HIGH TEMPERATURE CREEP BEHAVIOR OF INCONEL 617 AND HAYNES 230

BY

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THESIS

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ABSTRACT

The high temperature gas-cooled reactor (HTGR) has been selected for the Next Generation Nuclear Plant (NGNP) project. The structural materials of HTGR will serve in the temperature range of 760-950°C and withstand coolant pressure up to 7 MPa for a design life of 60 years. Currently, Ni-based Inconel 617 (Alloy 617) and Haynes 230 (Alloy 230) are considered to be the leading metallic candidate materials for applications in the HTGR due to their favorable mechanical properties at high temperatures.

In the present study, focus is placed on the biaxial, long term, high temperature creep behavior for Alloy 617 and Alloy 230. The creep mechanisms, including dislocation creep and grain boundary sliding, were found for the stress range of 10 to 55 MPa at 850°C, 900°C and 950°C for Alloy 617. In the contrast, only dislocation creep was found for Alloy 230 at 850°C, different from the creep mechanism with both grain boundary sliding and dislocation creep at 900°C and 950°C. Both Alloy 617 and Alloy 230 have similar creep rupture lives at 950°C and 900°C, while Alloy 230 has a longer rupture life than Alloy 617 at 850°C.

The equation \( \varepsilon = A \sigma \cosh^{-1}(1 + \tau t) + P \sigma^n t^m \) was used to simulate the creep curves for both alloys at temperatures of 850°C, 900°C, and 950°C. The simulation results have a reasonable agreement with experimental results at medium stress level, but overestimated the experimental result at low stress level and underestimated it at high stress level. The FEM simulation provides a better simulation result.
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CHAPTER 1-INTRODUCTION

The high temperature gas-cooled reactor (HTGR) is a promising Gen-IV reactor concept, in which the reactor core is graphite-moderated and helium-cooled. Its designed output temperature can reach up to 950°C, at which the energy conversion efficiency can exceed 50%, as opposed to the 33% efficiency of currently used light water reactors (LWRs) [1]. In addition to electricity generation, HTGR can produce hydrogen, a clean and green energy source, by taking advantage of the high outlet temperature through thermal-chemical processes [2]. Fig. 1.1 shows the structure of one of the HTGR designs.

Fig 1.1: Structure of HTGR [1]

Due to the harsh operating environment in the reactor core, the HTGR poses a great challenge for its structure materials. The major damage mechanism for structural materials used in the HTGR is the long-term creep damage due to its high operating temperature (~900°C on average) and high gas pressure (~7MPa) in the designed 60-year lifetime [3]. Alloy 617 and Alloy 230 are considered two of the most promising structural materials. Both of these alloys are nickel-based,
solid-solution strengthened materials that exhibit good strength at high temperatures, with acceptable corrosion resistance. Alloy 617 was first introduced in the early 1970’s by Special Metals [4] to achieve better high temperature performance than the previously developed Ni-Co-Cr alloys. Because of its desirable high temperature resistance, a large amount of data on Alloy 617 has been generated for a variety of applications such as gas turbines, chemical manufacturing, and power generation structures during the late 1970’s and early 1980’s [5-7]. Previous data shows that after long-time exposure at high temperature, the alloy remains tough enough since grain boundary carbides can significantly mitigate grain boundary sliding and impart resistance to creep [8]. Compared to Alloy 617, Alloy 230 is a relatively new solid-solution strengthened Ni-Cr-W alloy introduced in the early 1980’s as a competitor of Alloy 617 [9]. The addition of tungsten can produce a relatively low stacking fault energy, and the formation of tungsten-rich M₆C carbide can pin grain boundaries [10]. Alloy 230 displays even better oxidation corrosion (but not carburizing corrosion) and creep deformation resistance at elevated temperature, which makes it a candidate structural material for the HTGR. However, compared to Alloy 617, the amount of experimental data on its mechanical performance is limited. Table 1.1 lists the limiting chemical compositions of both alloys.

Table 1.1 Chemical compositions (Weight %) [11, 12]

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Ni</th>
<th>Cr</th>
<th>Co</th>
<th>Mo</th>
<th>Al</th>
<th>C</th>
<th>Fe</th>
<th>Mn</th>
</tr>
</thead>
<tbody>
<tr>
<td>617</td>
<td>44.5</td>
<td>20-24</td>
<td>10-15</td>
<td>8-10</td>
<td>.8-.1.5</td>
<td>.05-.15</td>
<td>3 max</td>
<td>1 max</td>
</tr>
<tr>
<td>230</td>
<td>57</td>
<td>20-24</td>
<td>5 max</td>
<td>1-3</td>
<td>.2-.5</td>
<td>.05-.15</td>
<td>3 max</td>
<td>.3-1</td>
</tr>
<tr>
<td></td>
<td>Si</td>
<td>S</td>
<td>Ti</td>
<td>Cu</td>
<td>B</td>
<td>P</td>
<td>La</td>
<td>W</td>
</tr>
<tr>
<td>617</td>
<td>1 max</td>
<td>.015</td>
<td>.6 max</td>
<td>.5 max</td>
<td>.006</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>230</td>
<td>.25-.75</td>
<td>.015</td>
<td>.1 max</td>
<td>.5 max</td>
<td>.015</td>
<td>.03 max</td>
<td>.05 max</td>
<td>13-15</td>
</tr>
</tbody>
</table>
CHAPTER 2-LITERATURE REVIEW

