The Effect of Tungsten on Creep Behavior of Tempered Martensitic 9Cr Steels

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The effect of tungsten on creep behavior and microstructural evolution was investigated for tempered martensitic 9Cr steels with various W concentrations from 0 to 4 wt pct. The creep rupture testing was carried out at 823, 873, and 923 K for up to 54 Ms (15,000 hours). The creep and creep rupture strength increased linearly with W concentration up to about 3 wt pct, where the steels consisted of the single constituent of the tempered martensite. It increased only slightly above 3 wt pct, where the matrix consisted of the tempered martensite and δ-ferrite. The minimum creep rate was described by a power law. The apparent activation energy for the minimum creep rate showed a tendency similar to the W concentration dependence of the creep-rupture strength and was larger than the activation energy for self-diffusion at high W concentrations above 1 wt pct. The martensite lath microstructure with fine carbides along lath boundaries was responsible for a high resistance to creep deformation. With increasing W concentration, the martensite lath microstructure became stabilized, which decreased the minimum creep rate and increased the apparent activation energy for the minimum creep rate.

I. INTRODUCTION

Tempered martensitic steels, such as modified 9Cr-1Mo (9Cr-1Mo-0.2V-0.08Nb-0.1C) and HT-9 (12Cr-1Mo-0.5W-0.3V-0.5Ni-0.2C), are being considered as candidate structural materials to austenitic type 316 stainless steel (SS) for application to first-wall and blanket-structure of demonstration and commercial fusion reactors.1-3 A primary reason for the use of tempered martensitic steels is a higher resistance to swelling compared to type 316 SS.4-6 Recently, tungsten stabilized, tempered martensitic Cr-W steels have become of interest as a replacement for conventional Cr-Mo steels, such as modified 9Cr-1Mo and HT-9, from the viewpoint of reduced radioactivation of fusion components.7,8 In order to simplify special waste storage of highly radioactive structures of a fusion reactor after service, fast decay characteristics of induced radioactivity are also imposed on the candidate steels. In particular, the concentrations of Mo and Nb in the candidate steels must be severely restricted, because they transmute to long-lived radioactive nuclides in high-energy fusion neutrons. The alloying elements that can be used in a reduced radioactivation steel include C, Cr, W, V, Ta, Ti, Mn, and Si. At present, Cr-W steels offer the best possibility for the base composition of reduced radioactivation bainitic or martensitic steels.9-11 Of the Cr-W steels, 9Cr-W steels are the most promising as structural materials because of their excellent high-temperature creep strength and toughness.12,13 We already showed that the creep-rupture strength of experimental steels of Cr-2W-0.1C (wt pct) with various Cr concentrations from 2 to 15 wt pct had its maximum at about 9 to 10 wt pct Cr at 873 K. In previous reports,14,15 we examined the microstructural evolution in the tempered martensitic 9Cr-W steels with various W concentrations from 0 to 4 wt pct during tempering and subsequent aging and the effect of microstructural evolution on creep behavior of the tempered martensitic 9Cr-2W steel. The microstructural evolution, such as precipitation behavior, in the tempered martensite during aging depended on the concentration of W and aging temperature. During creep of the 9Cr-2W steel at 873 K, the microstructural evolution (e.g., the recovery of excess dislocations, the agglomeration of M23C6 carbides, and the growth of martensite lath subgrains) occurred with the aid of stress, while it was negligibly small during aging in the unstressed condition. The minimum creep rate was shown to be influenced by the microstructural evolution. Therefore, because the rate of microstructural evolution in the 9Cr-W steels during aging depended on the concentration of W, the creep behavior would also be influenced by the W concentration. The fact that the creep behavior of bainitic or martensitic steels is affected by microstructural evolution has been reported by many researchers.16-19

The purpose of the present research is to investigate the effect of W on creep behavior of tempered martensitic 9Cr steels containing 0 to 4 wt pct W. The creep-rupture testing was carried out at 823, 873, and 923 K for up to 54 Ms (15,000 hours), and microstructural observations by transmission electron microscopy (TEM) were made. The apparent activation for the minimum creep rate was also obtained. The effect of W on creep behavior is discussed, taking into account the microstructural evolution.

II. EXPERIMENTAL PROCEDURE

Four kinds of 9Cr steels with different levels of W were used. The chemical compositions are given in Table I. Only the concentration of W was varied from 0 to 4 wt pct in the steel, while the other alloying elements were kept constant (0.1 wt pct C, 9Cr, 0.5Mn, and 0.3Si). According to the concentration of W, the steels are designated 9Cr, 9Cr-1W, 9Cr-2W, and 9Cr-4W. The preparation of the steel rods was described...
Table I. Chemical Composition of Steels Examined (Weight Percent)

