 5% of strain and of the one performed at the same temperature/stress but brought to rupture, (b) EBSD measurements realized after the interrupted test. Location of the specimens in the standard stereographic triangle (b). The arrow in the figures (b) indicates the direction of the applied stress.

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
2.5 Dissolution tests

Pure thermal exposure have been performed for 1h between 950°C and 1300°C using CMSX-4 specimens. These specimens have been machined out from specimens oriented along the <001> direction and crept for 100h at 1050°C and 140 MPa. This prior creep aging has the main purpose to generate a well developed N-type rafting, so as to lose subsequent γ' stereological analyses, especially the γ' volume fraction at equilibrium \( f_{\gamma'\text{eq}} \).

The stereological analysis procedure is described in the Appendix A and details on temperatures and measurements are given in the Appendix C. Micrographs taken in the primary dendrite and in the inter-dendrite spacing are analyzed separately in order to point out the consequences of rhenium segregation. These results –shown in figure 2.52– are presented in terms of average values and of differences between the average and the maximum/minimum measurements (the latter corresponding to error bars).

![Figure 2.52 – Volume fraction of precipitates at the thermodynamic equilibrium (\( f_{\gamma'} = f_{\gamma'\text{eq}} \)) as a function of temperature. Measurements carried out in present study after dissolution tests. D = primary dendrite arm and ID = interdendritic spacing.](image)

As expected, the volume fraction of precipitates in the dendrites is lower than in the inter-dendrite spacing, and this at each temperature. These trends are in agreement to what has been observed by Burgel et al (2000) (figure 1.18). Figure 2.53 shows the microstructure observed after the aging at 950°C, at 1200°C and at 1250°C, while the figure 2.53(a) and 2.53(b) contain the SEM observations of the material aged at 1265°C and 1300°C respectively. From 950°C to 1200°C the material presents a N-rafted microstructure. Wider γ-channel are observed with the increase of temperature, according to a larger γ' dissolution. At each temperature morphological irregularities are observed in the inter-dendrites spacing, according to the chemical heterogeneity described in the section 1.1.4. A less evolved microstructure is also observed in these areas, still as a result of the rhenium seg-
Mechanical response and microstructural evolution of CMSX-4 during creep, load-free thermal exposure and non-isothermal loading.

Regeneration discussed in (Reed et al., 2007b, Arnoux, 2009). At 1250°C few remaining rafted particles are observed. At 1265°C, in the primary dendrite arms precipitates are fully dissolved in the matrix. Few rafted particles are still observed in the inter-dendrite spacings until –at 1300°C– there is no trace any more of the coherent γ/γ′ initial microstructure.

![Microstructure at different temperatures](image)

Figure 2.53 – Microstructure observed after the dissolution tests performed at (a) 950 °C, (b) 1200 °C and (c) 1250 °C.

For modeling purposes the $f_{\gamma'}$ mapping is completed by the value at room temperature (given in Appendix A) and at 850°C. The latter is measured by processing the SEM post-mortem observations of the sample A tested at 750 MPa. At this temperature no
microstructural evolution is expected. A cubical microstructure is then observed at thermodynamic equilibrium. The overall data and the trend curve used for the modeling are presented in the figure 3.7.
2.6 Conclusions

Let us now conclude in two steps, first on what has been observed and described in present chapter, second on the modeling guidelines.

2.6.1 CMSX-4 mechanical response

Creep tests performed at 1050°C confirm that in the high temperature/low stress regime, the material response is not fundamentally affected by crystal orientation and stress magnitude (when deriving the constitutive modeling this phenomenon will be denoted as the isotropization of the material behavior at high temperature). Deformation and damage mechanisms are quite the same along each crystal orientation, so similar creep lifetimes, minimal rates and primary creep strains are observed. The slight differences observed at high temperatures are correlated to the different microstructure morphologies observed along each crystallographic direction. These results are in agreement with what already been observed in literature, as for example in (Khan and Caron, 1991, Reed et al, 1999, Matan et al, 1999b, Reed, 2006, Ghighi, 2013).

Monotonic tensile tests conducted at 1050°C confirm that the strain rate dependency of microstructural evolution and of ductility manifests through the test duration. Ductility is found to vary with the strain rate also along <111> crystals but, differently from what has been observed by Giraud (2013a), in this case tensile curves show signs of the presence of strain hardening. Hardening is also observed during the complex test. In both cases TEM observations are required to identify the operating deformation mechanisms.

Non-isothermal tests confirm what has already observed in the literature review of section 2.3. Moreover, the microstructural observations did not reveal any specific damage mechanism during repeated thermal shocks and a plastic damage occurs only at the very end of the tests. Microstructural evolution is main causes of the material degradation, even during the most critical engine operative conditions.

At a low temperature the anisotropy of the creep response is found to depend on the activation of the <112> {111} secondary octahedral slip systems and on the onset of twinning. If the first allows to explain almost all the aspects of the primary creep behavior (especially along crystals close to the theoretical [001] − [102] symmetry boundary, as shown by Rae et al (2000a)), the second seems to allow to rationalize the high secondary creep, the short lifetimes, the high final strains and the fracture surface morphology observed along the most misoriented samples. However, these two phenomena seem to be closely linked, especially in alloys containing rhenium and along crystals favorable to single slips. The complex stress state close to microstructural irregularities is also suspected to play a major role in the onset of twinning. Regarding the consequences of twinning on the CMSX-4 creep response, it is pointed out that the loss of the single grain character of the material causes a damage acceleration, mainly due to the interaction with the environment. Further TEM measurements are in progress, in order to characterize the twinning origin, kinetics and impact on the plastic strain.

As expected, dissolution tests show that the volume fraction of γ′ precipitates at the
thermodynamic equilibrium decreases with temperature (a precise curve is plotted for considered the CMSX-4). In a consistent manner with the literature, the $\gamma'$-dissolution is slower in the inter-dendrite spacing because of the higher $\gamma'$-like elements (Al,Ti,Ta).

### 2.6.2 Consequences on the modeling

No classical primary creep phase is observed during the creep tests at 1050°C. An initial quasi-zero rate is followed by a strain rate acceleration caused by the evolution of the $\gamma/\gamma'$ microstructure. Primary creep seems not to exist at this temperature or, when it is observed, it is characterized by negligible primary creep strain amplitudes. For this reason, primary creep and isotropic hardening will be neglected in the high temperature modeling of creep.

The creep response along highly misoriented crystals is found to be affected by the crystal lattice rotation at the higher stresses. Hence, this rotation should be modeled, but this feature is left to further studies.

Further microstructural degradations are observed at the end of the tests: the topological inversion of the $\gamma/\gamma'$ microstructure (occurring after $\gamma'$ rafting, see 1.2.4) and the partial rotation of the rafted particle extremities. These phenomena should also be modeled to correctly reproduce the tertiary creep phase, the creep lifetimes and final strains. For the sake of a relative simplicity we will model them in a phenomenological manner and group these phenomena with voids/cracks nucleation and growth (occurring at the very end of the tests) within Rabotnov-Kachanov scalar damage variable $D$.

The strain rate dependency of ductility and of microstructural evolution should be modeled, at least both along $<001>$ (Giraud, 2013a) and $<111>$. In this last case, strain hardening might also be introduced to correctly reproduce the monotonic tensile curve at the lowest strain rate.

No additional degradation process seems necessary to reproduce the CMSX-4 bulk response under in-service (non-isothermal) critical conditions at high/very high temperature ($T>900^\circ$C). Microstructural evolution, oxidation and plastic damage are in fact the only degradation sources observed.

In order to reproduce the creep response along misoriented samples in the low temperatures/high stress regime, the modeling would need the description of the plasticity produced by $<112>\{111\}$ slips, the crystal lattice rotation and the twinning.
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Chapter 3

A tensorial thermodynamic framework for microstructural evolutions in Single Crystal Superalloys

“Sympathy for the devil”
Rolling Stones

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
3.1 Introduction

This chapter presents the modeling of visco-plasticity and microstructural evolutions developed during this work for the considered CMSX-4 alloy and, more generally, for Ni base single crystal superalloys. The first section proposes an overview of the main objectives of the modeling and of the models already existing in literature for the coupling between visco-plasticity and microstrutural evolution for this class of materials. The modeling choices made in this study—in order to achieve the modeling objectives—are presented at the end of the same section (section 3.2). In the following sections the ingredients of the proposed modeling are described and the microstructural and mechanical curves identified on CMSX-4 are presented. They make use of the experimental results presented in section 2.2. The constitutive equations being implemented in Z-set Finite Element code, examples of structural computations performed are finally given. Note that the thermodynamics framework of the model is detailed in Appendix I.

We will often use the word plasticity. From a mechanical point of view it will, in fact, mean visco-plasticity.

3.2 Modeling objectives, existing models and modeling choices

Modeling objectives – As already mentioned in the general introduction, reproducing the creep and monotonic tension responses is the first objective of the modeling, especially at 1050°C, which is an interesting temperature from both an industrial and a scientific point of view. This is the reason why the assumptions underlying the modeling are mainly formulated on the basis of the behavior of CMSX-4 at this temperature. The experiments presented in section 2.2 show that microstructural evolutions govern the mechanical response at this temperature. For this reason, the description of these evolutions and the description of their coupling with visco-plasticity are a key point in the proposed model. In particular:

— according to le Graverend et al (2017), γ′-rafting is responsible of the plastic strain acceleration during creep up to the ≃ 2−5% of deformation. As shown in the section 1.3.2, γ′-rafting is also the cause of the softening observed during monotonic tensile tests at the lower strain rates. Reproducing this acceleration and this softening by the mean of the coupling between visco-plasticity and γ channel widening is one of the primary objective of the modeling;

— the above mentioned studies and the tensile tests presented in the section 2.2.2 show that at higher strain rates, almost no evolution of the cubic microstructure is observed. This has therefore to be reproduced by the modeling;

— as pointed out in literature by Matan et al (1999a), Sakaguchi and Okazaki (2018) (section 1.2.1), the kinetics of γ′-rafting is influenced by plasticity and, if a certain amount of plastic deformation is reached, rafting proceeds even at zero load. This dependency on plastic strains has to be introduced in the γ channel widening...
evolution law.

Regarding the dependency of the mechanical response to the crystal orientation, literature studies (Ghosh et al., 1990, Pollock and Argon, 1994, Kolbe et al., 1997, Kamaraj et al., 1998, Bettge and Österle, 1999, Reed et al., 1999) have shown that plastic deformation at high temperature develops in the matrix channel following different slip mechanisms along $<001>$ and $<111>$ (see section 1.1.2) and that, along other crystallographic directions, plasticity is produced by a combination of these two deformation mechanisms:

- $<011>$ \{111\} along $<001>$;
- $<011>$ \{001\} along $<111>$.

Similarly, both the literature studies (Golubovskii et al., 1987, Khan and Caron, 1991, Pollock and Argon, 1994, Peng et al., 1996, Sass et al., 1996) and the experimental study conducted during this thesis, show that different morphological changes happen to the $\gamma'/\gamma$ microstructure during creep straining along $<001>$ and $<111>$ orientations. Moreover, the microstructural evolution observed along misoriented crystals away from these two directions are assumed to result from a combination of these two ones (section 1.2.1):

- N-rafting along $<001>$;
- mechanical coarsening along $<111>$.

Thus, including the description of these phenomena in the modeling is fundamental to describe the anisotropy of the mechanical response. This is the reason why the modeling is primarily targeted to reproduce the response along $<001>$ and $<111>$ orientations. In the modeling proposed next, one will not attempt to model a rafting phenomenon having a non zero inclination with respect to $<001>$ as sometimes induced by the multiaxiality of the loading (see Caccuri et al. (2017) for experimental evidences). This is left to further studies.

**Existing models**

There already exist many phenomenological models accounting for microstructural evolutions (Svobda and Lukas, 1996, Tinga et al., 2009b, Cormier and Cailletaud, 2010a, Gaubert et al., 2010, Fedelich et al., 2012b, Ghighi et al., 2012, Giraud, 2013a, le Graverend et al., 2014a, Fan et al., 2015, Gaubert et al., 2015). In all these models visco-plasticity is formulated at the microscale by means of the crystal plasticity framework proposed by Mandel (1973), Hill and Rice (1972), Rice (1975), Asaro and Rice (1977), Asaro (1983), Peirce et al. (1983) for FCC crystals. The softening produced by the $\gamma$ channel widening is modeled by a size reduction of the elasticity domain. This is done by introducing in the visco-plasticity criterion the Orowan shear stress (presented in the subsection 1.1.3), defined as:

$$\tau_{\text{ORO}} = \frac{\theta GB}{w_{001}}$$ \hspace{1cm} (3.1)

with for instance $\theta = \sqrt{2/3}$ obtained when calculating the Orowan stress using the $\gamma$ channels width along the [001] direction (Benyoucef et al., 1993). The value of $\theta$ was assumed to be 0.5 in Fedelich et al. (2009) and 0.85 in Tinga et al. (2009b), i.e a value close to the $\sqrt{2/3}$ considered in the work of Cormier and Cailletaud (2010a).

The models proposed by Cormier and Cailletaud (2010b), Ghighi et al. (2012), Giraud (2013a) are known in SAFRAN group as the “Polystar” models. They aim to reproduce
the material response along $<001>$ during thermal cycling loadings, fast overheatings and coolings of single crystal superalloys. These models then only account for octahedral slips and for the $\gamma'$ dissolution and re-precipitation. During isothermal loadings, the $\gamma'$ dissolution leads to a decrease of the $\gamma'$ volume fraction until the thermodynamics equilibrium is reached. From this point the $\gamma'$ volume fraction does not evolve anymore and, in these initial Polystar models, the matrix channel widths do not evolve either. Then, the continuous plastic strain acceleration due to microstructural evolutions observed by le Graverend et al (2017) is not described. An additional softening has to be introduced in these formulations in order to reproduce the non-linearity of the creep curves. This is sometimes done by coupling the damage evolution law with visco-plasticity from the very first stages of creep Rabotnov (1969), one will prefer to introduce a damage threshold (next Chapter).

The Polystar formulation has been extended by le Graverend et al (2014a) to thermo-mechanical cyclic loadings. Constitutive equations modeling the $\gamma'$ rafting and coarsening (LSW-type) have been included but an additional softening due to damage has still to be introduced even when plastic damage does not fundamentally impacts the mechanical response (le Graverend et al, 2017).

The models proposed by Svobda and Lukas (1996), Tinga et al (2009b), Gaubert et al (2010), Fedelich et al (2012b), Fan et al (2015), Gaubert et al (2015) are aimed to reproduce the material behavior under isothermal loadings. These models thus all account for $\gamma'$ rafting and coarsening. The one proposed by Svobda and Lukas (1996) has been mainly addressed to reproduce the material response along $<001>$, while the other models have been conceived to describe the material response along any crystal directions. In the work of (Tinga et al, 2009b, Fedelich et al, 2012b, Fan et al, 2015, Gaubert et al, 2015) the cubic slips contribution to visco-plasticity has been introduced to reproduce the dependency of the mechanical response on the crystal direction loaded. This formulation, initially proposed by Méric et al (1991a), has been revisited by Tinga et al (2009b) and Fedelich et al (2012b). In the work of (Tinga et al, 2009b), it has been enriched with micromechanics elements to account for precipitate shearing and dislocation recovery climb. In the work of (Fedelich et al, 2012b), the visco-plastic properties of cubic slip systems have been made dependent on the visco-plastic properties of octahedral slips. This choice has been made consistently with the micromechanics nature of pseudo-cubic slips described in section 1.1.2. In Tinga and Fedelich modeling the $\gamma'$ rafting morphology dependency on the crystal orientation is also described. Creep curves along the $<111>$ direction are shown only in (Fedelich et al, 2012b), revealing that along this direction this model does not describe the non-linearity of the creep curves.

In the work of Gaubert et al (2010, 2015), the microstructural evolution are described by the phase field approach developed by Boussinot (2009). In Gaubert et al (2010), visco-plasticity only accounts for octahedral slips. The dependency of the rafted particles morphology on the crystallographic direction solicited and on the sign of the applied stress have been both described. Even if a good agreement with creep curves has been found, the estimation of $\gamma$ channels widths is only qualitative and the model has not been totally validated along $<111>$ and $<011>$ crystallographic direction. Cubic slips have been
introduced in Gaubert et al (2015) but this study is mainly oriented to improve the pre-
vision of the final microstructural morphology after creep along the $<011>$ orientation,
thus no comparison with creep curves is presented. However, generally speaking, phase field based models still present many issues. One is that only an Al-Ni binary system is considered, so the other alloy elements contribution to the diffusion kinetics is neglected. Other two points are, for example, that the phase field approach predicts an excessive de-
crease of the $\gamma'$ volume fraction during isothermal creep and that computations are very CPU consuming, especially for 3D simulations.

Finally, among all the mentioned studies, the importance of modeling the loss of Orowan hardening during monotonic tension loading seems to have been clearly high-
lighted only in the work of Tinga (2009a). Moreover, only the models proposed by Tinga et al (2009b), Gaubert et al (2015) describe the evolution of the three $\gamma$ channels. All the other models are based on a two-dimensional approximation of the microstructure. There-
fore, in these models only the horizontal and vertical $\gamma$ channel evolution is considered (of widths $w_1$ and $w_3$, as represented in figure 1.1(b)).

In conclusion, even if constitutive models including microstructural evolutions already exist, none of them contain all the elements required to achieve the above mentioned modeling objectives. Furthermore, the most detailed models, as for example those of le Graverend et al (2014a) and of Gaubert et al (2015), imply very time consuming computa-
tions. For instance, le Graverend et al (2014a) model is based on crystal plasticity, then all the constitutive equations (including those modeling microstructural evolutions) have to be integrated when computing the (visco-)plastic strain rate on each slip system. The number of integrations at each time step thus corresponds to the number of internal vari-
ables multiplied by the number of the active slips systems. It follows that accounting for slips, for isotropic hardening, for kinematic hardening and for microstructural evolution can bring to very time consuming computations. Hence, introducing cubic slips or a three dimensional description of the microstructure in this type of formulation would be even more computationally expensive. Similarly, the Gaubert et al (2015) approach, which in-
cludes octahedral and cubic slips and which also describes the load direction dependency of $\gamma'$ rafting, is not yet adapted to Finite Element computations on three dimensional ge-
ometries.

Modeling choices – The model proposed in this work is based on a three dimensional
description of the microstructure which is presented in the figure 3.1.

As it will be presented in the section 3.4, the widening of the matrix channels is effi-
ciently modeled at an intermediate scale, so-called mesoscale. Consequently, the micro-
structural evolutions are modeled at the Representative Volume Element (RVE) scale by using the novel dimensionless internal variable $\omega$ (section 3.4.4). This variable is a symmetric second order tensor representing the state of the microstructure (cubic, rafted,
etc..). In the (cubic) Natural Anisotropy Basis (NAB) of the material (which coincides with the crystal frame as shown in figure 3.1(a)) the main diagonal of $\omega$ contains the width of the $\gamma$ channels parallel to the principal crystallographic directions of the cubic cell, say $w_i = w_{ii}$ (no sum), all normalized by the width of the $\gamma$ channels at the studied
Load-free mechanical coarsening will also be modeled. For this scope it is assumed that the homothetic growth of $\gamma'$ particles follows the LSW theory:

$$\lambda^3 - \lambda_0^3 = Ct$$  \hspace{1cm} (3.3)

where $\lambda = \lambda(t)$ and $\lambda_0 = \lambda(t = 0)$ are the microstructure periodicity (see figure 3.1 or figure 3.2) after a given exposure $t$ at high temperature and in the initial state respectively, and $C$ is the growth kinetics at a given temperature, which is assumed to be of Arrhenius type.

A tensorial evolution law of type $\dot{\omega} = \ldots$, i.e. $\omega_{ij} = \ldots$, is derived next by revisiting, modifying and extending the formulation proposed by Tinga et al (2009b) (Appendix D, see also Epishin et al (2008)). It accounts for the $\gamma'$ rafting and coarsening kinetics. Heaviside functions will be introduced as prefactors as proposed evolution law in order to model the plastic strain threshold observed by Matan et al (1999a). The other features such as the limitation of the effect of the stress level at large stresses or as the crystal orientation dependency will be detailed next point by point.

The tensorial evolution law proposed for the variable $\omega$ is described in the section 3.4.5. Concerning visco-plasticity, this evolution is modeled in a phenomenological manner directly at RVE scale, using so-called Kelvin mode based macroscopic plasticity. This choice is made for two main reasons. The first is that the evolution law for $\omega$ is naturally adapted to be coupled to a Kelvin modes based description of the material anisotropy. The second is that the crystal plasticity framework (used in the literature models described in the previous section) defines an additional scale smaller than the cuboidal microstructure and $\gamma'$-rafting scale (described in the section 1.2). Kelvin mode based visco-plasticity formulations for single crystal superalloys have already been proposed by Bertram and

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Olschewski (1996), Mahnken (2002) and by Desmorat and Marull (2011). In the present Ph.D. the later formulation is revisited and enriched to obtain a direct link with the material deformation mechanisms presented in the section 1.1.2. Similarly to crystal plasticity, two macroscopic criterion functions are introduced: a first one accounting for octahedral slips and a second one accounting for pseudo-cubic slips mainly. The softening produced by the higher dislocation mobility in the wider $\gamma$ channels is modeled by a size reduction of the elasticity domain. This reduction is obtained by introducing the Orowan stress (presented in the section 1.1.3) in the two criterion functions. This coupling, already proposed in the crystal plasticity based models mentioned in the previous paragraph, is here formulated at the macro-scale and coupled with the 3D representation of microstructural evolutions: the quadratic norm of the tensor $\omega$ takes the place of the horizontal channel width (Eq. (3.4)).

$$\sigma_{\text{ORO}} = \frac{\kappa_{\text{ORO}} G}{\|\omega\|} \quad (3.4)$$

A material parameter $\kappa_{\text{ORO}}$, to be identified, is introduced. It is temperature dependent. A sensitivity analysis to the type of norm considered (quadratic or not...) is presented in the Appendix M.

**Definitions** – In the modeling, the von Mises norm will be used for symmetric second order tensors von Mises (1928),

$$\langle . \rangle_{eq} = \sqrt{\frac{3}{2} \langle . \rangle' : \langle . \rangle'} \quad \langle . \rangle' = \langle . \rangle - \frac{1}{3} \text{tr}(\langle . \rangle) I \quad (3.5)$$

$\langle . \rangle'$ means the deviatoric part.

Due to cubic symmetry von Mises norm will not be applied to total stress tensor (see next).

### 3.3 Kelvin modes based visco-plasticity of cubic materials

In order to model the cubic anisotropy encountered for single crystal superalloys, such as CMSX-4, the elasticity framework is used to present consistent mathematical tools: harmonic decomposition (Schouten, 1951, Backus, 1970, Spencer, 1970), Kelvin stresses and projectors (Kelvin, 1856, 1878, Cowin et al, 1991, Biegler and Mehrabadi, 1995, François, 1995, Bertram and Olschewski, 1996, Arramon et al, 2000, Mahnken, 2002, Desmorat and Marull, 2011). In the following subsections their use is described and objective stresses and plastic strains dedicated to cubic material symmetry are defined.

The harmonic decomposition of cubic elasticity tensor $\mathbb{E}$ is used in order to define the Kelvin projectors with no need to perform the spectral decomposition of $\mathbb{E}$.
3.3.1 Cubic elasticity parameters

The three independent components of a cubic elasticity tensor \( E \) (of components \( E_{ijkl} \), having major and minor indicial symmetries \( E_{ijkl} = E_{klij} = E_{jikl} \)) are Young’s modulus \( E \), Poisson’s ratio \( \nu \) and the shear modulus \( G \neq \frac{E}{2(1+\nu)} \), the bulk modulus being \( K = \frac{E}{3(1-2\nu)}. \)

In Natural Anisotropy (of figure 3.2):

\[
\begin{align*}
E_{1111} = E_{2222} = E_{3333} &= \frac{(1-\nu)E}{1 - \nu - 2\nu^2} \\
E_{1122} = E_{1133} = E_{2233} &= \frac{\nu E}{1 - \nu - 2\nu^2} \\
E_{1212} = E_{1313} = E_{2323} &= G
\end{align*}
\]

(3.6)

The other \( E_{ijkl} \) are either obtained from the indicial symmetries or are equal to zero.

3.3.2 Harmonic decomposition of cubic elasticity tensor

An elasticity tensor belonging to the cubic symmetry class can be recast as

\[
E = 2\mu \mathbb{I} + \lambda \mathbb{1} \otimes \mathbb{1} + \mathcal{H}
\]

\[
\text{tr} \mathcal{H} = \text{tr} \mathcal{H} = 0
\]

(3.7)

where the fourth order part \( \mathcal{H} = \mathcal{H}(E) \) is harmonic, i.e. is both

- totally symmetric: having the major and minor indicial symmetries of the elasticity tensor and the additional Cauchy symmetry \( H_{ijkl} = H_{kijl} \),
- traceless: \((\text{tr}_{12} \mathcal{H})_{ij} = H_{kij} = 0, (\text{tr}_{13} \mathcal{H})_{ij} = H_{kik} = 0\).

Eq. (3.7) is the harmonic decomposition of fourth order tensor \( E \) (Backus, 1970, Cowin et al, 1991, Baerheim, 1993, Forte and Vianello, 1996). Generalized Lamé constants \( \lambda, \mu \) are two invariants of the elasticity tensor: they are functions of the invariants \( \text{tr}(\text{tr}_{12} E) \) and \( \text{tr}(\text{tr}_{13} E) \),

\[
\begin{align*}
\lambda &= \frac{1}{15} \left( 2 \text{tr}(\text{tr}_{12} E) - \text{tr}(\text{tr}_{13} E) \right) \\
\mu &= \frac{1}{50} \left( -3 \text{tr}(\text{tr}_{12} E) + 3 \text{tr}(\text{tr}_{13} E) \right)
\end{align*}
\]

(3.8)

Practical expressions are given in the Appendix F. Note that \( \mathcal{H} = 0 \) is equivalent to \( \mu = \frac{E}{2(1+\nu)} = G \), i.e. to isotropic elasticity.

3.3.3 Kelvin stresses – Kelvin projectors

A cubic elasticity tensor \( E \) and, in an equivalent manner, its invert \( E^{-1} \) have three orthogonal eigentensors: the hydrostatic stress tensor \( \sigma^H \), and two deviatoric stress tensors \( \sigma^d \) and \( \sigma^\tau \) (\( \sigma^d \) diagonal in Natural Anisotropy Basis (shown in figure 3.1(a)), deviatoric, \( \sigma^\tau \) out-of-diagonal in this basis, also deviatoric), such as

\[
E : \sigma^H = \frac{E}{1-2\nu} \sigma^H, \quad E : \sigma^d = \frac{E}{1+\nu} \sigma^d, \quad E : \sigma^\tau = 2G \sigma^\tau
\]

(3.9)

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with the orthogonality properties \( \sigma^d : \sigma^H = \sigma^d : \sigma^\overline{d} = 0 \) and the deviatoric and total stresses partitions

\[
\sigma = \sigma^d + \sigma^\overline{d} \quad \text{and} \quad \sigma = \sigma^d + \sigma^\overline{d} + \sigma^H
\]  

(3.10)

Such partitions are objective in case of cubic material symmetry (see Bertram and Olschewski (1996)).

The second order tensors \( \sigma^H, \sigma^d \) and \( \sigma^\overline{d} \) are the so-called Kelvin stresses, associated with Kelvin moduli \( \frac{E}{1-2\nu} = 3K, \frac{E}{1+2\nu} = 2G \) (the three eigenvalues of \( E \), Kelvin (1856, 1878), Cowin et al (1991)). They are obtained thanks to Kelvin fourth order projectors \( \mathbb{P}^H, \mathbb{P}^d, \mathbb{P}^\overline{d} \) (François, 1995),

\[
\mathbb{P}^H : \sigma = \sigma^H = \frac{1}{3} tr \sigma \mathbf{1} \\
\mathbb{P}^d : \sigma = \sigma^d \\
\mathbb{P}^\overline{d} : \sigma = \sigma^\overline{d}
\]

(3.11)

with \( \mathbb{P}^H = \frac{1}{3} \mathbf{1} \otimes \mathbf{1} \) standard hydrostatic projector. The two other projectors can be build directly from the harmonic part \( \mathbb{H} = E - 2\mu \mathbb{I} - \lambda \mathbf{1} \otimes \mathbf{1} \) of cubic elasticity tensor as

\[
\mathbb{P}^d = \frac{1}{5} \left( \mathbb{J} + \frac{\mathbb{H}}{\mu - G} \right), \quad \mathbb{P}^\overline{d} = \mathbb{J} - \mathbb{P}^d, \quad \mathbb{J} = \mathbb{I} - \frac{1}{3} \mathbf{1} \otimes \mathbf{1}
\]

(3.12)

with \( \mu \neq G \) and \( \mathbb{H} \neq 0 \) for cubic symmetry class and \( \mathbb{P}^H + \mathbb{P}^d + \mathbb{P}^\overline{d} = \mathbb{I} \).

The Kelvin stresses can be used to particularize Hill equivalent stress to cubic symmetry as writing in Natural Anisotropy Basis

\[
\sigma_{\text{Hill}} = \sqrt{\frac{1}{2} \left[ (\sigma_{11} - \sigma_{22})^2 + (\sigma_{22} - \sigma_{33})^2 + (\sigma_{33} - \sigma_{11})^2 \right] + \frac{3}{2} h^2 (\sigma_{12}^2 + \sigma_{21}^2 + \sigma_{13}^2 + \sigma_{31}^2 + \sigma_{23}^2 + \sigma_{32}^2)}
\]

is equivalent to intrinsic writing

\[
\sigma_{\text{Hill}} = \sqrt{\frac{3}{2} \left( \sigma^d : \sigma^d + h^2 \sigma^\overline{d} : \sigma^\overline{d} \right)} = \sqrt{(\sigma^d)^2_{eq} + h^2(\sigma^\overline{d})^2_{eq}}
\]

(3.13)

(3.14)

with \( h \) a material parameter (\( h = 1 \) for isotropy, von Mises stress recovered). Following notations are used next for the particular von Mises norms

\[
\sigma_{eq}^d = (\sigma^d)^2_{eq} = \sqrt{\frac{3}{2} \sigma^d : \sigma^d} \quad \sigma_{eq}^\overline{d} = (\sigma^\overline{d})^2_{eq} = \sqrt{\frac{3}{2} \sigma^\overline{d} : \sigma^\overline{d}}
\]

(3.15)

with in Natural Anisotropy Basis (figure 3.1)

\[
\begin{cases}
\sigma_{eq}^d = \sqrt{\frac{1}{2} \left[ (\sigma_{11} - \sigma_{22})^2 + (\sigma_{22} - \sigma_{33})^2 + (\sigma_{33} - \sigma_{11})^2 \right]} \\
\sigma_{eq}^\overline{d} = \sqrt{\frac{3}{2} [\sigma_{12}^2 + \sigma_{21}^2 + \sigma_{13}^2 + \sigma_{31}^2 + \sigma_{23}^2 + \sigma_{32}^2 + \sigma_{13}^2]}
\end{cases}
\]

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The non quadratic norm (expressed in Natural Anisotropy Basis)
\[ \|\mathbf{\sigma}^d\|_a = 3 \left( |\sigma_{12}|^a + |\sigma_{21}|^a + |\sigma_{13}|^a + |\sigma_{31}|^a + |\sigma_{23}|^a + |\sigma_{32}|^a \right)^{1/a} \] (3.17)
is a generalization of equivalent stress \( \mathbf{\sigma}_{eq}^d \) such as i) \( \mathbf{\sigma}_{eq}^d = \|\mathbf{\sigma}^d\|_{a=2} \), ii) \( \|\mathbf{\sigma}^d\|_a = 0 \) for tension along the crystallographic orientation \(<001>\) and iii) \( \|\mathbf{\sigma}^d\|_a = \mathbf{\sigma} \) for tension along the crystallographic orientation \(<111>\).

### 3.3.4 Objective plastic strains from Kelvin projectors

The Kelvin projectors allow to define different strain tensors (total, elastic and plastic) dedicated to cubic symmetry class (François, 1995). When applied to the deviatoric plastic strain tensor they define in an objective manner two deviatoric tensors \( \mathbf{\varepsilon}^p_d \) and \( \mathbf{\varepsilon}^p_{\overline{d}} \) as
\[ \mathbf{\varepsilon}^p_d = \mathbb{P}^d : \mathbf{\varepsilon}^p \quad \text{and} \quad \mathbf{\varepsilon}^p_{\overline{d}} = \mathbb{P}^{\overline{d}} : \mathbf{\varepsilon}^p \] (3.18)
such as the partition \( \mathbf{\varepsilon}^p = \mathbf{\varepsilon}^p_d + \mathbf{\varepsilon}^p_{\overline{d}} \) is objective for cubic symmetry class. In such a plastic strain partition \( \mathbf{\varepsilon}^p_d \) is the diagonal part of \( \mathbf{\varepsilon}^p \) in Natural Anisotropy Basis (figure 3.1(a)), \( \mathbf{\varepsilon}^p_{\overline{d}} \) is the out-of-diagonal part of \( \mathbf{\varepsilon}^p \) in this basis, with the orthogonality property \( \mathbf{\varepsilon}^p_d : \mathbf{\varepsilon}^p_{\overline{d}} = 0 \).

In a consistent manner (see Desmorat and Marull (2011)), we define two accumulated plastic strains
\[ p^d = \int \sqrt{\frac{2}{3} \mathbf{\varepsilon}^p_d : \mathbf{\varepsilon}^p_d} \, dt \quad \text{and} \quad p_{\overline{d}} = \int \sqrt{\frac{2}{3} \mathbf{\varepsilon}^p_{\overline{d}} : \mathbf{\varepsilon}^p_{\overline{d}}} \, dt \] (3.19)
Usual (isotropic) accumulated plastic strain
\[ p = \int \sqrt{\frac{2}{3} \mathbf{\varepsilon}^p : \mathbf{\varepsilon}^p} \, dt \] (3.20)
is such as
\[ \dot{p} = \sqrt{\left( \dot{p}^d \right)^2 + \left( \dot{p}_{\overline{d}} \right)^2} \] (3.21)

### 3.3.5 Stresses and plastic strains for tensions along the \( <001> \) and along \( <111> \) crystallographic directions

The Kelvin stresses and strains decouple the constitutive equations for two particular loading often used for the identification of crystal plasticity of cubic superalloys (uniaxial loading along the \( <001> \) and \( <111> \) crystallographic orientation):
- \( \mathbf{\sigma}^d = \mathbf{\sigma}' \), \( \mathbf{\sigma}^\overline{d} = \mathbf{0} \), \( \mathbf{\varepsilon}^{\text{nd}} = \mathbf{\varepsilon}^p \) and \( \mathbf{\varepsilon}^{\text{nd}} = \mathbf{0} \) for tension along \( <001> \), for which \( (\mathbf{\sigma}^d)_{eq} = \mathbf{\sigma}_{eq} = \mathbf{\sigma} \), \( p^d = p \), \( p_{\overline{d}} = 0 \).
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- \( \sigma^d = 0, \sigma^\Delta = \sigma', \epsilon^{pd} = \epsilon^p \) and \( \epsilon^{p'd} = 0 \) for tension along \(< 111>\), for which \( (\sigma^\Delta)_{eq} = \sigma_{eq} = \sigma, p^d = 0, p^\Delta = p \).

This decoupling allows to represent independently – i.e. with two different subsets of material parameters – by a single multiaxial model the uniaxial responses of cubic superalloys both along the \(< 001>\) crystallographic orientation and along the \(< 111>\) crystallographic orientation (Desmorat and Marull, 2011).

3.3.6 Macroscopic Kelvin mode based cubic plasticity

For cubic material symmetry, the yield criterion is generally a function of the 3 Kelvin modes/stresses: \( \sigma^d \) (the diagonal deviatoric part of stress tensor in Natural Anisotropy Basis), \( \sigma^\Delta \) (the out of diagonal deviatoric part of stress tensor in Natural Anisotropy Basis) and hydrostatic stress \( \sigma^H = \sigma_{H111} = \frac{1}{3} \text{tr} \sigma \). Incompressible plasticity is assumed so that generic yield criterion does not depend on the hydrostatic stress, it writes

\[
f = f(\sigma^d, \sigma^\Delta) \quad f < 0 \rightarrow \text{elasticity}
\] (3.22)

Examples of such yield criteria for CMSX-4 and AM1 superalloys are given in Desmorat and Marull (2011) (see also section 3.3.6). Note just that in order to recover a non homogeneous field in torsion, out of diagonal Kelvin mode \( \sigma^\Delta \) has to act by means of cubic non quadratic norm for out of diagonal stress tensor (components in Natural Anisotropy Basis), with \( a \neq 2 \).

Normality combined with Kelvin projectors (E.5) gives plastic strain rate (Kelvin) partition

\[
\dot{\epsilon}^p = \lambda \frac{\partial f}{\partial \sigma} = \dot{\epsilon}^{pd} + \dot{\epsilon}^{p'd} \quad \left\{ \begin{array}{l}
\dot{\epsilon}^{pd} = \mathbb{P}^d : \dot{\epsilon}^p = n^d \dot{p}^d \\
\dot{\epsilon}^{p'd} = \mathbb{P}^\Delta : \dot{\epsilon}^p = n^\Delta \dot{p}^\Delta
\end{array} \right.
\] (3.23)

with \( \lambda \) the plastic multiplier.

It has been shown by Desmorat and Marull (2011) that the accumulated plastic strain \( p^d \) is representative of octahedral slip systems plasticity and that \( p^\Delta \) is representative of cubic slip systems plasticity (see Eq. (3.19)). One has:

- \( p^d = p, p^\Delta = 0 \) for tension/compression in direction \(< 001>\),
- \( p^d = 0, p^\Delta = p \) for tension/compression in direction \(< 111>\).

For cubic symmetry still, using (quadratic) von Mises norms \( \sigma_{eq}^d, \sigma_{eq}^\Delta \) and accumulated plastic strains \( p^d, p^\Delta \), Hill cubic criterion function and associated equivalent stress and accumulated plastic strain are simply

\[
f = \sigma_{Hill} - \sigma_y \quad \sigma_{Hill} = \sqrt{(\sigma_{eq}^d)^2 + h^2(\sigma_{eq}^\Delta)^2} \quad p^{Hill} = \int \sqrt{(p^d)^2 + h^2(p^\Delta)^2} \, dt
\] (3.24)

with \( \sigma_y = \sigma_{y001} \) the yield stress in \(< 001>\) direction and \( h \) Hill material parameter for cubic plasticity. Plastic power is then (Hill, 1950):

\[
\sigma : \dot{\epsilon}^p = \lambda \frac{\partial f}{\partial \sigma} = \sigma_{Hill} \dot{p}^{Hill}
\] (3.25)

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with $\dot{\lambda} = \dot{p}_{\text{Hill}}$.

Non quadratic cubic yield criterion functions have been identified in Desmorat and Marull (2011) for CMSX-2 superalloy,

$$f = \left( \frac{\sigma_{\text{eq}}}{\sigma_y} \right)^n + \left( \frac{\|\sigma^d\|_a}{\sigma_y} \right)^m - 1 \quad (3.26)$$

using for "diagonal" deviatoric stress tensor (quadratic) von Mises norm $\sigma_{\text{eq}}^d$ and for "out of diagonal" stress tensor non quadratic norm $\|\sigma^d\|_a$ (components in cubic Natural Anisotropy Basis). This choice allows to recover a non homogeneous stress field in torsion (see Nouailhas and Cailletaud (1995) for experimental evidences), as shown in the Appendix H. Material parameter $\sigma_y = \sigma_{001}$ is the yield stress in direction $<001>$. The yield stress for tension along the crystallographic direction $<111>$ is $\sigma_{111} = \sigma_{y111}$.

When the value $a = 2$ is considered for non quadratic norm $\|\cdot\|_a$, one gets $\|\sigma^d\|_a=2 = \sigma_{eq}^d$ then equal to von Mises norm of “out of diagonal” $\sigma^d$. Note that to take $a = 2$ simplifies the writing –for instance of the normality law– and as long as pure shear is not encountered this will be the choice made.

### 3.3.7 Two criterion function macroscopic modeling

In order to obtain a close link with Schmid based single crystal plasticity (see section 3.9), multi-criterion by nature, an alternative two criterion framework can be used, defined at RVE macroscopic scale by Cowin et al (1991), Bertram and Olschewski (1996), Mahnken (2002). We introduce

—— a first criterion function $f_1 = f_1(\sigma^d, \sigma^d)$, mainly function of “diagonal” deviatoric stress $\sigma^d$ representative of octahedral slip plasticity, also function of “out of diagonal” $\sigma^d$ (this choice will be justified in section 3.9),

—— a second criterion function $f_2 = f_2(\sigma^d)$ only function of ”out of diagonal” $\sigma^d$, representative of cubic slip plasticity.

For multiaxial loading cases, including tension-torsion testing, consider Hill criterion for $f_1$ (introducing parameter $h$ within definitions Eq. (3.24)),

$$f_1 = \sigma_{\text{Hill}} - \sigma_y \quad f_2 = \|\sigma^d\|_a - \sigma_y \quad \text{(hardening omitted)} \quad (3.27)$$

For the sake of simplicity $h = 0$ and $a = 2$ are set to retrieve the von Mises criterion (3.15) for non deviatoric stress tensor $\sigma^d$,

$$f_1 = \sigma_{eq}^d - \sigma_y \quad f_2 = \sigma_{eq}^d - \sigma_y \quad \text{(hardening omitted)} \quad (3.28)$$

Normality law then becomes

$$\dot{\varepsilon}^p = \dot{\varepsilon}_1^p + \dot{\varepsilon}_2^p \quad \begin{cases} \dot{\varepsilon}_1^p = \lambda_1 \frac{\partial f_1}{\partial \sigma} = \dot{p}_1^d \mathbf{n}^d + \dot{p}_1^d \mathbf{n}^d \\ \dot{\varepsilon}_2^p = \lambda_2 \frac{\partial f_2}{\partial \sigma} = \dot{p}_2^d \mathbf{n}^d \end{cases} \quad (3.29)$$

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with the two independent (visco-)plastic multipliers equal to \( \dot{\lambda}_1 = \dot{p}_1^{\text{Hill}} = \sqrt{(\dot{p}_1^d)^2 + \frac{1}{K_N}(\dot{p}_2^d)^2} \), \( \dot{\lambda}_2 = \dot{p}_2^d \).

As in case of Schmid single crystal visco-plasticity (see for example the formulation proposed by Méric et al. (1991a)), two different viscous stresses are introduced so that visco-plasticity evolution corresponds to:

\[
\dot{p}_1^{\text{Hill}} = \left\langle \frac{f_1}{K_N} \right\rangle^N, \quad \dot{p}_2 = \left\langle \frac{f_2}{K_N} \right\rangle^N
\]

(3.30)

with \( K_N, N, K, N \) Norton material parameters in cubic visco-plasticity.

For an uniaxial loading along the \(<001>\) crystallographic orientation Norton law is recovered. \( \sigma_y \) is the yield stress \( \sigma_{y001} \) along the crystallographic direction \(<001>\),

\[
|\dot{\varepsilon}^p| = \left\langle \frac{|\sigma - \sigma_y|}{K_N} \right\rangle^N \quad \text{(along the } <001> \text{ crystallographic direction, hardening omitted)}
\]

(3.31)

For an uniaxial loading along the \(<111>\) crystallographic direction a double viscosity model is obtained (corresponding isotropic framework given in de Bussac (1993)), as well as for Schmid single crystal visco-plasticity modeling when slip planes with different Schmid factors are activated,

\[
|\dot{\varepsilon}^p| = h \left\langle \frac{h|\sigma - \sigma_y|}{K_N} \right\rangle^N + \left\langle \frac{|\sigma - \sigma_y|}{K_N} \right\rangle^N \quad \text{(direction } <111> \text{, hardening omitted)}
\]

(3.32)

\( \sigma_y \) is the yield stress \( \sigma_{y111} \) in direction \(<111>\) when it is lower than \( \sigma_y/h \).

