Variation of creep resistance in ferritic steels by a heat treatment

G. González, R. Molina, M. Delavalle, L. Moro

Abstract

In the power plants, boiler pipes and heaters, are made with ferritic steels low alloy. These steels have a microstructure with fine stable alloy carbides that impede the movement of the dislocations, however it is inevitable that during long periods of service or very critical conditions, microstructural changes occur that are responsible for the loss of material strength. In the past decades the 1Cr-0.5Mo steel was used, but it has been replaced by ferritic steels containing higher amounts of Cr and Mo, with the addition of other micro alloying elements such as niobium, titanium and vanadium to increase their mechanical strength. The objective of this work is to study the creep behavior of 1Cr-0.5Mo steel and to compare its strength when prior to service it is subjected to different heat treatments that improve its conditions of service, as that is beneficial from the economical point of view. Tensile creep tests were performed at a temperature range between 843 and 893 K, and applied stresses between 131 and 205 MPa in the material reception conditions comparing its behavior with others that previously has undergone different heat treatments. From experimental data the characteristic parameters were calculated such as the creep coefficient of stress and activation energy. The microstructural variation of the original material was also analyzed, after heat treatment and creep samples were characterized by optical microscopy, scanning electron microscopy and analysis by dispersive X-ray spectroscopy, to evaluate the effects of kinetics changes occurred in the precipitated phases and the presence of microstructural damage, such as nucleation, growth and coalescence of micro cavities. The microhardness of the phases present in the different samples were also measured.

Keywords: 1Cr-0.5Mo steel; microstructure; creep; characteristic parameters

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1. Introduction

Power plants use fuel combustion or nuclear fission to produce steam pressure. The process of transformation of the steam kinetic energy in electric energy is performed in the turbines and generators. This procedure requires high service temperatures to be efficient and comply with the Carnot cycle [Viswanathan et al. (1988)].

It’s expected that the steam turbines, boiler pipes and heat exchangers operate longer than 30 years of service. Evans et al. (1985) indicate that the working conditions are at temperatures between 723 and 873 K, and variable pressure from 15 to 100 MPa.

Technologically, it is intended that the equipment and components operate correctly during the whole work period. The rupture of this equipment means a high maintenance cost for the industries. Oxidation, corrosion, fatigue and most importantly creep are the critical processes that affect the components integrity. This phenomenon, that occurs at high temperatures and tensions below the elastic limit, produces a progressive deformation as time goes which is reflected as a poorer mechanical strength and microstructure transformations [Viswanathan et al. (1988)].

Microstructure of low alloy carbon steel is stable despite being subjected to high temperatures and pressures. This stability helps the materials resistance to degradation. Ferritic steel has a structure with fine stable alloy carbides that prevents movements of the dislocations. However it is inevitable that transformations occur during long periods of service or very critical conditions, causing a loss of material strength. Some of these structure modifications can be: carbides precipitation and transformation, bainite/pearlite areas decomposition, changes in the carbide morphology and chemistry variations of the matrix. The predominance of a particular transformation depends on the microstructure of the material in its original state.

Badeshia studied the ferritic steel morphology after being subjected to different heat treatments and found that precipitated carbides, when the initial sample had bainite structure, were different from a material with an initial structure composed of bainite and ferrite [Bhadeshia (2001)].

In the past decades the 1Cr-0.5Mo steel was used but it has been replaced by ferritic steels containing higher amounts of Cr and Mo, with the addition of other micro alloying elements to increase their mechanical strength. The objective of this work is to study the creep behavior of 1Cr-0.5Mo steel and to compare its strength when prior to service it is subjected to heat treatment to improve its performance, as that is beneficial from the economic point of view.

2. Experimental method

Steel samples of 1Cr-0.5Mo were taken from seamless tubes of 73 mm outer diameter and 12 mm thickness. In order to analyze the microstructure of the specimens, the samples were attacked chemically with 2% Nital. Observation of the microstructure of the material was done by light and scanning electron microscopy (SEM), for the latter JEOL 35 CF microscope was used, which has an energy dispersive X-ray diffraction (EDX).
The microstructure of the steel in its as-received condition is shown in Fig. 1, where it is possible to see that the structure is essentially ferrite/bainite as well as a significant dispersion of carbides. It is known that the initial structure of a low alloy steel creep resistant, depends not only on its chemical composition but also on the cooling rate after the manufacturing process, and may consist of ferrite+pearlite, ferrite+pearlite+bainite or bainite+ferrite [Porter et al. (2001)].

