

NATIONAL NUCLEAR SECURITY ADMINISTRATION
OFFICE OF DEFENSE PROGRAMS



Tritium Focus Group 2013

Tritium Effects on Reservoir Materials

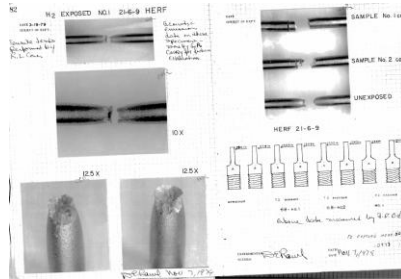


April 24, 2013

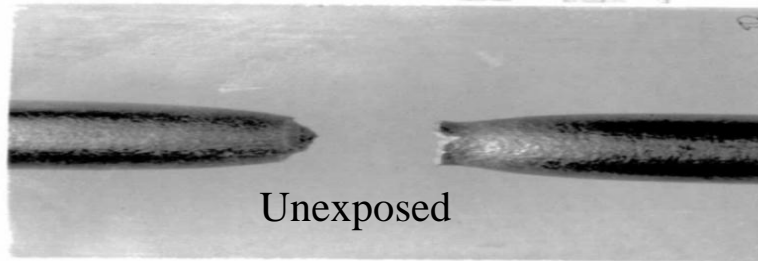
Michael Morgan
Senior Fellow Scientist
Savannah River National Laboratory



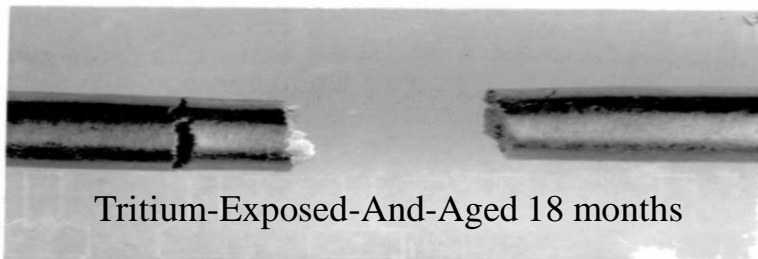
Tritium Embrittlement



Laboratory Notebook - Dave Rawl, 11/7/79



Unexposed



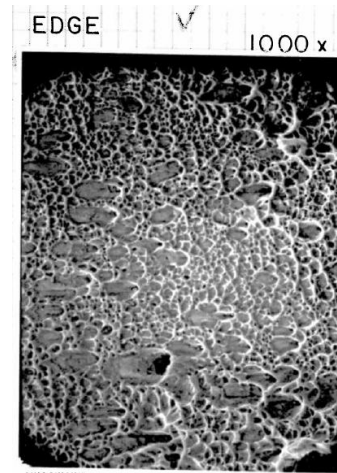
Tritium-Exposed-And-Aged 18 months



Tritium-Exposed-And-Aged 60 months

- Discovered at SRL in 1979.
- Aged tritium-exposed stainless steel samples much more embrittled than aged hydrogen-exposed samples
- Fracture occurred primarily along grain boundaries

Caused by the combined effects of hydrogen isotopes and the radioactive decay product of tritium, helium.



H2-Exposed



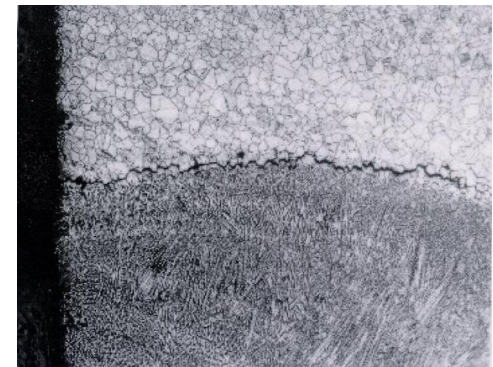
T2-Exposed

Program Background and Goals

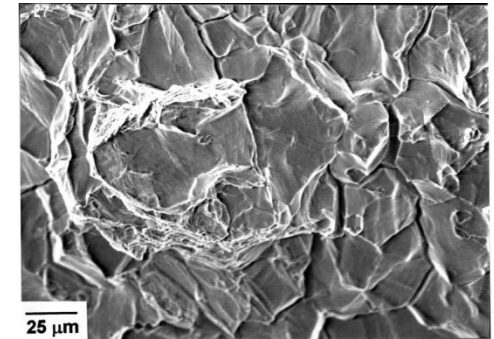
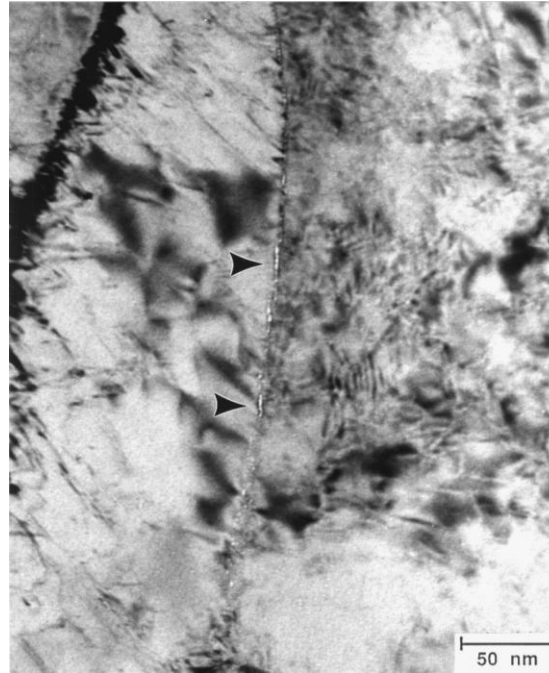
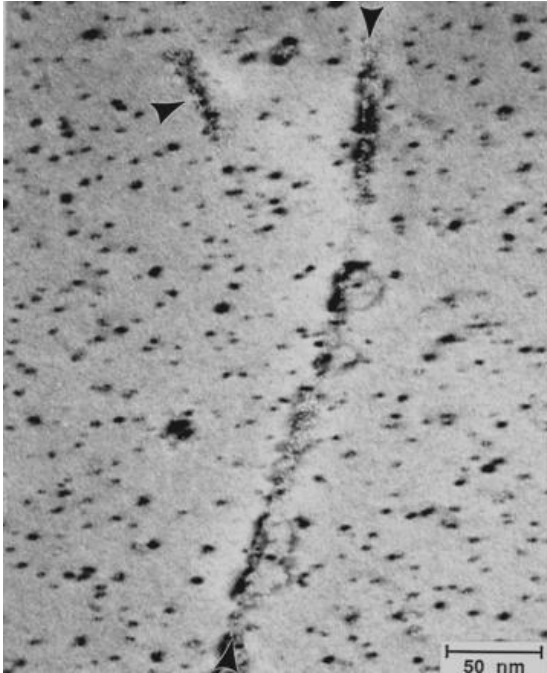
- **Tritium reservoirs are constructed from stainless steels and are used for the long-term containment of tritium gas in the weapons stockpile.**
- **Tritium and its decay product, helium-3, can cause cracking in tritium containment vessels.**
- **Tritium embrittlement phenomena is an aging phenomena and understood to be an enhanced form of hydrogen embrittlement that results over a long period of time from the development of a helium-hardened microstructure.**
- **The chief goal of the program is to measure those critical properties that are needed for structural integrity analysis and modeling.**

