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Precipitation of γ'' in Inconel 718 alloy from microstructure to mechanical properties.

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Abstract

This paper presents a coupled approach able to describe γ'' precipitation evolution and associated yield strength after various heat treatments in Inconel 718 alloy. The precipitation state is modeled *via* the implementation of classical nucleation and growth theories for plate-shaped particles. The precipitation model is validated through small-angle neutron scattering and transmission electron microscopy experiments. The precipitation size distribution serves as an input parameter to model the yield strength using a micromechanical model based on shear and bypass mechanisms accounting for the particular shapes of the precipitates. Results are in good agreement with measured yield stresses for various precipitation states. A complete simulated TTT diagram of the γ'' phase with the associated yield strength is proposed. The coupled model is finally applied to a series of non-isothermal treatments representative of welding (or additive manufacturing) from the peak aged state.

Keywords: Alloy (Inconel) 718, γ'' phase, Precipitation kinetics, Kampmann-Wagner Numerical (KWN) modeling, Scanning electron microscopy (SEM), Small Angle Neutron Scattering, Structural hardening alloys

1. Introduction

Inconel 718 is a nickel base superalloy mainly used in the aerospace industry to produce critical components for turbines, thanks to its excellent mechanical properties and corrosion resistance at high temperature [1]. Moreover, its good weldability, relative to other superalloys, makes it a very good candidate for assembly parts [2].

Welding or additive manufacturing processes are very complex processes during which the material is subjected to extreme thermomechanical loading, which involves microstructural evolution as grain growth and precipitation (or dissolution) of hardening phases. To optimize the final material properties it is essential to follow the evolution of the microstructure and mechanical properties in the Heat Affected Zone (HAZ).

Its outstanding mechanical properties are due to the fine precipitation of homogeneous hardening intermetallic phases in the nickel solid solution γ . These precipitates are the $L1_2$ face-centered cubic (fcc) structure γ' ($\text{Ni}_3(\text{Ti,Al})$) and DO_{22} body centered tetragonal (bct) structure γ'' (Ni_3Nb). The latter has the following orientation relationships [3]:

$$(001)_{\gamma''} \parallel \{001\}_{\gamma} \text{ and } [100]_{\gamma''} \parallel \langle 100 \rangle_{\gamma} \quad (1)$$

Oblak *et al.* [4] showed that γ' are coherent spherical precipitates and γ'' are coherent disc-shaped particles. This shape has been widely observed by numerous authors via TEM experiments; see [4–8] and more recently [9].

The characterisation of simultaneous γ' and γ'' precipitation is often complicated, as noted by Tian *et al.* [10]. These authors succeed in differentiating γ' and γ'' by using chemical contrast. They did not noted significant γ' precipitation after direct laser additive manufacturing. Several authors have noticed a coprecipitation phenomenon of γ' and γ'' particles. Kindrachuk *et al.* [11], in Inconel 706, and Philipps *et*

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al. [12] in Inconel 718 have shown that the elastic energy of γ'/γ'' is lower than for each precipitate alone. The mechanisms of co-precipitation of these precipitates have been studied recently by Shi *et al.* [13] using phase-field modelling trying to determine the conditions under which the γ'' precipitation is favoured by pre-existing γ' particles. This mechanism is also observed by Detor *et al.* [14] who showed a precipitation of γ'' following γ' . More recently Theska *et al.* [15] have studied various sequences of γ'/γ'' precipitation, especially in the early stages of precipitation [16] and correlated it with the alloy's properties [17].

Strengthening of Inconel 718 super alloys is mainly due to γ'' precipitates. They induce a considerable coherency strain (with a tetragonal distortion $c/a=2.04$), which explains their ellipsoidal disc shape [4]. Moreover, the predominant hardening effect of the γ'' can be also explained by its higher volume fraction ($f_{v\gamma''}/f_{v\gamma'} \approx 4$, which depends greatly on the (Ti+Al)/Nb ratio in the composition of the considered alloy [5].

It is therefore necessary to study the precipitation of the γ'' phase to better understand and predict the microstructure and mechanical behavior of Inconel 718 alloy. Since γ'' are fine and metastable particles, their quantitative characterization is not straightforward. Particle size distributions are relatively well documented thanks to extensive TEM studies [18–20]. However, particle density, volume fraction and/or solubility limits are much more difficult to characterize and very few experimental data are available [7].

The quantitative experimental data on γ'' stability (solubility product, surface energy) are rather scarce in the literature. Mons [21] has proposed a tentative TTT diagram from various literature values, which has been completed with more recent data and shown in Fig. 1. However, to the author's knowledge, no full precipitation time temperature diagram, and associated mechanical properties, is available in the literature. As it can be seen in Fig. 1, the precipitation C-curves for γ'' are rather incomplete and very scattered.

Devaux *et al.* studied the coarsening of γ'' precipitates [9] by extensive TEM image analysis and provided interesting information on the γ'' stability although in a limited temperature range. In a more recent work, Fisk *et al.* [26, 27] simulated the whole precipitation sequence of γ'' (nucleation, growth and coarsening) at 760°C using a mean radius precipitation model. They later used these data as entry parameters for the prediction of yield strength and hardening. The precipitate size distribution was post-calculated with the Lifshitz-Slyozov-Wagner (LSW) theory and accounted for by

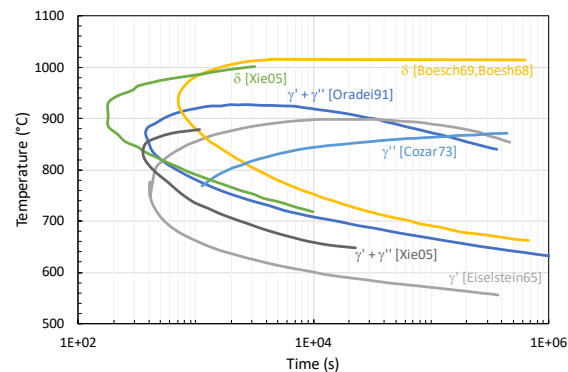


Figure 1: “Schematic” TTT diagram of 718 alloy (data from [Eiselstein65]: [22], [Xie05]: [23], [Boesch69,Boesch68]: [24, 25], [Cozar73]: [5],

weighting the fraction of sheared and bypassed precipitates. However, sheared and bypassed hardening amplitudes were estimated using a mean radius approach, which is known to be a correct estimation for isothermal treatment but fails to accurately describe non-isothermal precipitate size distribution [28–30]. More recently, Fisk *et al.* [31] completed their study and proposed a precipitation model adapted for non-isothermal treatments (*i.e.* welding), notably improving their model from a mean radius approach to an eulerian type full distribution description. Their results present a fair correlation with experimental results of the hardness in a welded joint. Furthermore, their precipitation model is only calibrated on mean radius literature data at a single temperature. A pure growth regime is observed up to 10^4 s which seems contradictory with experimental observations.

Moore *al.* [32, 33] recently proposed a model based on the Kampmann and Wagner numerical model (KWN) [34] for the precipitation of γ'' in nickel alloy 625 and 718. Their model allows to predict the shape factor of the precipitates and showed good results for the distribution of precipitates over several hours.

Matcalc simulations on alloy 718 were also proposed by Drexler *al.* [35] for the combined precipitation of γ' and γ'' . Yield stress prediction were proposed based on the hardening model developed by Ahmadi *et al* [36]. They proposed a simulated Time Temperature Precipitate diagram (represented in Fig. 1) which is in agreement with the measured onset of strengthening over a wide range of temperatures. However, they surprisingly show simultaneous precipitation of γ' and γ'' over all studied temperatures.

