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## CURRENT STATUS OF THE CHARACTERIZATION OF RPV MATERIALS HARVESTED FROM THE DECOMMISSIONED ZION UNIT 1 NUCLEAR POWER PLANT

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### **ABSTRACT<sup>1</sup>**

The decommissioning of Units 1 and 2 of the Zion Nuclear Power Station in Zion, Illinois, after ~ 15 effective full-power years of service presents a unique opportunity to characterize the degradation of in-service reactor pressure vessel (RPV) materials and to assess currently available models for predicting radiation embrittlement of RPV steels [1-3]. Moreover, through-wall thickness attenuation and property distributions are being obtained and the results to be compared with surveillance specimen test data. It is anticipated that these efforts will provide a better understanding of materials degradation associated with extending the lifetime of existing nuclear power plants (NPPs) beyond 60 years of service and subsequent license renewal. In support of extended service and current operations of the US nuclear reactor fleet, the Oak Ridge National Laboratory (ORNL), through the U.S. Department of Energy, Light Water Reactor Sustainability (LWRS) Program, coordinated procurement of materials, components, and other items of interest from the decommissioned Zion NPPs. In this report, harvesting, cutting sample blocks, machining test specimens,

test plans, and the current status of materials characterization of the RPV from the decommissioned Zion NPP Unit 1 will be discussed. The primary foci are the circumferential, Linde 80 flux, wire heat 72105 (WF-70) beltline weld and the A533B base metal from the intermediate shell harvested from a region of peak fluence ( $0.7 \times 10^{19} \text{ n/cm}^2$ ,  $E > 1.0 \text{ MeV}$ ) on the internal surface of the Zion Unit 1 vessel. Following the determination of the through-thickness chemical composition, Charpy impact, fracture toughness, tensile, and hardness testing are being performed to characterize the through-thickness mechanical properties of base metal and beltline-weld materials. In addition to mechanical properties, microstructural characterizations are being performed using various microstructural techniques, including Atom Probe Tomography, Small Angle Neutron Scattering, and Positron Annihilation Spectroscopy.

### **INTRODUCTION**

It is well known that components and structures in a NPP must withstand a very harsh operating environment, including time at temperature, stress from operational loads, neutron irradiation, and corrosive media [1]. Moreover, extending reactor service beyond 60 years will increase the effects of those stressors and possibly introduce new modes of degradation [4]. Although the numerous modes of degradation are complex and vary depending on location and material, understanding and managing materials degradation is key for the continued safe and reliable operation of NPPs. As noted in Volume 3 of the Expanded Materials Degradation Assessment (EMDA) [5], an important element of understanding aging-related degradation modes of the RPV and associated

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components is the examination of service-aged materials. And an important source of service-aged materials has been the LWRS Zion Harvesting Project (ZHP) [2]. This project is important because access to materials from active or decommissioned NPPs provides an invaluable resource for which there is limited operational data or experience to inform relicensing decisions and assessments of current degradation models to further develop the scientific basis for understanding and predicting long-term environmental degradation behavior.

The ZHP, in cooperation with ZionSolutions, LLC, a subsidiary of EnergySolutions, Inc., an international nuclear services company, was established in 2011 to coordinate the selective procurement of materials, structures, components, and other items of interest to the LWRS Program from the Zion Nuclear Station (a former nuclear generating facility), in support of extended service and current operations of the U.S. nuclear reactor fleet. The Zion Station is a decommissioned, two-unit, Westinghouse 4-loop PWR facility, with each unit capable of producing 1,040 MWe. The units were commissioned in 1973, permanently shut down in 1998, and placed into SAFSTOR (a method of decommissioning where a nuclear facility is placed and maintained in a condition that allows the facility to be safely stored and subsequently decontaminated to levels that permit release for unrestricted use) in 2010 [3]. Materials of high interest include low-voltage cabling, concrete, through-wall-thickness sections of the RPV, and other structures and components of interest to researchers evaluating aging management issues [5].

