HIGH TEMPERATURE DEFORMATION BEHAVIOR OF 718PLUSTM:

CONSIDERATION OF γ' EFFECTS

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ABSTRACT

The hot deformation behavior of 718Plus is modelled through a physically based hybrid dislocation density model which includes the effects of precipitating particles. It is well known that the service performance and hot-flow characteristics of this alloy are strongly dependent on the microstructure, particularly the grain size and second phase particles. Thus, comprehension and modelling of the hot flow behavior is an important task.

In precipitation hardening alloys (superalloys, microalloyed steels, etc.) it is particularly more challenging to model the microstructural evolution, in the processing windows, where material softening and precipitation processes take place, concurrently. In this work, the initial stages of the deformation is studied.

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A model based on dislocation density evolution is presented. As in conventional approaches, the dislocation density is considered as a competition between dislocation generation and dynamic recovery at the early stages of deformation. Recovery is assumed to be driven by glide and climb of dislocations which are considered to be proportional to the strain rate and damped by the existence of second phase particles.

It is known that under high temperature deformation conditions, 718Plus may undergo dynamic precipitation. Second-phase particles in the material may impede the grain boundary motion and contribute to an increase in flow-stress. To account for the dynamic precipitation, the present model combines experimental results and precipitation models to predict volume fraction and particle size.

The effect of aging is studied through TEM characterization of the samples from interrupted tests at the onset of Dynamic Recrystallization. Stress contribution due to different modes of dislocation precipitation interaction are modelled and integrated to the phenomenological dislocation density based hardening models

INTRODUCTION

Inconel 718Plus is introduced in early 2000's to respond the needs of increasing service temperatures while keeping the manufacturing costs moderate. Inconel 718Plus presents 50°C higher temperature capability over its ancestor Inconel 718 [1]. The chemical compositions of both alloys are given in Table 1.

TABLE 1. Chemical compositions in *wt. percent* of IN718, 718Plus.

Alloy	Ni	Cr	Mo	W	Co	Fe	Nb	Ti	Al
718	Bal	18.1	2.9	-	-	18	5.4	1	0.45
718Plus TM	Bal	18	2.8	1	9	10	5.4	0.7	1.45

The hot workability and the effect of processing routes on the final service performance of the alloy are

important areas of investigation, especially for the recently developed alloys. Regarding the hot working of 718Plus, the literature is still in development. In contrast, its predecessor Inconel 718 is well studied and a high number of reports can be found in the literature regarding its hot deformation characteristics under different conditions, with different initial microstructures.

A study regarding the hot working response of 718Plus was published previously by the authors [2]. This study is focused on a precipitate-free initial microstructure with relatively large grains (herewith this initial state is referred to as 'baseline'). The focus of the current study, on the other hand, is on the amount of γ' in the initial microstructure and its effect on the deformation behavior. This may be particularly of interest to design the last forging stages in industrial processing of the alloy. The previously-reported baseline behavior and model parameters may be useful to create contrast and understand some particularities found in the current study.

Unlike alloy 718, the main strengthening phase of 718PlusTM is γ' ($\text{Ni}_3[\text{AlNbTi}]$, L12, cubic) instead of γ'' (Ni_3Nb , D022, tetragonal). Above-mentioned increase of the temperature capability is believed to originate from the stability advantage of γ' over γ'' [3–6].

The importance of the δ/η phase, in terms of grain size control and stress rupture properties, is well known in alloy 718. Since the appearance and precipitation behavior of the grain boundary precipitates in 718PlusTM were found to be similar to that of δ phase of 718, they were initially identified as δ phase (Ni_3Nb , D0a, orthorhombic) as well [7–9]. However, it was later studied in depth by Pickering *et al.* [10] and reported that the grain boundary (GB) precipitates mainly consist of layers of η phase ($\text{Ni}_6\text{Nb}[\text{AlTi}]$, D024, hexagonal) and accompanied with fine δ layers. This nomenclature is not important given the focus of the current work.

A TTT diagram of the alloy was previously presented by Xie *et al.* [11]. A partially agreeing Precipitation Time Temperature (PTT) diagram was published by the current author [2]. According to these, γ' precipitation nose is in the range of 900°C - 925°C. Around 950°C the transformation line can be accepted as horizontally

asymptotic. According to the same TTT, supplied by Xie *et al.* [11] the δ solvus temperature is given as 1060°C.

Hot working consists of forming operations conducted at temperatures higher than $0.5T_m$. During these operations different metallurgical processes may take place concurrently and interact with each other. These processes include Work Hardening (WH), Dynamic Recovery (DRV), Dynamic Recrystallization (DRX), and may be accompanied by phase transformations etc. Thus, comprehension of the underlying mechanisms and their relationship with forming parameters is essential to design better processes and to optimize the service performance and production costs.