2.1 Creep Phenomenology

When a material is subjected to constant applied stress at elevated temperature, continuous deformation can occur. This time dependent deformation process is known as creep. Creep is usually defined as plastic deformation at relatively high temperature (greater than one-third to one-half of melting point on the absolute temperature scale) under constant load over a long time. Creep can significantly affect the property of metals and limit the applied stress they can withstand in engineering applications. Fig. 2.1 shows a typical creep curve.

![Typical creep curve](image)

As indicated in Fig. 2.1, a typical creep process has three regions, which are divided by its slope. The slope, also called creep rate, is generally accepted as the result of competition between strain hardening rate and recovery softening rate [13]. In the primary region, the slope of curve decreases with time and eventually approaches a nearly constant value, which
signifies that the strain hardening rate dominates this stage, but the recovery softening rate becomes more and more pronounced with time. In the secondary region, the creep rate keeps nearly constant, which indicates a balance between the strain hardening rate and the recovery softening rate. The average value of the creep rate during secondary creep is called the minimum creep rate. Since almost all of the creep deformation occurs in the secondary region, engineers are more interested in the creep characteristics in this stage. Typically, the creep rate in this stage, $\dot{\varepsilon}_s$, can be expressed by using a power law on the stress $\sigma$ and exponentially on the temperature $T$:

$$\dot{\varepsilon}_s = A \left(\frac{\sigma}{E}\right)^n \exp\left(-\frac{Q}{RT}\right),$$

(2.1)

where $A$ is a constant, $n$ is the stress exponent, $\sigma$ is external stress, $E$ is elastic modulus of the material, $R$ is the gas constant, and $Q$ is the activation energy characterizing the creep process. The stress exponent $n$ provides important information about creep mechanisms; the relationships between them are shown in table 2.1[14], where $Q$ represents different types of activation energies and $r$ is the inverse grain exponent. In the third region, the recovery softening rate becomes dominant and the creep rate rapidly accelerates to a relatively high level until the specimen fails.
2.2 Creep Mechanisms

Generally, the creep phenomenon can be divided into four types of mechanisms. The first is called dislocation glide, in which dislocations move along slip planes and overcome obstacles such as precipitates, solute atoms, and other dislocations by thermal activation. This mechanism occurs at a relatively high stress level, which satisfies the condition $\frac{\sigma}{G} > 10^{-2}$, where $G$ is shear modulus.

The second mechanism is called dislocation creep. Dislocation creep occurs by dislocation glide aided by vacancy diffusion. Usually, it occurs at the stress level $10^{-4} < \frac{\sigma}{G} < 10^{-2}$. The Gittus model [15] given to calculate the steady-state creep rate $\dot{\varepsilon}_s$ for this mechanism is based on the idea of balance between the strain hardening rate and the recovery softening rate, which agrees well with experiments.
where \( c_j \) is the concentration of jogs, \( D_v \) is the bulk or lattice self-diffusion coefficient, \( b \) is the Burgers vector of dislocation, \( k \) the is Boltzmann’s constant, and \( T \) the is temperature on the absolute scale.

The third mechanism is termed diffusion creep, and involves the flow of vacancies and interstitials through a crystal with applied stress. Diffusion creep is the controlling mechanism at high temperatures and relatively low stresses (usually \( \frac{\sigma}{G} < 10^{-4} \)). Nabarro and Herring developed a creep equation [16, 17] based on this stress-directed atomic diffusion to describe its secondary creep rate:

\[
\dot{\varepsilon}_s = \frac{14 \sigma b^3 D_v}{kT d^2},
\]

where \( d \) is the grain diameter and \( D_v \) is the lattice diffusion coefficient.

The last creep mechanism is grain boundary sliding, which is a shear process that occurs in the direction of grain boundaries. Increasing temperature and decreasing strain rate both promote the grain boundary sliding. Grain boundary sliding is not an independent process at high temperature. It is usually accompanied with diffusional creep. Grain boundary sliding does not contribute to steady state creep, but is important in initiating intergranular fractures.

Deformation mechanism map is a practical way to illustrate the various creep mechanisms for a particular metal. A typical deformation map developed by Ashby for a 0.1 mm grain size nickel is shown in Fig 2.2 [18].
The deformation maps are usually presented as normalized shear stress versus homologous temperature with lines of constant strain rate drawn within the particular fields of dominance. The various regions of the map indicate dominant deformation mechanisms for a specific condition of stress and temperature. The boundaries of these regions represent a condition of stress and temperature where the two deformation mechanisms are equally important. Although the deformation maps for many alloys have been developed [19], deformation maps for Alloy 617 and Alloy 230 have not been developed.

