Irradiation Effects in Oxide Nanoparticle Stability and Matrix Microstructure in ODS Steel Neutron Irradiated to 3 dpa at 500°C

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ODS steels are deemed to be among the most promising candidate materials for cladding and core internals of next generation nuclear reactors.

One of the major steps toward the implementation of Generation IV nuclear reactor systems is the development of appropriate materials for cladding and other core internal components that will be able to withstand high doses and temperatures. Oxide dispersion strengthened (ODS) steels are deemed as being among the promising candidate materials for cladding and core internals for advanced nuclear reactors, by virtue of their superior mechanical properties at high temperatures. These properties arise from a fine dispersion of Y-Ti oxide nanoparticles in the steel matrix, which serve as pinning points for dislocation motion and point defect annihilation sites. Additionally, these oxide nanoclusters are expected to trap transmutation-produced helium in small, high-pressure bubbles. To apply these materials in the extreme environments of next generation nuclear reactors, a complete understanding of their radiation response is necessary.

Project Description

In the proposed research, a 9%Cr ODS steel in unirradiated and irradiated (with neutron to 3 dpa at 500°C) conditions, were analyzed with high-resolution microscopy techniques for microstructural and microchemical changes induced by neutron irradiation. The analysis was performed utilizing Transmission Electron Microscopy (TEM), Scanning-TEM coupled with Energy Dispersive Spectroscopy (EDS), and Local Electrode Atom Probe (LEAP, also known as atomic probe tomography, [APT]). Many studies in literature have investigated the stability of the Y-Ti oxide nanoclusters in ODS steels using proton and heavy ion irradiations, but there have been relatively few studies the effects of neutron radiation on these oxide nanoclusters. Specifically, this research focused on the changes caused by the neutron irradiation on the size, density, and composition of the nanoclusters as well as on the formation of extended defects (dislocation

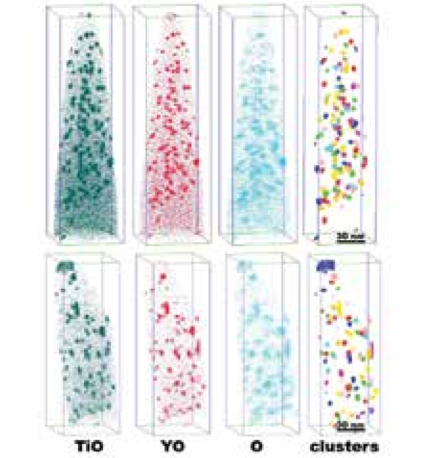

Figure 1. LEAP images showing elemental distribution maps at near-atomic level for the as-received (top row) and neutron irradiated (bottom row) ODS steels.

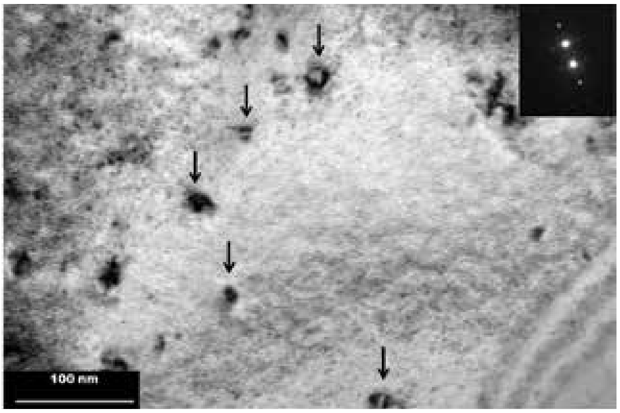

Figure 2. Two beam bright field image of a grain of the neutron irradiated ODS steels showing dislocation loops (identified are indicated by black arrows).

loops) and on the radiation-induced segregation of elements in the steel matrix. The synergistic deployment of multiple atomic resolution analytical techniques was intended to provide a comprehensive understanding of the response of this material to neutron irradiation. Understanding the influence of neutron irradiation-induced changes to the microstructure and microchemistry of ODS steels is key to the successful application of these steels as advanced fission reactor components, and is therefore highly relevant to the DOE-NE program.

