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EXTRACTION OF FRACTURE-MECHANICS AND TRANSMISSION-ELECTRON-MICROSCOPY SAMPLES FROM TRITIUM-EXPOSED RESERVOIRS USING ELECTRIC-DISCHARGE MACHINING

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Extraction of Fracture-Mechanics and Transmission-Electron-Microscopy Samples from Tritium-Exposed Reservoirs using Electric-Discharge Machining

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1.0 SUMMARY

The Enhanced Surveillance Campaign is funding a program to investigate tritium aging effects on the structural properties of tritium reservoir steels. The program is designed to investigate how the structural properties of reservoir steels change during tritium service and to examine the role of microstructure and reservoir manufacturing on tritium compatibility. New surveillance tests are also being developed that can better gauge the long-term effects of tritium and its radioactive decay product, helium-3, on the properties of reservoir steels. In order to conduct these investigations, three types of samples are needed from returned reservoirs: tensile, fracture mechanics, and transmission-electron microscopy (TEM). An earlier report demonstrated how the electric-discharge machining (EDM) technique can be used for cutting tensile samples from serial sections of a 3T reservoir and how yield strength, ultimate strength and elongation could be measured from those samples. In this report, EDM was used successfully to section sub-sized fracture-mechanics samples from the inner and outer walls of a 3T reservoir and TEM samples from serial sections of a 1M reservoir. This report fulfills the requirements for the FY06 Level 3 milestone, TSR 15.1 "Cut Fracture-Mechanics Samples from Tritium-Exposed Reservoir" and TSR 15.2 "Cut Transmission-electron-microscopy foils from Tritium-Exposed Reservoir" for the Enhance Surveillance Campaign (ESC). This was in support of ESC L2-1870 Milestone- "Provide aging and lifetime assessments of selected components and materials for multiple enduring stockpile systems".

2.0 BACKGROUND

Tritium and its decay product, helium-3, change the structural properties of stainless steels. Tritium-exposed steels generally exhibit higher yield strength, lower elongation and ductility, lower fracture toughness, and increased susceptibility to crack growth under sustained loads. The degree of property change depends on the tritium exposure history, because it results from the dissolution and diffusion of tritium into the steel and its radioactive decay to helium. Most of the quantitative data that is used to characterize the effects of tritium has been collected from long-term storage and burst tests of accelerated aged reservoirs. Indirect measurements using tensile and fracture toughness tests on samples exposed to tritium and aged prior to testing have also been used. Invariably, questions arise as to the exact condition of a particular reservoir because its tritium exposure history and material processing history is different from those used in simulated tests.

Today, modern numerical computational techniques like those used in the Enhanced Surveillance Campaign (ESC) are available to better predict reservoir aging and performance. However, these tools require accurate material property data. Until now, direct measurements of the effects of tritium on the properties of reservoir material were exceedingly difficult because the affected material is in the near-surface regions of the reservoir walls. The material properties in that region are highly dependent on reservoir geometry and the steep concentration gradients of tritium and helium. Techniques are needed to directly measure the effects of tritium on the properties of returned reservoirs for complete structural integrity assessments.

With the use of an Electric-Discharge Machine (EDM) very small samples can be sectioned from precise locations within components. Two previous Enhanced Surveillance Campaign programs (FY2004 and FY2005) demonstrated that sub-sized tensile specimens could be sectioned from components without adversely affecting mechanical properties [1, 2]. With this novel capability, characterization, including mechanical properties and microstructural information, of the thin tritium-exposed regions is now possible. In order to accurately assess the condition of the tritium-exposed regions and measure the cracking susceptibility of the material, new, more compact types of fracture toughness specimens will have to be developed. Dimensions and geometry of these samples will be dictated by the component size, shape, and depth of the tritium exposed material. Data obtained from the sub-sized fracture-mechanics specimens will complement existing shelf storage and burst test data for the Life Storage Program (LSP) and help answer questions about a specific reservoir's processing history and mechanical properties. Furthermore, TEM examinations are needed to reveal decay helium bubble morphology and forging microstructures.

