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γ' and γ" co-precipitation phenomena in directly aged Alloy 718 with high δ-phase fractions

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ABSTRACT

Co-precipitation of γ' and γ" is the main strengthening mechanism that provides superior high-temperature strength in directly aged Alloy 718 aerospace parts. Control of their morphology, fraction, and configuration might allow exposure to more demanding operation environments in next-generation aircraft engines. The density of geometrically necessary dislocations introduced during hot deformation has been shown to significantly affect the co-precipitate morphology of γ' and γ" in materials free of the δ-phase. However, the combined effects of geometrically necessary dislocation density and lower Nb content due to higher δ-phase fractions on co-precipitation behaviour and strengthening remain unknown. We verify these effects by hardness testing as a proxy for high-temperature strength in materials with 4.1 % δ-phase fraction. Deformation at 950 °C yields a remarkable increase of 12 % in hardness after direct ageing, explained by the prevalence of complex co-precipitate configurations. Deformation at 1000 °C decreases the δ-phase fraction and geometrically necessary dislocation density but achieves up to 19 % volume fractions of γ', leading to a predominance of monoliths and duplet co-precipitates and a better direct ageing response. Atom probe microscopy reveals the flux of elements during co-precipitation. We recommend a δ-annealing treatment before the final forging step for manufacturing stronger Alloy 718 aerospace parts.

1. Introduction

Ni-based superalloys, such as Alloy 718, have been the workhorse materials for aeroengine turbine disks for several decades due to their outstanding mechanical properties [1]. The excellent suitability of Alloy 718 for extreme environments is due to its polycrystalline, age-hardenable, and hierarchical microstructure, stable up to 650 °C [2]. Despite the development of several new multicomponent aerospace alloys in recent years, Alloy 718 remains unchallenged. It is expected to continue being one of the most commonly used materials in next-generation aeroengine designs [3]. Nevertheless, stricter regulations and societal demands for more environmentally friendly aircrafts have driven the need to further enhance aeroengines and their materials for superior performance. Lighter engines with smaller cores and larger bypass ratios can increase efficiency [4]. However, rotating components in smaller turbines spin faster, generating mechanical loads and temperatures that might exceed Alloy 718's current specifications. The preferred method to further improve its high-temperature mechanical strength is to adjust the thermo-mechanical processing route, which can be rapidly adopted by industry. Therefore, the approach adopted in this study is to engineer the strengthening mechanisms of Alloy 718 via advancing thermo-mechanical processing parameters.

Alloy 718 has a face-centred cubic (fcc) γ matrix, with grain boundaries decorated by the μ-scale δ phase with a D0 3 crystal and Ni 3 Nb chemical structure [5]. The δ phase has been shown to effectively pin grain boundaries, preventing grain growth and retarding inter-granular crack propagation during creep [6,7]. At the nm-scale, co-precipitation of the spherical γ' with a L12 crystal and Ni 3 (Al,Ti) chemical structure occurs concurrently with the lenticular-shaped γ" with a D0 22 crystal and Ni 3 Nb chemical structure [8,9]. The primary precipitation of δ phase depletes the γ matrix of Nb. Therefore, lower volume fractions of γ' are found when high δ fractions are present due to their similar chemical composition. This is generally undesirable as γ' is the main strengthening phase in Alloy 718. This phenomenon is more
pronounced in the low-Nb regions around the δ phase, with no co-precipitation of γ′ [10]. The sole precipitation of γ′ yields a local hardness 20 % lower than the remaining γ matrix [11]. Nonetheless, co-precipitation mechanisms in materials with high δ phase fractions due to associated chemical changes in the bulk of the γ matrix remain unclear.

Microstructural heterogeneities in Alloy 718 after industrial billet-to-part conversion include varying grain sizes, δ-phase fractions, and geometrically necessary dislocation (GND) densities [12]. Variations in grain size as a function of hot deformation parameters have been comprehensively investigated [13–18]. Variations in the δ phase fraction have been shown to impact peak strain and stress during forging. Furthermore, the δ phase plays an essential part during dynamic recrystallisation (DRX) via particle-stimulated nucleation (PSN) of newly recrystallised grains [19,20]. Post-forging heat treatments alleviate heterogeneities by modifying the grain size and dislocation density and by promoting the precipitation of γ″ and γ′. The traditional ageing process used over decades consists of solution annealing below the δ-solvus, followed by double-step ageing at 720 °C and 620 °C. This conventional ageing (CA) process has been less frequently applied since the introduction of direct ageing (DA) [21], a shorter and more economically viable treatment in which the material is subjected to the two ageing steps without prior solution annealing. In addition to delivering similar creep and fatigue life, parts subjected to DA have up to 10 % higher yield strength at 650 °C than CA parts [22].