Active Programs

- **Measure Properties of Actual Reservoir Forgings and Microstructures.**
- **Characterize Effect of Forging Process on Tritium Compatibility to Allow More Flexibility on Manufacturing Options.**
- **Measure Weldment and Heat-Affected Zone Properties.**
- **Future Efforts Should Concentrate on Properties of Aged Steels in High Pressure Hydrogen Environments.**
- **Continue to Provide Data for Crack Nucleation and Growth Modeling.**



Tritium Embrittlement Process

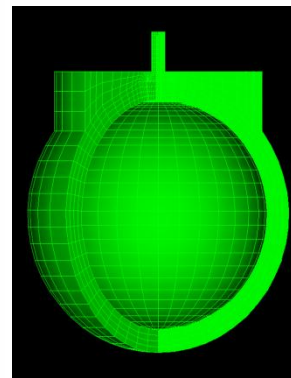
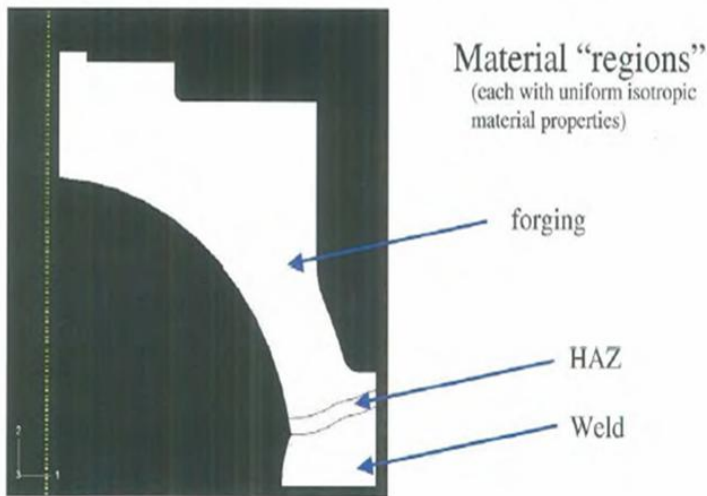
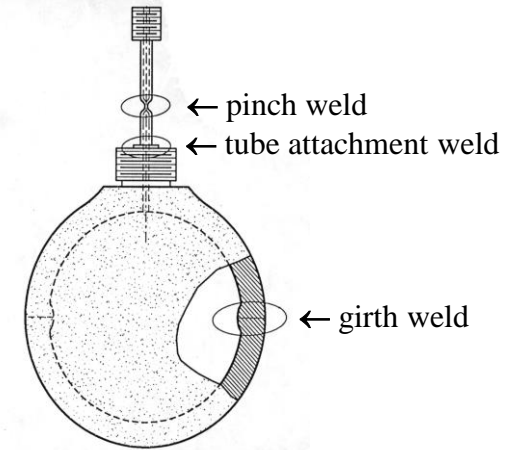


Decay helium precipitate as nanometer-sized bubbles that harden the microstructure

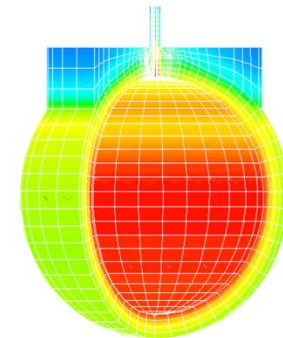
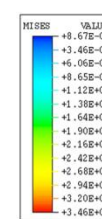
Embrittlement and Cracking of Grain Boundaries

Fracture Mechanics Approach

Design and evaluate tritium reservoirs such that any manufacturing defect cannot develop into a crack that could grow and cause the vessel to release its contents.