The purpose of this current work is to demonstrate that sub-sized fracture toughness specimens can be sectioned from within the tritium exposed material. Additionally, this work will show that thin samples (< 0.015 in thick), which will be used to produce transmission-electron-microscopy foils, can be serial sectioned from near defects, or other points of interest, within the tritium-exposed material. The work described below was performed under Enhanced Surveillance Campaign Tasks 15.1 and 15.2 for ESC L2-1870 [3].

3.0 DESCRIPTION OF SPECIMENS

3.1 Fracture-Mechanics Samples

New surveillance tests are being developed that can better gauge the long-term effects of tritium and its radioactive decay product, helium-3, on the properties of reservoir steels. The use of Type 316L stainless steel as a reservoir material of construction is relatively new, and therefore, little performance data in tritium service is available. In order to conduct comprehensive tritium aging studies, three types of samples sectioned from returned reservoirs are needed: tensile, fracture mechanics, and microscopy. Tensile specimens were successfully sectioned from a tritium-exposed reservoir in FY2005 [2]. The current work focused on design of sub-sized fracture-mechanics specimens in which the configuration and size were dictated by the reservoir geometries. Initially, non-radioactive material was used to evaluate the three different specimen designs. The various designs included a double edge notch, single edge notch, and modified grip single edge notch test specimens (Figure 1). Preliminary testing on the unexposed samples indicated that the double-edge-notched configuration would be the best fracture-mechanics sample configuration for existing tritium-exposed thin-materials-testing capabilities. The other two sample configurations were incompatible with the wedge grips that were available for tritium materials testing. Therefore, the double-edge notched specimen was selected for the current demonstration, though the other configurations could be machined from the reservoir in the future if needed. Test results for the three fracture-mechanics sample configurations will be documented in a future report.

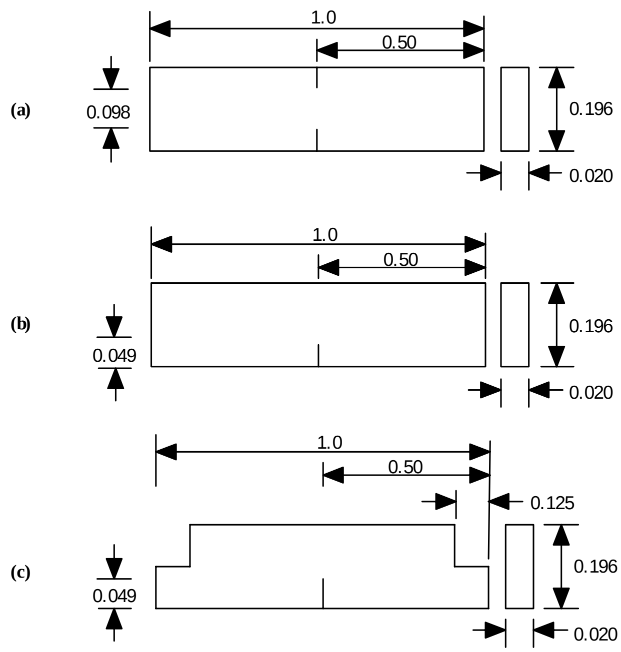


Figure 1. Dimensions (inches) of candidate fracture-mechanics specimens, a) double edge notch, b) single edge notch, and c) modified grip single edge notch test specimens.

3.2 Transmission-Electron-Microscopy Foils

Transmission-electron microscopy is needed for microstructural characterization of tritium exposed hardware. The technique is used to reveal differences in the decay helium bubble microstructures and the underlying forging microstructure. These differences are often related to changes in fracture properties of the stainless steels, e.g., Type 316L and 21-6-9. In order to perform these microstructural examinations, thin foils are needed from specific regions such as weld heat-affected zones, base metal, and tritium-exposed regions near defects. These foils must be thin enough for electron transmission and are described in Section 5.2.

