

Atom Probe Tomography as a Tool for Characterizing Irradiated Materials

M. K. Miller*, K. F. Russell*, D. T. Hoelzer*, L. Kovarik**, M. J. Mills**, A. G. Certain**, and T. R. Allen**

* Oak Ridge National Laboratory, Oak Ridge, TN 37831

** Materials Science and Engineering, The Ohio State University, Columbus, OH 43210

*** Materials Science Program, University of Wisconsin, Madison, WI 53706

Studies of the microstructures of neutron irradiated, reactor pressure vessel (RPV) steels have demonstrated that many important changes occur on the nanometer scale. Atom probe field ion microscopy and atom probe tomography (APT) have been key techniques in the understanding of the behavior of these irradiated materials due to their high spatial resolutions. For example, in copper-containing RPV steels, APT has demonstrated that ~2-nm-diameter Cu-enriched precipitates form within the grains and along dislocations during neutron irradiation [1]. In addition, P, and to a lesser extent C, segregation to dislocations is commonly observed. In low Cu or Cu-free RPV steels, Mn-, Ni- and Si-enriched precipitates are observed at higher fluences [2]. Characterization of the size, composition, and number density of these features by APT has enabled predictions to be made of mechanical properties for evaluating plant life extension and have provided the necessary microstructural information for modeling the material's behavior.

The remarkable thermal stability and tolerance to proton, heavy ion, and neutron irradiation of nanostructured ferritic steels, such as MA957, 12YWT, and 14YWT, have also been investigated by APT. These materials are candidates for use in the next generation of reactors under extreme environments. In these nanostructured ferritic steels (NFS), extremely high number densities of 2-nm-diameter Ti-, Y- and O-enriched nanoclusters have been detected by APT both in the grain interior and on grain boundaries. Examples of nanoclusters in the 14YWT material [Fe- 15 at. % Cr, 0.6% W, 0.26% Ti, 0.13% Y, 0.19% O] annealed for 1 h at 1000 °C are shown in Figs. 1 and 2 [3]. The solute partitioning between the ferrite matrix and the nanoclusters is shown in Fig. 3. In addition to the nanoclusters, low number densities of larger μm -sized precipitates are also present, Figs. 1 and 2. These coarse precipitates are often located on the grain boundaries and triple points. The grain boundaries are also preferential sites for solute segregation of Cr and W. Solute segregation and the presence of nanoclusters on the grain boundaries effectively pin the boundaries and consequently improve the creep properties of these alloys [4,5]. APT has also demonstrated that these nanoclusters are tolerant to high doses of irradiation. An example of a 14YWT specimen that was proton irradiated to a dose of 3 displacements per atom (dpa) is shown in Fig. 4. No significant changes in the size and number density of the nanoclusters were observed after irradiation. [6]

[1] M. K. Miller and K. F. Russell, *J. Nucl. Mater.* 371 (2007) 145.

[2] M. K. Miller, et al., *J. Nucl. Mater.* 385 (2009) 615.

[3] M. K. Miller, K. F. Russell and D. T. Hoelzer, *J. Nucl. Mater.* 351 (2006) 261.

[4] J. H. Schneibel, et al., *Scripta Mater.* 61 (2009) 793.

[5] R. L. Klueh, et al., *J. Nucl. Mater.* 341 (2005) 103.

[6] This research was sponsored by the U.S. Department of Energy, Division of Materials Sciences and Engineering. Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Scientific User Facilities Division, Office of Basic Energy Sciences, U.S. Department of Energy.