Phase field simulations of the precipitation kinetics in

123 alloy 718 have also been performed by Zhou *et al* [37]. 175
124 Although no correlation with experimental data was 176
125 provided, the proposed model interestingly allows to 177
126 simulate the precipitation of both γ' , γ'' and δ phases. 178
127 Phase field simulations along with *ab initio* computa- 179
128 tions have also been proposed [38, 39] to describe inter- 180
129 actions between the γ'' phase and dislocations. They 181
130 detail several complex mechanisms responsible for pre- 182
131 cipitation hardening of this alloy. 183

132 Ahmadi *et al* [40, 41] have developed a model to pre- 184
133 dict the mechanical properties of precipitation harden- 185
134 ing materials and applied it to alloy 718Plus [42]. They 186
135 notably distinguish the strengthening from weak and 187
136 strong shearable precipitates and were able to predict 188
137 the yield limit of the aforementioned alloy for various
138 aging times after heat treatment at 788 °C. 189

139 In summary, many studies on the precipitation kin- 190
140 etics and the consecutive hardening mechanisms have 191
141 been proposed for the γ'' hardened alloys. However, 192
142 most of these studies focus on long heat treatments, rep- 193
143 resentative of the typical aging treatments and service 194
144 life of the alloy for the most common aeronautical ap- 195
145 plications. Furthermore, the kinetics that would be ob- 196
146 served during short term thermal transient, representa- 197
147 tive of manufacturing processes, such as additive man- 198
148 ufacturing or welding have been far less studied. 199

149 In this paper, a multi-class precipitation KWN-type 199
150 model is proposed. Nucleation and growth equations 200
151 are adapted to disc shaped precipitates. The precip- 201
152 itation model is implemented in a "Lagrangian-like" 202
153 model class management software (PreciSo) [28, 29, 203
154 43], which provides a distribution density of precipitates 204
155 for non-isothermal heat treatments. 205

156 This model is then coupled with a yield strength 206
157 model, based on the work of Bardel *et al.* [43], taking 207
158 into account the whole precipitate distribution, the par- 208
159 ticular shape of the γ'' phase, their spatial distribution 209
160 and the competition of two mechanisms of interactions 210
161 with dislocations: bypassing and shearing. In order to 211
162 describe precipitation durations more representative of 212
163 manufacturing processes, experimental results are col- 213
164 lected for times ranging from a few minutes to a few 214
165 hours. 215

166 The simplicity of the proposed models allows their 216
167 integration in macroscopic scale modelling, typically 217
168 for processes simulation [44]. The KWN precipitation 218
169 model and the mechanical model remain yet physically 219
170 based and the model parameters can be linked, although 220
171 not straightforwardly, to physical properties of the mate- 221
172 rials. Finally those models have proven their robustness 222
173 and versatility to model various precipitation and hard- 223
174 ening phenomena (*e.g.* fast non-isothermal treatments).

In section 2, 2.2 and 2.3, the material, associated heat treatments and characterization techniques are presented. Several precipitation states, involving mainly the presence of the γ'' phase are characterized using the Transmission Electron Microscopy (TEM) and Small Angle Neutron Scattering (SANS) techniques. In section 3, the precipitation model, source and choice of material parameters are detailed. Numerical results are confronted, for various heat treatments, with the experimental ones and data from the literature. The mechanical model and comparison with experimental results are presented in section 4. Finally, the model is discussed and simulation results for non isothermal treatments are presented.

2. Materials, heat treatments and methods

2.1. Materials

The alloy 718 bench used in this study was supplied by Safran Aircraft Engines. Its chemical composition is given in Table 1.

The base material shape is a hot forged cylinder of diameter 250 mm and height 214 mm. All samples were taken from this cylinder at constant surface distance to ensure an homogeneous initial microstructure and grain size. The as-received material was previously annealed at 955°C during 1 h and then air-cooled.

2.2. Heat treatments

Heat treatments for each characterization technique have been chosen thanks to the approximate TTT diagram taken from the literature [21] (Fig. 1). The purpose was to maximise the presence of the γ'' phase while limiting the presence of the δ and, to a lesser extent, γ' phase.

The heat treatments were all conducted in a Nabertherm furnace, in an Argon neutral atmosphere to avoid oxidation during treatment. All performed heat treatments and expected phases are listed in Table 2.

Due to the massive geometry of the as-received cylinder (250 mm diameter), a Solution Treatment (ST) (1050°C/1h) followed by water quench (WQ), was further performed on smaller samples (cylinders of 15 mm diameters for the microscopy observations and 0.5 mm thick plates for SANS) to ensure a precipitation free state before any isothermal precipitation treatment.

This solution treatment was eventually followed by a precipitation treatment performed at temperatures T during time t and water quenched. In the following, all precipitation treatments are noted T/t, as-received samples are noted AR and as-received followed by solution treatment are noted AR+ST.

	Ni	Fe	Cr	Nb	Mo	Ti	Al	Co	Si	C	Others
wt%	53.72	18.22	17.84	5.42	2.91	0.93	0.46	0.16	0.11	0.02	0.21
at%	53.16	18.95	9.93	3.39	1.76	1.13	0.99	0.16	0.23	0.10	0.22

Table 1: Chemical composition of the Inconel 718 studied alloy (as provided by Safran Aircraft Engines - material certificate).

Experimental technique	Thermal treatment	Expected phase					
		γ	MC	γ'	γ''	δ	Laves
TEM	AR+ST+760°C/1h	✓	✓	✓	✓	✗	✗
	AR+ST+760°C/2h	✓	✓	✓	✓	✗	✗
	AR+ST+720°C/2h	✓	✓	✓	✓	✗	✗
	AR+ST+720°C/4h	✓	✓	✓	✓	✗	✗
SANS	AR+ST+760°C/30min	✓	✓	✓	?	✗	✗
	AR+ST+760°C/1h	✓	✓	✓	✓	✗	✗
	AR+ST+760°C/2h	✓	✓	✓	✓	?	✗
	AR+ST+720°C/9min	✓	✓	?	?	✗	✗
	AR+ST+720°C/18min	✓	✓	✓	?	✗	✗
	AR+ST+720°C/2h	✓	✓	✓	✓	✗	✗
	AR+ST+720°C/4h	✓	✓	✓	✓	✗	✗
Tensile tests	AR+ST+720°C/6h	✓	✓	✓	✓	?	✗
	AR	✓	✓	?	?	?	✗
	ST	✓	✓	✗	✗	✗	✗
	AR+ST+760°C/1h	✓	✓	✓	✓	✗	✗
	AR+ST+760°C/2h	✓	✓	✓	✓	✗	✗
	AR+ST+720°C/1h	✓	✓	✓	✓	✗	✗
	AR+ST+720°C/1h	✓	✓	✓	✓	✗	✗
	AR+ST+720°C/2h	✓	✓	✓	✓	✗	✗
	AR+ST+720°C/4h	✓	✓	✓	✓	✗	✗
	AR+ST+720°C/7h30min	✓	✓	✓	✓	✗	✗
AR+ST+720°C/15h15min	✓	✓	✓	✓	✗	✗	

Table 2: Heat treatments for each characterization technique. AR: as received. ST: Solid Solution Treatment (1050°C/1h/WQ)

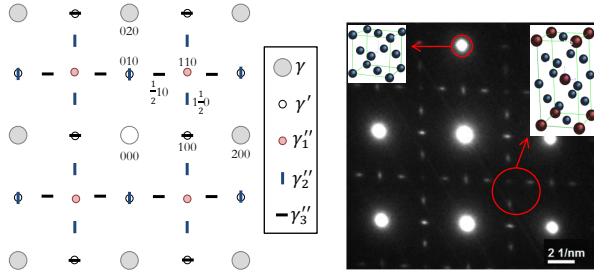


Figure 2: Right: schematic Selected Area Diffraction (SAD) patterns. $[001]_{\gamma}$ orientation. Left: experimental SAD patterns after $760^{\circ}\text{C}/2\text{h}$ precipitation treatment (see Table 2), zone axis: $[001]$.

2.3. Characterization techniques

In order to measure γ'' precipitates size distribution, two complementary techniques were used. Small Angle Neutron Scattering (SANS) measurements is based on the analysis of a large volume, providing statistically accurate data on precipitation radius distribution.

2.3.1. Transmission Electron Microscopy (TEM)

This technique was used to determine the γ'' phase size distribution and the shape factor $q = 2r_p/T_p$, where r_p is the radius and T_p is the thickness of the precipitate platelets. These results will be used as validation data for the precipitation modeling (see section 3). TEM characterizations were conducted on a JEOL 2010F microscope operating at 200 kV at the Consortium Lyonnais de Microscopie (CLYM) located at the University of Lyon (France).

Samples were extracted from the heat treated samples, manually polished to reach a thickness of 0.15 mm, and punched to extract usual TEM discs with a 3 mm diameter. Discs were then electro-polished in a Struers Tenupol using A2 Struers electrolyte at -15°C , and 24.5 V.

TEM allows a differentiation between the two hardening intermetallic phases γ' and γ'' , as the diffraction spots of the two phases can clearly be distinguished when the matrix is oriented in the $[001]$ zone axis [8, 19, 45, 46], see (Fig. 2).

With a $\langle 100 \rangle_{\gamma}$ orientation the three γ'' variants give three separate spots corresponding to the superlattice reflections at $\{100\}$, $\{110\}$ and $\{1\ 1/2\ 0\}$ positions [19]. To observe only the γ'' phase $\{1\ 1/2\ 0\}$ superlattice reflection is used, a dark field image reveals only one of the three variants. γ' particles are also observed although no evidence of co-precipitation have been found.