### 3.3.8 Saturating visco-plasticity law

The validity domain of the Norton’s law is often limited in terms of strain rate. Most often it applies properly in creep regime but less in tension at \( \dot{\varepsilon} \geq 10^{-3} \text{s}^{-1} \) and in dynamics cases of impacts for which a saturation of the viscous stress occurs. This can be simply modeled by means of power-exponential viscosity law, formulated like the one proposed in Eq. (3.33). For criterion function \( f_1 \), it reads:

\[
\dot{p}_1^{\text{Hill}} = \left\langle -\sigma_{v00} \ln \left( 1 - \frac{f_1}{\sigma_{v00}} \right) \right\rangle^N \quad \text{or} \quad \sigma_{v1} = \sigma_{v00} \left( 1 - \exp \left( -\frac{K_N}{\sigma_{v00}} \frac{\dot{p}_1^{\text{Hill}}}{N} \right) \right)
\]

(3.33)

Eq. (3.33) gives well a saturating viscous stress \( \sigma_{v1} \rightarrow \sigma_{v00} \) at high strain rate (such as \( f_1 = \sigma_{v1} \) in visco-plasticity). \( K_N, N \) are previous Norton’s parameters (identified along the \(<001>\) crystallographic direction at low strain rate), saturation value \( \sigma_{v00} \) is a material parameter, temperature dependent. At low strain rate one recovers \( \dot{p}_1^{\text{Hill}} \approx \left\langle \frac{f_1}{K_N} \right\rangle^N, \sigma_{v1} \approx K_N(\dot{p}_1^{\text{Hill}})^{1/N} \).
3.4 Tensorial variable and evolution law for microstructural evolutions

The microstructure evolution under a tensile load along [001] orientation –the crystallographic orientation taken as frame direction 1– is illustrated schematically in Fig. 3.2. The initial microstructure is assumed perfectly cubic. Geometric lengths \( w_i \) and \( \ell_i \) with \( i = 1, 2, 3 \) are respectively the widths of the three matrix channels along the direction \( i \) and the size of the \( \gamma' \) precipitates. The direction [001] \( \equiv 1 \) is usually assumed to coincide with the centrifugally stressed axis of blades. Thus, \( w_1 \) is the width of the \( \gamma \) channel normal to the loading axis. Similarly, \( \ell_1 \) is the size of the precipitates along the loading direction. Quantities \( w_2 \) and \( w_3 \), as well as \( \ell_2 \) and \( \ell_3 \), are the channel widths and the precipitates sizes measured along transverse directions \( [100] \equiv 2 \) and \( [010] \equiv 3 \). \( \lambda = \lambda_1 \) is finally defined the vertical periodicity of the microstructure.

Loading the material in tension along the [001] crystallographic direction, the \( \gamma' \) rafting proceeds towards the widening of the \( w_1 \) channels and the horizontal coalescence of the precipitates. Thus, \( w_2 \) and \( w_3 \) channel widths tend to become thinner (Fig. 3.2-2) until they vanish (Fig. 3.2-3). At the same time, the vertical size of precipitates decreases while the horizontal one increases. While the directional coalescence proceeds, the coarsening proceeds too. Thus, the final microstructure presents only non-zero vertical channel width \( w_1 \) and wider horizontal channels as well as an increased vertical periodicity.

The main question concerns the nature of thermodynamics variables for rafting. Do we need to choose a vectorial or a tensorial description of the microstructure evolution? For instance, it is easier to consider the \( w_i \) and \( \ell_i \) as principal values of second order tensors \( w_{ij} \) and \( \ell_{ij} \) (of general components \( w_{ij}, \ell_{ij} \)) rather than vectors, in order to introduce the Natural Anisotropic Basis –cubic, denoted next NAB and presented in the figure 3.1– of the single crystal within the mechanical modeling (see section 3.4.3).

3.4.1 Fedelich and coworkers variables for \( \gamma' \) rafting

Let us first summarize some key points present in Fedelich et al (2009, 2012a) concerning the status of the chosen variables for the mechanical description of \( \gamma' \) rafting.

At thermodynamics equilibrium, i.e. at low enough thermomechanical loading rate, the \( \gamma' \) precipitates volume fraction \( f_\gamma \) can be considered as a function of absolute temperature \( T \) only, with no delay (see Harada et al (1990), Royer et al (1998), Roebruck et al (2001), Eq. (3.34) plotted in Fig. 3.7 for \( f_\gamma^0 = 0.7 \) and two values of exponent \( n \),

\[
f_\gamma = f_\gamma^{eq}(T) = f_\gamma^0 \left[ 1 - \langle T^* \rangle_n^+ \right] \quad T^* = \frac{T - T_E}{T_S - T_E}
\]

with \( f_\gamma^{eq}(T) \) the equilibrium volume fraction function.

The increase of the periodicity length \( \lambda \) – measured along [001] tensile direction – has been found independent of the stress level for all temperatures. In isothermal cases, it is represented by a diffusion type function of temperature \( T \) and time \( t \) (\( \lambda_{001}^0 \): initial periodicity, \( Q_\lambda \): activation energy, \( \tau_\lambda \): characteristic time, \( k_B \): Boltzmann constant, exponent
Figure 3.2 – Microstructural evolution: 1) initial microstructure ($w_1 = w_2 = w_3 = w_{001}$), 2) intermediate microstructure ($w_1 > w_2 = w_3$), 3) rafted and coarsened microstructure ($w_1 > w_{001}, w_2 = w_3 = 0$).

$\lambda \approx 3$,

$$\lambda_{[001]} = \lambda(t, T) = \lambda_{001}^{0} \left[ 1 + \exp \left( -\frac{Q_{\lambda}}{k_{B}T} \frac{t}{T_{\lambda}} \right) \right]^{1/a_{\lambda}}$$  (3.35)

Eq. (3.35) generalizes Eq. (3.3). In such a modeling, the volume fraction $f'_{\gamma} = f'_{\gamma_{eq}}(T)$ is not considered as a thermodynamics variable, neither is explicitly the periodicity length $\lambda$. One can nevertheless remark that $\lambda$ satisfies the differential equation

$$\dot{\lambda} = \left( \lambda_{001}^{0} \right)^{a_{\lambda}} \lambda^{1-a_{\lambda}} \exp \left( -\frac{Q_{\lambda}}{k_{B}T} \right)$$  (3.36)

which can be interpreted as an evolution law of thermodynamics of irreversible processes, periodicity length $\lambda$ taken then the status of a state (internal) variable (Lemaitre and Chaboche, 1991).

$\gamma'$-rafting for tensile (creep) loading in principal orientation $<001>$ is mainly studied in literature and a dimensionless variable $\xi$, this time considered as a thermodynamics

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state variable, is defined in order to characterize the type of γ'-rafting/coarsening change in microstructure (Fedelich et al, 2006),

\[ \xi = \frac{w - w_{\text{cube}}}{w_{\text{raft}} - w_{\text{cube}}} \left\{ \begin{array}{l} w_{\text{cube}} = \lambda(t, T) \left( 1 - f_{\gamma eq}^{1/3}(T) \right) \\ w_{\text{raft}} = \lambda(t, T) \left( 1 - f_{\gamma eq}(T) \right) \end{array} \right. \]  

(3.37)

In Fedelich and co-workers works, the index of γ'-rafting \( \xi \) is considered as the single thermodynamics variable accounting for γ'-rafting in the constitutive modeling. The combined effects of γ' precipitates volume fraction (through temperature) and of periodicity length (through time and temperature) are taken into account within single definition (3.37) for \( \xi \) (within \( w_{\text{cube}} \) and \( w_{\text{raft}} \)).

### 3.4.2 Tinga et al variables for γ' rafting (Tinga et al, 2009b)

For the geometrical-mechanical description of γ' rafting Tinga et al use as internal variables the 3 dimensions \( \ell_1, \ell_2, \ell_3 \) of large γ' precipitates, of initial values \( \ell_i(t = 0) = \ell_0 \), for instance in an evolution law of the form

\[ \dot{\ell}_i = -\frac{\ell_i}{\tau_\ell} \frac{3}{2} \frac{\sigma_{ii}}{\sigma_{eq} + \sigma_e} \exp \left( -\frac{Q_\ell - \sigma_{eq} U}{k_BT} \right) \]  

(no sum over \( i \))  

(3.38)

with \( \sigma_{eq} \) von Mises equivalent stress, \( \sigma_{ii} = \sigma_{ii}^d \) the diagonal components of deviatoric stress tensor \( \sigma' \) in the cube Natural Anisotropy Basis. Material parameters are γ'-rafting characteristic time \( \tau_\ell \), numerical parameter \( \sigma_e \) (small, it avoids division by zero), activation energy \( Q_\ell \), function \( U = U(T) \). In Eq. (3.38) the multiplication by length \( \ell_i \) enforces automatically a constant volume of γ' precipitates \( V_{\gamma'} = \ell_1 \ell_2 \ell_3 = \ell_0^3 \), feature observed in experiments (Chen and Immarigeon, 1998, Ma et al, 2008).

A time/temperature diffusion like evolution is considered (Eq. (3.35) with \( a_\lambda = 3 \)), with the assumption \( \lambda_1 = \lambda_2 = \lambda_3 = \lambda(t, T) \) of an isotropic periodicity change, with the same remark as in previous section 3.4.1 on the thermodynamics status of variable \( \lambda \). The volume fraction of precipitates is also not an additional variable, it is \( f_{\gamma'} = \ell_1 \ell_2 \ell_3 / \ell_0^3 \). Finally, the widths \( w_i \) of the 3 orthogonal γ channels are determined from the unit cell geometry, \( w_i = \lambda - \ell_i \). They are not additional internal variables neither.

### 3.4.3 Tensorial variables for γ'-rafting

**Variables ℓ and e –** One can first consider that the large precipitates dimensions \( \ell_i \) are the components of a vector \( \ell = (\ell_1, \ell_2, \ell_3)^T \). This point of view is not pertinent in misoriented loading (i.e. out of the [001] crystallographic orientation) cases for which the rotation of the crystal (therefore of the Natural Anisotropy Basis) may occur (Mackay and Maier, 1982, Arakani et al, 1998, MacLachlan et al, 2001, Staroselsky et al, 2010, Ghighi, 2013, Stinville et al, 2015). This additional information is present within a 3 × 3 matrix or a second order tensor considered then as one of the pertinent internal variable for γ'-rafting. If \( \ell \) is chosen as a tensor principal values of such a variable \( \ell \) – which can
be diagonalized if chosen symmetric – are the large $\gamma'$ precipitates sizes $\ell_i$ in a possibly rotated basis.

Evolution law (3.38) for $\gamma'$-rafting can be rewritten in a tensorial form

$$\dot{\mathbf{e}} = -\frac{1}{\tau} \frac{3}{2} \frac{\mathbf{\sigma}^d}{\sigma_{eq} + \sigma_e} \exp \left( -\frac{Q \ell - \sigma_{eq} U}{k_B T} \right) \quad \ell = \ell_0 \exp(\mathbf{e})$$

(3.39)

where $\mathbf{\sigma}^d = \mathbb{P}^d : \mathbf{\sigma}$, with $\mathbb{P}^d$ a Kelvin projector, is diagonal deviatoric part of stress tensor $\mathbf{\sigma}$ in Natural Anisotropy Basis. The definition of symmetric second order tensor $\mathbf{e} = \ln(\ell/\ell_0)$ allows to recover constant volume of $\gamma'$ precipitates (from $\text{tr} \mathbf{\sigma}^d = 0$, $\text{tr} \mathbf{e} = 0$) as

$$V_\gamma = \det \ell = \ell_0^3 \det(\exp(\mathbf{e})) = \ell_0^3 \epsilon^{\text{tr}} = \ell_0^3$$

(3.40)

**Tensorial description of the matrix and microstructure** – The complementary quantities allowing for the geometric description of the evolving microstructure are the $\gamma$ channels widths $w_i$ and the periodicities $\lambda_i$ initially in the 3 natural anisotropy directions $1 \equiv [001]$, $2 \equiv [100]$, $3 \equiv [010]$. This means that two additional tensorial variables for the description of $\gamma'$-rafting are $\gamma$ the channel width tensor $\mathbf{w}$ and the periodicity tensor $\mathbf{\lambda}$ (assumed isotropic, $\mathbf{\lambda} = \lambda \mathbf{1}$, in Tinga et al (2009b)). Both $\mathbf{w}$ and $\mathbf{\lambda}$ are assumed symmetric second order tensors (see K). When the principal basis remains the initial Natural Anisotropy Basis:

$$\mathbf{w} = \begin{bmatrix} w_1 & 0 & 0 \\ 0 & w_2 & 0 \\ 0 & 0 & w_3 \end{bmatrix}_{\text{NAB}} \quad \mathbf{\lambda} = \begin{bmatrix} \lambda_1 & 0 & 0 \\ 0 & \lambda_2 & 0 \\ 0 & 0 & \lambda_3 \end{bmatrix}_{\text{NAB}}$$

(3.41)

The three tensors $\ell$, $\mathbf{w}$ and $\mathbf{\lambda}$ are not independent:

$$\ell + \mathbf{w} = \mathbf{\lambda}$$

(3.42)

The $\gamma'$ precipitates volume fraction is then not an independent variable either, it is :

$$f_\gamma = \frac{\ell_1 \ell_2 \ell_3}{\lambda_1 \lambda_2 \lambda_3} = \frac{(\lambda_1 - w_1)(\lambda_2 - w_2)(\lambda_1 - w_2)}{\lambda_1 \lambda_2 \lambda_3}$$

(3.43)

definition which can be generalized to complex $\gamma'$-rafting cases as intrinsic expression

$$f_\gamma = \frac{\det \ell}{\det \mathbf{\lambda}} = \frac{\det(\mathbf{\lambda} - \mathbf{w})}{\det \mathbf{\lambda}}$$

(3.44)

even when second order tensors $\mathbf{\lambda}$ and $\mathbf{w}$ do not remain coaxial.

### 3.4.4 On the choice of internal variables

The question of the choice of the best set of thermodynamic internal variables arises, $a)$ for the geometric description of $\gamma'$-rafting, $b)$ for the mechanical consequences of $\gamma'$-rafting. In case $a)$ the consideration of two tensorial variables among the three $\ell$, $\mathbf{w}$, $\mathbf{\lambda}$,
seems an adequate choice (but the question of the tensors coaxiality arises then). In case
b) different choices are pertinent, including scalars variables – for instance index of $\gamma'$-rafting $\xi$ (Fedelich et al., 2006, Tinga et al., 2009b, Fan et al., 2015), isotropic periodicity $\lambda$ or precipitates volume fraction $f_\gamma$ (Cormier and Cailletaud, 2010a, Ghiggi et al., 2012, Giraud, 2013a, le Graverend et al., 2014a). One has simply to be careful to the independence
of the variables retained.

One possible choice is (see Appendix I) to use tensorial variable $w$ and scalar variable
$f_\gamma$ as state thermodynamics variables for the mechanical description of microstructural
changes. Such a preliminary choice is based on the following remarks:
— the $\gamma$ channel width is the quantity that directly controls the Orowan stress. This
latter affects at first order the mechanical response (hardening) of the RVE, the
larger the channel width the lower the apparent yield stress; the three direction
widths $w_i$ can be measured with less difficulties than anisotropic precipitates shapes
$\ell_i$ or anisotropic periodicity lengths $\lambda_i$ in case of advanced $\gamma'$-rafting,
— if both the sizes of $\gamma'$ precipitates $\ell$ and the periodicities $\lambda$ are omitted, one cannot
determine (by means of Eq. (3.43)-(3.44)) the $\gamma'$ precipitates volume fraction; the
effect of $f_\gamma$ has then :
— either to be neglected (possible choice at thermodynamics equilibrium as it can
be considered at zeroth order as a function of temperature only),
— or to be set as the second variable for the mechanical description of microstructur-
tual changes (our choice for possibly high thermomechanical loading rates),
— the $\gamma'$ precipitates volume fraction $f_\gamma$ is a scalar, in comparison with tensorial
nature of $\ell$ and $\lambda$ (tensors difficult to measure, more costly choice for structural
Finite Element applications), it is standardly measured,
— the evolution kinetic of $\gamma$ channel width $w_{001}$ is at first order mainly related to vol-
ume fraction $f_\gamma$ with the same quantitative dependency measured for both coarsen-
ing and rafting processes. It has indeed already been shown for MC2 alloy, under
both isothermal and non-isothermal creep conditions that the $\gamma$ channel width only
(mainly) depends on the $\gamma'$ volume fraction for a sufficiently high temperature,
typically above $1000^\circ$C (Cormier and Cailletaud, 2010a).

This choice is fully consistent with the Polystar model of le Graverend et al (2014a)
which is the only model accounting for the totality of the microstructural evolutions ob-
served in Ni-based Superalloys in non-isothermal conditions. The most natural choice
would be then to use $w$ and $f_\gamma$ as state variables. However, as detailed in the Appendix I,
this choice is not the best one from a thermodynamics point of view. The reason is that,
as explained in the sections 1.2.3 and 1.4, the volume fraction of precipitates $f_\gamma$ and the
matrix channel width variations are related in a quite complex manner, especially dur-
ing non-isothermal loadings. To summarize, fast overheating and cooling bring to out of
thermodynamics equilibrium states and to
— the dissolution of the hardening phase, followed by the precipitation of the $\gamma'$ par-
ticles (that one could call tertiary $\gamma'$ precipitates);
— the reduction/re-widening of $\gamma$ channels.
As described in the Polystar model (Eq. (3.4.4)), the dissolution/re-precipitation ki-
netics depend on the temperature rate.

\[
\dot{f}_\gamma = \left(1 - \delta_\gamma e^{-\frac{\varepsilon}{\tau_\gamma}}\right) \frac{f_{\gamma\text{eq}}(T) - f_\gamma}{\tau_\gamma}
\]

(3.45)

with \(f_{\gamma\text{eq}}(T)\) the volume fraction of precipitates at equilibrium, \(\tau_\gamma\) and \(\delta_\gamma\) as material parameters. For the finer precipitates:

\[
f_{\gamma\text{eq}} - f_\gamma \leq 0 \Rightarrow \dot{f}_{\gamma\text{II}} = \frac{f_{\gamma\text{II}1} m_{sp}}{T_0} - \left(\frac{f_{\gamma\text{II}1}}{K_{s2}}\right) m_{sp}
\]

(3.46)

\[
f_{\gamma\text{eq}} - f_\gamma \leq 0 \text{ and } \dot{T} < 0 \Rightarrow \dot{f}_\gamma = -\frac{f_{\gamma\text{eq}} - f_\gamma - f_{\gamma\text{II}} \dot{T}}{\tau_s} - \left(\frac{f_{\gamma\text{II}1}}{K_{s1}}\right) m_{sp}
\]

(3.47)

\[
f_{\gamma\text{eq}} - f_\gamma \leq 0 \text{ and } \dot{T} \geq 0 \Rightarrow -\left(\frac{f_{\gamma\text{II}1}}{K_{s2}}\right) m_{sp}
\]

(3.48)

where (Giraud, 2013a):

— the first equation describes the dissolution of the small \(\gamma'\) precipitates during an overheating;
— the second describes the competition between precipitation of the \(\gamma_{\text{II}}'\) vs the Oswald ripening phenomenon during the cooling phase;
— the third equation describes the Oswald ripening which characterizes the isotherm stage after the overheating.

\(\dot{T}\) the temperature rate, \(\tau_s, T_0, K_{s1}, K_{s2}, m_{sp}\) as additional parameters.

Thus the widening/closing of \(\gamma\) channel depends on the volume fraction of the precipitates phase (by the function \(g(f_\gamma, f_{\gamma\text{II}})\), Eq. (3.49 and 3.50), to the temperature rate too. The following scalar dependency is considered in their modeling,

\[
w_{001} = g(f_\gamma, f_{\gamma\text{II}}) w_{001}^0 (\omega_{\text{mech}} + \omega_{\text{LSW}})
\]

(3.49)

where \(\omega_{\text{mech}}\) and \(\omega_{\text{LSW}}\) are non-dimensional variables accounting for widening of \(\gamma\) channel produced by \(\gamma'\) rafting and of the one produced by the Ostwald-ripening (LSW) respectively and where function \(g(f_\gamma, f_{\gamma'}\)) is the function describing the relation between the volume fraction variation of the secondary and tertiary precipitates population,

\[
g(f_\gamma, f_{\gamma'}) = f_{\gamma'}^{\text{auth}} - d_{tp} f_{\gamma\text{II}1}^{m_{sp}}
\]

(3.50)

Recall that at thermodynamics equilibrium, so that one defines

\[
w_{001} = g(f_{\gamma\text{eq}}(T), 0) w_{001}^0 = w_{001}(T)
\]

(3.51)

as the initial \(\gamma\) channel width as considered temperature for isothermal loading; \(w_{001}^0\) is the initial width of the \(\gamma\) channels measured at 20.

When using both \(f_\gamma\) as state variables, the dissipation due to microstructural evolution depends on both the evolution of \(f_\gamma\) and \(w\) \((f_\gamma = \ldots\text{ and } w = \ldots\), Eq. (3.45) and I.13), then
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on all the complex relations existing between the $\gamma'$ dissolution/re-precipitation and $\gamma'$-rafting kinetics. In order to describe the thermodynamics of microstructural evolution another choice is made in present work. Rewriting Eq. (3.49) in a tensorial form (to account for the 3D microstructure) and defining the non-dimensional variable $\omega = \omega_{\text{mech}} + \omega_{\text{LSW}}$ the following expression is obtained:

$$\mathbf{w} = g(f_{\gamma'}, f_{\gamma''}) w^{0}_{001} \omega$$

(3.52)

and then

$$\omega = \frac{\mathbf{w}}{g(f_{\gamma'}) w^{0}_{001}} \quad \omega_{ij} = \frac{w_{ij}}{w^{0}_{001}g(f_{\gamma'})}$$

(3.53)

The tensor $\omega$ then correspond to the tensor $\mathbf{w}$ but normalized by the product of the $\gamma'$ volume fraction and the horizontal matrix channel width at ambient temperature. This product corresponds to the $\gamma$ channel width of the cubic microstructure at the studied temperature $w^{0}_{001}$.

$$w^{0}_{001} = g(f_{\gamma'}) w^{0}_{001}$$

(3.54)

The difference between $w^{0}_{001}$ (at room temperature) and $w^{0}_{001}$ then results only by the dissolution of $\gamma'$ precipitates.

Isothermal creep is the first objective of the modeling, so only the dependence from the volume fraction of the larger precipitates is kept in Eq. (3.44). From a thermodynamic point of view, describing microstructural evolutions by the mean of only the one non-dimensional variable $\omega$ (the one defined in Eq. (3.44)) means that dissipation due to microstructural evolutions will neither depend on the interaction between the $\gamma'$ dissolution and $\gamma'$ rafting kinetics, nor on the temperature rate. However it is not the only advantage brought by the choice of using only one variable: in this way only one microstructural evolution law has to be integrated at each time step and only one variable is coupled with visco-plasticity. Further details about the thermodynamic of microstructural evolution and about the tensorial nature of the variable $\omega$ are presented in the Appendixes I and K.

3.4.5 Evolution law

According to its definition, the tensor $\omega$ has the same form of $\mathbf{w}$ (Eq. (3.41)) in the Natural (cubic) Anisotropy Basis of the material (NAB) (Eq. (3.55)).

$$\omega = \begin{bmatrix} \omega_1 & 0 & 0 \\ 0 & \omega_2 & 0 \\ 0 & 0 & \omega_3 \end{bmatrix}_{\text{NAB}}$$

(3.55)

Its main diagonal then contains the widths of the three matrix channels lying along the principal directions of the cubic frame and normalized by $w^{0}_{001}$. In order to describe
the γ′ rafting, mechanical coarsening and non-mechanical coarsening (Ostwald-ripening) the evolution law ˙ω = ... has to reproduce
— a decrease of the w_2 and w_3 channels when loading along the <001> crystallographic direction (figure 3.3, N-type rafting);
— an isotropic growth of the three matrix channels when loading along the <111> crystallographic direction (w_1 = w_2 = w_3, figure 3.4, mechanical coarsening);
— a growth of the only active channel on a N-type rafted microstructure and an isotropic growth of the three active matrix channels on a cubic microstructure (figure 3.5(a) and (b) load-free coarsening).

The evolution law proposed to model the three described phenomena consists of the sum of three contributions (Eq. (3.56)):

— ω_{raft}, which is the γ′ rafting contribution;
— ω_{mc} which accounts for mechanical coarsening;
— \sqrt{3} \omega_{LSW} \parallel \omega \parallel, which accounts for the load free coarsening.

\[ \hat{\omega} = \omega_{raft} + \omega_{mc} + \sqrt{3} \omega_{LSW} \parallel \omega \parallel \]

The pre-factor \sqrt{3} = ||1|| is such as if w \propto 1, ω_{LSW} = ω_{LSW} 1 with initial values ω_{LSW}(t = 0) = 1, ω_{raft} = 0, ω_{mc} = 0 and ω(t = 0) = 1.

As detailed in Appendix I:
— the evolution of matrix channels due to γ′ rafting is assumed to be proportional to the plasticity normal projected on the “deviatoric diagonal” Kelvin mode, ˙ω_{raft} \propto n_d. This formulation involves an increase of the channel perpendicular to the loading direction (w_1) during tensile rafting along the <001> crystallographic direction and a widening of the channel parallel to the loading direction during compression creep along the <001> crystallographic direction (w_3). Moreover, since the “deviatoric diagonal” mode is not activated when loading along the <111> crystallographic direction, no γ′-rafting is produced along this direction;
— the evolution of matrix channels due to γ′ mechanical coarsening is assumed to be proportional to the second order identity tensor 111. This formulation is consistent with the isotropic growth of the three matrix channels produced when loading along the <111> crystal direction. An Heaviside function dependent on the plasticity produced by the “out of diagonal” Kelvin mode is also added to model the plasticity threshold observed by Matan (section 1.2.1 Matan et al (1999a), Sakaguchi and Okazaki (2018)). Since the “out of diagonal” Kelvin mode is not activated when loading along the <001> direction, this formulation allow to reproduce the fact that no mechanical coarsening sets when loading along <001> oriented crystals;
— the evolution of γ channels produced by the load-free coarsening is made dependent on the rate between ω and its norm, \parallel \omega \parallel. This allows to reproduce the homothetic widening described in the subsection 1.2.2.

The main hypotheses underlying this formulation are the same assumed by (Tinga et
Figure 3.3 – Schematic representation of the $\gamma'$ N-type rafting: progressive closure of the $w_2$ and $w_3$ channels and contemporary widening of the $w_1$ channel.

— constant $\gamma'$ volume fraction during $\gamma'$ rafting, coarsening and Ostwald-ripening;
— isotropic periodicity $\lambda$;
— absence of periodicity evolution during $\gamma'$ rafting;
— in these hypotheses it is possible to assume that during N-type rafting $w_2 = w_3$.

Again, further details are given in the Appendix I.

The following section presents the detail of the $\omega$ evolution law and of the coupling between microstructural evolution and visco-plasticity. The creep and monotonic tension curves identified along $<001>$ and $<111>$ (by exploiting the tests presented in the section 2.2) are presented as well.

It has to be remarked that the proposed formulation also allows to reproduce P-type rafting during compression creep test along $<001>$ crystals, the passage from N-type rafting to P-type rafting observed by Ott and Mughrabi (1999), Tetzlaff and Mughrabi (2000), Giraud (2013a) and the $\gamma'$ rafting morpholgy observed in positive misfit alloys (these phenomena are described in the section 1.2.1). Results and discussion concerning these subjects are presented in the Appendix M

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3.4.6 3D index for rafting

An index of $\gamma$'-rafting ($\xi$, Eq. (3.37)) has been introduced in Fedelich et al (2006); it is mainly based on an uniaxial description of $\gamma$'-rafting, for which only one of the three $\gamma$ channel widths is considered (the one in the loading direction $w_1 = w_{[001]} = w_{001}$). Such an index of rafting is built so that $\xi = 0$ for coarsening ($\gamma$' precipitates remains cubes at $\ell_1 = \ell_2 = \ell_3$, $w_1 = w_2 = w_3$) and $\xi = 1$ for completed $\gamma$'-rafting in tension (at zero width channel $w_2 = w_3 = 0$). It introduces the two references states $w_{raft}$ and $w_{cube}$ at the given volume fraction $f_\gamma$ and periodicity $\lambda$ (in fact the periodicity in direction [100]).

For compression and for misoriented or multiaxial loading cases a pure geometric
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Figure 3.5 – Schematic representation of the $\gamma'$ coarsening acting on a cubic microstructure (a) and on a $\gamma'$-rafted microstructure (b).

definition is needed. Using the fact that deviatoric part $w' = 0$ in coarsening process, we propose to define such a geometric index of rafting as

$$x_{raft} = \frac{w_{eq}}{\text{tr} w} \quad w_{eq} = \sqrt{\frac{3}{2} \|w'\|} = \sqrt{\frac{3}{2} w' : w'} \quad (3.57)$$

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i.e. an index built from tensorial $\gamma$ channel width variable only $w$, of deviatoric part $w’ = w - \frac{1}{3} \text{tr} w \mathbf{1}$.

One has the following properties for $x_{\text{raft}}$:
- $x_{\text{raft}}$ is dimensionless,
- $x_{\text{raft}} = 0$ for $\gamma’$-coarsening ($w_{eq} = 0$),
- $x_{\text{raft}} = 1$ for $\gamma’$-rafting in uniaxial tension along [100] at diagonal $w = \text{diag}[w_1, 0, 0]$,
- $x_{\text{raft}} = \frac{1}{2}$ for rafting in uniaxial compression along [100] at diagonal $w = \text{diag}[0, w_2, w_3 = w_2]$.

Let us point out that the value $x_{\text{raft}} = 0$ uniquely correspond to coarsening (spherical tensor $w = w \mathbf{1}$), the value $x_{\text{raft}} = 1$ uniquely corresponds to complete $\gamma’$-rafting (rafting tensor $w$ with two eigenvalues equal to zero).

The link with index $\xi$ is given in the Appendix L.

**Remark** – Definition (3.57) is inspired from the definition of stress triaxiality $\text{TRIAX} = \frac{1}{3} \text{tr} \sigma / \sigma_{eq}$ in the field of ductile rupture and continuum damage mechanics (Rice and Tracey, 1969, Lemaitre and Chaboche, 1991).

### 3.5 Visco-plasticity coupled with tensorial $\gamma’$-rafting model

The thermodynamics framework and the ingredients for a constitutive modeling of visco-plasticity coupled to tensorial $\gamma’$-rafting have been developed in previous Sections. Let us summarize here the choices made to derive a multiaxial model for single crystal CMSX-4 superalloy at high temperature. Creep up to the secondary stage is the main domain of validity sought so that kinematic hardening and the coupling with damage are omitted in such a first model with tensorial $\gamma’$-rafting.

Concerning visco-plasticity, let us use the Kelvin modes based two criterion visco-plasticity framework of Section 3.3.7 for cubic single crystals. Let us recall the definitions $\sigma^d \overset{\text{def}}{=} \mathbb{P}^d : \sigma$ (also referred to as ”diagonal deviatoric stress”, diagonal in *Natural Anisotropy Basis*), $\sigma^d \overset{\text{def}}{=} \mathbb{P}^d : \sigma$ (also referred to as ”out of diagonal deviatoric stress” in *Natural Anisotropy Basis*), and partition $\sigma’ = \sigma^d + \sigma^\perp$. One also has Kelvin partition of the plastic strain tensor

$$\varepsilon^p = \varepsilon_1^p + \varepsilon_2^p$$

with $\varepsilon_1^p \overset{\text{def}}{=} \mathbb{P}^d : \varepsilon_1^p$ (diagonal in *Natural Anisotropy Basis*), $\varepsilon_1^p \overset{\text{def}}{=} \mathbb{P}^d : \varepsilon_1^p$ (out of diagonal in *Natural Anisotropy Basis*) and $\varepsilon_2^p = \mathbb{P}^d : \varepsilon_2^p = 0$.

Concerning microstructure degradation, we use the formulation in terms of $\gamma’$-rafting variable $\omega$ derived in sections I.1.4.2 and I.2.

No damage is introduced in the present version of the model, this coupling and the description of tertiary creep being left out to further work.

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3.5.1 Proposed model (Part I Mechanics)

The constitutive equation for cubic elasto-visco-plasticity coupled with tensorial γ'-rafting are:
— elasticity law coupled with γ'-rafting (with in practice the coupling of elasticity with γ'-rafting is neglected, $\kappa_\varepsilon \omega_{eq} << 1$)

$$\sigma = E : (\varepsilon - \varepsilon^p - \alpha (T - T_{ref}) \mathbf{1})$$  \(3.59\)

Hooke’s tensor $E$ has cubic material symmetry, $\alpha$ is the thermal expansion coefficient, $T_{ref}$ is reference temperature.

— stress partition in cubic symmetry

$$\sigma = \sigma^d + \sigma^\gamma + \frac{1}{3} \text{tr} \sigma \mathbf{1} \quad \sigma^d = \sqrt{\frac{3}{2}} \sigma^d : \sigma^d \quad \sigma^\gamma = \sqrt{\frac{3}{2}} \sigma^\gamma : \sigma^\gamma$$  \(3.60\)

Introducing equivalent von Mises norms $\sigma^d_{eq}$ and $\sigma^\gamma_{eq}$,

— criterion functions with saturated hardening and Orowan stress effect (Hill criterion for $f_1$, for the sake of simplicity quadratic norm $a = 2$ for $f_2$,

$$f_1 = \sigma_{\text{Hill}} - R_\infty - \kappa_{\text{Orowan}} G \|\omega\| - \sigma_y \quad f_2 = \sigma^\gamma_{eq} - R_\infty - \omega \kappa_{\text{Orowan}} G \|\omega\| - \sigma_y$$  \(3.61\)

where

$$\sigma_{\text{Hill}} = \frac{1}{\sqrt{2}} \left( \sigma^d \sigma^d + h^2 \sigma^\gamma \sigma^\gamma \right)$$  \(3.62\)

with $h$ Hill material parameter for cubic (visco-)plasticity. The expression of $f_2$ retained for multiaxial case (containing the non-quadratic norm of stress tensor) is given in the Appendix G

— plastic strain rate

$$\dot{\varepsilon}^p = p^\text{Hill}_1 \frac{\partial f_1}{\partial \sigma} + p^\gamma_2 \frac{\partial f_2}{\partial \sigma}$$  \(3.63\)

— accumulated plastic strain rates

$$p^\text{Hill}_1 = \sqrt{\frac{2}{3}} \left( e^{d_1} : e^{d_1} + \frac{1}{h^2} e^{d_2} : e^{d_2} \right) \quad p^\gamma_2 = \sqrt{\frac{2}{3}} e^{d_2} : e^{d_2}$$  \(3.64\)

— viscosity laws

$$p^\text{Hill}_1 = \left\langle \sigma_{\text{vsoo}} K_N \ln \left( 1 - \frac{f_1}{\sigma_{\text{vsoo}}} \right) \right\rangle^N_+$$  \(3.65\)

$$p^\gamma_2 = \left\langle \frac{f_2}{K_N} \right\rangle^N \frac{1}{\max \left( \kappa (\sigma^\gamma_{eq} - \sigma^\mu_{\text{lim}}), 1 \right)}$$  \(3.66\)

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The material parameters are: $E$, $\nu$, $G$ for elasticity, thermal expansion coefficient $\alpha$, yield stresses $\sigma_y$ and $\bar{\sigma}_y$, Hill parameter $h$, saturated hardenings $R_\infty$ and $\bar{R}_\infty$, parameter $\kappa_{oro}$ for Orowan stress effect, viscosity parameters $K_N$, $N$, $\bar{K}_N$, $\bar{N}$ for orientations $<001>$ and $<111>$ visco-plasticity. A tendency to saturation of the viscous stress will be obtained for $<001>$ orientation so that viscosity law (3.33) is used, introducing the saturation value $\sigma_{v,\infty}$ as material parameter. Two additional material parameters are $\bar{K}$ and $\sigma_{\mu,\infty}^\mu$.

Standard Norton law for $\dot{\rho}_2^\parallel$ was our initial modeling choice, but such a choice underestimates the stress level in monotonic tension (see Fig. 3.15b). This is why we did measure the value of $\bar{K}_{\text{Nexp}}$ in the $<111>$ tension tests—performed at different strain rates, Fig. 3.6a—by formula

$$\bar{K}_{\text{Nexp}} = \frac{\sigma_{\text{plateau}} - \sigma_{\text{OROini}}}{\left(\rho_2^\parallel\right)^{1/N}}$$  \hspace{1cm} (3.67)

once the Norton parameters identified from creep experiments (here $\sigma_y + R_\infty \approx 0$). In Eq. (3.67) the values of the stress $\sigma = \sigma_{\text{plateau}}$ and of the plastic strain rate $\dot{\rho}_2^\parallel$ are measured on the plateau of the stress-strain curve (see figure 3.6(a)); $\sigma_{\text{OROini}}$ is the “initial” value of the Orowan stress: since no microstructural evolution is observed at the high strain rates, the Orowan stress does not evolve either and so its value stays equal to the one computed on the cubic non-evolved microstructure (as $\|\omega\| \approx \sqrt{3}$).

The retained function describing the measured variation of the parameter $\bar{K}_{\text{Nexp}}$ is, with $K_N$ measured in creep,

$$\bar{K} = \bar{K}_N \left(\max \left[\bar{K} (\sigma - \sigma_{\mu,\infty}^\mu), 1\right]\right)^{1/N}$$  \hspace{1cm} (3.68)

It is plotted in Fig. 3.6b from $<111>$ monotonic datas of Fig. 3.6a.

Note that for the creep curves presented next the stress will remain low enough so that $\bar{K}_{\text{Nexp}} = \bar{K}_N$ and Norton viscosity law apply.

**Particular uniaxial cases (Mechanics)** — For an uniaxial loading along the crystal direction $<001>$, $\sigma_{ini} = |\sigma|$ and $p_1^\text{Hill} = p = \int |\dot{\varepsilon}^p| \, dt$ (with $\dot{\varepsilon}^p$ the uniaxial plastic strain), $\sigma^\parallel = 0$, $\sigma^\perp_{eqx} = 0$, $\rho_2^\parallel = 0$ so that, at low strain rate,

$$|\dot{\varepsilon}^p| \approx \left(\frac{\sigma - R_\infty - \frac{\kappa_{oro}}{\|\omega\|} - \sigma_y}{K_N}\right)^N$$  \hspace{1cm} (3.69)

with then $\sigma_y = \sigma_{y,001}$ the yield stress in direction $<001>$.

For an uniaxial loading in direction $<111>$, $\sigma^\parallel = 0$, $\sigma^\perp_{eqx} = 0$, $\rho^\parallel = 0$, $\sigma^\perp_{eqx} = |\sigma|$, $h \rho_1^\text{Hill} + \rho_2^\parallel = p = \int |\dot{\varepsilon}^p| \, dt$ (with $\dot{\varepsilon}^p$ the uniaxial plastic strain), so that, at low strain rate,

$$|\dot{\varepsilon}^p| \approx \left(\frac{\sigma - R_\infty - \frac{\kappa_{oro}}{\|\omega\|} - \sigma_y}{K_N}\right)^N + h \left(\frac{\sigma - R_\infty - \frac{\kappa_{oro}}{\|\omega\|} - \sigma_y}{K_N}\right)^N$$  \hspace{1cm} (3.70)

along the $<111>$ orientation.
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Figure 3.6 – a) $<111>$ stresses $\sigma = \sigma_{\text{plateau}}$ used in Eq. (3.67) and b) values of the parameter $K_{N_{\text{exp}}}$ computed by using (3.67) and the proposed variation of this parameter as a function of the stress (red line, Eq. (3.68)).

For small enough values of $h$, the right hand inside term can be neglected and Eq. (3.70) then come back to the Norton’s law (with then $\bar{\sigma}_y = \sigma_{y,111}$ the yield stress along the crystal direction $<111>$). The value $h \approx 0.5$ will be obtained in section 3.9 by cross identification with a Schmid law based single crystal visco-plasticity model.

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
3.5.2 Proposed model (Part II Tensorial evolution law for $\gamma'$-rafting)

To account for $\gamma'$ rafting and mechanical coarsening using a unified expression from the previous models Tinga et al (2009b), Cormier and Cailletaud (2010a), we propose:

$$\dot{\omega}^{\text{raft}} = K^{\text{raft}} \exp \left( -\frac{Q - \min(\sigma_{eq}^d, \sigma_{lim}^d) U_{raft}}{k_B T} \right) H(p_1^{\text{Hill}} - \varepsilon_{th}^p) \mathcal{H} \mathbf{n}^d \tag{3.71}$$

$$\dot{\omega}^{\text{mc}} = K^{\text{mc}} \exp \left( -\frac{Q - \sigma_{eq} U_{mc}}{k_B T} \right) H(p_2 - \varepsilon_{th}^p) \tag{3.72}$$

with either $\mathcal{H} = H(\omega: \mathbf{n}^d)$ in modeling $A$ or $\mathcal{H} = H(\mathcal{D})$ in modeling $B$.

In equation 3.71, the stress affects on $\gamma'$-rafting has been bounded replacing $\sigma_{eq}^d$ by $\min(\sigma_{eq}^d, \sigma_{lim}^d)$ in the Arrhenius-Tinga exponent (equation 3.38) in order to correctly model monotonic tension (higher magnitude of stress than creep).

As already discussed, a $\gamma'$-rafting threshold $\varepsilon_{th}^p$ and a mechanical coarsening threshold $\varepsilon_{th}^p$ are introduced according to Matan et al (1999a). In the next section, one will obtain $\varepsilon_{th}^p = 10^{-4}$ and $\varepsilon_{th}^p \approx 0^+$ from mechanical measurements. As $\gamma'$-rafting does not occur for tension along the crystal direction $<111>$, the contribution $\dot{\omega}^{\text{raft}}$ is made dependent on $\sigma_{eq}^d$ (vanishing for tension along the $<111>$ crystallographic orientation, see Eq. (3.60)). Full von Mises stress $\sigma_{eq}$, non zero for both $<001>$ and $<111>$ orientations, is assumed to drive the mechanical coarsening contribution $\dot{\omega}^{\text{mc}}$.

In the initial Polystar model $K^{\text{raft}}$ and $K^{\text{mc}}$ are functions of both the accumulated plastic strain and the loading rate. One considers next them as material parameters, temperature dependent only. Thermal/stress activation constants $Q$ and $U$ are assumed not to depend on temperature, $k_B$ is Boltzmann constant.

Matan et al plastic strain threshold (Matan et al, 1999a) is introduced through Heaviside functions $H$. It corresponds to the density of dislocations on octahedral slip systems necessary to relax the misfit stresses (so that $\varepsilon_{th}^p >> \varepsilon_{th}^p$). Once the $\gamma'$/$\gamma'$ lattice mismatch is relaxed with a given dislocation density, $\gamma'$-rafting can proceed, even in the absence of any applied stress. This phenomenon is not represented in Tinga approach.

For homothetic growth no threshold has been observed so that the evolution law (Cormier and Cailletaud, 2010a)

$$\dot{\omega}^{\text{LSW}} = \frac{K^{\text{LSW}}}{3\omega_{\text{LSW}}^2} \exp \left( -\frac{Q}{k_B T} \right) \tag{3.73}$$

consistent with both Eq. (3.3) and Eq. (3.36) is considered.