DA of Alloy 718 has been reported to have significant effects on the δ phase and γ′ precipitate fractions, as well as the γ′-γ″ co-precipitation configurations [23,24]. Industrially forged DA parts commonly have ~2.6 % and ~14 % volume fractions of δ phase and γ′ precipitates, respectively, with a predominance of duplet co-precipitates (γ′-γ″) and sandwich-like particles with a γ″-γ′-γ″ stacking sequence [23]. Their CA counterparts have higher fractions of δ phase (~4–5 %), while the γ′ fraction is reduced to ~11 %, with a majority of duplets and γ″-γ′-γ″ co-precipitates. While some degree of δ phase precipitation occurs during solution annealing in CA, the fraction of this phase in DA is mainly inherited from forging. In our recent work, Alloy 718 billet material with negligible fractions of δ phase was deformed at several strain rates and temperatures, leading to up to a 20 % fraction of γ′ precipitates after DA [25]. An inverse correlation was identified between the GND density generated during deformation and the number of duplet co-precipitates.

Multi-phase field models on co-precipitate nucleation and growth corroborate experimental investigations demonstrating higher thermal stability of specific co-precipitate configurations. Simulations by Sriman et al. [26] and Shi et al. [27] have shown that, on the one hand, higher Al/Nb ratios, common in CA materials due to low Nb availability in the γ matrix, favour initial γ′ precipitation followed by γ″. This mechanism has been verified via atom probe microscopy (APM) of the early stages of precipitation during ageing [28]. The opposite is valid for DA materials, with high concentrations of Nb in the γ matrix and preferential formation of γ″, which chemically drive the nucleation of γ′ shells in the γ″-γ′ co-precipitates. In a similar study, Cozar and Pineau [29] altered the chemical composition of Alloy 718 to obtain various co-precipitate configurations. They showed that a compact configuration with cuboidal γ′ coated with γ″ will coarsen slower during exposure to high temperatures. These findings demonstrate that advanced control over co-precipitation phenomena could indeed unlock Alloy 718 for temperatures above 650 °C.

The effects of deformation temperature and strain rate on morphology, chemistry, and configuration of the nanoprecipitates have recently received more attention, primarily due to previous challenges in conventional electron microscopy [30–36]. Higher δ phase fractions have been shown to affect co-precipitation [23]. However, a systematic approach to examining the strain rate and temperature effects on morphological and chemical aspects of γ′ and γ″ in materials with high δ phase fraction is currently missing. This paper investigates the effects of hot deformation parameters on co-precipitation in Alloy 718 with higher δ phase fractions than commonly found in industrial DA materials. We elucidate co-precipitation mechanisms by APM of the precipitates’ morphology, configuration, and chemical profile in DA conditions after deformation. These findings can support the development of better thermo-kinetic models, aiding the implementation of new processing routes to generate stronger and more thermally stable Alloy 718 parts.

2. Materials and methods

The nominal chemical composition of Alloy 718 is provided in Table 1. Samples were extracted from a 254 mm billet provided by voestalpine BÖHLER Aerospace GmbH & Co KG, Austria. Cylindrical samples (H 10 mm Ø 8 mm) were cut via electrical discharge machining from the 76 mm radial billet position. Samples were heat treated at 900 °C for 6 h to achieve 4.1 ± 0.6 % volume fraction of δ phase. The volume fraction was determined from the total area fraction of the δ phase in ten 120 × 90 μm² backscatter scanning electron (BSE) images. The Fiji software was used to distinguish the γ matrix from the δ phase via a colour threshold analysis [37]. A typical BSE image of the microstructure after a heat treatment is shown in Fig. 1.

The heat-treated samples were subjected to uniaxial compression in a Gleeble 3500. The deformation temperatures of 950 °C and 1000 °C were reached after heating at a rate of 5 °C/s and soaking for 300 s. The selection of these temperatures below the δ phase solvus temperature of 1025 °C [38,39] ensured that a certain δ phase fraction was retained after deformation. The strain rates used were 0.1, 1, and 10 s⁻¹, which are in similar ranges as during the industrial forging of DA turbine disks [40,41]. Once a height reduction of 70 % was reached, all samples were immediately water quenched in the Gleeble. In the following, various material conditions will be denoted by linking the forging temperature and strain rate, such as “700/0.1SR,” which designates the sample subjected to deformation at 1000 °C with a strain rate of 0.1 s⁻¹.

Deformed samples were cut along the deformation axis, mounted in conductive resin, and prepared for electron microscopy via standard metallography procedures for Ni-based superalloys [42]. DEFORM® (v 12.0) simulations in Fig. 2 show the effective strain in the cross-section of deformed samples. Regardless of the deformation parameters, the maximum effective strain is concentrated at the centre of the samples. Therefore, microstructural analyses and hardness testing were limited to the regions inside the black dashed rectangles in Fig. 2.

A ZEISS Auriga cross-beam scanning electron microscope was used for imaging at an accelerating voltage of 20 kV. Electron backscatter diffraction (EBSD) measurements were conducted using a Nordlys detector. The crystallographic data collection involved mapping multiple areas of 300 × 220 μm². This was achieved using a 4 × 4 camera binning and 0.3–0.5 μm step size. The acquired data was post-processed using the TSL OIM Analysis software 8 and the HKL Channel 5 software. A grain dilation clean-up procedure was applied with a 5° grain tolerance angle. Furthermore, a neighbour correlation clean-up procedure was carried out to measure the distribution of GNDs. This considered a grain tolerance angle of 5° and a minimum confidence index of 0.1. A Kuvahara-filter clean-up procedure was applied as a last step, with one iteration for every third neighbour and a maximum misorientation of 5° for neighbouring pixels. Finally, the distribution of GNDs and their average values were calculated using the TSL software function developed by Field et al. [43].