The chemical composition of the samples was determined by a plasma emission spectrometer, Spectromax Model X. Table 1 shows the measured concentration (% by weight) of the materials by element (% by weight).

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Cr</th>
<th>Mo</th>
<th>Si</th>
<th>Ni</th>
<th>Mn</th>
<th>Al</th>
<th>Cu</th>
<th>Others</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05</td>
<td>1.08</td>
<td>0.36</td>
<td>0.70</td>
<td>0.04</td>
<td>0.40</td>
<td>0.04</td>
<td>0.01</td>
<td>P, S &lt;0.02</td>
<td>Balance</td>
<td></td>
</tr>
</tbody>
</table>

1Cr-0.5 Mo steel exhibits good mechanical strength at high temperature due to the precipitation of carbides but low corrosion resistance at temperatures around 823 K for its low chrome content. Since its use is very interesting from an economic point of view, thermal treatment was performed to improve the resistance to creep. This treatment consisted of austenitization at 1223 K followed by cooling in air and then tempering at 1023 K for 3 hours and cooling in air again.

To determine the material behavior with regards to heat treatment and compared with untreated material, creep tests were performed on specimens of both materials. These tests were performed with tensile equipment where stress and constant temperature is maintained under the conditions of ASTM E 139 [American National Standard, ASTM E 139-11, (2011)].

The equipment has a main pivot rod, with constant radius cams. The selected stress depending on the distance to center pivot. The deformation of the calibrated area of the specimen is measured by two linear variable differential transformers (LVDT) Solartron with a 0.01 mm sensitivity, which produces a DC output with amplitude proportional to the displacement of a movable core. The output of the LVDT is acquired by an analog-digital system [American National Standard, ASTM E 139-11, 2011].

The creep test is carried out at stresses (σ) between 82 and 205 MPa and at temperatures in the range from 843 to 923 K. The equivalent strain rate is determined, which is related to the temperature and the applied stress, from equation or empirical power law which is expressed as

$$
\dot{\varepsilon} = A \sigma^n e^{-Q/RT}
$$

(1)
where $\dot{\varepsilon}$ is the equivalent strain rate, $A$ is a constant depending on the structure, $n$ is the strain exponent, $Q$ is the creep activation energy, $R$ is the universal gas constant, $T$ the absolute temperature [Kassner et al. (2004)].

Finally Vickers hardness of the original specimens as well as the thermally treated specimens before the creep test was measured. For this, a micro hardness tester, Future-Tech, FM-300 was used.

3. Results and discussion

Creep tests were performed at different stresses and temperatures to evaluate the behavior of the material when exposed to these conditions. The material was studied as cast and with the heat treatment, and graphics of the specific deformation were made according to the corresponding time of tests at temperatures of 843, 873 and 923 K, and an applied stress of 168 MPa. From this graphics the strain rate of the secondary creep zones (steady state) was calculated. Table 2 indicates the strain rates for stationary state.

<table>
<thead>
<tr>
<th>Steel</th>
<th>843 K</th>
<th>873 K</th>
<th>923 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>Without heat treatment</td>
<td>$2.9216 \times 10^{-10}$</td>
<td>$1.1503 \times 10^{-8}$</td>
<td>$3.5039 \times 10^{-6}$</td>
</tr>
<tr>
<td>With heat treatment</td>
<td>$1.9085 \times 10^{-10}$</td>
<td>$6.6965 \times 10^{-9}$</td>
<td>$3.0450 \times 10^{-7}$</td>
</tr>
</tbody>
</table>

Also, tests at temperatures of 873 K and stresses of 131, 168 and 205 MPa were conducted, getting the strain rate for stationary state, indicated in Table 3. According to the stated values in all cases of steels treated by heat, the strain rate decreases and increases the duration of the test, which also means an increase of the material strength.