2.3 Creep Rupture

The creep deformation always terminates in fracture after a sufficiently long-term loading. It is believed that the grain boundary voids and cracks that form in the third stage cause the final rupture of the material. Creep rupture is always intergranular due to voids and micro cracks
which form at the boundaries [20]. Two types of intergranular cracks often occur in creep. At high stress condition, wedge-shaped cracks (w-type cracks) at triple points of grain are often observed due to grain boundary sliding, as shown in Fig. 2.3 (a). At low stress, the round or elliptical cavities (r-type cracks) that form in the grain boundaries aligned normal to the tensile stress are generally considered to be the main fracture mechanism, and are shown in Fig. 2.3 (b). Further studies on their growth mechanism have shown that they grow by diffusion when they are of small size and by power-law creep as they become larger. As the strain rate is decreased or the temperature is raised the r-type cracks is favored over the w-type cracks [21].

![Fig. 2.3: Schematic illustration of creep fractures: (a) w-type cracks (b) r-type cracks](image)

2.4 Creep Behavior of Alloy 617 and Alloy 230

It has been shown [22] that the creep curve of alloy 617 follows a classical behavior at 850°C; however, no recognizable secondary creep regime was observed at 950°C. Recently, Roy et al. [23] found that with increasing applied stress levels and temperature, the steady-state region
became shorter and finally disappeared at 950°C, showing only a steeper tertiary creep curve. However, in a study by Chomette [24], non-classical creep behavior at 850 and 950°C were observed, with a strain rate drop at the beginning of the tests followed by a creep rate increase to a plateau before the onset of the tertiary creep. Microstructure examination of the crept alloy 617 showed that the intragranular carbides are effective in lowering creep rate, and the unstable intragranular carbides redissolve in favor of the more stable grain boundary carbides at 1000°C [25]. More recently, it has been found that in a creep-deformation environment, carbides observed in alloy 617 may dissolve and reprecipitate on boundaries in tension [25, 26]. It was believed that the difference in the stability of the carbides in compressive vs tensile environments is the driving force for the distribution [25]. Tung [27] did some creep experiments on both Alloy 617 and Alloy 230 at 850°C and 950°C and found that Tertiary creep takes a great part of both materials’ creep life. With increasing applied stress, the fraction of time for the secondary creep regime decrease. At 950°C, Alloy 617 and Alloy 230 had similar creep rupture life, whereas Alloy 230 exhibited a better creep rupture life than Alloy 617 at 850°C. However, besides Tung’s work, very limited amount of data for Alloy 230 were found and therefore, deformation mechanism is not fully understood up to now.
CHAPTER 3-EXPERIMENTAL APPROACH

The Alloy 617 and Alloy 230 creep tubes used in this experiment were fabricated by Century Tubes Inc and their composition is given in Table 3.1. The specimens used in this study are compact, with dimensions as shown in Fig. 3.1. The two ends of the specimens are sealed with two caps by using electron beam welding technology. A small hole was drilled in one cap for inner gas pressurizing.

Table 3.1 Chemical compositions of Alloy 617 and Alloy 230 for the specimens (Weight %)

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Al</th>
<th>B</th>
<th>C</th>
<th>Co</th>
<th>Cr</th>
<th>Cu</th>
<th>Fe</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>617</td>
<td>1.04</td>
<td>0.02</td>
<td>0.08</td>
<td>12.46</td>
<td>22.21</td>
<td>0.14</td>
<td>1.55</td>
<td>-</td>
</tr>
<tr>
<td>230</td>
<td>0.37</td>
<td>0.03</td>
<td>0.11</td>
<td>0.19</td>
<td>22.39</td>
<td>-</td>
<td>0.94</td>
<td>0.008</td>
</tr>
<tr>
<td></td>
<td>Mn</td>
<td>Mo</td>
<td>Ni</td>
<td>S</td>
<td>Si</td>
<td>Ti</td>
<td>W</td>
<td>La</td>
</tr>
<tr>
<td>617</td>
<td>0.07</td>
<td>8.97</td>
<td>53.02</td>
<td>0.01</td>
<td>0.17</td>
<td>0.29</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>230</td>
<td>0.5</td>
<td>1.36</td>
<td>BAL</td>
<td>0.02</td>
<td>0.38</td>
<td>0.01</td>
<td>14.26</td>
<td>-</td>
</tr>
</tbody>
</table>
This study was conducted at the temperature of 900°C, and the experiment conditions are shown in tables 3.2 and 3.3. The middle wall effective stress $\sigma_{\text{eff}}$ in the table is calculated by the following equation, which is based on von Mises criterion [28]:

$$\sigma_h = \frac{p R_i^2}{R_0^2 - R_i^2} \left(1 + \frac{R_o^2}{R_m^2} \right),$$  

(3.1)

$$\sigma_{\text{eff}} = \frac{\sqrt{3} R_0^2}{R_0^2 + R_m^2} \sigma_h,$$

(3.2)

where $R_i$ is the inner radius, $R_0$ is the outside radius, $R_m$ is the mid-wall radius, $p$ is the internal gas pressure and $\sigma_h$ is the mid-wall hoop stress.