Reservoir Model

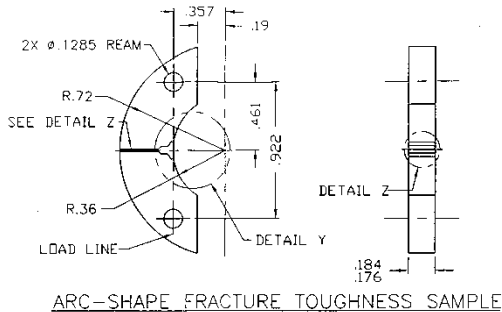


Stress Distribution

Experimental Procedure



Fabricate samples
From Forgings

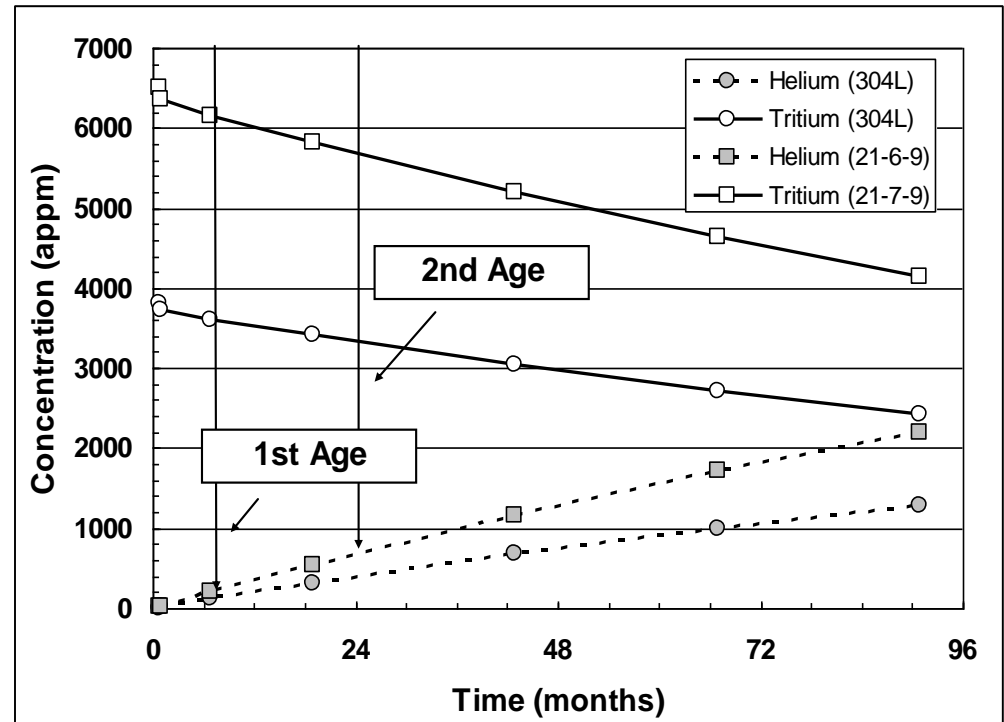
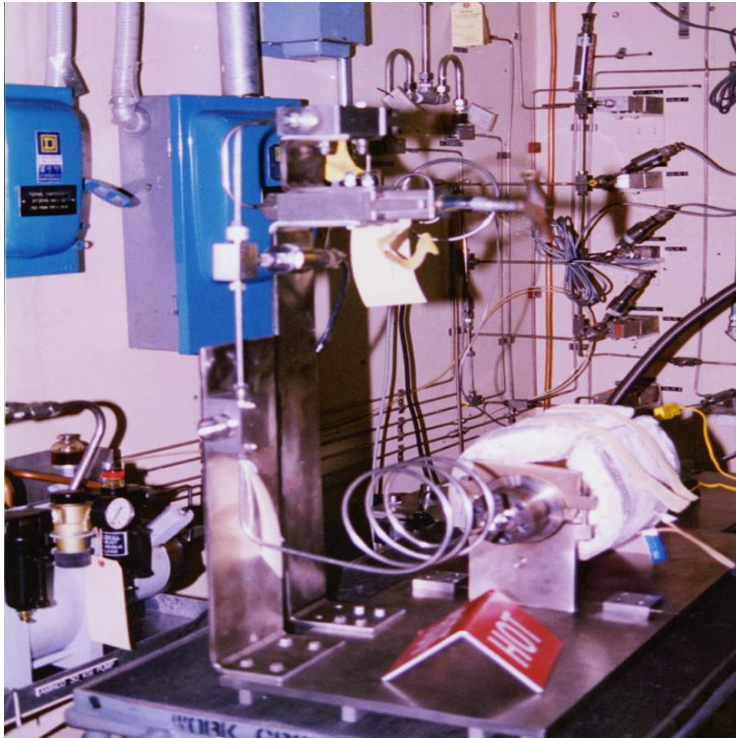


Hydrogen/Tritium
Exposure at 350C
and 5000 psi – Age
at -50 C to Build-in
Helium W/O Losing
Tritium



