Microstructural characteristics of forged and heat treated Inconel-718 disks

A. Chamanfar a,⁎, L. Sarrat a, M. Asadi b, A. Weck b, A.K. Koul c

a Département de Génie Mécanique, École de Technologie Supérieure, 1100 rue Notre-Dame Ouest, Montréal, Québec H3C 1K3, Canada
b Department of Mechanical Engineering, University of Ottawa, Ottawa, Ontario K1N 6N5, Canada
c Life Prediction Technologies Inc., 23-1010 Polytek Street, Ottawa, Ontario K1J 9J1, Canada

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Microstructure evolution from center to edge of the as-forged and heat treated Inconel-718 disks was investigated. Specifically, the evolution of primary carbides, grain size, γ′, γ″, δ, and secondary carbide particles was the focus of the current study. In fact, characterization of these microstructure features is essential for models predicting the creep and fatigue lives of the alloy. Accurate and reliable revealing of the grain boundaries in as-forged and heat treated Inconel-718 was made possible in this study by development of a new method. From microstructure investigations, nonuniformities in grain size, volume fraction, size and inter particle spacing of precipitates from center to edge were observed in both as-forged and heat treated disks. The microstructure nonuniformities resulted in significant variation in hardness from center to edge of the disks.

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1. Introduction

Inconel-718 is a Ni–Fe–Cr superalloy widely used as disk material in gas turbine and jet engines with service temperatures up to 650 °C because of its high temperatures strength and corrosion resistance [1–3]. The nominal chemical composition of Inconel-718 is 52.50 Ni–18.50 Fe–19.00 Cr–5.10 Nb–3.00 Mo–0.50 Al–1.01 Ti–0.08 C [3,4]. This alloy gains its high temperature strength mainly through precipitation of body-centered tetragonal (BCT) γ″, Ni3Nb, dispersed in the γ matrix [1]. Also, face-centered cubic (FCC) γ′, Ni3(Al, Ti), supply additional strength for the alloy [5]. γ″ usually precipitates at higher aging temperatures (720 °C) whereas γ′ precipitates at lower ones (620 °C) [6]. For the fully heat treated alloy, i.e., solution heat treated and double aged, the volume fraction of γ″ and γ′ are 20% and 5%, respectively [7,8]. The solvus temperatures of γ″ and γ′ are about 900–920 °C [3]. γ″ particles are metastable and long term exposure to temperatures above 650 °C, transform into the stable form of Ni3Nb, i.e., the δ phase, leading to degradation of mechanical properties [1,9]. Alloying elements such as Fe, Cr, Mo, Al, and Ti also strengthen the alloy through solid solution mechanisms [9]. In addition, MC-type carbide, M being mostly Nb, and orthorhombic δ, Ni3Nb, precipitate at grain boundaries and inhibit grain boundaries sliding at high temperatures thereby improving creep properties [5]. Aluminium and Cr form the protective impermeable Al2O3 and Cr2O3 oxide films that supply the corrosion resistance for the alloy [9].

Inconel-718 disk is manufactured through casting, forging, and heat treatment. The microstructure changes induced during the manufacturing process affects the performance of the alloy during service. Reliable revealing of different microstructural features is a key element for process optimization and development of material-based finite element models that predict the service capabilities such as fatigue and creep lives.

In order to predict the fatigue and creep lives in Inconel-718 disk, it is essential to characterize and quantify the various microstructural features in the alloy. Specifically, grain size, the morphology of primary and secondary carbides, γ″, γ′, and δ precipitates, their sizes and volume fractions, as well as their inter particle spacing at the grain boundaries (δ and secondary carbides) are the key input parameters for creep life modeling [10]. Comprehensive review of the literature indicated that characterizing and quantification of all of the microstructure constituents in Inconel-718 have been rarely carried out. The focus has been mainly on grain size evolution with temperature, strain, and strain rate of deformation on lab scale samples where deformation conditions have been carefully controlled [11–13].

The lack of quantitative data on microstructure of Inconel-718 can be partly due to difficulties encountered in revealing various microstructure constituents. For example, at the early stages of the current study, it was found that revealing clearly the grain boundaries in solution heat treated and/or hot forged Inconel-718 was very difficult when classical approaches proposed in
handbooks were used. Similarly, in a recent work [13], it can be observed that grain boundaries are not clearly revealed in Inconel-718 hot forged at 1100 °C and 0.1 s⁻¹ to strain of 0.03 and 0.05. Lack of proper revealing of grain boundaries is an important impediment in the reliable estimation of the grain size and morphology as a function of thermomechanical processing conditions.

