Influence of Heat Treatment on the Distribution of Ni$_2$Nb and Microsegregation in Cast Inconel 718 Alloy

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The influences of solution and homogenization heat treatments and grain size on the microstructure and mechanical properties of cast Inconel 718 alloy have been investigated. The microstructure of as cast In718 alloy consists of primary γ, eutectic (γ + NbC) and eutectic (γ + Ni$_2$Nb). Fine and coarse grain structure specimens were achieved by controlling casting temperature. The fine grain specimen has a higher volume fraction of Ni$_2$Nb, 4.99%, than the coarse grain one, 3.45%. The volume fraction of NbC is 1.0 to 1.3% in both fine and coarse grain specimens, and almost unchanged by solution heat treatment. In case of the fine grain specimen, the as cast volume fraction of Ni$_2$Nb decreases by solution treatment to 1.26% at the temperature of 1403 K for 0.5 h holding time and completely dissolve after 4 h at the same temperature. By increasing temperature to 1440 K, it takes only 2 h to vanish Ni$_2$Nb by solution treatment to 1.26% at the temperature of 1403 K for 0.5 h holding time and completely dissolve after 4 h at the same temperature. By test is selectively initiated at Ni$_2$Nb strength for both as cast and as solution treated in compare with fine grain ones. The volume fraction of Ni$_2$Nb phase has a significant influence on the tensile strength and strain values, while it has no effect on the yield stress and hardness measurements. The fracture crack during tensile test is selectively initiated at Ni$_2$Nb phase and propagated along it in interdendritic zone.

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1. Introduction

Inconel 718 alloy, an Nb-modified Fe–Cr–Ni-base superalloy, has been widely applied to gas turbine and related parts due to its good mechanical properties and structural stability at elevated temperatures (~650°C). Alloy 718 derives its good mechanical properties at elevated temperature from a fine dispersion of DO$_2$-ordered γ" and L1$_2$-ordered γ precipitates in a face-centered-cubic γ matrix. Both these precipitates have been described as Ni$_3$(Nb, Al, Ti) with varying levels of Nb, Al and Ti.

During the solidification process, some elements such as Ni, Cr and Fe partition to the dendrite core. While other elements for instance Nb and Ti tend to accumulate in the interdendritic liquid and then solidify as the eutectic phases.

The micro-segregation of elements and non-equilibrium phases that may influence the transformation behaviors and the properties can be eliminated by a solution and homogenization heat treatments.

The alloy composition and the as cast microstructure may influence elimination process of the as-cast elemental segregation by homogenization. Therefore the understanding of multicomponent bulk diffusion process and the effect of each step of standard heat treatment on the microstructure elements distribution of the alloy, for optimization of temperature and time at which the chemical homogeneity is achieved in Ni-base superalloys is essential in a wide variety of applications.

Analysis of microsegregation remaining after progressive heat treatment reveals that the dendritic and interdendritic regions homogenize at different rates.

In this study, an attempt to find out the optimum heat treatment conditions that affects the volume fraction of Ni$_2$Nb phase. Moreover the influence of solution heat treatment and also effect of as cast grain size on the mechanical properties are investigated.

2. Materials and Experimental Procedures

The chemical composition of Inconel 718 alloy used in this experiment is given in Table 1. The alloy was melted by an induction heating and cast into Y-block type samples under vacuum, Fig. 1. Fine and coarse grain samples were prepared by pouring the melt into preheated (1273 K) ceramic mold at 1633 and 1708 K, respectively. The specimens for heat treatment and mechanical properties were taken from the cast samples, as shown in Fig. 1.

Solution heat treatments were accomplished with heating rate of 0.17 K/s and holding at different temperatures of 1360, 1403 and 1440 K (±3 K) for time range of 0 to 20 h in a muffle furnace under Ar gas atmosphere. The finally quenched in water to room temperature (RT).

The microscopic specimens were prepared by standard metallographic procedures and etched in a 10% Oxalic acid in water solution. The microstructure of the as cast and the heat treated specimens were investigated by using a Nikon optical microscope fitted with a Polaroid digital microscope camera and a Shimadzu Electron Probe Micro Analyzer-1600. The microstructure photographs, which were taken by EPMA, were introduced to a computer to determine the volume fraction of Ni$_2$Nb phase, by using a soft ware program. Additionally EPMA was used for the assessment of the diffusion coefficients of Nb in solid and measuring segregation of alloying elements.

