EFFECT OF SOLUTION TREATMENT ON THE
STRESS RUPTURE PROPERTY OF MA ODS NI-BASE
SUPERALOY AT 760°C

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Introduction

The strengthening of mechanically alloyed (MA) oxide dispersion strengthened (ODS) Ni-base superalloys is obtained through the precipitation hardening by a Ni₅(Al, Ti), which is known as γ' phase, at the intermediate temperature region of 760—850°C. The γ' precipitates are ordered FCC (L1₂) structure and are coherent with the nickel-rich γ matrix having FCC structure. The size, volume fraction and distribution of the γ' precipitates are sensitively related to the heat treatment for precipitation and these parameters can significantly affect the mechanical properties of ODS Ni-base superalloys[1].

The MA ODS Ni-base superalloys in general are given three stages of heat treatments; (1) zone annealing treatment to develop a stable coarse-grained structure suitable for high stress-rupture properties; (2) solution heat treatment to dissolve the γ' so that it can subsequently be precipitated; (3) aging treatments to precipitate fine γ' carbides and borides at grain boundaries[2-5].

The aim of this study was to investigate the effect of solution heat treatment temperature on the γ' precipitate size and stress rupture property of MA ODS Ni-base superalloy. The deformation mechanism of ODS Ni-base superalloy was discussed by observing the interaction between dislocations and γ' precipitates at 760°C.

Experimental Procedure

An oxide dispersion strengthened Ni-base superalloy, which is designated as Alloy 92, was prepared at INCO Alloys International Inc. The elemental powders and master alloy powders were mechanically alloyed in an attritor ball mill. The mechanically alloyed powders were packed into mild steel cans and the cans were evacuated and sealed. The sealed cans were extruded into bars with 1.8cm in diameter at 1175°C with extrusion ratio of about 17:1. The nominal composition of the ODS Ni-base superalloy (Alloy 92) is listed in Table 1.
TABLE 1
The nominal composition (wt%) of experimental Alloy 92

<table>
<thead>
<tr>
<th></th>
<th>Ni</th>
<th>Cr</th>
<th>Al</th>
<th>W</th>
<th>Ta</th>
<th>Mo</th>
<th>Co</th>
<th>Ti</th>
<th>Zr</th>
<th>B</th>
<th>C</th>
<th>Y_{2}O_{3}</th>
<th>Re</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy 92</td>
<td>Bal.</td>
<td>8</td>
<td>6.5</td>
<td>6</td>
<td>3</td>
<td>1.5</td>
<td>5</td>
<td>1</td>
<td>0.15</td>
<td>0.01</td>
<td>0.05</td>
<td>1.1</td>
<td>3</td>
</tr>
</tbody>
</table>

The extruded bars were zone annealed at hot zone temperature of 1300°C with constant furnace travel speed of 9cm/hr and followed by three-stage heat treatment for the precipitation of γ' phase. The solvus temperature of γ' extended over 1070-1170°C and the solidus line spread over 1330-1390°C in Alloy 92[6]. This superalloy was solution treated at three different conditions. The superalloys were solution treated at two different temperatures of 1232°C and 1280°C for fixed time of 30 min. While, the solution treatment time was varied from 30 min. to 2 hours at the solution treatment temperature of 1280°C. Then followed by two stages aging treatments (950°C/2hrs/air cooling (AC) + 850°C/24hrs/AC) as following:

1. HTC1: 1232°C / 0.5hr / air cooling (AC) + 950°C / 2hrs / AC + 850°C / 24hrs / AC
   (Standard heat treatment applied to MA 6000 )
2. HTC2: 1280°C / 0.5hr / AC + 950°C / 2hrs / AC + 850°C / 24hrs / AC
3. HTC3: 1280°C / 2hrs / AC + 950°C / 2hrs / AC + 850°C / 24hrs / AC

The specimens for stress-rupture test were machined with a gage diameter of 0.45cm and gage lengths of 2.0 cm after three stages heat treatments. The stress-rupture test was conducted at 760°C with the tensile axis parallel to the extrusion direction. The γ' precipitates were observed by the scanning electron microscope (SEM) and transmission electron microscope (TEM). The specimens to observe microstructure by SEM were electropolished in a mixture of 17 ml of H_{2}O, 2ml of HNO_{3} and 1 ml of CH_{3}COOH. The thin foils for TEM were jet polished in a Struers Tenupole twin-jet electropolisher operating at 30V in a 9:1 mixture of ethanol and perchlolic acid between -50°C and -30°C.

Figure 1. This shows the process of zone annealing heat treatment under the influence of a high temperature gradient.