Hence, at the early stages of the present work a method was developed which allowed reliable and repeatable revealing of grain boundaries in samples extracted from different locations of as-forged and heat treated disks. The disks were sectioned from a nonisothermally forged Inconel-718 billet and a heat treatment was applied on one of them. Furthermore, metallography procedures were developed to reveal and determine reliably the size, volume fraction, and inter particle spacing of other microstructural features such as primary carbides, γ', γ, δ, and secondary carbide particles in different locations of the disks. Such data are key inputs for models predicting the creep life of the disks as presented in another publication.

2. Experimental material and procedures

Two Inconel-718 disks, 32.4 cm in diameter were received. They were sectioned from a nonisothermally forged Inconel-718 billet. One of the as-received disks was in the as-forged condition and the other in the heat treated condition, i.e., solution heat treated and double aged. The solution heat treatment was performed at 954 °C for 1 h followed by air cooling. The double aging was carried out at 718 °C for 8 h, followed by furnace cooling to 621 °C at a rate of 56 °C/h, second aging at 621 °C for 8 h, and then air cooling to room temperature.

Metallography samples were extracted from the center, midradius, and edge of the as-forged and heat treated disks by wire electrodischarge machining. After mounting, automated grinding and polishing procedures were used to prepare the samples for microstructure studies. The details of grinding and polishing procedures are provided in Table 1. For grinding 320, 600, 800, and 1200 SiC papers were used. After grinding, the samples were polished on a MD Nap cloth for 2 min while using 1 μm diamond suspension as polishing solution. Final polishing of the samples was carried out with colloidal silica suspension (OP-S) on a vibratory polisher for 12 h. Characterization of primary carbides was carried out in the as-polished condition using optical microscopy (OM). To reveal...
the grain boundaries the samples were swabbed with Kalling reagent (5 g CuCl₂, 100 mL HCL, and 100 mL ethanol) using a cotton stick. Grain size studies were performed using OM. For characterization of γ', γ', δ, and secondary carbide particles with a high resolution field emission gun scanning electron microscope (FEG-SEM), the samples were electroetched at 2 V for three seconds in a solution composed of 8 ml H₂SO₄ and 100 ml H₂O.

Grain size measurements were carried out using the intercept method. The volume fraction of different particles was determined using the ASTM: E562, assuming that area fraction is equal to volume fraction. Clemex® image analysis software was used for size and inter particle spacing measurements of different particles. To have statistically representative results, at least 6 micrographs were considered for grain size measurement and characterization of different particles. Furthermore, for measuring the sizes of primary carbides, γ', γ', δ, and secondary carbide particles on average 40, 37, 43, 47, and 23 particles were considered respectively for each location.

To relate microstructure evolution to mechanical properties at different locations in as-forged and heat treated disks, Rockwell A hardness measurements were carried out at least at five locations on each sample. The distance between each indent was greater than five times the indentation diameter. To compare the hardness data with literature, the Rockwell A hardness values were converted to Vickers hardness using ASTM: E140. The average of the Vickers hardness values for each sample was determined and reported in this study.

3. Results and discussion

3.1. Primary carbides

Fig. 1 shows the primary MC-type carbides observed in the as-polished microstructures of the samples extracted from center, midradius, and edge of as-forged and heat treated disks. The average diameter and volume fraction of these particles are presented in Fig. 2 for the investigated locations. It can be seen that, there is no significant change in the size and volume fraction of the primary MC-type carbides from center to edge in both disks. Moreover, for a given location the sizes and volume fractions of primary MC-type carbides in two disks were very close and the slight difference lies within the error range of experimental measurements. These can be related to the fact that primary MC-type carbides are very stable and do not degenerate, even at temperatures very close to the bulk melting point of the alloy.

Because of a relatively low volume fraction, about 1.5% (Fig. 2), and almost uniform distribution, primary MC-type carbides have little influence on the mechanical properties of superalloys [9]. However, these particles are important for grain size control during thermomechanical processing since they pin the grain boundaries and inhibit grain growth.