**Particular uniaxial cases (Tensorial evolution law for rafting)** — For an uniaxial loading along the direction $<001>$, $\sigma_{eq}^d = \sigma_{eq} = |\sigma|$, $\mathbf{n}^d = \frac{3}{2} \frac{\sigma}{\sigma_{eq}}$, $p = p_1^{\text{Hill}}$, so that

$$\dot{\omega} = K^{\text{raft}} \exp \left( -\frac{Q - \min(|\sigma|, \sigma_{lim}^d) U_{raft}}{k_B T} \right) H(p - \varepsilon_{th}^p) \mathcal{H} \mathbf{n}^d \tag{3.74}$$
For an uniaxial loading along the $<111>$ direction, $\sigma_{eq}^d = 0$, and $\sigma_{eq} = |\sigma|$ so that, at $\varepsilon_{th}^d = 0^+$,

$$\dot{\omega} = K_{mc} \exp \left( -\frac{Q - |\sigma|U_{mc}}{k_B T} \right) \Omega$$

(3.75)

### 3.5.3 Proposed model (Part III $\gamma'$ precipitates evolution / $\gamma$ channel width)

For the $\gamma'$ precipitates volume fraction $f_{\gamma'}$, the evolution law retained is the one of non-isothermal Polystar model previously identified by Giraud (2013a), Ghiggi (2013) and le Graverend et al (2014a),

$$\dot{f}_{\gamma'} = \left( 1 - \delta_{\gamma'} e^{-\frac{p_{\text{mean}}}{\tau_{\gamma'}}} \right) \frac{f_{\gamma'\text{eq}}(T) - f_{\gamma'}}{\tau_{\gamma'}}$$

(3.76)

Parameter $\delta_{\gamma'} = 0.8$ is such as $0 < \delta_{\gamma'} < 1$ in order to ensure $f_{\gamma'} = f_{\gamma'\text{eq}}(T)$ at thermodynamics equilibrium, at rate controlled by characteristic time $\tau_{\gamma'} = 200$ s$^{-1}$ and by parameter $e_{\gamma'} = 5 \times 10^{-3}$ at 1050°C; $f_{\gamma'\text{eq}}(T)$ is equilibrium volume fraction, function of the absolute temperature $T$ only. Instead of Eq. (3.34), we use for considered CMSX-4 superalloy a modified expression (plotted in Fig. 3.7):

$$f_{\gamma'\text{eq}}(T) = \left( f_0^{\gamma'} - (f_0^{\gamma'} - f_1^{\gamma'}) \left( \frac{T - T_0}{T - T_1} \right)_+ \right) \left( 1 - \left( \frac{T - T_1}{T_2 - T_1} \right)_+^2 \right)$$

(3.77)

with $f_0^{\gamma'} = 0.7$, $f_1^{\gamma'} = 0.48$, $T_0 = 1073$ K, $T_1 = 1418$ K, $T_2 = 1543$ K for the considered CMSX-4 alloy. All these parameters have been identified based on our own experiments done specifically for the purpose of this study (see section 2.5). Indeed, to achieve a reliable modeling of the creep strain rate measured in our experiments, the specimens used to characterize the evolution of $f_{\gamma'\text{eq}}$ have been extracted from a CMSX-4 bar having exactly the same chemistry and having received exactly the same solution and aging heat treatments as the creep specimen used for the experiments presented. These experiments consists in stereological analyzes of SEM pictures taken from different specimens first submitted to a creep deformation at 1050°C and 140 MPa for 100 hours whose aim is to develop a regular $\gamma'$-rafted structure (figure 2.7(a)). They were then held for 1 hour at different temperatures ranging from 850°C to 1300°C, then water quenched to be able to extract the gamma-prime volume fraction from area-fraction measurements. Note that stereological analyzes were performed in both the primary dendrite arms and in the interdendritic spacings, as shown in Fig. 3.7. The $\gamma'$ volume fraction is then constant from ambient temperature to around 800°C (Link et al, 2011).

As a matter of fact, once yielding at high temperature has taken place, a $\gamma'$ dissolution kinetics is observed, enhanced by the level of plasticity (Ghiggi et al, 2012, Giraud et al, 2013b, le Graverend et al, 2014a). The coupling with plasticity is written in terms of accumulated plastic strain $p_1^{\text{ema}}$ (representative of octahedral slips, with $e_{\gamma'}$ a material
3.6 Isotropization at very high temperature

Let us last point out an interesting feature due to the consideration of Hill (cubic) criterion for yielding function $f_1$ and of material parameter $h$: the possibility to model the observed visco-plastic isotropization at very high temperature (> 1100°C, Caron et al (1988), Hana et al (2010), Mataveli Suave at al. (2016)), where “isotropization” means the decrease of the creep strain rate and creep life anisotropy. In our proposed model an isotropization is simply gained by a temperature dependency of Hill parameter $h$ made such as

$$h = h(T) \rightarrow 1$$

if in the same time the temperature dependent sum yield stress + hardening $\bar{\sigma}_y(T) + \bar{R}_\infty(T)$ for criterion function $f_2$ is made larger than $\sigma_y(T) + R_\infty(T)$ for criterion function $f_1$, i.e. material parameters such as $\dot{\varepsilon}_2 \ll \dot{\varepsilon}_1$. Such an isotropisation of the viscoplastic...
properties is generally obtained under conditions of fast development of the gamma-prime rafting, without any degradation of the stability of the $\gamma'$-rafted structure (i.e. no $\gamma/\gamma'$ topological inversion (Epishin et al (2000, 2001)). Under such conditions, a creep life anisotropy factor of 2 at maximum is experimentally obtained.

### 3.7 Isothermal creep along $<001>$ and $<111>$ crystallographic directions at 1050°C

The time integration of the constitutive equations –by means of simple Euler explicit scheme for presented results– allows to calculate the model responses to any loading case. Note that, in 1D as well as in 3D, the proposed elasto-viscoplastic model with tensorial $\gamma'$-rafting only needs the coupled computation of the strain, stress, plastic strain and of dimensionless tensorial variable $\omega$ (constitutive equations of Sections 3.5.1 and 3.5.2). The determination of both the $\gamma'$ volume fraction $f'_{\gamma}$ and the physical $\gamma$ channel width tensor $w$ at any time can be computed in a post-processing manner (constitutive equations of Section 3.5.3, the coupling with mechanics being made through dimensionless tensor $\omega$ instead of width tensor $w$). Material parameters are identified on the experimental results presented in the chapter 2. These parameters are presented in the Appendix N (SS set of material parameters).

#### 3.7.1 $\gamma'$ precipitates volume fraction

For an isothermal loading, the thermodynamics equilibrium is quickly reached (small characteristic time $\tau_{\gamma'}$) and $f'_{\gamma} \approx f'_{\gamma eq}(T)$. At 1050°C, it is a $\gamma'$ precipitates volume fraction $f'_{\gamma} = 0.54$. For the next calculations the initial $\gamma$ channel width is $w_0(1050°C) = w_{001}(t = 0) = w_i(t = 0) = 80$ nm (measured). Using Eq. (3.78), one has at this temperature $g(f'_{\gamma}) = 1.533$. One gets then a 20°C $\gamma$ channel width $w_{001}^0 = 52$ nm, value very close to the standard 50 nm for CMSX-4 and MC2 superalloys (Benyoucef et al, 1993). We consider this as a validation of mixture law (3.78) for $g(f'_{\gamma})$ function.

#### 3.7.2 LSW homothetic growth

At 1050°C, only few experimental datas are available for load free LSW homothetic growth of the cuboidal microstructure (marks in Fig. 3.8 from reference Fedelich et al (2012a)). The fitting curves due to different authors for similar nickel based single crystal superalloys are also plotted

- by means of Eq. (3.73) for MC2 superalloy (curve from le Graverend (2013), $w_0(1050°C) = w(t = 0) = 80$ nm).
- by means of Eq. (3.35) with CMSX-4 material parameters from Fedelich et al (2012a) with here also $w_0(1050°C) = 80$ nm $(w = 80[1 + 0.198t]^{0.0745}$ nm with $t$ in hours),
The retained choice corresponds to Eq. (3.73)-(3.78). Even if the kinetics of the LSW phenomenon in CMSX-4 alloy differs from the one of the MC2 because of the presence of Rhenium, the kinetic of the Oswald ripening phenomenon is low and negligible compared to the γ'-rafting/coarsening. Thus, $K_{LSW} \exp\left(-\frac{Q}{k_B T}\right)$ has been chosen equal to $1.1 \times 10^{-6}$ s$^{-1}$, which helps to recover the curve identified by le Graverend (le Graverend, 2013) in figure 3.8.

![Figure 3.8 – Channel width evolution as a function of time $t$ in hours at 1050°C.](image)

### 3.7.3 $<001>$ creep

During tensile creep along the $<001>$ direction, γ'-rafting is developing perpendicular to the applied stress (Fig. 3.9c, γ width channel $w_1$ increases, $w_2 = w_3$ decrease to zero). In the present model the evolution is governed by a $K_{raft} = 1.7 \times 10^{-7}$ s$^{-1}$ and it is stress enhanced according to non zero Tinga et al material parameter $u_{raft} = \frac{U_{raft}}{k_BT} = 0.025$ within Arrhenius exponential. The mechanical responses are given in Fig. 3.9a-b for three creep stresses: 140 MPa, 180 MPa, 200 MPa. The black thin lines of the figures correspond to experiments performed on CMSX-4 specimens oriented close to a $<001>$ orientation with less than a 5° misorientation. One sees in Fig. 3.9a and 3.9b that the experimental curves are well reproduced by the model up to the 5% of strain, meaning that the creep rate increase only results from the microstructure degradation and not from a damage mechanism in the form of, e.g., cavitation (le Graverend et al, 2017). It is also seen from Fig. 6c that the evolutions of the gamma channel widths, using Matan et al threshold $\epsilon_{th} = 10^{-4}$, reproduce quite well the experimental observations at the end of the tests. For coupling visco-plasticity and microstructural degradation, an Orowan parameter $\kappa_{ORO} = 2.1 \times 10^{-3}$ MPa$^{-1}$ has been identified. The shear modulus of the material is $G = 96$ GPa. The index of γ'-rafting $x_{raft}$ (see Section 3.4.6) is plotted in Fig. 3.9(d). It shows
the completion of rafting phenomenon during the initial primary creep regime \( x_{\text{raft}} = 1 \) for fully rafted microstructure. The further nonlinearity of \( \dot{p} \) versus \( p \) curves (Fig. 3.9b) is due to LSW diffusion term. At each stress level tested, the widths of the \( \gamma' \) channels (corresponding to the experimental points on Fig. 3.9c) have been calculated as the average of the Gaussian distribution of the values measured on SEM micrographs, showed in Fig. 3.10a. The error bars correspond to the standard deviation of the distributions.

Figure 3.9 – [001] creep responses of CMSX-4 alloy at 1050°C: a) Creep curves \( \epsilon(t) \), b) \( \dot{p} \) vs \( p \) curves, c) \( \gamma \) channel width curves (upper lines: model \( w_1(t) > w_0 \), lower lines: model \( w_2(t) = w_3(t) < w_0 \), marks: measurements at 140 MPa and 200 MPa), d) Index of \( \gamma' \)-rafting \( x_{\text{raft}} = \frac{w_{\text{eq}}}{\omega} \) as a function of accumulated plastic strain \( p \).

### 3.7.4 \(< 111 > \) creep

For creep along direction \(< 111 >\) there is \( \gamma' \) coarsening but no \( \gamma' \) rafting, neither in experiments (Leidermark et al, 2010) (as it can be seen in Fig. 3.11 and which corresponds to black thin lines in Fig. 3.12) nor in the model \( (H(p_{\text{Hill}} - \epsilon_{h}^p) = 0 \) so that the \( K_{\text{raft}} \) term vanishes in evolution law for \( \omega \), thick lines in the Figures). The three \( \gamma \) channel widths \( w_i \)
Isothermal creep along $<001>$ and $<111>$ crystallographic directions at 1050°C

Figure 3.10 – $\gamma/\gamma'$ rafted microstructure at 1050°C along [001]: a) at 140 MPa after 101 h, b) at 140 MPa after 242.5 h, c) at 200 MPa after 34.9 h. Note that the gamma-prime phase appears in dark while the gamma matrix is in light gray.

Figure 3.11 – $\gamma/\gamma'$ rafted microstructure at 1050°C along [111]: a) at 180 MPa after 114 h, b) at 200 MPa after 66.9 h. Note that the gamma-prime phase appears in dark while the gamma matrix is in light gray.

are equal in the 3 crystallographic directions $[001] \equiv 1$, $[100] \equiv 2$ and $[010] \equiv 3$, with a zero index of rafting ($x_{raft} = 0$, Fig. 3.12c-d). A zero threshold $\varepsilon_{th}^p = 0^+$ allows to properly model the microstructure change. The evolution of the channel width has been modeled with a $K_{mc} = 1.4 \times 10^{-5} \text{s}^{-1}$. The coupling with the microstructure degradation is due to a non zero value of parameter $\Theta = 0.8$ (Fig. 3.12).

For creep in direction $<111>$ the same remarks than for direction $[001]$ apply: the nonlinearities, pronounced concerning the creep rate, are due to the microstructure change.

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with no need to introduce damage up to $\varepsilon^p = 0.05$ of plastic strain. Let us point out a key point in the success of the modeling: a zero plastic strain rate $\dot{\varepsilon}^p = 0$ is obtained by the model during the first hours of the creep test, up to $\approx 2.5\text{h}$ for $\sigma = 180\text{ MPa}$ creep test and up to $\approx 15\text{h}$ for $\sigma = 140\text{ MPa}$ creep test. There is no creep elongation as long as the Orowan stress contribution – parametrized by $\varpi$ – is large ($f^2 < 0$). When coarsening takes place, the term $\sigma_{\text{oro}}^{\parallel\omega}$ decreases so that apparent yield stress $\sigma_y + \mathcal{R}_\infty + \sigma_{\text{oro}}^{\parallel\omega}$ diminishes up to allow for creep (at $f^2 > 0$).

The identification presented in Fig. 3.12 is made at Hill parameter $h = 0.5$ (value theoretically justified in 3.9), with $\varpi = 0.8$ for the coupling with microstructure change. The value of the parameter $u_{\text{mc}} = \frac{U_{\text{mc}}}{k_B T}$ is set to 0.0012. Let us last point out that damage has not been introduced, it will be needed for a better modeling of creep at $\varepsilon^p > 0.03$.

Figure 3.12 – $<111>$ creep responses of CMSX-4 at 1050°C ($h = 0.5$, $\varpi = 0.8$): a) Creep curves $\varepsilon(t)$, b) $\dot{\varepsilon}^p$ vs $\varepsilon^p$ curves, c) $\gamma$ channel width curves $w_1(t) = w_2(t) = w_3(t)$, d) Index of rafting $x_{\text{raft}} = \frac{w_{eq}}{\text{tr} \mathbf{w}}$ as a function of accumulated plastic strain $p$. 

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3.8 Responses in monotonic tension

The tension curves are given in Fig. 3.13 for the two orientations $<001>$, $<111>$. We recall that material parameters are identified on the experimental results presented in chapter 2 and in Appendix N (SS set of material parameters). The proper modeling of $<001>$ tensile behavior at strain rate $10^{-3}s^{-1}$ strain rate is gained thanks to a saturation value of viscous stress for criterion function $f_1$ with identified viscosity parameter $\sigma_\infty = 600$ MPa (the curve computed without the saturating effect is shown in figure 3.14). A stress softening for the slowest tensile tests is observed in Fig.3.13a (at $10^{-5}s^{-1}$) whereas a plateau is observed for tensile tests at $10^{-4}$ and $10^{-3}s^{-1}$. This gradual stress softening for the slowest tension test results from the progressive degradation of the $\gamma'$ microstructure since this test is long enough to develop $\gamma'$ rafting (microstructures not shown here). Such a microstructure degradation is well-captured by the model since a decrease of the Orowan stress is observed under this strain rate conditions, while the Orowan stress is almost constant for the tests performed at $10^{-4}$ and $10^{-3}s^{-1}$. One also has to note that the stress peak observed just after yielding is not reproduced by our model since this peak results from the micromechanisms of dislocation deposition in vertical and then horizontal gamma-channel before recovery processes (e.g. dislocation climb) occur (Fedelich, 2002, Mughrabi, 2009). The softening rate in $<001>$ is related to the value of material parameters $U_\text{raft} \neq 0$ representing a stress enhanced $\gamma'$-rafting phenomenon (therefore a quicker softening of Orowan stress when $U_\text{raft}$ is taken larger). The softening produced by microstructural evolution is compensated by strain hardening during monotonic tension along $<111>$ (section 2.2.2). However, along this direction $\gamma'$-coarsening is observed when loading at at $10^{-5}s^{-1}$ (the result of this test are described in the section 2.2.2). Since no primary creep is observed during creep tests, no hardening is introduced in the modeling and so a slightly softening is reproduced at $10^{-5}s^{-1}$ (instead of a perfect plateau). Image analyses have been performed on SEM images of the specimen tested at $10^{-5}s^{-1}$ and the average $\gamma$ channel width has been measured. The Orowan stress (Eq. (3.4)) is computed by using this measure and the parameters identified by exploiting creep tests. The computed value is shown in the figure 3.13(b) (black dot) and compared to the Orowan stress variation predicted by the model (red dotted line). A good agreement between experimental data and the model prevision is found, thanks to modified viscosity law (3.66) for $<111>$ response in tension.

As anticipated in section 3.5.1, and as shown in figure 3.14(b), the standard Norton law $\dot{p}_2^N = \frac{f_2}{K_N}N$ underestimates the $<111>$ viscous stress at stress level higher than the one encountered in creep tests. To correctly describe the monotonic tensile tests along $<111>$, a stress dependent parameter $K_N$ has been computed by exploiting all the tensile tests realized and presented in the section 2.2.2.

The curves shown in figure 3.13(b) are computed using the corresponding viscosity law Eq. (3.66). Figure 3.15 presents the comparison between the model –using law (3.66)– and the experimental curve for the variable rate test. A good agreement is found between the model and the experiment, except after the phase at $10^{-5}s^{-1}$. During this phase it is expected that typical high rate deformation mechanisms set and these mecha-

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Figure 3.13 – a) $\langle 001 \rangle$ uniaxial tension curve at $10^{-5}\text{s}^{-1}$, $10^{-4}\text{s}^{-1}$ and $10^{-3}\text{s}^{-1}$ (Orowan stress also reported), b) $\langle 111 \rangle$ uniaxial tension curve at $10^{-5}\text{s}^{-1}$ and $10^{-3}\text{s}^{-1}$ (Orowan stress also reported) with $h = 0.5$, $\varpi = 0.8$. CMSX-4 at 1050°C (stress (model): thick lines, Orowan stress (model): dotted lines, experimental : thin black lines).

Nisms are not described by our modeling approach (see section 2.2.2).
3.9 Model rewritten in crystal plasticity framework

Single crystal material behavior is often modeled by means of the so-called crystal plasticity framework (Mandel, 1973, Hill and Rice, 1972, Rice, 1975, Asaro and Rice, 1977, Asaro, 1983, Peirce et al, 1983): the permanent strain $\varepsilon_p$ of the Representative Volume Element (RVE) of continuum mechanics is then the sum of the individual shearing contributions of the (active) slip systems, gathered by families. Criterion functions are formulated by the mean of the Schmid criterion, which is described in the following subsection.
Figure 3.15 – Complex tensile test curve obtained by modeling the $<111>$ viscosity by a Norton law and by computing the parameter $K_N$ with the equation 3.68.

### 3.9.1 Schmid criterion

In the subsection 1.1.2, it has been highlighted that plastic deformation is produced in crystals when dislocation loops propagate along the most closely packed directions contained in the most densely packed planes. For a dislocation loop to propagate, a threshold shear stress has to be overcome. This stress is commonly called “Critical Resolved Shear Stress” (CRSS or $\tau_c$). Once identified, its value is assumed to be equal for each plane belonging to a specific family (octahedral, pseudo-cubic and secondary octahedral in FCC crystals, as seen at page 13 and 16. The Schmid criterion (Schmid and Boas, 1935) states that plasticity is produced by a specific slip system if the shear stress acting on this system overcomes the CRSS. The shear stress acting on slip systems, called “Resolved Shear Stress” (RSS) and indicated by $\tau_g$, is computed by projecting the applied stress on each specific system $g$.

$$\tau_g = m_g \cdot \sigma \cdot n_g$$  \hspace{1cm} (3.80)

In Eq. (3.80), $n_g$ and $m_g$ are the unit vectors of the normal to the slip plane and the vector of the slip direction, respectively. The product $n_g \cdot m_g$ is the so-called Schmid factor. It is the product of the direction cosines of the stress projection on slip systems (figure 3.16 and Eq. (3.81)).

$$m_g = \cos^2 \chi^g \cos \theta^g$$  \hspace{1cm} (3.81)

Each octahedral, pseudo-cubic and secondary octahedral slip system is characterized by a Schmid factor (Ghosh et al, 1990). Systems having the highest Schmid factors are
then the first to be activated. The Schmid criterion then leads to write $g$ criterion functions at the slip plane scale, one for each slip system belonging to a family. An example is given in Eq. (3.82), where $g \in [1, 12]$ for octahedral / secondary octahedral systems and $g \in [1, 6]$ for pseudo-cubic slips.

\[ f_g = |\tau_g| - \tau_c = |m_g \sigma| - \tau_c \]

(3.82)

The original formulation, outcoming from the works of Mandel (1965, 1966), Hill (1966), Mandel (1973), has been widely used and extended over the years (Hutchinson, 1964, Hill, 1966, Rice, 1970, 1971, Hill and Rice, 1972, Asaro and Rice, 1977, Hill, 1979, Asaro, 1983, Peirce et al, 1983, Asaro, 1985). In (Méric et al, 1991a), isotropic and kinematic hardening have been added to criterion functions in order to describe the response of materials exhibiting more complex behaviors. For example, the interaction matrix has been introduced in order to account for the latent hardening coming from the dislocation interactions (Franciosi, 1985, Besson, 2001, Fivel and Forest, 2004, Gerard et al, 2013). A Cosserat theory for single crystals at finite deformation and curvature has been proposed recently by Forest (2012).

### 3.9.2 Crystal visco-plasticity

In standard crystal plasticity framework, the constitutive equation for elasto-visco-plasticity coupled with tensorial rafting are:

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— **elasticity law coupled with rafting**

\[
\sigma = \mathbb{E}(1 - \kappa_E \omega_E) : (\varepsilon - \varepsilon^p - \alpha(T - T_{\text{ref}}) \mathbf{1}) \approx \mathbb{E} : (\varepsilon - \varepsilon^p - \alpha(T - T_{\text{ref}}) \mathbf{1})
\]

(3.83)

with in practice the coupling of elasticity and of thermal expansion with rafting neglected.

— **resolved shear stresses** on octahedral and cubic slip systems \( g \) (Schmid and Boas, 1935)

\[
\tau^o_g = m^o_g \cdot \sigma \cdot n^o_g \quad \tau^c_g = m^c_g \cdot \sigma \cdot n^c_g
\]

(3.84)

with \( m^o_g, m^c_g \) the unit vectors of the slip directions and \( n^o_g, n^c_g \) the normals to the slip planes, \( \gamma^o_g \) the slip rates. In general multiaxial case, a maximum of 12 octahedral slip systems (with for each \( \tau^o_g = m^o_g \sigma \)) and of 6 cubic systems (with for each \( \tau^c_g = m^c_g \sigma \)) can be activated. \( m^o_g \) and \( m^c_g \) are the Schmid factor on octahedral and cubic slip systems (Ghosh et al, 1990).

- **In tension along the \( <001> \) crystal direction** there are 8 active octahedral slip systems at \( \tau^o = m^o_{001} \sigma \) with \( m^o_{001} = \frac{1}{\sqrt{3}} \), no active cubic slip systems.
- **In tension along the \( <111> \) crystal direction** there are 6 active octahedral slip systems at \( \tau^o_g = m^o_{111} \sigma \) with \( m^o_{111} = \frac{2}{3} \) and 3 active cubic slip systems at \( \tau^c_g = m^c_{111} \sigma \) with \( m^c_{111} = \frac{\sqrt{2}}{3} \).

— **plastic strain rate**

\[
\dot{\varepsilon}^p = \dot{\varepsilon}^{po} + \dot{\varepsilon}^{pc} = \frac{1}{2} \sum_{g=1}^{12} (n^o_g \otimes m^o_g + m^o_g \otimes n^o_g) \dot{\gamma}^o_g + \frac{1}{2} \sum_{g=1}^{6} (n^c_g \otimes m^c_g + m^c_g \otimes n^c_g) \dot{\gamma}^c_g
\]

(3.85)

allowing to define the total, octahedral and cubic macroscopic accumulated plastic strains as

\[
p = \int \sqrt{\frac{2}{3} \dot{\varepsilon}^p : \dot{\varepsilon}^p} \, dt \quad p^o = \int \sqrt{\frac{2}{3} \dot{\varepsilon}^{po} : \dot{\varepsilon}^{po}} \, dt \quad p^c = \int \sqrt{\frac{2}{3} \dot{\varepsilon}^{pc} : \dot{\varepsilon}^{pc}} \, dt
\]

(3.86)

— **viscosity laws** for octahedral and cubic slip systems (saturated hardening, coupling with Orowan stress),

\[
\tau_{\text{ORO}} = m^o_{001} \frac{k_{\text{ORO}} G}{\| \omega \|}
\]

(3.87)

\[
\dot{\gamma}^o_g = \left\langle \frac{-\tau_{voo}}{k^o} \ln \left( 1 - \frac{|\tau^o_g| - \tau_{\text{ORO}} - \rho^o}{\tau_{voo}} \right) \right\rangle + \frac{1}{k^o} \left[ \frac{1}{\max \{ \tau^o_g - \tau_{\text{lim}}, 1 \} } \right]
\]

(3.88)

\[
\dot{\gamma}^c_g = \left\langle \frac{|\tau^c_g| - \sigma^c \tau_{\text{ORO}} - \rho^c}{k^c} \right\rangle + \frac{1}{k^c} \left[ \frac{1}{\max \{ |\tau^c_g| - \tau_{\text{lim}}^c, 1 \} } \right]
\]

(3.89)

with, from macroscopic parameters of section 3.5.1, identified for the orientations \( <001> \) and \( <111> \)

\[
n^o = N, \quad \rho^o = m^o_{001} (\sigma_y + R_{\infty}), \quad k^o = m^o_{001} (8m^o_{001})^{1/N} K_N, \quad \tau_{voo} = m^o_{001} \sigma_{voo}
\]

(3.90)
Model rewritten in crystal plasticity framework

\[ n^c = \overline{N}, \quad \rho^c = m_{111}^c (\overline{\sigma} + \overline{R}_c), \quad k^c = m_{111}^c (3m_{111}^c)^{1/2}\overline{K}_N, \quad (3.91) \]

\[ \overline{\sigma}^c = \overline{\sigma} m_{111}^c, \quad \overline{k} = \frac{K}{m_{111}^c}, \quad k^c = m_{111}^c, \quad (3.92) \]

__microstructure degradation__, written at RVE macroscopic scale using "diagonal" Kelvin stress \( \overline{\sigma}^d = \mathbb{P}^d : \mathbb{\sigma} \) (projector \( \mathbb{P}^d \) defined in Eq. (E.5)), and its von Mises norm \( \sigma_{eq}^d = \sqrt{\frac{2}{3} \sigma^d : \sigma^d}, \)

\[ \dot{\omega} = \dot{\omega}^{raft} + \dot{\omega}_{mc} 1 + \sqrt{3} \dot{\omega}_{LSW} \frac{\omega}{\|\omega\|} \quad (3.93) \]

with

\[
\begin{align*}
\dot{\omega}^{raft} &= K_{raft} \exp \left( -\frac{Q - \min(\sigma_{eq}^d, \sigma_{lim}^{raft})}{k_B T} \right) H(p^o - \varepsilon_{th}^p) \mathcal{G} n^d \\
\dot{\omega}_{mc} &= K_{mc} \exp \left( -\frac{Q - \sigma_{eq} U_{mc}}{k_B T} \right) H(p^c - \varepsilon_{th}^p) \\
\dot{\omega}_{LSW} &= \frac{K_{LSW}}{3\omega_{LSW}} \exp \left( -\frac{Q}{k_B T} \right)
\end{align*}
\]

__volume fractions of \( \gamma' \) precipitates__, using crystal plasticity octahedral accumulated plastic strain \( p^o, \)

\[ \dot{f}_{\gamma'} = \left( 1 - \delta_{\gamma'} e^{-\frac{p^o \tau_{\gamma'}}{\varepsilon_{th}^p}} \right) \frac{f_{\gamma'}(T) - f_{\gamma'}}{\tau_{\gamma'}} \quad f_{\gamma'}(T) \text{ from Eq. (3.77)} \quad (3.95) \]

__\( \gamma' \) channel width tensor

\[ w = g(f_{\gamma'}) w^{0}_{001} \omega \quad g(f_{\gamma'}) = \frac{1 - f_{\gamma'}}{1 - f_{\gamma'}} \quad (3.96) \]

Note that the calculation of the \( \gamma' \) precipitates volume fractions \( f_i, f_s, \) and of the physical \( \gamma \) channel width tensor \( w \) can be made in a post-processing manner as they do not explicitly affect the mechanical behavior (they affect it but hidden inside global \( \gamma' \)-rafting variable \( \omega \)).

### 3.9.3 Tension along the \( <001> \) crystallographic direction

For an uniaxial loading along the \( <001> \) crystallographic direction, there are 8 active octahedral slip systems and no active cubic slip systems so that one exactly recovers Eq. (3.69)

\[ |\dot{\varepsilon}^p| = 8m_{001}^o \left( \frac{m_{001}^o |\sigma| - m_{001}^o \kappa_{001G} G}{\|\sigma\|} - p^o \right)^{n^o} + \left( \frac{|\sigma| - R_{oo} \kappa_{ooG} G - \sigma_o}{K_N} \right)^N <001> \text{ orientation} \quad (3.97) \]

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3.9.4 Tension along the $<111>$ crystallographic direction

For an uniaxial loading in direction $<111>$, the accumulated plastic rate is the sum of the contributions of 3 octahedral slip systems and of 6 cubic slip systems,

$$|\dot{\varepsilon}_P| = 3m_{111}^c \left( \frac{m_{111}^c |\sigma| - \sigma_{ORO} G}{k^c} \right) n^c + 6m_{111}^o \left( \frac{m_{111}^o |\sigma| - m_{111}^o \sigma_{ORO}}{k^o} - \rho^o \right) n^o \quad (3.98)$$

or, using Schmid factor $m_{111}^o = \frac{2}{3} m_{001}^o$ and parameters definition (3.91),

$$|\dot{\varepsilon}_P| = \left( \frac{|\sigma| - R_\infty - \sigma_y - \sigma_{ORO}}{K_N} \right) + \frac{1}{2} \left( \frac{2}{3} |\sigma| - R_\infty - \sigma_{ORO} - \sigma_y \right) <111> \quad (3.99)$$

which, making the approximation $\frac{1}{2} \approx \frac{2}{3} \approx h$, is found close to be Eq. (3.70) obtained from macroscopic Klevin modes based model of section 3.5.1.
3.10 Structures computations

The proposed model with tensorial representation of $\gamma'$-rafting is implemented in the Z-set finite element code. The code is presented in the Appendix Q. The set corresponding material behavior model being called “RaftX” in Z-set. The differential equations are solved with a fourth order Runge-Kutta explicit integration scheme. It has to be remarked that the code includes isotropic hardening (exponential law) but the associated material parameters are set to zero (see Appendix S), so there are no differences with the behavior presented in the present chapter. 3D structural computations are presented next, mainly in order to illustrate that the proposed constitutive model is ready to be used for the design of structures. Two sets of material parameters have been identified in this study for CMSX-4 (both are presented in the Appendix N):

— the first one, is dedicated to small strains (SS set of material parameters presented in the N, up to the 5% of creep strain, therefore with no damage). This has been used to compute the single gauss point responses presented in the previous sections (3.7 and 3.8);
— the second one, identified with the hypothesis of finite strains (locally, i.e. at Gauss point level the stress is updated by using the strain computed at the previous time step) is aimed to reproduce the material response on a wider deformation range including the coupling with damage (proposed in next chapter).

Even if all the finite element computations presented in the following subsections have been carried out in the small strains assumptions, the second set of parameters has been used. This has been done in order to account of the fact that creep tests are performed at constant imposed load. The input files of the simulations are presented in the Appendix S. In the following section several results are presented in terms of equivalent plastic strain. It is defined as in the following equation.

$$\varepsilon_{eq}^p = \sqrt{\frac{2}{3} \varepsilon^p : \varepsilon^p}$$  \hspace{1cm} (3.100)

3.10.1 Torsion of a thin walled tube

The experiment performed by Nouailhas and Cailletaud (1995) has shown that a non-homogeneous plastic deformation field is observed when loading in torsion a single crystal thin walled tube having its main axis aligned with the $<001>$ crystallographic orientation. This heterogeneity is due to the cubic symmetry both of the crystal and of its mechanical response. The stress orientation changes along the circumference, so different crystallographic orientations are loaded depending on the $\theta$ angle (figure 3.17).

As indicated in the Appendix H, at $\theta = \frac{\pi}{4} + \frac{k\pi}{2}$ from $<001>$, yielding first initiates for the $<110>$ orientations. Visco-plasticity models based on the Schmid criterion and including the cubic slips, correctly describe this phenomenon (Nouailhas and Cailletaud, 1995, Méric et al, 1991b, Fivel and Forest, 2004). In the present study, a non-quadratic norm is used for the equivalent stress within the visco-plastic criterion function $f_2$ (Eq. 3.3.3 and Appendix G). As explained in subsection 3.3.6 and in Appendix H, this choice...
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Figure 3.17 – Example of a single crystal thin walled tube, the vertical axis 2 corresponds to the axe of the tube and with the \([010]\) crystallographic orientation.

allows to recover the heterogeneous deformation field when a norm order \(1 < a < 2\) is chosen. Figure 3.18 presents the yield criterion function \(f_2\) computed when using the non-quadratic norm (Eq. 3.3.3). In the \(\sigma_{12}-\sigma_{23}\) plane, it has a crystal-plasticity like shape such as the one presented in Nouailhas and Cailletaud (1995) for \(1 < a < 2\), while \(a = 2\) corresponds to the homogeneous case.

Figure 3.18 – Theoretical yield surfaces predicted by computing the equivalent stress using a non-quadratic norm in the criterion function \(f_2\) Appendix (G).

For the finite element computations, a thin walled tube having a 1 mm thickness is considered. The geometry of the tube and its finite element approximation are presented
in the figure 3.19. The mesh (courtesy of C. Defaisse, Mines Paris Tech) is made up of 960 elements of type c3d20 (figure 3.19). They are 3D continuum elements with 20 nodes, thus the finite element model has 16320 degrees of freedom. The boundary conditions and the loading are presented in the figure 3.20. The displacements of the nodes belonging to the last two lines of elements are constrained. A rotation around the axis 2 is applied to all the nodes belonging to the elements contoured by the red rectangle in figure 3.20. The loading is such that a maximum of 2 rad is reached in 2 seconds (the loading rate is then equal to 1 rad/s). The results obtained with value \( a = 1.1 \) for non quadratic norm are shown in figure 3.22. Figure 3.21(a) and 3.21(b) contain the computed equivalent plastic strain field and accumulated plastic strain rate field respectively. Both present the expected heterogeneity at angle \( \theta = \frac{\pi}{4} + k \frac{\pi}{2} \). This first computation is realized with a simple Norton’s law (\( \bar{\kappa} = 0 \) and \( \bar{K}_{N_{exp}} = \bar{K}_{N} \), see section 3.5.1) while all the others are realized by modifying Norton law, making use of Eq. (3.68). Note that the norm order \( a \), in this context, acts like a parameter. It plays a main role on the visco-plastic response along crystallographic orientations different from \(<001> \) and \(<111> \) and also in the multiaxial case. The value of plastic deformation at \( \theta = \frac{\pi}{4} + k \frac{\pi}{2} \) – hence, the visco-plastic properties along the \(<011> \) – depends on the chosen \( a \), as shown in figure 3.23 and 3.24. Figure 3.21 presents the different global response that the structure has according to parameter \( a \).

![Figure 3.19 – (a) Schematic representation of c3d20 elements, (b) Geometry of the considered tube and its finite element mesh (axis 2 coincides with the vertical axis of the tube).](image)

The matrix channels sizes along the tube have been computed, they are not plotted as the total simulation time (2 seconds) is too short to observe a significant variation of \( \gamma \) channels: the three channels evolve together and their computed final value is 80.832 nm (when their the initial value at 1050°C is \( w_{001} = 80.83 \text{nm} \)).
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Figure 3.20 – Boundary conditions applied for computing the response of a single crystal thin walled tube subjected to torsion (courtesy of C. Defaisse, Mines Paris Tech).

Figure 3.21 – Global structure response torque $M_2$ vs rotation angle $\theta$. Loading rate of 1 rad/s.
Figure 3.22 – Equivalent plastic strain field (a) and accumulated plastic strain rate field contour of the accumulated plastic strain rate (s\(^{-1}\)) (a) (gauss point values) on a single crystal thin walled tube subjected to torsion. The axis 2 coincides with the vertical axis of the tube.
Figure 3.23 – Equivalent plastic strain field after 2s of torsion on a single crystal thin walled tube (gauss point values) computed by using different norm order a. Loading rate of 1 rad/s.

Figure 3.24 – Accumulated plastic strain rate field (s\(^{-1}\)) after 2s of torsion on a single crystal thin walled tube (gauss point values) computed by using different norm order a. Loading rate of 1 rad/s.

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
3.10.2 Creep at 1050°C

A finite element mesh of the specimens used for the creep tests presented in chapter 2 (subsection 2.2.1) has been realized with Abaqus and imported in Z-set. The geometry of the specimens is given in the Appendix A. The mesh (courtesy of prof. D. Ryckelynck, Mines Paris Tech) is presented in figure 3.25. It is made up of 21504 3D continuous element of type c3d20 (figure 3.19), corresponding to 276675 degrees of freedom. The average size of a mesh element in the gauge length is shown in figure 3.27. The mesh has been refined in the stress concentration zones in order to avoid geometrical discontinuities. A negative pressure of -140 MPa has been applied to the upper and lower faces of the heads (Fig. 3.25). Because of their dimensions, the heads of the specimen are more rigid than its gauge length. For this reason the surfaces where the pressure is applied do not deform. A constant force of 3956.4 N is then applied on each surface. Computations then account for the fact that real creep tests (presented in the chapter 2) have been carried out under a constant load. The maximum computation time corresponds to the observed creep lifetimes (see section 2.2.1). Figure 3.26 details the boundary conditions.

Figure 3.25 – Finite element model of the creep specimens and loading conditions applied to simulate creep.

It has been chosen to simulate the creep tests realized during the present Ph.D. at 1050°C and 140 MPa along $<001>$ and also the one performed by R. Giraud during his Ph.D. thesis along $<111>$ under the same loading conditions (Giraud, 2013a). In the first case the vertical axis $z$ coincides with one $<001>$ orientation, in the second one with $<111>$ orientation. It has to be remarked that in the experiment realized by Giraud a misalignment exists between the theoretical $<111>$ crystallographic orientation and the vertical axis of the tested specimen (D’ sample, figure 2.2). The simulation does not account for this difference.

The results are shown in the figures from 3.28 to 3.36. The equivalent plastic strain fields are presented in the figures 3.28 and 3.29. As observed in the experiments (section 2.2.1), plasticity develops more and faster along $<001>$ then along $<111>$. Plastic
strain localizes because of the softening induced by the microstructural degradation.

Figure 3.31 and 3.32 show the creep curves computed by considering the axial strains of three nodes (N1, N2 and N3). In the same plots the creep curve computed on a single gauss point is reported (denoted GP). These curves exhibit a structural effect, the three nodes deforming at different rates (not to far from the gauss point curve GP, also reported in the figure). The global displacement $u$ (normalized by the gauge length $L$) versus time is plotted in figure 3.30 for both $<001>$ creep at 1050°C MPa and $<111>$ creep at 140 MPa. This compares well with the experiments (experimental results are shown in section 2.2.1).

The $\gamma$ channels width distributions are presented in the figure 3.33 and 3.34. As expected after tensile creep along $<001>$, the channels normal to tensile direction widen (vertical width $w_3 = w_{33}$), especially where plastic deformation concentrates. At the same time transverse channels close ($w_1 = w_{11}$ and $w_2 = w_{22}$ become equal to zero). In the heads of the specimens the $\gamma$ channel widths do not evolve significantly (their value is close to the initial one of 80.83 nm). Consequently the rafting index $x_{raft}$ becomes equal to 1 (figure 3.35(a)). At the opposite, during creep along $<111>$, the isotropic evolution due to mechanical coarsening leads to a situation where the three channels have the same sizes (figure 3.34). Then, as shown in figure 3.35(b), the $\gamma'$-rafting index remains equal to zero. The $\gamma$ channel sizes presented in figure 3.34 correspond to the ones computed

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
Figure 3.28 – Equivalent plastic strain field (a) after 243h of creep along the $<001>$ crystal orientation and (b) 622h of creep along the $<111>$ crystal orientation.

Figure 3.29 – Accumulated plastic strain rate field ($s^{-1}$) (a) after 243h of creep along the $<001>$ crystal orientation and (b) 622h of creep along the $<111>$ crystal orientation.
Figure 3.30 – Global displacement-time response $u/L$ vs $t$: (a) creep along $<001>$ at 1050°C and 140 MPa (b) $<111>$ creep at 1050°C and 140 MPa.

Figure 3.31 – Creep test at 1050°C and 140 MPa along the $<001>$ crystal orientation: nodes selected to extract the creep curves (a), contour of the accumulated plastic strain rate ($s^{-1}$) after 243h (b) and stress strain curves for selected nodes (GP = Gauss Point).

After 197h (the time of the measurements of Fig. 3.12, we lack of later $\gamma$ channels size measurements). Note that the matrix channel widths are not bounded by the evolution law for mechanical coarsening (Eq. (3.75)) and probably should be, according to the stress level. This point is left to further work.
Figure 3.32 – Creep test at 1050°C and 140 MPa along the <001> crystal orientation: nodes selected to extract the creep curves (a), contour of the accumulated plastic strain rate (s⁻¹) after 622h (b) and stress strain curves for selected nodes (GP= Gauss Point).

Figure 3.33 – γ channels width (nm) after 234h of creep along the <001> crystal orientation at 1050°C and 140 MPa.
Figure 3.34 – $\gamma$ channels width (nm) after 197h of creep along the $<111>$ crystal orientation at 1050°C and 140 MPa.

Figure 3.35 – $\gamma'$-rafting index after creep at 1050°C: (a) after 234h of 140 MPa creep along $<001>$ orientation, (b) after 197h of 140 MPa creep along $<111>$ orientation.
Figure 3.36 – $\gamma$ channels width evolutions at different nodes and for Gauss Point (GP) calculation, (a) $\gamma'$-rafting phenomenon for $<001>$ creep at $w_3$ increasing and $w_1 = w_2 \rightarrow 0$, (b) mechanical coarsening for $<111>$ creep at $w_1 = w_2 = w_3$ increasing.
3.10.3 Bi-notched specimen

Inspired from the study presented in le Graverend et al (2014a) for the MC2 alloy, the creep response of a bi-notched specimen (geometry is shown in figure 3.37) has been computed for CMSX-4 at 1050°C. The corresponding experiment has not yet been performed so only qualitative comments will be made.

The finite element model (courtesy of J.-B. le Graverend, asst. prof. at Texas A&M University) are presented in figure 3.38. The vertical axis y (also axis 2) corresponds to the <001> crystallographic orientation. The mesh is made up of 3893 elements of type c3d4 (tetrahedral 3D continuous elements with 4 four nodes, 11679 total degrees of freedom). A negative pressure is applied to the upper surface in order to simulate tensile creep at 110 MPa in the notch sections (a force of 476 N is applied to the upper surface), bottom face has zero applied displacements. The loading path is presented in figure 3.39. The (real) loading duration is 40h (144000s).

Even if no experiments have been performed using CMSX-4 bi-notched specimens, let us remark that the results of the simulation are in qualitative agreement with what has been observed by le Graverend et al (2014a). The $\gamma/\gamma'$ rafting is faster where plasticity is higher (figures 3.40 and 3.41). Moreover, thanks to the proposed 3D description of visco-plasticity and microstructural evolutions, the effect of the real stress state and of the anisotropy of the material are accounted. In fact, even if the specimen is oriented along the <001> crystallographic direction, the $\gamma$ channel size distribution is very different from the one computed for the creep specimen oriented along <001> (figure 3.33). Figure 3.40 shows that $\gamma'$-rafting is predicted between the notches ($w_{22}$ while $w_{11}$ and $w_{33} \rightarrow 0$) while $\gamma'$-mechanical coarsening is predicted in the notch roots ($w_{22} = w_{33} = w_{11}$ widen).
Structures computations

Figure 3.38 – Finite element approximation of a bi-notched specimen (courtesy of asst. prof. J.-B. le Graverend)

Figure 3.39 – Loading path applied to simulate the creep response at 1050°C of a bi-notched specimen.