Deformed materials were directly aged to study the nanoscale precipitation evolution. All samples were placed in a tube furnace with a steady flow of Ar at 720 °C for 8 h, followed by continuous cooling for 2 h down to 620 °C and further holding for 8 h. At the end of DA, all samples were water quenched.

APM of the DA samples was carried out on deformed grains only, to investigate the effects of GND density and the δ phase fraction on γ′ and γ″ co-precipitation. Site-specific tips were prepared using a method where lift-outs are avoided, as proposed in [10]. Blanks (0.3 × 0.3 × 7 mm³) were cut from the cross-section of the deformed samples, ensuring
that one end was at the centre of the cross-section, i.e., within the black rectangles shown in Fig. 2. These blanks were placed in cylindrical Cu stubs, and their top end was annularly milled in a ThermoFisher Helios G4 Xe plasma-focused ion beam (PFIB). Once the rough annular milling was completed, EBSD maps of the tips were obtained to identify deformed grains, as shown in Supplementary Fig. S1, which includes inverse pole figure (IPF) and grain orientation spread (GOS) maps. Deformed grains were identified as those with a GOS $>1.1^\circ$ [44], as indicated by red arrows. After locating non-recrystallised grains, the distance to the apex was measured, and further annular milling was performed to reach the desired position. Secondary electron images of the tips were recorded to reconstruct the atom probe data via the tip profile. APM experiments were carried out in a CAMECA Invizo 6000 3D atom probe with a dual-beam laser operating at a pulse rate of 200 kHz, pulse energy of 400 pJ, detection rate of 4 %, detector efficiency of 62 %, and temperature of 50 K. The software AP Suite 6.3 was used to analyse the 3D datasets. A minimum volume of 1,300,000 nm$^3$ was analysed per sample. Isosurface objects enveloping (Al + Ti) ions and Nb ions were used to reveal $\gamma'$ and $\gamma''$ precipitates, respectively. Isovalues were determined through the average between the maximum and minimum concentration differential in the 1D composition profile in cylinders placed across five $\gamma'$-$\gamma''$ co-precipitates. Further information on this method is available in [45]. Once isovalues are determined, AP Suite lists the isosurfaces in each dataset, including their dimensions (x, y, z) and volume. The volume fractions of $\gamma'$ and $\gamma''$ precipitates were determined as the sum of the respective isosurfaces divided by the total volume of the tip. To calculate the average length of the major axis of the precipitates, the isosurfaces with the 10 % lowest volumes were

### Table 1
Nominal composition of Alloy 718.

<table>
<thead>
<tr>
<th>Element</th>
<th>Fe</th>
<th>Cr</th>
<th>Nb</th>
<th>Mo</th>
<th>Ti</th>
<th>Al</th>
<th>C</th>
<th>B</th>
<th>Ni</th>
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<td>wt.%</td>
<td>18.5</td>
<td>18.0</td>
<td>5.3</td>
<td>3.0</td>
<td>0.95</td>
<td>0.50</td>
<td>0.02</td>
<td>0.003</td>
<td>Bal.</td>
</tr>
<tr>
<td>at.%</td>
<td>19.2</td>
<td>20.1</td>
<td>3.3</td>
<td>1.8</td>
<td>1.15</td>
<td>1.08</td>
<td>0.01</td>
<td>0.002</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

![Fig. 1. Typical BSE image of Alloy 718 after heat treatment at 900 °C for 6 h, revealing a 4.1 % volume fraction of the needle-shaped δ phase.](image)

![Fig. 2. DEFORM® simulations of strain distribution on the cross-section of deformed samples at various temperatures and strain rates. Black dashed rectangles represent regions where microstructural analyses and hardness testing were performed.](image)
of temperature and strain rate can be observed in the GOS maps in Fig. 4. The GOS maps are colour-coded, ranging from blue to red (Fig. 4a), representing the recrystallised grains with the remaining deformed grains shaded in grey contrast. The corresponding IPF maps are provided as Supplementary Fig. S2. A ‘necklace’ microstructure is present in all conditions, with variations in the fraction of recrystallised grains quantified in Fig. 4b. The V-shape in the recrystallisation fraction at multiple orders of magnitude of strain rate has been widely reported for Alloy 718 [52–54]. This is primarily an effect of the correlation between the strain rate, strain hardening and DRX. At 0.1 s⁻¹, the nominal strain of 1.4 is reached after about ~7 s. Consequently, deformation is slow enough to enable the formation of recrystallised nuclei and their subsequent growth, leading to a higher degree of recrystallisation. In this case, there is an equilibrium between GND formation and annihilation, therefore, strain hardening is minimised. The duration of deformation is reduced by an order of magnitude at 1 s⁻¹ and to <0.07 s at 10 s⁻¹. Faster deformation activates different mechanisms. On the one hand, a strain rate of 1 s⁻¹ is rapid enough to induce more strain hardening than 0.1 s⁻¹; on the other hand, it is not enough to trigger DRX due to adiabatic heating, observed for the strain rate of 10 s⁻¹ (Fig. 4). This intermediate state leads to a lower recrystallisation fraction and smaller recrystallised grains than for strain rates of 0.1 and 10 s⁻¹. This phenomenon is also verified in the flow stress curves during deformation, as shown in Supplementary Fig. S3. The final stress at a strain of 1.4 is the highest for the strain rate of 1 s⁻¹, regardless of the deformation temperature. Further, the stress–strain curves indicate that the peak stress increases with strain rate. The exceedingly fast deformation at 10 s⁻¹ generates a high degree of strain hardening up to 0.2 – 0.3 true strain. However, adiabatic heating creates an ideal condition for accelerated DRX [55]. Subsequent recrystallisation and GND annihilation lower the flow stress, facilitating further deformation. Near the maximum strain, stress values at a strain rate of 10 s⁻¹ are even lower than for 0.1 s⁻¹.