3.2. Grain size evolution

The optical microstructure of the samples extracted from center to edge of the as-forged and heat treated Inconel-718 disks are presented in Fig. 3. It is worth noting that, initial metallography results indicated that revealing the grain boundaries in the hot forged and/or solution heat treated Inconel-718 was not possible using emersion etching or electroetching even after attempting different solutions. The developed method in the current work, swabbing the polished surface with Kalling reagent, resulted in clear revealing of the grain boundaries of the as-forged and heat treated Inconel-718 (Fig. 3). Such achievement was very useful for reliable microstructure characterization and determination of the grain size data as input for creep life prediction model [10]. The observation of the microstructure (Fig. 3) clearly indicated finer grains at the edge compared to the central region in both disks.

The evolution of the grain size from center to edge of the disks is shown in Fig. 4. For the as-forged disk, the grain size continuously decreases from the center to the edge of the disk. This is due to variation in temperature, strain, strain rate, and inter pass time during the nonisothermal forging process. For instance, in this process there is a temperature gradient from center to edge since the temperature at edge drops more quickly compared to the center because of heat loss through radiation to surrounding environment and conduction to cooler dies. Such variations in grain size can be interpreted in terms of Zener–Hollomon (Z) parameter which relates the dynamic recrystallized grain size (d₄₃) to temperature (T) and strain rate (c) of deformation. Specifically, for Inconel-718 this relationship has been formulated as [14]:

\[ \log(d_{4\%}) = -0.027\log(Z) - 7454/T + 8.49 \]  \( (1) \)

Where

\[ Z = c\exp(Q/RT) \]  \( (2) \)

In Eq. (2), Q and R are activation energy for hot deformation of the alloy and gas constant, respectively. Based on Eq. (1), for a constant strain rate, the lower the deformation temperature, the smaller is the dynamic recrystallized grain size. Thus, at the edge of the disk due to temperature drop, grains are finer. In fact, because of temperature drop, δ particles precipitate at the edge of the Inconel-718 disk, as it will be shown later, and inhibit grain growth by pinning the grain boundaries.

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**Fig. 2.** (a) average diameter and (b) average volume fraction of primary carbides in as-forged and heat treated (HTed) disks for various locations. The standard deviations of measurements are shown as error bars.
Similarly, there is strain gradient from center to edge in the as-forged disk. Depending on the size of the ingot, as the die deforms the ingot, the central region may not be strained and the deformation may be concentrated at the edge of the ingot. Thus, in the central region, strain can be below the critical value for the onset of dynamic recrystallization (DRX) and grain refinement may not take place. It is also noteworthy that the obtained results of grain size evolution in as-forged Inconel-718 billet are consistent with modeling results reported by Dandre et al. [15].

For the heat treated disk, the grain size for a given location was smaller than that of the as-forged disk (Fig. 4). This indicates that during heat treatment, static recrystallization (SRX), i.e. grain refinement, has taken place. It is important to note that the applied heat treatment did not lead to uniformity in grain size from center to edge of the disk. Thus, creep and fatigue properties depend on the location in the disk from which the sample is extracted. For instance, in Fig. 5, a schematic presenting the block taken from the heat treated disk for extracting creep samples and the corresponding microstructures at different locations in the creep sample are shown. Grain size evolution in the investigated locations is shown in Fig. 6. It can be seen that, there is a gradient in the grain size along the creep sample and by moving towards the edge of the disk the grain size decreases.

The evolution of grain size during forging of different superalloy components is very important since it significantly affects strength, creep, and fatigue crack initiation and growth rate. In general, one of the objectives of forging of superalloys is attainment of a uniform microstructure to have consistent mechanical properties. However, the observed variation in grain size indicates nonuniform microstructure from center to edge of the as-forged billet from which the disks were extracted. Since the applied heat treatment also did not lead to a uniform grain structure, the mechanical properties in the as-forged and heat treated disks will not be consistent. Indeed, as it is shown in Section 3.4, the mechanical properties are dependent on the location in the disks.