Starting from the late 60s and early 70s, many studies focusing on the deformation behavior of metals at high homologous temperatures were reported. A review of the studies focusing on mathematical modeling flow curves showing a single peak DRX behavior was published by Ebrahimi and Shafiei [12].

Apart from this, ample amount of studies can be found in the literature regarding the hot deformation behavior of microalloyed steels [13–22]. Although the precipitating phases are different in nature in Ni-base superalloys, these studies are particularly interesting from the perspective of current study because both 718Plus and microalloyed steels (especially with Nb) show similar precipitation - hot deformation behavior.

While one of the most comprehensive studies, regarding the effect of precipitation in microalloyed steels was published by Jonas and Weiss [15] in the late 70s, recently some models were constructed in order to include the effects of second phase particles. Backe *et al.* [23] put effort on modeling precipitation and its effects on dynamic recrystallization in Nb microalloyed steels. Tancrét *et al.* [24], modeled the effect of γ' influence on the retardation of DRX through a net driving force analysis.

This work uses the well known Estrin-Mecking-Bergstrom [25, 26] approach to define the dislocation density as a function of dislocation generation and annihilation/remobilization. In order to model the effect of second phase particles, a third term is added into the dislocation density balance equation relating the precipitate radius

and volume fraction directly to the dislocation density by damping the recovery term.

EXPERIMENTAL PROCEDURE

Cylindrical compression samples with 4, 5 and 7.5 mm diameter s were machined from a 718Plus billet with initial grain size of $62\ \mu\text{m}$. The height to diameter ratio was 1.5 for the samples with 7.5mm diameter while for the rest this value was 2. Baehr 805 A/D deformation dilatometer and Instron 4507 (fitted with Mo punches) were employed to conduct the tests.

Two sets of compression tests were performed. Set 1 consisted of four different temperature and strain rates, namely; 900, 950, 1000 and 1050°C , at strain rates of 0.001, 0.01, 0.1 and 1s^{-1} . Final strain was varied between 0.5 and 0.65 due to the intrinsic limitations of compression testing.

Tests of Set 2 were conducted at 920, 940 with strain rate of 0.01s^{-1} on the samples held at 850°C for a different amounts of time, similar to the approach given in [27]. Deformation was terminated at approximately 0.4 strain in order to stay below the maximum force capability of the equipment. Some samples were quenched slightly before the peak stress in order to preserve the structure before the DRX initiation.

For the Set 1, the cycle consisted of 3 steps. Firstly, the samples were subject to solutionizing for 1 hour at 1050°C and water quenched. The average grain size after this solutionizing treatment was measured to be $73\ \mu\text{m}$. Secondly, a 10-minute long extra solution treatment was applied to avoid any precipitating particles prior to the deformation. This extra solution treatment was followed by cooling down to the deformation temperature (at 1.5°C/s) for compression testing. Testing was started after allowing approximately 3 minutes for homogenization.

For the Set 2, samples solutionized at the same temperature (1050°C) for 1 hour, followed by water quench. Then samples were aged at 850°C (for different amounts of time) followed by water quench. Finally, samples were heated directly to the deformation temperature and compressed after allowing 3-minutes for homogenization.

Tests were interrupted at around $90\pm3\%$ of the peak stress.

The selection of the deformation temperatures in Set 2 were based on TTT diagram of 718Plus [2, 11, 28]. Since the nose of the γ' transformation is around 920°C , this is selected as one condition. Furthermore, since the transformation line for γ' on the TTT diagram becomes asymptotic at 940°C , this is picked as the second condition.

Since the aging temperature (850°C) and strain rate during deformation (0.01s^{-1}) are the same for all samples in Set 2, in the rest of the text these samples will be referred to with their deformation temperature and prior aging time e.g. a sample deformed at 920°C after 120 minutes of aging is named Sample 920°C -120'.

During the tests, boron nitride powder and Tantalum foils were used to reduce the sample-punch friction and after the compression tests, the samples are quenched with Argon purging with a rate of 7.5°C/s .

For transmission electron microscopy (TEM) analysis, the specimens were thinned with silicon carbide papers down to $20\text{-}30\ \mu\text{m}$ and electropolished with 5% perchloric acid in ethanol at 32 V at -10°C . Some samples were extracted through FIB; however, the final quality was observed to be inferior to the electropolished specimens. TEM examinations were performed with Jeol ARM 200F operated at 200 kV acceleration voltage. All samples were plasma cleaned in order to remove surface oxides and impurities before TEM.