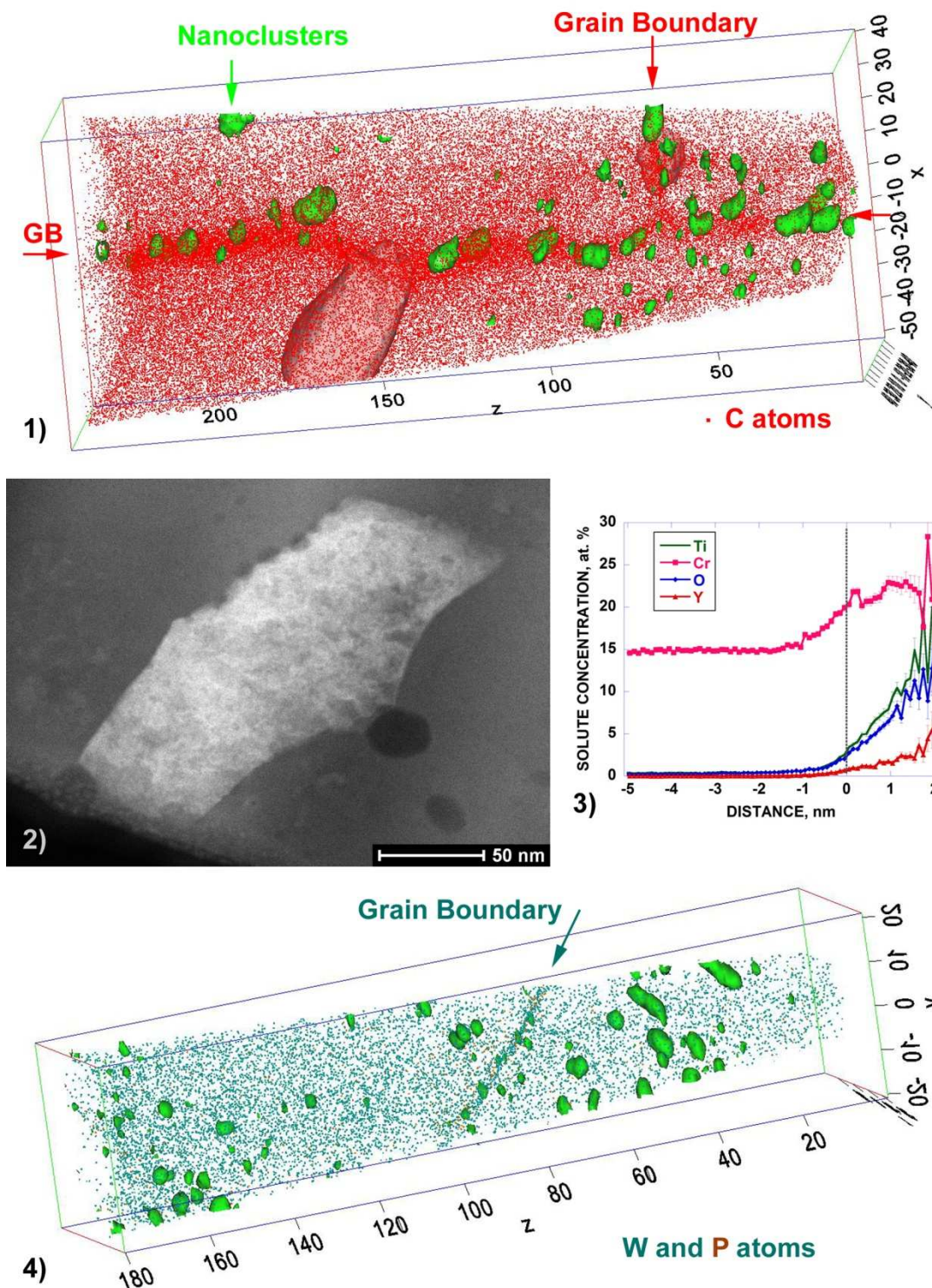


Fig. 1. Examples of nanoclusters, coarse precipitates, and grain boundary segregation in a 14YWT alloy annealed for 1h at 1000 °C. Fig. 2. TEM of a grain boundary decorated with coarse precipitates. Fig. 3. Solute partitioning between the nanoclusters and ferrite matrix. Fig. 4. The distribution of intragranular nanoclusters and W and P segregation to a grain boundary in a 14YWT alloy exposed to 3 dpa proton irradiation.