Before pressurizing the specimen, the specimen was pumped down by a mechanical pump to prevent inner wall oxidization. Then, the specimen was placed in the center of the heated furnace. The tubing was then pressurized with high purity argon and maintained a constant pressure monitored by a pressure gauge. The schematic diagram of the system is shown in Fig. 3.2.
**Table 3.2: Experimental conditions of Alloy 617**

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Inner Pressure (MPa)</th>
<th>Effective Stress $\sigma_{eff}$ (MPa)</th>
<th>Rupture Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>900</td>
<td>4.10, 2.46, 2.05</td>
<td>30, 18, 15</td>
<td>up to 1000 hr</td>
</tr>
</tbody>
</table>

**Table 3.3: Experimental conditions of Alloy 230**

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Inner Pressure (MPa)</th>
<th>Effective Stress $\sigma_{eff}$ (MPa)</th>
<th>Rupture Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>900</td>
<td>4.79, 4.10, 3.15, 2.46</td>
<td>35, 30, 23, 18</td>
<td>up to 1000 hr</td>
</tr>
</tbody>
</table>

After a period of time, the specimen was taken out of the furnace in order to measure the diameter change until rupture. The inner argon gas was released after the specimen was taken out of the furnace. After the specimen was fully cooled down to room temperature, the diameter change of the specimen was measured by laser scanner with the resolution of 0.5 μm. The diameter strain is calculated by the following equation:

$$
\varepsilon_{dia} = \frac{\Delta D}{D_0},
$$

(3.3)

where $D_0$ is the original diameter, $\Delta D$ is the diameter change, and $\varepsilon_{dia}$ is the diameter strain.
4.1 Biaxial Creep Behavior of Alloy 617

The biaxial creep behavior of Alloy 617 at 900°C is plotted in Fig. 4.1. With increasing applied stress, the rupture time decreases. From the plot, it can be seen that the creep strain development for the stress of 18 MPa is close to the one for 15 MPa. In addition, all the curves exhibit a large tertiary part of creep deformation, while the secondary regime is not distinct.

Fig. 4.1: Diameter strain vs. creep exposure time for Alloy 617 at 900°C
To illustrate the change in creep rate during the test, the creep strain-time curves were differentiated to obtain the creep strain rate using finite difference formula:

\[
\dot{\varepsilon}(\bar{t}_n) = \frac{\varepsilon_{n+1} - \varepsilon_n}{\bar{t}_{n+1} - \bar{t}_n}, \tag{4.1}
\]

\[
\bar{t}_n = \frac{t_{n+1} + t_n}{2}, \tag{4.2}
\]

where \( \varepsilon_{n+1} \) and \( \varepsilon_n \) represent the effective strain at the two consecutive exposure times \( t_{n+1} \) and \( t_n \), respectively; \( \dot{\varepsilon}(\bar{t}_n) \) is the creep strain rate and \( \bar{t}_n \) is the mean time of the two time intervals. The creep strain rate is plotted as a function of the average strain on a semi-log scale as shown in Fig. 4.2

Fig. 4.2: Creep strain rate vs. average creep time for Alloy 617 at 900°C
It should be noted that with increasing of applied stress, the steady state creep rate portion decreases. Fig. 4.3 (a)-(c) shows the cross-sectional view of SEM images near the fracture surface. Intergranular fracture was observed for all specimens.

Fig. 4.3 (a): Cross-sectional view of pressurized creep tubes near fracture surface under the applied stress of 30 MPa at 900°C for Alloy 617
Fig. 4.3 (b): Cross-sectional view of pressurized creep tubes near fracture surface under the applied stress of 18 MPa at 900°C for Alloy 617
Fig. 4.3 (c): Cross-sectional view of pressurized creep tubes near fracture surface under the applied stress of 15 MPa at 900°C for Alloy 617
4.2 Biaxial Creep Behavior of Alloy 230

The biaxial creep behavior of Alloy 230 at 900°C is plotted in Fig. 4.4. With increasing applied stress, the rupture time decreases. As shown in the plot, all the Alloy 230 creep curves exhibit a large tertiary portion of creep deformation. Even at the lowest stress, the secondary regime is not distinct. The creep strain rate is calculated using equations 4.1 and 4.2, and is plotted in Fig. 4.5 on a semi-log scale. It should be noted that with increase of applied stress, the steady state creep rate portion decreases. Fig. 4.6 (a)-(d) shows the cross-sectional view of SEM images near the fracture surface. Intergranular fracture was observed for all specimens.