The RPV is a potentially life-limiting component in light-water reactors (LWR) because replacement of the RPV is not considered a viable option [2]. Researchers studying the effects of radiation on RPV materials have long been interested in evaluating service-irradiated materials to validate physically-informed correlations of transition-temperature-shift predication models [6] and ASTM E900 [7]. Valo et al. [8] published the first postmortem examination of a beltline weld from the first commercial VVER-440 RPV, Novovoronesh-1. The goal was to perform an integrity assessment of the vessel to evaluate the effects of long-term, low-flux irradiation since long-term experimental studies are not feasible. In 2008, Viehrig et al. [9] reported on the postmortem examination of another VVER 440 RPV beltline weld. Using the Master Curve approach, as described in ASTM E1921 [10], on trepans acquired from the Greifswald Unit 1 RPV, they demonstrated that  $T_0$  varies through the thickness of the weld. For these reasons, the acquisition of segments (Phase 1) of the Zion Station Unit 1 RPV, cutting the segments into blocks (Phase 2) from the previously well-characterized beltline weld [11 - 13] and base metal [14], and machining the blocks (Phase 3) into mechanical (Charpy, compact tension, and tensile) test specimens and coupons for microstructural (transmission electron microscopy, atom probe tomography, small angle neutron scattering, and hardness)

characterization are expected to provide critical data to assess current radiation damage models [2,3,6, 7].

## ZION UNIT 1 RPV

The Zion Unit 1 RPV consisted of the head, three ring or shell sections composed of semicylindrical plates with two vertical welds, and a bottom plate as shown in a preliminary segmentation plan (Fig. 1). It had a height without the head plate of ~ 419 inches (1,064 cm). The vessel wall has an inner diameter of 173" (439 cm) and thickness of 8.8" (22.4 cm) over the beltline region. Including the cladding, the reactor vessel weighed about 700,000 lbs. (317,515 kg) and had a total activity of ~ 400 curies.

An important consideration in the evaluation of which RPV segments to harvest is the circumferential fluence. As shown at the bottom of Fig. 2 and highlighted in blue, the peak circumferential fluence varies approximately by a factor of three over a 45° arc from the vertical weld positions to midway between the vertical weld positions. Based on this variation, the optimum region of beltline weld to harvest would be a section midway between the upper (intermediate shell) and lower (lower shell) vertical welds.

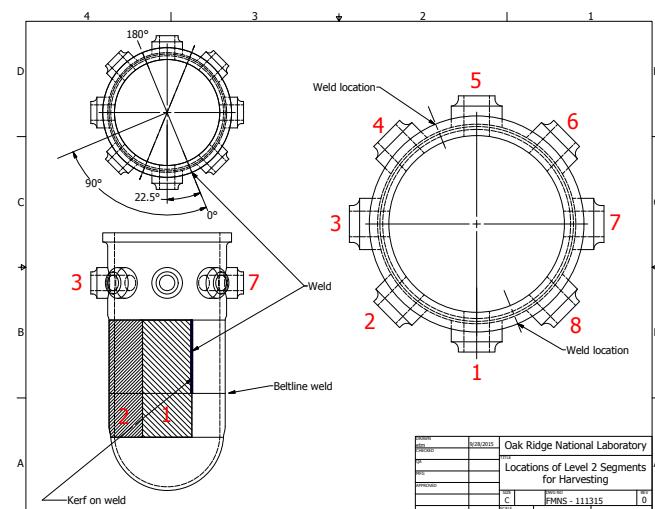


Fig. 1. Identification of Level 2 segments (1, 2, 5, and 6) collected as part of process to harvest Segment 1. Segment 1 contains the previously well-characterized base metal B7835-1 (intermediate shell) and WF-70 beltline weld [15].

## SEGMENTATION

The Zion Unit 1 RPV was cut into 16 segments over three levels with horizontal cuts in the nozzle section just above the intermediate shell and midway into the lower shell above the bottom plate. Level 1, which includes the inlet and outlet nozzles, was cut into eight 45° segments of 157.5" (400 cm) in height. Level 2 was also cut into eight 45° segments of 157.5" (400 cm) in height and 72.9" (185.2 cm) in length as measured from end to end of the outer diameter (Fig.1).

