Original Article

Correlation between grain boundary character distribution and δ-phase precipitation in nickel-based superalloy 718

Flávia da Cruz Gallo\textsuperscript{a,*}, Luiz Maurício Barreto de Azevedo\textsuperscript{a}, Cilene Labre\textsuperscript{b}, Leonardo Sales Araújo\textsuperscript{a}, Jean Dille\textsuperscript{a,c}, Luiz Henrique de Almeida\textsuperscript{a}

\textsuperscript{a} Metallurgical and Materials Engineering – COPPE/Federal University of Rio de Janeiro, Centro de Tecnologia, Bl.F, CEP: 21941-914, Rio de Janeiro, Brazil
\textsuperscript{b} Brazilian Center of Research in Physics – CBPF, R. Dr. Xavier Sigaud, 150, CEP: 22290-180, Rio de Janeiro, Brazil
\textsuperscript{c} Materials Engineering, Characterization, Processing and Recycling – 4MAT/Université Libre de Bruxelles, 50 Avenue FD Roosevelt, CP194/03, 1050 Brussels, Belgium

\textbf{ABSTRACT}

The nickel-based superalloy 718 after processing through three thermomechanical routes, i.e., forged, hot rolled, and cold rolled, was investigated considering its δ-phase (Ni3Nb-DOa) intergranular precipitation. The samples were subjected to different combinations of annealing and aging heat treatments to obtain different grain boundary character distribution (GBCD) as well as different δ-phase volume fractions and morphologies. While δ-phase precipitation can be beneficial to avoid grain growth during recrystallization, an increase in volume fraction can be detrimental in several aspects. Deliberate manipulation of GBCD through thermomechanical processing was found to be a key to improving Ni-based superalloy properties. The present study quantified δ-phase occurrences along the GBs based on electron backscattering diffraction analysis to identify preferential precipitation sites in consonance with GB misorientation angles. The results show that random high-angle boundaries with Σ > 29 are more populated with δ-phase than low coincidence site lattices (CSL), Σ < 29, special boundaries, focusing specifically on Σ3\textsuperscript{a} class of boundaries. Nonspecial triple junctions (0- and 1-CSL) are also preferential nucleation sites when compared to 2- or 3-CSL triple junctions.

© 2019 The Authors. Published by Elsevier B.V. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).

1. Introduction

The nickel-based superalloy 718 (UNS N07718) shows an excellent combination of high strength and good corrosion resistance at temperatures as high as 650 °C \cite{1,2}. The alloy's good mechanical properties are mainly because of the precipitation of a coherent, ordered, and metastable γ′-Ni3Nb intermetallic phase with a DO22 tetragonal structure \cite{3}.

\textsuperscript{*} Corresponding author.
E-mails: flaviagallosoglobo.com, flacruz@metalmat.ufrj.br (F.C. Gallo).
2238-7854/© 2019 The Authors. Published by Elsevier B.V. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).
In addition, an important microstructural aspect of alloy 718 is the formation of the stable Ni3Nb δ-D0α phase (δ-phase) during processing or service at a wide range of temperatures and with various morphologies, volume fractions, and distributions [4–6]. As demonstrated by Sundararaman et al. [4], at temperatures up to 900 °C, the δ-phase is formed from dissolution of the metastable γ and, above that temperature, the δ-phase can precipitate directly from the γ matrix.

A controlled precipitation of the δ-phase can be used to pin grain boundary (GB) movement during recrystallization and avoid grain growth [7]. This leads to a refined microstructure and an increase in rupture stress, fatigue resistance, and ductility [8]. In contrast, the increased volume fraction of δ-phase may result in loss of hardenability and plasticity and in deterioration of the fatigue performance of alloy 718 at room temperature, due to the reduction in volume fraction of γ [4,9–11]. Besides that, when the alloy is subjected to hydrogen-enriched environments, the presence of the δ-phase can also embrittle the material by acting as a preferential site for hydrogen trapping and fracture initiation [12]. However, Valle et al. [8] contradicted the previous statement by arguing that the δ-phase has no considerable influence on the mechanical strength of aged alloy 718 at room temperature.