From the temperature perspective, at 1000 °C, higher thermal activation energy enables a higher recrystallisation fraction than at 950 °C [56]. This is also related to the δ phase volume fraction and the PSN mechanism. At 950 °C, the ~4 % δ phase fraction yields a higher number of recrystallised nuclei at heterogeneous nucleation sites, i.e., the δ phase/γ matrix boundaries [57]. At 1000 °C, the lower fraction of δ phase implies that fewer recrystallised nuclei are generated, but they grow faster at this temperature. The PSN effect can be further exemplified by comparing the data from Fig. 4b with Alloy 718 without δ phase [25]. The absence of these particles is detrimental to DRX, particularly at 950 °C, where recrystallisation is as low as 3 % at 950C,1SR, or five times lower than for the samples with 4.2 % δ phase fraction. This impact is minimised at 1000 °C. In 1000C,1SR, there are 33 % recrystallised grains in the absence of δ phase, or 8 % less than in 1000C,1SR with 1.8 % δ phase fraction. Despite possible additional mechanisms, the faster decrease in flow stress after peak stress (Fig. S3) for all strain rates at 950 °C compared to the same strain rates at 1000 °C is likely contributed by the higher fraction of δ phase, corroborating its impact on facilitating DRX. Additionally, the increase in recrystallisation fraction at 1000 °C is not proportional for all strain rates. As seen in Fig. 4b, for 1 s⁻¹, the recrystallised fraction is 2.7 x higher at 1000 °C than at 950 °C, while at 10 s⁻¹, this variation is 1.6 x. A similar behaviour is observed in materials without δ phase [25], where recrystallisation increases by an order of magnitude at 1 s⁻¹ and is considerably lower for 10 s⁻¹. This indicates that for 1 s⁻¹, the impact of temperature and δ phase on DRX is more significant than for other strain rates.

Dislocation annihilation occurs during the formation and growth of recrystallised grains [58]. This leads to an inverse proportionality between the recrystallisation fraction and GND density. Fig. 5a shows the GND density maps, and Fig. 5b the corresponding GND densities. These plots exhibit roughly the inverse shape of Fig. 4b, indicating that samples with lower recrystallised fractions have the highest GND densities. These values are lower in samples without δ phase [25], except for
the 950C_1SR condition. Despite a much higher recrystallised fraction (15% vs. 3%), the non-recrystallised grains might contain more GNDs due to the presence of δ phase particles, which are sites of dislocation accumulation during deformation [59]. This effect is less prominent in other samples due to their comparably higher recrystallisation degree.

3.2. Effects of hot deformation on the morphology of γ' and γ″ and co-precipitation mechanisms

The deformation parameters affect many microstructural features, including the δ phase fraction (Fig. 3), recrystallisation fraction (Fig. 4) and GND density (Fig. 5). Subsequently, these microstructural characteristics impact co-precipitation phenomena during DA. The GND density has been shown to affect the configuration of co-precipitates [25], while the δ phase is known to reduce the γ′ precipitation [11]. In the following, these effects are systematically elucidated via APM. Tips were extracted from central regions of the DA samples (Fig. 2), and for consistency in the analyses, APM was performed in deformed grains selected via EBSD (Fig. S1). The reconstructions are shown in Fig. 6, which, for clarity, displays typical 750,000 nm³ volume sections selected from a minimum analysed volume of 1,300,000 nm³ per tip. Supplementary Table S1 shows the concentration isovalues used to envelop the (Al + Ti) and Nb-rich regions, i.e., γ′ and γ″ precipitates, respectively. Minor variations are observed, indicating the consistency of the method proposed by Theska et al. [45] to determine isovalues in APM datasets.

Fig. 4. (a) GOS over recrystallised grains on top of image quality maps of Alloy 718 samples deformed to 70% height reduction at 950 °C and 1000 °C with strain rates of 0.1 s⁻¹, 1 s⁻¹, 10 s⁻¹. A GOS value of 1.1° is the threshold above which grains are considered deformed. The recrystallised fractions are shown in (b).
Visual inspection of the reconstructions in Fig. 6 indicates that more \( \gamma' \) precipitates are present in the materials deformed at 1000 °C. This is confirmed in Fig. 7, where the average lengths of the major axes and volume fractions of the precipitates are presented. The size of \( \gamma' \) precipitates is larger in materials deformed at 1000 °C. Minor deviations are observed between different strain rates for \( \gamma' \) precipitates. For \( \gamma'' \) precipitates, a slight size reduction is found at a strain rate of 1 s\(^{-1}\). The volume fraction trend is similar to the length of the precipitates, where higher volume fractions of \( \gamma' \) precipitates and \( \gamma'' \) precipitates are present in materials deformed at 950 °C and 1000 °C, respectively. The fraction of \( \gamma'' \) precipitates also seems to inversely correlate with the GND density, where samples deformed at 1 s\(^{-1}\), i.e., with the highest GND densities, have the lowest \( \gamma'' \) fraction. This might be related to the slightly higher \( \delta \) phase fraction in these conditions.