<table>
<thead>
<tr>
<th>Steels</th>
<th>C</th>
<th>Cr</th>
<th>W</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>O</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>9Cr</td>
<td>0.104</td>
<td>8.96</td>
<td>--</td>
<td>0.49</td>
<td>0.30</td>
<td>&lt;0.002</td>
<td>0.003</td>
<td>0.009</td>
<td>0.001</td>
</tr>
<tr>
<td>9Cr-1W</td>
<td>0.101</td>
<td>9.01</td>
<td>0.99</td>
<td>0.48</td>
<td>0.29</td>
<td>&lt;0.002</td>
<td>0.004</td>
<td>0.011</td>
<td>0.002</td>
</tr>
<tr>
<td>9Cr-2W</td>
<td>0.100</td>
<td>8.92</td>
<td>1.92</td>
<td>0.48</td>
<td>0.28</td>
<td>&lt;0.002</td>
<td>0.003</td>
<td>0.012</td>
<td>0.002</td>
</tr>
<tr>
<td>9Cr-4W</td>
<td>0.101</td>
<td>9.09</td>
<td>3.93</td>
<td>0.50</td>
<td>0.29</td>
<td>&lt;0.002</td>
<td>0.002</td>
<td>0.006</td>
<td>0.002</td>
</tr>
</tbody>
</table>

elsewhere. The steel rods were austenitized and quenched and then tempered. Heat-treatment conditions, matrix phase, and precipitates present after tempering are given in Table II. The 9Cr, 9Cr-1W, and 9Cr-2W steels were observed to be tempered martensitic, while the 9Cr-4W steel contained both tempered martensite and δ-ferrite. The volume fraction of the δ-ferrite in the 9Cr-4W steel was 10 pct. The δ-ferrite was arranged in a bamboo structure in the matrix of tempered martensite. The bamboo had a diameter of about 5 μm and was distributed parallel to the rolling direction of the steel rods. Therefore, the δ-ferrite was oriented parallel to the stress direction in the creep-rupture testing. The tempered martensite consisted of lath subgrains that contained a high density of dislocations produced by the martensitic transformation during cooling after austenitizing. The width of the martensite laths was about 0.5 μm, and the average grain size of prior austenite was about 50 μm. The carbides, mostly M23C6, were about 0.1 μm or less in size and were distributed preferentially along lath boundaries, prior austenite grain boundaries, and martensite/δ-ferrite boundaries. The δ-ferrite grains contained no carbide and a much lower density of dislocations. The details of the microstructure of the steels are described elsewhere.

Creep-rupture testing was carried out at 823, 873, and 923 K for up to 54 Ms (15,000 h), using specimens of 6 mm in gage diameter and 30 mm in gage length. The creep curves were obtained by measuring the displacement of the pullrod by a dial gage. After testing, the longitudinal cross section of the specimens was observed metallographically by a 200 kV transmission electron microscope (JEM-200CX). The preparation of thin foils for the TEM observations was described elsewhere.

III. EXPERIMENTAL RESULTS

A. Creep-Rupture Strength

Figure 1 shows the creep rupture data for the 9Cr, 9Cr-1W, 9Cr-2W, and 9Cr-4W steels at 823, 873, and 923 K. The addition of W to the 9Cr steel causes a significant shift of the stress vs rupture time curves to higher stresses and longer rupture times. The creep rupture strength of the steels was evaluated from the rupture data and is shown in Figure 2 as a function of W concentration. The creep rupture strength of the 9Cr-4W steel, having the highest W concentration of the steels in this work, is as high as that of 9Cr-1MoVNb steel that has the highest creep-rupture strength of candidate ferritic/martensitic steels for fusion reactor structures. Taking into account that the martensite constituent extends to about 3 pct W and δ-ferrite forms at high W concentrations above 3 pct W in the 9Cr steel with 0.1 pct carbon, the creep rupture strength increases linearly with W concentration in the region of tempered martensite. However, the contribution of W to the increase in creep-rupture strength decreases at high W concentrations above 3 pct, where the steel consists of tempered martensite and δ-ferrite. This suggests that the creep rupture strength of the δ-ferrite is much lower than that of the tempered martensite. The contribution of W to the increase in creep rupture strength in the tempered martensite, $\Delta \sigma/\Delta C_w$, depends on test temperature. The $\Delta \sigma/\Delta C_w$ decreased with increasing test temperature: $\Delta \sigma/\Delta C_w = 44, 32, and 20$ MPa/wt pct W at 823, 873, and 923 K, respectively.

B. Creep Curve and Creep Rate

Figure 3 shows the creep curves and their creep rate curves for the 9Cr-1W, 9Cr-2W, and 9Cr-4W steels at 873 K and 137 MPa. The time to rupture was 150 (42), 1369 (380), and 14,175 ks (3937 hours) for the 9Cr-1W, 9Cr-2W, and 9Cr-4W steels, respectively. The creep curves consist of a primary or transition creep region, where the creep rate decreases with time, and of a tertiary or acceleration creep region, where the creep rate increases with time after reaching a minimum creep rate. In the initial stage of creep for up to 1 ks (about 20 minutes), the creep rates are not largely different among the steels, despite different W levels. At long times above 1 ks, the further decrease in creep rate with time becomes more significant with increasing W concentration.