One example on centered $\{1\ 1/2\ 0\}$ superlattice reflection dark field for each studied heat treatment are

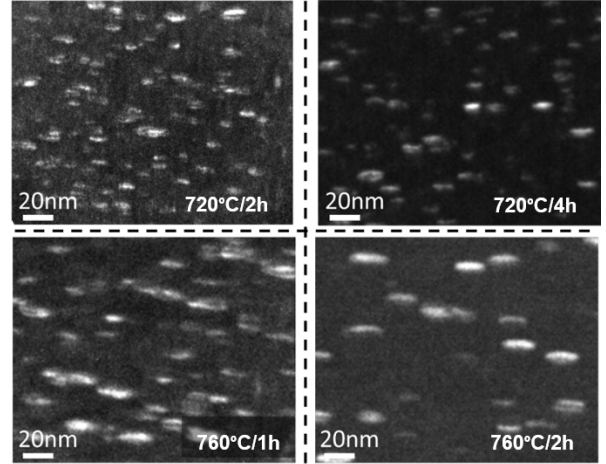


Figure 3: Dark field image using the $\{1\ 1/2\ 0\}$ superlattice reflection (see Fig. 2)

available in Fig. 3. Precipitates are homogeneously distributed in the matrix.

Dark field micrographs were used to determine the radius distributions for two temperatures at two different times. To plot these distributions, clearly visible particles on the dark field images were outlined with ImageJ software. A normalized statistical distribution is obtained, it is then multiplied by the total number of particles given by the model for each studied heat treatments. These experimental results will be represented further in section 3 and compared with modelling outputs.

2.3.2. Small-Angle Neutron Scattering (SANS)

SANS experiment were conducted at the Laboratoire Léon Brillouin (LLB) located in the Commissariat à l'Énergie Atomique (CEA) Saclay. The 7 meter long SANS spectrometer Paxy was used. The samples are rectangular shaped (1 cm \times 1 cm \times 1 mm).

The detector was placed in two different configurations (see Table 3).

The neutron scattering cross section $\frac{d\Sigma}{d\Omega}(q)$ is obtained from the total integrated neutron intensity after background subtraction and normalization by the sample transmission and thickness and by the solid angle. $\frac{d\Sigma}{d\Omega}(q)$ was assumed to be the sum of 3 contributions:

$$\frac{d\Sigma}{d\Omega}(q) = I_{\text{Porod}}(q) + I_{\gamma''}(q) + I_{\text{incoherent}} \quad (2)$$

$I_{\text{Porod}}(q)$ is a $1/q^n$ signal originating from large scale scattering length fluctuations (very large particles, grain

	Configuration 1	Configuration 2
Wavelength (nm)	0.6	0.96
Sample-detector distance (m)	2	5

Table 3: SANS configurations.

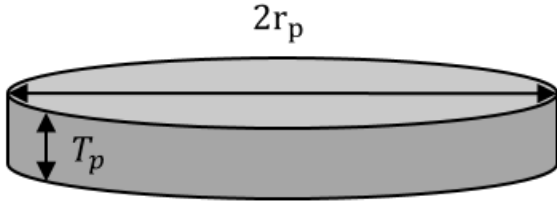


Figure 4: Disc-shaped γ'' precipitates

287 structure, dislocations, segregations...). $I_{\text{incoherent}}$ is a
 288 constant contribution to the signal, which originates
 289 both from the disorder in the solid solution (Laue scatter-
 290 ing) and from the incoherent scattering of the atomic
 291 species.

292 $I_{\gamma''}(q)$ is modeled by the signal of an assembly of
 293 discs of radius r_p and thickness T_p (Fig. 4) with a fixed
 294 aspect ratio and with a log-normal size distribution with
 295 a dispersion parameter of 20% [47, 48]. The aspect ratio
 296 $q = 2r_p/T_p$ is assumed to follow the expression given
 297 by eq. 18 based on experimental results (more on this in
 298 section 3).

299 To account for the interaction between the particles,
 300 resulting in a visible maximum of intensity in some of
 301 the SANS results, we introduce a structure factor in the
 302 decoupling approximation [49] and assume that a hard-
 303 sphere model can reasonably describe the interactions
 304 between these particles. While this may be a strong as-
 305 sumption, the validity of the structure factor model is of
 306 low influence on the obtained particles sizes if the fit is
 307 valid in the high q range where the structure factor is
 308 close to 1.

309 Figure 5 shows the SANS signal obtained on sam-
 310 ples aged at 720 °C and 760 °C for various ageing times
 311 (circles), along with the corresponding fitted intensities
 312 (solid lines).

313 2.3.3. Tensile tests

314 Cylindrical specimens of 6 mm diameter and 40 mm
 315 length were machined from the as-received cylinder.
 316 Samples were heat treated at various times and tem-
 317 peratures corresponding to the microstructure charac-
 318 terization (see Table 2) with 6 samples at 720 °C and

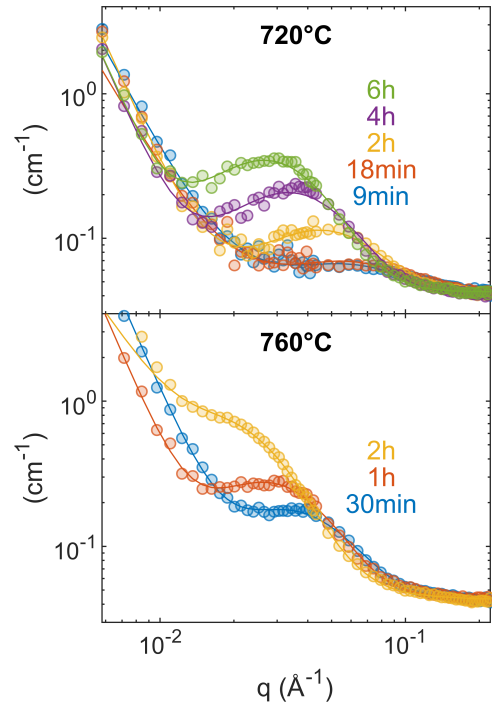


Figure 5: SANS experimental results vs fitted data for various heat treatments (circles) along with the corresponding fitted intensities (solid lines).

319 4 at 760 °C. Tensile tests were carried out on a Schenck 363
 320 Hydraulic 250 kN system with a constant strain rate of 364
 321 10^{-4}s^{-1} . During the tensile test, the strain-rate were 365
 322 controlled by an extensometer HZT071 for which the
 323 gauge length is 10 mm. The true strain is calculated
 324 by using the displacement measured by the extensome-
 325 ter. The experimental yield strengths at 0.2% strain (σ_y)
 326 are represented further in section 4 and compared with
 327 modelling outputs.

328 3. Precipitates modeling

329 3.1. KWN model with disc shaped precipitates

330 Classical Nucleation and Growth Theories (CNGTs)
 331 have been widely used to model the evolution of precipi-
 332 tate size distribution. A numerical model, origi-
 333 nally proposed by Wagner and Kampan [50] is generally
 334 implemented for spherical precipitates [51–53]. In the
 335 case of γ'' phase in 718 alloy, this hypothesis is hardly
 336 acceptable as those precipitates exhibit platelet shapes
 337 with shape factor as large as 5-8 (see Fig. 6). It is there-
 338 fore necessary to adapt the CNGT's equations to accu-
 339 rately model the precipitation of plate-shaped particles.
 340 The KWN model describes the evolution of the precipi-
 341 tate size distribution, discretized in a set of precipitates
 342 classes: at each time step, for each precipitate class i ,
 343 the precipitate radius r_{p_i} and the number N_i are calcu-
 344 lated.

345 This model is based on some important hypotheses:

- 346 • only the γ'' Ni₃Nb phase precipitates, whereas (γ')
 347 and δ phases are ignored. As no co-precipitation of
 348 γ'/γ'' is observed for the current alloy and under
 349 the studied conditions and although some studies
 350 suggest a role of the γ' precipitation especially in
 351 the early stages of γ'' , this effect is here neglected;
- 352 • the nucleation of γ'' in the γ matrix is homoge-
 353 neous;
- 354 • γ'' precipitates are assumed to be discs (Fig. 4);
- 355 • the aspect ratio $q = 2r_p/T_p$ depends linearly on the
 356 precipitate size;
- 357 • only the niobium diffusion in the γ matrix is con-
 358 sidered (*i.e.* nickel is massively present in the ma-
 359 trix and diffuses faster than niobium)

360 Due to the non-spherical shape of the precipitates,
 361 the energy balance of the classical nucleation theory is
 362 modified as follows.