### 3.3. Evolution of $\delta$, $\gamma^\prime$, $\gamma^\prime$, and secondary carbide particles

In literature, it is well established that $\gamma^\prime$ and $\gamma^\prime$ have disc shape and globular morphologies, respectively, whereas $\delta$ and NbC

![Fig. 3. Optical micrographs presenting the $\gamma$ grains in the samples extracted from the center, midradius, and edge of the as-forged and heat treated disks.](image)

![Fig. 4. Grain size evolution from center to edge of as-forged and heat treated Inconel-718 disks. The standard deviations of measurements are shown as error bars.](image)
Furthermore, it has been reported [9] that forging at temperatures below the final stages of the forging process. The morphology of grain boundaries by the final stages of the forging process. Consequently, dissolution occurred at this location during forging and no δ reprecipitation occurred on post-forge cooling (Fig. 7). In contrast, for center and edge of the as-forged disk, δ dissolution did not take place, as the temperature remained below δ solvus during the final stages of the forging process.

To have a better understanding of the evolution of the δ phase with location in the disks, the size, volume fraction, and inter particle spacing of the δ phase were determined and presented in Fig. 8. As shown in Fig. 8a, for the as-forged disk, the volume fraction of δ phase is slightly higher at edge compared to the center of the disk. This is due to higher cooling rates during nonisothermal forge and post-forge cooling, and thus, higher δ nucleation rate. Furthermore, it has been reported [9] that forging at temperatures below δ solvus can significantly change the precipitation kinetics and morphology of δ phase. Indeed, since higher amounts of deformation are applied at the edge of the billet compared to its center, accelerated nucleation and growth of δ will be expected at the edge [9]. This results in the higher volume fraction of δ phase at the edge compared to the center in the as-forged billet. Similarly, due to a higher growth rate, width and inter particle spacing of δ are higher at the edge than those at the center of the as-forged disk (Fig. 8b and c).

With applying the heat treatment, the volume fraction of δ phase increased in all three investigated locations of the disk (Fig. 8a). The highest volume fraction of δ phase was observed at midradius. This may be related to super saturation of γ matrix with Nb (since δ phase was completely dissolved at midradius for the as-forged billet) and subsequent precipitation of Nb during heat treatment as δ phase. The width and inter particle spacing of the δ phase at the edge of the disk did not change significantly with applying the heat treatment (Fig. 8b and c). This is probably because the solution heat treatment temperature (954 °C) is below the δ solvus. Thus, δ phase does not dissolve to precipitate with a different size or inter particle spacing. For the central region of the disk, it was found that the width of δ phase increased with applying the heat treatment but no significant change in δ inter particle spacing was observed. The obtained results clearly demonstrated that the amount of δ phase in Inconel-718 depends on the

![Figure 5](image1.png)

**Fig. 5.** (a) A schematic showing the extracted block from heat treated disk for creep sample preparation and location of metallography samples in the creep sample and (b) the optical micrographs of samples extracted from different locations in the creep sample.

![Figure 6](image2.png)

**Fig. 6.** Grain size evolution in the creep sample extracted from the heat treated disk as shown in Fig. 5. The standard deviations of measurements are shown as error bars.
thermomechanical history. In this study, the nonisothermal forging process and the applied heat treatment resulted in the precipitation of the \( \delta \) phase with a volume fraction of \( \sim 6\% \) to \( 8\% \) as shown in Fig. 8.

It is also important to note that the characteristics of the \( \delta \) phase vary from center to edge of the as-forged billet due to variation in thermomechanical history. The application of the selected heat treatment did not lead to a uniform precipitation of \( \delta \) phase from center to edge. This results in variation in mechanical properties from the center to the edge of the disks.

3.3.2. \( \gamma'' \) and \( \gamma' \)

The evolution of \( \gamma'' \) and \( \gamma' \) volume fractions with location along the diameter of the as-forged disk is shown in Fig. 9. The volume fraction of \( \gamma'' \) at center of the as-forged disk was the highest compared to midradius and edge whereas it was almost the same at midradius and edge locations. In contrast, the volume fraction of \( \gamma' \) increased continuously from the center to the edge in the as-forged disk. The \( \gamma'' \) and \( \gamma' \) phases precipitated either during nonisothermal forge or during post-forge cooling. Since there are variations in temperature, strain, strain rate, available alloying element for precipitation, and cooling rate from the center to the edge of the billet during forging and post-forge cooling, the volume fraction of \( \gamma'' \) and \( \gamma' \) will also vary accordingly.