4.0 EDM CONFIGURATION

The Brother HS-70 is a submerged type machine in which the part remains under water during the cutting operation (Figure 2). This minimizes splashing and the potential for operator contamination. Numerous modifications were necessary prior to beginning radiological operations. The most notable was the installation of an exhaust plenum. The exhaust system was designed to contain and remove tritium released via reservoir off-gassing and tritium evaporation from the deionized water.

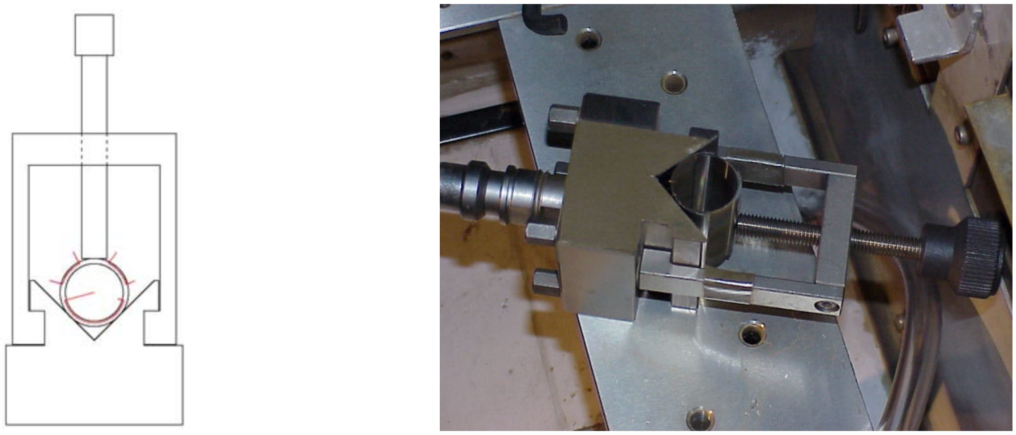
Figure 2. Brother HS-70A submerged type wire EDM with exhaust plenum.

A glove bag was also fabricated to enclose the 100 gallon water reservoir. A Femto-TECH® tritium monitor was permanently installed in the room to monitor the breathing zone around the EDM water bath, the water reservoir, and the wire collection area. Portable tritium monitors were also used by Radiological Control Operations (RCO) personnel to more closely monitor the zone above the plenum during sample transfer between the hood and the EDM and other locations in the laboratory, as required. All work was performed under a Hazard Analysis Package, including a detailed Job Hazards Analysis [4].

5.0 EDM SECTIONING

5.1 Fracture-Mechanics Specimens

An approximately 1.25 inch long portion of a 3T reservoir serial number was mounted in the “V” vice and placed in the EDM for sectioning. The specific EDM program code necessary to perform the cutting operation was written using the Computer Aided Design software, BobWire™. Once the desired cut path was drawn, it was converted to code for the EDM (Figure 3a). The goal of the initial cut was to section enough material circumferentially (0.020 inches thick) from the inner and outer walls to allow fabrication of several sub-sized fracture-mechanics specimens. To minimize the number of entries into the drained water bath, the EDM was programmed to sequentially cut the two small pieces from the outer diameter and the one larger piece from the inner diameter (Figure 3a). Figure 3b shows the surrogate part secured in the “V” vise after completion of the initial cutting process. The pieces were then secured in a set of modified vice grips that were mounted directly to the EDM table (Figure 4). Sub-sized fracture-mechanics specimens were then cut using a second EDM program. Dimensions of the sub-sized double edge notch fracture-mechanics specimen are shown in Figure 1a. Pieces sectioned from the surrogate hardware, including a final sub-size fracture mechanics-specimen, are shown in Figure 5.