The first step of the model is the determination of the
 free enthalpy (Gibbs energy) variation due to the appari-
 tion of a precipitate of volume V_p and surface S_p :

$$\Delta G = V_p \Delta g + S_p \Gamma = \frac{2\pi \Delta g}{q} r_p^3 + 2\pi \Gamma r_p^2 \left(1 + \frac{2}{q}\right) \quad (3)$$

Where Γ is the surface tension of γ'' precipitates, r_p
 and q are the radius and shape factor of platelets. Note
 that the assumption of thin-disk shaped particles instead
 of oblate ellipsoids does not significantly affect the ex-
 ternal surface of the precipitates, as well as their volume
 (approximation within a few percents of error). The
 driving force Δg is calculated from the solubility prod-
 uct K_S of Ni₃Nb:

$$\Delta g = -\frac{k_B T}{4v_{at}^{\gamma''}} \ln \left[\frac{X_{Ni}^{\gamma}{}^3 X_{Nb}^{\gamma}}{K_S} \right] \quad (4)$$

Where k_B is the Boltzmann's constant, T is the tem-
 perature, $V_{at}^{\gamma''}$ is the atomic volume of γ'' , X_{Ni}^{γ} and X_{Nb}^{γ}
 are the matrix atomic fraction of Ni and Nb, respec-
 tively. The solubility product is usually given by:

$$K_S = X_{Ni}^{\gamma e 3} X_{Nb}^{\gamma e} = 10^{-\frac{A}{T} + B} \quad (5)$$

$X_{Ni}^{\gamma e}$ and $X_{Nb}^{\gamma e}$ are the equilibrium atomic fraction of Ni
 and Nb in the matrix. Parameters A and B are constants
 and determined experimentally.

The critical radius r_p^* above which precipitates are
 stable can be expressed as follow:

$$r_p^* = -\frac{2\Gamma(q+2)}{3\Delta g} \quad (6)$$

The energy barrier for nucleation ΔG^* becomes:

$$\Delta G^* = \frac{16}{3} \pi \frac{\Gamma^3}{\Delta g^2} \frac{(q+2)^3}{18q} \quad (7)$$

Note that eqs. 6 and 7 are slightly different from
 eqs. (22) and (23) of Fisk *et al.* [27], for which disk
 shape precipitates are supposed spherical when $q = 1$.

The nucleation rate is given by the CNGT:

$$\frac{dN}{dt} = N_0 \beta^* Z \exp \left[-\frac{\Delta G^*}{k_B T} \right] \left[1 - \exp \left(-\frac{t}{\tau} \right) \right] \quad (8)$$

where $N_0 = 1/v_{at}^{\gamma}$ is the nucleation site density, V_{at}^{γ} is
 the matrix atomic volume. The condensation rate β^* ,
 the incubation time τ and the Zeldovitch factor Z are
 given by:

$$\beta^* = \frac{4\pi r_p^2}{a^4} \frac{X_{\text{Nb}}^{\gamma''}}{D_{\text{Nb}} X_{\text{Nb}}^{\gamma}} \quad (9)$$

$$\tau = \frac{2}{\pi \beta^* Z^2} \quad (10)$$

$$Z = \frac{v_{\text{at}}^{\gamma''}}{3\pi r_p^2} \sqrt{\frac{\Gamma q(q+2)}{2k_B T}} \quad (11)$$

a is the γ lattice parameter, D_{Nb} is the diffusion coefficient of Nb in the matrix, $X_{\text{Nb}}^{\gamma''} = 0.25$ is the Nb atomic fraction in the precipitate.

The growth equation for disc shaped particles has been adapted from the plate-shaped Zener-Hillert expression [54]:

$$\frac{dr_p}{dt} = \frac{q D_{\text{Nb}}}{2 r_p} \frac{X_{\text{Nb}}^{\gamma} - X_{\text{Nb}}^{\gamma_e}(r_p)}{\alpha X_{\text{Nb}}^{\gamma''} - X_{\text{Nb}}^{\gamma_e}(r_p)} \quad (12)$$

where α is the ratio of the matrix over the precipitate mean atomic volume $\alpha = v_{\text{at}}^{\gamma}/v_{\text{at}}^{\gamma''}$ and $X_{\text{Nb}}^{\gamma_e}(r_p)$ is the equilibrium Nb atomic fraction in γ at the precipitate/matrix interface for a given radius r_p .

In the original Zener-Hillert equation, a multiplicative term of $1 - r_p^*/r_p$ is present to account for capillarity effects. This term is removed here because the Gibbs-Thomson effect is explicitly accounted for (see next paragraph).

To determine the equilibrium solute fractions, the influence of interfaces on equilibrium has to be taken into account: *i.e.* Gibbs-Thomson effect. The Gibbs-Thomson formalism has been adapted for disc-shaped precipitates with the method available in [51]:

$$X_{\text{Ni}}^{\gamma_e^3}(r_p) X_{\text{Nb}}^{\gamma_e}(r_p) = K_S \exp\left[\frac{r_{p0}}{r_p}\right] \quad (13)$$

where r_{p0} is the capillarity radius. It can be expressed as follows:

$$r_{p0} = \frac{4v_{\text{at}}^{\gamma} \Gamma 2(q+2)}{k_B T 3} \quad (14)$$

At the end of each time step, after nucleation and growth calculations, remaining Nb in the matrix can be calculated *via* a mass balance:

$$X_{\text{Nb}}^{\gamma} = \frac{X_{\text{Nb}}^0 - X_{\text{Nb}}^{\gamma''} \alpha f_v}{1 - \alpha f_v} \quad (15)$$

X_{Nb}^0 is the total atomic fraction content of element Nb and f_v is the volume fraction of γ'' precipitates. The volume fraction f_v is calculated from the precipitate distribution given by the numerical model (r_{pi} and N_i):

$$f_v = 2\pi \sum_i \frac{r_{pi}^3 N_i}{q_i} \quad (16)$$

where q_i is the aspect ratio for the precipitate class i (of radius r_{pi}).

Implementation of nucleation and growth equations (eqs. 8 and 12) has been done using PreciSo software as described in [28, 29].

3.2. Calibration of the precipitation model

γ matrix parameters. Sundararaman *et al.* [55] have measured the γ lattice parameter in Inconel 718 with the XRD method. They obtained $a = 3.616 \times 10^{-10}$ m. The matrix mean atomic volume v_{at}^{γ} can then be determined as: $v_{\text{at}}^{\gamma} = a^3/4 = 1.182 \times 10^{-29}$ m³.

Niobium diffusion parameters. The evolution of niobium diffusion coefficient is classically described by an Arrhenius law:

$$D_{\text{Nb}} = D_{\text{Nb}}^0 \exp\left[-\frac{Q}{RT}\right] \quad (17)$$

Pavil *et al.* [56] or Karunaratne *et al.* [57] have proposed diffusion coefficients of niobium in pure nickel which are not necessarily valid for highly alloyed materials such as Inconel 718 [58]. Following the example of Devaux *et al.* [9] and Low *et al.* [59] the pre-exponential factor D_{Nb}^0 has been fixed to 8.8×10^{-5} m²·s⁻¹. The activation energy Q has been found equal to 272 kJ·mol⁻¹ by Devaux *et al.* [9] and 286 kJ·mol⁻¹ by Han *et al.* [60] for respective niobium weight fractions of 5.3% and 4.94%. Here, and in a similar way to Low *et al.* [59], Q was taken as 263 kJ⁻¹ for a niobium weight fraction of 5.42%.

γ'' precipitate parameters. The composition of γ'' precipitates has been measured by Miller *et al.* [61] using Atom Probe Tomography: they found 74 at% Ni, 17 at% Nb among many other elements. For the sake of simplicity, the stoichiometric binary Ni₃Nb composition is used here. The γ'' lattice parameters have been reported by Wagner and Hall [50], the resulting mean atomic volume is $v_{\text{at}}^{\gamma''} = 1.157 \times 10^{-29}$ m³.

Devaux *et al.* [9] calculated the γ/γ'' interfacial energy as $\Gamma = (95 \pm 17)$ mJ/m². Here, a value of 100 mJ/m², consistent with Devaux's calculation was chosen.

A comparison between the experimental aspect ratio versus mean radius evolution obtained in this study and the literature data is presented in Fig.6. TEM measured aspect ratios are in agreement with the literature

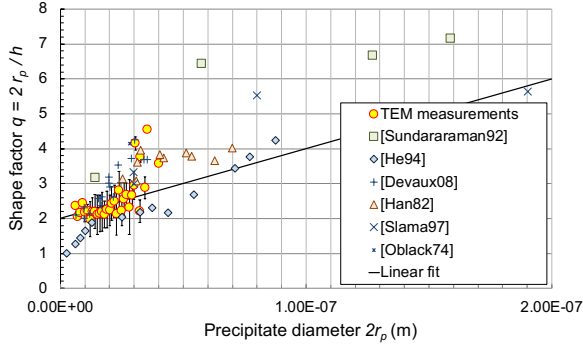


Figure 6: aspect ratio vs precipitate diameter. TEM results compared with literature data

460 results. A large discrepancy of values is observed from 461 the different sources, certainly due to the large period 462 of time over which data were collected (and associated 463 progresses in measurement techniques). For the sake of 464 simplicity a simple linear fit describing the evolution 465 of the aspect ratio with the precipitate radius has been 466 implemented in the model:

$$q = 2 \times 10^7 \text{ m}^{-1} \times 2r_p + 2 \quad (18)$$

467 In this approach, the precipitate aspect ratio q is up- 468 dated according to eq. 18 after each time step.

Various literature studies have proposed measure- 494 ments of niobium concentration in Inconel 718 matrix 495 at equilibrium [9, 35, 62, 63] with a rather large dis- 496 crepancy. Fisk *et al.* [31] have proposed a tempera- 497 ture dependant relation for the equilibrium concentra- 498 tion of niobium in their precipitation model. The sol- 499 ubility product of the γ'' phase is therefore classically 500 described as the following equation, consistent with 501 the literature data and the temperature dependency pro- 502 posed by Fisk *et al.* [31].

$$\log_{10}(Ks) = -\frac{A}{T} + B \quad (19)$$

469 with $A=3294 \text{ K}$ and $B=0.3997$.