At specific misorientations, the coincidence site lattice (CSL) concept relates to the reciprocal density of coincident atomic sites within two adjoining grain lattices and is denoted by Σ [13]. Being referred to as a reciprocal value, the lower the Σ number, the higher the degree of order between the lattices.

In recent years, the control of grain boundary character distribution (GBCD) through iterative thermomechanical processing (TMP) has become an important step in many stainless steel- and Ni-based superalloy property optimization [14–19]. Random high-angle boundaries (HABs), with Σ > 29, usually present higher susceptibility to intergranular degradation [20,21]. By increasing the length fraction of low CSL boundaries (3 < Σ < 29) and special triple junctions (TJs), the connectivity of random HABs was consequently decreased [22,23]. Hence, TJs adjoining at least two special low CSL boundaries are more desirable to break down the HABs network in the material and consequently decrease the conglomeration of the preferred path to failure propagation [17,24,25].

In face centered cubic (FCC) Ni-based alloys with low-to-medium stacking fault energy, annealing twins that present a {123} 60°/(111) orientation can readily be formed by properly designed TMP. Processing induces the formation of Σ3 while also promoting an intense interaction between these boundaries leading to a significant increase in the proportion of Σ9 and Σ27 through a phenomenon proposed by Randle [18] called “Σ3 regeneration model”. This group of special boundaries is known as Σ3ρ and represents the most common type of low CSL boundaries to the detriment of other Σ < 29 boundaries (e.g., Σ5, Σ7, Σ11, Σ13, Σ15, Σ17, Σ19, Σ21, and Σ25) [17].

Despite the expected low energy associated with low-angle and low-CSL boundaries, it is complex to establish a direct correlation between the Σ value and the GB energy [26]. Likewise, boundary mobility cannot be directly correlated with the Σ value or boundary energy [27]. As an example, depending on their tilt or twist nature, Σ3 boundaries present completely different mobilities and energies, although they are classified with the Σ notation [17].

Nonetheless, CSL is still the most common model used to relate the GB nature with intergranular phenomena, such as segregation, precipitation morphology and distribution, stress corrosion, cracking susceptibility, sensitization and hot corrosion, creep, and fatigue resistances [28–33].

These studies have shown that all Σ3 and Σ3ρ potentially have restricted responses to intergranular failure and are desirable.

In the light of the effect of GBCD in phase precipitation behavior, many researchers have focused on carbides and the way their morphology and distribution affect intergranular failure phenomena [30,34–39]. However, none have yet addressed δ-phase precipitation and its correlation to CSL boundaries. Even though Ida et al. [40] claim that there is a difference between the precipitation behaviors of δ-phase on GBs according to the GB energy and misorientation, they emphasize the difference between low angle (<15) and HABs. Carbide precipitation in Ni-based superalloys are more frequent in HABs and incoherent twins, and their morphology changes depending on the nucleation site [34,35].

Therefore, the aim of the present work was to assess the frequency of experimental observation of the δ-phase in alloy 718 and identify the systematic behavior of intergranular precipitation, particularly high-energy random HABs versus Σ3ρ special boundaries. Thus, the empirical data were based on three groups of alloy 718 samples processed using different TMP routes. The results showed that precipitation of the δ-phase at GBs is not homogenous and different GB characteristics can be correlated with different precipitation frequencies.

2. Materials and methods

To obtain different δ-phase volumetric fractions, different proportions of annealing twins and other Σ3ρ boundaries, two Ni-based superalloy 718 industrial products were used as starting materials: i.e., a 1 mm thickness hot-rolled sheet (HRS, UNS N07718) and a 3 mm hot-forged bar (FB, modified alloy 718). Two distinct chemical compositions were evaluated, being one under the regular specification for the UNS N07718 class, while the other one was a modified composition, which was used for an aircraft component. Among the effects of such compositional difference, the foremost significant for the present study is the effect of the higher Nb content on δ-phase precipitation, its fraction and character distribution. Table 1 presents the chemical composition of the materials.

