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Microstructural Features Affecting Properties and Aging of Tritium-Exposed Austenitic Stainless Steels

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1 EXECUTIVE SUMMARY

A project to implement a life-cycle engineering approach to tritium reservoirs has been initiated through the DOE - Technology Investment Projects. The first task in the project was to develop a comprehensive list of microstructural features that impact the aging performance of the tritium reservoirs. Each of the participating sites (SRNL, SNL, LANL, KCP) independently developed a list of features deemed integral to tritium reservoir performance based upon operational and design experience. An integrated list of features was ultimately developed by the project team that could be included in the modeling process.

The features of interest were chosen based upon their impact on the following key factors in controlling crack growth: (1) the H/He solubility or diffusivity within the materials, (2) the stress/strain state at the crack tip, (3) material threshold for crack extension, and (4) microstructure based fracture distance, commonly estimated by grain size for intergranular fracture. Wherever possible, key references were identified to substantiate the effects on the tritium embrittlement phenomenon of the various microstructural features. Each of these features was chosen based upon their impact to the cracking phenomenon of interest.[1]

The features chosen were typically associated with orientation, morphology, and distribution of phases and inclusions, grain and grain boundary characteristics, and initial mechanical properties. Phase and inclusion content and distribution were determined to play a key role in the cracking phenomenon. The presence of δ-ferrite in the weld and strain-induced martensite in the primarily austenitic matrix are known to facilitate hydrogen diffusion and the interfaces have been observed as a hydrogen assisted fracture path. The morphology, size, and distribution of inclusions and precipitates, particularly on the grain boundaries, influence cracking since they trap hydrogen and facilitate intergranular fracture. Compositional banding and nitrogen concentration were also included as features of interest. The microstructural features of interest included (1) grain size, shape, and orientation; (2) dislocation structure and distribution, or recovered vs. un-recovered. The grain size and orientation affect the grain boundary fracture stress and the hydrogen solubility and diffusion paths. The dislocation structure and distribution play a role in hydrogen trapping as well as potentially affecting the hydrogen assisted fracture path. The initial mechanical and physical properties that are to be included in the investigation are yield stress, fracture toughness, work-hardening capacity, threshold hydrogen cracking stress intensity and stacking-fault energy.

2 INTRODUCTION

A significant amount of work has been completed by the Dynamic Materials Program, Structural Integrity Program, and the Plant Directed Research Development and Demonstration (PDRD) to collect data and establish analytical models addressing tritium reservoirs. The current processes for the delivery of tritium reservoirs is currently controlled with field experience and an empirical approach to process control, whereas simulation tools may allow for improved and cost-effective life-prediction. However, the design guide for tritium reservoirs is based upon a wealth of specific laboratory data and experience. The value of simulation tools is to reduce design iteration and manufacturing cycle lead time on the design front of the life-cycle, while minimizing the impact of non-conformance during service.

The design guide for the tritium reservoirs has evolved from the initial structural design based criteria to the formal recognition of embrittlement phenomena based upon laboratory testing, life storage and surveillance, and manufacturing experience. The design guide is highly conservative and includes safety factors on short-term and long-term failure based upon data, and defines start of stable cracking as failure.[1] This project will leverage existing data, models and ongoing work to integrate a structure of models that depict the relationship between structure, property, and performance of the reservoir. This project will develop an operationally ready capability to prognosticate vulnerabilities of tritium reservoirs as a function of service and aging. This implementation will involve leveraging existing data and models, gathering additional data, and the development and validation of an integrated structure-property-performance model capable of integrity prognostics which include aging effects.

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[1] Cracking in tritium reservoirs is a combination of tritium-induced slow crack growth and hydrogen induced cracking. Even though the short term effects of tritium and hydrogen are similar, the decay of tritium to helium and its effect on cracking adds complexity to the cracking phenomenon.

The specific objectives of the project are the following:

(1) Accurate representation of the physical system

(2) Prognostication of reservoir vulnerabilities with validation

(3) Quantified definition of design envelope

(4) Identification of research and development needs

The first task in the project was to develop a comprehensive list of microstructural features that impact the aging performance of the tritium reservoirs. Each of the participating sites (SRNL, SNL, LANL, KCP) independently developed a list of features deemed integral to tritium reservoir performance based upon operational and design experience. An integrated list of features was ultimately developed by the project team that could be included in the modeling process.