In order to estimate the numerical performances of proposed modeling, we have finally carried out the same finite element computation by using different elasto-visco-plasticity constitutive equations developed for single crystal superalloys. For this purpose, the computation time of the model coupled to tensorial γ microstructure evolution proposed in this chapter – named ”RaftX” in Z-set code – has been compared to those of the following list.

— The version of the Polystar model proposed in (le Graverend, 2013). It accounts for γ/γ′ rafting and load-free coarsening, for γ′ dissolution and for (visco-)plasticity produced by octahedral slips (crystal plasticity). In the model proposed in le Graverend (2013) visco-plasticity is coupled to damage (with a revisited Rabotnov-Katchanov evolution law Rabotnov (1969)). However, in order to compare objectively Polystar and RaftX numerical performances, damage has been deactivated in Polystar.
— An isotropic Norton flow without threshold (identified on CMSX-4 at SAFRAN

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Helicopters Engines (SHE), details are contained in the internal technical notes (SHE) Hervier (2010) and Sahores and Hervier (2008)).

— A visco-plasticity model based on crystal plasticity and accounting for octahedral and cubic slips (of Méric-Cailletaud type without hardening (Méric et al, 1991a)). The material parameters have been identified starting from the sets of parameters proposed in the Appendix N (Small Strain set) and by applying the cross-identification equations presented in subsection 3.9.2.

The computation times are presented in the table 3.1.

<table>
<thead>
<tr>
<th>Model</th>
<th>CPU time</th>
</tr>
</thead>
<tbody>
<tr>
<td>RaftX (Graverend et al, 2014) without damage</td>
<td>3572s</td>
</tr>
<tr>
<td>Polystar (without threshold)</td>
<td>18540s</td>
</tr>
<tr>
<td>Norton (octahedral &amp; cubic slips)</td>
<td>2205s</td>
</tr>
<tr>
<td>Crystal plasticity</td>
<td>5561s</td>
</tr>
</tbody>
</table>

Table 3.1 – CPU time required to compute the bi-notched specimen response after 40h of creep at 1050°C and 101 MPa (figure 3.38) corresponding to four visco-plasticity model used for single crystal superalloys: RaftX (Desmorat et al, 2017), Polystar (version proposed in le Graverend et al (2014a), here used without damage), Norton flow and crystal plasticity accounting for octahedral & cubic slips (Méric-Cailletaud-like model (Méric et al, 1991a) without hardening).

The faster computation time is of course obtained by simple Norton’s visco-plasticity
Figure 3.41 – Components of the plastic deformation tensor $\varepsilon^p$ after 40h of creep at 1050°C and 101 MPa along a bi-notched specimen oriented along the $<001>$ crystallographic orientation.

model. The “RaftX” model developed in the present Ph.D. thesis has a promising computation time (recall that this is a single scale model, an internal variable, tensorial, being representative of the microstructure evolutions).

The time costs are only indicative at this stage, more computations are needed to unambiguously conclude an the model CPU time efficiency.

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3.11 Conclusions

Starting from already available models / sets of constitutive equations in the open literature (Tinga et al (2009b), Fedelich et al (2009, 2012a), Cormier and Cailletaud (2010b)) accounting for for the physical and mechanical descriptions of the $\gamma$-$\gamma'$ rafting, coarsening and the $\gamma'$ dissolution process, an original phenomenological model for nickel based single crystal superalloys having a $\gamma$-$\gamma'$ negative misfit (mostly Ni-base superalloys like CMSX-4 and SRR99) has been proposed. The description of the microstructure state and evolution by the mean of the tensorial variable $\omega$ (of components $\omega_{ij}$) has been proved to be consistent with the $\gamma$ channel evolution happening during creep and monotonic tension tests along $<001>$ and $<111>$. The proposed evolution law not only allows to account for the correct morphological changes but also for the different kinetics which characterize the $\gamma'$-rafting, mechanical coarsening and load-free coarsening. Finally, using a tensor variable to account for the 3D actual microstructural state (instead of a scalar one, see see Appendix M) has been found to be fundamental under alternated loadings.

Thanks to the correct description of microstructural evolutions and thanks to the coupling with visco-plasticity via the macroscopic Orowan stress, it has been possible to reproduce the softening of the creep and monotonic tensile responses up to quite important strains without introducing any damage law. The Kelvin mode based visco-plasticity framework introduced is a key point to model both the visco-plasticity and the microstructural evolutions dependency on the crystal orientation. The two criteria formulation and the introduction of a non quadratic norm into the pseudo-cubic yielding criterion has turned out to be consistent with crystal plasticity models and physics: a heterogeneous deformation field is obtained when loading a thin walled tube under torsion loadings. A formulation of the model in the crystal plasticity framework has also been given.

Moreover, the model has been implemented in the Z-set finite element code (Burlet and Cailletaud, 1991, Besson et al, 1998). Results of Finite Element computations using real geometries confirm the effectiveness of the modeling to reproduce 3D effects of both visco-plasticity and microstructural evolutions.
Chapter 4
Rate dependent ductility and damage threshold: application to CMSX-4

4.1 Introduction

This chapter deals with the modeling of damage during creep of CMSX-4 alloy and, more generally, of Ni base single crystal superalloys. As for the visco-plasticity modeling, isothermal creep and monotonic tension at 1050°C along the $<001>$ and $<111>$ crystallographic directions will be the final applications (motivations are listed in the General Introduction and in the section 3.2).

The damage modeling is intended to complete the description of the material response developed in chapter 3, so it has as main objectives a good description of both tertiary creep stages and evolution of the tensile ductility as a function of the strain rate. In subsections 1.3.1 and 2.2, it has been highlighted that the main cause of the initial plastic strain acceleration during creep of Ni base single crystal superalloys is the microstructure degradation at the precipitation scale (Fedelich et al, 2012b, le Graverend et al, 2017) and that this latter is also responsible for the softening observed during slow monotonic tension tests, section 1.3.2 (Gabrisch et al, 1994, Diologent et al, 2002, Cormier, 2006, Tinga, 2009a, Giraud, 2013a). Modeling this creep acceleration and softening by the mean of microstructural evolution and by its coupling with visco-plasticity has been the main objective of the chapter 3. Hence, the damage modeling is targeted to reproduce the additional degradation which is a consequence of the following (damaging) mechanisms:

— the shearing of $\gamma'$ particles, which is observed in the last stages of tertiary creep and at the end of monotonic tensile tests (Sugui et al, 2000, Link et al, 2005, Sarosi et al, 2007);
— the formation of TCP phases, as suggested by Reed et al (2007b);
— the $\gamma/\gamma'$ topological inversion, observed in the final stage of tertiary creep when the undissolved precipitates volume fraction is higher than 0.5 (Mughrabi, 1996,
Epishin and Link, 2004, Fredholm and Strudel, 1984, Caron et al, 2008);
— the nucleation, growth and coalescence of microvoids and microcavities observed
during the last stages of tertiary creep (Link et al, 2006, le Graverend et al, 2017).
As detailed in the section 2.3, oxidation also contributes to the degradation of mechanical
properties, especially during non-isothermal tests. However, no remarkable oxidation
effects have been observed during isothermal creep at 1050°C (see experimental results
in subsections 2.2.1 and 2.2.2), thus oxidation is not included in the present modeling.
Hence, in details, the damage modeling has to reproduce:
— an additional plastic strain acceleration in the last stages of tertiary creep, starting
from $\approx 2 - 5\%$ of plastic deformation (subsections 1.3.1, 2.2.1 and 3.7);
— an additional softening during monotonic tension, starting from a different plastic
strain ($\geq \approx 10\%$) according to the strain rate (see subsections 1.3.2);
— creep lifetimes (figure 2.7 and 2.2.2) and monotonic tension ductility;
In the present work, it has been chosen to model damage according with Continuum
Damage Mechanics (Hult, 1979, Lemaitre, 1992, Lemaitre and Desmorat, 2005). Hence,
damage is modeled at the mesoscale by introducing the homogenized isotropic scalar state
variable $D$. Classically, this internal variable accounts for the degradation of the mechan-
ical properties due to discontinuity creation and propagation in continua, as for example
the creation and growth of microvoids or microcracks. In this work, it is also supposed to
account for the degradation produced by all the above mentioned phenomena (i.e. shearing
of $\gamma'$ particles, $\gamma/\gamma'$ topological inversion, etc...). Its evolution is modeled using a rate
(kinetic) damage law $\frac{dD}{dt} = ...$ governed by anisotropic (cubic) plasticity and enhanced by
stress triaxiality. The coupling between damage and the visco-plasticity model presented
in the chapter 3, is made thanks to the effective stress concept. Furthermore, a novel
rate sensitive damage threshold is introduced in order to reproduce the (anisotropic) rate
dependency of the damage onset.

The details of the proposed model are presented in the form of a scientific article. This
is the reason why several elements of the modeling are recalled in detail in this chapter
even if they have already been presented/defined previously, especially in chapter 3. For
instance, the harmonic decomposition of the elasticity tensor and the Kelvin modes based
modeling of visco-plasticity have already been presented in the section 3.3. Similarly,
the description of the tensorial modeling of microstructural evolutions has already been
described in the section 3.4.

The material parameters are identified on the experimental results presented in the
chapter 2. They are presented in the Appendix N (FS parameter set). Finally, structure
computations are presented at the end of the chapter, in section 4.3.

4.2 Rate dependent ductility and damage threshold:
application to Nickel-based single crystal CMSX-4

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature:
a tensorial microstructure-sensitive approach
Rate dependent ductility and damage threshold: application to Nickel-based single crystal CMSX-4

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Abstract

A phenomenological damage model is proposed to account for the strain rate dependency of the damaging processes at high temperature. Mechanical softening during tertiary creep and monotonic tensile load are modeled by an isotropic scalar internal variable $D$, whose evolution is described using a rate damage law $\frac{dD}{dt}=-\gamma$ governed by plasticity and accounting for the enhancement by stress triaxiality. A novel rate sensitive damage threshold is introduced in order to reproduce the rate dependency of the onset of damaging processes. Damage evolution is coupled with the visco-plasticity model developed by the same authors for single crystal superalloys and accounting for microstructural evolution (like $\gamma$-rafting and coarsening) in Desmorat et al (2017). The curves presented in this article are identified at 1050$^\circ$C for the Ni base single crystal superalloy CMSX-4 but the proposed rate sensitive threshold modeling can be applied to other alloys showing a rate sensitive damage onset, as for example the single crystal superalloys MC2 (Cormier, 2006) but also the polycrystalline aggregate AD 730\textsuperscript{TM} (Thébaut et al, 2016).

Key words: damage mechanics, Orowan stress, rafting, rate sensitive ductility, tertiary creep, visco-plasticity

1. Introduction

Nickel-based single crystal superalloys are state of the art materials for the design of high temperature components of aero-engines and land-based gas turbines for power generation (Pollock and Tin, 2006, Reed, 2006). They are typically used for the manufacturing of first stages of blades and vanes in such gas turbines and they can even be used for the manufacturing of intermediate and low pressure (uncooled) turbine blades in the most advanced engines for aircraft propulsion. During service operations, different kind of damage may develop along a blade profile, involving creep and/or fatigue and/or oxidation assisted damage, depending on the type of engine and operating conditions. As an example, creep is the main life limiting solicitation for uncooled turbine blades while thermo-mechanical fatigue damage are mainly observed for internally cooled components (Zhang, 2016). In the present paper, a special focus will be paid to damage, with a special emphasis on identifying when its development, in the sense of a loss of the load bearing capacity, has a major contribution to creep acceleration and monotonic tension softening. Given the variety in creep curves shapes that can be encountered when changing creep conditions (Svoboda and Lukas , 1998, Reed et al., 1999, Matan et al, 1999a, Reed, 2006, Kindrachuk and Fedelich, 2012), it is often difficult to clearly identify when cavitation or development of cracks are really active based on the macroscopic creep curve. Damage, in the sense of a loss of load bearing section, may already be active at low temperature/high stress despite no clear tertiary creep stage is noticed (MacLachlan et al, 2002, MacLachlan and Knowles, 2001, MacLachlan et al, 2001), while at high temperature, a continuous creep acceleration may be noticed (Epishin et al, 2001, Matan et al, 1999b), without any noticeable increase in porosity, both in terms of pores density and pore volume fraction. Indeed, as noticed recently by le Graverend et al (2017) using MC2 first generation Ni-based single crystal superalloy followed by X-ray tomography, the onset of tertiary creep in the 900$^\circ$C-1100$^\circ$C temperature range results from the $\gamma/\gamma$ microstructure degradation, under the form of a topological inversion (Epishin et al, 2001, Matan et al, 1999b, Caron et al, 2008, Epishin et al., 2000). This kind of microstructure degradation may be qualified as a microstructural damage and is sometimes considered as a “true” damage (McLean and Dyson, 2000), even if not inducing a loss of stiffness or load bearing capacity. Reed et al (2007) also observed that the very steep tertiary creep at 1150$^\circ$C / 100 MPa in CMSX-4 Ni-based single crystal superalloy is not controlled by the initial pore volume/size. Indeed, creep lifetimes of this alloy under these creep conditions remain unaffected after applying a Hot Isostatic Pressing (HIP) treatment devoted to close pores. A similar conclusion was observed more recently by Steuer et al (2015) comparing creep properties in a wide temperature range (from 750$^\circ$C to 1200$^\circ$C) using AM1 Ni-based single crystal superalloys processed using either a Liquid Metal Cooling or a standard Bridgman solidification techniques (both techniques leads to different pore size distribution). Similar questions also arise about the monotonic tensile response. Many literature studies

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show that at high temperatures ($T > 900^\circ C$) and low strain rates ($\dot{\varepsilon} \approx 10^{-5} s^{-1}$) the monotonic tensile response of this class of materials is characterized by the same deformation/damage mechanisms observed during creep (Gabrisch et al., 1994, Cormier, 2006, Diolomenet, 2002, Giraud, 2013). Hence, damage mechanisms (i.e., cavitation and $\gamma'$ shearing) contribute to the degradation of the mechanical properties only when a certain amount of softening—and thus of plastic deformation—has already been produced. Different deformation/damaging mechanisms are instead observed during monotonic tensile tests performed at high temperature and higher strain rates. In this case no time is left to the microstructure to evolve and to cavities to diffuse. Thus, deformation is produced by the progressive filling of constant-size matrix channels by dislocations. Plastic damage, enhanced by necking, takes places only at the end of the tests. Overall creep and tensile test results thus suggest that the mechanical damage onset is a rate governed phenomenon.

In the context of Continuum Damage Mechanics (Lemaître and Chaboche, 1985, Lemaître, 1992), kinetic damage laws with loading dependent damage thresholds have been introduced: in the works of Lemaître and Doghri (1994) for High Cycle Fatigue and of Sermage et al. (2000), Desmorat and Otin (2008) and of Marulli (2011) for creep-fatigue. In these works, the loading dependency of the accumulated plastic strain at damage initiation is due to a formulation of the damage threshold in terms of stored energy density. The corresponding constitutive equations apply to creep and fatigue—of a $2\frac{1}{2}$ Cr Mo steel, of HA188 alloy and of AM1 and CMSX-2 single crystal superalloys—but they do not model the ductility in monotonic tension.

To answer the key question, which is when so-called mechanical damage starts to have a prominent contribution to the material mechanical response, one will next use a microstructure modeling approach recently developed for single crystal superalloys (Desmorat et al, 2017) and identified at $1050^\circ C$ for the CMSX-4 alloy. This visco-plastic model (whose key points are described in the section 7), accounts for the $\gamma'$-rafting, coarsening and dissolution. According with what observed by le Graverend et al. (2017), it is intended to describe the degradation of the mechanical response until the firsts stage of tertiary creep (up to $\approx 2 - 5\%$ of plastic deformation) and the first mechanical softening observed during slow monotonic tension (up to $\approx 10\%$ of plastic deformation). The objective of the continuum damage modeling proposed in this article is then to describe the last stage of creep as well as monotonic the tensile response of single crystal superalloys. The curves presented next are identified on the tests considered in Desmorat et al (2017), but specific experimental tests have been performed expressly for the present study in order to enrich the experimental database for the mechanical modeling.

Moreover, the formulation of Kelvin projectors for cubic materials will be based on the harmonic decomposition of the elasticity tensor $\mathbf{E}$ having cubic material symmetry (Backus, 1970, Onat, 1984, Olive et al., 2017b). This will allow us to compute Kelvin projectors without using the spectral decomposition.

Finally, it has to be highlighted that a rate sensitive ductility at high temperature has been observed not only for single crystal superalloys (Gabrisch et al, 1994, Cormier, 2006), but also for other materials presenting very different deformation/damage mechanisms. Examples are the nano-twinned crystal of pure copper (Lu et al, 2005), the oxide dispersion strengthened (ODS) steels (Steckmeyer, 2012) and the polycrystalline aggregates AD 730TM (Thébaud et al, 2016), which all show a positive rate sensitivity of the (mechanical) damage onset and, as a consequence, a positive rate sensitivity of the ductility. Thus, the damage model proposed in this article can be used for modeling the monotonic mechanical response of other materials, having visco-plastic properties different from the one observed for single crystal superalloys. Further details are given in next section, where the damage modeling issues for CMSX-4 and AD 730TM are described.

2. Background and motivations

As mentioned, a rate sensitive ductility has been observed on $\gamma'$ hardened superalloys such as the AD 730TM and the CMSX-4 (Figure 1 and Figure 3).

For the AD 730TM this rate dependency can be attributed to oxidation: higher loading rates lead to a shorter interaction with the environment and to a lower time to rupture (Figure 1, Thébaud et al (2016)),

- at high strain rate $\dot{\varepsilon}$, the specimens deform until the plastic damage—enhanced by necking—classically leads to rupture (Kachanov, 1958, Rabotnov, 1969); in this case oxidation does not play a main role in the damage process,
- inversely, during the tests performed at lower strain rate ($\dot{\varepsilon} = 10^{-4} s^{-1}$ in Figure 1), oxidation has enough time to develop and to contribute to the degradation of the material.

Figure 2 is a post-mortem observation of an AD 730TM Fin Grain (FG) specimen tested under tensile creep at 700°C and 250 MPa. Since AD 730TM FG is a polycrystal, oxidation takes place at grain boundaries and it is present even at 700°C.

CMSX-4 has no grain boundaries, it is a Ni based single crystal superalloy. Its oxidation is observed only at temperature in excess of 900°C and this phenomenon does not impact significantly the mechanical response of the material (Reed, 2006). At such temperature levels, the main causes of the material degradation are

- first, the microstructural evolution of the $\gamma'$ phase, i.e. the rafting phenomenon (Tien and Copley, 1971, Tien and Gamble, 1972, Caron and Khan, 1983, Draper et al., 1989, Pollock and Argon, 1994, Reed et al., 1999), see Fig. 5),
– second, after a non negligible threshold, the standard plastic damage (le Graverend et al, 2017, Link et al., 2006, Srivastava and Needleman, 2015).

Two types of microstructural evolution affect the CMSX-4 response: the rafting and coarsening of the hardening phase.

The first consist in a morphology change of the cubic $\gamma - \gamma'$ microstructure (Appendix C). The coexistence of the two $\gamma/\gamma'$ crystal phases having different lattice parameters produces internal residual stresses at the microscale (Pollock and Argon, 1994, Kamaraj, 2003). When the equilibrium of the internal stress is perturbed by the external loads, the potentials of the chemical species change and a diffusive phenomenon, enhanced by plasticity, takes place (Biermann et al., 1996, Epishin et al., 2000, Mughrabi, 2009). The mass transport direction depends on the crystal direction solicited and cause the migration of the hardening species from the more loaded to the less loaded matrix channels. During tensile creep along $<001>$ this leads to the saturation of vertical channels and to the widening of the horizontal ones (Figure 4). Precipitates then became plate-shaped and the $\gamma$ horizontal channel appear wider and longer.

The $\gamma'$ coarsening consist of an Oswald-ripening like phenomenon (Ostwald, 1897). This consists in a homothetic growth of the $\gamma - \gamma'$ (cubic or plate-shaped) microstructure which causes an additional widening of the active $\gamma$ channels (Figure 11).

These microstructural evolutions have a detrimental impact, time dependent, on the tensile response (Gabrisch et al, 1994, Diologent, 2002): as in the oxidation case, the diffusive process has time to set when the loading rate is low enough. For this reason a gradual softening is observed during the tensile test at $10^{-5}$s$^{-1}$ on a $<001>$ oriented CMSX-4 specimen (Figure 3a, the linear part of the softening up to $\varepsilon \approx 0.15$ is at zero damage). On the contrary, a higher loading rate leads to shorter time to rupture but larger rupture strain: the tensile tests at strain rates $\dot{\varepsilon} \geq 10^{-4}$s$^{-1}$ are too quick for the $\gamma'$ evolution to occur.

The main cause of the initial plastic strain (convex) acceleration in creep of CMSX-4 at 1050°C is the microstructure degradation (Fig. 4, schemes of Fig. 10 and 11). It is not mechanical damage: indeed it has been shown that microcracks or cavities growth takes place in a second stage (le Graverend et al, 2017). Thus, continuous damage $D$ in the sense of Continuum Damage Mechanics occurs only when a sufficient amount of plastic deformation, $p_D$, is reached (observations given in Fig. 4, 10 and 11). Such a damage threshold is also observed in monotonic tension (the onset of final acceleration of the softening in Fig. 3); concerning the tests at $\dot{\varepsilon} \geq 10^{-4}$s$^{-1}$, for which almost no microstructural evolutions take place, the samples deform first with a plateau (no damage); the plastic damage, enhanced by necking, occurs only in a second stage at large enough plastic strains ($> 0.18$).

figure1: AD 730™ fin grain: uniaxial tension tests at 750°C (Thébaud et al, 2016)

figure2: AD 730™ fin grain: post-mortem observation after creep 700°C and 250 MPa (Thébaud et al, 2016)
Figure 3: Stress-strain curves of CMSX-4 at different strain rates (1050°C). Computed Orowan stress $\sigma_{\text{ORO}}$ reported. (a) crystallographic orientation <001>, (b) crystallographic orientation <111>.

Figure 4: $\gamma/\gamma'$ microstructure evolution (rafting) of CMSX-4 Ni-based single crystalline superalloys: (a) initial morphology at 1050°C, $\gamma$ channel widths $w_1 = w_2 = w_3 = w_0$, (b) after 101 h of creep at 1050°C and 140 MPa along <001>, $w_1 > w_0$ and $w_2 = w_3 = 0$.

From a mechanical point of view, previous observations will allow us to define a damage threshold $p_D$ in terms of accumulated plastic strain $p$, such as long as the accumulated plastic strain remains below $p_D$ there is no damage.
damage:

\[ p < p_D \rightarrow D = 0 \]  

Contrary to standard results (Lemaitre, 1992, Sermage et al., 2000), the damage threshold will next be made dependent, in an original manner, on the accumulated plastic strain rate \( \dot{p} \). Remark that threshold \( p_D \)– is of the order of magnitude of a few percent in creep (assumed to be the limiting case \( \dot{p} \approx 0 \)),

– and reaches almost 0.2 (i.e. 20%) in tension at \( 10^{-3} \text{s}^{-1} \).

Proposed modeling will be applied to CMSX-4 superalloy, starting with isotropic formulations that do not represent the microstructural changes. In order to account for the rafting phenomenon and for the mechanical damage, both in creep and in tension at different strain rates, we use then the tensorial representation of the \( \gamma \) channel width proposed in (Desmorat et al., 2017). Kelvin modes based formulations of anisotropic (cubic) viscoplasticity (Cowin et al., 1991, Biegler and Mehrabadi, 1995, Francois, 1995, Bertram and Olschewski, 1996, Arramon et al., 2000, Mahnken, 2002, Desmorat and Marull, 2011) will be introduced and a link between Kelvin decomposition and harmonic decomposition of cubic elasticity made (Section 5).

The decrease of the yield stress and the acceleration of plastic strain rate due to \( \gamma' \) rating phenomenon are attributed to Orowan stress, inversely proportional to the \( \gamma \) channel width (Benyoucef et al., 1993, Fedelich et al., 2009, Tinga et al., 2009, Cormier and Cailletaud, 2010a, Staroselsky et al., 2011), denoted \( \sigma_{\text{ord}} \) when directly summed to yield stress \( \sigma_y \) in a macroscopic criterion function, possibly anisotropic. Fig. 3 shows that it evolves only at low strain rate \( \dot{\varepsilon} \leq 10^{-3} \text{s}^{-1} \) (dotted lines, CMSX-4 at 1050°C). Some definitions are given in the Appendix A.

3. Generalities on continuous damage

Desmorat, 2005). For both families of models, including for first kind the representation of void nucleation (Chu and Needleman, 1980), the damage rate is proportional to the accumulated plastic strain rate $\dot{p}$ and enhanced by the stress triaxiality

$$T_X = \frac{\sigma_{eq}^{\mu}}{\sigma_{eq}^2} = \frac{1}{3} \frac{\text{tr} \sigma}{\sigma_{eq}}$$

(2)

The stress level has no effect in Rice & Tracey’s and in Gurson’s void growth laws as void growth by mass conservation $\phi = (1 - \phi) \text{tr} \dot{\varepsilon}^p$ or enhanced by void nucleation is a rate independent equation, as

$$\phi = (1 - \phi) \text{tr} \dot{\varepsilon}^p + A \dot{p} \iff d\phi = (1 - \phi) \text{tr} d\dot{\varepsilon}^p + Ad\dot{p}$$

(3)

with $\phi$ the void volume fraction, $\varepsilon^p$ the plastic strain and where $A$ is the nucleation parameter (Chu and Needleman, 1980). For metals the elastic strain is negligible so that a rate independent ductility is obtained, even for the extension to visco-plasticity (Leblond et al, 1994, Besson, 2009).

The stress level has a strong effect in Lemaitre’s damage law when written

$$D = \left(\frac{Y}{S}\right) \dot{p} \quad Y = \frac{1}{2} \varepsilon^e : \varepsilon^e = \frac{1}{2} \sigma : E^{-1} : \sigma$$

(4)

with $Y$ the strain energy release rate density, $\varepsilon^e$ the elastic strain, $\sigma^e/(1 - D)$ the effective stress. According to the principle of strain equivalence (Lemaitre, 1971, Lemaitre and Chaboche, 1985) the elasticity law coupled with damage writes $\dot{\sigma} = \dot{E} : \varepsilon^p$ (with $E$ elasticity tensor of virgin material). The damage parameters are $S$, the damage strength, and $s$, the damage exponent. Damage law (4) leads then to a rate dependentductility (Lemaitre and Desmorat, 2005), but the higher the stress level the lower the ductility. Some authors prefer then to use—even in high strain rate visco-plasticity (Borvik et al, 2002)— the damage law

$$D = AR^s \dot{p} \quad R_s = \frac{2}{3} (1 + \nu) + \frac{2}{3} (1 - 2\nu) \left(\frac{\sigma_{eq}^2}{\sigma_{eq}}\right)^2$$

(5)

with $R_s$ Lemaitre’s triaxiality function, $\nu$ Poisson’s ratio, $A$ and $s$ as damage parameters. This evolution law may be rewritten (Lemaitre and Chaboche, 1985)

$$\dot{D} = \frac{R_s^2}{\epsilon_{pR} - p_D} \dot{p}$$

(6)

in order to make appear the accumulated plastic strain to rupture $\varepsilon_{pR}$ and the damage threshold $p_D$, both identified in monotonic tension in Eq. (6).

Damage law (5) is in fact derived from a rate independent analysis with isotropic elasticity when the effective stress saturates at the ultimate stress $\sigma_u$, a material constant then, so that (Lemaitre, 1992)

$$\dot{D} = \left(\frac{\sigma_{eq}^2 R_s}{2ES}\right) \dot{p} \approx AR^s \dot{p} \quad \text{with} \quad A = \left(\frac{\sigma_u^2}{2ES}\right)^s$$

(7)

Setting $A$ as a material parameter and using law $\dot{D} = AR^s \dot{p}$—with $s = 0$ in (Borvik et al, 2002) recovering then Chu and Needleman (1980) nucleation term—for visco-plasticity coupled with damage analyses leads to a constant ductility independent from the loading rate (but dependent of the stress triaxiality for $s > 0$). This is the damage law that we propose next to use (with $s = 1$ for the sake of simplicity). A first possibility to account for the rate dependency exists, usually dedicated to dynamics and impact (Johnson and Cook, 1985): to enforce a zero damage threshold (this needs to make nonlinear the damage law) and to consider as rate dependent the damage parameter $A$ (in an equivalent manner to make rate dependent the plastic strain to rupture $\varepsilon_{pR}$ within Eq. (6)). This would make difficult the modeling of tertiary creep which may exhibit quite large strains to rupture ($> 0.1–0.2$) after a secondary creep stage at very low strain rate.

We propose in present work to gain a positive rate sensitivity of the ductility by introducing a rate dependent damage threshold $p_D$ such as

$$p < p_D \implies \dot{D} = 0 \quad p \geq p_D \implies \dot{D} > 0$$

(8)

formulated in an equivalent manner by means of a rate dependent threshold criterion $f_D$ such as

$$f_D < 0 \implies \dot{D} = 0 \quad f_D \geq 0 \implies \dot{D} > 0$$

(9)

Let first assume isotropy and focus on the damage part of the modeling, owing for the increase of ductility with the strain rate in tension.
4. Damage modeling with rate dependent threshold – Isotropic case

We give next the constitutive equations of an elasto-visco-plastic damage model which represents a strain rate dependent ductility, increasing with the loading rate. Such a first model does no account for (cubic) anisotropy nor for the microstructure degradation observed for single crystal superalloys at high temperature. We use the remark that at sufficiently large strain rates the Orowan stress $\sigma_{ORO}$ evolves slowly (Fedelich et al., 2012b, le Graverend, 2013, Giraud, 2013, Desmorat et al, 2017) so that further simplifying assumption

$$\sigma_y + R + \sigma_{ORO} \approx k = \text{const} \quad (10)$$

can be made for a first modeling of monotonic loading, with scalars $\sigma_y$ and $R$ respectively the yield stress and the isotropic hardening. The value $k = 117 \text{ MPa}$ has been measured for CMSX-4 superalloy at 1050$^\circ\text{C}$ at strain rates $\dot{\varepsilon} = 10^{-4} \text{s}^{-1}$ and $10^{-3} \text{s}^{-1}$. The elasto-visco-plastic material parameters are those of (Desmorat et al, 2017).

4.1. Constitutive equations (isotropy, no microstructural degradation)

First modeling is isotropic with no microstructural degradation.

- **Effective stress / Elasticity**
  
  $$\tilde{\sigma} = \frac{\sigma}{1 - D} \quad \tilde{\sigma} = \mathbb{E} : (\epsilon - \epsilon^p) \quad (11)$$

  with $\mathbb{E}$ isotropic.

- **Criterion function**, such as $f < 0 \rightarrow$ elasticity, $f \geq 0 \rightarrow$ visco-plasticity,

  $$f = \tilde{\sigma}_{eq} - k \quad (12)$$

- **Normality rule**

  $$\dot{\epsilon}^p = \dot{\rho} \mathbf{n} \quad \mathbf{n} = \frac{3}{2} \frac{\dot{\sigma}'}{\tilde{\sigma}_{eq}} = \frac{3}{2} \frac{\sigma'}{\tilde{\sigma}_{eq}} \quad (13)$$

- **Viscosity law**

  $$\dot{\rho} = \left( \frac{\tilde{\sigma}_{eq}}{K_N} \ln \left( 1 - \frac{f}{\tilde{\sigma}_{eq}} \right) \right)^N \quad (14)$$

- **Rate dependent damage threshold criterion $f_D$ such as $f_D < 0 \rightarrow$ no damage, $f_D \geq 0 \rightarrow$ damage growth,**

  $$f_D = \hat{\rho} - 1 \quad \hat{\rho} = \sup \left[ \frac{p}{\rho_D} \exp \left( -B \left( 1 - \frac{\rho_0}{\hat{\rho}} \right) \right) \right] \quad (15)$$

  with $\hat{\rho}$ a dimensionless equivalent anelastic strain defined as positive monotonic ($\hat{\rho} \geq 0, \frac{d\hat{\rho}}{dt} \geq 0$). The corresponding damage threshold $p_D$ is plotted in Fig. 7 as a function of the accumulated plastic strain $\dot{\rho}$ in the case $\dot{\rho} = \text{constant}$.

- **Damage evolution law,**

  $$\dot{D} = AR_{c} \dot{\rho} \mathsf{H}(f_D) \quad R_e = \frac{2}{3}(1 + \nu) + 3(1 - 2\nu) \left( \frac{\sigma_H}{\tilde{\sigma}_{eq}} \right)^2 \quad (16)$$

  i.e. $\dot{D} > 0$ as soon as $f \geq f_D$. Damage growth is classically governed by (visco-)plasticity and enhanced by the stress triaxiality (but not by the stress level).

The material parameters of such an elasto-visco-plasticity coupled with damage model are:

- the (isotropic) elasticity parameters $E$, $\nu$ (add the thermal expansion coefficient $\alpha$ in case of anisothermal loading),
- the yield stress $k$, including saturated hardening and assumed constant Orowan stress,
- Norton’s viscosity parameters $K_N$, $N$, and $\sigma_{cvo}$ as the saturation value of the viscous stress,
- the damage threshold in creep $\rho_D$,
- $\rho_0$ and $B$ for the rate dependency of the damage threshold,
- the damage parameter $A$.

Some remarks are given in E.
4.2. Computed $< 001 >$ stress-strain curves

We present in Figure 6 the model response for tension tests at 1050°C along the $[001]$ crystal direction of CMSX-4 alloy at different strain rates: as true stress - true strain curves. The experiments reported are the curves of Fig. 3a. The strain rate effect on the damage threshold by Eq. (15) is clearly noticeable, the higher the strain rate the larger the damage threshold and the ductility. The damaging stage is linear due to the linearity of the damage evolution law to $\dot{p}$ and to constant viscous stress (obtaining a nonlinear softening is possible but this introduce an additional material parameter, thus this choice has not been made here). A critical damage $D_c = 0.15$ such as $D = D_c \rightarrow$ crack initiation is considered (the calculated curves —in red— end at $D = D_c$). The initial gradual softening of experimental response at $\dot{\varepsilon} = 10^{-5}\text{s}^{-1}$ is not reproduced. This softening is not due to damage but to the rafting phenomenon: a better modeling accounting for microstructural changes will be obtained in Section 8.

![Figure 6: First modeling of $< 001 >$ stress-strain curve up to critical damage $D_c = 0.15$ at different strain rates (CMSX-4 at 1050°C, $\dot{\varepsilon} = 10^{-5}\text{s}^{-1}, 10^{-4}\text{s}^{-1}, 10^{-3}\text{s}^{-1}$).](image)

5. Cubic elasticity from harmonic decomposition

In order to extend our damage modeling to the cubic anisotropy encountered for single crystal superalloys, such as CMSX-4, we now use the elasticity framework to present consistent mathematical tools: harmonic decomposition (Schouten, 1951, Backus, 1970, Spencer, 1970), Kelvin stresses and projectors (Kelvin, 1856, 1878, Rychlewski, 1984, Cowin et al., 1991, Biegler and Mehrabadi, 1995, François, 1995, Bertram and Olschewski, 1996, Arramon et al., 2000, Mahnken, 2002, Desmorat and Marull, 2011). We describe their use and define objective stresses and plastic strains dedicated to cubic material symmetry.

5.1. Cubic elasticity parameters

The three independent components of a cubic elasticity tensor $E$ (of components $E_{ijkl}$, having major and minor indicial symmetries $E_{ijkl} = E_{klij} = E_{ikjl}$) are Young’s modulus $E$, Poisson’s ratio $\nu$ and the shear modulus $G \neq \frac{E}{2(1+\nu)}$, the bulk modulus being $K = \frac{E}{3(1-2\nu)}$. In Natural Anisotropy Basis (of Figure 10):

$$
\begin{align*}
E_{1111} &= E_{2222} = E_{3333} = \frac{(1-\nu)E}{1-\nu-2\nu^2} \\
E_{1122} &= E_{1133} = E_{2233} = \frac{\nu E}{1-\nu-2\nu^2} \\
E_{1212} &= E_{1313} = E_{2323} = G
\end{align*}
$$

The other $E_{ijkl}$ are either obtained from the indicial symmetries or are equal to zero.

5.2. Harmonic decomposition of cubic elasticity tensor

An elasticity tensor belonging to the cubic symmetry class can be recast as

$$
E = 2\mu I + \lambda I \otimes I + \mathbf{H} \quad \text{tr}_{12} \mathbf{H} = \text{tr}_{13} \mathbf{H} = 0
$$

where the fourth order part $\mathbf{H} = \mathbf{H}(E)$ is harmonic, i.e. is both

- totally symmetric: having the major and minor indicial symmetries of the elasticity tensor and the additional Cauchy symmetry $H_{ijkl} = H_{ikjl}$,
\[
- \text{traceless: } (tr_{12} \mathbf{H})_{ij} = H_{kk} = 0, \ (tr_{13} \mathbf{H})_{ij} = H_{kk} = 0.
\]

Eq. (18) is the harmonic decomposition of fourth order tensor \(\mathbf{E}\) (Backus, 1970, Cowin et al., 1991, Baerheim, 1993, Forte and Vianello, 1996). Generalized Lamé constants \(\lambda, \mu\) are two invariants of the elasticity tensor: they are functions of the invariants \(tr(tr_{12} \mathbf{E})\) and \(tr(tr_{13} \mathbf{E})\),

\[
\begin{align*}
\{ \lambda = \frac{1}{15} (2 tr(tr_{12} \mathbf{E}) - tr(tr_{13} \mathbf{E})) \\
\mu = \frac{1}{36} (-tr(tr_{12} \mathbf{E}) + 3 tr(tr_{13} \mathbf{E}))
\end{align*}
\]

Practical expressions are given in the Appendix B. Note that \(\mathbf{H} = 0\) is equivalent to \(\mu = \frac{E}{(1+\nu)} = G\), i.e. to isotropic elasticity.

5.3. Kelvin stresses – Kelvin projectors

A cubic elasticity tensor \(\mathbf{E}\) and, in an equivalent manner, its inverse \(\mathbf{E}^{-1}\) have three eigentensors: the hydrostatic stress tensor \(\sigma^{H}\), and two deviatoric stress tensors \(\sigma^{d}\) and \(\sigma^{\tilde{d}}\) (\(\sigma^{d}\) diagonal in Natural Anisotropy Basis, deviatoric, \(\sigma^{\tilde{d}}\) out-of-diagonal in this basis, also deviatoric), such as

\[
\mathbf{E} : \sigma^{H} = \frac{E}{1-2\nu} \sigma^{H}, \quad \mathbf{E} : \sigma^{d} = \frac{E}{1+\nu} \sigma^{d}, \quad \mathbf{E} : \sigma^{\tilde{d}} = 2G \sigma^{\tilde{d}}
\]

with the orthogonality properties \(\sigma^{d} : \sigma^{H} = \sigma^{\tilde{d}} : \sigma^{H} = \sigma^{d} : \sigma^{\tilde{d}} = 0\) and the deviatoric and total stresses partitions

\[
\sigma' = \sigma^{d} + \sigma^{\tilde{d}} \quad \text{and} \quad \sigma = \sigma^{d} + \sigma^{\tilde{d}} + \sigma^{H}
\]

Such partitions are objective in case of cubic material symmetry (see Bertram and Olschewski (1996)).

The second order tensors \(\sigma^{H}, \sigma^{d}\) and \(\sigma^{\tilde{d}}\) are the so-called Kelvin stresses, associated with Kelvin moduli \(\frac{E}{1-2\nu}, \frac{E}{1+\nu}\) and \(2G\) (the three eigenvalues of \(\mathbf{E}\), Kelvin (1856, 1878), Cowin et al. (1991)). They are obtained thanks to Kelvin fourth order projectors \(\mathbf{P}^{H}, \mathbf{P}^{d}, \mathbf{P}^{\tilde{d}}\) (François, 1995),

\[
\begin{align*}
\mathbf{P}^{H} : \sigma &= \sigma^{H} = \frac{1}{3} tr(\sigma) \mathbf{1} \\
\mathbf{P}^{d} : \sigma &= \sigma^{d} \\
\mathbf{P}^{\tilde{d}} : \sigma &= \sigma^{\tilde{d}}
\end{align*}
\]

with \(\mathbf{P}^{H} = \frac{1}{3} \mathbf{1} \otimes \mathbf{1}\) standard hydrostatic projector. The two other projectors can be build directly from the harmonic part \(\mathbf{H} = \mathbf{E} - 2\mu \mathbf{I} - \lambda \mathbf{1} \otimes \mathbf{1}\) of cubic elasticity tensor as

\[
\mathbf{P}^{d} = \frac{1}{5} \left( \mathbf{J} + \frac{\mathbf{H}}{\mu - G} \right), \quad \mathbf{P}^{\tilde{d}} = \mathbf{J} - \mathbf{P}^{d}, \quad \mathbf{J} = \mathbf{I} - \frac{1}{3} \mathbf{1} \otimes \mathbf{1}
\]

with \(\mu \neq G\) and \(\mathbf{H} \neq 0\) for cubic symmetry class and \(\mathbf{P}^{H} + \mathbf{P}^{d} + \mathbf{P}^{\tilde{d}} = \mathbf{1}\).

The Kelvin stresses can be used to particularize Hill equivalent stress to cubic symmetry as writing in Natural Anisotropy Basis

\[
\sigma_{mn} = \sqrt{\frac{1}{2} (\sigma_{11} - \sigma_{22})^2 + (\sigma_{22} - \sigma_{33})^2 + (\sigma_{33} - \sigma_{11})^2} + \frac{3}{2} h^2 \left( \sigma_{12}^2 + \sigma_{21}^2 + \sigma_{13}^2 + \sigma_{31}^2 + \sigma_{23}^2 + \sigma_{32}^2 \right)
\]

is equivalent to intrinsic writing

\[
\sigma_{mn} = \sqrt{\frac{3}{2} (\sigma^{d} : \sigma^{d} + h^2 \sigma^{\tilde{d}} : \sigma^{\tilde{d}})} = \sqrt{(\sigma^{d})_{eq}^2 + h^2(\sigma^{\tilde{d}})_{eq}^2}
\]

with \(h\) a material parameter (\(h = 1\) for isotropy, von Mises stress recovered). Following notations are used next for the particular von Mises norms

\[
\sigma^{d}_{eq} = (\sigma^{d})_{eq} = \sqrt{\frac{3}{2} \sigma^{d} : \sigma^{d}}, \quad \sigma^{\tilde{d}}_{eq} = (\sigma^{\tilde{d}})_{eq} = \sqrt{\frac{3}{2} \sigma^{\tilde{d}} : \sigma^{\tilde{d}}}
\]

with in Natural Anisotropy Basis

\[
\begin{align*}
\sigma^{d}_{eq} &= \sqrt{\frac{3}{2} (\sigma_{11} - \sigma_{22})^2 + (\sigma_{22} - \sigma_{33})^2 + (\sigma_{33} - \sigma_{11})^2} \\
\sigma^{\tilde{d}}_{eq} &= \sqrt{\frac{3}{2} \sigma_{12}^2 + \sigma_{21}^2 + \sigma_{13}^2 + \sigma_{31}^2 + \sigma_{23}^2 + \sigma_{32}^2}
\end{align*}
\]
The non quadratic norm (expressed in Natural Anisotropy Basis)
\[ \|\sigma^d\|_a = 3 \left( |\sigma_{12}|^a + |\sigma_{21}|^a + |\sigma_{31}|^a + |\sigma_{23}|^a + |\sigma_{32}|^a \right)^{1/6} \]  
(28)
is a generalization of equivalent stress \( \sigma_{eq}^a \) such as i) \( \sigma_{eq}^a = \|\sigma^d\|_{a=2} \) ii) \( \|\sigma^d\|_a = 0 \) for tension in orientation \(< 001 > \) and iii) \( \|\sigma^d\|_a = \sigma \) for tension in orientation \(< 111 > \).