A diverse precipitation behavior of δ-phase is of major importance for this investigation inasmuch as the goal is to assess a trend in the distributive character of intergranular δ-phase precipitates under different manufacturing and processing conditions. Besides the difference in chemical composition, thermomechanical processing was also diversified to reinforce the empirical observations. Hence, the TMP of the materials followed three different routes:

HRS: The as-received HRS was subjected to heat treatments comprising solution annealing at δ-phase supersolvus temperature of 1050 °C for 30 min and aging at 900 °C for various aging times (i.e., 2, 6, and 24 h). Both steps were followed by water quenching. The studied samples are HRS-SA, HRS-2 h,
HRS-6 h, and HRS-24 h. CRS: The as-received HRS underwent TMP in a single cold-rolling step from 1.0 to 0.4 mm thickness (60% deformation) followed by annealing at a δ-phase subsolvs temperature of 975 °C for 75 min for recrystallization and overaging at 975 °C for 1 and 96 h followed by water quenching. The studied samples are CRS-1 h and CRS-96 h.

FB: The FB was solution annealed at a δ-phase subsolvs temperature of 954 °C for 1 h, aged at 760 °C for 5 h, cooled down to 649 °C and kept for 1 h, and then air cooled. Studied sample is FB.

Aging temperatures were chosen to embrace different δ-phase morphologies and aging times representing evolution in δ-phase volume fractions. According to the published time–temperature–precipitation diagrams for alloy 718 [1,41], HRS samples were aged at 900 °C. This temperature corresponds to the maximum kinetics for the precipitation of δ-phase and is related to a needle-like Widmanstätten precipitation structure. The CRS group was aged at a higher temperature, 975 °C, which usually leads to the formation of spheroidized δ particles with increased volume fraction. At temperatures lower than 900 °C, such as that of 760 °C used to age FB samples, GB platelets were observed frequently [1,5,9,41].

The δ-phase morphology was characterized by scanning electron microscopy (SEM) in the backscattered electron (BSE) imaging mode. Metallographic samples were etched by immersion for 90 s in a glyceregia solution of 60 ml HCl, 20 ml HNO3, and 40 ml glycerin.

For EBSD determination, samples prepared in an automatic polishing disc machine with a solution of colloidal silica and hydrogen peroxide (1:1) for 60 min were analyzed in a field-emission gun (FEG)-SEM equipped with an electron backscatter diffraction (EBSD) detector, with 15 kV accelerating voltage, spot size of 14, working distance of 15 mm and 0.1 μm step size. At least three nonadjacent fields with a size of 1000 by 1000 μm were scanned from each sample to achieve a sufficient grain boundary statistic, encompassing a minimum number of 500 grains each and representing the overall microstructure.

The EBSD data were processed using an open-source MATLAB® extension METEX Software Toolbox (version 5.1.1; METEX, Germany) [42]. The average grain size was determined from the EBSD data. The characterization of low CSL boundaries (3 < Σ < 29) was based on Brandon’s criterion (ΔΦ < 15° / Σ−1/2) [43] and all other non-CSL boundaries with misorientation angle ΔΦ > 10 were classified as random HABs. TJs were categorized as: 0-CSL (3 HABs), 1-CSL (2 HABs + 1 Σ3), and 2-CSL (1 HAB + 2 Σ3), 3-CSL (Σ Σ2). The area fraction of the δ-phase was also determined from the EBSD analysis based on the correlation between the collected Kikuchi patterns and phases in the crystallographic structures. More than 1000 δ-phase intergranular precipitates were investigated for experimental determination of their occurrence location and divided into six categories: i.e., random HABs (Σ > 29), special low CSL Σ3n, 3-CSL, 2-CSL, 1-CSL, or 0-CSL TJs.