The purpose of this report is to identify the key microstructural features that affect the aging and performance of tritium reservoirs. The emphasis is on those microstructural features that will be critical in modeling the flow and fracture properties of alloys used for tritium containment. This list of features will be the starting point for characterization of fielded reservoir steels and the development of microstructural models. The goal is to integrate the models into a decision tool that accurately depict the relationship between structure, property, and performance of the reservoir.

3 TECHNICAL APPROACH

The features of interest were chosen based upon their impact on the following key factors in controlling crack growth: (1) the H/He solubility or diffusivity within the materials, (2) the stress/strain state at the crack tip, (3) material threshold for crack extension, and (4) microstructure based fracture distance, commonly estimated by grain size for intergranular fracture. Wherever possible, key references were identified to substantiate the effects on the tritium embrittlement phenomenon of the various microstructural features. Each of these features was chosen based upon their impact to the cracking phenomenon of interest.

The microstructural features that affect cracking in tritium-exposed alloys fall into three categories: base metal, weldments, and heat affected zones. The most important base metal and heat-affected-zone features include dislocation substructure as well as grain orientation and composition. The substructure of dislocations determines the overall flow properties of the material and serves as trap sites for hydrogen isotopes and decay helium bubbles. Grain boundary properties are critical to tritium-influenced crack growth, which typically occurs along grain boundaries. For weldments, the δ-ferrite content in the weld, its morphology, size, and distribution will all affect the flow and fracture behavior of the weld and the hydrogen diffusion through the weld. Ferrite-austenite interfaces within the weld are important because they have been observed as fracture paths in hydrogen-and-tritium-exposed weldments.

The impact of the features on a micromechanical approach to fracture was considered.[2] The key elements of the micromechanical approach are: (1) crack-tip stress and/or strain fields, (2) critical local stress or strain for fracture (3) critical microstructure-based fracture distance, and (4) microstructure based fracture distance, commonly estimated by grain size for intergranular fracture. The effect of hydrogen on fracture is primarily incorporated into the model through the critical local stress or strain for fracture. The local stress or strain for fracture depends on hydrogen concentration. The hydrogen concentration can be governed by trapping at microstructural features. Thus, microstructural features impact the local stress and strain for fracture not only through their inherent resistance to fracture but also through their ability to promote locally high hydrogen concentrations.

3.1 Materials and Manufacturing

Tritium reservoirs are manufactured from Types 304L, 316L, and 21-6-9 stainless steels. The properties of these materials are affected by their forged microstructures and their exposure to tritium gas during service. The combination of tritium, decay helium and microstructure govern structural property and fracture mode changes in these alloys.

Tritium reservoirs are primarily manufactured through a high rate energy forging (HERF) process, followed by machining and welding processes. The HERF process is a near-net-shape forming process that is designed to provide a warm-worked structure of the required high yield strengths. The HERF process develops a distinctive deformation pattern in stainless steels, which is shown in Figure 1. The HERF process produces a distinctive partially recovered dislocation structure typical of material that is rapidly deformed and chilled prior to the onset of significant recovery. This partially recovered structure may impart improved hydrogen isotope compatibility. The flow lines of HERF and press forgings appear similar. The goal of the forging process is to maintain deformation-induced strengthening at room temperature. However, the competing processes of recovery and static/dynamic recrystallization can lead to a wide variation in strengths within the structure of the forging. Such variations in microstructures may not only lead to mechanical properties outside of acceptable limit for design requirements but will also cause variations in tritium compatibility.[3,4] In addition to the bulk metal of the reservoir, the weld areas are known to have a variant microstructure. Depending upon the weld technique, weld composition, and processing parameters, the microstructure within the fusion zone and heat affected zone can vary.

Figure 1: Flow Pattern in High Energy Rate Forging (HERF).