![Diagram](image)

Fig. 4.4: Diameter strain vs. creep exposure time for Alloy 230 at 900°C
Fig. 4.5: Creep strain rate vs. average creep time for Alloy 230 at 900°C
Fig. 4.6 (a): Cross-sectional view of pressurized creep tubes near fracture surface under the applied stress of 35 MPa at 900°C for Alloy 230
Fig. 4.6 (b): Cross-sectional view of pressurized creep tubes near fracture surface under the applied stress of 30 MPa at 900°C for Alloy 230
Fig. 4.6 (c): Cross-sectional view of pressurized creep tubes near fracture surface under the applied stress of 23 MPa at 900°C for Alloy 230
Fig. 4.6 (d): Cross-sectional view of pressurized creep tubes near fracture surface under the applied stress of 18 MPa at 900°C for Alloy 230
CHAPTER 5-CREEP SIMULATION

5.1 Simulation Equation Development

The experimental results are central to understand material properties at certain temperatures and stresses. However, these experimental conditions are limited and could not directly provide long-term creep life estimations. To understand the creep properties of both Alloy 617 and Alloy 230 comprehensively, a computational method to extend the existing experimental results to a larger range of temperatures and stresses is required. The experimental data from Tung [27] of Alloy 617 and Alloy 230 at 950°C and 850°C and data for both alloys at 900°C mentioned in chapter 4 were used for this simulation.

To develop a simulation equation, we begin with a description of the time-dependent relationship between stress and strain. Take the experimental data of Alloy 617 at 950°C and at a creep time of 30 hours for example (Fig. 5.1), we can describe its behavior as the sum of two strain components: First one describes a linear stress dependence at low stress levels, and second one describes a stronger stress dependence at higher stress levels.
The linear portion can be approximated by the relation $\varepsilon = C(t)\sigma$, where $t$ represents time. This fitting line is used to evaluate the slope $C(t)$ and is allowed to intercept the strain axis at nonzero values. The interception value is considered to be a measurement uncertainty. The portion that has a stronger dependence on stress can be represented by $\varepsilon = K(t)\sigma^n$. In this relation, $n$ is the stress exponent, which indicates the creep mechanism and can be procured from equation 2.1. Then, the total stress-strain relationship can be represented by:

$$\varepsilon = C(t)\sigma + K(t)\sigma^n$$

(5.1)

From the previous work by Tung [27], the value of $n$ for Alloy 617 at 950°C is 3.3. The values of $C(t)$ and $K(t)$ are determined from each set of strain-stress data by curve fitting. Table 5.1 provides the $C(t)$ and $K(t)$ for Alloy 617 at 950°C for different periods of time.
Table 5.1: Curve-fitting values of $C(t)$ and $K(t)$ for Alloy 617 at 950°C

<table>
<thead>
<tr>
<th>Time (h)</th>
<th>$C(t)$</th>
<th>$K(t)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>1.316e-7</td>
<td>1.81e-5</td>
</tr>
<tr>
<td>30</td>
<td>2.479e-8</td>
<td>8.333e-5</td>
</tr>
<tr>
<td>100</td>
<td>1.059e-9</td>
<td>2.524e-4</td>
</tr>
<tr>
<td>160</td>
<td>4.933e-10</td>
<td>5.525e-4</td>
</tr>
<tr>
<td>256</td>
<td>1.127e-10</td>
<td>7.648e-4</td>
</tr>
</tbody>
</table>

As suggested by Swindeman and Pugh [29], $C(t)$ and $K(t)$ can be represented as:

$$C(t) = A \cosh^{-1}(1 + rt)$$

$$K(t) = Pt^m$$

$A$, $r$, $P$ and $m$ are temperature dependent constants obtained from curve fitting. Finally, the experimental creep data can be represented as:

$$\varepsilon = A\sigma \cosh^{-1}(1 + rt) + P\sigma^n t^m$$

5.2 Simulation Result of Alloy 617

Figs. 5.2 and 5.3 show the fitting curve of $C(t)$ and $K(t)$ for Alloy 617 at 950°C. When fitting the $K(t)$, the natural log was taken on both sides of equation 5.3.
Fig. 5.2: Experimental results and numerical description of $C(t)$

Fig. 5.3: Experimental results and numerical description of $K(t)$
From the curve fitting shown above, the values of the constants $A$, $r$, $P$ and $m$ for Alloy 617 at 950°C are obtained. With the same procedure, the corresponding constants’ values for 900°C and 850°C are obtained. Table 5.2 lists these values at different temperatures for Alloy 617.

Table 5.2: Values of equation constants of Eq. 5.4 for Alloy 617

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>$A$</th>
<th>$r$</th>
<th>$P$</th>
<th>$m$</th>
<th>$n$</th>
</tr>
</thead>
<tbody>
<tr>
<td>850</td>
<td>0.008778</td>
<td>0.03115</td>
<td>9.261e-10</td>
<td>1.401</td>
<td>4.1</td>
</tr>
<tr>
<td>900</td>
<td>0.01239</td>
<td>0.02198</td>
<td>1.3219e-8</td>
<td>1.413</td>
<td>3.714</td>
</tr>
<tr>
<td>950</td>
<td>4.0897e-7</td>
<td>0.06541</td>
<td>1.317e-6</td>
<td>1.161</td>
<td>3.3</td>
</tr>
</tbody>
</table>

The simulation results for Alloy 617 are plotted in Fig. 5.4 (a)-(c). The red stars represent the experiment results.