### 3. Results and Discussion

#### 3.1. Microstructural aspects

The HRS samples aged at 900 °C, close to the maximum kinetics for δ-phase precipitation, presented needle-like precipitates, as shown in the SEM/BSE images in Fig. 1. After aging for 2 h, fine particles were observed in Fig. 1(a), growing either along the GB or toward the grain interior. A bulky MC-type carbide was also observed. After 6 h of aging, Fig. 1(b) shows thicker needles of the δ-phase, with some crossing the whole width of a grain and a colony of very finely spaced needles of the δ-phase.

Fig. 2 shows the SEM/BSE images of the CRS samples heat treated at 975 °C for 1 h (CRS-1h) and 96 h (CRS-96h), respectively. Above the temperature of the maximum kinetics of δ-phase precipitation in alloy 718, the phase morphology changes from being plate-like in the early stages of aging to coarser round-shaped particles as the aging time increases [41].

Comparing Fig. 2(a) and (b), the brighter bulky δ-phase coarsens with the increase in aging time.

Fig. 3 presents a SEM/BSE image of the modified alloy 718 FB sample. An intergranular δ-phase precipitation occurs along the GBs in a continuous manner as a result of the lower solution-annealing temperature if compared to the two previously studied conditions.

Local orientation measurements using orientation imaging microscopy (OIM) were performed to characterize the GB type and correlate it with the intergranular precipitation of the δ-phase. The OIM data sets were processed and the phase maps are presented in Fig. 4. The indexed δ-phase (green) within the γ matrix (white) is combined with the boundary distribution map to enclose the quantitative analysis of phase locations.

Fig. 4(a) and (b) shows the phase maps of samples (a) HRS-2 h and (b) HRS-24 h, respectively. Since the early stages of aging, the δ-phase is observed to nucleate mainly at random Σ > 29 HABs, which are labeled with black arrows. The TJs populated with particles are highlighted with circles.

Fig. 4(c–e) shows the EBSD-processed phase maps coupled with CSL boundary maps of samples CRS-1 h, CRS-96 h, and FB, respectively. Several δ particles identified with black arrows were precipitated in random Σ > 29 boundaries, although the majority were located at TJs, mainly 0-CSL and 1-CSL, as circled and labeled in the figures. Additionally, some low-CSL Σ3 and Σ9 boundaries were populated with δ particles and labeled with red and green arrows, respectively. The subscript ‘i’ relates to an incoherent Σ3 segment and the subscript ‘c’ relates to a coherent Σ3 segment. A common feature observed

### Table 1 – Chemical composition of the starting materials (wt.%).

<table>
<thead>
<tr>
<th></th>
<th>Ni</th>
<th>Cr</th>
<th>Fe</th>
<th>Nb</th>
<th>Mo</th>
<th>Al</th>
<th>Ti</th>
<th>Co</th>
<th>Mn</th>
<th>Si</th>
<th>C</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>HRS, UNS N07718</td>
<td>52.73</td>
<td>18.45</td>
<td>18.71</td>
<td>5.06</td>
<td>2.92</td>
<td>0.56</td>
<td>1.01</td>
<td>0.11</td>
<td>0.006</td>
<td>0.09</td>
<td>0.04</td>
<td>0.02</td>
</tr>
<tr>
<td>FB, modified alloy 718</td>
<td>46.6</td>
<td>19.52</td>
<td>18.81</td>
<td>6.08</td>
<td>2.26</td>
<td>0.453</td>
<td>3.86</td>
<td>1.49</td>
<td>0.331</td>
<td>0.221</td>
<td>0.065</td>
<td>0.277</td>
</tr>
</tbody>
</table>
in Fig. 4(a–e) is that most of the \( \Sigma^3 \) boundaries are free of \( \delta \)-phase precipitation. Table 2 summarizes the length fractions (%) of \( \Sigma^3 \) boundaries (\( \Sigma^3 + \Sigma^9 + \Sigma^{27} \)) and other low-CSL boundaries (\( \Sigma^5, \Sigma^7, \Sigma^11, \Sigma^13, \Sigma^15, \Sigma^17, \Sigma^19, \Sigma^21, \text{and} \Sigma^25 \)), area fraction of the \( \delta \)-phase (%), and average grain size (\( \mu m \)) for all studied samples.