3.2 Fracture Modes

The hydrogen-induced fracture mode for stainless steels is not unique and can include: (1) microvoid coalescence, (2) intergranular separation, (3) twin boundary parting, (4) cleavage, and (5) γ/α interfaces, as shown in Figure 2.[5,6] Reference 5 reported the following key parameters that control fracture mode for stainless steels:

a) Dimpled rupture is reported to be the primary mode of failure due to hydrogen, though the ductility may be low. However, dimple sizes change with a change in the yield stress of the materials.

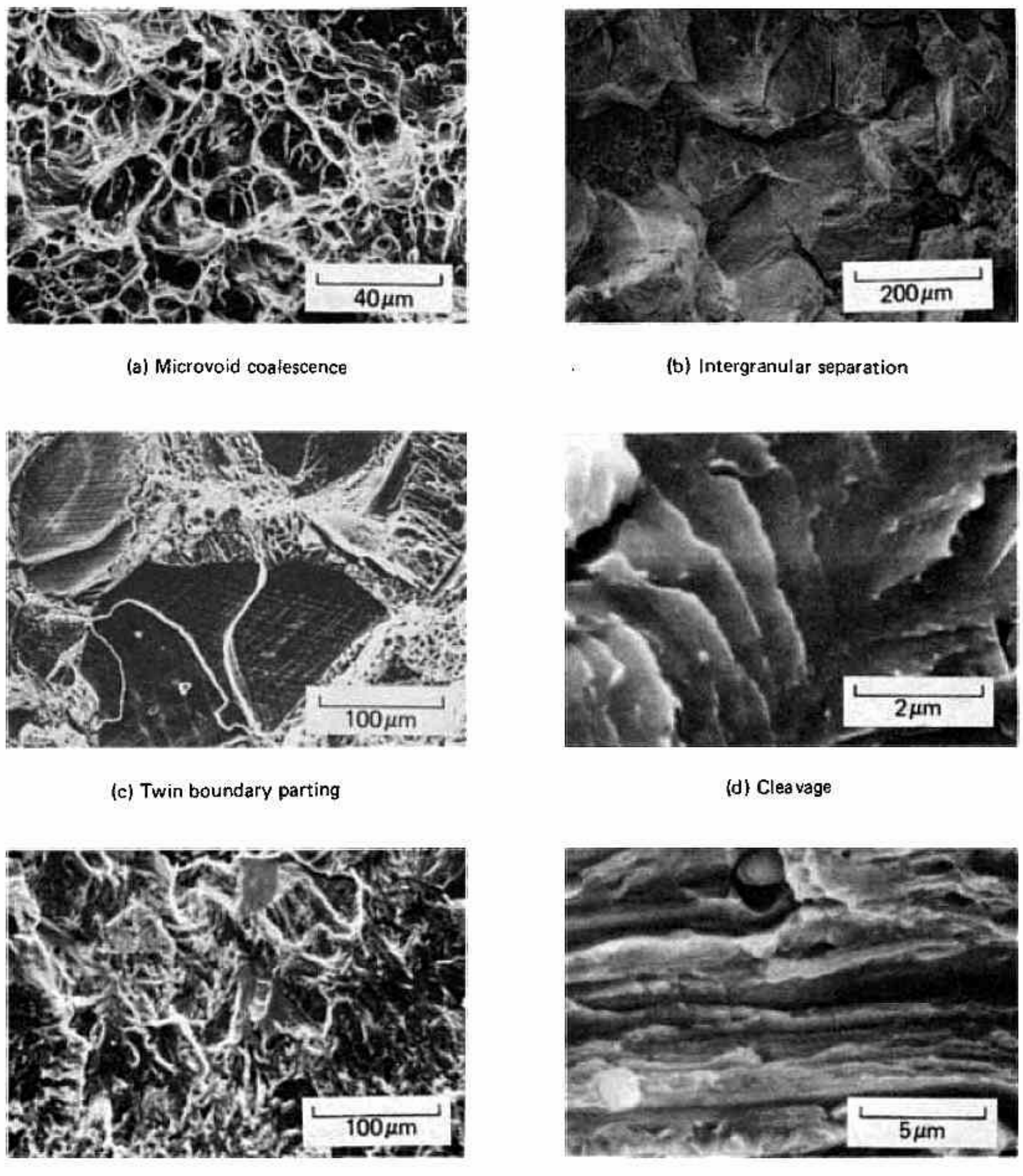
b) Intergranular separation is typical of alloys with 30-35% nickel as well as alloys that are sensitized or exhibit grain boundary precipitation.

c) Twin boundary parting is a strong function of temperature and composition, but may be present with or without hydrogen at temperatures in the 200-350K range for 304L.

d) Transgranular cleavage is evident in ferritic and martensitic stainless steels.

e) Propagation of cracks along phase interfaces (austenite-martensite, austenite-ferrite) is common at ambient temperatures. The austenite may transform to martensite in stainless steels that are unstable during deformation.