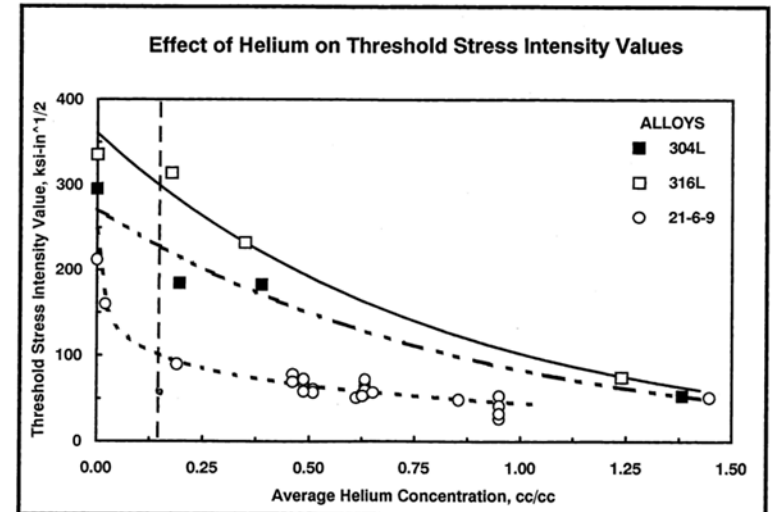
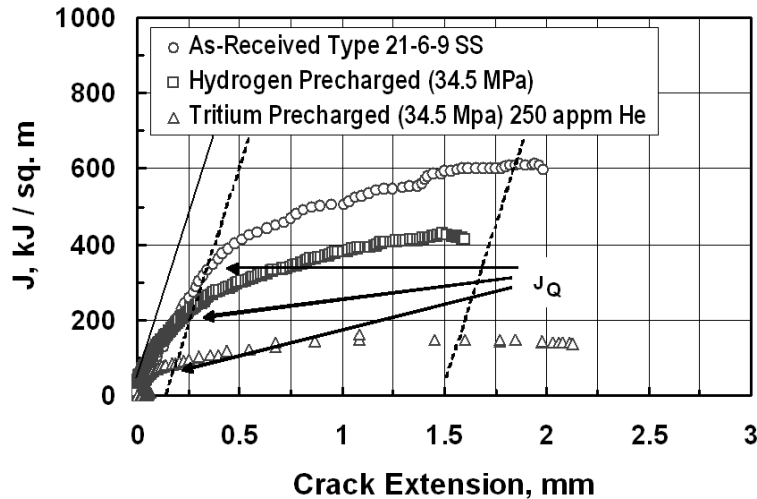
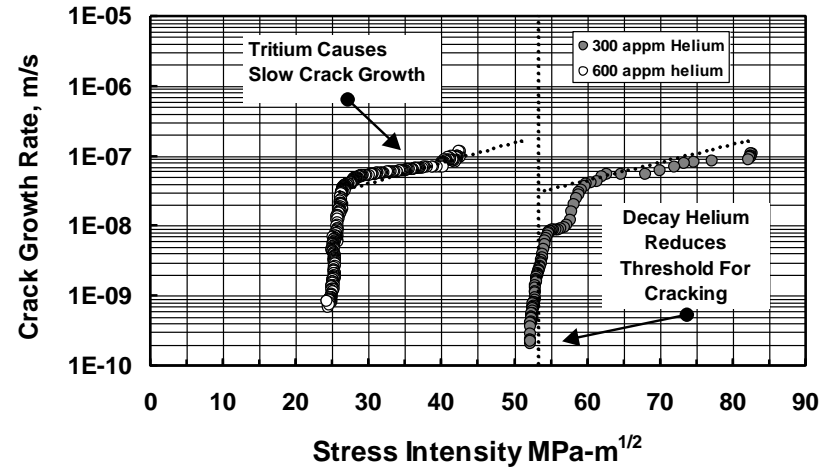
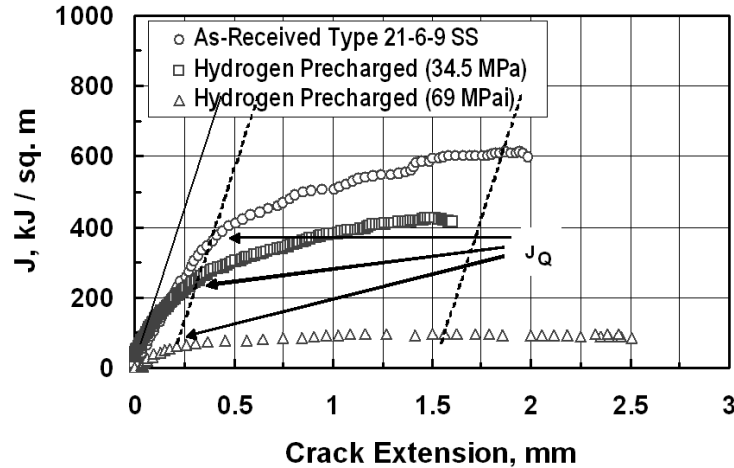
Test In Hood For
Containment of
Tritium Off-gassing

Tritium Exposures and Aging

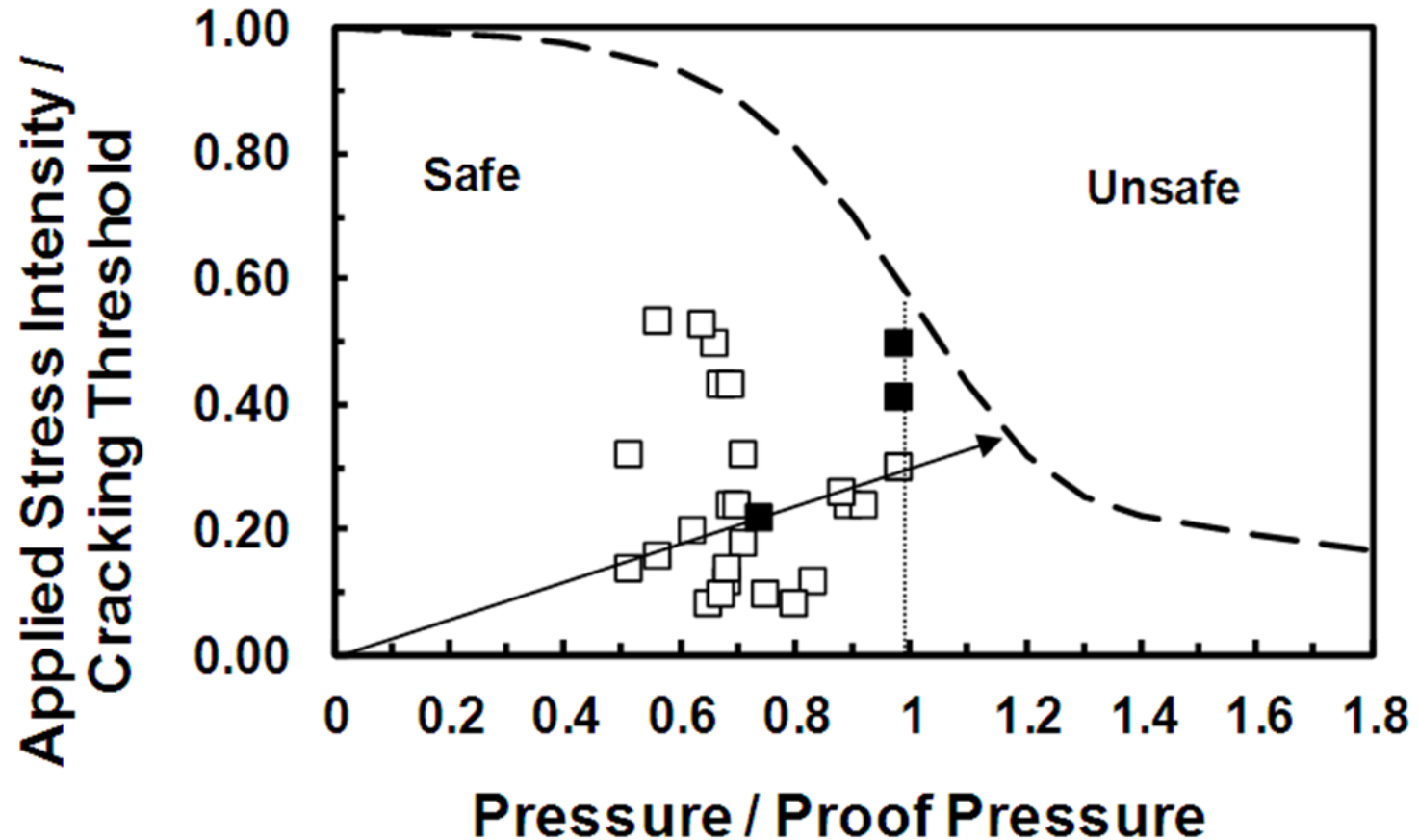


- Typically expose at 35 MPa and 350°C.
- Age to ~1000 appm He.
- 350°C is high enough for to saturate samples with tritium but low enough to minimize any change in microstructure.
- Aging at low temperature minimizes off-gassing losses.