Accomplishments

The objective of the proposed research was to develop a fundamental understanding of the structural response of a 9C%Cr ODS steel to neutron irradiation. Specific emphasis was placed on the size and size distribution of the nanoclusters before and after radiation, their compositional changes, as well as the formation of extended defects in the matrix, and radiation-induced elemental segregation.

Analysis of the size, size distribution and chemistry of the smallest precipitates in the matrix (size <5 nm), which were difficult to be imaged by conventional TEM and Scanning Transmission Electron Microscopy (STEM) techniques, was performed by LEAP. After irradiation, it was observed that the average size of the nanoclusters increased by a small fraction, from 2.58 ± 1.19 nm to 3.01 ± 1.71 nm. Correspondingly, the number density of the nanoparticles decreased slightly, from 1.8×10^{23} m^{-3} to 1.2×10^{23} m^{-3}. Figure 1 shows the LEAP 3-D reconstruction of the samples in the

Understanding the stability of the microstructure of oxide dispersion strengthened (ODS) steels under neutron irradiation is critically important to the application of these steels to next generation advanced nuclear reactors.

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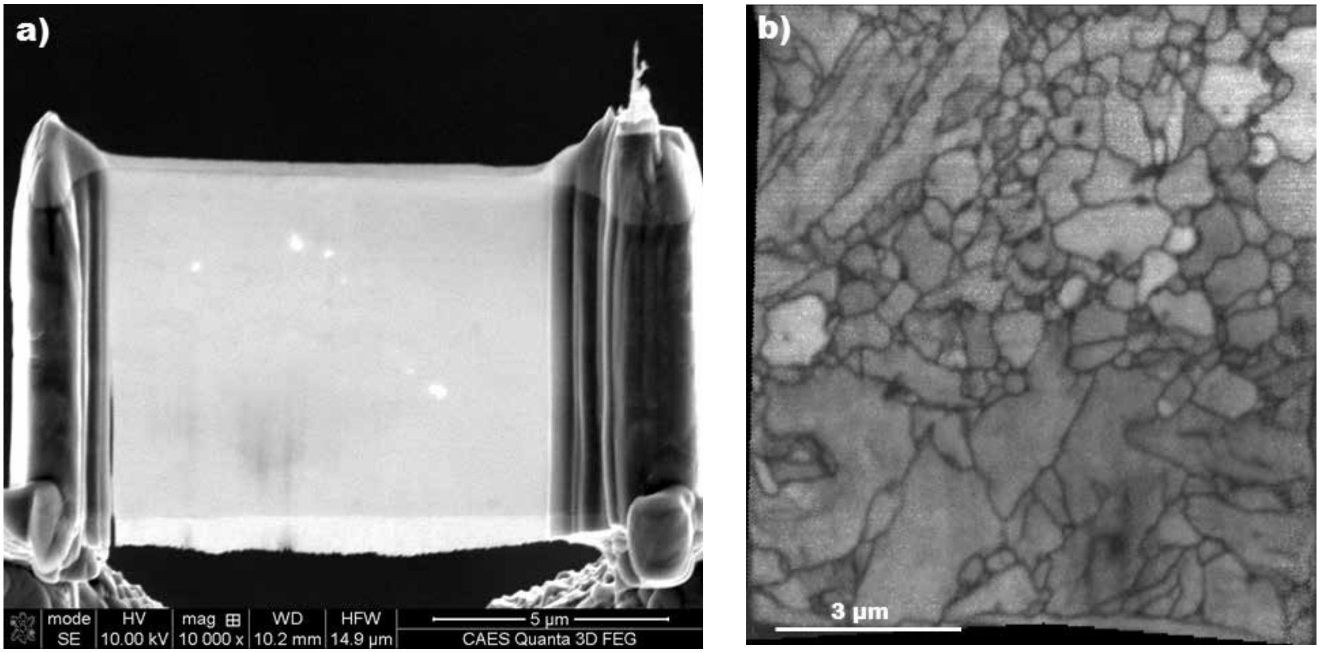