The tritium exposure effect on the fracture mode has been established in previous studies. The short-term effect of tritium is similar to that of hydrogen, but with its decay to helium, the fracture mode changes from dislocation glide to deformation twinning. This change occurs at much lower strains than in non-tritiated material, due to impedance of plastic deformation by dislocation motion by helium bubbles.[7] This is –particularly evident in HERFs as opposed to annealed samples.[4]



(a) Microvoid coalescence (b) Intergranular separation

(c) Twin boundary parting (d) Cleavage

(e) Fracture along γ/δ interface (f) Fracture along γ/α interface

Figure 2: Hydrogen Assisted Fracture Paths in Austenitic Stainless Steel. (Reproduced from Reference 5)

3.3 Grain & Grain Boundary Characteristics

The grain size, shape and orientation play a key role in controlling hydrogen/tritium assisted fracture. The grain size of the material affects several key parameters to crack growth. The grain size and grain boundary orientation can affect the boundary fracture stress and consequent cracking paths, as well as the trapping of hydrogen and tritium.[8] The intergranular fracture mode along the grain boundaries is reported in Types 304L, 316L, and 21-6-9 stainless steels, particularly when tested in hydrogen environments or when decay helium bubbles are present in the microstructure from tritium-exposure (Figure 3 and Figure 4) [7] A severe fracture mode change from ductile rupture to intergranular fracture has been reported with increasing helium-3 concentration in 21-6-9 stainless steel.[7] The fracture facets exhibited fine striations which correlated with the deformation twin band spacing.[9] Cracks oriented parallel to the long axis of the grain tend to be easier to propagate than those running across the long axis. The intergranular fracture appearance will reflect the underlining grain size, shape, and orientation as shown in Figure 3.

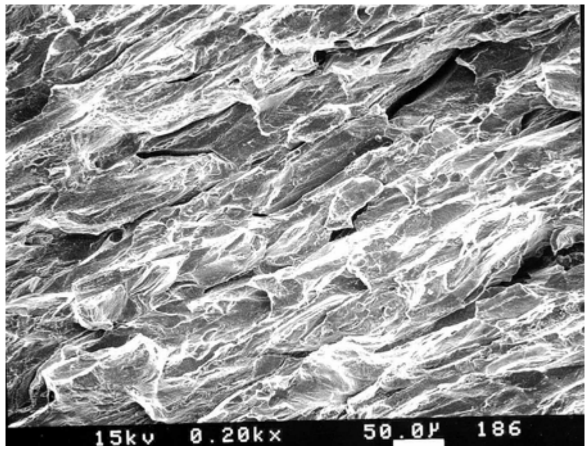


Figure 3: Hydrogen-Induced Intergranular Fracture in Type 21-6-9 Stainless Steel Reflecting the Orientation of the HERF Grain Structure.