The configuration of various co-precipitates as single particles (monoliths) or as duplets, triplets, or quadruplets depends on the nucleation sequence during ageing [28]. The effects of deformation parameters on the precipitates' configuration formed during DA are shown in Fig. 8. The most noticeable features are the predominance of \( \gamma' \) monoliths over \( \gamma' \) at 950 °C, the higher fraction of duplets at 1000 °C, and the increasing fraction of \( \gamma'' \) monoliths for higher strain rates at 1000 °C. Additional aspects, such as higher fractions of complex

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**Fig. 5.** (a) GND density maps of Alloy 718 samples deformed to 70 % height reduction at 950 °C and 1000 °C with strain rates of 0.1 s\(^{-1}\), 1 s\(^{-1}\), and 10 s\(^{-1}\). The GND densities are shown in (b).
The volume fraction of $\gamma'$ precipitates is generally unaffected by the hot deformation parameters when deformation-induced variations in $\delta$ phase fraction are low \cite{12,25}. Here, slightly larger volume fractions of $\gamma'$ precipitates are observed in materials deformed at 950 °C (Fig. 7), with a disproportionate deviation seen in 1000C_10SR. This material, with a 1.2 % volume fraction of $\delta$ phase (Fig. 3) and $51 \times 10^{12}/m^2$ GND density (Fig. 5), has the lowest fraction of $\gamma'$ precipitates among all conditions studied here, which is counterbalanced by the highest fraction of $\gamma''$ precipitates. Higher concentrations of Nb in the $\gamma$ matrix, due to $\delta$ phase dissolution in materials deformed at 1000 °C, contribute to the preferential formation of monoliths of $\gamma''$ precipitates \cite{60}. Nucleation of $\gamma'$ is then limited to pre-formed $\gamma''$ precipitates, and subsequent growth occurs as co-precipitates such as duplets and $\gamma'$-coated triplets, as previously verified in computational simulations \cite{26}. This is confirmed in Supplementary Table S2, which presents the overall chemical composition of the materials obtained from the APM reconstructions. Deviations are observed for Al and Nb, with average values of 2.7 ± 0.2 at.% of Nb and 2.3 ± 0.2 at.% of Al for materials deformed at 950 °C and 3.5 ± 0.4 at.% of Nb and 2.4 ± 0.4 at.% of Al for 1000 °C. Nevertheless, as explained next in section 3.3., some discrepancies are observed between the Nb content in the materials and the $\gamma'$ fraction, as exemplified by 1000C_1SR and 1000C_10SR, with respectively higher (4 at.%) and lower (3.2 at.%) Nb content (Table S1) in 1000 °C specimens.

Fig. 6. 50 × 50 × 300 nm$^3$ APM reconstruction with isosurfaces of DA samples deformed at strain rates of 0.1 s$^{-1}$, 1 s$^{-1}$, and 10 s$^{-1}$ at (a) 950 °C and (b) 1000 °C.

Fig. 7. Length of the major axis (top graph) and volume fraction (bottom graph) of $\gamma'$ and $\gamma''$ precipitates.
but with the lowest (16.8 %) and highest (19 %) \( \gamma^\prime \) fraction, respectively.

Characterisation of the nanostructure of Alloy 718 is intrinsically local. Thus, heterogeneities in the GND density are expected within the non-recrystallised grains investigated by APM (Fig. S1). These local variations are assumed to correlate with the GND densities shown in Fig. 5b. Hence, a different nanoprecipitation behaviour is expected in recrystallised regions, which are not explored here. For instance, 950°C_1SR has the highest GND density (208 \( \times \) 10^{12}/m²) and contains the lowest duplet fraction, which agrees with previous work [25]. Comparatively, the associated decrease in GND density and dissolution of \( \delta \) phase at 1000 °C yields a duplet fraction averaged for all strain rates of \( \sim 61 \% \), i.e., 10 % more than the average duplet fraction in samples deformed at 950 °C (~51 %). This takes place at the expense of \( \gamma^\prime \) monoliths, with a fraction decrease of 8.5 % between samples deformed at 950 °C and 1000 °C. In addition, the low duplet fraction in the materials deformed at 950 °C seems to drive the formation of complex configuration observed 950C_1SR and 950C_10SR with the most triplets and quadruplets.

