

***Technical Work Plan:  
Characterization of Weld Regions  
on a Full-Scale Cylindrical Mockup  
of an Interim Storage Container***

**Fuel Cycle Research & Development**

***Prepared for  
U.S. Department of Energy  
Used Fuel Disposition Program  
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December 23, 2014  
FCRD-UFD-2014-000710***



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## APPENDIX E

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Name/Title of Deliverable/Milestone/Revision No. Characterization of Weld Regions on a Full-Scale Cylindrical Mockup of an Interim Storage Container/ M4FT-15SN0805054

Work Package WBS Number 1.02.08.05

Responsible Work Package Manager Charles R. Bryan / *CRB* Dec. 23, 2014  
(Name/Signature) Date Submitted

Quality Rigor Level for Deliverable/Milestone <sup>2</sup>	<input checked="" type="checkbox"/> QRL-3	<input type="checkbox"/> QRL-2	<input type="checkbox"/> QRL-1 Nuclear Data	<input type="checkbox"/> Lab/Participant QA Program (no additional FCT QA requirements)
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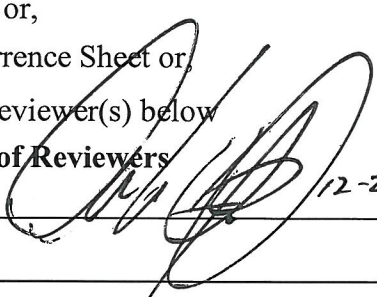
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## SUMMARY

Stress corrosion cracking (SCC) of interim storage containers has been indicated as a high priority data gap by DOE(Hanson, 2012), EPRI(2011), NWTRB(Rigby, 2010), and the NRC(2012, 2012a). Uncertainties exist both in terms of the environmental conditions that exist on the surface of the storage containers (as discussed below) and the electrochemical properties of the storage containers themselves. The goal of this task is to assess the effects of the manufacturing process on canister performance by evaluating the properties of a full-scale interim storage canister mockup. This mockup has been produced using the same manufacturing procedures as fielded spent nuclear fuel interim storage canisters.

In order for SCC to be a viable degradation mode, there must be three criteria that are met – there must be a sufficiently large stress in the material to support crack growth, the material itself must be susceptible to stress corrosion cracking, and the environment must be sufficiently aggressive to support crack initiation and propagation. The work outlined in this test plan is aimed at determining the first two of these criteria. Assessment of the residual stress will use a combination of three techniques. These include neutron diffraction, the contour method, and the deep-hole drilling method. Neutron diffraction will be explored via collaboration with LANL. The other two techniques will also involve collaboration with other groups capable of performing these measurements.

Evaluation of the electrochemical properties of the welded regions of the container will first involve an assessment of the microstructure of the regions at the longitudinal, circumferential, and repair welds via standard metallurgical techniques. The thermal cycling associated with the welding process will, in addition to altering the overall microstructure of the near-weld material, result in the precipitation of chromium carbides and the formation of chromium depleted regions along the grain boundaries. This effect, known as sensitization, will be particularly pronounced in the weld heat affected zone (i.e., the region near the weld fusion zone that has been impacted by the heat input from the welding process). The extent to which sensitization has taken place will be documented as a function of position from the edge of the weld fusion zone. This will be done both for the near surface regions, as well as through the thickness of the container wall. A volumetric assessment of the degree of sensitization will illustrate the extent of the region and illustrate the presence/absence of an active path for crack propagation through the material.

Establishing the susceptibility to SCC will require both the resistance to crack nucleation as well as the magnitude of crack propagation to be assessed as a function of the environmental conditions to which the container is subjected while exposed to stresses as defined by the full scale mock-up. A wide variety of experiments are planned to explore both aspects of the cracking process, as well as to define methods to produce relevant weld analog materials.

