The Nucleation of High Temperature Brittle Intergranular Fracture in 2.25Cr-1Mo Steel

C. A. HIPPSLEY and J. J. LEWANDOWSKI

An investigation of brittle intergranular crack nucleation at high temperatures in 2.25Cr-1Mo steel is presented. A series of tests was designed to allow the independent variation of segregating impurities (e.g., sulfur and phosphorus) and locally applied stress levels to determine the effects of applied stress on impurity enrichment and fracture of grain boundaries in as-quenched material. The study utilized blunt notched specimens with suitable finite element stress analyses, together with high resolution Auger electron surface analysis techniques. It is shown that the imposition of a tensile hydrostatic stress enhances the segregation of elemental sulfur to grain boundaries prior to the nucleation of high temperature brittle intergranular cracks. The location of enriched grain boundaries relative to the peak stress regions in notched specimens is discussed, and possible mechanisms for the observed sulfur enrichment presented.

I. INTRODUCTION

High temperature brittle intergranular fracture (HTBIGF) has been identified in recent years as a potential mode of failure in low-alloy steel heat-affected zone (HAZ) microstructures. It was first studied as a mechanism of stress-relief cracking but has since received attention as a mode of sustained load cracking in its own right. HTBIGF is manifested most clearly in material which has been austenitized at >1100 °C, and occurs in the presence of a stress-concentrator at temperatures between 300 and 650 °C.

The majority of investigations into this fracture mechanism have concentrated on crack propagation. Detailed study of fracture surfaces formed in vacuum has shown intergranular facets with periodic striations, suggesting a stepwise mode of crack propagation. This has been confirmed by acoustic emission measurements. Analysis of the surface chemistry at crack tips in specimens tested in air and under high vacuum (1.3 × 10⁻³ Pa) has indicated that cracking is associated with a local crack-tip enrichment of sulfur together with more uniform grain boundary segregation of phosphorus.

Several models for the cracking process have been proposed. One essential difference concerns the route by which sulfur enters the crack tip region. It has been suggested that sulfur is either enriched on crack faces and then driven into the tip by surface diffusion, or that it is driven to the crack tip internally under the influence of the crack tip stress field. It is possible to differentiate between these two types of mechanism by analysis of the nucleation stage of HTBIGF, i.e., the period over which sulfur is enriched sufficiently to enable local fracture of a grain boundary under the prevailing stress.

An initial study of HTBIGF in notched bend specimens has indicated that cracks can nucleate within the material in subnotch regions of elevated stress. Crack nucleation was associated with sulfur enrichment, and was affected by the amount of sulfur in solution after austenitizing. There was associated with sulfur enrichment, and was affected by the amount of sulfur in solution after austenitizing. There was a limited amount of evidence to support the view that sulfur is driven internally to grain boundaries under high temperature austenitizing (1300 °C) followed by either water quenching (leaving most sulfur in solution), or step cooling to 300 °C (allowing more sulfur to form precipitates). Table II summarizes the heat treatments used in the present study.

II. EXPERIMENTAL

A. Materials

Commercial purity 2.25Cr-1Mo steel of composition given in Table I was chosen for the present work, in accordance with previous studies. Heat treatments were chosen to vary the amount of sulfur available to segregate under stress at elevated temperatures and comprised high temperature austenitizing (1300 °C) followed by either water quenching (leaving most sulfur in solution), or step cooling to 300 °C (allowing more sulfur to form precipitates). The range of test conditions and notch geometries employed are given in Table III. Finite element analyses, which are currently available for these geometries, were used to derive the principal stress and strain distributions imposed within each specimen. In a typical test, one of the two specimen notches develops a significant crack, extending from the nucleation site in front of the notch tip back to link up with the notch, i.e., a ‘macrocrack’.


C. A. HIPPSLEY is with Materials Physics and Metallurgy Division, Harwell Laboratory, Oxfordshire, OX 11 ORA, England. J. J. LEWANDOWSKI is with the Department of Materials Science and Engineering, Case Western Reserve University, Cleveland, OH 44106.

VOLUME 19A, DECEMBER 1988—3005
Table I. Alloy Composition (Wt Pct)

<table>
<thead>
<tr>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>S</th>
<th>P</th>
<th>Cr</th>
<th>Mo</th>
<th>Sb</th>
<th>Sn</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.15</td>
<td>0.43</td>
<td>0.35</td>
<td>0.014</td>
<td>0.010</td>
<td>2.30</td>
<td>1.10</td>
<td>0.003</td>
<td>0.020</td>
</tr>
</tbody>
</table>

Table II. Summary of Heat Treatments

<table>
<thead>
<tr>
<th>Condition</th>
<th>Heat Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>austenitize 1300 °C, water quench</td>
</tr>
<tr>
<td>2</td>
<td>austenitize 1300 °C, step cool to 300 °C, oil quench</td>
</tr>
</tbody>
</table>

III. RESULTS

A summary of stress/strain calculations and surface chemical analyses (expressed as differential peak height ratios to the iron peak at 703 eV) is given in Table IV for each specimen and facet. Only the phosphorus, sulfur, and nitrogen levels were quantified, since some carbon and oxygen were adsorbed onto the specimens during exposure within the SAM, making quantitative analysis of these elements unreliable. However, it was still possible to distinguish between facets which had been exposed to the atmosphere during high temperature fracture (giving carbon and oxygen levels > iron level) and those which were exposed only by fracture within the SAM (giving carbon and oxygen levels < 15 pct iron level). Four types of intergranular facet were observed; all were relatively smooth in appearance (to the resolution of the SAM system). First, Figure 3 shows a ‘macrocrack’. Previous work suggests that in this type of crack, fracture nucleates in front of the notch tip, and then propagates in one direction to join up with the notch, while simultaneously traveling farther into the specimen. This type of facet was found in specimen T6 (Table IV), and a