The co-precipitation configuration population can be primarily related to their nucleation and growth sequences. This can be observed during the early stages of precipitation during DA, which is out of the scope of this research. However, the final shape of the precipitates also provides clues on the co-precipitation mechanism, as shown by APM and simulation studies [28,60,61]. The three-dimensional character of APM enables further examination of the morphology of the precipitates, as shown in Fig. 9. Information on the precipitates’ shape can be established considering their aspect ratio, oblateness and volume. The quadrants in Fig. 9 categorise their shapes as disk, sphere, lath, or rod [62]. Fig. 9a shows the plots for each condition considering the volume of a random sample of 75 \( \gamma \) and 75 \( \gamma^\prime \) precipitates within each dataset, while Fig. 9b shows the average aspect ratio vs. oblateness values. Most precipitates fall into the two top quadrants, with the aspect ratio having a narrower distribution than oblateness. \( \gamma^\prime \) precipitates tend to form as spheres, while \( \gamma^\prime \) precipitates have lower oblateness, i.e., they are closer to disk shape. This is a well-known phenomenon, where, on the one hand, the lattice misfit is small between the cubic crystal structure of both \( \gamma \) matrix and the \( \gamma^\prime \) precipitates, and the formation of spherical precipitates is energetically favourable [63]. On the other hand, the \( c \) parameter of the tetragonal lattice of \( \gamma^\prime \) precipitates has up to 3 % mismatch with the \( \gamma \) matrix [64], and this anisotropic misfit makes it favourable for growth to occur along the \( \gamma^\prime \) precipitates habit plane [65], i.e., into disk-shaped particles. The \( \gamma^\prime \) precipitates in 950C_1SR and 950C_10SR are the only ones with an average value entirely within the disk-shaped quadrant in Fig. 9b. As shown in the insets in Fig. 8 and demonstrated by Shi et al.[27] and Sriram et al. [26], \( \gamma^\prime \) precipitates in any of the two triplet configurations tend to be more disk-shaped due to the hard impingement imposed by the flat interface with the \( \gamma^\prime \) precipitates. For instance, in \( \gamma^\prime \)-\( \gamma^\prime \)-\( \gamma^\prime \) triplets, \( \gamma^\prime \), growth is restricted perpendicularly to both \( \gamma^\prime \)-coated surfaces, while in \( \gamma^\prime \)-\( \gamma^\prime \)-\( \gamma^\prime \) triplet, \( \gamma^\prime \) heterogeneously nucleates on \( \gamma^\prime \), and growth is restricted along just one surface. This is assumedly also valid for quadruplets, while it might not be applicable for duplets in cases where \( \gamma^\prime \) forms first, and its growth is unrestricted until \( \gamma^\prime \) heterogeneously nucleates on it. Therefore, all conditions with duplet fractions higher than 52.2 % have an oblateness average value higher than 0.5.

3.3. Effects of \( \delta \) phase and hot deformation on the chemical composition of \( \gamma^\prime \) and \( \gamma^\prime \) precipitates

The sequence of nucleation events during ageing is mainly determined by the chemical composition of the matrix, where the concentration of Al, Ti, and Nb provides indications about the precipitation mechanisms [29,63]. The correlation between these elements has been shown to drive the formation of specific co-precipitate configurations [66]. Fig. 10 shows the \( (Al + Ti)/Nb \) ratio of the \( \gamma \) matrix only, i.e., disregarding \( \gamma \) and \( \gamma^\prime \) precipitates, and of all phases in the APM reconstruction, i.e., \( \gamma + \gamma^\prime + \gamma^\prime \). The materials deformed at 950 °C exhibit higher ratios than the ones deformed at 1000 °C, primarily due to lower amounts of Nb (Table S1). However, the higher Nb availability in the latter does not necessarily translate into higher \( \gamma^\prime \) precipitate fractions,
where some discrepancies in this phenomenon are exemplified in 950C_10SR and 1000C_1SR. Even though 950C_10SR has roughly double the fraction of δ phase (3.5 % vs. 1.8 % in Fig. 3), surprisingly, both conditions have similar volume fractions of γ″ precipitates (16.6 and 16.8 % in Fig. 7). Similar GND densities (99 × 10^{12}/m^2 and 109 × 10^{12}/m^2, Fig. 5) are not a suitable variable to explain this phenomenon. The (Al + Ti)/Nb ratios of the γ matrix only show an excessive deviation for 1000C_1SR (Fig. 10). This is caused by the highest amount of residual Nb (1.29 at.% in the γ matrix compared to the other conditions (0.7 ± 0.1 at.% Nb on average). 1000C_1SR also presents the highest overall Nb amount (4 at.% in Table S2) but has the lowest fraction of γ precipitates amongst all samples deformed at 1000 °C (Fig. 7). This indicates that precipitation is not only a function of the chemical composition but also of how the elements are distributed in each type of precipitate, as explained in the following paragraphs. Furthermore, the impacts of the different δ phase fractions on the configuration of the precipitates are observed (Fig. 8), where 950C_10SR has 11.7 % more complex (γ′-γ″-γ′) triplets and quadruplets co-precipitates than 1000C_1SR. The latter condition, with more Nb, has higher fractions of γ″ monoliths and duplets, where the γ″ precipitates have shown to be richer in Nb than in other co-precipitate configurations [28], as confirmed in the following.