Hardness was measured by using MVK-H1 Akashi Hardness Tester (Akashi Co. Ltd.) under a load of 30 kg. Finally tensile tests were conducted at RT at nominal strain rate of 8.33 × 10$^{-6}$ m/s using a Shimadzu Universal Testing Machine (AG-5000E).

Table 1 Chemical composition of the Inconel 718 alloy (mass%).

<table>
<thead>
<tr>
<th>C</th>
<th>Nb</th>
<th>Ti</th>
<th>Cr</th>
<th>Fe</th>
<th>Ni</th>
<th>Mo</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.06</td>
<td>4.88</td>
<td>0.95</td>
<td>19.45</td>
<td>Bal.</td>
<td>52.65</td>
<td>3.06</td>
<td>0.56</td>
</tr>
</tbody>
</table>

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3. Results and Discussion

3.1 Structures of as-cast specimens

Figure 2(a) and (b) shows the macrostructures of the as cast In718 alloy for the fine and coarse grain specimens. In718 alloy poured at 1753 K has a grain size about 3.78 mm that corresponds to ASTM 7.5 while the specimen poured at 1663 K has a grain size of 0.19 mm that corresponds to ASTM 12. The microstructure of as cast In718 alloy is consists of primary $\gamma/C_13$, eutectic $(\gamma + NbC)$ and eutectic $(\gamma + Ni_2Nb)$.

Figure 3 demonstrates the solidification microstructure of the In718 cooled at 10 K/min. Both Back Scattered Electron (BSE) and Reflected Electron (RE) images are used to distinguish NbC from $Ni_2 Nb$. The volume fractions of $Ni_2 Nb$ and NbC in the coarse grain specimen were 3.45 and 1.00% while in fine grain specimen they were 4.99 and 1.30% respectively. Additionally the secondary dendrite arm spacing of coarse and fine grain was 58.9 and 46.8 $\mu m$, indicating that the cooling rate for the coarse grain specimen is lower than that of fine grain specimen.

EPMA analysis of primary $\gamma$, eutectic $\gamma$, $Ni_2 Nb$ and NbC phases are given in Table 2. There are two groups of alloying elements; the first group consists of Ni, Cr and Fe. It has a partition coefficient to the primary $\gamma$ ($k_\gamma$) higher than one and prefers to segregate into the dendrites of the primary $\gamma$. The second group has $k_\gamma$ lower than one, which contains Nb, Ti, and Mo. These elements selectively partition to the interdendritic regions. To obtain the partition coefficients for the alloying elements to primary $\gamma$, the specimens

Fig. 1 The shape of Y-block type casting and the dimension of tensile test specimen.

Fig. 2 (a) Macrostructure of the as cast alloy with coarse grain. (b) Macrostructure of the as cast alloy with fine grain.

Fig. 3 EPMA photograph (a) BSE and (b) RE images.
were melted in Ar atmosphere to 100°C above the liquidus temperature and kept for 600 s before cooling to just below liquidus and then hold for 1 h and terminally quenched in water.

By using EPMA, the micro-analysis of primary $\gamma$ and liquid were measured just before quenching. The partition coefficients of alloying elements to $\gamma \left( K_\gamma \right)$ were calculated as a ratio between the element composition in Solid, $y \left( C_s \right)$ and composition of the same element in liquid ($C_l$).

Figure 5 shows an example of EPMA line analysis, for alloying elements distribution in the matrix of as cast coarse grain specimen. Similar distribution behaviors of alloying elements are found in fine grain specimen.