Fig. 5.4 (a): Results of creep experiments and simulations for Alloy 617 at 950°C
Fig. 5.4 (b): Results of creep experiments and simulations for Alloy 617 at 900°C

Fig. 5.4 (c): Results of creep experiments and simulations for Alloy 617 at 850°C
As illustrated in the plots of Fig. 5.4 (a)-(c), the simulation in most of the cases has a reasonable agreement with the experimental results at the medium stress level. However, the simulation overestimates the creep development at low stress levels, and underestimates the creep development at high stress levels. The deviation primarily attributes to the parameter curve fitting that requires more long-term experimental data.

5.3 Simulation Result of Alloy 230

Using the same method that was used to attain $C(t)$ and $K(t)$ in section 5.2, the constants $A, r, P,$ and $m$ for Alloy 230 were obtained and listed in Table 5.3.

<table>
<thead>
<tr>
<th>Temperature ($^\circ$C)</th>
<th>$A$</th>
<th>$r$</th>
<th>$P$</th>
<th>$m$</th>
<th>$n$</th>
</tr>
</thead>
<tbody>
<tr>
<td>850</td>
<td>0.008027</td>
<td>0.009588</td>
<td>8.41e-23</td>
<td>1.835</td>
<td>11.1</td>
</tr>
<tr>
<td>900</td>
<td>0.006461</td>
<td>0.007775</td>
<td>3.177e-10</td>
<td>1.362</td>
<td>4.666</td>
</tr>
<tr>
<td>950</td>
<td>0.00867</td>
<td>0.04153</td>
<td>9.54e-6</td>
<td>1.152</td>
<td>2.0</td>
</tr>
</tbody>
</table>

Fig. 5.5 (a)-(c) shows the simulation results of Alloy 230, where the red stars represent the experimental results. As illustrated in the plot, the simulation results at high temperatures are more accurate compared to the ones at lower temperatures, and also are more accurate at high stress compared to the ones at lower stress. At temperatures of 900$^\circ$C and 950$^\circ$C, the tertiary portion is often underestimated.
Fig. 5.5 (a): Results of creep experiments and simulations for Alloy 230 at 950°C

Fig. 5.5 (b): Results of creep experiments and simulations for Alloy 230 at 900°C
5.4 Finite Element Analysis for Creep Behavior

A more accurate manner to simulate the creep behavior is to apply finite element method (FEM). FEM is a numerical technique for finding approximate solutions to boundary value problems. The commercial software, ABAQUS, was employed for the FEM analysis. ABAQUS/CAE provides numerical functions for both modeling and analysis of mechanical components and assemblies and allows visualization of the finite element analysis result. Creep, a nonlinear huge deformation problem, is especially suitable for simulation by ABAQUS. The ABAQUS model of the creep tube before deformation is shown in Fig. 5.6.
ABAQUS provides three basic models to describe creep behavior: the time-hardening, strain-hardening, and hyperbolic-sine models. However, all these models cannot precisely describe the three-stage creep development, so the implementation of user subroutines is needed to develop better simulation accuracy. The user subroutines in ABAQUS allow the program to be customized for particular applications and projects, and greatly extend the applicability of ABAQUS. With slight modifications to the constants’ values, equation 5.4 is used in the creep subroutine as the constitutive function. The dimension of pressurized tube defined in ABAQUS is of the same size as the one used in the experiment. Since the dimension of a sample near two end caps barely change in our experiment, the two ends are assumed to be fixed in the model as a boundary condition. The subroutine program is attached in the appendix. Fig. 5.7 shows the
pressurized creep tube deformed for 32MPa at 950°C after FEM modeling. By using the tool of XY-Data, we can pick a node in the center region of the deformed model and plot its moving magnitude, which can then be compared with our experimental results. These results of the comparison are shown in Fig. 5.8 (a)-(f), where the red line represents the simulation data.

Fig. 5.7: FEM modeling for creep strain development of a pressurized creep tube
Fig. 5.8 (a): FEM simulation results for Alloy 617 at 950°C
Fig. 5.8 (b): FEM simulation results for Alloy 617 at 900°C
Fig. 5.8 (c): FEM simulation results for Alloy 617 at 850°C
Fig. 5.8 (d): FEM simulation results for Alloy 230 at 950°C
Fig. 5.8 (e): FEM simulation results for Alloy 230 at 900°C
Fig. 5.8 (f): FEM simulation results for Alloy 230 at 850°C
CHAPTER 6-DISCUSSION

6.1 Creep Mechanism Analysis

The creep deformation of Alloy 617 and Alloy 230 shown in Figs. 4.1 and 4.4 indicates that tertiary creep regime constitutes the major portion of the creep life for all samples tested at 900°C, which is in accord with Tung’s observations at 850°C and 950°C [27]. With increasing applied stress, the time duration of the secondary regime decreases. For example, in Fig. 4.5 (a)-(d) of Alloy 230, the time duration of the secondary regime decreases from a few hundred hours to a few hours with increasing applied stress. Similarly, by comparing the same stress at different temperatures, it can be seen that the time duration of secondary regime decreases with increasing temperature.