Samples HR presented \( \Sigma^3 \) length fraction of around 40% from 0 to 6 h of aging. After 24 h of aging at 900 °C, the generation and multiplication of annealing twins was probably the most intense, achieving a length fraction of approximately 55% of \( \Sigma^3 \). An increased proportion of \( \Sigma^3 \) boundaries was achieved for CRS group samples, which underwent TMP to manipulate GBCD after 1 h of aging, and kept constant around 55% up to 96 h. A similar length fraction of \( \Sigma^3 \) boundaries was present in the forged FB sample. For all studied samples and conditions, the total-length fraction of low CSL, \( \Sigma^5, \Sigma^7, \Sigma^11, \Sigma^13, \Sigma^15, \Sigma^17, \Sigma^19, \Sigma^21, \) and \( \Sigma^25 \) boundaries ranged approximately between 5–10% of the \( \Sigma^3 \) length fraction. This finding (see Table 2) is in accordance with Randle’s proposal [17] and configures the reason why the present study avoids considering the occurrence of \( \delta \)-phase on low CSL boundaries other than the \( \Sigma^3 \) class.

No \( \delta \)-phase precipitates were observed in the solution-annealed material. The area fraction of the \( \delta \)-phase increased significantly in the HR group of samples from 0.2% after 2 h of aging to >2% after 24 h of aging at 900 °C. For the CRS group, a slight increase in area fraction from 1.1% after 1 h of aging to 1.8% after prolonged aging for 96 h was observed. Confronting this result with the values obtained for the HRS group, aging at 975 °C resulted in delayed kinetics of precipitation and growth. While aging at 900 °C resulted in around 2% of \( \delta \)-phase area fraction after 24 h of aging, overaging at 975 °C resulted in an equivalent fraction of precipitates only after 96 h of heat treatment. However, the FB presented over 3.5% of \( \delta \)-phase due to its modified chemical composition of alloy 718, with an increased Nb content.

While the HRS samples showed an enlarged average grain size of >11 \( \mu m \), the applied TMP notably effectively kept refined...
Fig. 4 – EBSD data processed using MTEX Software Toolbox (version 5.1.1) shows the γ matrix in white and δ particles in green in combination with the CSL boundary mapping (Σ3 = red, Σ9 = green, Σ27 = light blue, and HABs = black) for samples (a) HRS-2h; (b) HRS-24h; (c) CRS-1h; (d) CRS-96h; and (e) FB. Several δ particles at random HABs are labeled with black arrows, Σ3 (coherent and incoherent) and Σ9 precipitates are identified with red and green arrows, respectively, and δ particles at TJs are highlighted in circles.

Table 2 - Summary length fraction (%) of Σ3^n boundaries (Σ3 + Σ9 + Σ27), length fraction (%) of other low CSL boundaries, area fraction of δ-phase (%) and average grain size (μm) for all studied samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Σ3^n</th>
<th>HRS-2h</th>
<th>HRS-6h</th>
<th>HRS-24h</th>
<th>CRS-1h</th>
<th>CRS-96h</th>
<th>FB</th>
</tr>
</thead>
<tbody>
<tr>
<td>HRS-SA</td>
<td>40.01</td>
<td>±5.06</td>
<td>40.77</td>
<td>±3.40</td>
<td>40.19</td>
<td>±3.97</td>
<td>54.36</td>
</tr>
<tr>
<td>HRS-2h</td>
<td>4.31</td>
<td>±0.76</td>
<td>5.01</td>
<td>±1.75</td>
<td>3.78</td>
<td>±0.69</td>
<td>2.57</td>
</tr>
<tr>
<td>HRS-6h</td>
<td>4.99</td>
<td>±1.12</td>
<td>4.14</td>
<td>±0.54</td>
<td>4.00</td>
<td>±0.49</td>
<td>3.91</td>
</tr>
<tr>
<td>HRS-24h</td>
<td>55.06</td>
<td>±3.19</td>
<td>54.20</td>
<td>±1.99</td>
<td>54.20</td>
<td>±1.02</td>
<td>55.06</td>
</tr>
<tr>
<td>CRS-1h</td>
<td>3.60</td>
<td>±0.54</td>
<td>3.57</td>
<td>±0.15</td>
<td>3.57</td>
<td>±0.15</td>
<td>3.57</td>
</tr>
<tr>
<td>CRS-96h</td>
<td>4.25</td>
<td>±0.84</td>
<td>4.25</td>
<td>±0.84</td>
<td>4.25</td>
<td>±0.84</td>
<td>4.25</td>
</tr>
<tr>
<td>FB</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
grains in the CRS samples. The FB sample also presented a refined microstructure.