### Table 2: EPMA results for the as cast alloy, coarse grain

<table>
<thead>
<tr>
<th></th>
<th>Nb</th>
<th>Ti</th>
<th>Cr</th>
<th>Fe</th>
<th>Ni</th>
<th>Mo</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Primary $\gamma$</td>
<td>0.15</td>
<td>0.3</td>
<td>1.14</td>
<td>1.30</td>
<td>1.14</td>
<td>0.85</td>
<td>1.05</td>
</tr>
<tr>
<td>Eutectic $\gamma$</td>
<td>2.12</td>
<td>0.64</td>
<td>20.80</td>
<td>20.25</td>
<td>52.09</td>
<td>2.46</td>
<td>0.52</td>
</tr>
<tr>
<td>NbC</td>
<td>6.44</td>
<td>1.40</td>
<td>19.39</td>
<td>16.73</td>
<td>51.29</td>
<td>3.23</td>
<td>0.54</td>
</tr>
<tr>
<td>Ni$_2$Nb</td>
<td>76.46</td>
<td>7.52</td>
<td>1.09</td>
<td>0.85</td>
<td>2.48</td>
<td>ND</td>
<td>0.01</td>
</tr>
<tr>
<td>Ni$_2$Nb</td>
<td>0.85</td>
<td>1.50</td>
<td>14.28</td>
<td>12.45</td>
<td>37.32</td>
<td>6.50</td>
<td>0.17</td>
</tr>
</tbody>
</table>

Fig. 4 Influence of heat treatment on the microstructure of fine grain specimen: (a) As cast specimen. (b) Solution heat treated specimen at 1403 K for 0.5 h. (c) Solution heat treated specimen at 1403 K for 4 h.

3.2 Influence of heat-treatment on structure and distribution of elements

Figures 4(b) and (c) demonstrate the changes in the microstructure of fine grain specimen after solution heat treatment at 1403 K for 0.5 and 4 h, respectively. By increasing heating time, the eutectic phases tend to dissolve into the matrix and its volume fraction decreases.

The solution heat treatment conditions has no effect on the volume fraction of NbC, while the volume fraction of Ni$_2$Nb phase ($V_i$) was lowered to 1.26 from 4.99% in the as cast fine grain specimen and to 1.35 from 3.45% in case of as cast coarse grain specimen at 1403 K for 0.5 h.

By increasing the holding time from 0.5 to 4 h, the volume fraction of Ni$_2$Nb is decreased to 0% as shown in Fig. 4(c). Figure 6 shows the effect of solution treatment temperature and time on the $V_i$ of Ni$_2$Nb for coarse and fine grain specimens. As the temperature increases from 1403 to 1440 K the time required vanishing Ni$_2$Nb phase was shortened to 2 h. However at temperature of 1360 K, it takes about 10 h to almost disappear Ni$_2$Nb from the microstructure. On the
other hand, the volume fraction of NbC was almost unchanged by solution heat treatment.\textsuperscript{13}

The distribution of alloying elements after the solution treatment at 1403 k for 4 h is shown in Fig. 7. It can be seen that the segregation of the alloying elements has been eliminated to lower extent, in compare with as cast conditions, and Ni\textsubscript{2}Nb phase has been vanished.

### 3.3 Solution Kinetics of eutectic Ni\textsubscript{2}Nb

To study the solution kinetics of non-equilibrium Ni\textsubscript{2}Nb phase, Singh and Flemings model has been used.\textsuperscript{14} In this model dendrite arms are assumed platelike and solute distribution within them sinusoidal. The diffusion equation is described as follows:\textsuperscript{14}

\[
g + a = g_o + a e^{-\left(\pi^2 D_s/4l_o^2\right)} \tag{1}
\]

Where:

- \( g \): Volume fraction of Ni\textsubscript{2}Nb at time (t).
- \( g_o \): Volume fraction of Ni\textsubscript{2}Nb at time (t_0).

And

\[
a = \frac{C_M - C_o}{C_p} \tag{2}
\]

- \( C_p \): Uniform concentration of second phase plates in the interdendritic region.
- \( C_o \): Overall alloy composition.
- \( C_M \): Maximum solute content of the primary phase.