MODELING APPROACH

Work Hardening of Initial Material

In the formalism of work hardening, the following relationship given in Eq. (1), known as Taylor's Equation is accepted to well form the bridge between the flow stress (σ) and dislocation density (ρ).

$$\sigma = \sigma_i + \mu\alpha b\sqrt{\rho} \quad (1)$$

In this relationship, m is orientation/texture factor, α is a proportionality factor, μ is the shear modulus and b is the magnitude of the Burgers vector.

σ_i is usually referred to as friction stress. The definition of this term is somewhat ambiguous [29] even if some authors relate it to the contribution of grain size hardening, precipitation hardening, solution hardening etc. Within this study, as the model results of the aged samples will be compared with the reference (solutionized) condition, the only contribution is assumed to be originating from precipitation hardening. Thus, it will be named σ_p .

To be fully described, the second term in Eq. (1) necessitates a model describing the evolution of the dislocation density fed in. To this end, a model initially proposed by Estrin and Mecking and Bergstrom is adapted and implemented in this study. According to this, the dislocation evolution in the time domain through the Eq. (2):

$$\frac{d\rho}{dt} = U \frac{d\varepsilon}{dt} - \Omega \rho \left[1 - \frac{g}{\sqrt{\rho}} \right] \frac{d\varepsilon}{dt} \quad (2)$$

where t is time and ε is the strain. U is the athermal work hardening in the form of $(= \frac{m}{bL})$. L is the mean free path of dislocations, which they can travel until immobilized by a barrier (pile up) and/or obstacle (second phase/precipitate).

Second term in the Eq. (2) represents dynamic recovery. Ω is a strain independent material constant representing the dislocation re-mobilization in the absence of precipitation. Note the damping term attached to this term which represents the effect due to the precipitation which was first proposed by Engberg *et al.* to limit the softening due to climb only [16, 18, 23]. Here g can be expanded as $g = C (f_{prec}/r_{prec})$, where f_{prec} and r_{prec} represent volume fraction and radius of the second phase particles and C is a parameter, similar to the disloca-

tion density factor introduced by Dutta et al. [20]. Here, it is important to mention that in this work, only up to moderate volume fractions of γ' with spherical morphology are considered, agreeing with the TEM observations.

The mean free path of dislocations is calculated based on the subcell diameter and precipitation. They were assumed to additively contribute to the immobilization of the dislocations which is given in Eq. (3).

$$\frac{1}{L} = \frac{\sqrt{\rho}}{K_{cell}} + \frac{K_{prec}}{L_{prec}} \quad (3)$$

where K_{cell} and K_{prec} are parameters which define the immobilization effectiveness [30]. The original model [31] assumes an evolution from L_{start} to L_{end} . In the current case, this is assumed to be constant, which is a fair considering the original treatment was on ferrite. Moreover, for the solutionized material it is assumed that the cell spacing is smaller than precipitate spacing. Therefore, the hardening contribution from the precipitates is ignored in this case. The exact value of L_{prec} is calculated based on a statistical representation [32] of randomly distributed spherical particles whose reduced form is shown in Eq. (4).

$$L_{prec} = \sqrt{\frac{\log(3)}{2\pi Nr} + \frac{8}{3}r^2} - \frac{8}{3}r^2 \quad (4)$$

where N and r are the number density and particle radius respectively.

Furthermore, in order to define both Eq. 4 and Eq.2, one needs a description of the volume fraction and particle size of the precipitates. These can be obtained through numerical models for precipitation or based on experimental data. The radius data presented by Radis and Zickler *et al.* [33, 34] considering static precipitation are found to under-estimate the second particle radius when compared to the current findings below 940 °C. Thus, the models they presented are calibrated according to γ' radius observed in the deformed samples in this study.

Accordingly, the volume fraction and precipitate radius values are summarized on Table 2.

TABLE 2. γ' Volume Fraction and Radius of Deformed Alloy

Temperature	Vol. Fraction	Radius
$< 940^{\circ}\text{C}$	0.091	$3.1843 t^{1/3}$
$> 940^{\circ}\text{C}$	0.091	$2.9143 t^{1/3}$

MATLAB 2017a was used for the numerical treatment and estimation based on the experimental hot flow curves. The temperature dependence of the magnitude of Burgers vector (b) is ignored and taken as 2.56×10^{-10} m. The constants m and α are assumed to be strain independent and taken as 3.1 (close to the 12 slip system value, 3) and 0.15 respectively. Initial dislocation density, ρ_0 is taken as 1×10^{12} m/m³. The values for hardening recovery rate for Set 2 are calculated based on the previously reported Zener-Hollomon (Z) dependency for Set 1 [2]. Temperature dependency of the shear modulus is considered through Eq. (5) [35].

