Effect of Long-Term Service Exposure at Elevated Temperature on Microstructural Changes of 5Cr-0.5Mo Steels

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Effects of long-term service exposure at elevated temperature on microstructural changes have been studied for both virgin and service-exposed process heater tube pipes of 5Cr-0.5Mo steels used in oil refineries. Samples selected for this study had experienced a nominal temperature range of 450 °C to 500 °C for about 20 to 25 years. Two different initial virgin microstructures were taken and designated by steel A and steel B. The virgin microstructure of steel A exhibited fine platelets of fibrous or hairlike M₇C carbides within the ferrite grains and occasionally irregularly shaped M₂₃C₆ carbides, both along the grain boundaries and at the lath interiors, and very few spheroidally shaped M₇C carbides, either along the grain boundaries or at the grain interiors. In addition, regular geometrically shaped M₃C₆ carbides, such as hexagonal, square, and triangular types, were observed to form at the grain interiors. The virgin steel B microstructure exhibited predominantly M₃C₆ carbides, either along the grain boundaries or at the lath boundaries. Occasionally, fine plates of M₇C₆ carbides were also observed within the laths. The position, shape, distribution, and type of carbides in virgin steel A changed significantly due to 220,000 hours of service exposure in the temperature range of 450 °C to 500 °C. Massive M₃C₆ carbides precipitated along the grain boundaries. In addition, regular geometrically shaped M₇C₆ carbides, such as hexagonal, square, and triangular types, were observed to form at the grain interiors. The virgin steel B microstructure exhibited predominantly M₃C₆ carbides, either along the grain boundaries or at the lath boundaries. Occasionally, fine plates of M₇C₆ carbides were also observed within the laths. The position, shape, distribution, and type of carbides did not change significantly due to 172,000 hours of service exposure in the temperature range of 450 °C to 500 °C. The average interparticle spacings of the carbides increased from 0.35 to 1.2 μm due to 172,000 hours of exposure.

I. INTRODUCTION

A 5Cr-0.5Mo steel is widely used in petrochemical industries, primarily because of its high strength and corrosion resistance against oils and crudes containing hydrogen sulfide and other corrosive agents. This steel is conventionally given an austenitizing treatment in the temperature range of 925 °C to 970 °C followed by either air cooling or furnace cooling and subsequently tempering in the range of 650 °C to 700 °C, which is considerably higher than the maximum operating temperature. Due to the long-term service exposure of this steel at elevated temperature, microstructural changes in terms of carbide coarsening, formation of more stable carbides either from solid solution or at the expense of the existing carbides, and precipitate-free zone formation are expected to occur. The microstructural changes due to long-term exposure might cause mechanical property degradation.

In recent years, there has been considerable interest paid by the metallurgists and engineers in extending the service period of a component used at elevated temperatures in power plants, petrochemical industries, chemical and fertilizer plants, etc., beyond its design life due to current economic considerations. Among the various methods developed so far, the metallographic method appears to be the most attractive method. Because it requires a small sample, the method could be made nondestructive; in principle, the metallographic method measures the damage micro-mechanism directly. Therefore, for the estimation of the service life of a component by means of the metallographic method, microstructural information of long-term service-exposed material is required. Several reports and technical articles have already been published on microstructural changes due to long-term service exposure of 1Cr-0.5Mo, 0.5Cr-0.5Mo-0.25V, 2.25Cr-1Mo, and 1Cr-1Mo-0.25V steels. However, information relating to microstructural changes due to long-term service exposure of 5Cr-0.5Mo steel at elevated temperatures is scarce and sparsely distributed in the literature.

Therefore, the present work was undertaken to strengthen the microstructural information in the literature for 5Cr-0.5Mo steel subjected to long-term service exposure at elevated temperatures. Such microstructural information could be helpful for assessing the service life of a component beyond its design life.

II. EXPERIMENTAL PROCEDURE

The steel samples used for the present investigation are commercial grade 5Cr-0.5Mo steels. The chemical composition in weight percent of the steels is given in Table I. The heat treatments were performed by the tube manufacturers and are unknown to the authors. The samples were collected from virgin and service-exposed process heater tubes used in oil refineries. The insides of the tube were exposed in a crude oil environment for a long time in the temperature range of 450 °C to 500 °C. During crude oil processing at elevated temperatures, petroleum coke deposits in the inside of the tube; as a result, the inside diameter of the tube decreases. Initially, these coke deposits were removed from the inside mechanically at regular intervals according to the planned shutdown of the plants. But presently, these are removed by heating the tube at about 700 °C from outside. In addition, during startup and shutdown of the plant, the process heater tubes experience low cycle
Table I. The Chemical Composition (Weight Percent) of the Steels Investigated

<table>
<thead>
<tr>
<th>Steel Designation</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steel A</td>
<td>0.11</td>
<td>0.38</td>
<td>0.36</td>
<td>5.57</td>
<td>0.44</td>
</tr>
<tr>
<td>Steel B</td>
<td>0.12</td>
<td>0.40</td>
<td>0.44</td>
<td>5.19</td>
<td>0.48</td>
</tr>
</tbody>
</table>

Fatigue deformation as well. The microstructural characterization of the service-exposed tubes was performed only from the outer portion of the process heater tubes, since the aim of this work was to study the effect of long-term service exposure on microstructural changes. After collecting samples from the tubes, all samples were polished by conventional metallographic methods and etched with 2 pct nital. Replicas were made by the conventional carbon extraction replica preparation method. Thin foils were also made from the thin slices of the virgin and service-exposed samples. Thin slices were cut using a low speed diamond saw. These were then mechanically thinned to less than 0.1-mm thickness using emery paper. Thin foils were then prepared by double jet polishing in a twin jet electropolisher using an electrolyte containing 10 pct perchloric acid and 90 pct glacial acetic acid. Carbon extraction replicas and thin foils were then examined in a PHILIPS* 400 EM transmission electron microscope using operating voltages of 100 and 120 kV.

III. RESULTS

A. Hardness

The hardnesses of virgin steel A and steel B were measured using a Vickers hardness tester and found to be 151 and 190 HV, respectively.

B. Virgin Microstructure

1. Steel A

Figure 1 is a very low magnification transmission electron micrograph (TEM) taken from a carbon extraction replica revealing predominantly fibrous-type carbides, fine platelets, occasional irregularly shaped carbides, and spheroidally shaped carbides, either along the grain boundaries or at the grain interiors. Figure 2(a) is a higher magnification carbon extraction TEM replica of area A, shown in Figure 1, exhibiting fine platelets (0.75- to 3-μm long) of carbides. A corresponding selected area diffraction pattern (SADP) is shown in Figure 2(b). Analysis of the pattern identifies the fine platelets as M$_2$C. Figure 3(a) is also a carbon extraction replica TEM showing fibrous-type carbides. A corresponding SADP from the fibrous-type carbides, as shown in Figure 3(b), identifies the fibrous carbides as M$_3$C. In addition to fibrous carbides, grain boundary carbides are also observed in Figure 3(a). Figures 4(a) and (b) are two carbon extraction replica TEMs showing grain boundary carbides at two different locations. Corresponding SADPs are shown in Figures 4(c) and (d), respectively. Analysis of the patterns identify the carbides as M$_3$C. Based on this study, steel A was probably annealed.