Typical Effects of Hydrogen, Tritium, and Decay Helium on Fracture Toughness Properties



Failure Assessment Approach for Pressure Vessels Requires Fracture Mechanics Data



Characterize Tritium Compatibility of Reservoir Forgings

Characterize Tritium Effects on Steels Forged Using Mechanical, Screw, Hydraulic and High-Energy-Rate Forging Processes

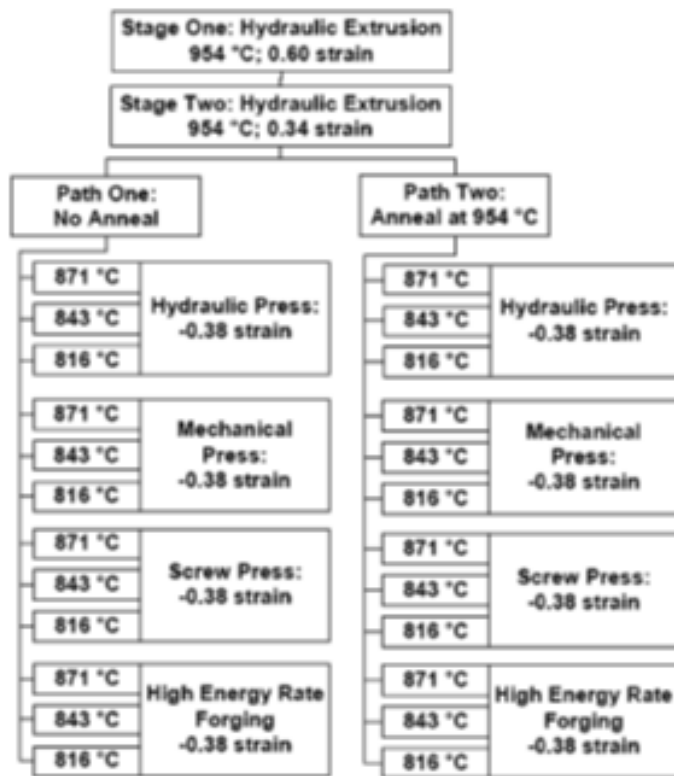


Fig. 1. Flowchart matrix for 304L stainless steel processing experiments.

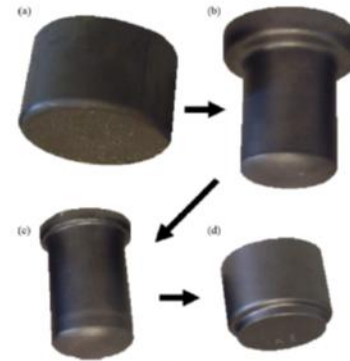


Fig. 3. Product of each step for 304L stainless steel: (a) billet prepared for first extrusion, (b) after first extrusion, (c) after second extrusion, and (d) after final forging.

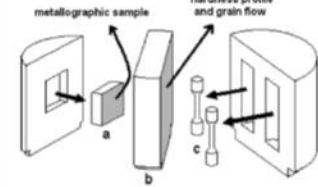
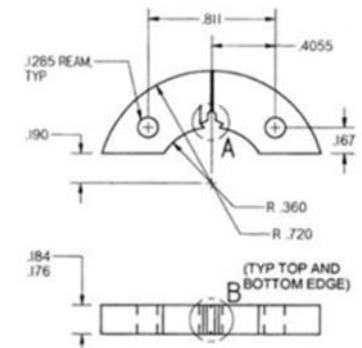
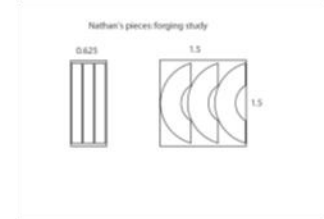


Fig. 4. Exploded view of final forging shape, showing the locations of each test sample: (a) metallographic imaging sample, (b) hardness mapping sample, and (c) tensile test samples.



Project Y642 Enhanced Fracture Toughness Facility

Fracture Toughness Information Used in GTS Design

SRNL is seeking to establish an enhanced mechanical testing capability in 774-A. It will be used for fracture toughness testing of tritium exposed-and-aged stainless steel samples in high pressure hydrogen gas, for the Enhanced Surveillance Campaign. This will support reservoir integrity and lifetime assessment activities with the Gas Transfer System Design Agencies (LANL & SNL). Fracture toughness is a critical material property that quantifies a material's resistance to crack propagation. Current testing involves the tritium charging and aging of fracture samples, followed by testing these tritiated samples in air. SRS has thus obtained valuable data on the materials and manufacturing methods used for current and potential future reservoirs. Such data is needed to apply modern design and aging assessments using fracture mechanics and the application of current codes and standards as required by law



Current Techniques May Yield Non-Conservative Results

Analysis of data needs for reservoir behavior models within the NSE indicates that fracture mechanics data obtained from tests in air may be non-conservative, and do not adequately represent service conditions for reservoirs. Current test samples contain residual tritium and helium from tritium decay in the microstructure. Helium is known to accentuate hydrogen embrittlement. However, the residual tritium is not sufficient to represent the hydrogen/tritium gas environment for slow crack growth in reservoirs. It is further believed that a "high" pressure gas environment is best. Experiments are

needed to confirm this belief and establish the appropriate test conditions.