Upon completion of the residual stress measurements, the mock-up will be sectioned to perform stress corrosion cracking initiation tests. These coupons, in addition to being critical for the UFD program, are also of great interest to outside parties such as EPRI and the various academic groups working on NEUP programs. Meetings will be held with each of the interested parties to assess their needs. Utilizing the information from each party, a larger prioritized list of coupons will be assembled. The needs of the UFD program will be given first priority, followed by the DOE funded NEUP groups and EPRI



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## ACRONYMS

ASME B&PVC	American Society of Mechanical Engineers Boiler and Pressure Vessel Code
ASTM	American Society for Testing and Materials
DOE	Department of Energy
EDM	Electric Discharge Machining
EPR	Electrochemical Reactivation
EPRI	Electric Power Research Institute
FCRD	Fuel Cycle Research and Development
NEUP	Nuclear Energy University Programs
NRC	Nuclear Regulatory Commission
NWTRB	Nuclear Waste Technical Review Board
SCC	Stress Corrosion Cracking
UFD	Used Fuel Disposition
XRD	X-Ray Diffraction



# TECHNICAL WORK PLAN:

## EVALUATION OF THE FULL-SCALE INTERIM STORAGE CONTAINER MOCKUP

### 1. INTRODUCTION

Stress corrosion cracking (SCC) of interim storage containers has been indicated as a high priority data gap by DOE(Hanson, 2012), EPRI(2011), NWTRB(Rigby, 2010), and the NRC(2012, 2012a). Uncertainties exist both in terms of the environmental conditions that exist on the surface of the storage containers (as discussed below) and the electrochemical properties of the storage containers themselves. The goal of this task is to assess the effects of the manufacturing process on canister performance by evaluating the properties of a full-scale interim storage canister mockup. This mockup has been produced using the same manufacturing procedures as fielded spent nuclear fuel interim storage canisters.

The material used to construct the storage containers, as well as the manner through which the containers are constructed (i.e., formed and welded plates) dictates not only the susceptibility of the material to localized attack such as SCC, but also provides the driving force (i.e., the stress) for crack propagation in the form of residual stresses from welding and, to a lesser extent, forming of the metal plates used to build the storage container. The primary material of construction across the various cask designs in use today is 304SS, a material that has been demonstrated throughout the literature to be susceptible to chloride induced stress corrosion cracking.

Due to the carbon content of 304SS, it is prone to a phenomenon known as sensitization when welded. The thermal cycling associated with welding facilitates the precipitation of chromium carbides. As the chromium diffusion rate is significantly faster along grain boundaries than within the bulk, these carbides tend to grow at the grain boundaries. In time, chromium depleted zones are formed along the grain boundaries. These chromium depleted zones are more susceptible to corrosion than the surrounding grains, and serve as a preferential site for localized corrosion to initiate, potentially providing a crack initiation site (and fast growth path) for stress corrosion cracking. The overall degree of sensitization is thus a function of the carbon content of the material, and reduced carbon content alloys (e.g., L grade stainless steels such as 304L) are less susceptible.

In order to assess the implications of the environment that develops on the surface of an interim storage container, it is essential that the susceptibility of the container, in particular, the weld regions, be understood. As such, a full-scale mockup storage container has been fabricated. The following discussion outlines the various techniques which will be used to determine:

1. The near surface and through-thickness residual stress states associated with the welds used to construct the container
2. The near-surface and through-thickness residual stress states associated with the as-formed walls of the container (i.e., far from the welds)
3. The degree of sensitization associated with the various weldments used to form the container

### 2. Detailed Description of the Full-Scale Mockup

There are three primary vendors in the US for welded stainless steel interim storage systems – Areva-TN, Holtec, and NAC. Typical canister designs were reviewed to determine materials and methods of

construction. Nearly all designs used 304SS (for older containers) and welding was multi-pass, predominantly utilizing the submerged arc welding process with a double-V edge preparation. A single canister design was selected, and a cylinder (without end plates) was purchased from a fabricator that has produced canisters under the direction of one of the vendors. The mock-up whose evaluation is described in this document is based upon the TransNuclear NUHOMS 24P design. This container design is employed at the Calvert Cliffs nuclear power station, which was the first site surveyed by EPRI for the dust composition on the surface of the containers (Gellrich, 2013).

The mock-up, pictured schematically in Figure 1 below, consists of three cylindrical shells, each being 48 inches high, 67.2 inches in diameter, and with a wall thickness of 5/8 inch. Each shell was formed by cold forming a plate into a cylinder, then making a single longitudinal weld to form the cylinder. The three cylinders were then welded together to form a single large cylinder 12 feet in length with two circumferential welds. All of the welds were formed via the submerged-arc welding process and were multi-pass. The inner diameter was welded first, followed by the outer diameter.