470 All precipitation model parameters are listed in Ta- 471 ble 4.

472 3.3. Precipitation simulation: results and discussion

473 From an initial supersaturated solid solution, three 474 isothermal precipitation treatments have been modeled 475 at 660, 720 and 760 °C. Figure 7 shows the compari- 476 son between predicted and experimental mean radii evo- 477 lution with time. Predicted values of precipitate radi- 478 us show the classical growth ($\propto t^{1/2}$) and coarsening 479 ($\propto t^{1/3}$) regimes. It can be noticed that increasing

Parameter	Value	Ref.
a (m)	3.616×10^{-10}	[55]
v_{at}^γ (m^3)	1.182×10^{-29}	[55]
D_{Nb}^0 (m^2/s)	8.8×10^{-5}	[57], [9]
Q (kJ/mol)	263	[9], [60], [59]
$v_{at}^{\gamma''}$ (m^3)	1.157×10^{-29}	[50]
Γ (mJ/m^2)	100	[9]
A (K)	3294	[31], this work
B	0.3997	[9, 31, 35, 62, 63], this work

Table 4: Precipitation model input parameter synthesis

480 temperature accelerates the precipitation kinetics as the 481 three investigated temperatures range in the diffusion 482 limited domain (lower part of the C-curve).

For the three investigated temperatures, simulated 483 radii are in good agreement with SANS and TEM mea- 484 surements, as well as other experimental data from the 485 literature. The model predicts a growth to coarsening 486 transition at approximately 100 s for the three studied 487 temperatures and almost all experimental values fall in 488 the coarsening regime, as in the study of Fisk *et al.*, for 489 which a mean radius model [27] is used. However, in 490 a more recent paper, Fisk *et al.* used a more elabo- 491 rate precipitation model (KWN-type) [31] that is com- 492 pared with data only in the pure growth regime (below 493 2×10^4 s) with another set of simulation parameters at 494 750 °C only.

The volume fraction and number density of precip- 495 itates as a function of time are given in Fig. 8 for the 496 three investigated temperatures: 660, 720 and 760 °C. 497 Note that the final precipitate volume fraction at 760 °C 498 is approx. 9%, whereas Fisk *et al.*[27, 31] find a value 499 of 12% at 760 °C and Drexler *et al.* [35] a value of \approx 500 10% at 720 °C and 11% at 620 °C. There is however 501 a larger disagreement on the characteristic precipitation 502 time t_{50} (time to precipitate 50% of the maximum vol- 503 ume fraction), for which we find 50 s and Fisk *et al.* find 504 1000 s at 760 °C. However, Drexler *et al.* found a t_{50} of 505 about 700 s at 800 °C, which is in good agreement with 506 the 400 s from our model. Note that the precipitation 507 time is extremely sensitive to temperature in the upper 508 part of the C-curve (above 750 °C), as shown in the TTT 509 diagram presented later on.

Experimental precipitate distributions were obtained 510 from TEM. They are compared with modeled distri- 511 butions in Fig. 9, where a general agreement can be 512 observed. Experimental distributions exhibit a tail for 513 large precipitates that is not predicted by the precip- 514 itation model. The model also predicts more small 515 precipitates. The experimental size distribution has a

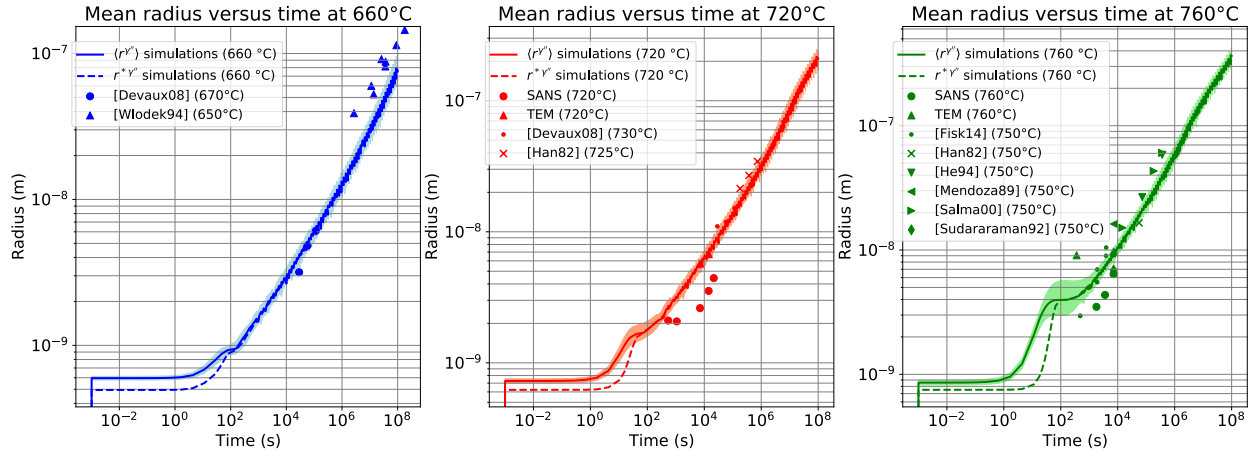


Figure 7: Mean radius as a function of time for different treatment temperatures. Simulation versus experimental data (shaded colors represent simulation results at $\pm 10^\circ\text{C}$).

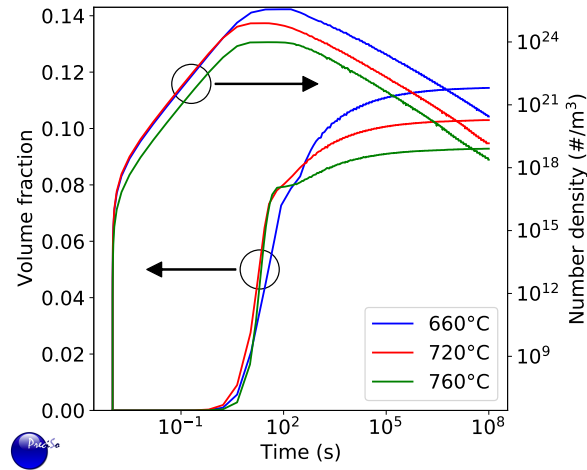


Figure 8: Simulated volume fraction and number of precipitates versus of time and treatment temperature.

519 log-normal shape, whereas the simulation has a LSW
 520 shape. These differences are classically observed and
 521 have rarely been explained (see ref. [64]). Some possi-
 522 ble causes of are: (i) small precipitates are not seen by
 523 TEM; (ii) large precipitates seen by TEM may be the
 524 results of diffusion short-circuits not accounted for in
 525 the precipitation model; (iii) LSW shape is based on the
 526 non-impingement of diffusion fields, which may not be
 527 the case at high precipitate volume fraction.

528 4. Yield strength estimation

529 4.1. Yield strength model: presentation and calibration

530 In this section a model based on the interaction be-
 531 tween dislocations and defects is proposed in order to
 532 calculate the yield strength. The influence of the γ' pre-
 533 cipitates, not modelled in this study, on the mechanical
 534 properties is neglected as currently assumed in the lit-
 535 erature. The classical formulation, originally proposed
 536 by Friedel [65], and improved by Kocks *et al.* [66] and
 537 Deschamps *et al.* [67] is used to take into account the
 538 precipitation state:

$$\sigma_y = \sigma_0 + \Delta\sigma_{SS} + \Delta\sigma_p \quad (20)$$

539 where σ_0 is the yield strength of the base material. It
 540 includes Peierls-Nabarro stress, the forest dislocation
 541 contribution, the Hall-Petch effect, and the solid so-
 542 lution strengthening of alloying elements, except Nb .
 543 This contribution is supposed constant during all heat
 544 treatments since the initial solution treatment anneals
 545 the dislocations and leads to a relatively large grain size

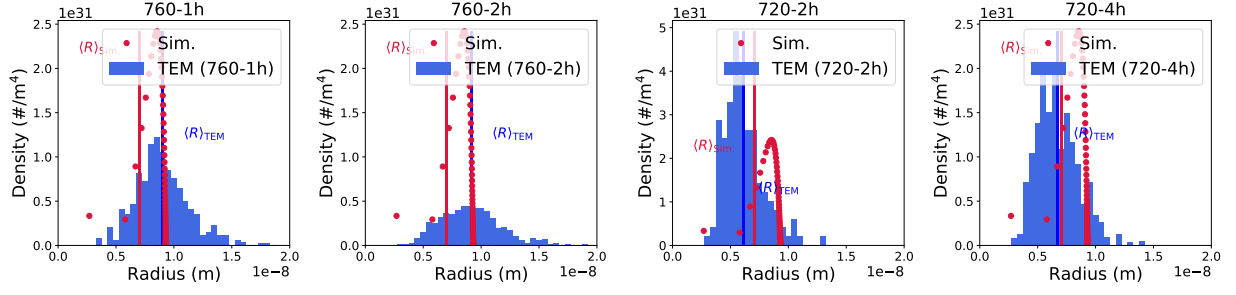


Figure 9: Precipitate radius distribution: simulation vs experimental data from TEM.