$$\mu(T) = \mu_0 \exp \left(1 - \frac{T - 300}{T_m} \right) \quad (5)$$

Precipitation Stress and its Components

During (hot) deformation when a dislocation meets a precipitate, it can either loop around the particle, or cut through it. Former is usually called Orowan mechanism, latter occasionally referred to as precipitation shearing. The stress contribution (the stress necessary to make a dislocation overcome all the obstacles on its way) of these mechanisms are given by Eq. (6) and Eq. (7) respectively.

$$\sigma_{Loop} = \frac{2.43\mu b\gamma}{4\pi(L_p - 2r)} \beta \ln\left(\frac{L_p - 2r}{2b\gamma}\right) \quad (6)$$

$$\sigma_{Cut} = \frac{m \mu b \gamma}{r \sqrt{\pi w_q}} k_c \sqrt{f} \left(\frac{2 \gamma_{APB} w_r r}{\mu b^2 \gamma} \right)^{3/2} \quad (7)$$

where μ is shear modulus, b is the magnitude of Burgers vector, L_p is the interparticle spacing, r is the precipitate radius and γ_{APB} is the energy density of the planar anti-phase boundary. w_r and w_q are constants [36]. β and k_c are calibration constants taken from the approach of Fisk *et al.* [30].

Although the activity of these mechanisms can be observed qualitatively through TEM observations, the contribution of each cannot be quantified. Because of this, the overall contribution is assumed to be a weighted average of the two as given in Eq. (8).

$$\sigma_{Total} = f_{Cut} \sigma_{Cut} + (1 - f_{Cut}) \sigma_{Loop} \quad (8)$$

where f_{Cut} is the volume fraction of precipitates passed by shearing mechanism. A straightforward solution would be obtained through calculating the fraction of sheared (cut) precipitates by using a size distribution function and assuming a critical size for mechanism transition from cutting to looping. However, in the current case, an inverse approach is used and f_{Cut} is reported as a function of aging time. Constants β and k_c are found based on the assumption that after two hours of aging at $850^\circ C$ the dominating mechanism is particle looping.

RESULTS AND DISCUSSION

The current text intends to report the two parallel investigations i.e. the characterization and the modeling the deformation behavior from a perspective which prioritize γ' precipitation. To this end, first, some highlights of the TEM/STEM characterization of the undeformed, deformed, aged and un-aged specimens will be given and later the model results will be presented. Some of the results, especially those regarding the effect of γ' on the deformation behavior play an important role in the selection of suitable models and/or selection of plausible values of certain model parameters. These are highlighted in the text when necessary.

Hot Deformation Behavior of Unaged Specimens

The flow curves of the tests belonging to Set 1 described in the Experimental Procedure are given in Fig. 1. In general, flow curves show a classical single peak DRX behavior. No cyclic DRX behavior is observed. Since at higher temperatures the stress to initiate the DRX is lower and precipitation is unfavorable, the nucleation of new strain free grains is expected to be easier. Thus, this is an expected scenario to observe. Moreover, the DRX kinetics are expected to be higher at high temperatures because of the higher grain boundary mobility. When these facts are considered, the obtained results within Set 1 can be considered as natural.

Moreover, it can be said that the strain curves at 900°C and 950°C generally show lower hardening slopes when compared to previously published data [37]. Unfortunately these authors did not share the complete set of data, so it is not possible to make a better comparison.

The material constants defining the work hardening of the material and detailed discussion of the general hot flow behavior is presented elsewhere [2]. Previously apparent values of Ω and U are given as a function of Z . This relationship can be defined through $\Omega = K_{\Omega} Z^{m_{\Omega}}$ and $(\alpha' b)^2 U = K_U Z^{m_U}$. Where K_{Ω} and m_{Ω} were reported as 8020, -0.1155; K_U and m_U as 5.083e-06 and 0.1069, respectively. These DRV rate parameters do not take into

account the precipitation effects as they are apparent in nature.

FIG. 1. Flow curves of Set 1 obtained at 900, 950, 1000, 1050°C and 0.001, 0.01, 0.1 and 1s⁻¹ strain rates.

FIG. 2. Hot flow response of different samples aged at 850 °C for different times. Upper image: 920 °C, $0.01s^{-1}$; lower image: 940 °C, $0.01s^{-1}$.

The Effect of Gamma Prime on the Hot Deformation Behavior

As mentioned earlier, in this investigation an attempt has been made to follow the effects of γ' particles on the high temperature flow behavior. Tests within Set 2 were conducted on samples solutionized at 1050 °C for 1 hour followed by water quenching, then aging at 850 °C different times, finally water quenching prior to the

FIG. 3. Effect of aging on different characteristic points of the flow curve: Strain needed for critical stress (ϵ_C), critical stress (σ_C) and yield strength (σ_Y) are plotted as a function of aging time.

deformation. Resulting flow curves are presented in Fig. (2) until 0.35-0.45 strain.