5.4. Objective plastic strains from Kelvin projectors

The Kelvin projectors allow to define different strain tensors (total, elastic and plastic) dedicated to cubic symmetry class (François, 1995). When applied to the —here deviatoric— plastic strain tensor they define in an objective manner two deviatoric tensors \( \epsilon^{p\alpha} \) and \( \epsilon^{p\beta} \) as
\[
\epsilon^{p\alpha} = \mathbb{P}^d : \epsilon^p \quad \text{and} \quad \epsilon^{p\beta} = \mathbb{T}^d : \epsilon^p
\]
(29)such as the partition \( \epsilon^p = \epsilon^{p\alpha} + \epsilon^{p\beta} \) is objective for cubic symmetry class. In such a plastic strain partition \( \epsilon^{p\alpha} \) is the diagonal part of \( \epsilon^p \) in Natural Anisotropy Basis, \( \epsilon^{p\beta} \) is the out-of-diagonal part of \( \epsilon^p \) in this basis, with the orthogonality property \( \epsilon^{p\alpha} : \epsilon^{p\alpha} = 0 \).

In a consistent manner (see Desmorat and Marull (2011)), we define two accumulated plastic strains, one \( \rho^\alpha \) associated to the ”deviatoric diagonal” Kelvin mode, another to the ”deviatoric out of diagonal” Kelvin mode \( \rho^\beta \)
\[
\rho^\alpha = \int \frac{2}{3} \epsilon^{p\alpha} : \epsilon^p \, dt \quad \text{and} \quad \rho^\beta = \int \frac{2}{3} \epsilon^{p\beta} : \epsilon^p \, dt
\]
(30)
Usual (isotropic) accumulated plastic strain \( \rho = \int \sqrt{2} \epsilon^p : \epsilon^p \, dt \) is such as
\[
\dot{\rho} = \sqrt{(\rho^\alpha)^2 + (\rho^\beta)^2}
\]
(31)
5.5. Stresses and plastic strains for tensions along \(< 001 > \) and along \(< 111 > \)

The Kelvin stresses and strains decouple the constitutive equations for two particular loading often used for the identification of crystal plasticity of cubic superalloys (uniaxial loading in orientations \(< 001 > \) and \(< 111 > \)):
\[
- \sigma^d = \sigma^r, \sigma^\alpha = 0, \epsilon^{p\alpha} = \epsilon^p \quad \text{and} \quad \epsilon^{p\beta} = 0 \quad \text{for tension along} \ (< 001 > , \text{for which} \ (\sigma^d)_{eq} = \sigma_{eq} = \sigma, \rho^\alpha = \rho, \rho^\beta = 0.
\]
\[
- \sigma^d = 0, \sigma^\alpha = \sigma^r, \epsilon^{p\alpha} = \epsilon^p \quad \text{and} \quad \epsilon^{p\beta} = 0 \quad \text{for tension along} \ (< 111 > , \text{for which} \ (\sigma^d)_{eq} = \sigma_{eq} = \sigma, \rho^\alpha = 0, \rho^\beta = \rho.
\]
This decoupling allows to represent independently —i.e. with two different subsets of material parameters— by a single multiaxial model the uniaxial responses of cubic superalloys both in crystallographic orientation \(< 001 > \) and in orientation \(< 111 > \) (Desmorat and Marull, 2011). The material parameters for orientation \(< 111 > \) will be next overlined. For example if \( \sigma^r, K_N, \eta, \epsilon^{p\alpha}, B, \rho_0, A \) are material parameters for orientation \(< 001 > \), the parameters (having the same role) for orientation \(< 111 > \) will be denoted \( \overline{\sigma}^r, \overline{K}_N, \overline{\eta}, \overline{\epsilon}^{p\alpha}, \overline{B}, \overline{\rho}_0, \overline{A}. \)

6. Damage threshold and evolution law for cubic symmetry class

Let us use the decoupling of the mechanical responses obtained for orientations \(< 001 > \) and \(< 111 > \) when accumulated plastic strains \( \rho^\alpha \) and \( \rho^\beta \) are introduced within visco-plasticity coupled with damage constitutive equations.

6.1. Rate dependent damage threshold for cubic material symmetry

We define for cubic materials the dimensionless equivalent anelastic strain \( \hat{\rho} \) as
\[
\hat{\rho} = \sup_i \left[ \frac{\rho^{p\alpha}}{\epsilon^{p\alpha}} \exp\left(-B(1 - \frac{\rho_0}{\rho^{p\alpha}})\right) + \frac{\rho^{p\beta}}{\epsilon^{p\beta}} \exp\left(-\overline{B}(1 - \frac{\overline{\rho}_0}{\rho^{p\beta}})\right) \right]
\]
(32)
The damage threshold criterion is still expressed as
\[
f_D = \hat{\rho} - 1
\]
(33)and is such as \( f_D < 0 \) → no damage, \( f_D \geq 0 \) → damage growth.

We have then the main two practical particular cases:
– for monotonic tension (including creep) in orientation \(<001>\): 
\[ p^d = p = \epsilon_p, \ p^\beta = 0, \] 
so that in such an uniaxial case writing \( f_D \geq 0 \rightarrow D > 0 \) gives

\[ p \geq p_D^{001>}(\dot{\rho}) = \epsilon_p^0 \exp \left( B \left( 1 - \frac{\dot{\rho}_0}{\dot{\rho}} \right) \right) \rightarrow D > 0 \]  
(34)

– for monotonic tension (including creep) in orientation \(<111>\): 
\[ p^d = 0, \ p^\beta = p = \epsilon_p \] 
so that in such a case to write \( f_D \geq 0 \rightarrow D > 0 \) gives

\[ p \geq p_D^{111>}(\dot{\rho}) = \epsilon_p^0 \exp \left( B \left( 1 - \frac{\dot{\rho}_0}{\dot{\rho}} \right) \right) \rightarrow D > 0 \]  
(35)

The material parameters \( \epsilon_p^0, B, \dot{\rho}_0 \), are then those of the damage threshold rate dependency measured in direction \(<001>\) (monotonic loading, Figure 7). The material parameters \( \epsilon_p^0, \ B, \dot{\rho}_0 \), are those of the damage threshold rate dependency measured in direction \(<111>\) (Figure 8).

Figure 7: Damage threshold \( p_D \) as a function of the accumulated plastic strain rate \( \dot{\rho} \) for \(<001>\) CMSX-4 (bullets: experiments, solid line: Eq. (15)).

Figure 8: Damage threshold \( p_D \) as a function of the accumulated plastic strain rate \( \dot{\rho} \) for \(<111>\) CMSX-4 (bullets: experiments, solid line: Eq. (15)).

In order to determine the damage growth condition for the other crystallographic orientations or for multiaxial loading simply apply criterion (32)-(33) with \( p^d \) and \( p^\beta \) defined by Eq. (29)-(30).

6.2. Damage evolution law for cubic material symmetry

We now extend the damage law \( \dot{D} = AR\dot{\rho} \) to cubic material symmetry as:

\[ \dot{D} = R\left( A\dot{p}^d + A\dot{p}^\beta + A_{\text{dis}} \sqrt{\dot{p}^d \dot{p}^\beta} \right) H(f_D) \]  
(36)
with previous threshold function $f_D$ (Eq. (32)-(33)) such as $f_D < 0 \rightarrow$ no damage, $f_D \geq 0 \rightarrow$ damage increase. When introduced, the additional damage parameter $A_{dis}$ induces a faster damage increase for loadings outside of the [001] and [111] directions. The extra term $A_{dis}(p^d p^{\beta^2})^{1/2}$ vanishes $\forall A_{dis}$ in uniaxial loading performed either in crystallographic orientation $<001>$ or in orientation $<111>$. As for original Rice & Tracey’s, Gurson’s and Lemaitre’s laws, the damage evolution law (36) does not introduce a strain rate dependency by itself: $p^d$, $p^\beta$ and $(p^d p^{\beta^2})^{1/2}$ are all homogeneous to $s^{-1}$, i.e. to $\dot{\rho}$ (Eq. (37)).

$$dD = R_h(\dot{A} d\rho^d + \bar{A} d\rho^\beta + A_{dis} \sqrt{d\rho^d d\rho^\beta}) H(f_D)$$ (37)

Function $R_h$ is Lemaitre’s triaxiality function extended to cubic symmetry (Winter et al, 2009):

$$R_h = \chi + (1 - \chi) \frac{\sigma_{H}}{\sigma_{na}}$$

$$\sigma_{na}^2 = (\sigma_{eq}^d)^2 + h^2 (\sigma_{eq}^\beta)^2$$ (38)

$h$ and $\chi \in [0, 1]$ being material parameters. For the sake of simplicity, and due to the difficulty to measure and model the stress triaxiality effect of anisotropic materials (Benzerga and Besson, 2001, Benzerga, 2002, Winter et al, 2009), we choose next to work with initial (isotropic) Lemaitre’s triaxiality function (5), i.e. to set in previous equation $\chi = \frac{3}{4}(1 + n)$, $h = 1$, $\sigma_{na} = \sigma_{eq}$, therefore $R_h = R_c$.

We have then the main two practical particular cases:

- **for monotonic tension (including creep) in orientation $<001>$**: $R_c = 1$, $p^d = p = \epsilon_p$, $p^\beta = 0$ and
  $$\dot{D} = A \dot{\rho} H(p - p_{<001>}^{<001>}(\rho))$$ (39)

- **for monotonic tension (including creep) in orientation $<111>$**: $R_c = 1$, $p^d = 0$, $p^\beta = p = \epsilon_p$ and
  $$\dot{D} = \bar{A} \dot{\rho} H(p - p_{<111>}^{<111>}(\rho))$$ (40)

which exhibit anisotropy of the damage growth when $\bar{A} \neq A$. The damage threshold functions $p_{<001>}^{<001>}(\rho)$ and $p_{<111>}^{<111>}(\rho)$ are given by Eq. (34)-(35)

The curves damage $D$ versus accumulated plastic strain $\rho$ obtained for monotonic tension of CMSX-4 along the $<111>$ direction are plotted in Fig. 9 (with then $p = \rho^{\beta^2}$). A critical damage $D_c = 0.15$ is assumed in order to recover the observed (experimental) time to rupture ($t_r$) when performing gauss point simulation (anticipating Figure 15). For the structural computations, we will allow for the strain localization phenomenon to take place by setting a large value for critical damage, $D_c = 0.999$. The damage parameters are: $\epsilon_{pD}^0 = 0.05$, $\varepsilon_{pD}^2 = 0.025$, $\dot{\rho}_0 = 2 \times 10^{-6} s^{-1}$, $\dot{\rho}_0 = 2.5 \times 10^{-6} s^{-1}$, $B = 1.35$, $\bar{B} = 2.21$, $A = 2.8$, $\bar{A} = 2.5$, $A_{dis} = 0$. The slopes of the curves are independent from the loading rate (but they differ, equal to $A$ for $<001>$ and to $\bar{A}$ for $<111>$). The experiments are plotted thanks to the plateaux $\sigma_{strain}$ of the curves of Figure 3 for $<111>$ orientation using formula $D_{exp} = 1 - \sigma_{strain}/\sigma$ (Lemaitre, 1992). The increase of ductility with $\dot{\epsilon}$ is attributed to the rate dependent damage threshold. The corresponding stress-strain curves are those of Fig. 12.

Figure 9: $D(\rho)$ curves in monotonic tension at different strain rates up to critical damage $D_c = 0.15$ (CMSX-4 at 1050°C, $<111>$ crystallographic orientation).
7. Full damage model with microstructure degradation and rate dependent threshold

In order to obtain a close link with Schmid based single crystal plasticity (Schmid and Boas, 1935, Hill and Rice, 1972, Rice, 1975, Asaro, 1983, Peirce et al., 1983, Asaro and Lubarda, 2006), multi-criterion by nature, a two criterion framework inspired from (Cowin et al., 1991, Bertram and Olschewski, 1996, Mahnken, 2002) has been introduced by Desmorat et al (2017) and applied to CMSX-4 single crystalline superalloy. It allows for the modeling of visco-plasticity coupled with microstructure degradation (without "mechanical" damage $D$). The material parameters of this initial model are kept, they are those for CMSX-4 at 1050°C given in (Desmorat et al, 2017).

We now couple this initial model to damage by means of the principle of strain equivalence (Lemaitre, 1971): the stress in the elasticity law as well as in criterion functions is replaced by the effective stress $\tilde{\sigma} = \sigma/(1 - D)$ and damage evolution law (36) is considered with rate dependent threshold (32)-(33). The isotropic hardening is assumed saturated ($R = R_\infty$).

7.1. Tensorial $\gamma$ channel width variables $w$ and $\omega$

The effect of the microstructure change/degradation has not to be mistaken with mechanical damage $D$, representative of the microcracks and voids present within RVE of Continuum Mechanics. Rafting phenomenon is represented by a tensorial variable: the $\gamma$ channel width second order (symmetric) tensor $w$, of initial value $w(t=0) = w_0 \mathbf{1}$ (i.e. $w_{ij}(t) = w_0 \delta_{ij}$) in terms of components, eigenvalues denoted $w_i$ in Fig. 5). We have $w_0 \approx 80$ nm for CMSX-4 at 1050°C. Such an initial value $w_0 = w_0(f_{\gamma'}(T))$ is temperature dependent through the change of $\gamma'$ volume fraction with $f_{\gamma'} \approx 0.7$ at room temperature, $f_{\gamma'} \approx 0.55$ at 1050°C (Roebuck et al, 2001, Fedelich et al., 2012b, Desmorat et al, 2017). The mechanical effect of the microstructure degradation is quantified by macroscopic Orowan stress, inversely proportional to the norm $\|w\| = \sqrt{w}$ of $\gamma$ channel width tensor. This is therefore inversely proportional to the norm of dimensionless tensor $\omega = w/w_0$, taken as a state variable of thermodynamics

$$\sigma_{\text{ORO}} = \frac{\kappa_{\text{ORO}} G}{\|\omega\|} \quad \omega = \frac{w}{w_0}$$

with $\kappa_{\text{ORO}}$ a material parameter and $G$ the shear modulus. The model is completed by an evolution law $\dot{\omega} = \ldots$ for variable $\omega$. The details concerning the derivation of the full set of constitutive equations, with no damage, can be found in (Desmorat et al, 2017).

Figure 10: Schematic microstructural evolution in tension creep along the $<001>$ direction in Natural Anisotropy Basis: 1) initial microstructure ($w_1 = w_2 = w_3 = w_0$), 2) intermediate microstructure ($w_1 > w_2 = w_3$), 3) rafted and coarsened microstructure ($w_1 > w_0$, $w_2 = w_3 = 0$).

Figure 11: Schematic microstructural evolution in pure coarsening mode (i.e. pure thermal expansion): 1) initial microstructure ($w_1 = w_2 = w_3 = w_0$), 2) final microstructure ($w_1 = w_2 = w_3 > w_0$).
7.2. Proposed model (Part I Mechanics)

The first part of constitutive equations for cubic elasto-visco-plasticity coupled with tensorial rafting and damage are:

-弹性律与损伤耦合
  \[ \tilde{\varepsilon} = \frac{\sigma}{1 - D} \quad \tilde{\varepsilon} = E : (\varepsilon - \varepsilon^p) \]
  \[ (42) \]
  其中Hooke’s tensor E of cubic symmetry class. It defines the Kelvin projectors \( P^d \) and \( P^\pi \) by Eq. (23).

- 有效应力分解在立方对称中
  \[ \tilde{\sigma} = \tilde{\sigma}^d + \tilde{\sigma}^\pi + \frac{1}{3} \text{tr} \tilde{\sigma} \mathbf{1} \]
  \[ (43) \]
  引入等效有效维氏应力 \( \tilde{\sigma}_{eq} = \sigma_{eq} / (1 - D) \) 和 \( \tilde{\sigma}_{eq}^\pi = \sigma_{eq}^\pi / (1 - D) \), 与 \( \tilde{\sigma}^d = P^d : \tilde{\sigma} \), \( \tilde{\sigma}^\pi = P^\pi : \tilde{\sigma} \),

- 条件函数与饱和硬化和Orowan应力效应：Hill准则对于 \( f_1 \)，为了简化起见取一个平方模对于 \( f_2 \)（如果需要通过替换 \( \tilde{\sigma}_{eq}^\pi \) 为 \( ||\tilde{\sigma}||_\infty \)，Desmorat et al (2017)),
  \[ f_1 = \tilde{\sigma}_{\text{res}} - R_{\infty} - \frac{\kappa_{\text{amo}} G}{||\omega||} - \sigma_y \]
  \[ f_2 = \tilde{\sigma}_{eq} - \tilde{\sigma}_{\text{res}} - \frac{\kappa_{\text{amo}} G}{||\omega||} - \sigma_y \]
  \[ (44) \]
  其中 \( \tilde{\sigma}_{\text{res}} = \sqrt{\frac{3}{2} (\tilde{\sigma}^d : \tilde{\sigma}^d + h^2 \tilde{\sigma}^\pi : \tilde{\sigma}^\pi)} \]
  \[ (45) \]

- 塑性应变率和正常 \( n^d \)
  \[ \dot{\varepsilon}^p = \dot{p}_1^{nu} \frac{\partial f_1}{\partial \sigma} + \rho_2^\pi \frac{\partial f_2}{\partial \sigma} \quad n^d = \left( \frac{\partial f_1}{\partial \sigma} \right)^d \]
  \[ (46) \]

- 累积塑性应变率
  \[ \rho_1^{nu} = \sqrt{\frac{2}{3} \left( \dot{\varepsilon}_1^p : \dot{\varepsilon}_1^p + \frac{1}{h^2} \dot{\varepsilon}_1^\pi : \dot{\varepsilon}_1^\pi \right)} \quad \rho_2^\pi = \sqrt{\frac{2}{3} \dot{\varepsilon}_2^\pi : \dot{\varepsilon}_2^\pi} \]
  \[ (47) \]

- 黏度定律，用不同的方式表达 \( f_1 \) 和 \( f_2 \) 来代表高应变速率

\[ \rho_1^{nu} = \left( - \frac{\sigma_{\text{vco}}}{K_N} \ln \left( 1 - \frac{f_1}{\sigma_{\text{vco}}} \right) \right)^N \quad \rho_2^\pi = \left( \frac{f_2}{K_N} \right)^N \left( \frac{1}{\max \left( \tilde{\sigma}_{eq}^\pi - \tilde{\sigma}_{\text{th}}, 1 \right)} \right) \]
  \[ (48) \]

The material parameters are: \( E \), \( v \), \( G \) for elasticity, yield stresses \( \sigma_y \) and \( \tilde{\sigma}_y \), Hill’s parameter \( h \), saturated hardenings \( R_{\infty} \) and \( \tilde{R}_{\infty} \), parameters \( \kappa_{\text{amo}} \) and \( \sigma_y \) for Orowan stress effect, Norton’s viscosity parameters \( K_N \), \( N \), \( \tilde{K}_N \), \( \tilde{N} \) and high strain rate viscosity parameters \( \sigma_{\text{vco}} \) and \( \tilde{\kappa}_{\text{th}} \).

7.3. Proposed model (Part II Microstructure degradation)

The second part of constitutive equations concerns the microstructure degradation occurring at high temperature and the evolution law for the γ channel width variables \( \omega \) and \( w \).

- 进化律对于无量纲 γ 粒子宽度变量 \( \omega \)
  \[ \dot{\omega} = \dot{\omega}^{\text{raft}} + \dot{\omega}_{\text{mc}} 1 + \sqrt{3} \dot{\omega}_{\text{LSW}} \frac{\omega}{||\omega||} \]
  \[ (49) \]
  其中}$raft$ (通过张量 \( \omega^{\text{raft}} \))，机械粗化效应（通过等效张量 \( \omega_{\text{mc}} \)) 和等温增长效应（通过控制等温粗化 \( \gamma \) 粒子的应变）在热应力下存在（Lifshitz and Slyozov, 1961, Wagner, 1961）

\[ \begin{aligned}
\dot{\omega}^{\text{raft}} &= K_{\text{raft}} \exp \left( t_{\text{raft}} \min (\tilde{\omega}_{eq}^d, \omega_p^p) \right) H \left( \rho_1^{nu} - \dot{\varepsilon}_1^p \right) \mathbf{n}^d \\
\dot{\omega}_{\text{mc}} &= K_{\text{mc}} H (\rho_2^\pi - \hat{\dot{\varepsilon}}_{\text{th}}) \\
\dot{\omega}_{\text{LSW}} &= \frac{K_{\text{LSW}}}{3 \omega_{\text{LSW}}^d} 
\end{aligned} \]
  \[ (50) \]

The initial values are: \( \omega_{\text{LSW}} (t = 0) = 1 \), \( \omega^{\text{raft}} (t = 0) = 0 \), \( \omega_{\text{mc}} (t = 0) = 0 \) and \( \omega (t = 0) = 1 \). See Appendix F for the stress triaxiality effect.
• \(\gamma\) channel width tensor

\[
\mathbf{w} = w_0 \omega \tag{51}
\]

gained from initial temperature dependent value \(w_0 (w_0 = 80\text{nm for CMSX-4 at 1050°C})\),

• minimum eigenvalues \(\omega_i\) (resp. \(\omega_j\)) of second order tensor \(\mathbf{\omega}\) (resp. \(\mathbf{w}\)) bounded to zero.

Note that a particular Heaviside function \(\mathcal{H}\) (of values 0 or 1) is introduced in evolution law (50), with most often \(\mathcal{H} = 1\). As described in (Desmorat et al, 2017) the value \(\mathcal{H} = 0\) is set only when needed to enforce an always positive intrinsic dissipation. The material parameters for microstructure evolution are \(w_0, K_{\text{alp}}, u_{\text{eff}}, \varepsilon_{\text{line}}^{\beta}, \kappa_{\text{th}}^{\beta}, K_{\text{mc}}, \kappa_{\text{th}}^{\nu}\) and \(K_{\text{LSW}}\). The plasticity normal \(n^d\) is a deviatoric second order tensor introduced by Tinga et al (2009), it is defined Eq. (46). Second order tensor \(\omega^{\mu
u} = (\omega^{\mu
u})^{\gamma}\) is deviatoric.

7.4. Proposed model (Part III Damage)

The constitutive equations for final (damage) part of the model are those derived in Section 6.2 with \(R_\varepsilon = R_\varepsilon\). They are recalled here.

• Rate dependent threshold criterion,

\[
f_D = \hat{P} - 1
\]

such as \(f_D < 0\) \(\rightarrow\) no damage, \(f_D \geq 0\) \(\rightarrow\) damage growth, with

\[
\hat{P} = \sup_i \left[ \frac{p^d_i}{\rho^d} \exp\left(-B\left(1 - \frac{\rho^d_i}{\rho^d}\right)\right) + \frac{p^p_i}{\rho^p} \exp\left(-B\left(1 - \frac{\rho^p_i}{\rho^p}\right)\right) \right] \tag{53}
\]

• Damage evolution law

\[
\dot{D} = R_\varepsilon \left( A \dot{\sigma}^d + \bar{A} \dot{\sigma}^p \right) \mathcal{H}(f_D) \tag{54}
\]

with Lemaitre’s triaxiality function

\[
R_\varepsilon = \frac{2}{3} (1 + \nu) + 3(1 - 2\nu) \left(\frac{\sigma_{\text{eq}}}{\sigma_{\text{eq}}^0}\right)^2 \tag{55}
\]

Failure by crack initiation occurs when the damage \(D\) reaches the critical value \(D_c\) (measured almost equal to \(D_c = 0.15\) from all \(< 001\) and \(< 111\) creep and tension tests). Material parameters \(\varepsilon_{\text{line}}^{pD}\) and \(\varepsilon_{\text{line}}^{dD}\) are the damage thresholds in creep, respectively measured in \(< 001\) and in \(< 111\) creep, \(\rho_{001}, \rho_{111}, B, \bar{B}\) are the rate dependency parameters, \(A, \bar{A}\) are the damage parameters.

8. Creep and monotonic failure of cubic CMSX-4 superalloy at 1050°C

Let us finally present the responses of the proposed set of constitutive equations (parts I, II and III) coupling microstructural changes, visco-plasticity and mechanical damage. Rupture still corresponds to \(D = D_c = 0.15\), the damage value at which are stopped the computations, whatever the loading type or direction.

8.1. Computed stress-strain curves

The true stress-true strain tension curves are plotted in Fig. 12. The increase of ductility with the strain rate is well obtained, due to proposed rate dependent damage threshold (the plateaux or the gradual (linear) softening are at zero damage). It is here recalled that there is no isotropic (nor kinematic) hardening in the modeling (parameters \(R_{\infty} = \bar{R}_{\infty} = 0\) so that one can expect a better fit of the pre-plateau curves for \(< 111\) orientation if introduced. Also, the modeling of the initial (quick) softening at strains lower than 0.03 is less to further work (note that this usually requires a multi scale modeling (Tinga et al., 2009, Kindrachuk and Fedelich, 2012).

8.2. Computed microstructure degradation in creep

The microstructure evolution of the different specimens tested in creep is plotted in Fig. 13 and compared to experiments with a good agreement. The \(w_i\) are the 3 principal components of the \(\gamma\) channel width tensor, here in Natural Anisotropy Basis, either measured (bullets) or computed (lines).

• For \(< 001\) orientation: longitudinal mean channel width \(w_1\) measured in the loading direction increases when the transverse widths \(w_2 = w_3 \rightarrow 0\). This is a macroscopic representation of the phenomenon of rafting. The effect of the stress level is well obtained for creep in \(< 001\) orientation, enhanced by the damage at plastic strain larger than 0.05.

• For \(< 111\) orientation: the 3 components remain equal \(w_1 = w_2 = w_3\) and increase with time. This is the representation of the phenomenon of mechanical coarsening, here stress independent.

Note that under loading the LSW homothetic growth term \(\omega_{\text{LSW}}\) (Eq. (49)) has a negligible contribution. The effect of damage on the microstructural changes is quite small.
8.3. Computed creep curves

The creep responses are given in Fig. (14), also with a very good agreement. We have made the choice of a critical damage independent from the loading direction, therefore identical for $<001>$ creep and for $<111>$ creep. This is why the creep curves for $<111>$ crystallographic orientation look shorter than the experimental ones. The time to failure is nevertheless very accurate for both orientations.
9. Conclusion

A phenomenological damage model has been proposed to describe the (anisotropic) strain rate sensitivity of damage onset and evolution in single crystal superalloys. A novel strain rate sensitive damage threshold has been introduced, which allows to describe the damage onset dependence on the strain rate during creep and monotonic tension. Starting from (Desmorat and Marull, 2011), a novel formulation of the Kelvin projectors is proposed for cubic material symmetry as well. This latter, based on the harmonic decomposition of the elasticity tensor, allows to determine the Kelvin projectors for cubic material symmetry without solving the eigenvalues problem.

The curves obtained for the CMSX-4 at 1050°C along the <001> and <111> crystallographic directions show that, thanks to this model, tertiary creep, time to rupture and ductility can be quite accurately described for single crystal superalloys. However, the proposed damage can be extended to other materials, deforming and degrading by mechanisms different from the ones observed in single crystal superalloys but still exhibiting a rate dependent ductility.

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A. Definitions

Heaviside function $H(x)$ is such as $H(x) = 0$ for $x < 0$ and $H(x) = 1$ for $x \geq 0$. The positive part of scalar $x$ is then

$$\langle x \rangle = x H(x) = \max(0, x)$$

(56)
The hydrostatic and deviatoric parts $A_H$ and $A'$ of a second order tensor $A$ are

$$A_H = \frac{1}{3} \text{tr} A \quad A' = A - A_H I$$

The von Mises stress $\sigma_{eq}$ and the accumulated plastic strain $\varepsilon_p$ are

$$\sigma_{eq} = \sqrt{\frac{2}{3}(\sigma' : \sigma')} \quad \varepsilon_p = \int \sqrt{\frac{2}{3}(\varepsilon' : \varepsilon')} \, dt$$

with $\sigma'$ and $\varepsilon'$ respectively the stress deviator and the (traceless) plastic strain tensor.

**B. Harmonic decomposition in Natural Anisotropy (cubic) Basis**

In Natural Anisotropy Basis (NAB) the compliance tensor $S = E^{-1}$ of a material having cubic material symmetry has for matrix representation

$$[E^{-1}] = \begin{bmatrix}
\frac{1}{E_{1111}} & \frac{1}{E_{1122}} & \frac{1}{E_{2222}} & 0 & 0 & 0 \\
\frac{1}{E_{1122}} & \frac{1}{E_{1111}} & \frac{1}{E_{2222}} & 0 & 0 & 0 \\
\frac{1}{E_{2222}} & \frac{1}{E_{1122}} & \frac{1}{E_{1111}} & 0 & 0 & 0 \\
0 & 0 & 0 & \frac{1}{E_{1212}} & 0 & 0 \\
0 & 0 & 0 & 0 & \frac{1}{E_{1212}} & 0 \\
0 & 0 & 0 & 0 & 0 & \frac{1}{E_{1212}}
\end{bmatrix}_{NAB}$$

where $1/2G = 2S_{1212} = 1/2E_{1122}$.

The generalized Lamé constants of harmonic decomposition are

$$\begin{cases}
\lambda = \frac{1}{2} (E_{1111} - 2E_{1212} + 4E_{1122}) \\
\mu = \frac{1}{2} (E_{1111} + 3E_{1212} - E_{1122})
\end{cases}$$

The harmonic fourth order part $H$ carries both the third elasticity parameter and the Natural Anisotropy Basis (NAB) of cubic symmetry. It has for matrix representation in Natural Anisotropy Basis still (Auffray et al., 2014)

$$[H] = \delta \begin{bmatrix}
8 & -4 & -4 & 0 & 0 & 0 \\
-4 & 8 & -4 & 0 & 0 & 0 \\
-4 & -4 & 8 & 0 & 0 & 0 \\
0 & 0 & 0 & -8 & 0 & 0 \\
0 & 0 & 0 & 0 & -8 & 0 \\
0 & 0 & 0 & 0 & 0 & -8
\end{bmatrix}_{NAB}$$

with

$$\delta = \frac{1}{20} (E_{1111} - 2E_{1212} - E_{1122})$$

Components $E_{ijkl}$ are given in Eq 5.1.

**C. Rafting, mechanical coarsening, homothetic growth**

When a creep tensile test is performed vertically on $<001>$ oriented specimen (along direction 1 in Fig. 4), the hardening alloy elements migrate from the horizontal matrix channel to the vertical matrix channel and the constitutive matrix elements migrate from the vertical to the horizontal ones. At some point all the hardening alloy elements are in the vertical channels and all the ductile elements are in the horizontal ones. The $\gamma'$ precipitates became plate-shaped (Figure 4, plates oriented normally to the $<001>$ oriented tensile load): the horizontal matrix channels of an initial cube grow while the others two channels disappear (see also the schematic microstructural evolution of Fig. 11). This morphological evolution of the $\gamma'$ phase is called N-shaped $\gamma'$-coalescence (Tien and Copley, 1971, Tien and Gamble, 1972, Fredholm and Strudel, 1984, MacKay and Ebert, 1985, Caron et al., 1988, Henderson et al., 1998, Pollock and Argon, 1994). As for the rafted microstructure the mean free path of dislocations increases, the rafting process results in a loss of the hardening properties of the material.

Since the direction of such a diffusive phenomenon depends on the internal stress state, the $\gamma'$ coalescence depends on the crystallographic orientation and it tends to respects the crystal symmetry. A different morphology of the degraded microstructure is observed if the loading is performed along $<111>$ crystallographic orientation, called "mechanical coarsening" of the $\gamma'$ phase (Fig. 16) as the $\gamma$ channel widths equally increase in the three space directions. At constant temperature still but with no applied load another type of microstructural change may occur: standard (load free) Oswald ripening (Ostwald, 1897). This diffusive phenomenon is denoted "homothetic growth" (as it takes place at zero stress the shape of $\gamma'$ matrix does not evolve, it remains cubic) and be assumed to follow the LSW theory (Lifshitz and Slyozov, 1961) by contribution $\omega_{LSW}$ within Eq. (50) with $K_{LSW} = 1.10^{-6} \text{s}^{-1}$ at 1050°C. At this temperature it has been observed that the microstructural changes under load develop so quickly so that a continuous acceleration of the macroscopic plastic strain is observed (modeled in Section 8).
Figure 16: $\gamma/\gamma'$ microstructure evolution of CMSX-4 Ni-based single crystalline superalloys: (a) initial morphology at 1050°C, $\gamma$ channel widths $w_1 = w_2 = w_3 = w_0$, (b) morphology after 67 h of creep at 1050°C and 200 MPa along $<111>$, $w_1 = w_2 = w_3 > w_0$.

D. Non quadratic norm in criterion function $f_2$

If non quadratic norm is used within second criterion function of (44), setting

$$f_2 = \|\tilde{\sigma}^d\|_a - R_{\infty} - \kappa_{\text{c}} G \|\omega\| - \tilde{\sigma}_y$$

(63)

we still have have $\dot{\epsilon}^p = \rho_{1nn} \frac{\partial f_1}{\partial \sigma} + \rho_{2} \frac{\partial f_2}{\partial \sigma}$ but with

$$\rho_{2} = 2 \left( \frac{1}{6} \sum_{i \neq j} |\tilde{\sigma}_{ij}|^{\frac{2}{3 \alpha}} \right)$$

(64)

and

$$\tilde{\sigma} : \dot{\epsilon}^p = \tilde{\sigma}_{\text{eq}} \rho_{2} + \|\tilde{\sigma}^d\|_a \rho_{2}^{\tilde{\sigma}}$$

(65)

Recall that $\|\tilde{\sigma}^d\|_a = 3 \left( \frac{1}{6} \sum_{i \neq j} |\tilde{\sigma}_{ij}|^{\frac{1}{3 \alpha}} \right)^{\frac{3}{2 \alpha}}$.

A value $\alpha \in [1, 2]$ allows to model a non homogeneous plastic strain field in torsion (Fig. 17), as observed experimentally by Nouailhas and Cailletaud (1995). The prescribed rotation is such that the damage $D$ remains equal to zero at all points. Remark that the uniaxial model response for applied stresses in orientation $<001>$ and in orientation $<111>$ remains for any value of parameter $\alpha$ the same as the one of simpler (quadratic) case $\alpha = 2$, $\|\tilde{\sigma}^d\|_2 = \tilde{\sigma}_{\text{eq}}$.
E. Remarks on damage modeling proposed in Section 4

• The viscosity law (14) recovers Norton’s law \( \sigma_v \approx K_N p^{1/N} \) at low strain rates, with \( f = \sigma_v \) equal to the viscous stress when yielding. Remark also that law (14) is inverted into

\[
\sigma_v = \sigma_{v0} \left( 1 - \exp \left( - \frac{K_N}{\sigma_{v0}} p^{1/N} \right) \right)
\]  

(66)

a form which exhibits the saturation of the viscous stress \( \sigma_v \) (to \( \sigma_{v0} \)) at high strain rate.

• In creep (at low strain rate) one has \( 1 - \frac{\dot{\rho}}{\rho} < 0 \) so that \( \dot{P} \approx \rho \dot{\epsilon}_{pD}^0 \) and one recovers standard constant threshold \( p < p_D = \epsilon_{pD}^0 \rightarrow \) no damage (Lemaitre, 1992).

• The material parameter \( B \) is related to both the damage threshold in creep (at low strain rate) \( \epsilon_{pD}^0 \) and the damage threshold in tension at infinitively high strain rate \( \epsilon_{pD}^\infty \) as

\[
B = \ln \left( \frac{\epsilon_{pD}^\infty}{\epsilon_{pD}^0} \right)
\]  

(67)

At high strain rate \( 1 - \frac{\dot{\rho}}{\rho} \approx 1 \), one has then \( \dot{P} \approx p e^{-B} / \epsilon_{pD}^0 \) so that the damage threshold becomes

\[
p < p_D = \epsilon_{pD}^0 e^B = \epsilon_{pD}^\infty \rightarrow \) no damage.
\]  

(68)

• Function \( R_v \) is Lemaitre’s triaxiality function, \( T_X \) being the stress triaxiality, with the property \( R_v = 1 \) in uniaxial loading. Depending on the applications the triaxiality function \( R_v \) can be changed into \( R_v = \frac{2}{3}(1 + \nu) + 3(1 - 2\nu)(T_X)^2 \) or into Rice & Tracey’s triaxiality function \( \exp(\frac{1}{2}T_X - \frac{1}{2}) \).

Figure 17: Accumulated plastic strain field map in torsion. Geometry = tube of length 12 mm, of diameter 4 mm, of thickness 1 mm. Left and right nodes rigidly linked, prescribed rotation \( \theta = 2 \) rad around [010]-axis.
• Note last that Lemaitre’s original stress—and stress triaxiality—dependency can be included into the proposed damage evolution law, setting

\[ D = \left( \frac{Y}{
u} \right) \dot{\rho} \mathbf{H}(f_D) = A \tilde{\sigma}_{eq}^2 \dot{\rho} \mathbf{H}(f_D) \quad A = (2ES)^{-s} \]  

(69)

but such a generalization will not be needed.

F. Triaxiality effect on microstructure evolution

The stress triaxiality has an effect on the microstructure evolution, compressive loads inducing slower evolutions than positive ones. A way to model this is to replace the (effective) von Mises equivalent stress \( \tilde{\sigma}_{eq}^2 \) in evolution law (50) by a Drucker-Prager stress, i.e. by setting

\[
\begin{align*}
\dot{\omega}^{aff} &= K_{aff} \exp \left( \omega^{aff} \min(\tilde{\sigma}_{DP}^{eq}, \sigma_{lim}^{eq}) \right) \mathbf{H}(\rho^{eq} - \tilde{e}_{th}^{eq}) \mathbf{n}^d \\
\dot{\omega}^{inc} &= K_{inc} \exp \left( \omega^{inc} \min(\tilde{\sigma}_{DP}^{eq}, \sigma_{lim}^{eq}) \right) \mathbf{H}(\rho^{eq} - \tilde{e}_{th}^{eq}) \\
\dot{\omega}_{LSW} &= \frac{3\omega^{aff}}{\lambda} \\
\end{align*}
\]

(70)

where

\[ \tilde{\sigma}_{DP}^{eq} = \zeta \tilde{\sigma}_{eq}^{2} + (1 - \zeta) \text{tr} \tilde{\sigma} \]  

(71)

and

\[ \tilde{\sigma}_{DP}^{eq} = \zeta \tilde{\sigma}_{eq}^{2} + (1 - \zeta) \text{tr} \tilde{\sigma} \]  

(72)

with \( \zeta \) a material parameter (possibly different in each definition).

References


4.3 Structures computations

The full model, accounting for damage evolution and its coupling with viso-plasticity and microstructural evolutions, has been implemented in the Z-set finite element code at the LMT Cachan, starting of course from the code with no damage presented in the Appendix Q. The code is presented in the Appendix R. The corresponding material behavior model is called “RaftXD” in Z-set (D for Damage). As for the RaftX set of constitutive equations, the RaftXD set is also solved by using a fourth order Runge-Kutta explicit integration scheme. The code includes isotropic hardening (exponential law) but the associated material parameters are set to zero at considered temperatures, as shown in the Appendix S. In this Appendix the input files are presented too. Computations have been carried out in the finite strain hypothesis by updating the nodes position at each time step (commands “*** behavior raftx lagrange rotate” in the RaftXD.mat file and “*** mesh updated lagrangian” in the Z-set inputs files for creep) and seeking the equilibrium between external and internal forces (these latter found by the constitutive equations).

3D structural computations are presented next. We have chosen to simulate the creep tests realized during the present Ph.D. at 1050°C and 140 MPa along <001> and also the one performed by R. Giraud during his Ph.D. thesis along <111> under the same loading conditions (Giraud, 2013a). As in subsection 3.10.2, the axis z coincides with one <001> orientation, in the second one with <111> orientation. Again the simulation does not account for the difference between the orientation of the tested specimens and the theoretical crystallographic <111> orientation.

In the following section several results are presented in terms of equivalent plastic strain ($\varepsilon_{eq}$, defined Eq. (3.100)).

The geometry of the specimens, the finite element mesh, the boundary and loading conditions applied, are the same as the ones used in the subsection 3.10.2. The following computations are aimed to reproduce the creep tests presented in the chapter 2, which have been realized under constant force. Thus the material parameters used for the following computations are the ones which have been identified under the assumption of Finite Strains (set FS, see Appendix N). For structural computations the value of the critical damage $D_c$ is assumed to be equal to 0.999 instead of 0.15 and the failure corresponds to the numerical divergence of the computation. This coincides with 196h for the simulation along <001> and with 328h along <111>, while the observed lifetimes are 243h and 622h respectively. This difference is in part the result of the enhancement of damage by the stress redistribution and in part the result of the numerical instabilities which can appear when simulating a softening behavior (see figures from 4.1 to 4.3).

Figure 4.4 shows the creep curves computed by considering the axial strains of the nodes N1, N2 and N3 (their position is shown in the same figures) and the creep curve computed on a gauss point when simulating creep along the <001> direction. The curves computed on nodes result as accelerated because of the structural (geometric) effect on the stress state.
Figure 4.1 – Equivalent plastic strain field computed by the full model (accounting for visco-plasticity, microstructural evolution and damage) (a) after 196h of creep along the $<001>$ crystal orientation and (b) 328h of creep along the $<111>$ crystal orientation.

Figure 4.2 – Accumulated plastic strain rate field (s$^{-1}$) computed by the full model (accounting for visco-plasticity, microstructural evolution and damage) (a) after 196h of creep along the $<001>$ crystal orientation and (b) 328h of creep along the $<111>$ crystal orientation.
Figure 4.3 – Damage field computed by the full model (accounting for visco-plasticity, microstructural evolution and damage $D$, of evolution law Eq. (35) of page 11 of the article), (a) after 196h of creep along the $<001>$ crystal orientation and (b) 328h of creep along the $<111>$ crystal orientation.
Figure 4.4 – Results of creep simulations at 1050°C and 140 MPa along the $<001>$ crystal orientation computed by the full model (accounting for visco-plasticity, microstructural evolution and damage): nodes selected to extract the creep curves, contour of the equivalent plastic strain $\varepsilon_{eq}^p$ after 196h and stress strain curves for selected nodes (GP= Gauss Point).

The $\gamma$-channel distributions at the end of the simulations are presented in figures 4.5 and 4.6. As expected, after tensile creep along $<001>$, the channels normal to tensile direction widen (vertical width $w_3 = w_{33}$), especially where plastic deformation concentrates. At the same time transverse channels close ($w_1 = w_{11}$ and $w_2 = w_{22}$ become equal to zero). The $\gamma'$-rafting index contours presented in the figure 4.7 show that $\gamma'$ rafting completion is obtained along $<001>$ and that coarsening progress along $<111>$. Figure 4.8 presents the matrix channel evolution curves extracted on the same nodes (N1, N2 and N3) of figure 4.4(a). The evolution of $\gamma$ channels computed on a gauss point is also reported (GP). The evolution of $w_1$, $w_2$ and $w_3$ are faster in the nodes than on the gauss point simulation. Moreover, N2, N3 and GP curves show a non-linear end. This final acceleration is due to finite strains and damage (figure 4.8(a)). Figure 4.9(b) shows the evolution of $\gamma$ channels computed on the nodes N1, N2 and N3 (figure 4.9(a)) but this time during creep along the $<111>$ crystal direction. Even in this case the curves extracted from the nodes evolve faster compared to the one computed on a single gauss point. This is due to the plastic strain enhancement during the 3D computation.

The normalized global displacement $u/L$ ($L = 14mm$ corresponds to the gauge length) is plotted in figure 4.10 as a function of the time.
Figure 4.5 – $\gamma$ channels width (nm) computed using the full model (accounting for visco-plasticity, microstructural evolution and damage) after 196h of creep along the $<001>$ crystal orientation at 1050°C and 140 MPa.

Figure 4.6 – $\gamma$ channels width (nm) computed using the full model (accounting for visco-plasticity, microstructural evolution and damage) after 197h of creep along the $<111>$ crystal orientation at 1050°C and 140 MPa.

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
Figure 4.7 – $\gamma'$-rafting index after creep at 1050°C computed by the full model (accounting for visco-plasticity, microstructural evolution and damage): (a) after 196h of 140 MPa creep along $<001>$ orientation, (b) after 197h of 140 MPa creep along $<111>$ orientation.