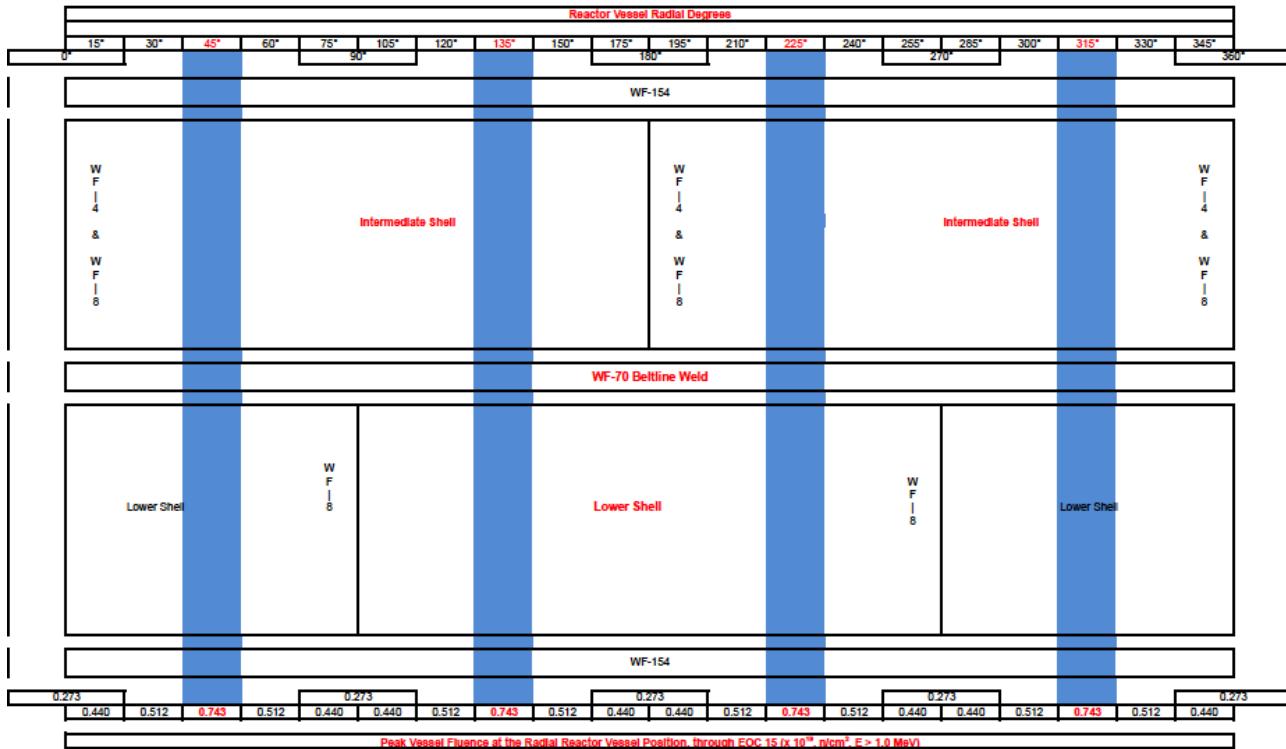


Fig. 2. Vessel fluence along the circumferential weld of the Zion Unit 1 RPV through End of Cycle (EOC) 13 ( $\times 10^{19}$  n/cm $^2$ , E > 1.0 MeV) is noted at the bottom of the diagram. Blue bands highlight the highest fluence regions [16].