A few details can be noted immediately by observing the flow curves. Firstly, a convergence in the flow behavior with longer aging times observed after almost two hours of aging. Secondly, some curves (920°C-0', 940 °C-0' and 940 °C-10') present a serration/double yield at the very initial stages of deformation. This is observed in some of the flow curves of Set 1 as well.

The effect of aging seems to manifest itself on the flow curve as if there was an increase of the Z. This is

accompanied by the vanishing of the aforementioned-serration/double yield and a slight change in the slope of the curves in the elastic section. The slope change and the “double yielding” behavior was also observed by Wen et al. [38] who compared the work hardening behavior of a Ni-base superalloy in the solutionized and aged state. This might be related to the diffusion of the γ' forming elements and deformation speed; however, this is out of the scope of the current investigation.

In 1979, Jonas and Weiss [27] presented a report, stating that the consideration of yield strength to observe the effect of precipitation and/or to estimate the precipitation kinetics is not the optimum method due to its insensitivity / oversensitivity. In the current case, yield strength against the aging time is presented; however, they are not used in any quantitative/numerical evaluation. Furthermore, these authors [27] found that strain to peak stress is a more reliable parameter to track the effects of precipitation. The evolution of critical stress, yield strength and strain to critical stress is given in Fig. (3). The critical above analysis is found based on the formulation of Najafizadeh and Jonas [39].

Conversely to the findings of Jonas and Weiss [27], here the strain to critical stress does not decrease to converge a value. The decrease of the critical strain in their case (earlier start of DRX) is explained by the lack of dynamic precipitation. When the sample is held before deformation, as the static precipitation evolves, the matrix remains depleted of precipitate forming elements, thus reducing the potential of dynamic precipitation.

In the current investigation, the opposite is observed, i.e. longer aging times result in delays in the critical strain. While this is not the core point of the current work, this might a result of a combination of few facts. First, the precipitating phases in Nb microalloyed steels (Nb(C, N)) and superalloys are different in nature when their relationship with the matrix is considered. The coherency/semi-coherency for the precipitates in microalloyed steels is usually lost for precipitate sizes larger than 10 nm [40]. Thus, with aging, precipitates formed prior to the deformation might not contribute the retardation of DRX as efficient as the dynamically precipitating ones.

Secondly, probably as Andersson et al. and Srinivasan et al. [28, 41] reported, after solutionizing at relatively higher temperatures (above 1012 °C) the subsequent δ/η precipitation becomes very sluggish (due to the growth of the original grains). This makes the microstructure starve the preferential DRX nucleation sites. Due to the absence of GB precipitates, the effect of γ' on DRX onset potentially become more pronounced. However, further investigation would be necessary to clarify this.

Modeling the Observed Behavior

Table (3) shows the model parameters. A comparison of measured and computed stress-strain curves for 0, 30 and 120 minutes of aging are shown in Fig. (4). Here the model is calibrated only for no or little aging times when compared to industrial routines (up to 8 or 16 hours of aging). The resultant mean error over the strain interval averaged through all conditions is 11.37 MPa, which is acceptable considering the modifications made to the base models. Although the deformation is assumed to be homogeneous, to account for the effect of precipitation, it was necessary to introduce more model parameters. While the number of parameters is quite high relative to the simplistic models, the model is still applicable. To simplify the treatment, the factor C can be accepted as a constant when the deformation duration is short relative to the precipitation kinetics. Plausible values for this factor is given in Table (3).

To evaluate how f_{Cut} evolves with aging time, the contribution of second phase particles is considered through Eq. (6) and (7) based on the change in σ_p of Eq. (1). This is summarized in Fig. (5). When calculating the contribution of yield stress at room temperature, the critical radius/aging time can be found by equating the Eqs. (6) and (7). When this is applied to the current case, one finds the critical aging time to be 4000 seconds (taking $\beta = 1$ and $k_c = 1$). However, this would not correlate with the findings of the TEM study (this will later be shown in Fig (9) where after 1200 seconds of aging it is possible to see bowing mechanism). After calibrating

FIG. 4. Measured and calculated flow curves of solutionized and aged (30 and 120 mins.) samples. Dashed lines represent calculated values.

Eqs. (6) and (7) and using the Eq. (8) one finds that about 1200-1800 seconds of aging Orowan mechanism is active. The critical radius for similar alloys is reported to be around 12 nm [42, 43] which is in line with the current findings. However, the very same alloy was previously studied by Zickler *et al.* for room temperature yield strength predictions and they reported particle shearing as the main mechanism governing the precipitation contribution. This raises the question whether high temperature deformation favors Orowan mechanism or not. Furthermore the validity of the equations Eq. (6) and (7) to evaluate the particle strengthening at high temperatures can be questioned. From a physics based modeling, there should not be any calibration constants associated with contributions independent of strain.