Figure 4.8 – $\gamma$ channels width evolutions at different nodes and for Gauss Point (GP) calculation. (a) $\gamma'$-rafting phenomenon for $<001>$ creep at $w_3$ increasing and $w_1 = w_2 \to 0$. Curves computed by the full model (accounting for visco-plasticity, microstructural evolution and damage).
Figure 4.9 – γ channels width evolutions at different nodes and for Gauss Point (GP) calculation, (a) position of the nodes N1, N2 and N3 (b) mechanical coarsening for \(<111>\) creep at \(w_1 = w_2 = w_3\) increasing. Curves computed by the full model (accounting for visco-plasticity, microstructural evolution and damage).

Figure 4.10 – Global displacement-time response \(u/L\) vs \(t\) computed by the full model (accounting for visco-plasticity, microstructural evolution and damage): (a) creep along \(<001>\) at 1050°C and 140 MPa (b) \(<111>\) creep at 1050°C and 140 MPa.

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
4.4 Conclusions

As already said in the section 9 of the article, an original phenomenological damage model is proposed to describe the (anisotropic) strain rate sensitivity of damage onset and evolution in single crystal superalloys. Moreover, a novel strain rate sensitive damage threshold has been introduced. This law allows to describe the damage onset dependence on rate during tertiary creep and monotonic tension. Based on the previous work of Desmorat and Marull (2011), a novel formulation of the Kelvin projectors is proposed for cubic material symmetry as well. This latter, based on the harmonic decomposition of the elasticity tensor, allows to compute Kelvin projectors for cubic material symmetry simply starting from the three independent parameters $E$ (measured along the $<001>$ direction), $G$ and $\nu$. Curves identified for the CMSX-4 at $1050^\circ$C along the $<001>$ and $<111>$ crystallographic directions show that, thanks to this model, tertiary creep, time to rupture and ductility can be quite accurately described for single crystal superalloys. However, the proposed damage can be extended also to other materials deforming and degrading by mechanisms different from the ones observed in single crystal superalloys but still showing a rate dependent ductility.

Apart from what has already been presented in section 9, several further comments can be done about the proposed damage modeling. Different rupture lifetimes have been observed after the creep tests performed at $1050^\circ$C and at 140,180 and 200 MPa (figure 2.7 of subsection 2.2.1). At the same time the three specimens tested at these stress levels have provided very similar elongations to failure ($\approx 12 - 15\%$), as it can be seen from the experimental curves reported in the figures 3.9 and 3.12. From a mechanical point of view, this means that the stress intensity does not impact directly the creep damaging process. For this reason no stress level contribution is included in the damage evolution law (but a stress triaxiality effect through Lemaitre triaxiality function $R_\nu$ is included, in Eq. 36 of the article).

During creep, damage is mainly governed by plasticity. At the higher stresses plasticity develops faster and so damage does, thus the samples deform more rapidly and creep lifetimes are shorter. At the lowest stress plasticity, and then plastic damage, develops slowly so $\approx 600h$ are required to reach failure. For this reason, the proposed damage evolution law depends on the accumulated plastic strain rate. Since the main dependence of damage phenomenon during creep is on plasticity, no additional rate-sensitive effects have to be introduced.

Then, a damage threshold has been used to model the mechanical damage onset during creep, not dependent on the plastic strain at the correspond low rates. On the contrary, the damage onset strongly depends on the strain rate during monotonic tension. A change in the deformation/damaging mechanism is observed between $10^{-5}s^{-1}$ and $10^{-4}s^{-1}$. At $10^{-5}s^{-1}$ microstructural degradation enhances plasticity development and cavity diffusion, thus in this case, damage sets at lower plastic strains than when loading at lower rates. For this reason, different plastic strain thresholds, varying according with the strain rates, have then been considered and the following damage criterion has been formulated (see equations 31 and 32 and the article):

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
\[ f_D = \sup_t \left[ p_d^{\frac{\rho_d}{\varepsilon_{\rho D}}} \exp \left( -B \left( 1 - \frac{\dot{\rho}_0}{\dot{\rho}_d} \right) \right) + \frac{p_d^{\frac{\rho_d}{\varepsilon_{\rho D}}}}{\varepsilon_{\rho D}} \exp \left( -B \left( 1 - \frac{\dot{\rho}_0}{\dot{\rho}_d} \right) \right) \right] - 1. \] (4.1)

The curves presented in the sections 8 of the article, show that this choice allows to correctly model both creep lifetimes and ductility. The parameter \( D_c \) (critical damage) has been introduced in order to recover the creep lifetimes and the ductility during gauss point simulations, in order to provide a tool to predict failure during fast design computations. For the 3D structural computations presented, we have preferred to let the localization phenomenon to take place: the value of this parameter was set to 0.999. Structural failure corresponds in this case to the divergence of the computation.

The curves computed by structural computations performed on a finite element approximation of the real creep specimens do not correspond exactly to the ones computed on a single integration point, exhibiting the sensitivity of the 3D formulation to structural effects. The material parameters used for these computations have been identified by performing Gauss point simulations in the hypothesis of final strains. Identifying the material parameters by optimizing the Finite Element response with respect to the experimental one should be considered in order to improve the Finite Element prevision. Mesh sensitivity should be investigated too. The average size of a mesh c3d20 element has been presented in the figure 3.27.

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
General conclusion and perspectives

“My way”
Sex Pistols, cover of Frank Sinatra’s song
The scope of the present Ph.D. was to develop a physically based mechanical model accounting for Ni-base single crystal superalloys. During typical high temperature in-service conditions \(T \geq 900^\circ\text{C}\), these materials are subjected to creep and their visco-plastic response is affected by microstructural evolutions and specific damage mechanisms. Special attention has been given to the CMSX-4 superalloy used to manufacture high pressure turbine blades of Safran helicopter engines.

After a literature review pointing out missing information and data required for its modeling (section 1.6 of chapter 1), the second step of presented work has been to plan and realize an experimental campaign on CMSX-4 superalloy (chapter 2). The literature review revealed that not enough data were available at 1050°C, which was the target temperature for the modeling, thus creep and monotonic tensile tests have been carried out at this temperature (presented in the subsections 2.2.1 and 2.2.2). Similarly, not enough information were available in the low temperatures/high stress domain \(T < 900^\circ\text{C}\), so that further creep tests have been carried out at 850°C (presented in the section 2.4). The role of microstructural evolution and damage mechanisms during repeated One Engine Inoperative events (OEI events, defined page ) was not been explored exhaustively (so that the modeling of failure under non-isothermal conditions will be a natural perspective to this work). But as they will be needed some non-isothermal tests have been carried out as well (presented in the subsection 2.3). Since the \(\gamma'\) dissolution phenomenon is highly sensitive to the chemical composition of the alloy and even if data characterizing the \(\gamma'\) volume fraction evolution with temperature were already available in literature for CMSX-4, it has been decided to perform dissolution tests and to generate data specifically for the present study (results for considered CMSX-4 alloy presented in the subsection 2.5). We point out that, apart from the modeling considerations and scopes, two interesting conclusions have been drawn from the experimental results. The first is that microstructural evolutions have been found to be the main cause of degradation during repeated OEI events. In fact, as explained in paragraphs 2.3 and 2.3, no particular damage mechanisms have been observed during post-mortem observations. The second is that twinning develops during creep at 850°C and along along the \(\langle 011 \rangle\), \(\langle 112 \rangle\) and \(\langle 111 \rangle\) crystallographic directions (subsection 2.4.1). This deformation mechanism is quite unusual and rarely observed in single crystal superalloys.

The third step has consisted in the modeling of the visco-plastic response of CMSX-4 in the high temperature range \((T<900^\circ\text{C},\ \text{chapter 3})\). Since the visco-plastic properties of single crystal superalloys at high temperature are affected by microstructural evolutions, a key point for the modeling has been to represent these evolutions and their coupling with visco-plasticity. Both literature studies and the experiments performed during this Ph.D. (chapter 1 and 2 respectively), have shown that different deformation mechanisms and \(\gamma/\gamma'\) evolutions occur when loading along the \(\langle 001 \rangle\) and \(\langle 111 \rangle\) crystallographic direction and that, moreover, the response along others directions results from a combination of these phenomena. For this reason, the modeling has been developed to primary describe the material response along these two directions. A phenomenological model has been proposed for the \(\gamma\)-channels evolution by representing it by means of a second order tensorial variable (section 3.4). This model allows to account for the \(\gamma'\)-rafting and
General conclusion and perspectives

Coarsening, as well as for the $\gamma'$-dissolution. The law describing the evolution of the $\gamma/\gamma'$ microstructure is formulated according to the (cubic) anisotropy of the material, which in the proposed model is described via the Kelvin decomposition of the elastic tensor (section 3.3). This formulation allows to reproduce the morphological evolution of $\gamma/\gamma'$ microstructure according to the crystal direction loaded (section 3.4.5). Microstructural evolutions have been coupled to both

- a phenomenological model of visco-plasticity which is also formulated by using the Kelvin decomposition,
- a crystal plasticity model accounting for both octahedral and cubic slips.

Both models are equivalent for uniaxial loadings, possibly complex, along $<001>$ and along $<111>$ directions so that they are denoted next as “the model”. For the Kelvin mode based formulation, two visco-plastic criterion functions have been introduced in order to recover the multi-criterion nature of the crystal plasticity at the meso-scale (section 3.5.1). The loss of structural hardening due to the evolution of the $\gamma/\gamma'$ microstructure has been modeled as a size reduction of the yield surface by introducing the Orowan stress (subsection 1.1.3) in each criterion function (Eq. 3.61). The model has been identified on the tests performed during the present Ph.D. work (chapter 2) and on the tests realized by R. Giraud during his Ph.D. thesis (Giraud, 2013a). The comparisons between the curves identified and the experimental results show that the model allows to correctly recover the visco-plastic behavior of the CMSX-4 at 1050°C. In particular, the creep response of CMSX-4 along $<001>$ and $<111>$ at 1050°C is characterized by an acceleration of the plastic strain since the first stages of creep. The proposed model allows to recover this acceleration only thanks to the modeling of microstructural evolution and visco-plasticity. As highlighted in the section 3.11, we consider this as a major achievement of present work.

The final step of the work has been to propose a damage model and to couple it with visco-plasticity and microstructural evolutions (chapter 4). The main scope of the damage modeling was to correctly predict both the creep lifetimes and the ductility in tension. The understanding of the damage mechanisms and of their dependence on the strain rate (sections 1.3.2 and 2.2.2) has allowed to propose a new phenomenological damage evolution law for CMSX-4 and also a novel expression for the damage threshold, which is rate dependent. Since this formulation allows to correctly recover both the creep lifetimes and the ductility in tension at different strain rates, the second modeling objective of the present Ph.D. has been achieved.

It has to be remarked that the proposed model has been encoded in the Z-set finite element computer code. Two versions of the code exist. A first one accounting for visco-plasticity and microstructural evolution (Appendix Q, programmed with the help of Tonya Rose at Safran Tech) and a second one also accounting for damage (Appendix R, programmed by myself on the basis of previous code). In both cases, the model has shown good abilities for finite element computations (sections 3.10 and 4.3).
Perspectives

Further developments of the presented work naturally concern the modeling of the creep response along crystallographic direction different from \(<001>\) and \(<111>\), the response under non-isothermal and more complex loading paths (possibly multiaxial, non-proportional), the description the creep response at low temperature and high stress level (for which the isotropic and kinematic hardening will not have a negligible effect). The thermodynamics framework allows to account for microstructural evolutions under non-isothermal loading (Appendix I) and under alternated loading (Appendix M). However, further investigations and modeling development should be carried out to describe the mechanical response under particular isothermal loading loadings. For example, compressive creep should be investigated and modeled as well as the role of the loss of structural hardening during cyclic loading (including creep-fatigue).

The material parameters have been identified at 850°C, 1050°C and 1200°C (values and curves are presented in the Appendix P), but the performances of the model under non-isothermal loading (including OEI events) has not been tested. This is left to further work.

Multiaxiality, other crystallographic orientation

In chapter 3, a comparison between the experiments and the model has been presented only for the \(<001>\) and \(<111>\) crystallographic directions. Some features for the description of the mechanical response along others crystallographic directions have already been introduced in the Kelvin mode based model, i.e. the Kelvin stresses representative in a phenomenological way of the octahedral and of the cubic slips, the two criterion functions with two parameters \(h\) a Hill parameter and \(a\) for the use of a non quadratic norm— not sensitive in \(<001>\) and \(<111>\). A numerical optimization of the sets of parameters needs to be performed for both our Kelvin mode based formulation and for our crystal plasticity based formulation, including additional crystallographic orientations (see for instance the work of Olivier et al (2017). The formulation of our model given in the crystal plasticity framework in section 3.9.2 of chapter 3, even if seen as more computer time consuming than a macroscopic model, has not to be neglected for the practical extension to multiaxiality.

The mechanical response along crystallographic directions different from \(<001>\) and \(<111>\) can be influenced by the rotation of the crystal lattice (mainly at low temperatures, as explained in the section 1.5 and 2.4 but also at high temperatures, as suggested in the paragraph 2.2.1). This rotation involves a change in the local stress state and, consequently, in the plasticity and microstructural evolution development. Thus, modeling the crystal lattice rotation and the rotation of the \(\gamma/\gamma'\) microstructure may prove of primary importance to describe the mechanical response along unstable crystal directions. The evolution of the microstructure under complex multiaxial stress state has not been tested either. As a consequence of a complex local stress state, the extremities of the \(\gamma'\)-rafted particles sometimes tend to be inclined compared to the \(<100>\) crystallographic
directions (as shown at the paragraph 2.2.1 and in the works of Reed et al (2007a), Caccurini e al (2017)). In this case the microstructure state cannot be described by a diagonal matrix channel because the $\gamma$ channel widths are not parallel to the principal direction of the (cubic) Natural Anisotropy Base but by a full second order tensor. Out-of diagonal components of $\gamma$ channel width tensor $w$ have, then, to be induced by the loading and by a therefore enriched evolution law.

**Low temperatures/high stress domain**

Experimental results performed in the low temperatures/high stress domain open up many modeling/experimental perspectives. At low temperatures (T$<$900°C) primary creep has to be modeled. Its dependence to the crystallographic direction and the applied stress magnitude should be modeled too (section 2.4). Creep experiments performed at 850°C (section 2.4) have shown that the creep response at low temperatures and high stress level along the $<011>$, $<112>$ and $<111>$ crystallographic directions is influenced by twinning development. However, the mechanisms governing the twinning onset and kinetics have not been still clarified, so further experiments and observations should be performed to investigate these aspects. Furthermore, since the presence of twinning bands involves a loss of the single crystal character of the material at Representative Volume Element scale, (visco-)plasticity and damage mechanisms are affected by the presence of grain boundaries (the boundaries between the twinning bands and the single crystal structure). The consequences of twinning on plasticity and damage mechanisms has to be investigated and modeled.

**3D computations, finite strains**

There are some differences between the curves computed with the finite element simulations of the testing specimens meshed in 3D and the experimental results considered as given at a material point. This difference has been anticipated (two sets of material parameters, one SS under the small strain assumption, one FS under the finite strain assumption) but no FE computation has been considered in the identification procedure. Considering some when identifying will improve the robustness of the modeling. However numerical difficulty such as mesh sensitivity may arise (Saanouni et al, 1989). Then, the mesh sensitivity should be investigated and non-local techniques should be considered. The set of material parameters can be optimized by minimizing the difference between the global response of the structure and the experimental results, using for example the model reduction technique applied to model identification proposed by Olivier et al (2017) at Centre des Matériaux, Mines ParisTech. Further (high-temperature) development of the Digital Image Correlation (DIC) followed by weighted Finite Element Method Updating (FEMU) (Mathieu et al, 2015, Guery et al, 2016) could also brings to more accurate estimation of the material parameters. Apart from material parameters, some differences between the computations and the experimental may be due to an early localization. We leave to further work the study of the instabilities due to the rafting and damage processes and of
Constitutive equations have been developed first in the small strain hypothesis (so a Lagrangian formulation was adopted). Finite strains have been accounted by gaussian point simulations by correcting the actual stress with the strain computed at the previous time step. In finite element computations, the extension to Finite Strains is done by re-actualizing the Lagrangian formulation at each time step by updating the position of the nodes. However, a proper finite strain formulation has to be considered, as for the example the formulation proposed by Mandel (1973) for crystal plasticity, which is used in Fivel and Forest (2004), Forest (2012). Crystal rotations and Finite Strains could be gained together by adapting the Cosserat formulation proposed for crystal plasticity in (Forest et al, 1997).

Finally, it has to be remarked that the constitutive equations have been encoded by an explicit integration scheme. A fourth order Runge-Kutta technique is used and the equilibrium equations are solved by use of the initial elastic matrix. Even if the performance of the model in term of numerical cost are already satisfying, some computational time could be saved. For explicit integration techniques, a choice would be to accelerate the convergence by using the residual-based acceleration techniques, as for example the ones described in (Serge, 2005, Ramiére and Helfer, 2015). Another choice would be the number of iterations by using an implicit integration scheme or by calculating the tangent coherent operator. However, these two solutions introduce a further computational effort, the solution of a linear system of equation in the first case and the assembling of the tangent coherent operator in the second case. Hence, the minimum CPU time would be a trade-off between the introduced improvement and the additional computational effort. Moreover, there exists many numerical techniques which allow to save computational time by operating at the Finite Element resolution level. Among them, the parallelism techniques in which the tangent matrix construction and factorization is distributed on several CPUs (MUltifrontal Massively Parallel sparse direct Solver developed by Amestoy et al (2000)). There also exist techniques which reduce the cost related to the solution of the equilibrium system of equations (when using an implicit integration scheme), as for instance the Proper Orhtogonal Decomposition (POD) presented in Carlberg et al (2011) and the Proper Generalized Decomposition based non-linear solvers (Ladevéeze, 2012) (PGD). Finally, others techniques of model reduction help to reduce the CPU time dedicated to both the construction of the tangent operator and the solution of the equilibrium equations. Recent examples are the global/local method developed by Blanchard et al (2017), the “A-priori hyper-reduction method” proposed by (Ryckelynck, 2012) the Empirical Interpolation Method (EIM) Barrault et al (2004) and the “Reference Points Method” (Capaldo et al, 2017).
Appendix A

Experimental procedures and observation techniques

A.1 Material

Specimens tested in this study are cast from CMSX-4 bars solidified from the same Master Heat. These bars have been all subjected to the same solution and thermal treatments. This choice is made on purpose. The experimental results are in fact intended to provide a solid database for the model identification and testing. Then it is convenient not to introduce any scatter related to the chemistry or to the initial size of the microstructure. The chemical composition of the tested alloy is reported in the table A.1. Table A.2 contains the details of the solution heat and thermal aging treatments. The initial volume fraction and size of precipitates, as well as the initial matrix channels width, are presented in the Table A.3.

Confidential

Table A.1 – Nominal chemical composition (in weight%) of the CMSX-4 alloy tested in this study.

Confidential

Table A.2 – Solution heat and aging treatments performed on the CMSX-4 alloy tested in this study. GFC = for Gas Furnace Cooling, AQ = Air Quench.
A.2 Experimental procedures

A.2.1 Creep tests

Cylindrical creep specimens with a 14 mm gauge length and a 4 mm diameter in the gauge section are used to perform all creep tests. The geometry is shown in Figure A.1.

![Figure A.1 – Geometry of the specimens used to perform tensile creep tests.](image)

Creep rigs used for tests in air are equipped with a radiant furnace. Temperature is controlled with an S-type thermocouple. The extremities of the thermocouple are placed in two holes machined close to the gauge length. It allows a bulk temperature control without interfering with microstructural evolutions. An example of the bores is given in Figure A.2 but for a prismatic specimen. The same is realized for the cylindrical ones.

During creep tests at 850°C, the displacement in the gauge length is measured with a Linear Variable Differential Transformer and loading is realized after a three hours soak time. During creep tests at 1050°C displacements is measured with a laser contactless extensometer (Figure A.3) and loading is realized after a reduced soak time.

The accuracy of displacement measurements is of 3–6 μm. This estimation accounts for the extensometer accuracy and for the fluctuation of the temperature control, equals to (± 2°C).

All creep tests have been carried out under constant load mode. A load cell allows to follow the load evolution. Data acquisition is launched before the application of the load.
Figure A.2 – From Ghighi (2013): example of thermocouple slots on a prismatic specimen.

Figure A.3 – Laser contactless extensometer (From Ghighi (2013).)
It is worth mentioning that grips/lines in these are assembled with rotules, allowing the creep specimens to deform freely. Indeed, no bounding condition are imposed by forcing, for example, lines to remain perfectly aligned. Creep tests in high vacuum have been performed with an instron 1271 machine, equipped with a radiant furnace. Tests have been performed at pressure of $\sim 10^{-7}$ mbar.

### A.2.2 Monotonic tensile tests

Cylindrical specimens presented in figure A.1 are also used for monotonic tensile tests. These tests are realized with an instron 8562 servo-hydraulic machine (Figure A.2.2). It is equipped with a 100 KN load cell. Heating is ensured by a resistive furnace and temperature is controlled by two thermocouples, one for the upper side of the furnace and the other for the lower side.

Deformation is tracked by a Linear Variable Displacement Transducer (LVDT) and by an epsilon extensometer (leaning on the gauge length) having ceramics arms.

### A.2.3 Non-isothermal tests

Two non-isothermal tests have been performed using MAATRE rig (Mécanique et Aérothermique d’Aube de Turbine Refroidies). This is a burner rig designed to mimic the in-service loading conditions in high temperature components of gas turbines. The basic operative principle is schematically described in Figure A.5. More details about the the rib features can be found in Mauget et al (2014).
The specimens used for non-isothermal creep tests have a prismatic gauge length section and a gauge length of 14 mm (figure A.6). These specimens are aligned within the [001] crystal axis with a ±5° primary misorientation. The geometry is shown in Figure A.6. Prismatic specimens are chosen for non-isothermal tests for three main reasons. First, they allow to reproduce the thin-wall oxidation effect observed on high temperature blades. Second, they allow to obtain a quasi-homogeneous temperature in the gauge volume even during thermal shocks. Third, exposing the thickness of the specimen to the gas flow reduces the trailing edge re-circulation and hence, drag stresses.

The temperature is controlled on the sample surface with a spot welded platinum S type thermocouple. A camera measures the displacements between two platinum markers having a 1 mm diameter and positioned at ±2 cm from the gauge length center. Total and plastic strain are then post processes based on the video-extensometric measurement. Details are shown in Figure A.7.

A.2.4 Dissolution tests

Two creep specimens (orientation A, Figure 2.2 Chapter 2) are crept at 1050°C − 140 MPa for 100h in order to obtain a rafted microstructure and to saturate the plasticity effect on the γ′ volume fraction rate (Giraud et al, 2013b) (section 1.2.3). Specimens are
obtained by cutting the crept specimens longitudinally in two parts and then by sectioning it into half-discs having a 2 mm thickness (Figure A.8). Thermal agings are realized using a Nabertherm 1800 resistive furnace. Temperature is assured with a $\pm 2^\circ$C precision. Because of the size of the specimens no soaking time is required.

A.3 Observation techniques

Mirror surface finish has been realized by performing mechanical polishing up to 4000 SiC with grit papers and then by polishing with 3µm and 1 µm diamond solutions. A final chemical etching with acqua ragia ($1/3 \text{HNO}_3 + 2/\text{HCl} / \text{vol. part}$) has been used to reveal the $\gamma/\gamma'$ microstructure. Scanning Electron Microscopy (SEM) is realized using a Jeol 7000F microscope. For Electron Back Scatter Diffraction (EBSD) an OIM-TSL system installed on a Jeol 6100 SEM is used. Transmission Electron Microscopy (TEM) is performed by using a Jeol 3010 microscope equipped with a LaB6 filament and used at an accelerating tension of 300kV. Surface are refined to 15 µm by mechanical polishing and final preparation to achieve electron transparency has been done by ion milling using GATAN PIPS equipment.
Appendix B

Post-processing of the experimental curve and image analysis

B.1 Post-processing of experimental curves

Creep curves are smoothed using the Savitzky-Golay filter (Savitzky and Golay, 1964). The mobile average technique is used to smooth the results of non-isotherm tests. Variable windows widths are used to avoid altering the initial and final shape of the curves.

B.2 Image analysis

Image analysis is performed on the SEM observations of the tested specimens in order to measure:
— the $\gamma$-channel widths at the end of tests;
— the $\gamma'$ volume fraction (at equilibrium) variation with temperature;
— the connectivity number (Caron et al, 2008).
Details are presented below.

B.2.1 $\gamma$ channel width

Manual image analysis of the post-mortem SEM observations is used to quantify the $\gamma$ channel widths after high temperature tests. The measurements took after creep tests performed along A and D crystallographic orientation (see figure2.2, chapter 2) are further used to identify the parameters of the model. More than 150 measures are manually taken on high magnification images (higher than x5000 magnification). They are used to build the frequency histogram and to identify a gaussian distribution. After creep along the specimens of type A (figure2.2) a N-rafted microstructure is observed. For this reason only the horizontal channels are measured. On the contrary, along the specimens of type D, three channels remain active because of mechanical coarsening, but only two are visible in 2D imaging. The Gaussian distributions of the two channels are built separately.
in order to verify the deviation from the mechanical coarsening hypothesis of isotropic growth. Since small deviations are observed, a final unique Gaussian distribution is built. A finer investigation could be carried out by analyzing the cross section images, as for example the figures 2.17(a) and 2.17(c) presented in the chapter 1.

Figure B.1 – Exemple of the $\gamma$ channel measurement by manual image analysis along A: (a) horizontal channel $w_1$; (b) frequency histogram and Gaussian distribution of measurements.

Small difference between the $w_1$ and $w_2$ distribution were anyway expected (see figure B.2 (b) and (c)). D samples are not perfectly aligned with the theoretical $<111>$ crystal direction, thus the stress in $\gamma$ channels is non exactly the same in the three channels. The non-perfect loading symmetry corresponds to an asymmetry of the plasticity development in the channels and of the channel width evolution.

B.2.2 Equilibrium $\gamma'$ volume fraction

The area fraction of specimens at the thermodynamic equilibrium is measured by processing the SEM images of the aged specimens with the Visiolog software. Assuming that the length of the rafted particles is higher than the raft thickness (in agreement with Diologent et al (2002)), the area fraction of $\gamma'$ is assimilated to the volume fraction.

In order to highlight the effect of Rhenium segregation, three images from the primary dendrite arms and three images of the dendritic microstructure are analyzed separately. On each image, the surface fraction of precipitates is computed as the ratio of the number
Figure B.2 – Example of the γ channel measurement by manual image analysis along D: (a) frequency histogram and Gaussian distribution of $w_1$ and $w_2$ measurements; (b) frequency histogram and Gaussian distribution of $w_1$ measurements; (c) frequency histogram and Gaussian distribution of $w_2$.

of pixels occupied by precipitates and the total number of pixels of the image. Images

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
having a magnification of 5000 are chosen for the analysis. Before the measurement, these are subjected to:

– contour smoothing;
– contrast and light correction, in order to accentuate the difference between the two phases;
– binarization;
– erosion and reconstruction, in order to remove the hyper fine $\gamma''$-phase precipitated during the cooling from the aging to the ambient temperature.

Results are presented in the figure 2.52 of chapter 2 and 3.7 of chapter 3.

### B.2.3 Connectivity and phase inversion

The connectivity number ($N_{A(\gamma)}$) is measured after dissolution tests and high temperature creep tests by following the procedure suggested in Fredholm and Strudel (1984), Caron et al (2008). Measurements are reported in the Figures B.3 and B.4. All the microstructures having a negative connectivity number have an inverted topology. According to Caron et al (2008), they corresponds to conditions where an equilibrium $\gamma'$ volume fraction higher than $\simeq 0.5$ is observed (figure 2.52 of the chapter 2).

![Figure B.3 – Measurements of the connectivity number ($N_{A(\gamma)}$) performed after dissolution tests. TI = Topologically Inverted.](image-url)
Figure B.4 – Measurements of the connectivity number ($N_{A(\gamma)}$) performed after several creep tests at 1050°C. TI = Topologically Inverted.
Post-processing of the experimental curve and image analysis

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
Appendix C

Experimental results

C.1 Misorientation of the tested samples

<table>
<thead>
<tr>
<th></th>
<th>θ (°)</th>
<th>ρ (°)</th>
</tr>
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<tbody>
<tr>
<td>A</td>
<td>1.3</td>
<td>3.3</td>
</tr>
<tr>
<td>B</td>
<td>23.7</td>
<td>2.3</td>
</tr>
<tr>
<td>B’</td>
<td>21.4</td>
<td>9.2</td>
</tr>
<tr>
<td>C</td>
<td>45.1</td>
<td>6.3</td>
</tr>
<tr>
<td>C’</td>
<td>44.2</td>
<td>0.9</td>
</tr>
<tr>
<td>D</td>
<td>52.9</td>
<td>44.3</td>
</tr>
<tr>
<td>D’</td>
<td>52.8</td>
<td>41.9</td>
</tr>
<tr>
<td>E</td>
<td>29.7</td>
<td>38.9</td>
</tr>
</tbody>
</table>

Table C.1 – Location of all the crystal tested in the standard stereographic triangle (Laue convention).

Figure C.1 – Location of all the crystal tested in the standard stereographic triangle.

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
C.2 Creep and monotonic tensile test results

All the results are presented in term of true stress and true strain.

Symbols and definitions:
— creep lifetime \( t_{\text{rup}} \);
— rupture elongation \( A\% = 100 \frac{L_f - L_i}{L_i} \), where \( L_f \) is the specimens length measured after rupture, \( L_i \) the nominal length and \( L_g \) the gauge length (equal 14mm);
— \( S\% \) is the cross section area reduction. This is computed as \( S\% = 100 \frac{S_f - S_i}{S_i} \), where \( S_f \) is the final cross section area computed in the hypothesis of circular section and using the measured final diameter. \( S_i \) is the initial cross section area computed using the nominal diameter;
— ellipticity of the cross section, \( e = \frac{D_f - D_i}{D_i} \);
— when the cross section presents a non-homogeneous necking, \( S_{\text{min}}\% \) and \( S_{\text{max}}\% \) are measured instead of \( S\% \). \( S_{\text{min}}\% \) and \( S_{\text{max}}\% \) are measured by substituting to \( D_f \) the minimum measured diameter \( D_{\text{min}} \) and the maximum measured diameter \( D_{\text{max}} \) respectively;
— primary creep amplitude \( \varepsilon_0 \). In the present work this is defined as the strain corresponding to the minimum creep rate (see Figure C.2). A deviation of \( \simeq 0.1 \) is associated at each measure;
— minimum creep rate \( \dot{\varepsilon}_{\text{min}} \).

Figure C.2 – Strain corresponding to the minimum creep rate. Example on the creep curve along A at 850°C and 750 MPa.

<table>
<thead>
<tr>
<th>( T ) (°C)</th>
<th>( \sigma ) (MPa)</th>
<th>( t_{\text{rup}} ) (h)</th>
<th>( A% )</th>
<th>( S_{\text{min}}% )</th>
<th>( S_{\text{max}}% )</th>
<th>( e % )</th>
<th>( S% )</th>
<th>( \varepsilon_0 ) (%)</th>
<th>( \dot{\varepsilon}_{\text{min}} ) (s(^{-1}))</th>
<th>testing conditions</th>
</tr>
</thead>
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<td>Confidential</td>
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</tbody>
</table>

Table C.2 – Summary of creep tests performed at 1050°C. NM = Non-Measurable. AL = Air Laboratory.

<table>
<thead>
<tr>
<th>( T ) (°C)</th>
<th>( \sigma ) (MPa)</th>
<th>( t_{\text{rup}} ) (h)</th>
<th>( \varepsilon_0 )</th>
<th>( \dot{\varepsilon}_{\text{min}} ) (s(^{-1}))</th>
<th>testing conditions</th>
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<tr>
<td>Confidential</td>
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</table>

Table C.3 – Summary of the results of creep tests performed by Giraud (2013a) at 1050°C and recalled in the present work (chapter 2. AL = Air Laboratory.

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
Table C.4 – Summary of creep tests performed at 850°C. AL = Air Laboratory. IV = In-Vacuum.

Table C.5 – Summary of interrupted creep tests performed at 850°C. AL = Air Laboratory.

Table C.6 – Results of the monotonic tensile tests along D.

C.3 Monotonic tensile tests

C.4 γ channel widths

Table C.7 – γ′ channel widths measured after high temperature tests along A and D.

C.5 Non-isothermal tests

Table C.8 – Summary of the non-isothermal tests realized on the MAATRE rig.

C.6 Precipitate volume fraction at the thermodynamic equilibrium

Measurements are carried out on SEM images after dissolution tests. In the Table C.9 “max var” and “min var” are defined as the differences between the max and the min measures values with respect to the average. These differences correspond to the error bars of the Figures 2.52 presented in the Chapter 2.
### Table C.9 – Equilibrium γ' volume fraction variation with temperature measured by image analysis in the dendrite and inter-dendrite spaces. NM = Non-Measurable.

<table>
<thead>
<tr>
<th>T (°C)</th>
<th>$f_{\gamma_{eq}}$</th>
<th>max var D</th>
<th>min var D</th>
<th>$f_{\gamma_{eq}}$</th>
<th>max var ID</th>
<th>min var ID</th>
<th>test type</th>
</tr>
</thead>
</table>

 Confidential
Appendix D

From Tinga et al evolution law to $\dot{w} = ...$

Let us consider uniaxial tension or compression along orientation $1 \equiv [001]$. Diagonal deviatoric part of stress tensor $\sigma^d$ (in cubic Natural Anisotropy Basis) is equal to stress deviator $\sigma'$ and defines normal $n^d$, as

$$\sigma' = \sigma^d = \frac{2}{3} \sigma \begin{bmatrix} 1 & 0 & 0 \\ 0 & -\frac{1}{2} & 0 \\ 0 & 0 & -\frac{1}{2} \end{bmatrix} = \frac{2}{3} \sigma_{eq} n^d \quad \sigma^d_{eq} = \sigma_{eq} = |\sigma| \quad (D.1)$$

Evolution law (3.38) gives a diagonal second order tensor $\ell$ which commutes with normal $n^d$. Altogether with the assumption of constant volume of precipitates $V' = \ell_1 \ell_2 \ell_3 = \ell_L \ell_T^2 = \ell_0^3$ inherent to Eq. (3.38) this defines

$$\ell = \begin{bmatrix} \ell_L & 0 & 0 \\ 0 & \ell_0^3/\sqrt{\ell_L} & 0 \\ 0 & 0 & \ell_0^3/\sqrt{\ell_L} \end{bmatrix}_{NAB} \quad \mathbf{L} = \mathbf{n}^d \cdot \mathbf{l} = \mathbf{n}^d \cdot \mathbf{L} = \text{sgn}(\sigma) \begin{bmatrix} \ell_L & 0 & 0 \\ 0 & -\ell_0^3/2\sqrt{\ell_L} & 0 \\ 0 & 0 & -\ell_0^3/2\sqrt{\ell_L} \end{bmatrix}_{NAB}$$

where we set $\ell_1 = \ell_L$ and $\ell_2 = \ell_3 = \ell_T = \ell_0^3/\sqrt{\ell_L}$ respectively the longitudinal and the transverse sizes of large $\gamma'$ precipitates. Evolution law (3.38) becomes

$$\dot{\ell} = -\frac{1}{\tau_\ell} \frac{\sigma_{eq}}{\sigma_{eq} + \sigma_e} \exp\left(-\frac{Q_\ell - \sigma_{eq} U}{k_B T}\right) \mathbf{L} = -\frac{1}{\tau_\ell} \frac{\sigma_{eq}^d}{\sigma_{eq}^d + \sigma_e} \exp\left(-\frac{Q_\ell - \sigma_{eq}^d U}{k_B T}\right) \mathbf{L} \quad (D.3)$$

Using in present uniaxial case the fact that in tension $\text{sgn}(\sigma) = 1$, $\ell_L < \ell_0$ due to $\dot{\ell}_1 < 0$ (from Eq. (3.38)), and in compression $\text{sgn}(\sigma) = -1$, $\ell_L > \ell_0$ (from Eq. (3.38)), one gets

$$\mathbf{L} = \frac{2\ell_0^{3/2} + \ell_L^{3/2}}{3\sqrt{\ell_L}} \mathbf{n}^d + \text{sgn}(\sigma) \frac{\ell_L^{3/2} - \ell_0^{3/2}}{3\sqrt{\ell_L}} \mathbf{l} = \frac{2\ell_0^{3/2} + \ell_T^{3/2}}{3\sqrt{\ell_L}} \mathbf{n}^d - \frac{\ell_L^{3/2} - \ell_0^{3/2}}{3\sqrt{\ell_L}} \mathbf{l} \quad (D.4)$$

Eq. (3.38) can be rewritten in a tensorial form valid for tensile of compressive loading in

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any \(<001>\) directions,

\[
\dot{\ell} = \frac{1}{\tau_\ell} \frac{\sigma_{eq}^d}{\sigma_{eq}^d + \sigma_e} \exp \left( - \frac{Q_L - \sigma_{eq}^d U}{k_B T} \right) \left[ \frac{\ell_L^{3/2} - \ell_0^{3/2}}{3 \sqrt{\ell_L}} \right] \left[ 1 - \frac{2 \ell_L^{3/2} + \ell_0^{3/2}}{3 \sqrt{\ell_L}} \right] \mathbf{n}^d
\]  

(D.5)

Combined with an anisotropic periodicity growth \(\dot{\lambda} = \dot{\lambda}' + \frac{1}{3} \text{tr} \dot{\lambda} \mathbf{1} \geq 0\) (growth meaning that \(\dot{\lambda}\) is a definite positive second order tensor), one obtains an evolution law for \(\gamma\) channel width \(\dot{w} = \dot{\lambda} - \dot{\ell}\) as the sum of a deviatoric part (interpreted as rafting contribution, including homothetic growth) and of a spherical or isotropic part (coarsening, including homothetic growth). As furthermore second order periodicity tensor \(\dot{\lambda}\) also has equal transverse elongations \(\lambda_2 = \lambda_3 = \lambda_T\), it is of the form

\[
\dot{\lambda} = \dot{L} \mathbf{n}^d + \dot{\lambda}_{iso} \mathbf{1} \quad \dot{\lambda}_{iso} = \frac{1}{3} \text{tr} \dot{\lambda}
\]

(D.6)

so that evolution law (3.38) combined with anisotropic periodicity growth ends up in to

\[
\dot{w} = \dot{w}_{\text{raft+hom}}' + \dot{w}_{\text{coars+hom}}
\]

\[
\dot{w}_{\text{raft+hom}}' = \left[ \frac{1}{\tau_\ell} \frac{2 \ell_L^{3/2} + \ell_0^{3/2}}{3 \sqrt{\ell_L}} \frac{\sigma_{eq}^d}{\sigma_{eq}^d + \sigma_e} \exp \left( - \frac{Q_L - \sigma_{eq}^d U}{k_B T} \right) + \dot{L} \right] \mathbf{n}^d
\]

\[
\dot{w}_{\text{coars+hom}} = \dot{\lambda}_{iso} - \frac{1}{\tau_\ell} \frac{\ell_L^{3/2} - \ell_0^{3/2}}{3 \sqrt{\ell_L}} \frac{\sigma_{eq}}{\sigma_{eq} + \sigma_e} \exp \left( - \frac{Q_L - \sigma_{eq} U}{k_B T} \right)
\]

(D.7)

The \(\gamma\) rafting contribution is therefore proportional to the normal \(\mathbf{n}^d\) to yield surface \(f = \sigma_{eq}^d - \sigma_y\). A periodicity growth term \(\dot{\lambda}_{iso} \geq 0\) large enough is needed to ensure coarsening.

It is important to point out that, even if only \(<001>\) loading are considered here, one has anticipated the fact that misoriented \(\gamma\) rafting is also governed by the diagonal deviatoric part of stress tensor \(\sigma^d\) in Natural Anisotropy Basis: even if one has the equality \(\sigma_{eq} = \sigma_{eq}^d\) in \(<001>\) uniaxial loading cases, rafting contribution in Eq. (D.7) is written in terms of invariant \(\sigma_{eq}^d\). For coarsening the whole stress components may act so the full von Mises stress \(\sigma_{eq}\) is kept.

**Remark** If parameter \(\sigma_e\) is not small, the term \(\frac{\sigma_{eq}^d}{\sigma_{eq}^d + \sigma_e}\) (resp. \(\frac{\sigma_{eq}}{\sigma_{eq} + \sigma_e}\)) introduces, through hardening, a dependency to accumulated plastic strain \(p^d\) (resp. \(p\)) that tends to saturate.
Appendix E

Kelvin spectral decomposition of elasticity tensor

The elasticity fourth rank tensor $\mathbb{E}$ (resp. its Voigt matricial representation $[E]$) has eigenvalues $\Lambda^{(I)}$ and corresponding second rank symmetric eigentensors $e^{(I)}$ (resp. eigenvectors $\hat{e}^{(I)}$) are solutions of the eigenproblem

$$\mathbb{E} : e^{(I)} = \Lambda^{(I)} e^{(I)}, \quad e^{(I)} : e^{(J)} = \delta_{IJ}$$  \hfill (E.1)

with $\delta_{IJ}$ the Kronecker symbol. The couples $(\Lambda^{(I)}, e^{(I)}$ or $\hat{e}^{(I)}$) are the Kelvin modes (or strains here), the eigenvalues $\Lambda^{(I)}$ are the Kelvin moduli (Rychlewski, 1984). These moduli are at most six (Cowin et al, 1991), they are only two in case of isotropic elasticity ($3K$ and $2G$ if $K$ and $G$ are the bulk and shear moduli).

Kelvin decomposition of Hooke tensor is then simply the following re-writing

$$\mathbb{E} = \sum_{I=1}^{6} \Lambda^{(I)} e^{(I)} \otimes e^{(I)} \quad \mathbb{E} = \sum_{k=1}^{N<6} \Lambda^{k} P^{k} \quad \mathbb{S} = \mathbb{E}^{-1} = \sum_{k=1}^{N<6} \frac{1}{\Lambda^{k}} P^{k} \quad P^{k} = \sum_{I/\Lambda^{(I)}=\Lambda^{k}} e^{(I)} \otimes e^{(I)}$$  \hfill (E.2)

There is always a family of six orthogonal eigentensors $e^{(I)}$ but some eigenvalues can be multiple, depending on the material symmetry. The terms of identical moduli $\Lambda^{(I)} = \Lambda^{k}$ are grouped to form Kelvin projectors (François, 1995), if $\mathbb{S}$ is compliance tensor, with $N$ the number of different Kelvin moduli. The projectors $P^{k}$ are unique for a given elasticity tensor $\mathbb{E}$.