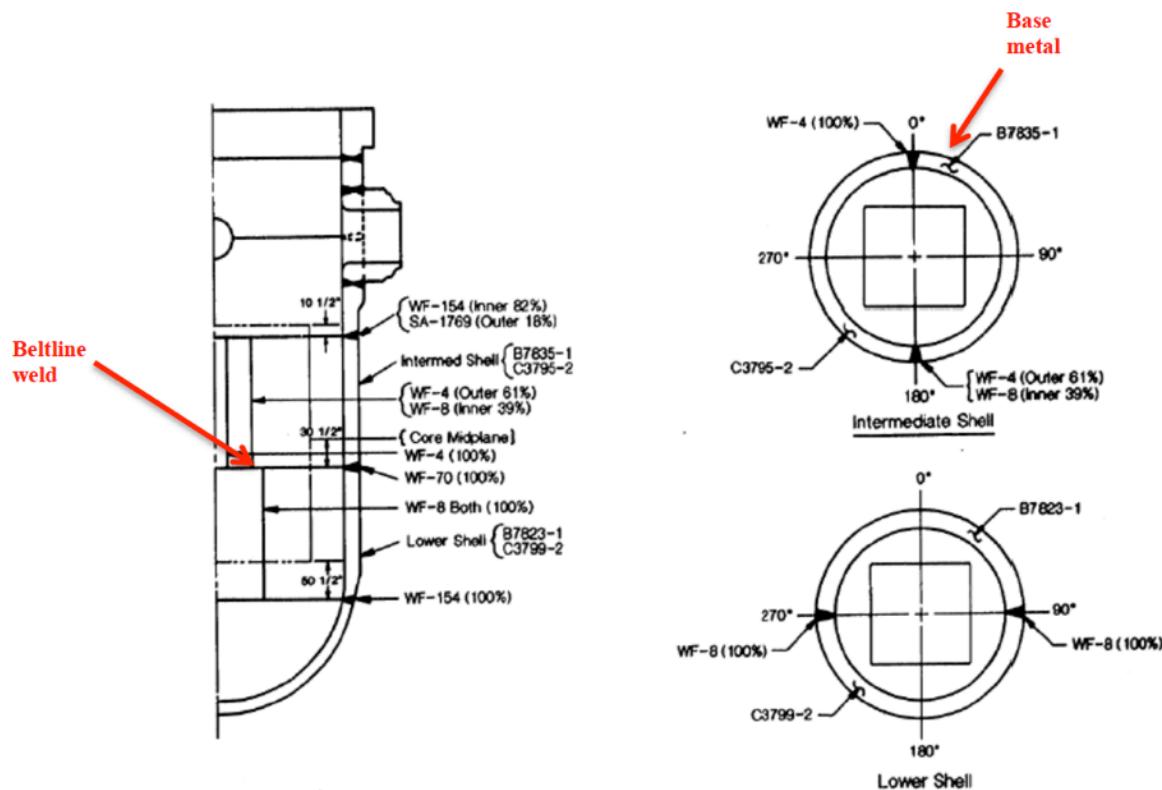


Fig. 3. Location of materials (beltline weld W-70 and base metal, B7835-1) used in the fabrication of the Zion Unit 1 beltline (intermediate shell to lower shell), two vertical welds above the beltline, and base metal heats [17].

Because the vessel could not be rotated  $22.5^\circ$  after the nozzle segments were cut due to the location of the overhead bridge, the level 2 segments, which include the intermediate shell and a portion of the lower shell and the WF-70 beltline weld, were cut along the same vertical lines as the nozzle cuts, i. e., at the two vertical welds of the intermediate shell, directly above the two vertical welds of the lower shell, and in the middle of the peak circumferential fluence (Figs. 1 and 2). Moreover, four segments (including segments 1 and 2) also contained the well-characterized base metal B7835-1 in the intermediate shell (Figs. 1 and 3). The beltline pieces cut from the vessel are 8.8" (22.4 cm) thick including a 5/16th inch (7.9 mm) stainless steel cladding on the internal surface. Each piece weighs approximately 28,000 lbs. (12,727 kg).



Fig. 4. Vertical cut using an oxy-propane torch of a Zion Unit 1 RPV Level 2 Segment [15].

### ZION RPV PHASE 1: HARVESTING

Zion Unit 1 Beltline Weld Segment 1, with dimensions of 157.5" x 72.9" x 8.8" (400 x 185.2 x 22.4 cm) and containing the previously well-characterized WF-70 beltline weld and base metal heat B7835-1 (Fig. 3), was cut from just below the circumferential weld between the nozzle section and the intermediate shell to just above the circumferential weld between the lower shell and the bottom plate using an oxy-propane torch (Fig. 4). The right edge (as viewed from the outer wall of the RPV) begins at the  $0^\circ$  / WF-4 vertical weld (Figs. 2 and 3). Segment 1 was removed using a gripper crane and positioned on the "down-ender" frame (Fig. 5) to allow proper positioning in the steel-shipping box. Segment 5 was loaded face up and the matching segment (opposite side segment: Segment 1) was loaded face down (Figs. 6 and 7) to provide "clam-shell" shielding for shipment to the EnergySolutions Memphis Processing Facility (Figs. 8 & 9) for cutting the segment into specimen blocks [1].



Fig. 5. Gripper crane and "down-ender" frame for repositioning segments into the shipment box [15].



Fig. 6. Loading the matching segment (opposite side segment) face up into the shipping container box [15].



Fig. 7. Loading Segment 1 face down onto to Segment 5 (opposite side segment) into the shipping container box [15].



Fig. 8. Closing the steel box containing opposite side segment pairs for shielding and eventual loading into the rail car [15].



Fig. 9. Crane for loading 2 steel shipping boxes holding 2 pairs of Zion Unit 1 RPV segments into the railcar that was transported to the Memphis Processing Facility [15].