TABLE 3. Model parameters and results.

Parameter	$920^{\circ}\text{C} - 0.01\text{s}^{-1}$	$940^{\circ}\text{C} - 0.01\text{s}^{-1}$	Unit
α	0.15	0.15	-
b	2.56×10^{-10}	2.56×10^{-10}	meters
m	3.1	3.1	-
C (when constant)	0.11	0.08	-
Ω	33.82	37.35	-
U	8.020×10^{-4}	7.318×10^{-4}	-
ρ_0	1×10^{12}	1×10^{12}	m/m^3
K_{prec} (no hold)	0	0	-
μ	Eq. (5)	Eq. (5)	GPa
w_q	0.75	0.75	-
w_r	0.82	0.82	-
γ_{APB}	0.111	0.111	J/m^2
β	0.29	0.31	-
k_c	0.08	0.09	-

TEM and STEM Observations

Figure (6) shows a series of TEM and STEM images of an undeformed sample aged at 850°C for 30 minutes. A bright field TEM image giving an overview of the microstructure is given in (a). γ' precipitates accompanied by sparse amount of twins are observed together with stacking faults (SF).

FIG. 5. Contributions of different particle hardening mechanisms given together with the calculated increase in σ_p of Eq. (1).

In general, all observed samples point out two different families of γ' . One consisting of fine ($<10\text{nm}$) densely distributed population, the other consisting of larger, spherical (faceted) and homogeneously distributed precipitates; different in size depending on the aging time. The appearance of two different γ' population is found to be more pronounced for samples in Set 2 (deformed after pre-aging). This is in line with the findings of Whitmore et al. [44] where the authors analyzed single and double aged 718Plus and reported both treatment routines result in a very fine (1-2 nm) γ' as a secondary precipitate population. The smaller γ' size for their case is expected as the aging temperature was below 790°C for Whitmore *et al.* [44].

These γ' particles can be observed both in Fig. (6-b1 & b2) and (6-c). The former pair represents bright field

(BF) TEM images and corresponding dark field (DF) TEM image recorded using (100) superlattice reflection. The latter is bright field and High Angle Annular Dark Field (HAADF) STEM images recorded in a region where γ' is observed accompanied with planar defects which might be twins, SF or Anti-Phase boundary (APB).

As mentioned in the experimental section, Set 2 samples were deformed up to near critical strain after different aging treatments. Bright field and corresponding dark field TEM images using (001) superlattice reflection from two different regions of the 920°C-20' (for terminology selection, refer back to the *Experimental Procedure* section) sample are shown in Fig. (7).

These images are recorded on the same grain. While Fig. (7-a2) shows fine and densely distributed γ' , Fig. (7-b2) shows spherical particles having average size of 39.8 nm. Even though most of the observed regions have the latter form of spherical γ' (see also Fig. (8 for EDX maps), it is thought-provoking that there are regions within the same grain where a finer and denser γ' population is observed. This inhomogeneity can be either associated with that of the deformation or due to a zone depleted of γ' forming elements. As highlighted by Militzer *et al.* [45], vacancy concentration levels during hot deformation might attain as much as 40 times that of equilibrium concentrations for a given temperature. Thus, any inhomogeneity in the dislocation density within a grain might cause local variations in precipitation characteristics. For the calculations performed within this study, only the spherical γ' population is considered to have an effect on the dislocation path.

Figure (8) gives the elemental maps (EDX maps) recorded on samples 920°C-20' (a) and 940°C-30' (b). γ' can be associated with the Cr depleted and Al/Ti rich zones. Only Cr, Al and Ti elements are presented to highlight the contrast. As mentioned in the experimental section, 920 and 940 °C are the temperatures at which the nose and asymptote of γ - γ' transformation line is located on the TTT diagram, respectively. Based on Fig. (8-b), the γ' obtained during pre-aging treatment is preserved to a large extent during deformation at 940 °C. In contrast, γ' observed in 920°C-20' is much well defined, leading one to consider the contribution of deformation to γ'

(a)

(b1)

(b2)

(c1)

(c2)

FIG. 6. TEM images after solutionizing at 1050°C followed by 30 minutes aging at 850°C: (a) Low magnification image giving an overview of the microstructure, (b) Bright field (left) and corresponding dark field (right) images recorded along $[011]\gamma // [011]\gamma'$ zone axis. (c) STEM images: bright field (right) and corresponding High Angle Annular Dark Field (left).