The elastic energy density reads

$$w_{e} = \frac{1}{2} \mathbf{e} : \mathbb{E} : \mathbf{e} = \frac{1}{2} \mathbf{\sigma} : \mathbb{S} : \mathbf{\sigma} = \frac{1}{2} \sum_{k=1}^{N} \Lambda^{k} \mathbf{e}^{k} : \mathbf{e}^{k} = \frac{1}{2} \sum_{k=1}^{N} \frac{1}{\Lambda^{k}} \mathbf{\sigma}^{k} : \mathbf{\sigma}^{k}$$  \hfill (E.3)

with $\mathbf{e}$ and $\mathbf{\sigma}$ the strain and stress tensors and where $\mathbf{\sigma}^{k} = P^{k} : \mathbf{\sigma}$. For a given material symmetry, this equation defines in a unique and objective manner the Kelvin stress $\mathbf{\sigma}^{k}$ as the projection of the stress tensor on the $k^{th}$ Kelvin mode. If the same projection is made...
Kelvin spectral decomposition of elasticity tensor

for the strain, i.e. \( \mathbf{e}^k = \mathbb{P}^k : \mathbf{e} \), the elasticity law \( \mathbf{\sigma} = \mathbb{E} : \mathbf{e} \) is equivalent to \( \mathbf{\sigma}^k = \Lambda^k \mathbf{e}^k \) \( \forall k \) (no sum).

the Kelvin eigentensors are (in the material (cubic) Natural Anisotropy Basis)

\[
\begin{align*}
\mathbf{e}^{(1)} &= \begin{bmatrix}
\frac{1}{\sqrt{3}} & 0 & 0 \\
0 & \frac{1}{\sqrt{3}} & 0 \\
0 & 0 & \frac{1}{\sqrt{3}}
\end{bmatrix}_{NAB}, & \mathbf{e}^{(2)} &= \begin{bmatrix}
\frac{1}{\sqrt{2}} & 0 & 0 \\
0 & -\frac{1}{\sqrt{2}} & 0 \\
0 & 0 & \frac{1}{\sqrt{2}}
\end{bmatrix}_{NAB}, & \mathbf{e}^{(3)} &= \begin{bmatrix}
\frac{1}{\sqrt{6}} & 0 & 0 \\
0 & \frac{1}{\sqrt{6}} & 0 \\
0 & 0 & -\frac{2}{\sqrt{6}}
\end{bmatrix}_{NAB} \\
\mathbf{e}^{(4)} &= \begin{bmatrix}
0 & \frac{1}{\sqrt{2}} & 0 \\
\frac{1}{\sqrt{2}} & 0 & 0 \\
0 & 0 & 0
\end{bmatrix}_{NAB}, & \mathbf{e}^{(5)} &= \begin{bmatrix}
0 & 0 & 0 \\
0 & 0 & \frac{1}{\sqrt{2}} \\
0 & \frac{1}{\sqrt{2}} & 0
\end{bmatrix}_{NAB}, & \mathbf{e}^{(6)} &= \begin{bmatrix}
0 & 0 & \frac{1}{\sqrt{2}} \\
0 & 0 & 0 \\
\frac{1}{\sqrt{2}} & 0 & 0
\end{bmatrix}_{NAB}
\end{align*}
\]

They defines the three the projectors \( \mathbb{P}^k \) – renamed \( \mathbb{P}^H, \mathbb{P}^d, \mathbb{P}^\ddagger \) – in an objective and unique manner,

\[
\begin{align*}
\mathbb{P}^{k=1} &= \mathbb{P}^H = \mathbf{e}^{(1)} \otimes \mathbf{e}^{(1)} \\
\mathbb{P}^{k=2} &= \mathbb{P}^d = \mathbf{e}^{(2)} \otimes \mathbf{e}^{(2)} + \mathbf{e}^{(3)} \otimes \mathbf{e}^{(3)} \\
\mathbb{P}^{k=3} &= \mathbb{P}^\ddagger = \mathbf{e}^{(4)} \otimes \mathbf{e}^{(4)} + \mathbf{e}^{(5)} \otimes \mathbf{e}^{(5)} + \mathbf{e}^{(6)} \otimes \mathbf{e}^{(6)}
\end{align*}
\]

so that one has the strain partition \( \mathbf{\sigma} = \mathbf{\sigma}^H + \mathbf{\sigma}^d + \mathbf{\sigma}^\ddagger \) with Kelvin stresses

\[
\begin{align*}
\mathbf{\sigma}^H &= \mathbb{P}^H : \mathbf{\sigma} = \frac{1}{3} \text{tr} \mathbf{\sigma} \mathbf{1} & \mathbf{\sigma}^d &= \mathbb{P}^d : \mathbf{\sigma} & \mathbf{\sigma}^\ddagger &= \mathbb{P}^\ddagger : \mathbf{\sigma}
\end{align*}
\]

also defined in an objective and unique manner. In cubic Natural Anisotropy Basis, \( \mathbf{\sigma}^d = (\mathbf{\sigma}^d)' \) is diagonal deviatoric part of \( \mathbf{\sigma} \) and \( \mathbf{\sigma}^\ddagger = (\mathbf{\sigma}^\ddagger)' \) is out of diagonal deviatoric part of \( \mathbf{\sigma} \).

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
Appendix F

Normal forms of cubic elasticity and harmonic fourth order tensor

In Natural Anisotropy Basis (NAB) the compliance tensor $S = E^{-1}$ of a material having cubic material symmetry has for matrix representation

$$[E^{-1}] = \begin{pmatrix}
\frac{1}{E} & -\nu & -\nu & 0 & 0 & 0 \\
-\nu & \frac{1}{E} & -\nu & 0 & 0 & 0 \\
-\nu & -\nu & \frac{1}{E} & 0 & 0 & 0 \\
0 & 0 & 0 & \frac{1}{2G} & 0 & 0 \\
0 & 0 & 0 & 0 & \frac{1}{2G} & 0 \\
0 & 0 & 0 & 0 & 0 & \frac{1}{2G} \\
\end{pmatrix}_{NAB} \tag{F.1}$$

where $1/2G = 2S_{1212} = 1/2E_{112}$. The generalized Lamé constants of harmonic decomposition are

$$\begin{align*}
\lambda &= \frac{1}{5}(E_{1111} - 2E_{1212} + 4E_{1122}) \\
\mu &= \frac{1}{5}(E_{1111} + 3E_{1212} - E_{1122})
\end{align*} \tag{F.2}$$

The harmonic fourth order part $H$ carries both the third elasticity parameter and the Natural Anisotropy Basis (NAB) of cubic symmetry. It has for matrix representation, in Natural Anisotropy Basis still (Auffray et al., 2014)

$$[H] = \delta \begin{pmatrix}
8 & -4 & -4 & 0 & 0 & 0 \\
-4 & 8 & -4 & 0 & 0 & 0 \\
-4 & -4 & 8 & 0 & 0 & 0 \\
0 & 0 & 0 & -8 & 0 & 0 \\
0 & 0 & 0 & 0 & -8 & 0 \\
0 & 0 & 0 & 0 & 0 & -8
\end{pmatrix}_{NAB} \tag{F.3}$$

with

$$\delta = \frac{1}{20}(E_{1111} - 2E_{1212} - E_{1122}) \tag{F.4}$$

Components $E_{ijkl}$ are given in Eq 3.3.1.
Normal forms of cubic elasticity and harmonic fourth order tensor

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
Appendix G

Non quadratic norm in criterion function $f_2$

If non quadratic norm is used within second criterion function of (3.61), setting

$$f_2 = \|\sigma^T\|_a - \mathcal{R}_\infty - \sigma \frac{k_{iso} G}{\|\omega\|} - \sigma_y$$

(G.1)

we still have have $\dot{\varepsilon}^p = p_1 \frac{\partial f_1}{\partial \sigma} + p_2 \frac{\partial f_2}{\partial \sigma}$ but with

$$p_2^T = 2 \left( \frac{1}{6} \sum_{i \neq j} |\varepsilon_{ij}|^{\frac{a-1}{a}} \right)^{\frac{a}{a-1}}$$

(G.2)

and

$$\sigma : \dot{\varepsilon}^p = \sigma_{inl} p_{inl}^T + \|\sigma^T\|_a p_2^T$$

(G.3)

Recall that $\|\sigma^T\|_a = 3 \left( \frac{1}{6} \sum_{i \neq j} |\sigma_{ij}|^{a} \right)^{\frac{1}{a}}$.

A value $a \in [1,2]$ allows to model a non homogeneous plastic strain field in torsion (section 3.10.1), as observed experimentally Nouailhas and Cailletaud (1995). The uni-axial model response remains the same as the one of simpler case $a = 2$, $\|\sigma^T\|_2 = \sigma_{eq}^T$ for applied stresses in orientation $<001>$ and in orientation $<111>$.
Non quadratic norm in criterion function $f_2$
Appendix H

Heterogeneous field in torsion

Consider here the thin tube of longitudinal axis in [001] (= e_z = e_3) orientation tested in torsion in Nouailhas and Cailletaud (1995). Unit vectors e_k of cartesian basis are set parallel to each <001> directions, defining Natural Anisotropy Basis of present cubic material. Radial and tangential unit vectors are e_r = \cos \theta e_1 + \sin \theta e_2, e_\theta = -\sin \theta e_1 + \cos \theta e_2.

At homogeneous shear strain \gamma = 2\epsilon_\theta; applied in torsion on the tube the non zero stress components are \sigma_{13} = \sigma_{31} = G\gamma \cos \theta, \sigma_{12} = \sigma_{21} = -G\gamma \sin \theta so that diagonal deviatoric stress tensor \sigma^d = 0 and out of diagonal deviatoric stress tensor \sigma^d = \sigma. Non quadratic norm \|\sigma^d\|_a introduced in section 3.3.6 is at the elasticity limit

\[ \|\sigma^d\|_a = 3^{1-\frac{1}{a}}G\gamma(\cos \theta)^a + (\sin \theta)^a \frac{1}{a} \geq 1 \quad (H.1) \]

It is constant for a = 2 (homogeneous von Mises stress field in torsion at \sigma^d_{eq} = \sqrt{3}G\gamma = const), it is heterogeneous for a \neq 2 with

- for a > 2: the maximum value of the yield criterion \bar{f} is reached when radial basis vector e_r is aligned with <100> orientation (at angle \theta = k\frac{\pi}{2}, k integer), the minimum value of \bar{f} is reached when radial basis vector e_r is in direction <110> (at angle \theta = \frac{\pi}{4} + k\frac{\pi}{2} from <100> orientation).

- for 1 \leq a < 2: the location of minima and maxima of \bar{f} are inverted, the minimum value being reached at \theta = k\frac{\pi}{2}, the maximum value at angle \theta = \frac{\pi}{4} + k\frac{\pi}{2} from <001>: yielding will initiate for the <110> orientations, at \frac{\pi}{4} from <100> orientations.

The second case is the one observed in experiments (Nouailhas and Cailletaud, 1995) so that a value of material parameter a \in [1,2] has to be considered (a simple choice being a = 1).

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Appendix I

Thermodynamics framework

I.1 Thermodynamics of rafting

For nickel-based single crystal superalloys at high temperature, the decrease in mechanical properties associated with the γ' rafting mainly results from the widening of γ channels where the plastic deformation concentrates (Pollock and Argon, 1994). Indeed, during creep at high temperature and low stress, typically in between 1000 and 1200 °C, depending on the alloy chemistry, the end of the primary creep stage corresponds to γ' completion. The decreasing creep strain rate during this primary creep stage directly results from an easier dislocation mobility in the γ channels which can be evaluated by the Orowan Stress, defined as in the :

\[ \sigma_{\text{ORO}} \propto \frac{GB}{w_{001}} \]  

(I.1)

with \( B \) the Burgers vector magnitude (= 0.254 nm), and \( w_{001} \) the γ channel width along a \( <001> \) direction (i.e in a (100) or a (010) plane).

Since dislocations under high temperature conditions are mainly gliding in the matrix on octahedral slip systems, a correction factor \( \theta \) is considered for Orowan stress at (octahedral) slip systems scale (see equation (3.1)), with for instance \( \theta = \sqrt{2/3} \) obtained when calculating the Orowan stress using γ channels width along the [001] direction (Benyoucef et al, 1993). The value of \( \theta \) was assumed to be 0.5 in Fedelich et al (2009) and 0.85 in Tinga et al (2009b), i.e a value close to the \( \sqrt{2/3} \) considered in Cormier and Cailletaud (2010a).

I.1.1 Dislocation bypassing energy density

We propose to derive a thermodynamics framework for rafting directly at the RVE scale, without the use at this stage of the single crystal plasticity framework. We nevertheless need to make the conceptual difference between plasticity due to the octahedral slip systems, which are classically affected by Orowan stress, and plasticity due to the pseudo-cubic slip systems, less affected by Orowan stress. In order to do so at RVE scale,
we use Kelvin modes based definitions of section 3.3 and introduce two accumulated plastic strains representative of $<001>$ slip systems ($p^d$) and of $<111>$ slip systems ($p^\vec{d}$) (Desmorat and Marull, 2011). Both accumulated plastic strains $p^d$ and $p^\vec{d}$ are mean values over the RVE. Corresponding "deviatoric diagonal" $\varepsilon^{bd}$ and "deviatoric out of diagonal" $\varepsilon^{d\vec{d}}$ Kelvin modes based plastic strains are close to octahedral $\varepsilon^{po}$ and cubic $\varepsilon^{pc}$ plastic strains of single crystal plastic framework (see section 3.9). Assuming Orowan stress behaves as isotropic hardening and using a mixture law, the corresponding plastic work reads, with $\rho$ the density,

$$
\rho \psi_{oro} = f_{\gamma} \sigma_{oro} p^d = (1 - f_{\gamma'}) \sigma_{oro} p^d \propto (1 - f_{\gamma'}) \frac{GB}{w_{001}} p^d
$$

(I.2)

generalized into dislocation bypassing energy density,

$$
\rho \psi_{oro} = \kappa_{oro} g(f_{\gamma'}) \frac{w_{001}}{||w||} \left( p^d + \sigma p^\vec{d} \right) \frac{dg}{df_{\gamma'}} < 0
$$

(I.3)

where mean anisotropic Orowan effect over the RVE is simply introduced through quadratic norm $||w|| = (\text{tr} w^2)^{1/2}$ of $\gamma$ width channel tensor $w$, phenomenological function $g(f_{\gamma'})$ of $\gamma'$ precipitates volume fraction, of parameter $\kappa_{oro}$. By means of material constant $\Theta$, one has introduced in Eq. (I.3) a macroscopic second order contribution of the accumulated plastic strain $p^\vec{d}$ representative of $<111>$ material response (Eq. 3.19). The value $\Theta \approx 0.8–1$ will be obtained in section 3.7.4. Quantity $w_{001} \approx 50$ nm is the initial (room temperature) mean value of the $\gamma$ channels width, the corresponding initial value for $\gamma'$ precipitates volume fraction is $f_{\gamma'}^0$. Material parameter $\kappa_{oro} = \kappa_{oro}(T)$ is temperature dependent.

The function $g(f_{\gamma'})$ is a decreasing function of the precipitates volume fraction and it is normed such as $g(f_{\gamma'}^0) = 1$. Two possible expressions are given here, first one corresponding to (linear) mixture law, second one the power law used (and measured) in the Polystar model (Cormier and Cailletaud, 2010a),

$$
g(f_{\gamma'}) = \frac{1 - f_{\gamma'}}{1 - f_{\gamma'}^0} \quad \text{or} \quad g(f_{\gamma'}) = \left( \frac{f_{\gamma'}^0}{f_{\gamma'}} \right)^{m_{\gamma'}}
$$

(I.4)

with the properties $g(f_{\gamma'}^0) = 1$ and $\frac{dg}{df_{\gamma'}} < 0$

An important remark is that as the precipitate volume fraction changes, the rafting and the coarsening all act in energy density (I.3) as single (tensorial) dimensionless mechanical rafting variable

$$
\omega = \frac{w}{g(f_{\gamma'}) w_{001}} \quad \text{or} \quad \omega_{ij} = \frac{w_{ij}}{g(f_{\gamma'}) w_{001}^0}.
$$

(I.5)

Thus, in the same spirit than for rafting (scalar) variable $\xi$ in Fedelich and coworkers constitutive models, one proposes next to give to dimensionless (tensorial) variable $\omega$ the status of single thermodynamics state variable for microstructure degradation, possibly

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anisotropic. In this way the dislocation bypassing energy density at RVE scale can be rewritten as

$$\rho \psi_{\text{oro}} = \kappa \psi_{\text{oro}} \frac{G}{\|\gamma\|} (p^d + \gamma p^d)$$ \hspace{1cm} (I.6)

with the physical $\gamma$ channels widths later determined as

$$w = g(f') w^0_{001} \omega \quad \text{or} \quad w_{ij} = g(f') w^0_{001} \omega_{ij}$$ \hspace{1cm} (I.7)

which is nothing else than a tensorial extension (with second order tensors $w$, $\omega$ of component $w_{ij}$, $\omega_{ij}$) of initial (scalar) Polystar equation for tension along [001] (Cornier and Cailletaud, 2010a) (presented in the (3.49)) constitutive equation shown to apply to thermo-mechanical loading at $\dot{T} \neq 0, f' \neq 0$. Dimensionless scalar variable $\omega_{\text{Polystar}} = \omega_{\text{mech}} + \omega_{\text{LSW}}$ is the sum of a mechanical rafting/coarsening contribution $\omega_{\text{mech}}$, stress and/or plastic strain dependent, and of the diffusion term $\omega_{\text{LSW}}$ consistent with Eq. (3.3).

I.1.2 Coupling elasticity-rafting

Pineau (1976) by using a micro-mechanics approach and more recently Tinga et al (2009b) by using unit cell Finite Element computations have shown that the strain energy during a creep test decreases as yielding and rafting proceed (see also (Fan et al, 2015)). As there is a lack of experimental evidences on the rafting-elasticity coupling, one can simply assume that elasticity remains cubic and sets

$$\rho \psi_e = \frac{1}{2} (\varepsilon - \varepsilon') : \mathbb{E} (1 - \kappa E \omega_{eq}) : (\varepsilon - \varepsilon') = \frac{1}{2} \varepsilon^e : \mathbb{E} (1 - \kappa E \omega_{eq}) : \varepsilon^e$$ \hspace{1cm} (I.8)

where $\varepsilon^e$ is elastic strain tensor and where one has introduced the elasticity-rafting coupling parameter $\kappa E$. No effect is considered when coarsening ($\omega_{eq} = \sqrt{\frac{3}{2} \|\omega'\|}$ is the norm of deviatoric part $\omega' = \omega - \frac{1}{3} \text{tr} \omega 1$), if needed replace $\omega_{eq}$ by $\|\omega\|$. The thermodynamics framework derived next will consider such a coupling (in order to recover the energetic contribution to rafting driving force derived in Fan et al (2015)) but the final model proposed in section 3.5 will neglect it ($\kappa E \omega_{eq} << 1$, Eq. (3.59)).

Thermo-mechanical terms are omitted in Eq. (I.8). Considering the unconstrained misfit $\delta_u$, as a difference in reference temperature for $\gamma$ and for $\gamma'$ phases allows to define a thermo-elastic misfit contribution $-3 f' K' \delta_u \text{tr} \varepsilon = 3 f' K' \text{tr} \delta_u |\delta_u| \text{tr} \sigma$ (see J), proportional to both the absolute value of the misfit $|\delta_u|$ and to the stress (as in Nabarro et al (1995), Fedelich (2002)). Following Fedelich et al (2006, 2009) for high temperature visco-plastic processes, such a contribution will also be neglected, this time compared to next misfit energy $\rho \psi_u$.

---

1. negative for considered CMSX-4 superalloy
I.1.3 Misfit energy density $\rho \psi_u$

The misfit energy density $\rho \psi_u = \frac{1}{2} (\kappa_u - \kappa_\omega \omega_{eq}) \delta_u^2$ (Fedelich et al., 2006), ($\xi$ is defined in the equation 3.37)), with the –here negative– unconstrained mistfit, has the following properties: $\rho \psi_u$ remains constant during coarsening (as then $\xi = 0$), it decreases when rafting takes place (as then $\xi \rightarrow 1$). It is worth remarking that the dependency of material parameters $\kappa_u$ and $\kappa_\xi$ with respect to $f'_\gamma$ has not been exhibited in pioneering works. In most models (Fedelich et al., 2006, Tinga et al., 2009b, Fedelich et al., 2009, 2012a, Fan et al., 2015), thermodynamics equilibrium is assumed with biunivoque (instantaneous) relationship between $f'_\gamma$ and temperature $T$ so that the dependencies with respect to $f'_\gamma$ is hidden into temperature dependency $\kappa_u = \kappa_u(T)$, $\kappa_\xi = \kappa_\xi(T)$.

A first possibility for tensorial rafting framework is to rewrite $\rho \psi_u$ as a function of the 3D dimensionless index of rafting $x_{raft}$ defined in Eq. (3.57) (also such as $x_{raft} = 0$ when coarsening and $x_{raft} \rightarrow 1$ when rafting). Thermodynamics will not allow us to do so (impossibility to prove in any case the positivity of the intrinsic dissipation).

Instead we propose to simply use the remark that one has a zero deviatoric part $w' = \omega' = 0$ when coarsening and write misfit energy density as

$$\rho \psi_u = \frac{1}{2} (\kappa_u - \kappa_\omega \omega_{eq}) \delta_u^2 \quad \omega_{eq} = \sqrt{\frac{3}{2}} \|\omega'\|$$  \hspace{1cm} (I.9)

with $\kappa_u$ and $\kappa_\omega$ as temperature dependent material parameters. Misfit energy is expressed as a function of deviatoric part $\omega'$ of mechanical rafting variable and of temperature; therefore it remains constant during a pure isotropic coarsening process and it decreases during the gamma-prime rafting process, as wanted. Compared to Fedelich’s expression it is now dependent on the intensity of rafting (the $\gamma$-channels widths). Note that at thermodynamics equilibrium, one has $f'_\gamma = f'_{\gamma eq}(T)$ so that Eq. (I.9) becomes equivalent to alternative formulation $\rho \psi_u = \frac{1}{2} (\kappa_u - \kappa_\omega w_{eq}) \delta_u^2$ with an adequate definition of temperature dependency of parameters $\kappa_u = \kappa_u(T)$, $\kappa_\omega = \kappa_\omega(T)$, $\kappa_w = \kappa_w(T)$. Remark that present parameter $\kappa_\omega$ has the same dimension but differs in value from $\kappa_\xi$ parameter introduced by Fedelich and coworkers.

I.1.4 Thermodynamics potential

The thermodynamics potential, here Helmholtz free energy density $\rho \psi$, is function of the total strain $\varepsilon$, of the temperature $T$ and of internal variables $\Psi_K$. The thermodynamics forces are (Lemaitre and Chaboche, 1991)

$$\sigma = \rho \frac{\partial \psi}{\partial \varepsilon} \quad s = -\frac{\partial \psi}{\partial T} \quad \mathcal{A}_K = \rho \frac{\partial \psi}{\partial \Psi_K}$$  \hspace{1cm} (I.10)

with s the specific entropy and $\mathcal{A}_K$ the associated variables to internal variables $\Psi_K$.

The intrinsic dissipation is then (“•” meaning multiplication for scalar variables, scalar product “:” for tensorial variables)

$$\mathcal{D} = -\sum_K \mathcal{A}_K \cdot \Psi_K$$  \hspace{1cm} (I.11)
Thermodynamics of rafting

and is positive when the second principle of thermodynamics is satisfied.

I.1.4.1 \( w \) and \( f' \gamma \) as state variables

The question is then the proper definition of the internal variables for the considered material and applications. Let us follow \textit{a priori} choice made in section 3.4.4, and first consider as internal variables

- macroscopic plastic strain \( \varepsilon^p \) (associated with \(-\sigma\)) instead of the plastic slips over each slip system; note that crystal plasticity constitutive equations will be derived in section 3.9,
- macroscopic isotropic hardening variables \( p^d \) (associated force: isotropic hardening \( R \)) for octahedral-like slip systems and \( p^\overline{d} \) (associated force: isotropic hardening \( \overline{R} \)) for cubic-like slip systems,
- tensorial rafting variable \( w \) (associated force: \( W \)),
- \( \gamma' \) precipitates volume fraction \( f' \gamma \) (associated force: \(-F' \gamma\)).

The state laws are

\[
\sigma = \rho \frac{\partial \psi}{\partial \varepsilon} = -\rho \frac{\partial \psi}{\partial \varepsilon^p} \quad R = \rho \frac{\partial \psi}{\partial p^d} \quad \overline{R} = \rho \frac{\partial \psi}{\partial p^\overline{d}} \quad W = -\rho \frac{\partial \psi}{\partial w} \quad F' \gamma = -\rho \frac{\partial \psi}{\partial f' \gamma}
\]  

(I.12)

The intrinsic dissipation is therefore the sum of a plastic contribution (Lemaitre and Chaboche, 1991, Desmorat and Marull, 2011) and of a rafting contribution (which includes \( \gamma' \) precipitates evolution):

\[
D = D_{\text{plast}} + D_{\text{raft}} \quad \left\{ \begin{array}{l}
D_{\text{plast}} = \sigma : \dot{\varepsilon}^p - R \dot{p}^d - \overline{R} \dot{p}^\overline{d} \\
D_{\text{raft}} = W : \dot{w} + F' \gamma \dot{f' \gamma}
\end{array} \right.
\]  

(I.13)

The dissipation \( D_{\text{plast}} \) is always positive if criterion functions \( f_1 = \sigma_{eq}^d - R - \sigma_y \), \( f_2 = \sigma_{eq}^\overline{d} - \overline{R} - \sigma_y \) – including Orowan contributions – are considered altogether with standard visco-plasticity loading/unloading conditions (Eq. (3.30), as one has then

\[
D_{\text{plast}} = (\sigma_{eq}^d - R) \dot{p}^d + (\sigma_{eq}^\overline{d} - \overline{R}) \dot{p}^\overline{d} \geq 0
\]  

(I.14)

as \( \sigma' = \sigma^d + \sigma^\overline{d} \), and from Eq. (3.29), \( \sigma^d : n^d = \sigma_{eq}^d \dot{p}^d \) and \( \sigma^\overline{d} : n^\overline{d} = \sigma_{eq}^\overline{d} \dot{p}^\overline{d} \). Presented modeling corresponds to the case of a vanishing Hill parameter \( h \). The case \( h \neq 0 \), \textit{i.e.} of a Hill criterion function \( f_1 = \sigma_{\text{Hill}} - R - \sigma_y \), is simply obtained by replacing \( \sigma_{eq}^d \) by \( \sigma_{\text{Hill}} \) and \( (p^d, p^\overline{d}) \) by \( (p_{\text{Hill}}^d, p_{\text{Hill}}^\overline{d}) \) (see section 3.3.7).

Concerning the dissipation due to microstructure degradation \( D_{\text{raft}} \), the thermodynamics does not give specific limitations (one only has to enforce \( D \geq 0 \)).

But note that at thermodynamics non equilibrium, the \( \gamma' \) precipitates volume fraction –measured as well as modeled within initial Polystar model– may either increase and decrease (Cormier and Cailletaud, 2010a), depending on the temperature rate history. It is indeed not possible to write \( f' \gamma = f' \gamma_{eq}(T) \) at any time of a thermo-mechanical loading

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since out of equilibrium states generating fine precipitates are possible in cases of fast heating and fast cooling rates. On heating, fine and coarse precipitates are dissolving, the dissolution kinetics being higher for a fine precipitate. On cooling, both fine and coarse precipitates volume fractions increase at different kinetics, depending on the cooling rate and on the temperature variation from the high temperature state (depending on the driving force for fine $\gamma'$ nucleation in the $\gamma$ channels) (Cormier and Cailletaud, 2010a, Giraud et al, 2013b, le Graverend et al, 2014a). This fact makes impossible to satisfy separately, i.e. mechanism by mechanism / internal variable by internal variable, the positivity of each intrinsic dissipations $W : \dot{w}$ and $F_{\gamma'} \dot{f}_{\gamma'}$. This means that from a thermodynamics point of view, the evolution (rate) laws $\dot{w} = \ldots$ and $\dot{f}_{\gamma'} = \ldots$ for internal variables $w$, $f_{\gamma'}$ are coupled in a quite complex manner in order to always satisfy the positivity of the dissipation.

From the remarks

i) that the $\gamma$ width channel may increase and decrease in a reversible way by slow (at equilibrium) temperature variation,

ii) according to i), that there is no evolution law of the form $\dot{w} = \ldots$ in Polystar constitutive equations handling out of thermodynamics equilibrium states but rate independent equation (3.49),

iii) that the contribution of all rafting variables can be included in Helmholtz free energy density within single variable $\omega$,

we propose in next section an alternative thermodynamics framework based on single tensorial mechanical rafting variable $\omega$.

I.1.4.2 Tensorial mechanical rafting variable $\omega$ as state variable

Helmholtz free energy density is assumed to be a function of total strain $\varepsilon$, of temperature $T$, of macroscopic plastic strain $\varepsilon^p$, of the two macroscopic isotropic hardening variables $p^d$ and $p^\overline{d}$ and of tensorial mechanical rafting variable $\omega$,

$$
\rho \Psi = \frac{1}{2} (\varepsilon - \varepsilon^p) : \varepsilon + (1 - \kappa_E \omega_{eq} \varepsilon^p) + \left( R_\infty + \frac{\kappa_{ORD} G}{\|\omega\|} \right) p^d
$$

$$
+ \left( R_\infty + \frac{\kappa_{ORD} G}{\|\omega\|} \right) p^\overline{d} + \frac{1}{2} \left( \kappa_u - \kappa_{\omega_0} \omega_{eq} \right) \delta u^2
$$

(I.15)

with $\varepsilon$ Hooke’s tensor having cubic elasticity symmetry (material parameters $E$, $\nu$ and $G$) and where $\omega_{eq} = \sqrt{\frac{3}{2}} \|\omega\|$ is von Mises norm of symmetric second order tensor $\omega$. The rafting-elasticity coupling parameter $\kappa_E$ is introduced but have in mind that the term $\kappa_E \omega_{eq}$ will ne neglected.

For the sake of –relative– simplicity the mechanical parts of isotropic hardenings are assumed saturated through material parameters $R_\infty$, $R_\overline{\infty}$ (if not, replace them by functions of $p^d$ and $p^\overline{d}$), the thermal expansion and heat capacity contributions have been omitted.
The state laws are
\[ \sigma = \rho \frac{\partial \psi}{\partial \epsilon} = E (1 - \kappa_E \omega_q) : \epsilon^e \quad R = \rho \frac{\partial \psi}{\partial \rho^d} = R_\infty + \kappa_{\text{OD}} G \]

with elastic strain \( \epsilon^e = \epsilon - \epsilon^p = \epsilon - \epsilon^{pd} - \epsilon^{rd} \), and for rafting:
\[ \Omega = -\rho \frac{\partial \psi}{\partial \omega} = \kappa_{\text{OD}} \frac{G}{||\omega||} \omega \left( \rho^d + \sigma \rho^d \right) + \sqrt{\frac{3}{8} \left[ \kappa_E \epsilon^e : \epsilon^e + \kappa_\omega \delta_{ij} \right]} \omega' \]

\( -\Omega \) is the thermodynamics force associated with the internal variable \( \omega \).

The dissipation due to rafting, previous Eq. (I.13), becomes
\[ D_{\text{raft}} = \Omega : \dot{\omega} \]
a simple expression which includes \( \gamma' \) precipitates contribution.

Dissipation \( D_{\text{raft}} \) has to be positive for any loading, isothermal or not, proportional or not.

### 1.2 Positive intrinsic dissipation from generic tensorial rafting evolution law

One aims at modeling rafting, coarsening and homothetic \( \gamma \) channels width growth from a tensorial evolution law \( \dot{\omega} = \ldots \). The question is whether it is possible to propose a generic expression that always satisfies the second principle of thermodynamics. A first possibility is to use the framework of standard generalized materials (Halphen and Nguyen, 1975, Lemaitre and Chaboche, 1991) and to write \( \dot{\omega} = \frac{\partial F}{\partial \Omega} \), with potential of dissipation \( F \) convex in \( \Omega \). This proves to be not general enough. For instance Tinga type evolution law (3.38) does not satisfy such a standard framework (see D).

A more general non standard evolution law is derived next, written in terms of dimensionless variable \( \omega \).

#### 1.2.1 LSW homothetic growth (hg), loading independent

The initial microstructure is represented by a spherical \( \gamma \) channel width tensor \( \omega = w_{001} \) (i.e. \( w_{ij} = w_{001} \delta_{ij} \) in terms of components) with \( w_{001} \approx 50 \text{ nm} \) at room temperature for the considered CMSX-4. At zero load, the cuboidal morphology of the microstructure is kept but temperature enhanced diffusion controlled coarsening \( \dot{w}_1 \approx \dot{w}_2 \approx \dot{w}_3 > 0 \) takes place (standard LSW theory Lifshitz and Slyozov (1961), Wagner (1961), see Section 3.2): the channels width tensor evolves in a homothetic growth \( \dot{\omega} \propto 1 \) (i.e. \( \dot{w}_{ij} = \delta_{ij} \)).

After a first mechanical loading, having induced creep deformation, a continuation of anisotropic \( \gamma \) channel widths growth has been observed at load removal (Matan et al, 1999a): if rafting has taken place during an initial creep loading performed in direction
1 ≡ [001] and if a large enough plastic strain has been reached, the rafting phenomenon continues. This means in terms of widths evolutions rates that \( \dot{w}_1 \geq 0 \), \( \dot{w}_2 \approx \dot{w}_3 < 0 \) and \( w_2 \approx w_3 \rightarrow 0 \) at long times, evolutions that cannot be described from a law of the spherical type \( \dot{\omega} \approx 1 \).

In order to properly model both standard LSW coarsening and Matan et al rafting continuation phenomenon at load removal, we prefer –instead of \( \dot{\omega} \approx 1 \)– to define homothetic growth as a \( \gamma \) channel width growth proportional to second order \( \gamma \) channel width tensor \( w \) as

\[
\dot{\omega}_{\text{hg}} = \dot{G} \frac{w}{\|w\|} = \dot{G} \frac{\omega}{\|\omega\|} \quad \dot{G} \geq 0, \quad \frac{\partial \dot{G}}{\partial \sigma} = 0, \quad \frac{\partial \dot{G}}{\partial \epsilon_p} = 0, \quad \frac{\partial \dot{G}}{\partial \dot{\epsilon}_p} = 0 \quad (I.19)
\]

where homothetic growth function \( \dot{G} \) is furthermore assumed to be independent from the intensity of the mechanical loading. Recall is made here that \( \gamma \) channel width tensor \( w \) is itself proportional to dimensionless tensor \( \omega \). The positivity of \( \dot{G} \) will be justified next. Law (I.19) does not ensure negative \( \dot{\omega}_2 \) and \( \dot{\omega}_3 \) by itself but it has the sought asymptotic property \( \dot{w}_2 \approx w_3 \rightarrow 0 \) at long times. Additional coarsening/rafting contributions (see Sections I.2.2 and I.2.3) are necessary to enforce the observed feature \( \dot{w}_1 \geq 0 \), \( \dot{w}_2 \approx \dot{w}_3 < 0 \), and \( w_2 \approx w_3 \rightarrow 0 \) at long times, both during tensile loading and at load removal.

### I.2.2 Mechanical coarsening (mc)

We define mechanical coarsening as the spherical growth of the \( \gamma \) channel width due to mechanical loading,

\[
\dot{\omega}_{\text{mc}} = \dot{C} \mathbf{1} \quad \dot{C} \geq 0 \quad (I.20)
\]

i.e. the loading enhanced contribution of the \( \gamma \) channel width growth proportional to second order unit tensor \( \mathbf{1} \), with \( \dot{C} \) a function of loading intensity through the stress or and the plastic strain. In a consistent manner with the observations made in Matan et al (1999a), a plastic strain threshold for rafting will be introduced, enforcing \( \dot{C} = 0 \) as long as accumulated plastic strain remains below a threshold (as long as \( \epsilon_p < \epsilon_p^{\text{th}} \) with \( \epsilon_p^{\text{th}} \) a material parameter, Heaviside term \( H(\rho_2^d - \epsilon_p^{\text{th}}) \) in Eq. (3.72).

### I.2.3 Rafting (raft)

When rafting is due to a tensile stress in direction \( 1 \equiv [001] \), one has in Natural Anisotropy Basis (NAB)

\[
w = \begin{bmatrix} w_1 & 0 & 0 \\ 0 & w_2 & 0 \\ 0 & 0 & w_3 \approx w_2 \end{bmatrix} = g(f_{\gamma}) w_{[001]} \omega \quad (I.21)
\]

and the longitudinal \( \gamma \) channel width \( w_1 \) grows, the transverse channels widths equally shrinks to zero (\( \dot{w}_1 \geq 0 \) and \( w_2 \approx w_3 \rightarrow 0 \)). Rafting leads then to a non spherical tensorial...
rafting variable \( \omega \) (therefore \( w \)) having a non zero deviatoric part \( \omega' = \omega - \frac{1}{3} \text{tr} \omega \mathbf{1} \neq 0 \) of the form (using equality \( w_2 = w_3 \))
\[
\begin{bmatrix}
1 & 0 & 0 \\
0 & -\frac{1}{2} & 0 \\
0 & 0 & -\frac{1}{2}
\end{bmatrix}
\]

\[
\propto \dot{\mathbf{e}}^{pd}
\]

\( \text{(I.22)} \)

\( \text{i.e. proportional to plastic strain rate tensor } \dot{\mathbf{e}}^{p} = \dot{\mathbf{e}}^{pd} \text{ as in present uniaxial tension case} \)
\[
\dot{\mathbf{e}}^{p} = \dot{\rho} \mathbf{n} = \dot{\rho}^{d} \mathbf{n}^{d} \quad \mathbf{n} = \mathbf{n}^{d} = 
\begin{bmatrix}
1 & 0 & 0 \\
0 & -\frac{1}{2} & 0 \\
0 & 0 & -\frac{1}{2}
\end{bmatrix}
\]

\( \text{NAB} \)

\( \dot{\mathbf{e}}^{pd} = 0 \) (I.23)

One emphasizes in Eq. (I.23) that out of diagonal part in \textit{Natural Anisotropy Basis} of strain rate tensor is zero, \( \text{i.e. using the notations of Kelvin mode based plasticity (see Section 3.3)} \)
\[
\dot{\mathbf{e}}^{pd} = \dot{\mathbf{e}}^{pd} = \mathbf{P}^{d} : \dot{\mathbf{e}}^{p} = 0 \quad \dot{\rho} = \dot{\rho}^{d}, \dot{\rho}^{d} = 0 \text{ as is present case yielding is due only to octahedral slip systems.} \]

In a complementary manner, an uniaxial tensile stress is applied along \( <111> \) direction gives
\[
\begin{bmatrix}
1 & 1 & 1 \\
1 & 1 & 1 \\
1 & 1 & 1
\end{bmatrix}
\]

\( \text{NAB} \)

\( \sigma = \frac{1}{3} \dot{\mathbf{e}}^{pd} \propto \begin{bmatrix}
0 & 1 & 1 \\
1 & 0 & 1 \\
1 & 1 & 0
\end{bmatrix}
\]

\( \text{NAB} \)

and experimentally only leads to coarsening (Golubovskii et al, 1987) at \( \omega' = 0 \). This means that cubic plasticity (through \( \dot{\mathbf{e}}^{pd} = \dot{\rho}^{d} \mathbf{n}^{d} \)) does not contribute to rafting.

A quite generic multiaxial evolution law for deviatoric part \( \omega' \) is then
\[
\dot{\omega}' \propto \dot{\mathbf{e}}^{pd} \propto \mathbf{n}^{d}
\]

\( \text{(I.25)} \)

The question of the time dependency or independency of rafting evolution law is not addressed here so that at this stage one simply writes \( \dot{\omega}' = \dot{\mathcal{R}} \mathbf{n}^{d} \). The question of the sign of the proportionality factor \( \dot{\mathcal{R}} \) (for rafting) needs an answer. Two possibilities, addressed in more details in Section M are:

---

- In \textit{modeling A} we enforce a positive dissipation due to rafting by itself \( \left( \mathcal{D}^{\text{raft}} \geq 0, \text{as } \mathcal{D}^{\text{plast}} \geq 0 \text{ this implies } \mathcal{D} \geq 0 \right) \),

- In \textit{modeling B}, we allow the possibility to \( \mathcal{D}^{\text{raft}} \) to become negative but to enforce a positive global dissipation \( \mathcal{D} \geq 0 \).

Next proof (given in Section I.2.5) of the positivity of intrinsic dissipation for \textit{modeling A} will use a positive proportionality factor \( \left( \text{i.e. } \omega' = \dot{\mathcal{R}} \mathbf{n}^{d}, \dot{\mathcal{R}} \geq 0 \right) \) at positive scalar product \( w : \mathbf{n}^{d} \geq 0 \) equivalent to \( \omega : \mathbf{n}^{d} = \omega_{ij} n^{d}_{ij} \geq 0 \); it will use a vanishing deviatoric rate \( \omega' = 0 \) \( \left( \text{i.e. } \dot{\mathcal{R}} = 0 \right) \) at strictly negative \( \omega : \mathbf{n}^{d} < 0 \) so that we propose to write
\[
\dot{\omega}^{\text{raft}} = (\dot{\omega}^{\text{raft}})' = \dot{\mathcal{R}} \mathcal{H} \mathbf{n}^{d} \quad \dot{\mathcal{R}} \geq 0
\]

\( \text{(I.26)} \)

where

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— for modeling A

\[ \mathcal{H} = H(\omega : n^d) \quad \text{or} \quad \begin{cases} \mathcal{H} = 1 \text{ if } \omega : n^d \geq 0, \\ \mathcal{H} = 0 \text{ if } \omega : n^d < 0 \end{cases} \quad (I.27) \]

— for modeling B the Heaviside function applies to intrinsic dissipation \( D \) itself,

\[ \mathcal{H} = H(D) \quad \text{or} \quad \begin{cases} \mathcal{H} = 1 \text{ if } D \geq 0, \\ \mathcal{H} = 0 \text{ if } D < 0 \end{cases} \quad (I.28) \]

For compression in direction 1 \( \equiv [001] \)

\[ n = n^d = \begin{bmatrix} -1 & 0 & 0 \\ 0 & \frac{1}{2} & 0 \\ 0 & 0 & \frac{1}{2} \end{bmatrix} \quad \text{NAB} \quad (I.29) \]

so that \( \dot{\gamma} > 0 \) implies \( \omega_1 < 0, \omega_2 = \omega_3 > 0 \) as observed in the work of Fredholm and Strudel (1987) (Fig. 1.15, P-type coalescence in compressive loading).

Expression (I.26) is consistent with the observations: rafting during uniaxial compression creep tests (along \( <001> \)) is developing in the form of rods and/or plates almost aligned with the stress axis, the so-called P-type rafting (Tien and Copley, 1971, Ott and Mughrabi, 1999, Giraud et al, 2012) described in the Introduction. Once the uniaxial loading changes to a tensile stress, the former P-type is progressively broken to develop a \( \gamma' \) rafting (Xingfu et al, 2009).

Note again that a threshold \( \varepsilon_{\text{th}}^{p} \) in term of accumulated plastic strain has been observed Matan et al (1999a) so that one will later enforce \( \dot{\gamma} = 0 \) as long as \( \varepsilon^{p} < \varepsilon_{\text{th}}^{p} \) by means of multiplication by Heaviside term \( H(p_1^{\text{Hill}} - \varepsilon_{\text{th}}^{p}) \) in Eq. (3.71).

I.2.4 Generic evolution law for tensorial internal variable \( \omega \)

To sum up a generic evolution law for rafting, coarsening and homothetic \( \gamma \) channel width growth, is

\[ \dot{\omega} = \dot{\gamma} \mathcal{H} n^d + \dot{\gamma} \mathcal{I} + \dot{\gamma} \frac{\omega}{\|\omega\|} \quad \text{w} = g(\gamma') w_{001}^{0} \quad (I.30) \]

where normal \( n^d = \text{dir}(\mathbf{\varepsilon}^{\text{nd}}) = \mathbf{\varepsilon}^{\text{nd}} / p^d \) has to be seen in single crystal plasticity models as the macroscopic (mean) plastic strain direction due to active octahedral slip systems only (not the cubic ones). Evolution law (I.30) is consistent with Tinga et al (2009b) evolution law (as shown in the Appendix D). It applies to nickel-based single crystal superalloys having a negative misfit \( \delta_u \).

