Effect of irradiation on strengthening of a model Fe-9Cr oxide dispersion strengthened alloy

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INTRODUCTION

The growing energy demand will increasingly call upon advanced nuclear reactors to supply safe and reliable energy worldwide. However, structural and cladding materials in these reactors will be subject to extreme conditions of up to a few hundred displacements per atom (dpa) at temperatures as high as 600°C. Oxide dispersion strengthened (ODS) alloys are leading candidates for these components because their dispersion of Y-Ti-O nanoclusters provides high strength at elevated temperatures and dimensional stability under irradiation. But recent work [1-2] has suggested that the oxides are unstable under irradiation, which has serious implications on mechanical performance. Thus, the objective of this work is to understand the change in strengthening mechanisms of an Fe-9Cr ODS alloy under irradiation.

We use two approaches to assess strengthening of a proton-irradiated and neutron-irradiated Fe-9Cr ODS. First, nanoindentation directly measures changes in yield strength. Second, microstructure measurements (published in ref. [1]) are passed through models of two strengthening mechanisms. Results of these two approaches are compared to infer the strengthening mechanisms in the irradiated ODS alloy.

EXPERIMENTAL METHODS & RESULTS

Materials & Irradiations

A rod of Fe-9Cr ODS (Table I) was fabricated by the Japan Atomic Energy Agency. The rod was heat treated at 1050°C for 1 hour, air cooled, then tempered at 800°C with subsequent air cooling, for a fully martensitic final structure. The rod was cut by electrical discharge machining into bars or discs for irradiation. These specimens were mechanically and electropolished.

Specimens were irradiated to 3 dpa at 500°C using 2.0 MeV protons (dose rate $\sim 10^3$ dpa/s) at the Michigan Ion Beam Laboratory or fast neutrons ($\sim 10^7$ dpa/s) at the Advanced Test Reactor. No temperature shift was employed because it was of interest to this work to isolate the effect of irradiating particle type and dose rate, by keeping all other irradiation experiment parameters fixed. Proton irradiation damage was calculated using Stopping and Range of Ions in Matter (SRIM). Microstructure analysis of proton-irradiated specimens was conducted on the approximately constant region of the damage profile (Fig. 1) at a depth of $\sim 10$ µm, to avoid surface effects and the damage peak near $\sim 20$ µm. Neutrons experience little attenuation through the specimen thickness, so their damage profile can be assumed constant.

![Damage profiles of 2.0 MeV protons and neutrons](image)

**Fig. 1.** Damage profiles of 2.0 MeV protons and neutrons (shown at an arbitrary magnitude) on Fe-9Cr ODS.

Nanoinindentation

Nanoindentation was performed on a Hysitron TI 950 TriboIndenter fitted with a Berkovich tip at CAES. Nanoindents were made in depth-controlled mode, at depths ranging from 50 nm to 1100 nm. The plastic zone sampled by a nanoindent is $\sim 5x$ the depth of the indent itself, so the deepest indents sampled hardness at a depth of $\sim 5.5$ µm from the surface. It should be noted that these depths are shallower than the $\sim 10$ µm depth for microstructure characterization, but indentation is limited by the 25 µN maximum load that can be applied. A minimum of 15 indents was made at each depth to ensure repeatability and statistical certainty. All indents were performed with a 20 second loading time, a five second hold time, and a 20 second unloading time that accounted for thermal drift and creep.

The as-received, proton-irradiated, and neutron-irradiated specimens all exhibited high hardness values from 50-100 nm indents. This is attributed to the surface

<table>
<thead>
<tr>
<th>Chemical Composition (wt%, bal. Fe)</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Ni</th>
<th>Cr</th>
<th>W</th>
<th>Ti</th>
<th>Y</th>
</tr>
</thead>
<tbody>
<tr>
<td>[Y$_2$O$_3$] = 1.27 x [Y]</td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>0.14</td>
<td>0.048</td>
<td>0.05</td>
<td>0.06</td>
<td>8.67</td>
<td>1.95</td>
<td>0.23</td>
<td>0.28</td>
</tr>
</tbody>
</table>

TABLE I. Chemical Composition of 9-Cr ODS
roughness of the specimens being on the same order as the indent depth. Once these surface effects are overcome, the hardness of all three specimens becomes relatively constant (Fig. 2). The change in yield stress (in MPa) can be calculated directly from change in hardness (in GPa):

\[ \Delta \sigma_y = 3.06 \Delta H \]  
Eq. 1

Following this formulation, nanoindentation results suggest proton-irradiation induced softening of -42 MPa and neutron irradiation-induced hardening of 108 MPa.