FIG. 7. TEM bright field (left) and corresponding dark field (right) images of aged (850 °C for 20 mintues) and deformed (920 °C, 0.01s⁻¹) sample. Both dark field images were recorded using (001) weak superlattice reflection.

growth when the deformation takes place in the precipitation window (below 940 °C). The effect of deformation on the nucleation of γ' might be considered based on Fig. (7-a2); however, further examination would be necessary. Furthermore, considering the overall volume fraction of γ' before deformation, forced nucleation of γ' is of low probability.

(a)

(b)

FIG. 8. EDX maps of aged and deformed: (a) 920°C-20', (b) 940°C-30'

Observations on the Aged and Deformed Specimens One of the objectives of TEM characterization in this study is to understand which dislocation-particle interaction mechanism(s) is active. This is particularly important when evaluating the effect of aging on the integrated/overall mechanical response of the alloy.

To this end, the weak-beam technique has been used. Based on the observations, dislocation bowing and looping can be clearly observed, while cutting cannot be ruled out. Figure (9) shows a set of bright field images obtained from sample 920°C-20'. While Fig. (9-a) gives an overview, the upper arrow on the image given in Fig. (9-b2) shows a dislocation bowing around a precipitate and the lower one shows a dislocation cutting a precipitate (the dashed line given is $[\overline{112}]$). This result is interesting because Zickler et al. [34] reported the experimental and predicted room temperature yield strength of 718Plus for aging times up to 200 minutes at 825 °C. These authors highlight that the predictions based on Orowan mechanism overestimate the experimental values more than 1.5 times for all aging times. In their work they conclude that Orowan mechanism becomes active only after 50 minutes of aging time at 825 °C. Compared to this, in the current case while 20 minutes pre-aging time is rather short, traces of Orowan mechanism can be clearly observed.

Furthermore, Fig. (10) shows bright field and weak beam dark field images of 920°C-20' sample. In the DF images (Fig. (10)-b1&b2) some characteristic fringes are clearly visible. There may be many reasons causing the formation of these fringes. They could be attributed to Moiré, to stacking fault bounded by partial dissociations, Shockley dislocations resulting from perfect dislocation dissociation, etc. The fringes could appear during the shearing of precipitates by the matrix dislocations, recalling the Condat-Decamps mechanism [46,47].

It is well established [48] that the stacking fault alternate parallel bright and dark fringes, the outer of which does not have the same contrast, when the image is recorded in dark field mode, the image is called asymmetric. The insert in dark field image of Fig. (10-a2) point out a series of alternate bright and dark fringes, the outer fringes have the same bright contrast. Based on this behavior, the stacking fault are excluded, thus we are left

with the Moiré fringes. In the current case, the inter-fringes as well as the misorientation between the orthogonal direction to the fringes and the reflections (001) of the γ' (or the γ -matrix). A slight disorientation of less than 1 degree was detected. This configuration strongly argues in favor of mixed Moiré fringes (translation + rotation) obtained by the superposition of the matrix and precipitates. The departure to the exact cube-cube relationship should be attributed to stress relaxation during the shearing of the precipitates by the matrix dislocations.

Additionally, a bright field TEM image of 940°C-0' sample (sample with no prior aging) is shown in Fig. (11), presenting abundant $a/2\langle 110 \rangle$ matrix dislocations with evidence of cross slip (note the rigged appearance of the dislocations). This is an interesting detail as no significant precipitation is observed in this sample. Under normal circumstances, an increase in cross-slip probability, thus an increase in recovery parameter Ω , would be expected in an aged alloy relative to the solutionized one. Based on this in the current case, Ω is assumed to be independent of precipitation.

Morphology of γ' and Its Relationship with γ Matrix The γ' precipitates adopt an usual cube-on-cube orientation relationship with the matrix where it develops as derived from the analyzed SAED diffraction patterns. This relationship can be expressed by the parallelism between the corresponding planes and can be expressed as follows:

$$(002)_{\gamma} // (001)_{\gamma'}$$

$$(1\bar{1}1)_{\gamma} // (1\bar{1}1)_{\gamma'}$$

$$(2\bar{2}0)_{\gamma} // (1\bar{1}0)_{\gamma'}$$

Trace analysis indicates that the γ' phase develop only one variant in each matrix grain (see Fig. (7)-b2 as well). Figure (12) shows a high resolution image of a 30 minutes aged sample at 850°C after solutionizing at 1050°C. The inset is the corresponding FFT recorded along $[011]_{\gamma} // [011]_{\gamma'}$.

(a)

(b1)

(b2)

FIG. 9. (a) Bright field TEM image with approximate zone axis $[011]$. (b2) A portion of the image shown on (a) highlighting interaction modes.

FIG. 10. Bright field (a1 & b1) and weak beam dark field (a2 & b2) TEM images of 920°C-20' sample after deformation. Weak beam image ($g=3g$) $g=001$ recorded along zone axis close to [011].

