EFFECT OF SOLUTION TREATMENT ON HIGH TEMPERATURE CREEP BEHAVIOR OF INCOLOY 800H

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Abstract- Incoloy alloy 800H is solution strengthened Iron-Nickel-Chromium (Fe-Ni-Cr) superalloy with good strength and excellent resistance to oxidation and carburation for high temperature applications such as nuclear reactors, gas turbines and heat-treating industries. Hence, high temperature creep property becomes an important aspect to be considered during material selection for such applications. The primary objective of this paper is to study the effect of microstructural variation of Incoloy 800H on the high temperature creep properties. The materials were heat treated at temperature 900°C, 1050°C, 1150°C and 1200°C for 3 hours and followed by water quench. The grain sizes varied from 94.28 to 380.95 μm after the solution treatment at various temperatures. High temperature creep tests were performed on the heat-treated samples at temperature 800°C with constant stress of 100 MPa. The creep curves results show primary, secondary and tertiary stages. The 800H alloy shows an increase in steady state creep rate with increase in grain size of the solution treated sample. The samples after creep tests were analyzed using optical microscope, scanning electron microscope (SEM), energy dispersive spectrometer (EDS) and transmission electron microscope (TEM). Recrystallizations of the grains were also observed near the fracture surface and it was also found that the samples failed in ductile mode with cavitation at the grain boundaries.

Keywords- Incoloy 800H, High Temperature Creep, Superalloy.

I. INTRODUCTION

Nickel-based superalloys are widely used for components operating at high temperature. Nickel-based superalloys known as Incoloy 800H are widely used in aircraft, power-generation turbines, rocket engines, nuclear power and chemical processing plants, such as turbine discs for high pressure stages [1,2]. Due to the significant importance of materials used in high-temperature services, Fe-Ni-Cr alloys are widely used to replace stainless steels by modifying and adding some other elements making it to be heat-resistant alloys.

Incoloy 800 is a solid solution strengthened iron-nickel base superalloy which is extensively used in high temperature environment [3]. Austenitic alloy 800H has the same basic composition as Incoloy alloy 800 (Fe–20Cr–32Ni) but modified to control the carbon content (0.05–0.10 w%) and grain size. Incoloy 800H give higher creep-rupture strength due to the grain size [4]. Based on its advantages such as high strength and corrosion resistant at high temperatures, alloy 800H was selected as one of the potential candidate alloys for Generation IV supercritical water-cooled nuclear plant designs and also being considered for nuclear reactor systems [5,6]. The microstructure show fully austenitic matrix containing several types of precipitates which are found in the austenitic matrix and grain boundaries [3]. Since alloys Incoloy 800H are being used and exposed to critical environment, it is important to fully understand the creep damage behaviour for the design and safety evaluation process of the alloy.

Crev failure is a major safety concern for materials working under high temperature. Creep usually happens at temperature roughly higher than 0.4Tm, where Tm is the absolute melting temperature of the alloy [7,8]. At temperature above 0.4Tm, the dislocations are thereby no longer constrain to move only on their slip planes, but process of climb is the principle of this free dislocation movement [9]. The most widely reported creep damage mechanisms that occur in the bulk of specimens or components are cavities growth at grain boundaries interactions [1]. Even a few millimeters of crack induced by creep may also lead to total failure/fracture if the crack is not probed and controlled. Lee et al[10] suggested that increase in steady state creep rate by increasing the grain size is dominated by the role of the barrier to the dislocation loop which is generated by the cross slip and also grain boundaries act as barrier to dislocation movement.

This paper reports the effect of grain size on creep properties of Incoloy alloy 800H at 800°C with 100MPa stress. The effect of temperature on high temperature creep properties of Incoloy 800H based on the steady state creep rate has been investigated and the corresponding microstructures were observed using standard characterisation equipment.

II. MATERIAL AND METHODS

The as-received material known as Incoloy 800H in the form of plate shape with chemical composition (wt. %) shown in Table 1 was used in this research work. The specimens were cut by using EDM wire cut into rectangular form then followed by heat treatment process. The dimension of the specimens was in accordance to ASTM E8. The specimens were subjected to various solution treatment temperatures to produce different range of grain size that is determined by using the mean linear intercept method. Specimens were solution treated at 900 °C, 1050 °C, 1150 °C, 1200 °C for 3 hours, respectively.
followed by water quenching. Table 2 shows the solution treatment conditions and the measured grain size. High temperature creep test machine (MAYES TC 20) was used for the creep test. The load on the specimen was carefully applied to ensure that it was transferred to the specimen axially without bending. Constant load creep tests were carried out at 800°C in air with stress of 100MPa. The temperature was controlled at ±3°C during creep tests. A linear variable differential transformer (LVDT) was used for measuring the strain elongation. The strain elongation versus time result is then used to plot the “S” creep curve. The slope of secondary stage in creep curves was taken as steady state creep rate. In order to perform metallographic examination, the specimens were mechanically ground, polished and etched in etching reagent consists of Glycerol (2): Hydrochloric Acid (3): Nitric Acid (1). Optical microscope, SEM and TEM were used to examine the microstructures and the fracture surface. EDS was also used for compositional analysis.

