GRAND PRIZE
GAS-ATOMIZED CHEMICAL RESERVOIR ODS FERRITIC STAINLESS STEELS
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ABSTRACT

Gas-atomization reaction synthesis was used to surface oxidize ferritic stainless steel powders resulting in an ultrathin (<100 nm) metastable chromium-enriched oxide shell. Subsequently, the shell was dissociated and served as an oxygen reservoir for the formation of uniformly dispersed nanoscale Y-(Ti, Hf) oxide precipitates during heat treatment of the as-consolidated powders.

Powders in the as-atomized and heat-treated conditions were characterized and compared using several electron microscopy techniques. Scanning (SEM), auger (AES), and transmission (TEM) electron microscopy were used in conjunction with energy dispersive spectroscopy (EDS) and X-ray powder diffraction (XRD) to effectively characterize the oxide layers and the size, shape, location, and chemical composition of the precipitates formed during heat treatment. The goal of these analyses was to determine the composition of the oxide shell and to confirm the exchange of oxygen from the shell to a distribution of finely spaced and highly stable nanoscale oxide particles. Selected examples of the use of these techniques are shown.

Metallographic Analysis

Chemical compositions of the bulk powders are shown in Table I. The three alloys (top to bottom in Table I), will be referred to as A, B, and C. The relative sizes and spheroidal shape of the as-atomized particles are seen in Figure 1 along with the chemical compositions of the surface shells measured at increasing depth from the particle surface using AES.

### TABLE I. CHEMICAL COMPOSITION OF AS-ATOMIZED ALLOYS

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Fe (a/o)</th>
<th>Cr (a/o)</th>
<th>W (a/o)</th>
<th>Ti (a/o)</th>
<th>Hf (a/o)</th>
<th>Y (a/o)</th>
<th>O (a/o)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CR-118Ti-Y</td>
<td>Bal. 15.84</td>
<td>-</td>
<td>0.50</td>
<td>-</td>
<td>0.20</td>
<td>1.67</td>
<td></td>
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<tr>
<td>CR-144Hf-Y</td>
<td>Bal. 16.16</td>
<td>0.94</td>
<td>-</td>
<td>0.27</td>
<td>0.08</td>
<td>0.23</td>
<td></td>
</tr>
<tr>
<td>CR-156Y-Hf</td>
<td>Bal. 15.84</td>
<td>-</td>
<td>-</td>
<td>0.11</td>
<td>0.18</td>
<td>0.38</td>
<td></td>
</tr>
</tbody>
</table>

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SEM images of cross sections of the three alloy powders mixed with 75 v/o Cu powder and cold isostatically pressed (CIPed) are shown in Figure 2. The alloy particles are the round dark-gray particles in the matrix of lighter-gray copper particles. Initial evidence of phase precipitation is visible as a light-gray network in a few of the alloy particle cross sections.

Additionally, the three alloy powders were consolidated by hot isostatic pressing (HIP) and heat treated for formation of the stable Y-(Ti,Hf) oxides. Examples of the as-HIPed microstructures of each alloy are shown in Figures 3 and 4. In Figure 3, the as-HIPed microstructures of the three alloys are compared using SEM imaging. Grain boundaries, alloy segregation, and residual porosity are appar-
ent in the micrographs as variations in gray scale.

The TEM images in Figure 4 show the locations and distribution of the nanoscale oxides throughout the same set of HIPed alloys shown in Figure 3. The number and location of the small dark features appear to change from sparse and uniform to more frequent and located along or near grain boundaries.

Further examples of the effectiveness of metallographic analysis in characterizing the alloys are
displayed in Figures 5 and 6. SEM images of the heat-treated alloys (1,300°C for 1 h) are compared in Figure 5. Residual precipitates are seen as either dark (titanium-enriched oxides), almost pinpoint features in the A alloy, to whitish, high-aspect-ratio features (iron–hafnium intermetallic particles) in alloys B and C.

A heat-treated sample (1,200°C for 2.5 h) of alloy B, CR-144Hf-Y, was used to demonstrate chemical analysis mapping using energy-filtered transmission electron microscopy (EFTEM), Figure 6.

**SUMMARY**

Examples of the metallographic techniques used to characterize these alloys have been presented. They demonstrate the effectiveness of metallography as a means of providing the information necessary to make sound and informed judgments about the alloys and their viability to perform in the intended applications.