Figure 3. (a) SEM image of a TEM sample of the neutron irradiated specimen, and (b) image quality map of the same sample as obtained by t-EBSD analysis. This analysis was used to determine grain boundary character distribution of the samples.

as-received and irradiated conditions. The chemistry of very small nanoclusters was also investigated. It was found that both the Ti/Y and (Ti+Y)/O ratios decrease after radiation, going from 1.22 to 1.09 and from 1.37 to 1.29, respectively.

Nanoclusters of sizes greater than 5 nm, were identified by means of STEM Z-contrast imaging, by virtue of their differences in chemical composition with respect to the matrix (the presence of Y and Ti in these precipitates was confirmed by EDS). It was noted that, in accordance with the results obtained from LEAP analysis for the smallest precipitates, the average size decreased slightly and the number density correspondingly increased going from 7.62±2.0 nm and 2.17 x 10^{22} m^{-3} to 8.77±2.1 nm and 2.01 x 10^{23} m^{-3}, respectively, in the as-received and irradiated conditions. The very small changes in the sizes of the nanoclusters after radiation generally indicates that the nanoclusters are very stable under irradiation.

Defect analysis was also performed on the sample after irradiation. Two beam condition images close to [001], [011], and [111] zone axis were acquired and the dislocation loops identified were quantified. A typical micrograph showing dislocation loops in the neutron irradiated sample is shown in Figure 2. The analysis performed yielded an estimate defect size of 9.1 nm ± 2 nm and a density of 3.3 x 10^{21} m^{-3}.

Finally, elemental segregation across grain boundaries was performed on a few grain boundaries of the neutron irradiated sample. To reveal the nature of the grain boundary under investigation, t-EBSD maps were collected from the samples under investigation.

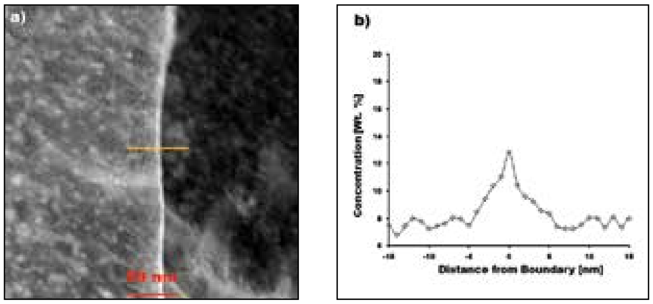

Figure 4. Segregation profile of Cr across a high angle grain boundary (θ=49°C) of the neutron irradiated specimen. (a) STEM image,(b) segregation profile obtained obtained form EDS analysis.

Figure 3 shows an SEM image of the TEM sample from the irradiated specimen along with the image quality maps obtained from t-EBSD analysis. Figure 4 shows a STEM-EDS line scan performed along a high-angle grain boundary of the irradiated specimen. This analysis shows that after radiation, high-angle grain boundaries are prone to radiation-induced segregation phenomena, with enrichment of Cr and, consequently, depletion of Fe, observed at the grain boundaries.

Future Activities

The research is completed.

Publications and Presentations

1. Mairov, A., J. He, K. Sridharan, T. Allen, 2015, "Irradiation Effects in Oxide Nanoparticle Stability in Oxide Dispersion Strengthened (ODS) Steel," *Oral presentation at the Metallurgical Society (TMS) Annual Meeting, March 15–19, 2015, Orlando, Florida*.

2. Mairov, A., J. He, K. Sridharan, 2015, "Structural Effect in Oxide Dispersion Strengthen (ODS) Steel Neutron Irradiated to 3 dpa at 500°C," *Microscopy and Microanalysis*, Vol. 21, Supplement S3, 2015 (Proceedings of Microscopy & Microanalysis 2015).

Distributed Partnership at a Glance	
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