### 3.2. Quantitative analysis of GB precipitation of δ-phase

Table 3 presents the compilation of the experimentally observed frequency of δ-phase precipitation occurrence (%) as classified by GB type (HABs and Σ3 boundaries) as well as by TJs (0-CSL, 1-CSL, 2-CSL, and 3-CSL) for all studied samples. At least 1000 δ particles were statistically computed and for each studied condition, a fraction (%) was calculated based on the number of particles recorded by GB location. The particles were considered to be either nucleating at a GB or a TJ, with no double counting.

Considering the insignificant proportion of the non-Σ3 CSL under Σ < 29 (Σ5, Σ7, Σ11, Σ13, Σ15, Σ17, Σ19, Σ21, and Σ25) in alloys’ GBCD (see Table 2), as well as the substantial relevance of Σ3 boundaries, specially annealing twins by the sigma-regeneration process [18] in the manipulation of character distribution, other low-CSL boundaries were disregarded from the quantitative analysis of δ-phase precipitation according to the GB characteristic.

For HRS samples, the vast majority of δ-phase occurrences are at random Σ > 29 HABs, with a peak fraction after aging for 6 h, with >85% of the accounted intergranular δ precipitates. The fraction of precipitates populating Σ3 GBs become more appreciable only in the HRS-24 h sample (~20%). It is possible to note the abrupt increase in δ-phase area fraction in the HRS-24 h sample, which leads to a saturation of possible nucleation sites for that aging time (Table 2). No particles were observed in 3-CSL TJs for any aging time and only <3% of precipitates occupied 2-CSL TJs after 24 h.

The CRS samples that underwent TMP to manipulate GBCD presented with reduced grain size and consequently lower connectivity of HABs due to the increased proportion of Σ3. The precipitation of the δ-phase was significantly concentrated in random Σ > 29 HABs and TJs, particularly the nonspecial 0- or 1-CSL ones. After 6 h of aging, almost 50% of δ-phase occurrences were at random Σ > 29 HABs and >31% at nonspecial 1-CSL TJs.

In the FB sample, having higher Nb content, the presence of δ-phase along Σ3 boundaries represented around 11% of the total, similar to the other samples’ percentage. Although the proportion of δ at random HABs decreased, this reduction was compensated by a more intense precipitation at nonspecial TJs, therefore not altering the trend of δ precipitation. Nonspecial TJs (0- and 1-CSL) as well as random Σ > 29 HABs represent majority of occurrences sites, summing up >75% of the particles, close to the values observed for HRS-24 (76,24%), CRS-1 h (78,62%) or CRS-96 h (80,85%).

A systematic understanding of the factors affecting the intergranular precipitation of the δ-phase can be achieved when analyzing the results in terms of GB misorientation and energy.

Ida et al. [40] studied a Ni-Fe-Nb alloy and noted that δ-phase precipitates at GBs rather than the grain interior; however, there is no occurrence in low-angle boundaries with misorientation angles of <15. The great scattering found regarding δ-phase nucleation and growth at HABs with misorientation angles between 15 and 60 was not addressed.