When the heat treatment is at a temperature close to the solvus, \( C_o = C_M \) and eq. (7) simply is:

\[
g/g_o = e^{-\left(\pi^2 D_s/4l_o^2\right)} \tag{3}
\]

As the first approximation we assumed that the change in Ni\textsubscript{2}Nb follows the equation (3) during the solution treatment. The diffusion coefficient of Nb in solid, \( D_s \),\textsuperscript{15} in eq. (3) is \( D_s = 1.04 \times 10^{-6} \exp\left[-\frac{202.5}{R}T\right] \) where \( T \) is the measuring temperature (K) and \( R \) is the gas constant (8.31 J/mol-K). According to Singh and Fleming’s model the value of \( l_o \) in eq. (3) is one half of dendrite arm spacing, therefore \( l_o \) is equal to 29.5 \( \mu \)m, in case of coarse structure specimen. However the calculated line, using \( l_o = 29.5 \mu \)m in eq. (3), for the solution of Ni\textsubscript{2}Nb with time is not coincident, but it is away from the experimental results as shown in Fig. 8. Since the diffusion layer dominating the solution of Ni\textsubscript{2}Nb could be narrower than a half of dendrite arm spacing as expected from Fig. 5, therefore \( l_o \) is lower than 29.5 \( \mu \)m. As a result the calculated solution line of Ni\textsubscript{2}Nb largely shifts to a lower level and fits the experimental data at 1440 and 1403 K as \( l_o \) is assumed to be 10 and 14 \( \mu \)m respectively, as shown in Fig. 8. This could be due to the real solution of Ni\textsubscript{2}Nb might take place in three dimensions, which would reduce the apparent diffusion layer thickness, \( l_o \), in one simple dimensional model.

### 3.4 Influence of solution treatment on mechanical properties

The as cast fine and coarse grain specimens have the same
hardness measurement 233 HV₃₀, while they have tensile strengths of 752 and 607 MPa respectively, as shown in Table 3. The difference in distribution of eutectic phase Ni₂Nb gives a minor influence on the hardness, but a significant effect on tensile strength. These higher values of hardness and tensile strengths for as cast coarse and fine grain specimens could be attributable to the γ” etc precipitated during the slow cooling in the preheated mold. Additionally, the yield stress and ductility is affected by the grain size where both are found to be 477 MPa and 33.8% for fine grain and 352 MPa and 39.4% for coarse grain, as mentioned in Table 3. The tensile strength and yield strength of fine grain specimens are higher than the equivalent coarse grain specimen. The grain size has a significant influence on the tensile and yield stress values; the grain size of the microstructure has an inverse relation with tensile ($\sigma_b$) and yield strength ($\sigma_y$).

Additionally, Fig. 9 demonstrates the stress–strain curves for as cast specimen and the specimens solution treated at 1403 K for 0.5 and 4 h. As the solution treatment time increases both the tensile strength and elongation increase. This increment in strength and ductility could be related to the decrease in $V_f$ of Ni₂Nb, as shown in Fig. 10. The microstructure of the tensile test specimen just before the failure indicates that the crack is initiated at the eutectic Ni₂Nb and then propagated along it, as demonstrated in Fig. 11.

### 4. Conclusions

In the present work, the solution of non-equilibrium Ni₂Nb and homogenization processes have been investigated on fine and coarse grain In718 alloy castings. The main results were as follows:

1. By controlling pouring temperature, both fine and
Coarse grain cast-specimens were prepared. The fine grain microstructure has a volume fraction of Ni$_2$Nb phase of 4.99% higher than that in coarse grain one, 3.45%. The grain size of fine and coarse specimens is 12 ASTM and 7.5 ASTM, respectively.

2) Solution heat treatment has no influence on NbC volume fraction whilst the volume fraction of Ni$_2$Nb is decreased by increasing the heating temperature or time. Ni$_2$Nb phase completely disappears after 2 h at 1440 K and 4 h at 1403 K.

3) Diffusion data is applied in the calculation of solution kinetics of Ni$_2$Nb during solution treatment and it was reasonably when compared with the experimental one.

4) Both as cast fine and coarse grain structures have the same hardness value (233 Hv), while tensile strength ($\sigma_t$) and yield stress ($\sigma_{0.2}$) of fine structure are higher than those of coarse one.

5) After the solution treatment at 1403 K for 4 h, $\sigma_t$ and $\sigma_{0.2}$ were 694 and 312 MPa for the fine grain, which are higher than those of coarse grain 524 and 252 MPa. Thus the fine grain gives higher strength.

6) Crack initiates and propagates along Ni$_2$Nb, thus the less the Ni$_2$Nb volume fraction, the higher the elongation and $\sigma_B$.

REFERENCES