The stress exponent calculated by equation 2.1 for both Alloy 617 and Alloy 230 at 900°C are shown in Figs. 6.1 and 6.2, respectively,

Fig. 6.1: Stress exponent analysis for Alloy 617 at 900°C
Fig. 6.2: Stress exponent analysis for Alloy 230 at 900°C

For Alloy 617 at 900°C, the stress exponent is measured to be 3.407, indicating that a viscous glide mechanism is the rate controlling step [30]. From the previous work by Tung, the stress exponent \( n \) is measured to be 4.1 at 850°C and 3.3 at 950°C, so there is no significant change in stress exponent for these two temperatures. The stress exponent analysis results show that at all three temperatures, Alloy 617 maintains similar creep mechanisms, which can be defined to be a combination of a viscous glide mechanism and a dislocation diffusion mechanism. Although viscous glide mechanism dominates all the three temperatures, the dislocation diffusion becomes more significant as decreasing studied temperature.

For Alloy 230 at 900°C, the stress exponent is measured to be 4.666, indicating the climb-controlled dislocation creep [31]. The stress exponent \( n \) is measured to be 11.1 for 850°C and 2 for 950°C by Tung, which indicates a significant change in the creep mechanism between the two temperatures. It was believed that the high stress exponent is attributed to the disruption of
dislocation motion by carbides precipitated within the material. The stress exponent dramatically decrease from 850°C to 950°C is because that as the temperature rises, fewer carbides are precipitated, so that the interaction of dislocation with carbides is weakened, and consequently the stress exponent decreased. With increasing temperature, the grain boundary sliding mechanism has a greater impact on the creep life, which further decreases the value of the stress exponent. Previous studies by Tung’s [27] showed that grain boundary sliding was not the dominant creep mechanism at 950°C. The most likely creep mechanism is the combination of dislocation creep and grain boundary sliding. The current study confirmed that the combined creep mechanism is also dominant at 900°C, while the change of creep mechanism occurs at 850°C where dislocation creep becomes most significant.

From SEM images of Figs. 4.3 and 4.6, many intergranular creep fractures are observed. These intergranular creep fractures originate at triple junctions of grain boundaries and propagate along grain boundaries perpendicular to the hoop stress along the cross section. Additionally, many intergranular voids and cavities can also be found at grain boundaries, which suggest that nucleation, growth, and coalescence of intergranular creep voids and cavities are micro-mechanisms leading to creep rupture. Voids and micro cracks always form at grain boundaries mainly due to impurity segregation to the boundaries and the interaction of grain boundary sliding with geometrical constraints. With increasing temperature and stress, it becomes much easier for the voids to form and grow. This trend will further decrease the connection parts of grain boundary perpendicular to the hoop stress, and can cause further decrease of steady state creep stage time, which is exactly in accord with the experimental results.

The creep life vs. applied stress for both alloys at all three temperatures is shown in Fig. 6.3. Observation of Fig. 6.3 leads to the conclusion that the creep life for Alloy 617 and Alloy 230 is
similar at 950\(^\circ\)C and 900\(^\circ\)C, whereas Alloy 230 has better creep properties than Alloy 617 at 850\(^\circ\)C. From Fig. 6.3, similar slopes for Alloy 617 at all three temperatures further justify the assumption that a similar creep mechanism is shared, while the distinct slope for Alloy 230 at 850\(^\circ\)C indicates a different creep mechanism from the other two studied temperatures.

![Graph showing creep life of Alloy 617 and Alloy 230 at 850\(^\circ\)C, 900\(^\circ\)C, and 950\(^\circ\)C](image)

Fig. 6.3: Creep life of Alloy 617 and Alloy 230 at 850\(^\circ\)C, 900\(^\circ\)C, and 950\(^\circ\)C
6.2 Evaluation of the Empirical Relation of Predicting Long-term Creep Life from Short-term Testing

The Monkman-Grant relation [32] provides a correlation between the steady-state creep rate and creep rupture time, and is described as:

\[ t_r \dot{\varepsilon}_{\text{min}}^{\alpha} = C_0 , \] (6.1)

where \( \alpha \) and \( C_0 \) are material constants, independent of stress and temperature. Figs. 6.4 and 6.5 show creep life as a function of minimum creep rate for Alloy 617 and Alloy 230.