546 that therefore slightly changes during subsequent treat-
 547 ments. $\Delta\sigma_{SS}$ and $\Delta\sigma_p$ are the contributions of Nb in
 548 solid solution and γ'' precipitates, respectively. These
 549 two contributions are linearly summed up according to
 550 Kocks *et al.* [66].

551 4.1.1. Solute niobium content contribution $\Delta\sigma_{SS}^{Nb}$

552 Mishima *et al.* [68] studied the effect of various sol-
 553 ute elements in Nickel alloys on yield strength. They
 554 proposed the following relation:

$$555 \Delta\sigma_{SS}^{Nb} = A_{Nb} \sqrt{X_{Nb}^{\gamma}} \quad (21)$$

556 where X_{Nb}^{γ} is the Nb atomic concentration (in at%) and
 $A_{Nb} = 1.17 \times 10^9 \text{ Pa}\cdot\text{at}\%^{-0.5}$ is a constant.

557 4.1.2. Constant contribution σ_0

558 To determine the yield strength of the base material,
 559 the annealed sample yield strength (treatment AR+ST,
 560 see 2) is used. In this precipitate free sample, $\sigma_y = \sigma_0 +$
 561 $\Delta\sigma_{SS}^{Nb} = 352 \text{ MPa}$. From the Nb content of the alloy and
 562 using eq. 21, the constant contribution σ_0 is estimated
 563 at 137 MPa.

564 4.1.3. Precipitation hardening $\Delta\sigma_p$

565 The precipitate hardening can be written as fol-
 566 lows [66, 67]:

$$567 \Delta\sigma_p = \frac{M\bar{F}}{b\bar{L}} = \frac{M}{b\bar{L}} \frac{\sum_i N_i(r_{pi})F_i(r_{pi})}{\sum_i N_i(r_{pi})} \quad (22)$$

568 where \bar{F} is the mean obstacle strength and \bar{L} is the mean
 569 particle spacing. To take into account the whole γ''
 570 radius distribution, the summation of each precipitate
 571 class i contribution is performed using $N_i(r_{pi})$, the num-
 572 ber density of precipitates of radius r_{pi} and $F(r_{pi})$ the
 573 force acting on precipitates of radius r_{pi} .

574 Depending on their radius, precipitates can be either
 sheared (small precipitates) or bypassed (large ones).

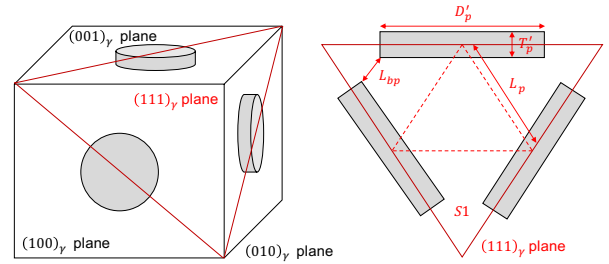


Figure 10: Disposition of plate precipitates in the slip plane

575 *Bypassed precipitates.* For bypassed precipitate class i ,
 576 the force acting on the dislocation is:

$$577 F_i^{bp}(r_{pi}) = 2\beta Gb^2 \quad (23)$$

578 where β is a constant parameter usually assumed to be
 579 equal to 0.5 ([67, 69]). However, according to Brown *et*
 580 *al.* [70], $\beta = 0.25$ is a more realistic value, which will
 be used here.

581 The mean particle spacing is a key parameter. It
 582 strongly depends on the precipitate density, orientation
 583 and shape. For precipitate plates oriented in the $\{100\}_\gamma$
 584 and $\{111\}_\gamma$ slip planes, Nie *et al.* [71] proposed a rela-
 585 tion between the distance between particle centres L_p
 586 and the space left for the dislocation to bypass precipi-
 587 tates L_{bp} (see Fig. 10):

$$588 L_{bp} = L_p - \frac{D'_p}{2} - \frac{\sqrt{3}}{2} T'_p \quad (24)$$

589 where D'_p and T'_p are the mean bypassed precipitate di-
 590 ameter and thickness as seen by the dislocation moving
 591 in the slip plane (see Fig. 10). Thickness T'_p is related
 592 to the precipitate thickness through the angle θ between
 $\{100\}_\gamma$ and $\{111\}_\gamma$ ($\cos \theta = 1/\sqrt{3}$):

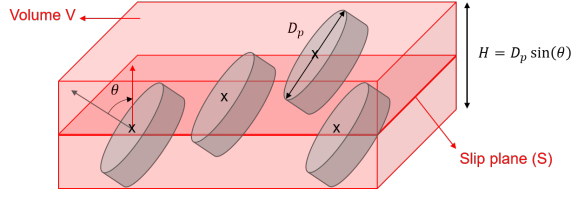


Figure 11: Intersection of the slip plane with plates of diameter D_p in the parallelepiped of thickness H

$$T'_p = \frac{T_p}{\sin \theta} = T_p \sqrt{\frac{3}{2}} = \frac{3}{\sqrt{2}} \frac{\sum_{i>i_c} N_i r_{pi} \frac{r_{pi}}{q_i}}{\sum_{i>i_c} N_i} \quad (25)$$

where r_{pi} , N_i and q_i are the radius, the number and aspect ratio of precipitate class i , and i_c is the critical precipitate class at which the dislocation changes from shear to bypass mechanism.

The average intersect diameter of a plate of diameter D_p intersecting a given plane is $\pi D_p/4$. The mean diameter D'_p is then given by:

$$D'_p = \frac{\pi D_p}{4} = \frac{\pi}{2} \frac{\sum_{i>i_c} N_i r_{pi}}{\sum_{i>i_c} N_i} \quad (26)$$

To determine the distance L_p between particles centres, the number of precipitates per unit area in the slip plane N_a is determined in two different ways. First, Fig. 10 shows that $3/2$ precipitates are lying in the equilateral triangle of surface $L_p^2 \sqrt{3}$, leading to:

$$N_a = \frac{\sqrt{3}}{2L_p^2} \quad (27)$$

Then, N_a can be estimated considering the number of particles intersecting a plane of unit surface. The centre of these particles is located within a parallelepiped of thickness $D_p \sin \theta$ (see Fig. 11). The number of precipitates per unit area is then related to the number of precipitates per unit volume N_v via:

$$N_a = N_v D_p \sin \theta = N_v D_p \sqrt{\frac{2}{3}} = \sqrt{\frac{8}{3}} \sum_{i>i_c} N_i r_{pi} \quad (28)$$

with N_v the number of precipitates per unit volume.

The distance L_p can then be expressed as:

$$L_p = \sqrt{\frac{3}{4 \sqrt{2} \sum_{i>i_c} N_i r_{pi}}} \quad (29)$$

Finally, using eqs. 24, 25, 26 and 29, L_{bp} can be expressed with the precipitation model outputs:

$$L_{bp} = \sqrt{\frac{3}{4 \sqrt{2} \sum_{i>i_c} N_i r_{pi}}} - \frac{\pi \sum_{i>i_c} N_i r_{pi}}{4 \sum_{i>i_c} N_i} - \frac{3}{\sqrt{2}} \frac{\sum_{i>i_c} N_i \frac{r_{pi}}{q_i}}{\sum_{i>i_c} N_i} \quad (30)$$

Sheared precipitates. For sheared precipitates, Friedel's approach [65] is modified to account for non-spherical precipitates. The intersection surface between precipitates and the slip plane is assumed to be a rectangle of surface $D'_p T'_p$ (see Fig. 10). A disc of radius r_p^{eq} and equivalent surface is considered, leading to:

$$r_p^{eq} = \sqrt{\frac{D'_p T'_p}{\pi}} = \left(\frac{3}{2}\right)^{\frac{1}{4}} \frac{r_p}{\sqrt{q}} \quad (31)$$

The force acting on the sheared precipitates of mean intercept radius \bar{r} is given by:

$$F_i^{sh} = kGb\bar{r} \quad (32)$$

The constant term k that drives the shear force is commonly derived from the critical radius r_c , assuming that for a precipitate of equivalent radius r_c , shearing and bypassing forces acting on the precipitate are equal. Chaturvedi *et al.* determined this transition radius as $r_c = 12$ nm through creep tests [6] and this value is commonly used in the literature [26, 31]. It is also consistent with the TEM observation (Fig. 3). This would lead to a value of k close to 0.015. The constant term k could also be determined from refs. [4]. In this study, the antiphase boundary energy $\Gamma_{\gamma''} = kGb/2 = 296$ mJ/m² was measured, leading to $k = 0.029$. By adjustment with the yield stress experimental results and consistently with the literature, the value of k was finally chosen equal to 0.02.