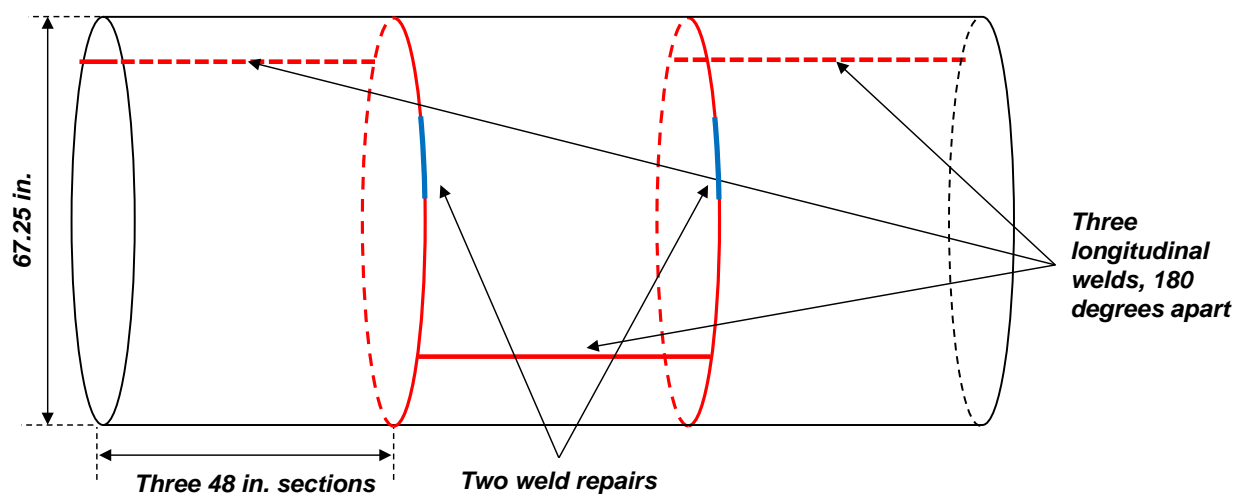


Figure 1: Schematic representation of the full scale mock storage container manufactured at Ranor

Production of the container was done at Ranor (located in Westminister, MA), the fabrication facility where the containers located at the Calvert Cliffs site were produced. Use of the same manufacturer was critical as, while the overall design is owned by Areva-TN, the production methodologies used are proprietary to Ranor. In other words, Areva-TN specified the overall design (i.e., material, overall dimensions, etc.) but Ranor developed the methodology to actually build each storage container.

Three plates of dual-certified 304/304L stainless steel were used to construct the container. The thickness of each plate was verified via ultrasonic inspection prior to being welded. The weld filler metal was 308L SS, as typically used when welding 304. The composition of the 304/304L plates and the 308L filler material are in the table below.

Table 1: Composition of 304L Plate and 308L Filler Metal Used to Construct Mock-Up

	C	Co	Cr	Cu	Mn	Mo	N	Ni	P	S	Si
Plate Material (304/304L)	0.0223	0.1865	18.1000	0.4225	1.7125	0.3180	0.0787	8.0270	0.0305	0.0023	0.2550
Weld Filler (308L) (lot 1)	0.014	--	19.66	0.16	1.70	0.11	0.058	9.56	0.025	0.010	0.39

<b>Weld Filler (308L) (lot 2)</b>	0.012	--	19.71	0.192	1.730	0.071	0.053	9.750	0.024	0.012	0.368
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In addition to the composition of the materials of construction, the parameters for each weld pass were documented. These include the current, voltage, travel speed, heat input, and interpass temperature. All welds used a double-V edge preparation with a 30 degree bevel.

In addition to welds formed under nominal conditions, discussions with Transnuclear and Holtec have indicated that for actual storage containers there will likely be repaired regions along the welds, at regions where the nondestructive testing indicated that the weld did not conform to the criteria in ASME B&PVC Section III, Division 1, Subsection NB. As such, all of the welds were subjected to a full radiographic inspection. While none of the welds contained indications requiring repair, a region of each circumferential weld was subjected to a repair procedure on the outer diameter. The purpose of the repaired regions is to allow determination of typical weld residual stresses and degrees of sensitization typical repair regions.. Repair regions have been identified by numerous researchers as having dramatically elevated residual stresses when compared to unrepaired portions of a weld (e.g., Bouchard 2005; Dong, 2002 and 2005;, Elcoate 2005, George 2005, Hossain 2006 and 2011,).