The chemical profiles of various co-precipitate configurations are depicted via their corresponding (Al + Ti)/Nb ratios in Fig. 11. One can anticipate that γ′ monoliths will exhibit the highest ratio, while γ″ monoliths are likely to exhibit lower ratios. The (Al + Ti)/Nb ratios in γ″ monoliths are constant (Fig. 11a), despite varying concentrations of Nb in the samples (Table S1). This indicates γ″ monoliths’ preferential formation prior to other configurations when Nb is still fully available in the γ matrix [60]. Also, γ′ precipitation might precede γ′ in non-recrystallized regions, as Nb has been shown to diffuse faster than Al.
Fig. 10. \((Al + Ti)/Nb\) ratio of the \(\gamma\) matrix only (blue), and \(\gamma + \gamma' + \gamma''\) (black and red).

Fig. 11. \((Al + Ti)/Nb\) ratio of the precipitates in their different configurations. (a) monoliths, (b) duplets, (c) \(\gamma' - \gamma' - \gamma''\) triplets, and (d) \(\gamma'' - \gamma' - \gamma''\) triplets.
along dislocations. For γ′ monoliths, there is an inverse correlation between the (Al + Ti)/Nb ratio and the amount of Al in the samples. Low Al content in the sample seems to cause the formation of Nb-depleted γ′ monoliths, increasing the (Al + Ti)/Nb ratio. Samples 950C_0.1SR and 1000C_10SR illustrate this. Both conditions have the lowest concentration of Al (2 at.% in Table S2), and their γ′ monoliths show the highest (Al + Ti)/Nb ratios (2.60 and 2.53, respectively). These γ′ monoliths have the lowest concentration of Nb, i.e., 6.1 and 6.4 at.%, respectively. The Nb concentration in γ′ monoliths in other conditions is at least 7.3 at.%. Comparably, 1000C_1SR with 40 % more Al (2.8 at.% in Table S2) shows the lowest (Al + Ti)/Nb ratio (1.9) due to the enrichment of Nb in these precipitates. A similar phenomenon has been observed in alloys with a 2:1 Al/Ti ratio, where γ′ precipitates enriched in Nb were predominantly formed [67].

The trend above between the (Al + Ti)/Nb ratio and the amount of Al is also valid for the other γ configurations (Fig. 11b-d). The (Al + Ti)/Nb ratios in γ′ in duplets and γ′-γ″ triplets are similar and lower than in γ′ monoliths, as these co-precipitate configurations share one face with γ′ precipitates. During the formation of co-precipitates, Al content decreases in the γ′ precipitates while Nb increases [26,27]. This phenomenon is accentuated for the γ′-γ″ triplets, where a dual γ′-γ″ interface is present, and Nb concentration in γ′ increases to as much as 10.2 at.% in 1000C_1SR, reducing the (Al + Ti)/Nb ratio to lowest values (1.49) for γ′ precipitates.

The increase in Nb concentration in the samples deformed at 1000 °C (Table S1) reduces the γ′ fraction, and particularly the monolith fraction. Alongside, γ′ fraction decreases (Fig. 7), mainly assuming monolith or duplet configurations (Fig. 8). The (Al + Ti)/Nb ratios in the various configurations of γ′ show an inverse correlation with their γ′ counterparts, except for monoliths and γ′-γ″ triplets in the conditions deformed at 1000 °C. This is expected. While one co-precipitate gets enriched in a particular element, the counterpart co-precipitate must get depleted in the same. This is exemplified in the γ′-γ″ triplets, where Al influx from γ′ into γ″ increases the (Al + Ti)/Nb ratio to 0.44 in the γ′ while decreasing the ratio to 1.64 in the γ′. In materials deformed at 1000 °C, the high Nb concentration in the matrix makes it energetically easier for the γ′ in the γ′-γ″ triplets to obtain Nb from there, instead of from the γ′ precipitates.

3.4. Hardness increment during DA as a function of deformation parameters

DA has been shown to mitigate heterogeneities in mechanical properties inherited from manufacturing [12]. In Alloy 718, hardness has been demonstrated to correlate well with yield strength at 650 °C, i.e., one of the most critical requirements for turbine disk applications [68]. Fig. 12 shows the hardness (HV1) of the six conditions considered here in their as-deformed states and after DA. The columns show the percentual increment in hardness from as-deformed to DA condition. Increases in hardness (−40 %), even though 950C_0.1SR has 2.5 % more γ′ precipitates. Its comparably lower fractions of complex configurations (triplets and quadruplets in Fig. 8) and the associated lack of dislocation shear resistance demonstrated via density-functional-theory [70] justify the low hardness increment in 950C_0.1SR. This is further observed in samples 950C_10SR and 1000C_1SR, with similar γ′ fractions (16.6 and 16.8 %, respectively). Despite 1000C_1SR having 4.4 % more γ′ monoliths than 950C_10SR, an 11.7 % higher fraction of tripods and quadruplets in 950C_10SR leads to both showing a hardness increment of ~182 HV. Therefore, the effects of the precipitates’ configuration on hardness can be experimentally determined as ~12 % of tripods and quadruplets corresponding roughly to the hardening achieved by ~4 % γ′ monoliths. In this assumption, GND density and grain size are presumed not to change during DA, as previously demonstrated [23]. Besides, the increased presence of tripods might be beneficial for thermal stability. This phenomenon has been shown by Qiao et al. [67], where Nb enriched γ′ precipitates, like those in tripods, showed slower coarsening rates at 800 °C.