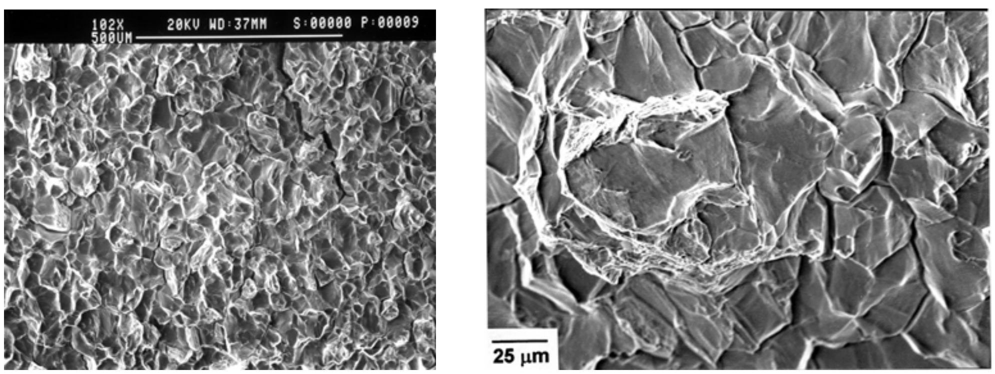


Figure 4: Examples of Tritium-Induced Intergranular Fracture in Type 304L and 21-6-9 Stainless Steels.

The grain boundaries can also act as trap sites for hydrogen, facilitating intergranular fracture. For example, the fracture mode of nickel containing hydrogen in solid solution remains a ductile shear rupture characteristic of the nickel specimens without solute hydrogen.[10] Dydrogen had little effect on ductility and fracture mode, provided there was a uniform distribution hydrogen mobility during the fracture process,. However, fracture was observed to take place by an intergranular mode if hydrogen segregation to the grain boundaries was allowed prior to the low temperature testing.

Grain boundary composition and second phases will affect grain boundary strength in a number of different ways [11,12]. Type 304 stainless steel have been shown to be more susceptible to hydrogen cracking when sensitized with the preferred path for hydrogen cracking along grain boundaries, attributed to the combined effects of impurity segregation and hydrogen.[13] It was surmised that deformation-induced martensite at Cr-depleted grain boundaries promoted the intergranular fracture. Sensitization of austenitic stainless steels has been shown to result in a strong non-uniformity of local chromium content, with phosphorus, nitrogen, and sulfur segregating to areas depleted of chromium.[14] A higher percent of intergranular fracture was obtained after sensitization and deuterium charging. Others have shown that grain boundary impurities like phosphorus and sulfur facilitate intergranular fracture.[15,16]

3.4 Compositional Effects

Several compositional characteristics play a role in the hydrogen and tritium induced cracking of stainless steels. Nitrogen is known to play a key role by affecting hydrogen and tritium solubility. Nitrogen also affects the slip mode by decreasing stacking fault energy.[5,17], It has been widely hypothesized that the stacking fault energy plays a strong role in HE susceptibility.[18,19] Hydrogen cracking may be facilitated by decreased stacking fault energy which causes increased slip planarity.

Flow lines in HERF austenitic stainless steels have been correlated to elemental inhomogeneities. [20] Areas in which flow lines occur correspond to austenite compositions that are the last to solidify during cooling from the melts. High-Energy-Rate-Forgings of Type 304L and 21-6-9 stainless steels appear to have increased elemental inhomogeneity and enhanced flow lines. These flow lines promote orientation-dependent fracture toughness, and It is commonly observed that fracture toughness was lower when crack propagation was parallel to the flow lines.[21],22] Compositional banding will also create local variations in all composition dependent microstructures and properties (such as ferrite, martensite, and stacking fault energy) which will impact fracture modes like those discussed above.

3.5 Dislocation Substructure

The dislocation substructure is one of the chief microstructural features affecting the tritium compatibility and aging behavior of stainless steels. This is largely because dislocations serve as trapping sites for hydrogen isotopes [23,24] and, after tritium decay, helium bubbles.[25-27] The trapping of hydrogen isotopes along the dislocations may be beneficial by preventing segregation to the grain boundaries. The substructure of dislocations will influence the subsequent helium bubble microstructure after tritium exposure and decay. Rocky Flats reports on the HERF microstructure indicate that there is a distinct transition from dislocation cells to sub-grains in the temperature region around 0.5 T_m, depending upon the strain rate of forging.[28]

Heavily worked stainless steels exposed to tritium can show transgranular fracture (Figure 5). This apparently results when decay helium bubbles associated with the dislocation substructure of highly deformed steels make transgranular fracture more favorable than intergranular fracture.[29]