(a) (b)

Figure 3. Sketch showing wire cutting path (red) and material sections cut from the OD and ID of the surrogate hardware, 1/2” SCH 5 pipe, (a) and the vise securing surrogate hardware (b).

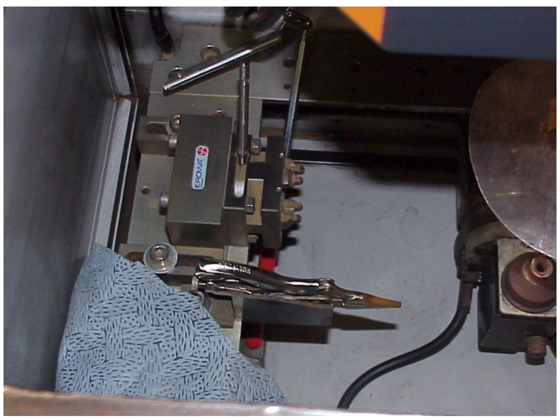


Figure 4. Modified vice grips used to secure small pieces to the EDM machining table.

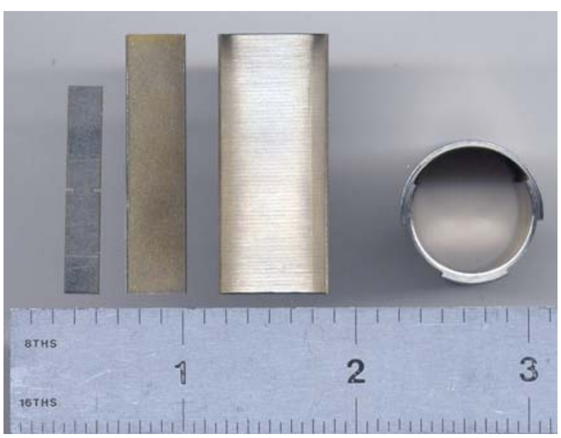


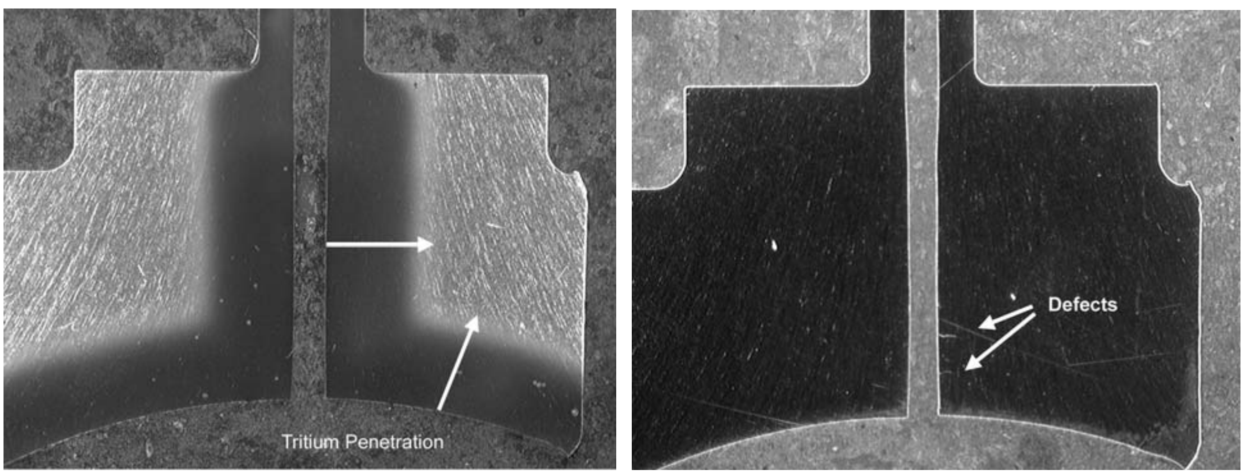
Figure 5. Photograph showing surrogate hardware (1/2” SCH 5 pipe), material sections from the ID and OD, and double edge notch fracture-mechanics specimen.