### **3. Weld Residual Stress**

In addition to a susceptible material and a sufficiently aggressive environment, the nucleation and growth of a stress corrosion crack requires the presence of a sufficiently large stress. In the case of interim storage containers, the stress existing within the structure will predominantly be residual stresses resulting from the forming of the metal plates into a cylinder and the subsequent welding of the panels. The latter are likely to be the largest in magnitude, and are the result of the constraint placed by the structure of the container (and any additional fixtures used during fabrication) on the weld as it solidifies. A wide variety of methods are available for residual stress measurement, as summarized in NUREG-2162 (Benson, 2014). Techniques are typically based on measurement of elastic strains in the crystal structure using diffraction of either x-rays or neutrons, or on strain measurements made upon mechanically altering the material being investigated (allowing stress relaxation to occur). The techniques vary in terms of their sensitivity and depth of penetration into the substrate metal. The most appropriate technique for assessing the mock-up storage container is one capable of measuring the stresses through the thickness of the container wall. Furthermore, the technique must allow for evaluation of large sections, as it is desired that the stress state be measured prior to and following the sectioning of the container into smaller samples for use in corrosion or stress corrosion cracking experiments.

Three techniques have been initially targeted for use in this study. These include neutron diffraction, the contour method, and the deep-hole drilling method. Neutron diffraction will be explored via collaboration with LANL. The other two techniques will also involve collaboration with other groups capable of performing these measurements.

#### **3.1 Deep Hole Drilling**

A variety of hole-drilling based techniques are available for the evaluation of the stress state in a metal sample. Typically, these techniques involve the attachment of strain gauges to the surface of the material being measured, which are monitored while a hole is precisely drilled in the material. As the hole is drilled, the local constraint within the structure is relaxed, allowing stress relaxation to occur, resulting in surface deformation which is captured by the strain gauges. A similar drilling technique involves the use of a surface strain gauge around which a core is cut into the material, allowing the center pillar to relax. These techniques, while they can be accurate, are only sensitive to the near-surface stresses in the material.

The deep hole drilling differs from other hole-drilling techniques in that it does not rely on the use of surface strain gauges. A small diameter hole is precisely drilled via a gun drill through the material. An air gauge is then used to precisely characterize the diameter of the hole along its length. Next, electric discharge machining (EDM) is used to cut an overcore around the aforementioned hole. As the core is cut, the constraint placed on the metal immediately adjacent to the central hole is relaxed, resulting in local lateral displacement of the material. The inner diameter of the hole is then re-characterized and the resulting change in diameter, due to the loss of constraint around the hole, is recorded. From these strains, the original residual stress state within the material as a function of depth can be calculated.

The calculations used for standard deep hole drilling are based upon the assumption that the stress relaxation leading to the measured displacements is entirely elastic in nature. When large stresses are present, this is not true, and plastic deformation of the material can result, hindering the ability of the technique to resolve stress. For a heavily constrained weld, such as the circumferential weld on the interim storage containers, it is anticipated that the residual stress levels will be very high – approaching the yield strength of the material – and as such, the traditional analysis will not work. To compensate for this, the deep hole drilling technique must be modified. (Mahmoudi, 2009 and 2011) In the modified technique, the EDM core is cut in steps. After each step, the inner diameter of the hole is characterized via the air probe. By measuring the deformation of the inner diameter of the hole at the depth of the core cut, the effect of plasticity can be addressed. The resulting residual stress distribution, while being lower in vertical resolution than the traditional measurement, is able to resolve large residual stresses. This modified technique is known as incremental deep hole drilling

The deep hole drilling technique is semi-destructive in nature. The drilling of the hole and the EDM overbore is obviously destructive to that region, but the site is small (1.5mm diameter hole, 6mm diameter overbore), but the remainder of the mockup will be undisturbed. Perhaps more importantly, the deep hole drilling technique can be employed without cutting the mock-up into smaller pieces. While the use of surface strain gauges can help measure the stress relaxation associated with cutting a section of the weld from the mockup, the cutting process introduces an additional level of complexity and adds to the uncertainty of the residual stress measurement.

### 3.2 Contour Method

As with hole drilling techniques, the contour method involves the removal of constraint from the system, and the measurement of the resulting relaxation displacements. In the contour method, the specimen being evaluated is fixtured securely in place, and a cut is made across the region where the stress state is to be assessed. A coordinate measuring machine is then used to precisely measure the deviations of the cut surface resulting from the stress relaxation associated with making the cut. Mathematically, the deviations (i.e., strains) are converted into the residual stress state that existed prior to being cut. In essence, the amount of stress required to force the surface flat is calculated.

