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Annual Progress Report
(May 2001 – May 2002)

Hydrogen-Induced Embrittlement of Candidate Target Materials for Applications in Spallation-Neutron-Target Systems
AAA Task-4

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June 25, 2002
Hydrogen-Induced Embrittlement of Candidate Target Materials for Applications in Spallation-Neutron-Target Systems

Introduction

The primary objective of this task is to evaluate the effect of hydrogen on environment-assisted cracking of candidate target materials for applications in spallation-neutron-target (SNT) systems such as accelerator production of tritium (APT) and accelerator transmutation of waste (ATW). The materials selected for evaluation and characterization are martensitic stainless steels including Alloy HT-9, Alloy EP 823 and Type 422 stainless steel. The susceptibility to stress corrosion cracking (SCC) and hydrogen embrittlement (HE) of these materials are being evaluated in environments of interest using tensile specimens under constant load and slow-strain-rate (SSR) conditions. Further, the localized corrosion behavior of these alloys is being evaluated by electrochemical polarization techniques. The extent and morphology of cracking and localized corrosion of the tested specimens are being determined by optical microscopy and scanning electron microscopy (SEM). The concentration of hydrogen resulting from cathodic charging will be analyzed by secondary ion mass spectrometry (SIMS).

Personnel

The current project participants are listed below.

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Accomplishments

Experimental heats of Alloys HT-9, EP-823 and 422 (three heats of each alloy) have been melted and processed into round bars at the Timken Research Laboratory, Canton, Ohio. Thermal treatments have also been completed on one heat each of these three alloys at this vendor’s heat- treating facility. The chemical compositions of these alloys, and thermal treatments performed on them are shown in Table I.

Table I

Chemical Composition of Martensitic Stainless Steels+

<table>
<thead>
<tr>
<th>Material/Heat No.</th>
<th>C</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Cu</th>
<th>V</th>
<th>W</th>
<th>Cb</th>
<th>B</th>
<th>Ce</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy HT-9/2048*</td>
<td>0.18</td>
<td>0.40</td>
<td>0.012</td>
<td>0.004</td>
<td>0.20</td>
<td>12.26</td>
<td>0.49</td>
<td>1.00</td>
<td>0.01</td>
<td>0.30</td>
<td>0.46</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>Alloy HT-9/2049</td>
<td>0.19</td>
<td>0.41</td>
<td>0.012</td>
<td>0.004</td>
<td>0.20</td>
<td>12.31</td>
<td>0.50</td>
<td>1.00</td>
<td>0.01</td>
<td>0.30</td>
<td>0.47</td>
<td>--</td>
<td>--</td>
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<tr>
<td>Alloy HT-9/2050</td>
<td>0.20</td>
<td>0.39</td>
<td>0.012</td>
<td>0.004</td>
<td>0.20</td>
<td>12.32</td>
<td>0.50</td>
<td>0.99</td>
<td>0.01</td>
<td>0.29</td>
<td>0.47</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>422 Stainless Steel/2051*</td>
<td>0.21</td>
<td>0.55</td>
<td>0.013</td>
<td>0.005</td>
<td>0.51</td>
<td>12.83</td>
<td>0.73</td>
<td>0.98</td>
<td>0.002</td>
<td>0.22</td>
<td>0.93</td>
<td>--</td>
<td>--</td>
<td>--</td>
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<tr>
<td>422 Stainless Steel/2052</td>
<td>0.20</td>
<td>0.56</td>
<td>0.013</td>
<td>0.005</td>
<td>0.51</td>
<td>12.77</td>
<td>0.74</td>
<td>0.98</td>
<td>0.002</td>
<td>0.22</td>
<td>0.92</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>422 Stainless Steel/2053</td>
<td>0.20</td>
<td>0.54</td>
<td>0.012</td>
<td>0.004</td>
<td>0.49</td>
<td>12.76</td>
<td>0.74</td>
<td>0.97</td>
<td>0.002</td>
<td>0.22</td>
<td>0.92</td>
<td>--</td>
<td>--</td>
<td>--</td>
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<tr>
<td>Alloy EP-823/2054*</td>
<td>0.16</td>
<td>0.55</td>
<td>0.014</td>
<td>0.004</td>
<td>1.09</td>
<td>11.70</td>
<td>0.66</td>
<td>0.74</td>
<td>0.002</td>
<td>0.30</td>
<td>0.60</td>
<td>0.24</td>
<td>0.009</td>
<td>0.04</td>
</tr>
<tr>
<td>Alloy EP-823/2055</td>
<td>0.16</td>
<td>0.57</td>
<td>0.014</td>
<td>0.004</td>
<td>1.14</td>
<td>11.71</td>
<td>0.67</td>
<td>0.74</td>
<td>0.002</td>
<td>0.31</td>
<td>0.58</td>
<td>0.23</td>
<td>0.009</td>
<td>0.04</td>
</tr>
<tr>
<td>Alloy EP-823/2056</td>
<td>0.14</td>
<td>0.56</td>
<td>0.013</td>
<td>0.005</td>
<td>1.11</td>
<td>11.68</td>
<td>0.66</td>
<td>0.73</td>
<td>0.002</td>
<td>0.30</td>
<td>0.62</td>
<td>0.22</td>
<td>0.009</td>
<td>0.05</td>
</tr>
</tbody>
</table>

