Atom Probe Tomography of the Early Stages of Phase Decomposition

Michael K. Miller

Microscopy and Microanalytical Sciences Group,
Metals and Ceramics Division,
Oak Ridge National Laboratory,
P.O. Box 2008, Oak Ridge, TN 37831-6376, USA.

The technique of atom probe tomography (APT) provides a microanalytical tool to investigate and characterize the solute distribution of an alloy at the atomic level. The x, y, and z coordinates and the mass-to-charge ratio, and hence the elemental identity, of the atoms in a small volume are determined in the mass spectrometer equipped with a position-sensitive single atom detector. The volume analyzed is typically ~10 to ~20 nm square by ~100 to ~250 nm long and contains between 10^5 to 10^6 atoms. This microanalytical technique enables the solute distribution during the early stages of decomposition to be characterized. The analytical techniques to perform these characterizations will be discussed with examples taken from a study of the embrittlement that occurs during service due to copper impurities in the pressure vessels steels used in nuclear reactors.

An atom probe tomography characterization of the copper and nickel distributions in a model Fe- 1.1 at. % Cu-1.4% Ni alloy has been performed after isothermal ageing for various extended times at low temperatures. After an initial solution treatment of 0.5 h at 1000°C, the materials were aged for 8 h at 850°C and water quenched. The specimens were then isothermally aged at 300, 400, 500 °C and times up to 10,000 h. The specimens were quenched to room temperature in water after each isothermal ageing treatment. Copper-enriched precipitates were observed at all annealing temperatures including the material annealed for 4,000 h at 300°C. These results will be compared to similar results from a neutron irradiated submerged arc weld from the HSSI fifth irradiation series (Weld 73W) that contained 0.27 at. % Cu, 0.57% Ni, and 1.58% Mn. This weld was examined after 5 conditions; after a typical stress relief treatment of 40 h at 607°C, after neutron irradiation to a fluence of 2×10^{23} n m^{-2} (E > 1 MeV), and after irradiation and isothermal anneals of 0.5, 1 and 168 h at 454°C. The matrix composition, and the size, composition and number density of the ultrafine copper-enriched precipitates that formed under neutron irradiation and the change in these parameters with post irradiation annealing treatments will be described.

This research was sponsored by the Division of Materials Sciences, U. S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp. and by the Office of Nuclear Regulatory Research, U. S. Nuclear Regulatory Commission under inter-agency agreement DOE 1886-N695-3W with the U. S. Department of Energy. This research was conducted utilizing the Shared Research Equipment (SHaRE) User Program facilities at Oak Ridge National Laboratory.