The calculated stress field represents one stress direction – perpendicular to the cut surface. In order to get the other two stress directions, X-ray Diffraction (XRD) measurements are made, mapping the other two stress states over the exposed surface.

The contour method is destructive in nature and requires that the region being measured be extracted from the mock-up container. However, the resulting stress distribution is a high resolution map across the entire cut surface. In addition, the use of external strain gauges when extracting the piece of material to be analyzed enables the stress relaxation not captured by the contour measurement itself to be added back in, such that the initial stress state can be accurately estimated.

### 3.3 Neutron Diffraction

Diffraction-based measurements allow the stress state to be evaluated with little or no cutting of the material being evaluated. The region being analyzed is hit with a collimated beam of neutrons. The

neutrons are diffracted from the sample and measured via panel detectors. Using Bragg's law, the diffracted neutrons are used to measure the lattice spacing of the stressed sample. The lattice spacings are then compared to similar measurements taken from an unstressed sample. Based upon the distortion of the stressed sample relative to the unstressed sample, the strain field within the structure can be measured. These strains are then converted to normal stresses through the use of Hooke's law in three dimensions.

By stepping the sample through the neutron beam, the stress distribution in a two-dimensional slice through the sample can be measured. As mentioned above, the use of neutron diffraction requires the availability of an unstressed sample. That sample must have the same metallurgical condition as the region being measured – this includes the microstructure as well as the elemental composition. Deviations in either will add to the uncertainty of the reported stresses. When dealing with specimens that have a uniform structure (chemically and microstructurally) developing the reference sample is relatively straightforward. Unfortunately, in the case of a weld, the structure is extremely non-uniform, progressing from what may be a uniform grain structure in the bulk metal, to a dendritic structure in the weld fusion zone. As a result, the reference sample must be cut from a segment of the weld nominally identical to that for which the stress state is being analyzed. The reference sample is typically a thin slice, which has been further cut into a comb pattern, allowing for the relief of any residual stress within the slice.

Another complication with neutron diffraction is the size of the chamber. There are no chambers available into which the full-scale mockup would fit. As such, large structures such as the mockup must be cut down into a ring, allowing 1-2 feet of material on either side of the weld to be present. Surface-mounted strain gauges will be employed as the sample is cut, such that any stress relieved by the cutting process can be added back in to the final calculated stress distribution. Thus, while neutron diffraction is ideally non-destructive, for large structures this is not the case. Due to physical limitations of the system in which the analysis will be performed, some cutting must take place.

### 3.4 Stress Measurement Locations and Methods for the Mock-Up

In order to completely characterize the residual stresses associated with the welds in the mock-up, a combination of the three techniques described above will be employed. The combination of the three measurements will be used to mitigate the errors/uncertainties associated with any one. Unfortunately, no one facility is able to do all of the measurements. As a result, the container must be cut in half, allowing one circumferential weld to be shipped to each location. The container will be cut at Ranor in the center of the middle segment (i.e., at the midpoint between the two circumferential welds) as shown in Figure 2. One section will be sent to the facility performing contour measurement and deep hole drilling measurements, and the other to the facility doing neutron diffraction measurements.