+Melted and Processed at Timken Research.
*Thermal Treatments Performed:
Austenitized: 1850°F/ 1 hr/ OQ
Tempered: 1150°F/ 1.25 hr/ AC
(In batches) 1150°F/ 1.75 hr/ AC
1150°F/ 2.25 hr/ AC
OQ: Oil Quenched
AC: Air Cooled

Tensile specimens have been machined from all heat-treated bars by a qualified machine shop located in Pennsylvania (Laboratory Testing, Inc.). Simultaneously, efforts are well underway to machine small cylindrical specimens to perform electrochemical polarization experiments. Meanwhile, the construction of the “Materials Performance Laboratory (MPL)” has been completed. MPL has numerous materials-related testing capabilities including evaluation of SCC/HE susceptibility under both constant load and SSR conditions, evaluation of localized corrosion (pitting and crevice) behavior using potentiostats, performance of thermal treatments, sample preparation and metallographic evaluations using a high-resolution optical microscope.
The room-temperature mechanical properties of Alloy EP-823 have been determined by using calibrated MTS equipment. The mechanical properties data are shown in Table II. Further, SCC tests of Alloy EP-823 are currently ongoing in SAW(M) environment at ambient temperature using calibrated proof rings (Table III). The experimental setups for constant-load SCC testing and electrochemical polarization studies are shown in Figures 1 and 2, respectively. A typical calibration curve showing potentiodynamic polarization behavior of Type 430 ferritic steel in 1N sulfuric acid solution at 30°C is illustrated in Figure 3.

Table II

<table>
<thead>
<tr>
<th>Material/Heat No.</th>
<th>Sample ID</th>
<th>YS (ksi)</th>
<th>Avg.YS (ksi)</th>
<th>UTS (ksi)</th>
<th>Avg. UTS (ksi)</th>
<th>% El</th>
<th>% RA</th>
<th>Hardness</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy EP-823/2054</td>
<td>S1</td>
<td>110.7</td>
<td>110.5</td>
<td>130.0</td>
<td>129.77</td>
<td>4.96</td>
<td>59.28</td>
<td>Re 30</td>
</tr>
<tr>
<td></td>
<td>S2</td>
<td>109.6</td>
<td>128.9</td>
<td>129.77</td>
<td></td>
<td>5.18</td>
<td>62.19</td>
<td>Re 30</td>
</tr>
<tr>
<td></td>
<td>S3</td>
<td>111.3</td>
<td>130.4</td>
<td></td>
<td></td>
<td>5.68</td>
<td>61.52</td>
<td>Re 30</td>
</tr>
</tbody>
</table>

*Thermal Treatments Performed:  
Austenitized (1850°F/1hr/OQ)  
Tempered (1150°F/1.25hr/AC)

Table III

<table>
<thead>
<tr>
<th>Material/Heat No.</th>
<th>Sample ID</th>
<th>Environment</th>
<th>Temperature</th>
<th>Applied Stress/Load</th>
<th>Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy EP-823/2054</td>
<td>S4</td>
<td>SAW (M)</td>
<td>Ambient</td>
<td>95% YS / 5312 lbs</td>
<td>In Progress</td>
</tr>
<tr>
<td></td>
<td>S5</td>
<td>SAW (M)</td>
<td>Ambient</td>
<td>90% YS / 5042 lbs</td>
<td>In Progress</td>
</tr>
<tr>
<td></td>
<td>S6</td>
<td>SAW (M)</td>
<td>Ambient</td>
<td>90% YS / 5042 lbs</td>
<td>In Progress</td>
</tr>
<tr>
<td></td>
<td>S7</td>
<td>SAW (M)</td>
<td>Ambient</td>
<td>95% YS / 5312 lbs</td>
<td>In Progress</td>
</tr>
</tbody>
</table>

SAW:  Simulated Concentrated Acidic Water  
M: Modified 2<pH<3
Figure 1. Constant-Load SCC Test Setup

Figure 2. Electrochemical Polarization Test Setup
Figure 3. Potentiodynamic Polarization Curve for Type 430 Ferritic Stainless Steel in 1N H₂SO₄ Solution at 30°C, Scan Rate: 0.166 mV/sec (ASTM G 5)

Problems

No problems are anticipated.

Status of Funds

Expenditures incurred during the first year are within the target amount allocated.

Plans for Year 2

- Continue SCC/HE testing of all three types of martensitic stainless steels.
- Perform heat treatments of remaining heats of all three test materials.
- Perform localized corrosion testing using electrochemical techniques.
- Perform metallurgical evaluations including microstructural characterizations.
- Conduct failure analyses using SEM.
- Prepare technical/scientific papers for presentations and publications.