**DISCUSSION**

Irradiation-induced changes in yield strength can generally be modeled by dispersed barrier hardening (Eq. 2), in which pre-existing and irradiation-induced obstacles inhibit dislocation motion through the material. The contribution of obstacle type \( i \) \((i = \text{loops, voids, precipitates, etc.})\) to the total yield stress is:

\[ \Delta \sigma_i = \alpha_i M \mu b \sqrt{Nd} \]  
Eq. 2

where \( \alpha_i \) is the obstacle-specific strength parameter, \( M \) the Taylor factor, \( \mu \) the shear modulus, \( b \) the Burgers vector, \( N \) the number density of obstacle type \( i \), and \( d \) the average size of obstacle type \( i \).

Yield stress can also be affected by solid solution strengthening, in which solute atoms hinder dislocation motion. In engineering alloys, the irradiation-induced change in solid solution strengthening is generally negligible compared to that of dispersed barrier hardening. However, due to the irradiation instability of oxide nanoclusters in ODS alloys, solid solution strengthening cannot be ignored. The contribution to yield stress attributed to solute specie \( i \) is [3]:

\[ \sigma_{SS,i} = K_i \times C_{SS,i} \times 100 \]  
Eq. 3

where \( K_i \) is the strengthening coefficient of \( i \) (MPa/at%) and \( C_{SS,i} \) is the at% of element \( i \) in solid solution.

The total change in yield strength is calculated using either a linear sum or a root-square sum of all \( \Delta \sigma_i \) and \( \sigma_{SS,i} \) contributions. The linear sum approach is most accurate when all obstacles to dislocation motion are weak, whereas the root-square sum is most accurate when all obstacles are strong. The ODS studied herein contains a mixture of both weak and strong obstacles, so both the linear sum and root-square sum are calculated as bounding approaches.

The individual irradiation strengthening contributions of dislocation loops, voids, oxide nanoclusters, and the solid solution, are based on the microstructure (ref. [1]) and are shown in the colored bars in Fig. 3. The net result of all of these microstructural contributions, then, using either the linear sum or root-square sum approach, is shown as a black bar in Fig. 3. When compared to the measured change in yield strength from nanoindentation (red dashed lines, Fig. 3), it is clear that the root-square sum method predicts strengthening more accurately than does the linear sum method. This is true for both the proton irradiation and neutron irradiation condition. In addition, it can be seen that solid solution strengthening makes a large contribution to the total strengthening of the neutron-irradiated specimen (orange portion of colored bars, Fig. 3). This is due to considerable oxide nanocluster dissolution under neutron irradiation [1]. Upon dissolution, oxide species Y, Ti, and O enter the matrix, and thus significantly change the solid solution strength of the material. Thus, irradiation-induced oxide instabilities require that solid solution strengthening be considered as a strengthening mechanism.
SUMMARY & FUTURE WORK

A model Fe-9Cr ODS steel was irradiated to 3 dpa at 500°C using either 2.0 MeV protons or a fast neutron spectrum. Both irradiation types induced the formation of dislocation loops and voids. Both proton and neutron irradiation resulted in dissolution of oxide nanoclusters, though neutron irradiation led to much more extensive oxide nanocluster dissolution.

The irradiated microstructure was used to predict irradiation hardening by considering both the precipitation strengthening and solid solution strengthening mechanisms. Due to more extensive oxide nanocluster dissolution under neutron irradiation, the solid solution strengthening mechanism must be considered in order for the predicted change in yield strength to be comparable to that measured by nanoindentation.

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REFERENCES