

Project Number: 152506

Project Title: Imaging and Quantification of Hydrogen Isotope Trapping in Stainless Steels

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Sponsoring IA: Nuclear Weapons

Annual Report Project Purpose

Hydrogen isotope transport through and embrittlement of pressure vessels ultimately limit component lifetime. Understanding hydrogen interactions with structural materials requires knowing not only equilibrium properties (temperature-dependent lattice solubility, diffusivity, and permeability of hydrogen), but also transient properties. Because it enhances local hydrogen concentration, defect trapping greatly reduces fracture toughness and changes deformation properties. It also makes the apparent diffusivity of hydrogen many orders of magnitude different from the equilibrium lattice diffusivity. However, trapping is typically a weak point in current simulations. Usually, only a single trapping mechanism is assumed at most, while multiple trapping mechanisms operate in real microstructures at temperatures of interest. The experimental characterization of trapping has been limited and has focused on high strength, ferritic steels. Austenitic alloys that are candidate materials and have lower equilibrium permeability values for hydrogen have not been analyzed with the same depth. While the trapping to large features, including grain boundaries and incoherent particles, has been imaged indirectly, smaller features (e.g. precipitates and solute atoms) have not been studied in detail due to fundamental resolution limitations of most analysis techniques.

The location of hydrogen isotopes will be imaged in austenitic stainless steel using local-electrode atom-probe (LEAP) tomography and trapping energies will be measured by thermal desorption spectroscopy. LEAP tomography has sub-nanometer resolution and excellent compositional sensitivity due to pulse counting techniques. Site-specific sample preparation is possible using focused-ion beam and model materials can be used to characterize trapping at low number density features. These unique capabilities of LEAP tomography make it promising for the study of hydrogen isotopes, but it has not yet been used to image trapping in austenitic stainless steels. The experimental work will be compared with first-principles calculations of the binding energy of hydrogen isotopes to solid solution elements in stainless steels and to coherent precipitate/matrix interfaces.

Annual Report Current FY Accomplishments

I have observed deuterium, for the first time, using local-electrode atom-probe tomography in stainless steel and model ultra-fine-grained aluminum specimens. Protium replaced deuterium during electropolishing in large ratios, so I've polished tips of 21-6-9 stainless steel with varying nitrogen content and have charged them with high pressure deuterium gas. I will also attempt electropolishing with deuterated acids.

I have measured deuterium trapping energies in 21-6-9 stainless steel and 304 stainless steel using thermal desorption spectroscopy.

Model samples of grain-boundary engineered nickel have been prepared by deuterium charging, characterizing grain boundaries, and focused-ion-beam milling from these boundaries. Atom-probe tomography will reveal why the fracture properties of these engineered alloys differ substantially from as-received nickel.

Annual Report Significance

A detailed understanding of hydrogen isotope transport in structural materials will help in the design and service of GTS reservoirs and materials for energy generation, storage, and transport, including fusion and other renewable energy and energy security programs. New opportunities for continuing this work through NNSA, DOE/EERE, or industrial partnerships are anticipated.