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I.2.5 Dissipation due to rafting – Positivity of intrinsic dissipation

Using state law (I.17), and generic evolution law (I.30) the intrinsic dissipation due to rafting becomes

\[ D_{\text{raft}} = \left( \kappa_{\text{oro}} \frac{G}{\|\omega\|_2} \frac{\omega}{\|\omega\|} (p^d + \sigma p^t) + \sqrt{\frac{3}{8} \left[ \kappa_\varepsilon \varepsilon^e : \varepsilon^e + \kappa_\omega \delta^2_{\omega} \right] \frac{\omega'}{\|\omega'\|}} \right) : \left( \hat{R} \hat{H} n^d + \hat{C} \hat{1} + \hat{G} \frac{\omega}{\|\omega\|} \right) \]

(I.31)

so that, with the fact that normal \( n^d = (n^d)' \) is deviatoric and therefore that \( \omega : n^d = \omega' : n^d \),

\[ D_{\text{raft}} = \hat{R} \left( \kappa_{\text{oro}} \frac{G}{\|\omega\|_3} (p^d + \sigma p^t) + \sqrt{\frac{3}{8} \left[ \kappa_\varepsilon \varepsilon^e : \varepsilon^e + \kappa_\omega \delta^2_{\omega} \right] \frac{\omega'}{\|\omega'\|}} \right) (\omega : n^d) \hat{H} + \hat{C} \kappa_{\text{oro}} \frac{G \text{tr} \omega}{\|\omega\|_3} (p^d + \sigma p^t) 

+ \hat{G} \left( \kappa_{\text{oro}} \frac{G}{\|\omega\|_2} (p^d + \sigma p^t) + \sqrt{\frac{3}{8} \left[ \kappa_\varepsilon \varepsilon^e : \varepsilon^e + \kappa_\omega \delta^2_{\omega} \right] \frac{\omega'}{\|\omega'\|}} \right) \]

(I.32)

with all terms always positive except scalar product \( \omega : n^d \). Let us first check whether the dissipation due to rafting \( D_{\text{raft}} = \Omega : \hat{\omega} \) –which includes \( \gamma \) precipitates evolutions– remains positive or not.

When \( \omega : n^d \geq 0 \) in case of modeling A –i.e. when Heaviside function \( \hat{H} = H(\omega : n^d) = 1 \)– one has \( (\omega : n^d) \hat{H} = (\omega : n^d)_+ \geq 0 \): the dissipation due to microstructure degradation \( D_{\text{raft}} \) is then positive for any rafting / mechanical coarsening / homothetic growth evolution law of generic form (I.30) under natural assumption \( \hat{R} \geq 0, \hat{C} \geq 0 \) and \( \hat{G} \geq 0 \). This includes any complex loading without rafting \( (w' = 0) \) and any monotonic loading \( (w' \) coaxial with constant normal \( n^d \) such as creep loadings of Sections 3.7.3 and 3.7.4.

In the other loading cases, both dissipations \( D_{\text{raft}} \) and \( D \) may become negative if \( \hat{H} = 1 \) is set when \( \omega : n^d < 0 \). In order to avoid this in complex loading cases, we will consider modeling B which enforces \( \hat{H} = 0 \) to avoid negative dissipation by setting \( \hat{H} = H(\hat{D}) \) and by considering assumption \( \hat{R} \geq 0, \hat{C} \geq 0, \hat{G} \geq 0 \). In such a modeling B, the dissipation due to rafting may become negative, the positivity of intrinsic dissipation being then gained thanks to a large enough plastic dissipation \( D_{\text{plast}} > 0 \).

To sum up, for both modeling A and B, the intrinsic dissipation \( D \) is shown to be always positive, for any loading, monotonic or not, proportional or not, isothermal or not. But this is not the case of the dissipation due to rafting \( D_{\text{raft}} \) only when modeling B is considered. Examples and consequences are detailed in Section M.

I.2.6 At thermodynamics equilibrium

We have mentioned a few times some consequences of thermodynamics equilibrium. The first of them, implicit in this work, is the possibility to define temperature and more generally thermodynamics state variables at RVE scale. But a second consequence is encountered for CMSX-4 and MC2 superalloys at temperature rate below \( 10 \)–\( 20 \)\degree\text{C}/min\(^{-1}\) and strain rates below \( 10^{-4} \)s\(^{-1}\) in case of strain controlled tests: at low thermomechanical
loading rate, a microstructure chemical equilibrium is reached, especially in the matrix which is the phase mostly affected in terms of chemical composition by the dissolution/precipitation of γ′ particles. The constitutive phases are stabilized at an equilibrium volume fractions, only function of temperature for γ′ precipitates, \( f_{\gamma'} = f_{\gamma'\text{eq}}(T) \) (Eq. (3.34) or (3.77), Fig. 3.7).

This second consequence allows for thermodynamics modeling (at equilibrium) at macroscopic scale without the explicit introduction of volume fractions of constitutive phases, as often done (Tinga et al, 2009b, Fedelich et al, 2009, 2012a, Fan et al, 2015): these volume fractions are naturally replaced by corresponding temperature dependencies of the material parameters. In this case, generic evolution law (I.30) becomes (isothermal case, \( \dot{\mathcal{R}} \geq 0, \dot{\mathcal{C}} \geq 0, \dot{\mathcal{G}} \geq 0, \delta_a < 0 \):

\[
\dot{w} \approx g(f_{\gamma'\text{eq}}(T))w_{001}^0 \omega = w_0(T) \left[ \dot{\mathcal{R}} \mathcal{H} n^d + \dot{\mathcal{C}} 1 + \dot{\mathcal{G}} \frac{w}{\|w\|} \right] \quad (\text{I.33})
\]

\textit{i.e.} a rafting law directly expressed in terms of γ channel width tensor rate \( \dot{w} \) with \( w_0(T) = g(f_{\gamma'\text{eq}}(T))w_{001}^0 \) a function of temperature homogenous to a length and at room temperature equal to initial γ channel width \( w_{001}^0 \). For considered CMSX-4, \( w_{001}^0 \approx 50 \) nm, \( w_0(1050^\circ\text{C}) \approx 80 \) nm.
Appendix J

Thermo-elastic terms

Additional thermo-mechanical terms have to be considered. If the thermo-elastic effect of misfit is derived from simple mixture law (Lemaitre et al, 2009), potential $\rho \psi_{TM}$ is added to isothermal potential defined in Eq. (I.15), with $T_{ref}$ the thermo-elastic reference temperature,

$$\rho \psi_{TM} = -3 f_{\gamma} K_{\gamma} \alpha_{\gamma} (T - T_{ref}) \text{tr} \mathbf{e} - 3 f_{\gamma} K_{\gamma} (\alpha_{\gamma} (T - T_{ref}) + \delta_{u}) \text{tr} \mathbf{e} - \rho C_{e} (T \ln \frac{T}{T_{ref}} - T + T_{ref})$$  \hspace{1cm} (J.1)

or, introducing $K$ and $\alpha$ the homogenized bulk modulus and thermal expansion coefficient,

$$\rho \psi_{TM} = -3 K \alpha (T - T_{ref}) \text{tr} \mathbf{e} - 3 f_{\gamma} K_{\gamma} \delta_{u} \text{tr} \mathbf{e} - \rho C_{e} (T \ln \frac{T}{T_{ref}} - T + T_{ref})$$  \hspace{1cm} (J.2)

so that $f_{\gamma}$ the $\gamma'$ precipitates volume fraction, $K_{\gamma}$ the elastic bulk modulus of such a $\gamma'$ phase, $\delta_{u}$ the unconstrained misfit (homogeneous to a strain); $C_{e}$ is the material specific heat capacity assumed constant over a large temperature range. Latent heat due to phase change has been neglected.

From state law $\mathbf{\sigma} = \rho \frac{\partial \psi}{\partial \mathbf{e}}$ one gets

$$\mathbf{e} = \mathbf{E}^{-1} : \mathbf{\sigma} + \alpha (T - T_{ref}) \mathbf{1} + \mathbf{e}^{pl} + f_{\gamma} K_{\gamma} \delta_{u} \mathbf{1}$$
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Appendix K

Tensorial $w$ and $\omega$

In the general case, symmetric $\gamma$ channel width variable $w$ may be non diagonal, of components $w_{ij} = w_{ji}$. It is endowed by the change of basis rule $\bar{w} = Q \cdot w \cdot Q^T$, i.e. $\bar{w}_{ij} = Q_{ik} Q_{jl} w_{kl}$, with $Q \in SO3$ a rotation matrix ($\det Q = 1$). This defines $w$ as a second order tensor and ensures the frame independence (objectivity) of the free energy density (I.15) as then dimensionless variable $\omega = w / g(f_\gamma) w_{001}^0$ is also a symmetric second order tensor (satisfying change of basis $\bar{\omega} = Q \cdot \omega \cdot Q^T$) and as

$$\rho \psi(e, e', \omega, p^d, p'^d, T) = \rho \psi(Q \cdot e \cdot Q^T, Q \cdot e' \cdot Q^T, Q \cdot \omega \cdot Q^T, p^d, p'^d, T) \quad \forall Q \in SO3$$

with $p^d, p'^d, T$ scalar variables. This means that second order tensor $w$ may be considered as a 2-linear mapping

$$w : \mathbb{R}^3 \times \mathbb{R}^3 \to \mathbb{R}, \quad (n, m) \mapsto w(n, m) := w_{ij} n_i m_j$$

If $n$ is a unit vector $w_{nn} = w(n, n)$ is the mean $\gamma$ channel width in direction $n$. 

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Appendix L

Link with Fedelich’s index of rafting $\xi$  
(Fedelich et al, 2012b)

For an uniaxial loading performed in direction $1 \equiv [001]$ (the most important one from a technological point of view), the transverse channel widths remain equals, $w_2 = w_3 = cw_1$ with $c$ a scalar, pure coarsening being $c = 1$ and complete rafting $c = 0$. In cubic Natural Anisotropy Basis,

$$
\mathbf{w} = \begin{bmatrix}
w_1 & 0 & 0 \\
0 & cw_1 & 0 \\
0 & 0 & cw_1
\end{bmatrix}
$$

and $\mathbf{w} = \begin{bmatrix}
\frac{2}{3}(1-c)w_1 \\
0 & -\frac{1}{3}(1-c)w_1 & 0 \\
0 & 0 & -\frac{1}{3}(1-c)w_1
\end{bmatrix}
$ and $\text{tr} \mathbf{w} = (1+2c)w_1$, $w_{eq} = \sqrt{\frac{3}{2}} \| \mathbf{w}' \| = (1-c)w_1$ so that the 3D index of rafting $x_{RAFT}$ is simply related to $c$ (and reciprocally) as $x_{RAFT} = \frac{w_{eq}}{\text{tr} \mathbf{w}} = \frac{1-c}{1+2c}$ or $c = \frac{1-x_{RAFT}}{1+2x_{RAFT}}$. Note that this relationship between $c$ and $x_{RAFT}$ applies to any more complex rafting / coarsening / homothetic growth process ensuring $w_2 \approx w_3$.

In order to make a link with Fedelich rafting index $\xi$, let us place ourselves in the same state of comparison than for definition (3.37) and use the fact that both definitions of coarsening expression $w_{cube}$ and of complete rafting expression $w_{RAFT}$ are made at given volume fraction $f_{\gamma}'$ and at given periodicity $\lambda$. Isotropic periodicity $\lambda_i = \lambda$ is assumed so that, from Eq. (3.43),

$$
f_{\gamma}' = \left(1 - \frac{w_1}{\lambda}\right) \left(1 - \frac{w_2}{\lambda}\right) = \left(1 - \frac{w_1}{\lambda}\right) \left(1 - \frac{cw_1}{\lambda}\right)^2
$$

The definition (3.37) is valid for tensile loading. From the assumption $w_1 = w = w_{cube} + \xi (w_{RAFT} - w_{cube})$ it is possible to write $\frac{w}{\lambda} = 1 - f_{\gamma}'^{1/3} + \xi (f_{\gamma}'^{1/3} - f_{\gamma}')$, so that we get explicitly $c = c(f_{\gamma}', \xi)$ as a function of $f_{\gamma}'$ and $\xi$.

Finally inverting $c = \frac{1-x_{RAFT}}{1+2x_{RAFT}}$, the index of rafting $x_{RAFT} = \frac{w_{eq}}{\text{tr} \mathbf{w}}$ is related to both the $\gamma'$ precipitates volume fraction and index of rafting $\xi$ as

$$
x_{RAFT} = \frac{1-c(f_{\gamma}', \xi)}{1+2c(f_{\gamma}', \xi)} = x_{RAFT}(f_{\gamma}', \xi)
$$

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Eq. (L.3) applies to tensile loading in \(<001>\) direction. Recall that it is an approximate expression obtained thanks to the assumption of equal pericities in the three directions of space \(\lambda_1 = \lambda_2 = \lambda_3 = \lambda\). It gives back \(x_{raft} = 0\) when \(\xi = 0\) (coarsening) and \(x_{raft} = 1\) when \(\xi = 1\) (rafting).
Appendix M

Thermodynamics of rafting under complex loading

In the aim of both checking the ability of the model to reproduce complex microstructure evolutions and the positivity of intrinsic dissipation, we have simulated a complex experiment starting first by a compression creep test at 1050°C for 100 hours and then, a stress reversal. Experimental data for such experiments can be found in Tetzlaff and Mughrabi (2000). In Section I.2.3, we have proposed two possibilities to ensure the positivity of intrinsic dissipation: modeling A with as argument of Heaviside function \( H \) the scalar product \( \omega : n^d \) (Eq. (I.27), this strong assumption enforces the positivity of the dissipation due to rafting itself, as illustrated in Fig. M.1d) and modeling B with as argument of \( H \) the total dissipation \( D \) (Eq. (I.28), \( D \geq 0 \) but negative values of the dissipation due to rafting are accepted, see Fig. M.1d).

Recall first that these two possibilities of modeling give exactly the same responses in creep and in monotonic tension: the responses of Section 3.7 are those of both modeling A and B. Within these cases both \( D_{raft} \geq 0 \) and \( D \geq 0 \).

Differences only occur for complex loading cases, for instance with load reversal, when rafting takes place. In order to emphasize them and to retain one modeling (it will hereafter be modeling B) we study here an academic loading made of a first creep stage in compression, 100 hours at \( \sigma = -140 \) MPa, followed by a 10 hours long load reversal at stress rate \( \dot{\sigma} = 7.78 \times 10^{-3} \) MPa s\(^{-1}\) up to a second creep stage, this time at positive stress \( \sigma = 140 \) MPa (other positive \( \sigma \) and \( \dot{\sigma} \) give similar results with different phenomenon rates). The loading direction is \([001] \equiv 1\).

During the initial stage in compression a P-type rafting is well obtained (see Fig. 1.15, compressive case \( w_1 = 0 \) and \( w_2 = w_3 > w_0 \)) with an index of rafting \( x_{raft} = 0.5 \). This situation is obtained up to time \( t = 100 \) h of the start of the load reversal (the microstructure evolution is given in Fig. M.1a). The microstructure evolution rate, stress enhanced, is much lower in the load reversal stage: the crossing of the elasticity domain and the corresponding low stress level decrease quite much the rate of rafting, rate which even vanishes when \( H = 0 \) for thermodynamics considerations (as then \( \omega_{raft} = 0 \)). For both modeling A and B the intrinsic dissipation \( D \) remains positive (as proven in Section I.2.5).
but not the dissipation due to rafting $D_{\text{raft}}$ for modeling $B$ at $H = H(\mathcal{D})$ (see Fig. M.1d).

The main difference between both modeling approaches is obtained in the second creep stage at positive stress:

— for modeling $A$, the (slow) microstructure evolution is due to LSW homothetic growth only as then $\omega : n^d < 0$ and $H = 0$ from $t = 105$ h up to the end: the microstructure remains P-type rafted at $x_{\text{raft}} = 0.5$, feature which is not consistent with the experimental results of Tetzlaff and Mughrabi (2000).

— for modeling $B$, the stage at $H = 0$ is short (between $t = 107$ h and $t = 108.7$ h) and rafting takes place again for $t > 108.7$ h, up to fully N-type rafted microstructure at $x_{\text{raft}} = 1$: this is in full agreement with the experimental results of Tetzlaff and Mughrabi (2000) on similar nickel based single crystal superalloys with a negative misfit.

Figure M.1 – and : modeling $A$ with $H = H(\omega : n^d)$, and : modeling $B$ with $H = H(\mathcal{D})$.

a) Evolution of $\gamma$ channel widths $w_1$ (solid lines), $w_2 = w_3$ (dotted lines), b) Evolution of index of rafting $x_{\text{raft}} = w_{\text{eq}} / trw$, c) Evolution of intrinsic dissipation $\mathcal{D}$ (logarithmic scale to underline the positivity of $\mathcal{D}$), d) Evolution of dissipation due to rafting $D_{\text{raft}}$.

Let us conclude on the thermodynamics features. Both modeling approaches are fully equivalent in standard (constant) creep and in monotonic loading cases, either tensile or compressive. Both modeling always satisfy the second principle of thermodynamics but, the most striking result is that modeling $B$ allows for a negative dissipation due to rafting (compensated by positive plastic dissipation $D_{\text{plast}}$ to ensure $\mathcal{D} \geq 0$). The expression for
Heaviside term $\mathcal{H}$ chosen to ensure a positive intrinsic dissipation has a major role on the rafting evolution: modeling A with $\mathcal{H} = \mathcal{H}(\omega : \mathbf{n}^d)$ spuriously blocks the tensile evolution after a pre-compression stage of rafting (of P-type), when modeling B with $\mathcal{H} = \mathcal{H}(\mathcal{D})$ allows to properly model the change from P-type rafting in compression to N-type rafting in tension. This is the modeling to retain.

**Positive misfit alloy**  Note last that the present work gives also the possibility to model the mechanical/rafting behavior of single crystals superalloys at positive misfit $\delta_u$ (mostly Co-based superalloys (Tanaka et al, 2012, Pyczak et al, 2013)). For those, the N-type rafting occurs in compression while the P-type rafting occurs in tension (Mughrabi (2014)). The evolution law for tensorial $\gamma$ channel width variable has to be changed into

$$\dot{\omega} = -\text{sgn}(\delta_u)\mathcal{R}_H(\mathcal{D}) \mathbf{n}^d + \dot{\mathcal{C}} \mathbf{1} + \dot{\mathcal{G}} \frac{\omega}{||\omega||}$$

With $\text{sgn}(\delta_u) = \frac{\delta_u}{||\delta_u||}$ and where the Heaviside function $\mathcal{H} = \mathcal{H}(\mathcal{D})$ ensures the positivity of the dissipation. As shown in figure M.2, microstructural evolutions are the opposite of those shown in figure M.1. N-rafting is reproduced under compression creep and P-rafting is reproduced under tensile creep.

**Norm sensitivity**  Creep and monotonic tensile tests can be correctly described by using the infinity norm (Eq. M.1) instead of the quadratic one (slightly modifications to parameters have to be done), as shown in figure M.3.

$$||\omega|| = \max_i |\omega_i|$$  \hspace{1cm} (M.1)

However, using the infinity norm does not allow to describe the microstructural evolution happening under low alternated loadings. Under the same loading of figure M.1, Orowan ripening governs the evolution of the microstructure until, during the traction phase, $\gamma'$ rafting takes over. Thus, the P-rafting and the P-rafting/N-rafting transition is not reproduced (figure M.4). It has to be highlighted that using the infinity norm corresponds to account of just the wider channel evolution (for example just the horizontal channel $\mathcal{w}_1$ during N-rafting, see figure 3.2). Hence, this result proves that even when just $\gamma'$ rafting takes place (simulations are performed along <001>) only a tensorial formulation of microstructural evolution allows to account for the very evolution observed in the material.

Results obtained by using the unitary norm (Eq. M.2) are presented in the figure M.5.

$$||\omega||_1 = \omega_1 + \omega_2 + \omega_3$$  \hspace{1cm} (M.2)

Again, creep and monotonic tension can be quite well described but no good agreement with literature (Ott and Mughrabi, 1999, Tetzlaff and Mughrabi, 2000, Giraud, 2013a) is found under alternating loadings (figures M.5 and M.6).
Thermodynamics of rafting under complex loading

Figure M.2 – and : modeling A with $H = H(\omega : n^d)$, and : modeling B with $H = H(D)$.

Example of curves computed for a positive misfit alloy: a) evolution of $\gamma$ channel widths $w_1$ (solid lines), $w_2 = w_3$ (dotted lines), b) evolution of index of rafting $x_{raft} = w_{eq} / \text{tr} w$, c) evolution of intrinsic dissipation $D$ (logarithmic scale to underline the positivity of $D$), d) evolution of dissipation due to rafting $D_{raft}$.
Figure M.3 – Creep and monotonic tension curves obtained by computing the Orowan stress with the infinity norm: a) creep curves, b) microstructural evolution during creep and c) monotonic tensile curves. (Model: red lines, experimental: black lines and Orowan stress: red dotted lines.

Figure M.4 – Curves computed by using the infinity norm (Eq. M.1) in computing the Orowan stress: Evolution of $\gamma$ channel widths $w_1$ (solid lines), $w_2 = w_3$ (dotted lines), b) Evolution of index of rafting $x_{RAFT} = w_{eq}/\text{tr} W$. 

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Figure M.5 – Creep and monotonic tension curves obtained by computing the Orowan stress with the unitary norm: a) creep curves, b) microstructural evolution during creep and c) monotonic tensile curves. (Model: red lines, experimental: black lines and Orowan stress: red dotted lines.)

Figure M.6 – Curves computed by using the unitary norm (Eq. M.2) in computing the Orowan stress: Evolution of $\gamma$ channel widths $w_1$ (solid lines), $w_2 = w_3$ (dotted lines), b) Evolution of index of rafting $x_{raft} = w_{eq} / tr w$
Appendix N

Parameters at 1050°C

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Appendix O

Set of material parameter with $h = 0$

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Set of material parameter with $h = 0$

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Appendix P

Parameters at 850°C and 1200°C
Parameters at 850°C and 1200°C

Figure P.1 – Curves identified at 850°C and along the <001> direction: (a) γ channel widths, (b) creep curves and (c) monotonic tensile curves.

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Figure P.2 – Curves identified at 850°C and along the $<111>$ direction: (a) monotonic tensile curves, (b) creep curves and (c) γ channel widths.

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Parameters at 850°C and 1200°C

Figure P.3 – Curves identified at 1200°C: (a) γ channel widths, (b) creep curves and (c) monotonic tensile curves.

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Figure P.4 – SEM images of the $\gamma/\gamma'$ microstructure after tests at $1200^\circ$C along the $<001>$ crystal direction: (a) after 5 minutes of thermal aging, (b) after 1h of thermal aging and (c) after 1h of creep at 120 MPa.
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Appendix Q

RaftX code in ZéBuLon (no damage)

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Appendix R

RaftXD code in ZéBuLon
(visco-plasticity model RaftX
coupled with damage)

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RaftXD code in ZéBuLon (visco-plasticity model RaftX coupled with damage)

Visco-plasticity and damage modeling of Single Crystal Superalloys at high temperature: a tensorial microstructure-sensitive approach
Appendix S

Input files

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Appendix T

Thermodynamics features

The thermodynamics framework for elasto-visco-plasticity coupled with microstructural evolutions has been presented in the Appendix I. This latter has been developed without considering the Hill criterion, the damage nor the isotropic hardening, which are instead included in the Z-Set code of the full model (see the Appendix R). Moreover, in Appendix I the coupling between elasticity and microstructural evolutions has been theoretically considered while it is neglected in practice because of the lack of experimental data. Here, the thermodynamics framework of the model used for the applications is presented, thus Hill visco-plasticity, damage and isotropic hardening are included (while the coupling between elasticity and microstructural evolutions is neglected).

Table T.1 presents the list of the thermodynamics variables.

<table>
<thead>
<tr>
<th>State Variables</th>
<th>Observable Variables</th>
<th>Internal Variables</th>
<th>Thermodynamics Forces</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total strain</td>
<td>$\varepsilon$</td>
<td>$\varepsilon''_1$</td>
<td>Stress $\sigma$</td>
</tr>
<tr>
<td>Temperature</td>
<td>$T$</td>
<td>$\varepsilon''_2$</td>
<td>Stress $-\sigma$</td>
</tr>
<tr>
<td>Plastic strain  1</td>
<td></td>
<td>$r$</td>
<td>Isotropic hardening $R$</td>
</tr>
<tr>
<td>Plastic strain  2</td>
<td></td>
<td>$\tau$</td>
<td>Isotropic hardening $\bar{R}$</td>
</tr>
<tr>
<td>Isotropic hardening variable</td>
<td>$r$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Isotropic hardening variable</td>
<td>$\tau$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dimensionless $\gamma$ channel width</td>
<td>$\omega$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Damage</td>
<td>$D$</td>
<td>$\Omega$</td>
<td>Strain energy density release rate $-Y$</td>
</tr>
</tbody>
</table>

Table T.1 – Thermodynamics variables.
State potential

\[ \rho \psi = \frac{1}{2} (\varepsilon - \varepsilon_1^p - \varepsilon_2^p) : \mathbb{E} (1 - D) : (\varepsilon - \varepsilon_1^p - \varepsilon_2^p) + R_\infty \left\{ r + \frac{1}{b} \left[ \exp \left( -br - 1 \right) \right] \right\} + \frac{\kappa_{\text{oro}} G}{\| \omega \|} r \]

\[ + \bar{R}_\infty \left\{ \bar{r} + \frac{1}{b} \left[ \exp \left( -\bar{b}\bar{r} - 1 \right) \right] \right\} + \frac{\bar{\omega} \kappa_{\text{oro}} G}{\| \omega \|} \bar{r} + \frac{1}{2} (\kappa_u - \kappa_{\text{io} \omega_{eq}}) \delta_u^2 \]

Thermodynamic forces

— Elasticity

\[ \sigma = \rho \frac{\partial \psi}{\partial \varepsilon} = \mathbb{E} (1 - D) : (\varepsilon - \varepsilon_1^p - \varepsilon_2^p) \rightarrow \bar{\sigma} = (\varepsilon - \varepsilon_1^p - \varepsilon_2^p) : \mathbb{E} \]

then

\[ \bar{\sigma} = \mathbb{E} : (\varepsilon - \varepsilon_1^p - \varepsilon_2^p). \]

— Isotropic hardening

\[ R = \rho \frac{\partial \psi}{\partial r} = R_\infty \left( 1 - e^{-br} \right) + \frac{\kappa_{\text{oro}} G}{\| \omega \|} \]

\[ (T.3) \]

\[ (T.4) \]

\[ \bar{R} = \rho \frac{\partial \psi}{\partial \bar{r}} = \bar{R}_\infty \left( 1 - e^{-\bar{b}\bar{r}} \right) + \frac{\bar{\omega} \kappa_{\text{oro}} G}{\| \omega \|}. \]

— Plastic strains

\[ \rho \frac{\partial \psi}{\partial \varepsilon_1^p} = -\bar{\sigma} \]

and

\[ \rho \frac{\partial \psi}{\partial \varepsilon_2^p} = -\bar{\sigma}. \]

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— Damage

\[ Y = -\rho \frac{\partial \psi}{\partial D} = \frac{1}{2} (\varepsilon - \varepsilon^p) : \mathbb{E} : (\varepsilon - \varepsilon^p). \]

— Microstructural evolutions

\[ \Omega = -\rho \frac{\partial \psi}{\partial \omega} = \kappa_{\text{oro}} \frac{G}{\|\omega\|^2} \frac{\omega}{\|\omega\|} (r + \omega \bar{r}) + \sqrt{\frac{3}{8}} \kappa_\omega \delta_a \frac{\omega \delta_a}{\|\omega\|^2}. \quad (T.6) \]

Plastic strain partition

\[ \varepsilon = \varepsilon^e + \varepsilon^p \quad \varepsilon^p = \varepsilon_1^p + \varepsilon_2^p \quad (T.7) \]

Effective stress

\[ \tilde{\sigma} = \frac{\sigma}{1 - D} \quad (T.8) \]

Criterion functions

The two criterion functions are the following:

\[
\begin{cases}
  f_1 = \tilde{\sigma}_\text{Hill} - R - \kappa_{\text{oro}} \frac{G}{\|\omega\|} - \sigma_y \\
  f_1 < 0 \text{ or } f_1 < 0 \text{ elasticity} \\
  f_1 = \sigma_v \geq 0 \text{ viscoplasticity} \\
  f_2 = \| \tilde{\sigma}^T_a \|_a - R - \kappa_{\text{oro}} \frac{G}{\|\omega\|} - \sigma_y \\
  f_2 < 0 \text{ or } f_2 < 0 \text{ elasticity} \\
  f_2 = \sigma_v \geq 0 \text{ viscoplasticity} \\
  \|	ilde{\sigma}^T_a\|_a = 3 \left( \frac{1}{6} \sum_{i \neq j} \left| \tilde{\sigma}_{ij} \right|^a \right) \quad (T.9)
\end{cases}
\]
\[ \dot{\sigma}_{\text{Hill}} = \sqrt{\frac{3}{2}} \left( \sigma^d : \sigma^d + h^2 \sigma^d : \sigma^d \right) \] (T.10)

Viscosity laws:

\[ \dot{p}_{\text{Hill}}^1 = \left\langle -\frac{\sigma_{\text{voo}}}{K_N} \ln \left( 1 - \frac{f_1}{\sigma_{\text{voo}}} \right) \right\rangle^N \] (T.11)

\[ \dot{p}_{\text{Hill}}^2 = \left\langle \frac{f_2}{K_N} \right\rangle^N \frac{1}{\max \left( \kappa \left( \sigma_{eq}^d - \sigma_{lim}^\mu \right), 1 \right)} \] (T.12)

**Internal variables evolution**

\[ \dot{\lambda}_1 = -\dot{\lambda}_1 \frac{\partial f_1}{\partial R} = \dot{\lambda}_1 \quad \dot{\lambda}_2 \frac{\partial f_2}{\partial R} = \dot{\lambda}_2 \]

**T.1 Plastic strain rates**

\[ \dot{\varepsilon}_{\text{p}}^1 \]

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Plastic strain rates

\[ \dot{\varepsilon}_p^1 = \lambda_1 \frac{\partial f_1}{\partial \sigma} \]

\[ = \frac{\lambda_1}{1 - D} \frac{\partial \sigma_{\text{Hill}}}{\partial \sigma} = \frac{\lambda_1}{1 - D} \frac{\partial}{\partial \sigma} \left[ \frac{3}{2} (\sigma^d : \sigma^d + h^2 \sigma^T : \sigma^T) \right]^{1/2} \]

\[ = \frac{\lambda_1}{1 - D} \left[ \frac{3}{2} (\sigma^d : \sigma^d + h^2 \sigma^T : \sigma^T) \right]^{-1/2} \frac{\partial}{\partial \sigma} \left[ \frac{3}{2} (\sigma^d : \sigma^d + h^2 \sigma^T : \sigma^T) \right] \]

\[ = \frac{\lambda_1}{\sigma_{\text{Hill}}(1 - D) ^{2/2}} \frac{3}{2} \left[ \frac{\partial (\sigma^d : \sigma^d + h^2 \sigma^T : \sigma^T)}{\partial \sigma} \right] \frac{\partial \sigma_{\text{Hill}}}{\partial \sigma} \]

\[ = \frac{\lambda_1}{\sigma_{\text{Hill}}(1 - D) ^{2/2}} \left[ \frac{3}{2} (\sigma^d : \sigma^d + h^2 \sigma^T : \sigma^T) \right] - \frac{1}{2} \frac{\partial}{\partial \sigma} \left[ \frac{3}{2} (\sigma^d : \sigma^d + h^2 \sigma^T : \sigma^T) \right] \]

\[ = \frac{\lambda_1}{1 - D} \left( \frac{3}{2} \sigma_{\text{Hill}} + h^2 \frac{3}{2} \sigma_{\text{Hill}}^T \right) \]

where \( n_1 = \frac{3}{2} \sigma_{\text{Hill}} \) et \( n_1^T = \frac{3}{2} \sigma_{\text{Hill}}^T \)

\[ \dot{\varepsilon}_2^p = \frac{\lambda_1}{1 - D} \left( n_1^T + h^2 n_1^T \right) \]

\[ \dot{\varepsilon}_2^p = \lambda_2 \frac{\partial f_2}{\partial \sigma} = \frac{\lambda_2}{1 - D} \frac{\partial |\sigma_{\text{Hill}}|}{\partial \sigma} \]

by components

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\begin{align*}
\dot{\varepsilon}_{12}^p &= \frac{\lambda_2}{1 - D} \frac{\partial}{\partial \sigma_{12}} \left[ 3 \left( \frac{[\sigma_{12}]^a + [\sigma_{21}]^a + [\sigma_{13}]^a + [\sigma_{31}]^a + [\sigma_{23}]^a + [\sigma_{31}]^a}{6} \right) \right] \\
&= \frac{3\lambda_2}{a(1 - D)} \left( \frac{[\sigma_{12}]^a + [\sigma_{21}]^a + [\sigma_{13}]^a + [\sigma_{31}]^a + [\sigma_{23}]^a + [\sigma_{31}]^a}{6} \right)^{\frac{1}{a - 1}} \\
&= \frac{3\lambda_2}{a(1 - D)} \left( 3 \left( [\sigma_{12}]^a + [\sigma_{21}]^a + [\sigma_{13}]^a + [\sigma_{31}]^a + [\sigma_{23}]^a + [\sigma_{31}]^a \right) \right)^{\frac{1}{a - 1}} \left\{ (\sigma_{12})^{a - 1} \text{sign}(\sigma_{12}) \right\} \\
&= \frac{\lambda_2}{a(1 - D)} \frac{3^{1 - a}}{3^{1 - a}} - \frac{\lambda_2}{a(1 - D)} \frac{3^{1 - a}}{3^{1 - a}} \\
&= \frac{\lambda_2}{(1 - D) \left( \left\{ \sigma_\pi \right\}^a \right)^{a - 1}} \left\{ (\sigma_{12})^{a - 1} \text{sign}(\sigma_{12}) \right\}
\end{align*}

Then, more generally, \( \dot{\varepsilon}_{2i}^p = 0 \) (with no sum) and for \( i \neq j \)

\[ \dot{\varepsilon}_{2i}^p = \frac{\lambda_2}{1 - D} \frac{3^a}{6} \left\{ \sigma_{ij} \right\}^{a - 1} \text{sign}(\sigma_{ij}) \]

the normal is \( \tilde{n}_{2i} = 0 \) (no sum) and

\[ \tilde{n}_{2i} = \frac{3^a}{6} \left\{ \sigma_{ij} \right\}^{a - 1} \text{sign}(\sigma_{ij}) \quad (T.13) \]

and so for \( i \neq j \)

\[ \dot{\varepsilon}_{2ij}^p = \frac{\lambda_2}{(1 - D)} \tilde{n}_{2ij} \]

### T.2 Accumulated plastic strain rates

\( \dot{p}_{1}^{\text{Hill}} \)

The plastic power is

\[ \boldsymbol{\sigma} : \dot{\varepsilon}_1^p = \dot{p}_{1}^{\text{Hill}} \bar{\sigma}_{1}^{\text{Hill}} = \boldsymbol{\sigma} : \frac{\lambda_1^{\text{Hill}}}{1 - D} \left( n_1^d + h^2 n_1^d \right) = \frac{\lambda_1^{\text{Hill}}}{1 - D} \bar{\sigma}_{1}^{\text{Hill}} \]

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we find that →  \( \dot{\rho}_p \) is

\[ \dot{\rho}_2 = \frac{\lambda_2}{1 - D} \]

The plastic power \( \tilde{\sigma} : \tilde{\varepsilon}_2^p =\)

\[ \dot{\rho}_2 \frac{\lambda_2}{1 - D} \]

\( \tilde{\sigma} : \tilde{\varepsilon}_2^p =\)

\[ \frac{\lambda_2}{1 - D} \frac{3^a}{6} \left( \frac{1}{\|	ilde{\sigma}\|_a} \right)^{a-1} \sum_i \sum_j |\tilde{\sigma}_{ij}|^{a-1} \text{sign}(\tilde{\sigma}_{ij}) \tilde{\sigma}_{ij} \text{ for } i \neq j \]

\[ \frac{\lambda_2}{1 - D} \frac{3^a}{6} \left( \frac{1}{\|	ilde{\sigma}\|_a} \right)^{a-1} \sum_i \sum_j |\tilde{\sigma}_{ij}| \text{ for } i \neq j \]

for \( i \neq j \)

\[ \sum_i \sum_j |\tilde{\sigma}_{ij}|^{a-1} = \frac{6}{3^a} \|	ilde{\sigma}\|_a \]

\[ \tilde{\sigma} : \tilde{\varepsilon}_2^p =\]

\[ \frac{\lambda_2}{1 - D} \frac{3^a}{6} \left( \frac{1}{\|	ilde{\sigma}\|_a} \right)^{a-1} \frac{6}{3^a} \|	ilde{\sigma}\|_a \]

\[ \frac{\lambda_2}{1 - D} \|	ilde{\sigma}\|_a \]

then

\[ \dot{\rho}_2 = \frac{\lambda_2}{1 - D} \]

(T.14)
T.3 Dissipation

\[ D = - \sum K \mathcal{A}_K \cdot \mathbf{\dot{v}}_K = D_{\text{plast}} + D_{\text{endo}} + D_{\text{raft}} \]

— Dissipation due to plasticity is:

\[ D_{\text{plast}} = \sigma : \dot{\varepsilon}^p_1 + \sigma : \dot{\varepsilon}^p_2 - R \dot{r} - \overline{R} \dot{r} \]

\[ = (1 - D) \left[ \overline{\sigma} : \dot{\varepsilon}^p_1 + \overline{\sigma} : \dot{\varepsilon}^p_2 - R \dot{p}^\text{Hill}_1 - \overline{R} \dot{p}^\text{Hill}_2 \right] \]

\[ = (1 - D) \left[ (f_1 + \sigma_y) \dot{p}^\text{Hill}_1 + (f_2 + \overline{\sigma}_y) \dot{p}^\text{Hill}_2 \right] \]

so

— in elasticity, \( D_{\text{plast}} = 0 \)

— in plasticity \((1 - D) \left[ (f_1 + \sigma_y) \dot{p}^\text{Hill}_1 + (f_2 + \overline{\sigma}_y) \dot{p}^\text{Hill}_2 \right] > 0.\)

— Dissipation due to damage

\[ D_{\text{endo}} = Y \dot{\mathcal{D}} > 0 \]

where \( Y \) is the elastic energy and \( \dot{\mathcal{D}} \) is defined positive.

— Dissipation due to \( \gamma' \)-rafting is

\[ D_{\text{raft}} = \mathbf{\Omega} : \mathbf{\dot{\omega}} \]

\[ D_{\text{raft}} = \left( \kappa_{\text{cro}} \frac{G}{\|\omega\|^2} \frac{\omega}{\|\omega\|} (r + \omega \overline{r}) + \sqrt{\frac{3}{8}} \kappa_{\omega} \delta^2_{\omega} \|\omega\| \right) : \left( \dot{\mathcal{H}} \mathbf{n}_1^d + \dot{\mathbf{C}} \mathbf{1} + \dot{\mathbf{G}} \frac{\omega}{\|\omega\|} \right) \]

\[ \mathbf{n}_1^d = (\mathbf{n}_1^d)' \text{ so } \omega' : \mathbf{n}_1^d = \omega : \mathbf{n}_1^d \]

\[ \omega' : \mathbf{1} = 0 \]

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\[ \omega : 1 = \text{tr} \omega \]
\[ \omega : \omega = \| \omega \|^2 \]
\[ \omega' : \omega = \omega' : \omega' = \| \omega' \|^2 \]

\[
\mathcal{D}_\text{raft} = \dot{\mathcal{K}} \left( \frac{G}{\| \omega \|^3} \left( r + \overline{\omega} r \right) + \sqrt{\frac{3}{8} \frac{\kappa_\omega \delta^2}{\| \omega' \|}} \right) \left( \omega : \mathbf{n}_1 \right) \mathcal{H} + \dot{\mathcal{C}} \frac{G \text{tr} \omega}{\| \omega \|^3} \left( r + \overline{\omega} r \right) \\
+ \dot{\mathcal{G}} \left( \frac{G}{\| \omega \|^2} \left( r + \overline{\omega} r \right) + \sqrt{\frac{3}{8} \left[ \kappa_\omega \delta^2_{u} \right]} \| \omega' \| \right).
\]

(T.15)

Based on the same assumptions/observations presented in the Appendix I, it results \( \mathcal{D}_\text{raft} \geq 0 \).
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Titre : Visco-plasticité endommageable des superalliages monogranulaires base Ni à haute température : approche couplée à une représentation tensorielle de la microstructure

Mots clefs : CMSX-4, Fluage, Ductilité, représentation tensorielle des évolutions microstructurales, effets de vitesse sur le seuil d’endommagement

Résumé : Un modèle phénoménologique 3D de visco-plasticité couplé avec les évolutions microstructurales et l’endommagement est proposé pour les superalliages monogranulaires base Nickel, les matériaux des aubes de turbine à haute pression de moteurs d’hélicoptères. L’anisotropie de la mise en radiaxes, la croissance et la dissolution de la phase durcissante sont modélisés. Une variable tensorielle et sa loi d’évolution permettent la description de la variation de la largeur des couloirs de matrice. Ce travail s’appuie sur la décomposition en modes de Kelvin du tenseur d’élasticité. Cette décomposition conduit également à une description multi-critère méso-copique de la visco-plasticité cubique. Une formulation en (visco-)plasticité cristalline a été également proposée. Une loi d’endommagement avec seuil de type $dD/dt = \ldots$ est formulée pour la modélisation du fluxage tertiaire et pour la prévision de la ductilité en traction. Une expression originale du seuil d’endommagement rend compte des effets de vitesse sur l’amorçage de l’endommagement par visco-plasticité. Une étude expérimentale a été conduite sur le CMSX-4, l’alliage au centre de cette étude, parallèlement aux travaux de modélisation. Des essais de dissolution ont été réalisés afin de mesurer la variation de la fraction volumique des précipités avec la température. Les mécanismes de déformation du matériau ont été observés en fluxage isotherme à 850°C et 1050°C selon les principales directions cristallines du triangle stéréographique standard et constituent une base d’identification pour le modèle, la réponse mécanique du matériau obtenue. Trois essais de traction ont été réalisés selon la direction $<111>$, deux à vitesse de chargement constante, le troisième à vitesse variable. Des essais cycliques thermiquement de type 150h-moteur ont été réalisées sur le banc MAATRE. Des analyses EBSD et MET ont été réalisées sur les échantillons orientés selon les directions cristallines $<011>$, $<111>$ et $<112>$ et testés à 850°C. Ces analyses ont montré que le mâclage est le principal mécanisme de déformation pour des déformations supérieures à 1–2% selon ces orientations cristallines en fluxage à cette température et à haute contrainte ($>400$ MPa). Enfin, le modèle a été implanté dans le code à Ellement Finis ZéBuLon, sans ou avec endommagement, et des calculs de structures ont été réalisés.

Title : Visco-plasticity and damage modeling of single crystal superalloys at high temperatures: a tensorial microstructure-sensitive approach

Keywords : CMSX-4, Creep, Ductility, Tensorial $\gamma/\gamma'$ evolutions, Rate dependent (plastic damage threshold

Abstract : A 3D phenomenological model coupling visco-plasticity, microstructural evolutions and damage is proposed for Ni-based single crystal superalloys, which are widely used materials for high pressure turbine blade in helicopter engines. The anisotropy of the $\gamma/\gamma'$-rafting, the $\gamma/\gamma'$-coarsening and the dissolution of the hardening $\gamma'$ phase are modeled. A tensorial variable and its evolution law allow to describe the variation of the $\gamma$ channels. The modeling is based on the Kelvin decomposition of the elasticity tensor. This decomposition leads to a phenomenological multi-criterion description of the cubic visco-plasticity. A formulation based on the single crystal (visco-)plasticity framework is also proposed. A damage law of type $dD/dt = \ldots$ is introduced in order to model the tertiary creep stage and the ductility. A novel rate sensitive damage threshold is introduced in order to account for the rate sensitivity of the damage onset by visco-plasticity. An experimental study has been carried out on the CMSX-4 alloy, which is the material of main interest in this study, in parallel to the modeling work. Dissolution tests have been carried out to measure the $\gamma'$-volume fraction variation with temperature. The deformation mechanisms of the CMSX-4 alloy have been observed by performing tensile creep tests at 1050°C and 850°C. These tests have also constituted a database for the model identification. Three tensile tests have been realized along the $<111>$ crystal direction, two at constant strain rate and the third by varying the strain rate. Non-isothermal creep tests reproducing the 150h-type engine test have been performed on the MAATRE bench. EBSD and TEM analysis have been realized on the specimens oriented along the $<011>$, $<011>$ and $<112>$ crystal directions and crept at 850°C. These analysis have shown that micro-twinning governs the deformation along these crystal directions during creep at this temperature and at high stresses ($>400$ MPa) for deformation in excess of 1–2%. Finally, the model (with and without damage) has been encoded in the ZéBuLoN Finite Element solver and structure computations have been performed.