A lower DA hardening effect in materials with high GND densities, i.e., low hardness increment in materials with comparably higher as-deformed hardness, has been previously shown in industrially forged disks [12]. Here, this is observed mainly in samples deformed at the strain rate of 1 s⁻¹, with the highest as-deformed hardness and highest GND densities but some of the lowest hardness increments (41 % and 51 % for 950 °C and 1000 °C, respectively). Coarse γ′ precipitates formed on dislocation structures, that are often reported in materials air-cooled after forging, tend to coarsen during DA. Hence, in addition to losing coherency and decreasing their strengthening effects, coarse precipitates remove Nb from the γ matrix and reduce the fraction of typical γ′ precipitates and co-precipitates [60]. These coarse precipitates are less likely to form in the present study, as the materials were water quenched after deformation. However, preferential growth of γ′ precipitates on dislocations might still occur during DA, which must be deleterious for the DA strengthening effect. Nevertheless, despite the lowest hardness increment, these samples still have the highest hardness after DA. Further investigation is required to understand whether variations in the DA parameters would generate higher hardness increments in materials with these microstructural characteristics.

Interestingly, the hardness of as-deformed materials in this study is 7 to 12 % higher than in materials without 6 phase [25]. A combination of varying GND densities and recrystallization fractions might explain this, in addition to the hardening effect of 6 phase particles, which are up to
80% harder than the γ matrix [11]. After DA, hardness is 1 to 4% higher than in materials with no δ phase, as the DA effect on hardness increment is superior in the latter due to higher fractions of γ precipitates. Here, the highest hardness of 543 HV is 12% higher than the hardest part of a DA turbine disk (483 HV) [12,71]. This is a considerable increase, possibly corresponding to more than 100 MPa increment to the high-temperature yield strength and potentially allowing turbine disks to rotate faster, yielding better aero-engine efficiency. It is, however, important to mention that the duplex microstructure, consisting of deformed and recrystallised grains, might partially contribute to this strengthening. This type of microstructure has been shown to be applicable for turbine disks of Alloy 718, Astroloy, and Rene 95, delivering superior rupture ductility in the latter [72–74]. Therefore, we recommend future studies to explore fully recrystallised materials to complement the findings of the current paper. In addition, our findings can be used to enhance thermo-kinetic models of co-precipitation and must be further explored to thoroughly explain the co-precipitation behaviour in various starting microstructures.

4. Conclusions

Alloy 718 was heat treated to generate a ~4.1% volume fraction of δ phase. This material was used to determine the impacts of deformation temperatures (950 °C and 1000 °C) and strain rates (0.1, 1, and 10 s⁻¹) on the chemical and morphological aspects of γ and γ′ co-precipitation during DA processing. Their strengthening effects approximated via hardness testing reveal dependence on specific co-precipitate configurations. The key findings include:

1. The δ phase is mostly retained after deformation at 950 °C. These materials have the lowest Nb concentration (2.6 to 3 at.%) in the γ matrix. Their (Al + Ti)/Nb ratio is the highest, leading to the preferential formation of γ′ monoliths and duplexes (γ′-γ). The δ phase is reduced by over half at 1000 °C, and Nb concentration increased up to 4 at.%. Lower (Al + Ti)/Nb ratios cause fractions of duplexes to increase by ~10% and γ′ monoliths to more than double in some cases.

2. A strain rate of 1 s⁻¹ yields the lowest recrystallised fraction and highest GND densities for both deformation temperatures. Atom probe microscopy from deformed grains confirms a trend of fewer complex co-precipitates (triplets and quadruplets) to as much as ~28% in condition 950C_1SR. The highest hardness of 543 HV is 12% higher than in typical industrially forged monoliths to more than double in some cases.

3. The effect of DA processing on the hardness is superior for materials deformed at 1000 °C. Samples with the highest fractions of γ′ and γ″ monoliths show the lowest and highest hardness increments after DA, respectively. Complex co-precipitates introduce a degree of strengthening similar to γ′ monoliths at a ~3:1 ratio. Despite different grain size distributions, GND densities and δ phase fractions, DA mitigates hardness variations to only 30 HV. The maximum hardness of 543 HV is 12% higher than in typical industrially forged turbine disks.

Our findings provide a novel understanding of co-precipitation as a function of thermo-mechanical processing and δ phase fraction in DA Alloy 718. Based on these results, a δ annealing treatment before the final forging step of Alloy 718 is shown to deliver superior strength, which is beneficial for manufacturing superior aerospace parts.

CRediT authorship contribution statement

Vitor V. Rielli: Writing – review & editing, Writing – original draft, Visualization, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. Ehsan Farabi: Writing – review & editing, Investigation, Formal analysis, Conceptualization. Floria Godor: Writing – review & editing, Software, Resources, Conceptualization. Christian Gruber: Writing – review & editing, Software, Resources, Conceptualization. Aleksandar Stanojevic: Writing – review & editing, Resources, Conceptualization. Bernd Oberwinkler: Writing – review & editing, Supervision, Resources, Funding acquisition, Conceptualization. Sophie Primig: Writing – review & editing, Supervision, Resources, Funding acquisition, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

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