### Table 1. Composition of the Incoloy 800H.

<table>
<thead>
<tr>
<th>Element</th>
<th>Fe (wt%)</th>
<th>Ni</th>
<th>Cr</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Ti</th>
<th>Cu</th>
<th>S</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>800H</td>
<td>46.6</td>
<td>30.3</td>
<td>20.6</td>
<td>0.08</td>
<td>0.3</td>
<td>0.6</td>
<td>0.49</td>
<td>0.06</td>
<td>0.002</td>
<td>0.49</td>
</tr>
</tbody>
</table>

### Table 2. Solution treatment temperature and grain size after solution treatment

<table>
<thead>
<tr>
<th>Solution Treatment (°C)</th>
<th>Grain Size (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ST 900 °C</td>
<td>94.28 µm</td>
</tr>
<tr>
<td>ST 1050 °C</td>
<td>122.81 µm</td>
</tr>
<tr>
<td>ST 1150 °C</td>
<td>222.22 µm</td>
</tr>
<tr>
<td>ST 1200 °C</td>
<td>380.95 µm</td>
</tr>
</tbody>
</table>

### III. RESULTS AND DISCUSSION

#### 3.1. Effect of different solution treatment on microstructures.

Fig. 1 shows the microstructure and for as-received material before solution treatment with an average grain size of 95.47µm. The as-received microstructure shows mainly austenitic structure (Fe-Ni-Cr) with the presence of precipitates. Based on EDS analysis, the elements identified in the precipitates are Ti, N and C which is shown in Fig. 1 (b). It was found that the cubic shape precipitate is rich in titanium and nitrogen and been identified as titanium nitride or carbonitride. The irregular shape precipitate is rich in titanium and carbon, and been identified as titanium carbide (TiC) [14]. These precipitates formed during the casting process.

Fig. 2 and Fig. 3 show the optical microstructure of the specimens that have undergone solution treatment at 900°C, 1050°C, 1150°C and 1200°C with an average grain size of 94.28µm, 122.81µm, 222.22µm and 380.95µm respectively. The grain size was determined using the mean linear intercept method. Increase in solution treatment temperature result in larger grain size of the austenitic structure.
3.2. Effect of grain size on creep properties

The rupture time was first predicted by referring to Larson-Miller parameter curve to compare between the experimental results and in the literature [11]. Fig. 4 shows the creep curve with primary, secondary and tertiary stages clearly observed. The linear stage is called steady state creep rate. The steady state creep rate of solution treated specimens at 900°C, 1050°C, 1150°C and 1200°C were 0.0000505% strain/s, 0.0005667% strain/s, 0.00022167% strain/s and 0.00027186% strain/s respectively. Fig. 5 shows the graph of the creep rate as a function of grain size. The creep rate increases sharply from solution treated sample at 900°C to 1050°C. For sample solution treated at 1050°C to 1150°C, the creep rate slightly decrease and almost become constant at 1200°C. The increase of steady state creep rate was dominated by the role of the barrier to the dislocation loop, which was generated by cross slip. The steady state creep rate increased with increase of grain size representing lower creep resistance, since grain boundaries acted as barriers of dislocation movement [10]. The highest creep resistance was shown with grain size of 94.28 µm.
Fig. 6 shows the fracture surfaces of the alloy at varying with grain sizes and the materials failed in ductile manner. The fracture surface show ductile dimples and some cavities. Fig. 7 to Fig 10 show the cross section fracture area of the specimens with varying grain sizes after creep test. At the fracture area, recrystallized grains was observed. It shows that the overall fracture mode of the specimens was transgranular, however, intergranular fracture was observed at the recrystallized grains. Small cavities were found at the grain boundaries of the recrystallized grains. During creep deformation, dislocations form in the material causing energy stored in it. Through three main mechanisms: recovery, recrystallization and grain coarsening (subsequent to recrystallization) this energy can be release. Creep is a deformation accompanied only by dynamic recovery that involves the rearrangement of dislocation to lower their stored energy. The dislocation rearrangement take place by annihilation of dislocation line length in the subgrain interior, such as by Frank network coarsening and formation of low-angle subgrain boundaries [12]. In dislocation hardening process, dynamic recovery is not the only (dynamic) restoration mechanism that may occur. Recrystallization process can also occur and also able to reduce the flow stress and “restore” the metal. Dynamic recrystallization or recrystallization during deformation normally take place outside the creep realm and mostly can be observed at relatively high strain-rate. High temperature creep may be considered as hot working process (high temperature and high strain-rate) and involves both dynamic recovery and recrystallization process [13].

Fig.6. SEM micrographs of the fracture area for solution treated specimens after creep test; (a) 900°C, (b) 1050°C, (c) 1150°C and (d) 1200°C.

Fig.7. SEM micrographs of the cross section of the fracture area for solution treated specimen at 900°C after creep test ; (a) magnification x250 (b) magnification x2000 .
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The recrystallized grains and precipitates were observed using TEM. Fig. 11 shows dislocations pile-up at the precipitates but no accumulation of dislocations in the recrystallized grains because the recrystallized grains are new grains and which are free from dislocations due to the recovery and recrystallization processes took place when the alloy were exposed to high temperature. Fig. 12 shows several types of precipitates found in the recrystallized grain. Fig. 13 shows EDS analysis of the square type and the irregular shape precipitates indicating it is Titanium rich and it can be suggested that the precipitates are Ti-based in the form of titanium carbide.
The effect of various grain sizes on creep properties of Incoloy 800H was investigated. The research work can be concluded as follows:

1. Increase the solution treatment temperature result in increase in the grain size of the austenitic structure.
2. The steady state creep rate increase with increase in grain size because grain boundaries act as barriers of dislocation mobility.
3. The fracture surface show ductile dimple fracture mode. Cross section fracture show transgranular fracture but intergranular fracture at the recrystallized grains at the fracture area.

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