## CUTTING SAMPLE BLOCKS FROM SEGMENT 1

As shown in Figs. 10 and 11, plans to cut seven blocks, varying in length from approximately  $5.7 \times 2.0 \times 8.8$  inches ( $144.8 \times 50.8 \times 223.5$  mm) to  $7.6 \times 3.0 \times 8.8$  inches ( $193.0 \times 76.2 \times 223.5$  mm) to  $11.25 \times 3.0 \times 8.8$  inches ( $285.8 \times 76.2 \times 223.5$  mm) and designated "F," "C," and "CF," using a diamond wire saw were developed to obtain sufficient through-vessel mechanical test specimens from the beltline weld and base metal to assess radiation embrittlement models and for possible annealing and re-irradiation experiments. The block designations correspond to the type of samples to be machined. For example, "F" blocks for fracture toughness, "C" blocks for Charpy and tensile specimens, and the "CF" block for alternating rows of fracture toughness and Charpy specimens.

The process of cutting sample blocks from the beltline weld and base metal above the weld sections of Segment 1 began with locating the exact position of the beltline weld and a determination of the slope across the outer diameter of the segment. Due to the high probability that the oxy-propane flame cut along the vertical direction may have obscured the weld, two 1" (25.4 mm) holes were drilled approximately 6" (152.4 mm) above and below the estimated position of the beltline weld and 2.75" (69.8 mm) from either edge of the segment. A wire saw was then placed through the holes to cut out two small sections in order to provide fresh surfaces to be etched to locate the exact position of the weld and to determine what the slope (if any) of the weld line is relative to the horizontal cut of the segment. Following the removal of the two small sections, chemical etching, using a 10% Nital (nitric acid / alcohol) solution, revealed the weld cross-section and position approximately 2" (50.8 mm) below the estimated position.

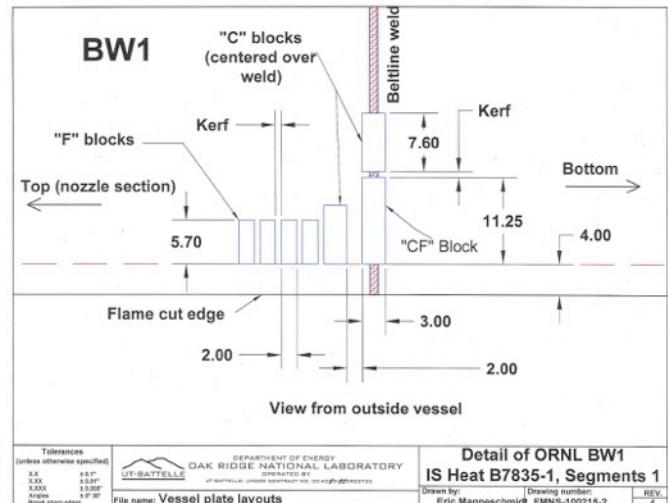


Fig. 10. Location of 5 base metal and 2 beltline blocks cut from Zion Unit 1 RPV Segment 1.

Once the position and slope of the weld were determined, a thin metal template was placed over the segment and aligned with the beltline weld (Fig. 12). The template was designed based on the location of the blocks relative to the beltline weld and base metal and the location of the 13 holes used to thread the wire to cut the blocks, including the kerf, along the lines as seen in Fig. 11. The holes were used for inserting the diamond wire to cut the 2 beltline weld and 5 base metal blocks from Segment 1 as shown in Fig. 13. With the completion of Cut 11 (Fig. 13), the CF block was removed from Segment 1 (Figs. 14 and 15) using a lift magnet. Similarly, the remaining blocks were successfully cut and lifted out from Segment 1.

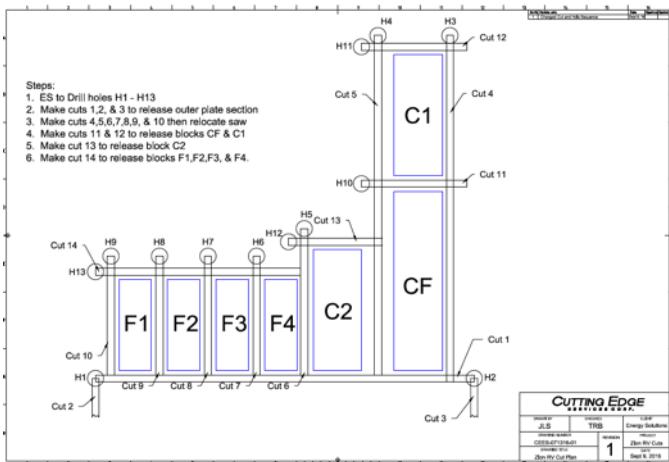


Fig. 11. Cut plan (based on Fig. 10) noting the blocks (blue outline), the wire saw access holes and cut [15].