Accounting for the averaged sheared surface of plates, for the orientation of the shear plane and assuming that the force only depends on the sheared surface, the average force acting on the dislocation is then:

$$F^{sh} = \frac{\sum_{i<i_c} N_i F_i^{sh}}{\sum_{i<i_c} N_i} = kGb \left(\frac{3}{2}\right)^{\frac{1}{4}} \frac{\sum_{i<i_c} N_i r_{pi} q_i^{-0.5}}{\sum_{i<i_c} N_i} \quad (33)$$

If precipitates are sheared, the distance L_{sh} between obstacles that interact with the dislocation line depends on the radius of curvature of the dislocation, which depends itself on the applied stress. The argument of Friedel [65] is that, on average the area A swept by the dislocation after breaking free of an obstacle contains

649 exactly one obstacle, so $AN_a = 1$. The area A can be es-
 650 timated from geometrical considerations (see ref. [72]
 651 for calculation detail). The former consideration leads
 652 to an estimation of the spacing of precipitates centres.

653 The average force acting on the dislocation is then:

$$\begin{aligned} L_{sh} &= \sqrt{\frac{4\Gamma_l}{\sqrt{3}}} F^{sh-0.5} L_p \\ &= F^{sh-0.5} \sqrt{\frac{\Gamma_l \sqrt{3}}{\sqrt{2}}} \left(\sum_{i < r_c} N_i r_{pi} \right)^{-0.5} \end{aligned} \quad (34)$$

654 with Γ_l the line tension $\Gamma_l = \beta G b^2$.

655 *Summary.* For bypassed precipitates:

$$\begin{aligned} \Delta\sigma_p^{bp} &= 2M\beta G b \left[\sqrt{\frac{3}{4\sqrt{2} \sum_{i > r_c} N_i r_{pi}}} \right. \\ &\quad \left. - \frac{\pi \sum_{i > r_c} N_i r_{pi}}{4 \sum_{i > r_c} N_i} - \frac{3 \sum_{i > r_c} N_i \frac{r_{pi}}{q_i}}{\sqrt{2} \sum_{i > r_c} N_i} \right]^{-1} \end{aligned} \quad (35)$$

656 For sheared precipitates:

$$\begin{aligned} \Delta\sigma_p^{sh} &= M (kG)^{\frac{3}{2}} \left(\frac{3}{2} \right)^{\frac{1}{8}} \sqrt{\frac{b}{\Gamma_l}} \left(\sum_{i < r_c} N_i r_{pi} \right)^{-0.5} \\ &\quad \left[\frac{\sum_{i < r_c} N_i r_{pi} q_i^{-0.5}}{\sum_{i < r_c} N_i} \right]^{\frac{3}{2}} \end{aligned} \quad (36)$$

657 The precipitate shearing and bypassing contributions
 658 are added according to a quadratic summation law as
 659 in refs. [67] and [66], although other authors (see e.g.
 660 [31]) use a linear summation. The result is then put into
 661 eq. (20) to obtain the overall yield strength.

662 The input parameters for the yield strength model are
 663 summarized in Table 5.

664 4.2. Yield strength estimation results and discussion

665 The comparison between experimental and simulated
 666 yield strength is shown in Fig. 12 for precipitation treat-
 667 ments performed at 660, 720 and 760 °C. The point at
 668 0 s holding time corresponds to the AR+ST treatment
 669 (solid solution).

670 The experimental yield strength was determined from
 671 tensile tests and error bars corresponds to twice the stan-
 672 dard deviation. The predicted values of tensile strength
 673 are in very good agreement with the experimental data
 674 for 720 °C and 760 °C.

Parameter	Value	Source
σ_0 (MPa)	137	This study
M	3.06	[67, 73–75]
G (GPa)	80	[76]
b (m)	2.54×10^{-10}	[4]
A_{Nb} (MPa.at $\%^{-0.5}$)	1170	[68]
β	0.25	[70]
r_c (nm)	12	[6]
k	0.02	[4, 6]

Table 5: Yield strength model input parameters

675 Figure 12 shows all contributions of hardening
 676 (eq. (20)). The contribution of niobium solute atoms
 677 decreases from 215 MPa to 120 MPa for 720 °C and to
 678 146 MPa for 760 °C as precipitation occurs (see Fig. 8).

679 The precipitate hardening contributions $\Delta\sigma_p^{bp}$ and
 680 $\Delta\sigma_p^{sh}$ depend on precipitate size distributions. The by-
 681 passed precipitates contribution remains zero until the
 682 largest precipitate class reaches the critical radius r_c .
 683 At this moment $\Delta\sigma_p^{sh}$ reaches its maximum value and
 684 decreases until most precipitate size classes exceed r_c ,
 685 leading to a negligible contribution of sheared precip-
 686 itates $\Delta\sigma_p^{sh}$. The maximum total yield strength oc-
 687 curs when the mean radius reaches r_c , as usually ob-
 688 served for structural hardening alloy with sheared and
 689 bypassed precipitates.

690 This coupled approach between a multi-class precip-
 691 itation model and a hardening model provides a re-
 692 markable agreement in the final yield strength predic-
 693 tion. Note that the precipitation model output (precip-
 694 itate size distribution) has been checked and validated
 695 independently by TEM and SANS observations.

696 The whole precipitate size distribution contributes
 697 to the structural hardening because the average dis-
 698 tance between precipitates directly depends on precip-
 699 itates number density, which itself is not satisfactory
 700 described by a mean radius precipitation model (see
 701 [28, 29]).

702 The yield strength model presented in this section is
 703 a simplification of the true physical phenomenon acting
 704 in the alloy 718. Indeed, Oblak *et al.* [4, 77] observed
 705 that shearing of γ'' precipitates was possible only con-
 706 sidering doublets or quadruplets of dislocations. They
 707 proposed a model, based on the mechanical equilibrium
 708 of two (or four) interacting dislocations shearing γ''
 709 precipitates. Unfortunately, this model leads to a lower
 710 hardening with a quadruplet than with one single dis-
 711 location, which is not satisfactory to explain the very
 712 good resistance of γ'' precipitates. Later, Sundarara-
 713 man *et al.* [78] completed this model but again the

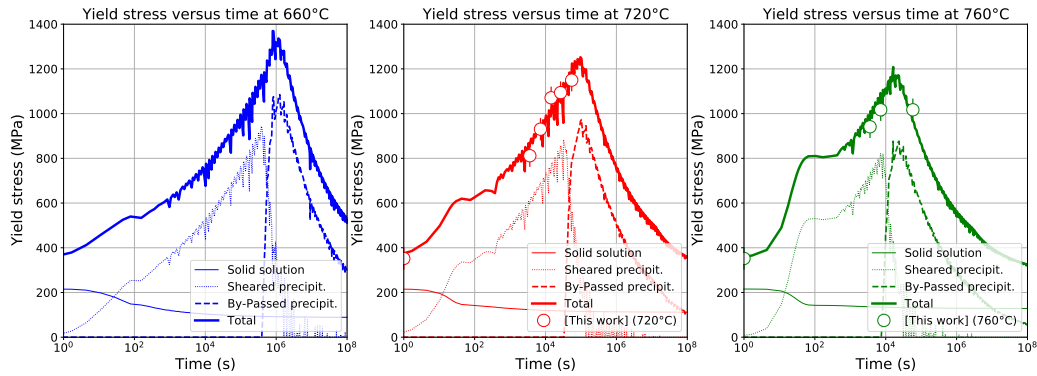


Figure 12: Yield strength in function of holding time: simulation versus experimental data

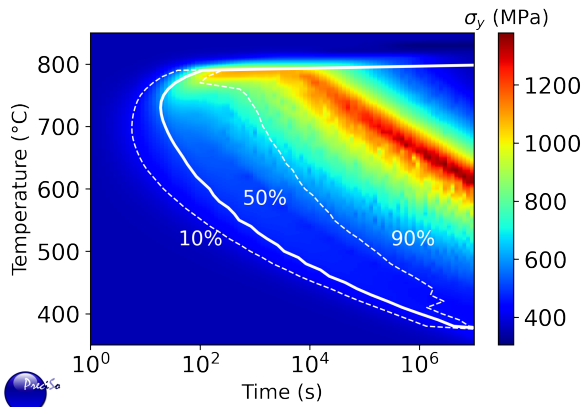


Figure 13: TTT diagram coupled with yield strength model. The model shows that although precipitation is rather fast, the hardening peak is obtained after relatively long treatment.