Rohrer [26] noted that despite the CSL model not being a direct predictor of GB energy, some particular tilt and twist directions represent low-energy configurations for HABs and were associated especially with Σ3 coherent twins in some different FCC materials. Wasnik et al. [44] observed an unexpected behavior in 304 and 316L stainless steels, where materials with both low and high random HAB fractions showed relatively better resistance to sensitization and intergranular corrosion. Therefore, it is common sense that all Σ3 potentially present special properties, i.e., mainly Σ3 coherent twins and Σ9 boundaries [17]. Li et al. [35] also claim that both GB misorientation and GB planes determine the GB energy. Annealing twins terminated by (111) planes as well as Σ9 tilt boundaries have the lowest energies.

In addition, minimum GB diffusivity can be associated with CSL misorientations for both tilt and twist boundaries, as demonstrated by Monzen et al. [45]. Oudriss et al. [46,47] studied the atomic diffusion of hydrogen in pure Ni and claim that it is often accelerated in interfaces with increased structural disorder, which can be associated with high-energy random Σ > 29 HABs. Inversely, low-CSL special boundaries are potential trapping sites for hydrogen due to the ordered structure, accommodation of defects, and low free volume.

The diffusion of Nb within the Ni matrix is claimed to be the determining micromechnism for δ-phase precipitation according to calculations of average activation energy [48]. The results compiled in Table 3 are in accordance with a potentially favored elemental diffusion through random Σ > 29 HABs, where precipitation nucleation and growth of δ-phase are preferred, as well as nonspecial TJs (0-CSL and 1-CSL).

Additionally, Trillo and Murr [49,50] claimed that there is a critical interfacial energy required to the onset of intergranular precipitation. That critical energy would be somewhere between corresponding energy of coherent Σ3 boundaries (segments usually free of precipitation) and the
energy associated with incoherent $\Sigma 3$ segments, where there are precipitates. That statement aligns with the observed in Fig. 4, where the incoherent segments of $\Sigma 3$ boundary (labeled as $\Sigma 3i$) are more susceptible to be populated by $\delta$ particles, whereas the majority of coherent $\Sigma 3$ boundaries (labeled as $\Sigma 3c$) are free of precipitation. Moreover, the authors [49,50] observed that the previous degree of cold deformation widens the misorientation angle interval where precipitation occurs. This is in accordance to the quantitative results presented in Table 3, especially when comparing HRS-24h with CRS-96h samples. It is possible to note that for the TMP group of samples (CRS), there was considerable overall increase in the presence of particles at special TJs (3-CSL and 2-CSL) for prolonged aging times.

4. Conclusions

Two distinct starting materials subjected to different TMP presented differences in $\delta$-phase intergranular precipitation, such as morphology, size, and distribution, due to the mechanisms of nucleation and growth. The following conclusions can be stated from the results and discussion presented:

- $\Sigma 3^\circ$ special boundaries are less susceptible for $\delta$-phase precipitation in alloy 718 in all studied conditions. High-energy $\Sigma > 29$ random HABs are preferable sites for $\delta$-phase precipitation, representing up to 85% of all $\delta$ particles at one of the conditions;
- Incoherent $\Sigma 3$ boundary segments are preferentially populated with $\delta$ particles despite the coherent segments of $\Sigma 3$-annealing twins;
- Special TJs (2-CSL and 3-CSL) are also improbable $\delta$-phase precipitation sites. Several samples did not show any $\delta$ particles at those TJs, contrasting with the nonspecial TJs (i.e., 0- and 1-CSL TJs), which summed up to 40% of the intergranular $\delta$ particles in one of the studied samples;
- Despite the variation of samples’ compositions (regular and modified UNS07718), subjected to different TMPs, a similar behavior was evidenced regarding the correlation between intergranular $\delta$-phase occurrence and grain boundary character distribution.

Conflcits of interest

The authors declare no Conflcits of interest.

Acknowledgments

The authors would like to thank LABNANO/CBPF for technical support during electron microscopy work, Roberta M. de Santana from CEPFL for metallographic preparation, and CAPES for the financial support.

References