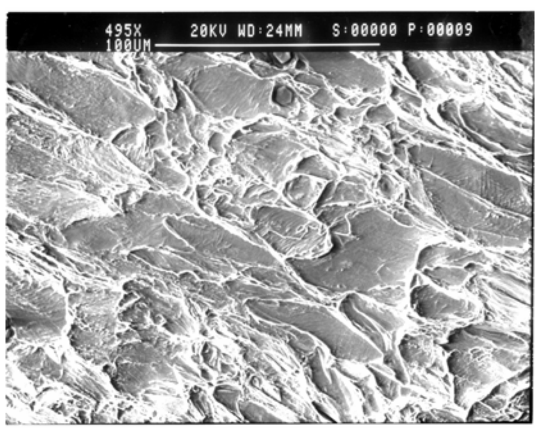


Figure 5: Tritium-Induced Transgranular Fracture through Heavily Forged 21-6-9 Steel.

Note also that the dislocation density and substructure will affect the steel's propensity for twinning deformation. As helium builds-in from tritium decay, twinning becomes favored as plastic flow from dislocation motion is impeded The onset of deformation twinning has been related to crack nucleation in tritium exposed stainless steels. Finally, the complete absence of dislocations, i.e. annealed microstructures, have a much greater tendency for tritium embrittlement.[30]

Figure 6: Highly Oriented Deformation Pattern in HERF Type 304L Stainless Steel.

3.6 Second Phases, Particles and Inclusions

The presence of second phases, particles, and inclusion also play a role in the hydrogen and tritium induced crack growth in austenitic stainless steels. These are transgranular features in contrast to the grain boundary characteristics mentioned above.

The formation of strain-induced martensite in Type 304L steel is identified as a feature of interest but its real impact seems to be during deformation.[31] The formation of martensite during deformation facilitates increased hydrogen diffusion and may play an important role in crack nucleation. Louthan saw more rapid penetration of tritium by autoradiography after martensite formation.

Inclusion morphology, size and distribution are important to the dimpled rupture process (Figure 7).[23] In the case of hydrogen or tritium exposed alloys, they will be important as trap sites for hydrogen and possibly as micro-crack nuclei. In alloys resistant to hydrogen-induced intergranular fracture, the dimpled rupture process tends to be prevalent.

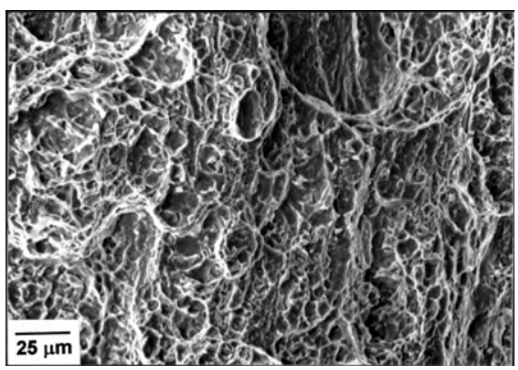
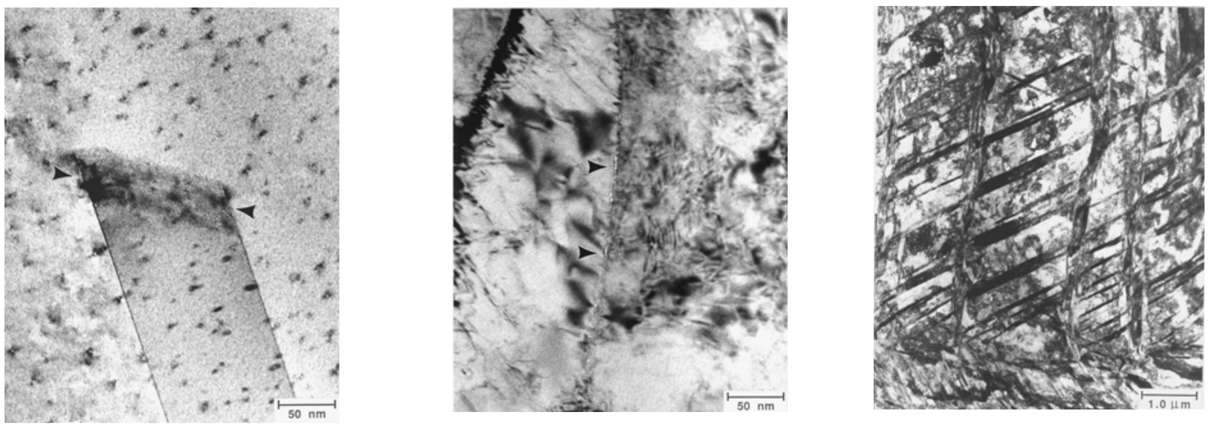