The equivalent sphere diameters of the disc-like \( \gamma'' \) precipitates, for various locations in the as-forged billet, are shown in Fig. 10a. The diameters of \( \gamma' \) precipitates with globular morphology for different locations in as-forged billet are presented in Fig. 10b. In general, with increasing the particle size, the hardness increases up to a peak value. It then decreases with further increase in the particle size [17]. The particle size at which the highest hardness is attained is known as the "strongest particle diameter", and its value depends on the volume fraction of the precipitates [18]. Based on the methodology presented in Ref. [18], the strongest particle diameters for \( \gamma'' \) and \( \gamma' \) precipitates were determined and the results are presented in Fig. 10 for comparison purposes. Fig. 10 indicates that, there is not significant change in the equivalent diameter of \( \gamma'' \) from center to edge of the as-forged billet. However, the equivalent diameter of \( \gamma' \) is significantly larger (3.3 times) than the strongest particle diameter.

In contrast, from center towards midradius, the diameter of \( \gamma' \) precipitates decreases, Fig. 10b. This is probably due to higher cooling rates experienced from the center to the edge during post-forge cooling cycle of the billet resulting in finer precipitates in the midradius. The average diameter of \( \gamma' \) precipitates at the edge is almost the same as that at the midradius. In contrast to \( \gamma' \), the diameter of \( \gamma'' \) at midradius and edge are relatively close to the strongest particle diameter. In summary, due to variations in thermomechanical history from center to edge of the as-forged billet, different characteristics for \( \gamma'' \) and \( \gamma' \) precipitates were observed. For the heat treated disk, \( \gamma' \) and \( \gamma'' \) particles at center, midradius, and edge were too small to be clearly resolved even by high resolution FEG-SEM (Fig. 7).

3.3.3. Secondary carbides

In the as-forged disk, secondary NbC carbides were only detected at the edge (Fig. 11). However, after applying the heat treatment, they were identified in the three investigated locations as

Fig. 7. High resolution FEG-SEM images of particles at the center, midradius, and edge of as-forged and heat treated disks.
shown in Fig. 11. The volume fraction, diameter, and inter particle spacing for secondary NbC carbides are presented in Fig. 12. The volume fraction and diameter of the secondary carbides at midradius are almost the same as those of the center in the heat treated disk, Fig. 12a and b. In contrast, these values are significantly higher for the edge location compared to the midradius and the center. This can be related to the presence of more residual plastic work at edge of the as-forged billet compared to the other two locations and thus enhancement in diffusion upon subsequent heat treatment. This statement is supported by Fig. 12a and b where it can be seen that the volume fraction and size of secondary carbides at edge increase after the heat treatment.

3.4. Hardness evolution

The hardness evolution versus location in as-forged and heat treated disks are depicted in Fig. 13. The target hardness, often reported in the literature, for solution heat treated and double aged Inconel-718 (430 HV [7]) is also shown as a horizontal line for comparison. For both disks, the hardness increases from the center towards the edge. The volume fraction and size of γ′, γ′, δ and NbC carbides, γ′–γ misfit, γ′–γ misfit, grain size, and stored energy (residual plastic work) are factors contributing to the hardness evolution [22,23]. For the as-forged disk, the increase in hardness from center to edge can be related to four distinct factors: (i) continuous grain refinement from center to edge (Fig. 4); (ii) continuous increase in the γ′ volume fraction from center to edge (Fig. 9); (iii) proximity of γ′ diameter to the strongest particle diameter at midradius and edge (Fig. 10); and (iv) presence of secondary carbides at the edge of the as-forged disk (Fig. 12). Similarly, hardness increase from center to edge in the heat treated disk can be attributed to three factors: (i) finer grains at edge compared to midradius and center (Fig. 4); (ii) higher volume fraction of secondary carbides at edge compared to the two
other locations (Fig. 12); and (iii) increase in the volume fraction of \(\delta\) phase from center to midradius (Fig. 8). Also, for a given location, the higher level of hardness in heat treated disk compared to the as-forged disk is due to the presence of finer grains (Fig. 4), higher \(\delta\) volume fraction (Fig. 8), and higher volume fraction of secondary carbides (Fig. 12).