During this cutting operation, the tritium concentration level in the air around the EDM was monitored continuously. Air monitoring indicated that tritium concentration in this area did not exceed 5 µCi/m^3. The background reading before the cutting operation began was < 1 µCi/m^3. The suspension limit per the Radiation Work Permit was 40 µCi/m^3.

During the cutting operation, several wire breakages occurred at several locations. The first wire break occurred when the wire initially contacted the outer surface. This situation may have resulted due to the presence of a nonconductive layer, e.g., foreign material or surface oxidation, on the outer surface of the part. Several other wire breaks occurred while the wire was well into the cut. These breaks may have been caused by the movement of the part as internal residual stresses were relieved. This may have caused the thickness variation observed in several of the samples. Wire breaks were not observed when cutting the surrogate material, 1/2” SCH 5 annealed pipe.

5.2 Microscopy Specimens (TEM)

A metallurgical specimen prepared from a portion of a 1M reservoir containing several internal defects was serially sectioned using the EDM. Photographs of the metallurgically mounted



(a) (b)

Figure 6. Micrographs of a section from 1M reservoir showing a) depth of tritium penetration and b) defects around fill bore.

specimen are shown in Figure 6. In order to section this part, the hot mounting material (phenolic resin) had to be removed. This operation was performed in the Tritium Facility. One half of the part was serially sectioned from the outer diameter to the edge of the fill stem. Seven pieces were removed, and are shown in Figure 7. Except for the outermost section, which was intentionally cut thicker, all sections were nominally 0.011 inches thick. The various sections contained material from both high and low tritium-exposed regions. Section number 6 contained the defects shown in Figure 6b. During this EDM cutting operation, the tritium concentration level in the air around the EDM was monitored continuously. Tritium concentration in breathing air in the room did not exceed 5 µCi/m^3.

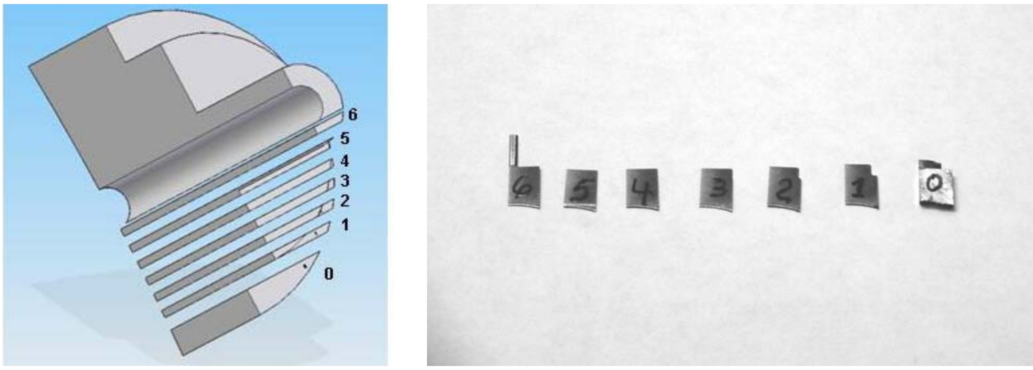


Figure 7. Schematic showing orientation of sections (not to scale) and EDM samples cut from 1M reservoir metallurgical sample.

Microstructures in the region(s) of interest, including high and low tritium-exposed regions and regions adjacent to the defects, will be characterized using TEM (Figure 8). TEM samples will be prepared from the six thin slices sectioned from the reservoir. Three to four, 3 mm diameter disks will be punched from each slice. Thin foils samples suitable for examination in the TEM will be prepared from these disks using a jet-polishing technique. The electrolyte used will be a 57% methanol, 39% butylcellosolve, and 4% perchloric acid solution at an applied voltage of 35 V DC and 243 K. All microscopy will be performed using a JEOL 2010 operating at 200 kV.