Symmetry Analysis It is well established, through the group theory, that the morphology of phases, taking place in any medium (solid, liquid or gas) is of great importance in materials science. Understanding of both the equilibrium shape and the habit plane adopted by these phases in their medium is widely investigated [49–56].

FIG. 11. Bright field TEM image of 940°C-0' sample. $(a/2)_{\gamma} [110]_{\gamma}$ type of matrix dislocations.

We will apply this group theory, based on the symmetry analysis, to explain the shape developed by the γ' phase and its variant number.

When the point symmetry groups of γ matrix and γ' precipitates are considered (G^{γ} and $G^{\gamma'}$), if the precipitating phase adopts an orientation relationship with the matrix, the intersection point group (H) will be represented by their common symmetry elements. Usually denoted as $H = G^{\gamma} \cap G^{\gamma'}$. H , the subgroup of the matrix and of the precipitate point groups the morphology of the precipitate together with the variant number n of the subgroupings. The latter helps to assess the number of variant(s) taking place in the matrix and satisfying the orientation relationship adopted between the matrix and the precipitate. The variant number n is the ratio $n = m/h$ where m and h are the orders $G^{\gamma} H$, respectively. This analysis is summarized in Table (4). Accordingly, the point group of the matrix (G^{γ}) is $\frac{4}{m} \bar{3} \frac{2}{m}$ which is the same as that of the γ' ($G^{\gamma'}$). Obeying to the orientation relationship, the superimposition of the symmetry elements of the two $\frac{4}{m} \bar{3} \frac{2}{m}$ point groups preserve all symmetry elements leading to $H = \frac{4}{m} \bar{3} \frac{2}{m}$.

FIG. 12. High Resolution TEM (HRTEM) image and its corresponding FFT recorded along $[011]_{\gamma} // [011]_{\gamma'}$ zone axis.

The fact that both G^{γ} and $G^{\gamma'}$ having the order of 48 leads to the variant number $n = 1$, indicating that there is only one variant of the γ' formation in γ matrix. This is in line with the observations i.e. faceted spherical and cubic γ' in γ depending on the volume fraction. In the case of 718Plus, we observe mostly fine or faceted spherical precipitates, see Figs. (7-b2) and EDX maps in Fig. (8-a).

TABLE 4. Determination of H , the intersection point group, the elements of which are common to the point groups of γ and γ' phases. n is the number of variants; m , p and h are the orders of the point groups of matrix, precipitate and intersection group respectively.

Orientation	Superimposed	Shared	$H = G^\gamma \cap G^{\gamma'}$
Relationship	symmetry elements	symmetry elements	with $n = \frac{m}{h}$
$(002)_\gamma // (001)_{\gamma'}$	$\frac{4}{m} \parallel \frac{4}{m}$	$\frac{4}{m}$	
$(1\bar{1}1)_\gamma // (1\bar{1}1)_{\gamma'}$	$\bar{3} \parallel \bar{3}$	$\bar{3}$	$\frac{4}{m}\bar{3}\frac{2}{m}$
$(2\bar{2}0)_\gamma // (1\bar{1}0)_{\gamma'}$	$\frac{2}{m} \parallel \frac{2}{m}$	$\frac{2}{m}$	with
$n = 48/48 = 1$			

CONCLUSIONS

The hot flow behavior of solutionized and pre-aged 718Plus is investigated. Two different sets of tests are performed: Set 1 which consists of solutionized samples being deformed at 16 different conditions (4 strain rate x 4 temperature) and Set 2 which consists of solutionized and pre-aged samples being deformed at two different conditions (1 strain rate x 2 temperature). Up to two hours of aging times are investigated which might be of interest to design the final passes of industrial forging operations.

A homogeneous work hardening model based on the long-standing dislocation density theory is proposed which includes the effect of precipitation as a damping factor of recovery rate. The evolution of precipitate volume fraction and radius is deduced based on the experimental findings reported in the literature and are calibrated according to the TEM observations. The proposed model is fitted to the experimental stress strain curves up to the critical stress. The flow stress contribution of different dislocation precipitation interaction mechanisms are

evaluated and it is found that Orowan mechanism is activated around 1200 seconds aging time after which it quickly starts to dominate.

A symmetry analysis of $\gamma - \gamma'$ is conducted and the variant number of γ' phase is reported as 1, meaning only faceted spherical or cubic γ' is possible. Within this study, only very fine (< 10 nm) or larger spherical γ' (size depends on aging time) is observed. An alteration in the precipitation kinetics compared to the previously reported static kinetics is observed; however, left out of the scope of the current study. Based on the TEM observations, after 1000 seconds of aging time; bowing, looping and cutting mechanisms are observed to be co-active.

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