Figure 2. Optical micrograph of Alloy 92 showing coarse elongated microstructure after zone annealing (1300°C, 9cm/hr).
TABLE 2
The chemical composition (wt%) of matrix and gamma prime precipitates of Alloy 92

<table>
<thead>
<tr>
<th></th>
<th>Ni</th>
<th>Al</th>
<th>W</th>
<th>Mo</th>
<th>Ti</th>
<th>Cr</th>
<th>Co</th>
<th>Others</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matrix</td>
<td>66</td>
<td>5.8</td>
<td>6.0</td>
<td>1.8</td>
<td>1.0</td>
<td>7.8</td>
<td>4.9</td>
<td>7.1</td>
</tr>
<tr>
<td>Precipitates (γ')</td>
<td>70</td>
<td>12.2</td>
<td>7.6</td>
<td>0.9</td>
<td>1.6</td>
<td>2.3</td>
<td>2.8</td>
<td>3.0</td>
</tr>
</tbody>
</table>

**Results and Discussion**

To make the ODS Ni-base superalloy (Alloy 92) suitable for use at high temperature, the as-extruded fine-grained microstructure needs to be transformed into an elongated coarse-grained microstructure. The elongated coarse-grained microstructure can be obtained by secondary recrystallization through the gradient annealing. For most MA ODS superalloys, the recrystallization is performed under the influence of a temperature gradient as shown in Fig. 1, which causes the formation of elongated large grains with a high grain aspect ratio (GAR). This process is known as directional recrystallization or zone annealing. Typical microstructure of zone annealed specimen in Fig. 2 showed a coarse elongated microstructure that was developed during the gradient annealing treatment.

The specimens were undergone a furnace cooling after peak temperature zone passed during zone annealing as illustrated in Fig. 1. During the furnace cooling, the gamma prime precipitates were precipitated in gamma matrix with the compositions as analyzed in Table 2. The large irregular γ' precipitates with an average cube size of about 1.8μm were observed as shown in Figure 3. It is reported that the high stress-rupture resistance at 760°C was obtained at the precipitate's size of below 1μm[7-9]. This indicates that solution treatment temperature needs to be high enough to dissolve the pre-existing coarse gamma prime so that it can be reprecipitated into a more finer size. The microstructure of Alloy 92 after three different solution treatments were observed and the morphologies of gamma prime precipitates after gamma prime heat treatments were shown in Fig. 4. The HTCl treatment resulted in a precipitation of cuboidal shape fine gamma

![Figure 3. SEM micrograph showing a coarse γ' precipitate formed during the furnace cooling after zone annealing heat treatment (1300°C, 9cm/hr).](image-url)
Figure 4. SEM micrographs show the variation of γ' particle size with solution heat treatment, (a) 0.5hr / 1232°C [HTC1], (b) 0.5hr / 1280°C [HTC2] and (c) 2hrs / 1280°C [HTC3].

Prime particles of about 0.30μm in cube size in addition to the unresolved coarse residual gamma prime of about 0.9μm precipitated during the furnace cooling after zone annealing (Fig. 4(a)). The HTC2 treatment showed a similar morphology with HTC1 treatment, while the average size was about 0.41μm and the number of unresolved coarse residual gamma prime was much reduced compared to HTC1 (Fig. 4(b)). The HTC3 treatment resulted in a uniform cuboidal γ' precipitates of about 0.53μm without any unresolved coarse gamma prime precipitates (Fig. 4(c)).

In order to compare the high temperature creep resistance, Alloy 92 heat treated with three different conditions were stress-rupture tested at 760°C. The stress-rupture property of Alloy 92 after three different heat treatments are plotted in Fig. 5. Fig. 6 shows the variation of 100hrs rupture-strength of Alloy 92 as a function of the gamma prime particle size. The highest stress rupture strength was obtained at the cuboidal γ' size of about 0.41μm. The typical deformation microstructures at 760°C of Alloy 92 containing γ' precipitates with size in the range of 0.30–0.53μm are shown in Fig. 7. When γ' size was about 0.41μm, the γ' precipitates were sheared by 1/3<112> type dislocations with forming superlattice intrinsic and extrinsic stacking faults as shown in Fig. 7 (c) and (d). This shear deformation mode of gamma prime showed the highest creep strength among the three different heat treatments. When the γ' size was decreased to 0.3μm, the deformation was occurred by an extensive slip through the matrix. The stress-rupture life was decreased as the γ' size decreased from 0.41μm to 0.3μm as shown in Fig. 6. As the precipitate size increased to 0.53μm, the Orowan bowing of dislocation around the gamma prime precipitates was promoted and resulted in a decrease in stress-rupture life as shown in Fig. 7.

Figure 5. The stress-rupture properties of Alloy 92 comparing with the MA 6000 at 760°C.
Conclusions

The effect of solution treatment on the γ' precipitate's size and the stress-rupture property of ODS Ni-base superalloy was investigated at the intermediate temperature of 760°C. When γ' size was about 0.41μm by HTC2 solution treatment condition, the γ' precipitates were sheared by 1/3<112> type dislocations with
forming stacking faults. When the γ' size was decreased to 0.30μm by HTC1 condition, the deformation was occurred by extensive slip through the matrix. As the precipitate's size increased to 0.53μm by HTC3 condition, the Orowan bowing of dislocation around the gamma prime precipitates was promoted. The highest stress-rupture property of Alloy 92 was obtained at cuboidal γ' size of about 0.41μm by solution treatment at 1280°C for 0.5hr. (HTC2). The shear deformation mode at about 0.41μm gamma prime size showed the higher creep strength than extensive matrix slip at 0.3μm γ' size (HTC1) and Orowan bowing at 0.53μm γ' size(HTC3). These showed that the sizes of γ' precipitates were closely related to the heat treatment for solutioning and this parameter could significantly affect the stress-rupture property of ODS Ni-base superalloy at the intermediate temperature.

References