Establish Unique Capability

This capital project seeks to modify a cell in the existing Fracture Toughness Tester Facility in building 774-A for testing tritiated samples in high pressure hydrogen. The cell was designed for this type of work (clean samples in hydrogen) and is constructed with steel plate walls and ceiling for explosion protection, hydrogen monitoring, and remote operation. The project will upgrade the facility for handling tritiated samples. Installation of radiological hoods and enclosures, new test equipment, a high pressure test vessel, and the associated pumps and piping will be required. This will establish a unique capability in the NSE here at SRS.



Funding Needed

SRS completed a conceptual design, formal cost estimate, and project schedule for this facility in FY2011. The Cost estimate is \$6.6M. The project will take up to 24 months of effort to complete long-lead procurements, lab



Crack Tip Depletion From Off-Gassing Losses

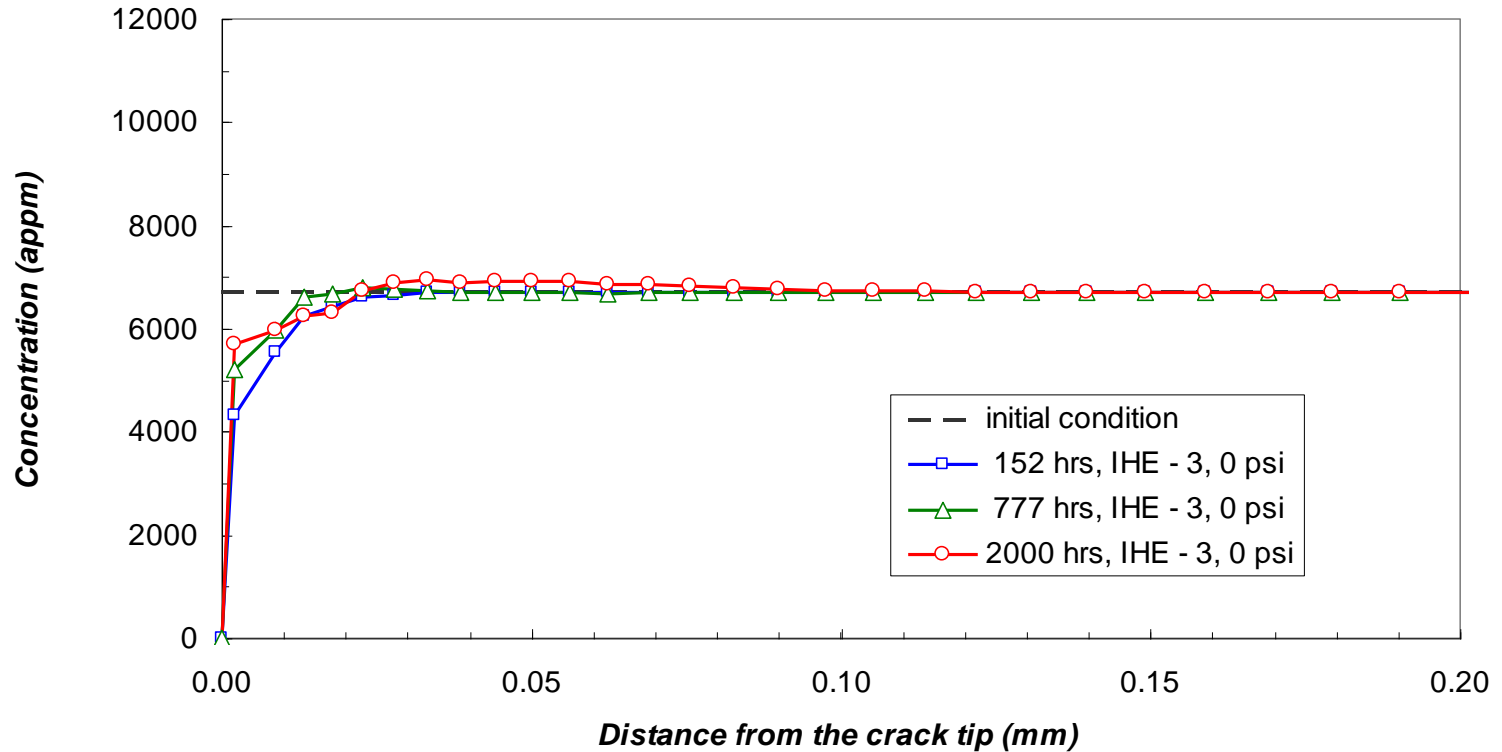


Fig.6 Distribution of hydrogen concentration under 0 psi with IHE (IHE -3)

Crack Tip Enhancement From External Source (5000 psi)