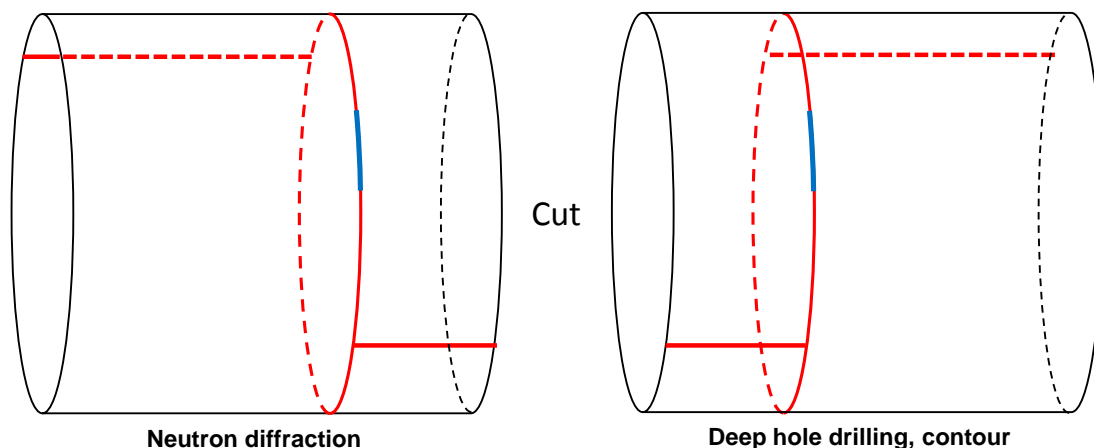


Figure 2: Plan for sectioning the container into two segments, one for neutron diffraction work and the other for the more destructive deep hole drilling and contour method measurements.

Measurements will be made in five different regions, as illustrated schematically in Figure 3.

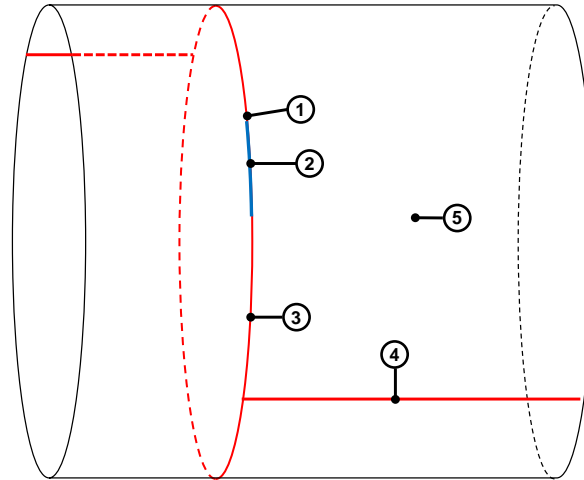


Figure 3: Regions for residual stress measurements. (1) End of repair region, (2) Center of repair region, (3) Circumferential weld, (4) Longitudinal weld, and (5) Base metal

Measurements will be made of the circumferential and longitudinal welds, as well as at the end and across the center of the repair regions. Finally, the stress state of the base metal will be measured. The following measurements are planned.

*Circumferential weld and Longitudinal weld:*

- a. Deep hole drilling measurement in center of fusion zone
- b. Deep hole drilling measurement in heat affected zone (immediately adjacent to fusion zone)
- c. Contour measurement perpendicular to weld centerline
- d. (optional) Contour measurement along weld centerline (parallel to centerline)

*Center and end of weld repair region:*

- a. Contour measurement perpendicular to weld centerline
- b. (optional) Contour measurement parallel to weld centerline (in center of weld)
- c. (optional) Deep hole drilling measurement in center of weld fusion zone (center of repair)

*Base metal:*

- a. Deep hole drilling measurement

When performing the deep hole drilling measurements, the holes will be drilled prior to any sectioning for removing contour measurement coupons. Each time the container is cut, the holes will be re-characterized. Once they are at their final size, the EDM overcore will be performed, and then the holes characterized one final time. As a result, the series of measurements will provide an assessment of the degree of stress relaxation associated with the gradual reduction in size of the workpiece. This information will be of critical importance when determining how the welds will be sectioned for SCC initiation testing as described below.

## 4. Weld Metallurgical Condition and Degree of Sensitization

Assessment of the microstructure of the regions at the longitudinal, circumferential, and repair welds will be performed using standard metallurgical techniques. An effort will be made to perform these analyses in the same basic regions as the residual stress measurements such that the two can be correlated.

The thermal cycling associated with the welding process will, in addition to altering the overall microstructure of the near-weld material, will result in the precipitation of chromium carbides and the formation of chromium depleted regions along the grain boundaries. This effect, known as sensitization, will be particularly pronounced in the weld heat affected zone (i.e., the region near the weld fusion zone that has been impacted by the heat input from the welding process). The extent to which sensitization has taken place will be documented as a function of position from the edge of the weld fusion zone. This will be done both for the near surface regions, as well as through the thickness of the container wall. A volumetric assessment of the degree of sensitization will illustrate the extent of the region and illustrate the presence/absence of an active path for crack propagation through the material.