Figure 7: Fracture by Dimpled Rupture Typical of Unexposed Alloys. The Size, Shape and Distribution of Particles and Inclusions

3.7 Decay Helium Bubble Microstructures

The short-term effects of tritium and hydrogen on cracking in stainless steels is similar, however, the decay of tritium to helium with time has an added effect. When tritium decays within an exposed alloy, helium bubbles precipitate homogeneously within the matrix and on various defects throughout the microstructure (Figure 8). This means that the helium bubble microstructure is influenced by the starting material microstructure. This has important consequences for tritium-induced crack paths.

Helium bubbles are obstacles for dislocation motion, thereby increasing the yield strength of forged and annealed stainless steels.[3,4] This increase in yield strength causes deformation twinning to dominate the deformation of the metal, beginning upon yielding and reaching high densities at small strain, which is particularly evident in HERFs.[7] Helium clusters have been observed on incoherent twin boundaries. Coherent twins and grain boundaries may contain helium bubble clusters which are likely below the resolution of the characterization technique.



(a) (b) (c)

Figure 8: Helium Bubble Microstructure in Tritium-Exposed-And-Aged 21-6-9 Stainless Steel: (a) Strain Fields Associated With Helium Bubbles give rise to the "Black Dots" in the Matrix and Clusters of Helium Bubbles at Incoherent Twin Boundaries; (b) Bubble Growth and Coalescence on a Grain Boundary; and (c) Deformation Twinning in Forged Microstructure.

3.8 Weldments and Heat-Affected-Zones

Weldments in stainless steel reservoirs are critical areas with regard to hydrogen and tritium induced cracking. The δ-ferrite volume fraction, morphology, orientation, size, and distribution are the most important microstructural feature affecting the flow and fracture behavior.[32-34] Weld ferrite can provide an easy diffusion path for hydrogen. Additionally, ferrite-austenite interfaces have been observed as fracture paths in hydrogen exposed steels. In weld heat-affected zones, hydrogen-induced cracking occurs along grain boundaries. Carbide precipitation at grain boundaries of sensitized microstructures plays an important role in that it depletes chromium from the matrix and serves as hydrogen and helium trap sites. In addition, the helium bubble microstructure is affected by the weld ferrite (Figure 9).

Figure 9: Typical Weld Ferrite Morphology in Type 304L Stainless Steel

3.9 Solid State Welds

There has been little work conducted on the effects of hydrogen and tritium on solid state weldments. The fracture morphologies of the unexposed steels showed that ductile fracture occurred by the microvoid nucleation and growth process (Figure 10). The microvoids on the fracture surfaces of the welded steels were much smaller and more closely spaced that those found on the base material fracture surfaces. The surface condition of the steel, prior to welding and the amount of upset appear to be important factors affecting weld microstructure and their behavior in hydrogen environments.35