<table>
<thead>
<tr>
<th>Steel</th>
<th>131 MPa</th>
<th>168 MPa</th>
<th>205 MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>Without heat treatment</td>
<td>$1.4615 \times 10^{-8}$</td>
<td>$4.0487 \times 10^{-9}$</td>
<td>$6.6482 \times 10^{-7}$</td>
</tr>
<tr>
<td>With heat treatment</td>
<td>$6.5646 \times 10^{-9}$</td>
<td>$7.0919 \times 10^{-9}$</td>
<td>$2.7329 \times 10^{-8}$</td>
</tr>
</tbody>
</table>

Activation energy ($Q$) can also be calculated from the power law of eq. (1). Based on tests performed at different temperatures and under a constant stress, strain rate as a function of the reciprocal of the temperature is plotted in Fig. 2. Results were adjusted by a linear regression through a line of slope proportional to $Q$. 

![Fig. 2. Logarithmic plot to determine the activation energy ($Q$) for: (a) as-received condition (b) heat treated material.](image-url)
With a similar procedure, creep strain rate values as a function of the mechanical stresses are also represented for a constant temperature of 873 K, in Fig. 3. Results were adjusted by a linear regression through a line whose slope allows obtaining the values of the stress exponent \( n \).

The values of the activation energies and values of the stress exponent obtained are shown in Table 4.

<table>
<thead>
<tr>
<th>Steel</th>
<th>Activation Energy (kJ/mol)</th>
<th>Stress Exponent</th>
</tr>
</thead>
<tbody>
<tr>
<td>1Cr-0.5 Mo Without heat treatment</td>
<td>764</td>
<td>2.13</td>
</tr>
<tr>
<td>With heat treatment</td>
<td>418</td>
<td>7.15</td>
</tr>
</tbody>
</table>

Fig. 4 shows the structure of the material after austenization at 1223 K, showing a matrix of ferrite with grains bigger than in the original state, dispersed bainite and a higher number of precipitates in the grain boundaries.

Fig. 5 shows the structure from the thermal treatments, which evidences a matrix of equiaxed grains of ferrite and dispersed grains of bainite; the number and size of these colonies increase with higher temperature.

Fig. 6 shows the structure of a carbide. In the material that was thermally treated, is higher the precipitation of carbides.
Hald et al. (2003) presented a map indicating different stable phases of precipitate in Cr-Mo steels and suggested that the stable carbides are $M_3C$, $M_2C$, $M_7C_3$. 
In Fig. 7 the EDX analysis of carbide for the material thermally treated is shown. The values of Vickers hardness obtained are shown in Table 5. These results can be explained by the fact that at 873 K carbides of type M₂C are stable and responsible for the creep resistance of Cr-Mo steel. It has been found that M₂C, M₄C and M₂₃C₆ coexist after thermal treatment [Dobrzanski et al. (2007)].

Table 5. Values Vickers hardness.

<table>
<thead>
<tr>
<th>Material</th>
<th>As cast</th>
<th>Austenitized</th>
<th>Tempering</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vickers hardness</td>
<td>164</td>
<td>153</td>
<td>184</td>
</tr>
</tbody>
</table>

4. Conclusion

From the obtained results we can conclude that:
- As a result of the thermal treatment it can be observed that when increasing duration of the heating process the number of colonies of bainite and precipitated carbides in the microstructure increases. This is related to increasing values of hardness.
- The creep tests done on the material in its original state exhibit lower values for the creep coefficient of stress and activation energy than those obtained on steel with a higher content of chromium. This explains its lower mechanical strength.
- Microhardness values in austenitized steel decrease, and after tempering increase from the original steel values, where a structure with a larger quantity of precipitated carbides is seen.
- On the basis of the results obtained, it is deduced that the heat treated steel improves its service conditions, specially its creep resistance.

Acknowledgements

The authors thank the economic collaboration of the Secretaría de Ciencia y Tecnología de la Facultad Regional Bahía Blanca (UTN) and Fundación Hermanos Agustín y Enrique Rocca, which allowed this publishing.

References