Samples taken from the container will be prepared metallographically and evaluated electrochemically for the degree of sensitization. Evaluation will be done through either the single loop electrochemical reactivation (EPR) test or double-loop EPR test. For the single loop test, as defined in ASTM G108 "Standard Test Method for Electrochemical Reactivation (EPR) for Detecting Sensitization of AISI Type 304 and 304L Stainless Steels", the surface to be analyzed is polarized anodically such that the surface is activated. This results in enhanced dissolution of the chromium depleted grain boundaries, while the remainder of the grain is rendered passive. The net charge associated with dissolution of the chromium depleted regions along the grain boundaries is determined based upon the total current passed during the aforementioned polarization. This technique requires characterizing the microstructure of the material (specifically, the grain size), such that the overall charge per unit area of grain boundary can be calculated, the magnitude of which defines the extent of sensitization. The second technique is a modification of the first, and is more suitable for instances where the surface finish of the material being evaluated is less well defined, or measurement of the grain size within the material is difficult. This technique, developed by Akashi et al. (ASTM, 1995) is known as the double-loop EPR technique. In this method, the sample is essentially subjected to the same polarization as the single loop EPR, but it is applied twice. The ratio of the peak currents extracted from the first and second polarization is then recorded. The magnitude of this ratio is directly related to the degree of sensitization of the material. For an un-sensitized microstructure, the first polarization passivates the sample, such that the peak current from the second polarization is considerably lower than the first. However, in the case of a sensitized microstructure, the chromium depleted zones are not passivated by the first polarization, and the peak current for the second polarization will be large, approaching the value of the first polarization. The magnitude of the ratio is used to assess the degree of sensitization, with the value approaching 1 for heavily sensitized materials.

In the event that the electrochemical techniques are insufficient to define the degree of sensitization of the container wall material, alternate methods will be pursued, such as those defined in ASTM A262 "Standard Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels". This specification provides a series of immersion tests designed to activate grain boundaries such that the extent of attack can be assessed either via metallography or weight change measurement.

Positional mapping of the degree of sensitization will be accomplished by selectively mapping regions of the surface using plating tape or a similar material. The exposed region will be selected such that a sufficient number of grains are evaluated in each test. Replicate measurements will be accomplished by grinding the surface upon the completion of the each successive set of experiments, such that the region immediately below the first set of measurements can be performed. This procedure will be repeated for

each of the regions where the stress distribution is assessed (i.e., the circumferential, longitudinal, and repair welds).

## 5. Stress Corrosion Cracking Susceptibility

Establishing the susceptibility to SCC will require both the resistance to crack nucleation as well as the magnitude of crack propagation to be assessed as a function of the environmental conditions to which the container is subjected while exposed to stresses as defined by the full scale mock-up. A wide variety of experiments are planned,

1. The information learned from the residual stress measurements, combined with the electrochemically determined degree of sensitization will enable the fabrication of material simulating the weld heat affected zone. A Gleeble will be used to replicate the thermomechanical processing to which the heat affected zone has been subjected. These samples will then be used in simple U-bend experiments, where marine aerosols are deposited on the surface of the bent portion of the sample (replicating the surface deposits observed via in-service inspections, as well as predicted worst-case deposits), then subjected to combinations of humidity and temperature typical of coastal ISFSI sites. These experiments will be used to evaluate SCC initiation under relevant conditions and with relevant materials and stresses.
2. Crack propagation studies will be conducted using compact tension specimens where the microstructure has been modified so as to accurately simulate the condition in the heat affected zone of actual storage containers, and tensile stresses will cover the range of residual stresses measured in the mockup welds. The surface deposits and exposure conditions will be similar to those explored for U-bend specimens.

Specimens taken from the large scale mockup, sized so as to maintain a residual stress distribution similar to that for the as-received condition will be used for crack initiation and propagation studies. A combination of worst case and field representative deposits will be placed on the surface of the samples, after which they will be subjected to relevant temperature and humidity exposure. It should be noted that these samples will be limited in number, and as such the aforementioned tests will be used to define the conditions used to evaluate specimens taken from the mock-up.

## 6. Weld Sample Dissemination to Interested Parties

Upon completion of the residual stress measurements, the mock-up will be sectioned to perform stress corrosion cracking initiation tests. These coupons, in addition to being critical for the UFD program, are also of great interest to outside parties such as EPRI and the various academic groups working on NEUP programs. Meetings will be held with each of the interested parties to assess their needs. Utilizing the information from each party, a larger prioritized list of coupons will be assembled. The needs of the UFD program will be given first priority, followed by the DOE funded NEUP groups and EPRI.

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