714 shear strength for four dislocations was found smaller
 715 than for the crossing of one single dislocation. In this
 716 study a "classical" single dislocation model was consid-
 717 ered with an antiphase boundary energy of the γ'' phase
 718 equal to the value measured by [77] and used by [55]
 719 and [78]. More sophisticated approaches have been re-
 720 cently proposed by Ahmadi *et al.* [36, 40–42]. How-
 721 ever, they lead to similar results, justifying thus the use
 722 of our simpler approach.

723 The precipitation and yield strength models used to
 724 plot the TTT diagram for γ'' precipitation reaction, su-
 725 perimposed with the evolution of yield stress. This dia-
 726 gram is presented in Fig. 13.

727 From this TTT diagram, we can see a much faster
 728 precipitation compared to previous TTT diagrams (see
 729 figure 1). However, considering the yield strength val-

730 ues represented at every point of this diagram, it can
 731 be observed that the maximal yield strength is reached
 732 much later than the precipitation equilibrium volume
 733 fraction, apart from temperatures near 780 °C, for which
 734 the δ phase is expected to replace γ'' . For most temper-
 735 atures, the yield strength reaches its maximal value for
 736 isothermal treatments between 3 h and 3000 h. The ap-
 737 parent contradiction with previous literature data on pre-
 738 cipitation kinetics can now be explained. The numerous
 739 small precipitates appearing quickly are very weak ob-
 740 stacles to the dislocations displacement, which explains
 741 the weak hardening despite the important precipitation.
 742 Precipitates growth (by coarsening) is then necessary
 743 to reach the maximum hardening. Moreover, recent ex-
 744 periments and simulations performed by Drexler *et al* [35]
 745 confirm this relatively fast (and weak) precipitation.

746 The yield strength prediction on this diagram is in
 747 good agreement with experimental observations of rela-
 748 tively slow hardening for this alloy. The 718 alloy
 749 is indeed known to be an alloy with a slow hardening
 750 behavior compared to others nickel based superalloys
 751 hardened by the γ' phase [2].

752 Industrial hardening treatments typically involve a 8
 753 to 10 h holding between 720 and 760 °C from a super
 754 saturated solid solution state, which is in agreement
 755 with the TTT diagram. Moreover, Ahmadi *et al.* [36]
 756 reach a maximum yield stress after 25 hours at 718 °C,
 757 which exactly corresponds to the hardening peak at this
 758 temperature in Fig. 13.

759 5. Non-isothermal prediction of precipitation and 760 yield strength

761 The knowledge of the precipitate size distribution is
 762 also needed to account for non-isothermal treatments as

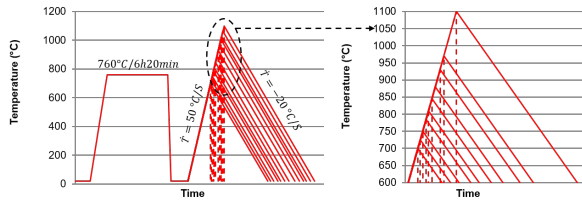


Figure 14: Non-isothermal treatments tested: heating rate of 50 °C up to variable maximum temperature T_{Max} , followed by (i) 20 °C (full line) and, (ii) instantaneous cooling (dashed line).

shown in refs [29, 29, 30]. In this section, the coupled microstructural and yield strength predictions are tested on non-isothermal treatments, which are representative of welding or additive manufacturing processes at various points of the heat affected zone.

From a super saturated solid solution, a precipitation treatment at 760 °C during 6 h 20 min is performed followed by a quick cooling (water quench) (*i.e.* corresponding to the hardness peak). After that, various non-isothermal thermal transients are tested. First a constant heating rate of 50 °C·s⁻¹ is applied up to variable maximum temperature T_{Max} . Two different cooling rates are then applied: 20 °C·s⁻¹ and instantaneous cooling to freeze the microstructure (that would correspond to a water quench). Maximal reached temperatures T_{Max} lie between 700 and 1100 °C. The studied heat treatments are presented in Fig. 14.

At the initial state of peak hardness, the precipitate volume fraction is maximum and equal to 11% with a mean radius close to 14 nm. For heat treatments up to 800 °C this state remains unchanged as the time and temperature are too low to induce any change. Above this temperature, volume fraction and precipitate radius drop as the precipitates start to dissolve. From a maximum temperature of roughly 1000 °C all precipitates have been dissolved at T_{Max} . After water quench, no precipitate remains. After cooling at 20 °C·s⁻¹, nucleation of small precipitates occurs during cooling, leading to a volume fraction of 2%.

Concerning mechanical properties, the initial state represents the hardening peak, essentially due to precipitate strengthening by bypass mechanism. As for the microstructure, nothing occurs until a maximal temperature of 800 °C. Then, a drop of mechanical properties, associated to precipitates shrinkage, is observed. For T_{Max} ranging between 800 °C and 1000 °C, followed by a water quench, the shearing contribution increases as precipitates shrink. Above a reached temperature of 1000 °C only the solid solution hardening remains

as precipitates are fully dissolved. When the heating is followed by a slow cooling, the material remains at high temperature for a longer time and therefore precipitates continue to shrink for a while, leading to a lower precipitate contribution to the overall yield limit. This phenomenon is in competition with the nucleation that occurs during cooling at lower temperatures. However the effect of this secondary precipitation is quite limited (around 100 MPa).

From the non affected zone to the most affected zone (for T_{Max} temperatures larger than 1000 °C), the yield stress drops from 1300 MPa to approx. 450 MPa (200 MPa for the solid solution $\Delta\sigma_{SS}^{Nb}$ and 150 MPa for the small secondary sheared precipitates contributions respectively).

6. Conclusion

A coupled precipitation and yield strength model was developed to describe the microstructural and strengthening evolution occurring during any heat treatment of a 718 Ni based alloy.

A recently developed precipitation model was improved to account for plate-shaped particles. This model gives the particle size distribution, later used as input data for the prediction of the yield strength. The precipitation model was calibrated by SANS and TEM analyses on isothermally treated samples.

All parameters of the coupled models were taken from the literature or experimentally determined. An excellent estimation of the yield strength was obtained by the coupled approach for isothermal treatments. The TTT diagram, coupled with the yield strength model, illustrates the slow structural hardening of the 718 alloy despite the fast precipitation of the γ'' phase.

Non-isothermal treatments, representative of different positions around a weld joint or for any point of part built by additive manufacturing, have been simulated. It has been observed that the yield strength strongly depends on the competition between the dissolution of the precipitates presents before the welding and the nucleation of new precipitates during the cooling.

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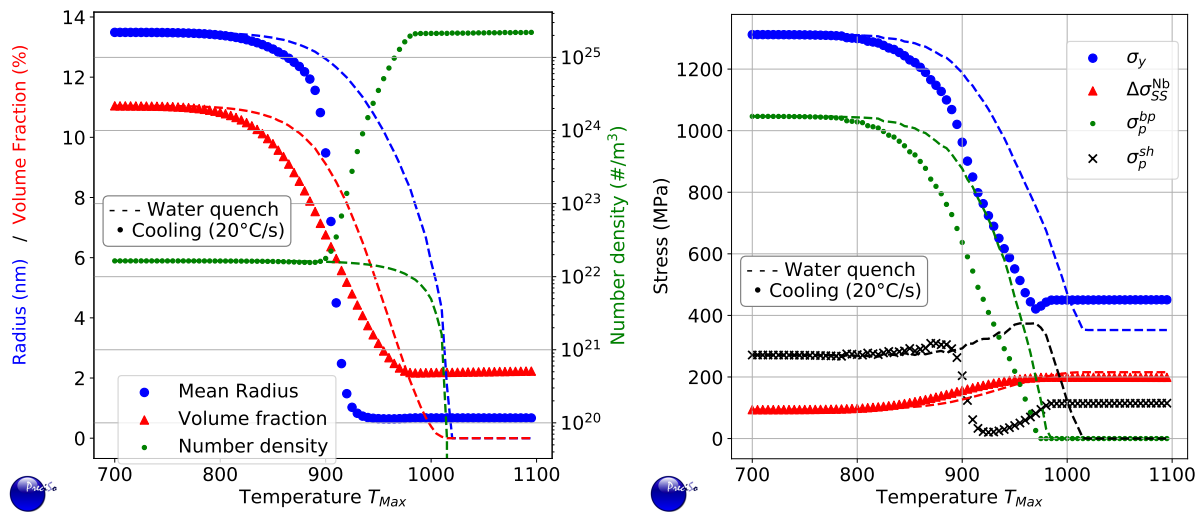


Figure 15: Microstructural and yield strength prediction for non-isothermal treatments

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855 8. Bibliography

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