According to SEM images shown in Fig. 7, the applied heat treatment does not appear to be optimized in terms of precipitation of \(\gamma''\) and \(\gamma'\) with proper size and morphology. The \(\gamma''\) and \(\gamma'\) in the as-forged disk were dissolved and reprecipitated as very fine particles upon applied heat treatment (Fig. 7). Considering the strongest particle diameter for \(\gamma''\) and \(\gamma'\) reported in Fig. 10, the very fine \(\gamma''\) and \(\gamma'\) particles in heat treated disk are not effective in strengthening the alloy. Thus, the target hardness was not achieved in the heat treated disk. Indeed, although the applied heat treatment is used by different researchers for Inconel-718 [8,24–30], it is totally different from the two recommended ones by Special Metal Corporation, the manufacturer of the alloy [31]:

![Fig. 10. Evolution of (a) \(\gamma''\) equivalent diameter and (b) \(\gamma'\) diameter in as-forged disk from center to edge. The standard deviations of measurements are shown as error bars. The strongest particle diameter extracted from Ref. [18] is also presented for comparison.](image)

![Fig. 11. Lower magnification SEM images of particles at the center, midradius, and edge of as-forged and heat treated disks.](image)
1- Solutionizing at 927–1010 °C followed by fast cooling, normally water quenching, plus aging at 718 °C for 8 h, furnace cooling to 621 °C at a rate of 55 °C/h, holding at 621 °C for 10 h, followed by air cooling.

2- Solutionizing at 1038–1066 °C followed by fast cooling, normally water quenching, plus aging at 760 °C for 10 h, furnace cooling to 650 °C at the rate of 57 °C/h, holding at 650 °C for 10 h, followed by air cooling.

Comparing the applied heat treatment with the first recommended one (Fig. 14), it can be observed that after solution heat treatment the material was air cooled instead of being water quenched. This change in the heat treatment led to the precipitation of very fine particles during air cooling, thus, full precipitation of \(\gamma''\) and \(\gamma'\) particles, and thus, full hardness of the alloy was not achieved (Fig. 13).

Additionally, for full precipitation of \(\gamma''\) and \(\gamma'\), and thus, full strengthening, the precipitates constituents, i.e., Nb, Al, and Ti, must be dissolved in the matrix. With the applied heat treatment, Nb precipitated as \(\delta\) phase and NbC. In fact, solution heat treatment of Inconel-718 at 960 °C for 1 h results in the precipitation of the \(\delta\) phase with a volume fraction of 2.3% [1]. In contrast, in the current study, almost a similar solution heat treatment has led to the precipitation of the \(\delta\) phase with a volume fraction of 6–8% (Fig. 8a). Thus, extra precipitation of \(\delta\) phase has depleted the matrix from Nb, consequently, complete precipitation of \(\gamma''\) has not taken place (Fig. 7) and for this reason full strength of the alloy has not been achieved (Fig. 13).

Thus, in order to achieve full precipitation of \(\gamma''\), the solution heat treatment must be carried out at temperatures significantly above \(\delta\) solvus (995 °C [9]) to dissolve some of the \(\delta\) phase in the \(\gamma\) matrix and provide enough Nb for strengthening through \(\gamma''\) precipitation. Consistent with the above analysis, it is reported [32] that solution heat treatment of Inconel-718 at 1024 °C for 0.5 h plus double aging (similar to double aging conducted on the current study Inconel-718) has led to uniform precipitation of \(\gamma''\) and \(\gamma'\) throughout the matrix and attainment of a hardness (423 HV) very close to the target value (430 HV). Furthermore, it is noted [5] that conducting the solution heat treatment above
4. Conclusions

The following conclusions can be drawn from this study:

1. Methods were proposed to clearly and reliably reveal microstructure constituents in as-forged and heat treated Inconel-718 disks. The methods allowed for a detailed examination of the influence of the process parameters on microstructural characteristics of the investigated alloy.

2. Due to variation in thermomechanical conditions from the center to the edge of the billet (from which the disks were sectioned) during nonisothermal forge, variations in grain size and characteristics of the particles were observed.

3. The applied heat treatment resulted in grain refinement. However, the nonuniformities in grain size and characteristics of the particles persisted after applying the heat treatment. As a result, for as-forged and heat treated conditions, variation in hardness from center to edge was observed.

4. Although the applied heat treatment is common for Inconel-718, it was not effective in achieving the required hardness. This heat treatment led to dissolution and reprecipitation of $\gamma'$ and $\gamma$ as very fine particles with a size significantly smaller than the strongest particle diameter.

5. To attain the required hardness, increasing the solution heat treatment temperature from $954 \degree C$ to $1035 \degree C$, water quenching (instead of air cooling) following the solution heat treat ment, and increasing the double age time from 8 h to 10 h are recommended.

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