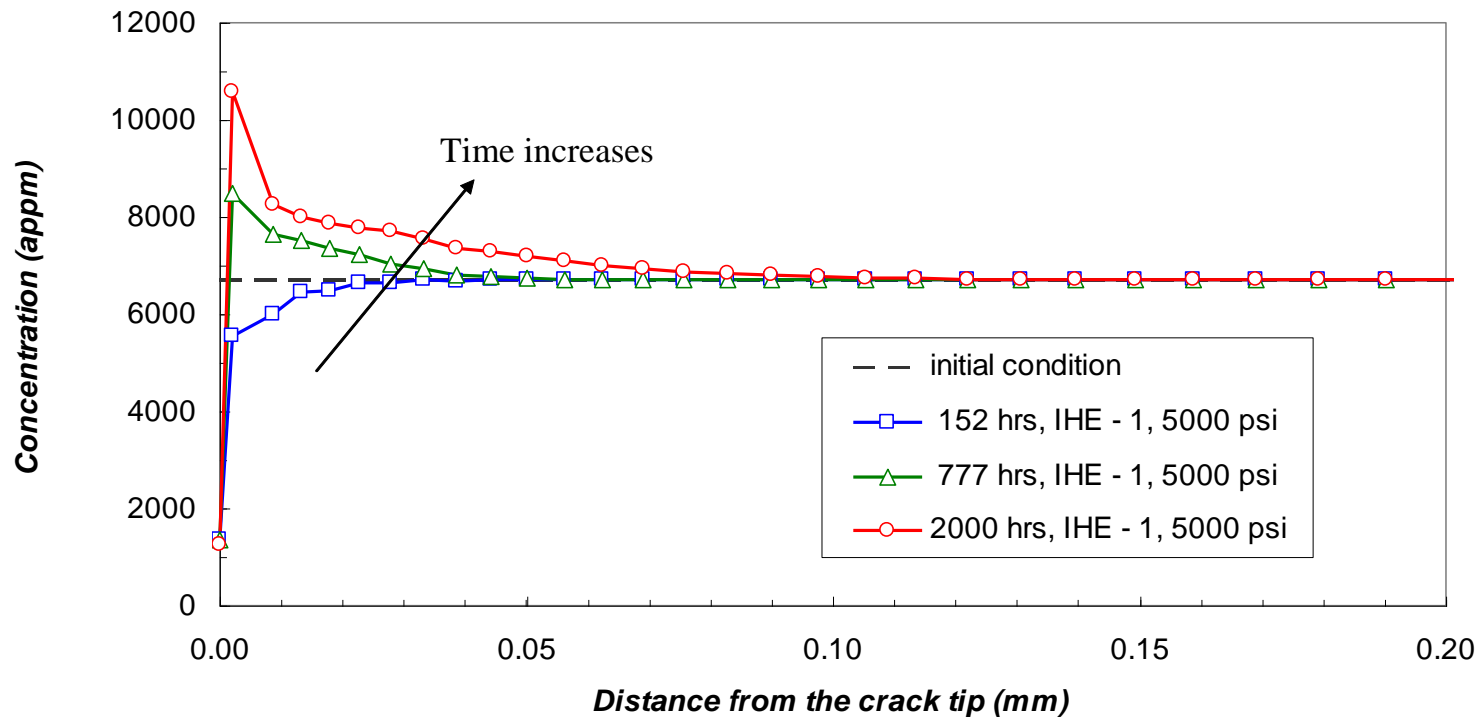
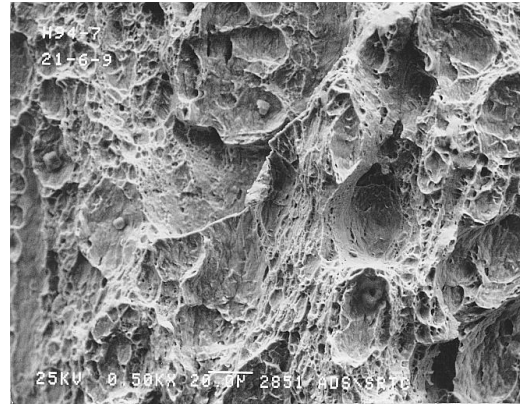


Fig.8 Distribution of hydrogen concentration under 5000 psi (340 atm) with IHE (IHE – 1)

Cracking Behavior For Stainless Steel Tested in High-Pressure Hydrogen Gas

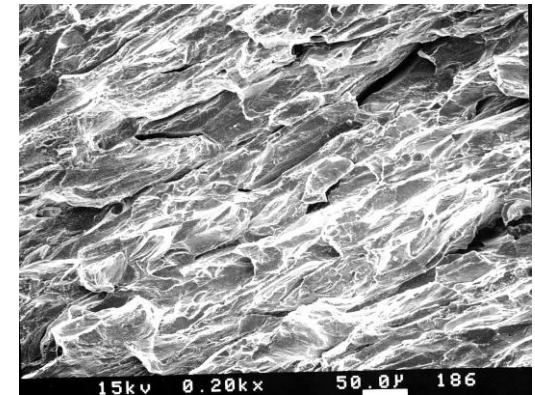


Hydrogen or Tritium Pre-charged Tested in Air

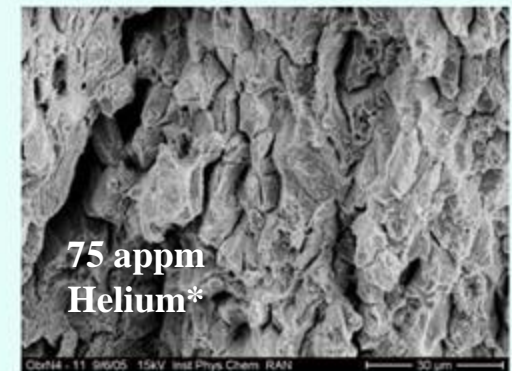
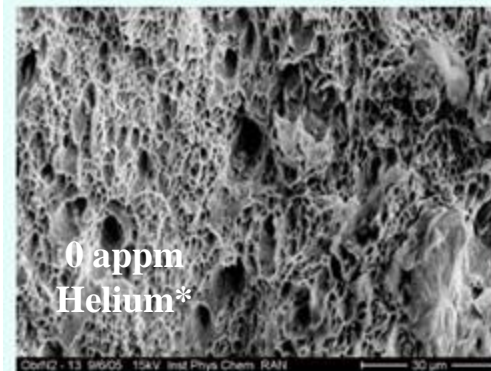