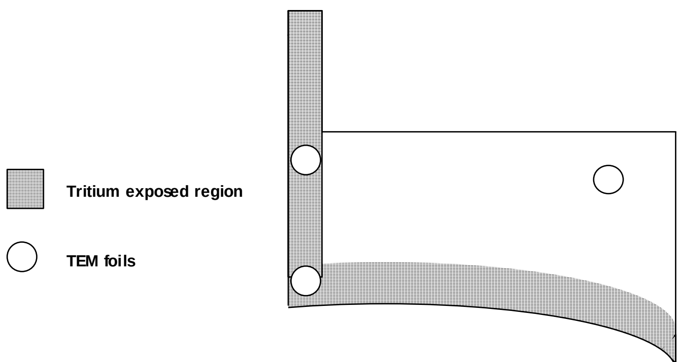


Figure 8. Schematic showing location of TEM foils cut from high and low tritium-exposed regions (not to scale).

6.0 RESULTS AND DISCUSSION

The EDM was used successfully to section sub-sized fracture-mechanics samples from the inner and outer walls of a 3T reservoir and TEM samples from serial sections of a 1M reservoir. The fabrication of these specimens will allow the investigation of the long-term effects of tritium and its radioactive decay product, helium-3 on structural properties and microstructure of reservoir steels.

This work demonstrated the capability of the EDM to safely remove very small pieces from precise locations within tritium-exposed components. The ability to remove only the material necessary reduces operator exposure and tritium releases to the environment. The local exhaust surrounding the EDM, which contained the tritium as it evolved during the cutting operation, and Hazard Analysis Package, including detailed procedures and JHA, ensured the safe handling and cutting of tritium-exposed components. This work also demonstrated its ability to perform serial sectioning with a minimal amount of wasted material. In addition to providing TEM foils, there is enough material in each of the individual sections to perform additional evaluations, such as optical metallography or tritium/helium-3 analyses.

Wire breaks occurred several times during the sectioning of the 3T reservoir. Surface contamination and/or oxidation and residual stresses may have caused the breaks. Although the wire breaks slowed the cutting operation, the automatic rethreading feature of the Brother HS70-A allowed the cutting to proceed without significant operator intervention. The EDM has demonstrated a significant advantage over conventional cutting techniques and should be considered for future surveillance activities.

This report fulfills the requirements for the FY06 Level 3 milestone, TSR 15.1 “Cut Fracture-Mechanics Samples from Tritium-Exposed Reservoir” and TSR 15.2 “Cut Transmission-electron microscopy foils from Tritium-Exposed Reservoir” for the ESC. This was in support of ESC L2-1870 Milestone-“Provide aging and lifetime assessments of selected components and materials for multiple enduring stockpile systems”.

7.0 CONCLUSIONS

The following are conclusions based on this work:

1) Electric-discharge machining is a viable technique for sectioning and extracting from tritium-exposed hardware fracture-mechanics and TEM samples.

2) The electric-discharge-machining equipment and sectioning techniques developed here can be used to improve routine sectioning of tritium-exposed hardware in existing Savannah River reservoir surveillance activities..

3) The results demonstrated that the EDM tritium confinement system and the hazard analysis packages developed during the course of this work ensure the safe handling and sectioning of tritium-exposed hardware in SRNL facilities.

8.0 FUTURE WORK

Future work should include the following activities:

1) Investigate the effect of EDM on the mechanical properties, morphology, and tritium/helium-3 profiles.

2) Evaluate the cause of wire breaks and develop mitigation strategies.

3) Machine mechanical test specimens and TEM samples from weldments, heat-affected zones, and base metal of tritium-exposed reservoir materials, especially Type 316 stainless steel.

4) Continue to develop techniques for cutting fracture toughness samples from tritium exposed components.

5) Develop EDM practices for enhanced failure analysis through serial metallography.

9.0 REFERENCES

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10.0 ACKNOWLEDGMENTS

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