Fig. 6.4: Relationship between minimum creep rate and rupture time for Alloy 617
Fig. 6.5: Relationship between minimum creep rate and rupture time for Alloy 230

From the plots of Figs. 6.4 and 6.5, we can see that the Monkman-Grant relation provides an acceptable method for predicting the long-term creep life of Alloy 617 at the studied temperatures. However, for Alloy 230, the material constants ($\alpha$ and $C_0$) seem to be temperature dependent. Temperatures 900°C and 950°C share one group of constants, while temperature 850°C uses another group. This phenomenon may be evidence in support of the hypothesis that the creep mechanism for Alloy 230 is similar at 900°C and 950°C, but is different at 850°C. Tables 6.1 and 6.2 list the values of $\alpha$ and $C_0$. 
Table 6.1: Constant values in the Monkman-Grant Equation of Alloy 617

<table>
<thead>
<tr>
<th>α</th>
<th>C₀</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.91957</td>
<td>0.00258</td>
</tr>
</tbody>
</table>

Table 6.2: Constant values in the Monkman-Grant Equation of Alloy 230

<table>
<thead>
<tr>
<th>Temperature (℃)</th>
<th>α</th>
<th>C₀</th>
</tr>
</thead>
<tbody>
<tr>
<td>900 &amp; 950</td>
<td>1.5731</td>
<td>7.85e-8</td>
</tr>
<tr>
<td>850</td>
<td>0.6887</td>
<td>0.2575</td>
</tr>
</tbody>
</table>
CHAPTER 7-CONCLUSIONS

Biaxial thermal creep deformation studies using pressurized creep tube technology were carried out at 900°C. Based on the results of the experiments, creep modeling is conducted to predict creep behavior at different temperatures and stresses. Creep mechanisms were analyzed at 900°C with comparison to the results at 950°C and 850°C from previous studies by Tung [27]. The conclusions are as follows:

1. The three regimes of creep curves of both Alloy 617 and Alloy 230 are not distinct at 900°C. The tertiary regime constitutes the major portion of the creep lives. With increasing temperature and stress, the scope of the secondary regime decreases.

2. The equation $\varepsilon = A \sigma \cosh^{-1}(1 + rt) + P \sigma^n t^m$ was used to simulate creep strain development for both alloys at temperatures of 850°C, 900°C, and 950°C. The simulation results have a reasonable agreement with experimental results at medium stress level, but were overestimated at low stress level and underestimated at high stress level. The FEM simulation provides a better simulation result.

3. Similar creep mechanisms were found at 850°C, 900°C and 950°C for Alloy 617. In the contrast, the creep mechanism for Alloy 230 at 850°C is different from the one at 900°C and 950°C. Both Alloy 617 and Alloy 230 have similar creep rupture lives at 950°C and 900°C, while Alloy 230 has a longer creep life compared to Alloy 617 at 850°C.

4. The Monkman-Grant relation provides an acceptable method for predicting the long-term creep life for Alloy 617. However, the material constants for Alloy 230 seem to be temperature dependent. This can be attributed to the dramatic creep mechanism change from 850°C to creep behavior at higher temperature.
CHAPTER 8-FUTURE WORK

Previous uniaxial stress creep studies by Tung [27] showed that the creep damage was more severe in biaxial stress state compared to uniaxial stress state. Since the damage is attributed to creep void development, it is important to consider the impact of biaxial stress-state on nucleation and growth rate of creep voids. Therefore, the future work will focus on the creep model development in both biaxial and uniaxial stress state.

Another way to deal with the failure of the accelerated creep damage related to creep cavities is to consider the propagation of macroscopic cracks. This process takes up the largest fraction of the creep rupture life. The initial macroscopic cracks inside the materials may come from some metallurgical defects, manufacturing processes and most likely from the evolution of grain boundary carbides. Therefore, fracture mechanics should be used as a fundamental tool to study the microscopic crack formation processes and the macroscopic crack growth mechanisms. In the present study, a good starting point is to consider the stress intensity factor based on biaxial stress state. It is necessary to evaluate the intensity of stress concentration at the crack tip and the corresponding crack propagation.
REFERENCES


APPENDIX

Subroutine for ABAQUS

SUBROUTINE CREEP(DECRA, DESWA, STATEV, SERD, EC, ESW, P, QTILD, 
1 TEMP, DTEMP, PREDEF, DPRED, TIME, DTIME, CMNAME, LEXIMP, LEND, 
2 COORDS, NSTATV, NOEL, NPT, LAYER, KSPT, KSTEP, KINC)

C
  INCLUDE 'ABA_PARAM.INC'

C
  CHARACTER*80 CMNAME

C
  DIMENSION DECRA(5), DESWA(5), STATEV(*), PREDEF(*), DPRED(*), 
 1 TIME(2), 
 2 COORDS(*), EC(2), ESW(2)

C
  DO 3 KK=1,5
  3 DECRA(KK)=0.D0

A= 
P= 
Rm= 
Rn= 
r= 

C
  DECRA(1)=A*QTILD*r/SQRT(2*r*TIME(1)+r**2*TIME(1)**2)*DTIME+
1 Rm*P*QTILD**Rn*TIME(1)**(Rm-1)*DTIME 
  DECRA(5)=A*r/SQRT(2*r*TIME(1)+r**2*TIME(1)**2)*DTIME+
1 Rn*Rm*P*QTILD**((Rn-1)*TIME(1)**(Rm-1))*DTIME

C
  RETURN

END