High Toughness - Ductile Rupture

Tested in High-Pressure Hydrogen



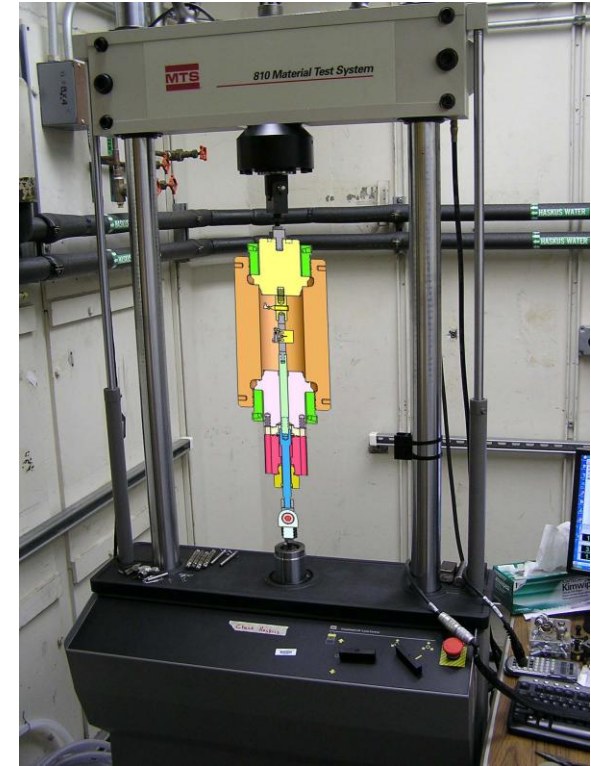
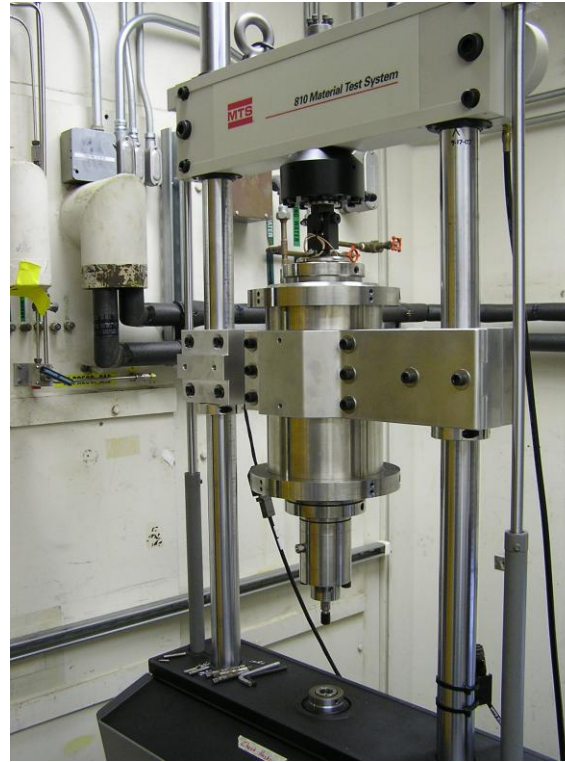
Low Toughness-Brittle Intergranular Fracture



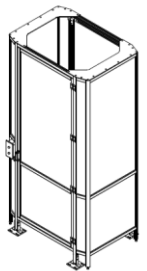
*Boitsov, Kanashenko, Causey, Tritium Science and Technology Conference, Rochester, NY 2007 in the Russian equivalent of Type 321 Stainless Steel

Testing in High-Pressure Hydrogen Environments

Until now, tritium effects have been investigated by measuring properties of pre-charged samples tested in air.

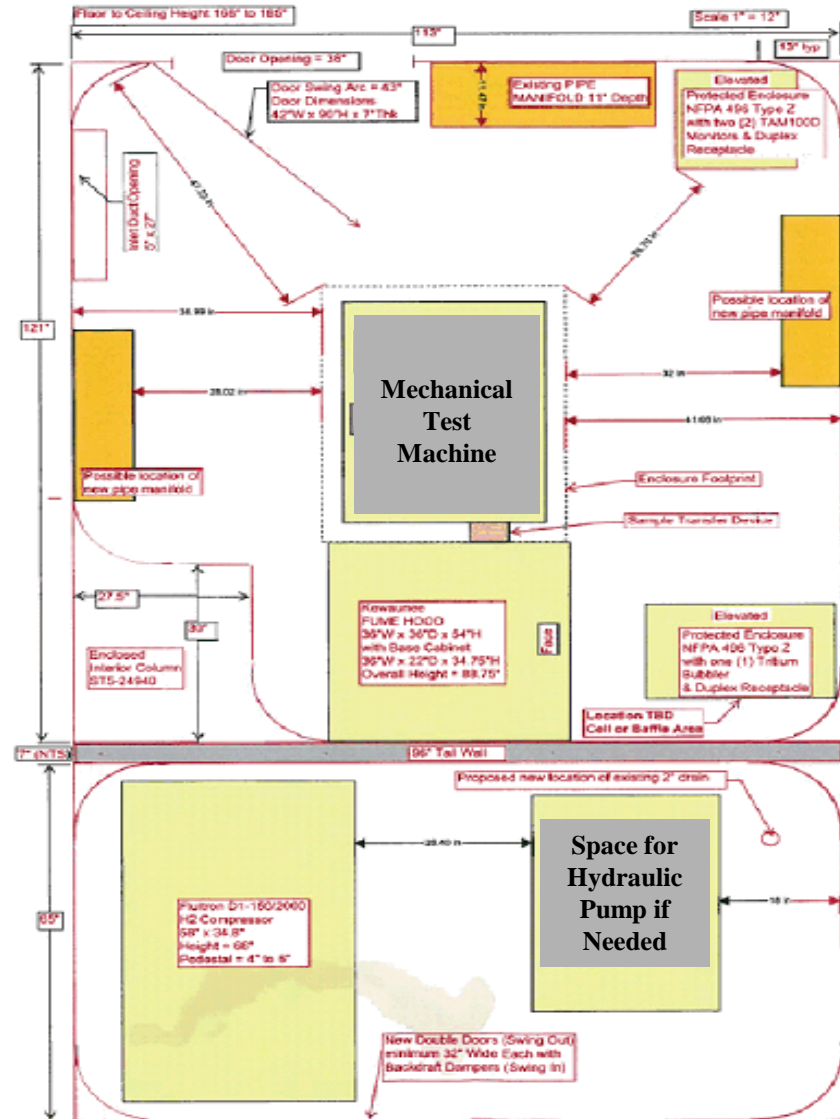


Design and construct a laboratory so that the properties of tritium-charged-and-aged samples can be measured in high-pressure hydrogen environments.



Conceptual Design-Modify Existing Laboratory

- Remote operation from control room outside cell
- Tritium hood, testing machine, high-pressure vessel, and hydraulic pump will be located within cell
- Includes tritium monitoring and confinement and manifolds for automatic control valves and pumping high pressure hydrogen gas into vessel on mechanical test machine.



Summary

- The purpose of the Tritium Effects on Materials program is to provide fracture mechanics data on tritium-exposed steels to the Design Agencies for modeling and evaluation of tritium reservoirs;
- Until now, these properties have been obtained using hydrogen and tritium pre-charged samples from forgings tested in air;
- Current programs are designed to measure tritium and decay helium effects on actual reservoir forgings;
- Programs include three steel types, four forging processes, multiple yield strengths, weldments and plans for HAZ.
- Future programs will characterize tritium effects in high-pressure gas environments.