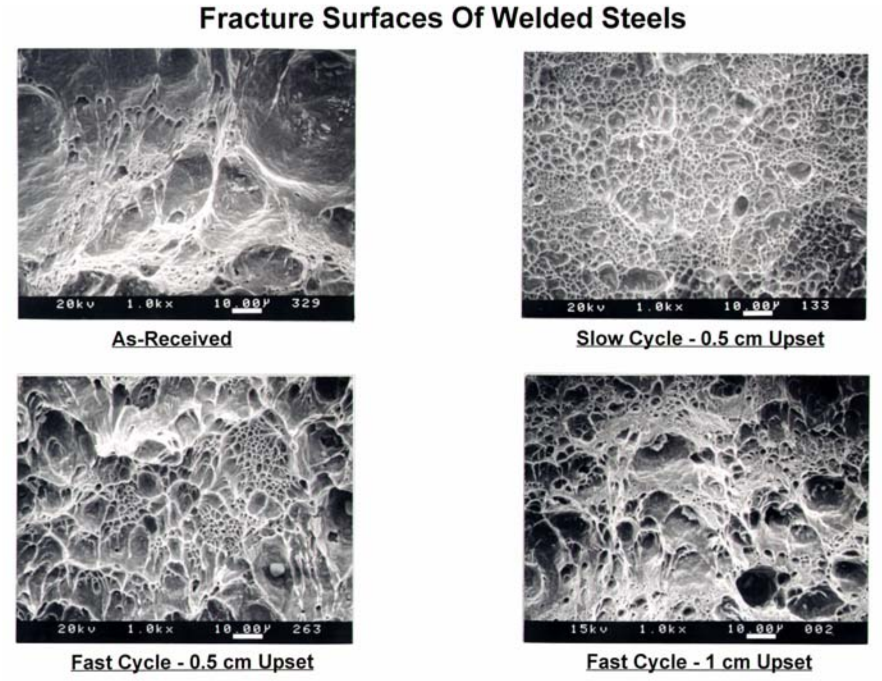


Figure 10: Fracture Appearance of Solid State Weldments

4 MODELING APPROACH

The modeling approach and capabilities were considered when determining the list of microstructural features. The initial simplified modeling approach is summarized in Figure 11.

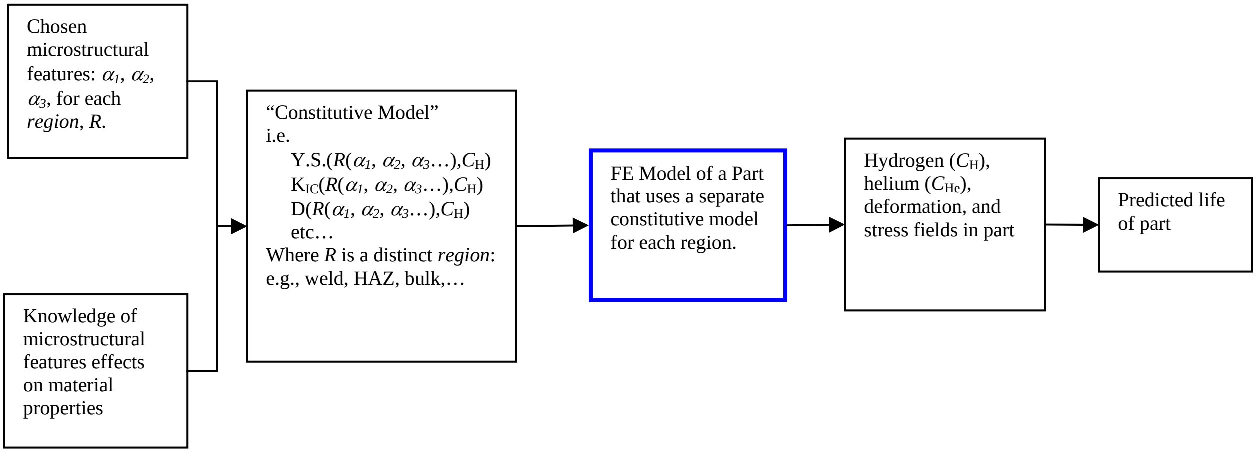


Figure 11: Summary of Modeling Approach

The constitutive models, in addition to being functions of helium and hydrogen content (C_H and C_{He}, respectively), are also functions of microstructural parameters. However, the microstructural parameters are not allowed to vary continuously over the part; instead, there are discrete regions of distinct microstructure (e.g. weld, bulk, HAZ). In general, continuum models of hydrogen enhanced fracture assume that failure occurs when a critical hydrogen concentration is locally attained.

5 CONCLUSION

A project to implement a life-cycle engineering approach to tritium reservoirs has been initiated through the DOE - Technology Investment Projects. The first task in the project was to develop a comprehensive list of microstructural features that impact the aging performance of the tritium reservoirs. The features chosen were typically associated with orientation, morphology, and distribution of phases and inclusions, grain and grain boundary characteristics, and initial mechanical properties. This list of features will be the starting point for characterization of fielded reservoir steels and the development of microstructural models. The goal is to integrate the models into a decision tool that accurately depict the relationship between structure, property, and performance of the reservoir.

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