Fig. 12. A metal template was aligned with the beltline weld (chalk line) and holes for threading the diamond wire [15].



Fig. 13. Wire saw cutting parallel to the beltline weld [15].



Fig 14. Lift magnet used to remove the cut blocks [15].



Fig. 15. CF block with orientation marked (Y axis is perpendicular to the beltline weld with the arrow pointing to the top of the segment and the X axis is parallel to the weld with the arrow pointing to the right of the segment) [15].

Upon completion of the cutting phase of the project, the seven RPV blocks cut from the Zion Unit 1 RPV Segment 1 at the MPF, were loaded into two 55-gallon drums along with individual lift magnets and the drums placed into a B-25 box with 1" steel shielding beneath and 1" steel shielding around the side of the drum. Sand was added into the B-25 box as a filler material and provided additional shielding and box transported to BWXT, Lynchburg, VA for machining the mechanical test specimens, coupons for chemical characterization and microstructural characterization samples.

## MACHINING SAMPLES FROM SEGMENT 1 BLOCKS

Prior to receiving the seven Segment 1 blocks, a detailed review of the machining plans was performed, including the removal of the cladding, the block orientation identifying the location of the high fluence edge, how to best fit the cut plan into the actual dimensions of the cut blocks, unique sample identification numbers, surface finish, the final identification of the C and F reserve blocks, block cutting order (2 F blocks, followed by C2 and CF blocks) and the estimated schedule for the project.

Following receipt of the Zion Unit 1 RPV sample blocks at BWXT the seven blocks were inspected and radiation levels verified, and the stainless steel cladding removed. This simple process was performed with an electrical discharge machining (EDM) using a 0.010" (.25 mm) diameter wire. The cut with the kerf created a straight ~0.1" (2.5mm) cut above the apex of the cladding and approximately 0.35" (8.9 mm) into the block from the outside of the cladding. The cladding thickness was measured manually to verify that no remnant pieces remained on the RPV steel block. A rough sketch of the cut is shown in Fig. 16.

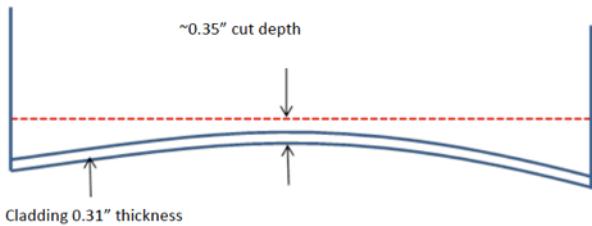


Fig. 16. Sketch of cladding cut off from Segment 1 blocks [18]

## MACHINING THE BASE METAL F BLOCKS:

As shown in Fig. 17, a total of 56 compact tension specimens were machined from each of two F blocks. The base metal F block layout is such that 4, 0.5T C(T) specimens were machined from each row oriented along the circumferential direction of Segment 1 (parallel to the beltline weld) and 14 specimens were machined through the thickness of the vessel segment.

## MACHINING THE BASE METAL C2 BLOCK:

As shown in Fig. 18, a total of 255 Charpy-size (10 x 10 x 55 mm) bars were machined from base metal block C2. Fifteen specimen bars were machined from each row oriented along the circumferential direction of the segment (parallel to the beltline weld) and 17 bars were machined through the thickness of the vessel segment. From those 255 Charpy-sized bars, 239 CVN specimens (10 x 10 x 55 mm) were machined. The remaining 16 Charpy-size bars were machined into 128 SS3 tensile specimens (25.4 x 5 x 0.76 mm) and 64 coupons (10 x 10 x 0.5 mm) for chemical and microstructural characterization (Fig. 19).

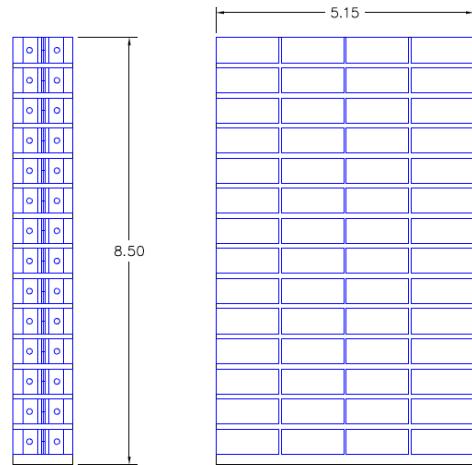


Fig. 17. Layout for machining compact tension specimens from "F" blocks from RPV Segment 1.

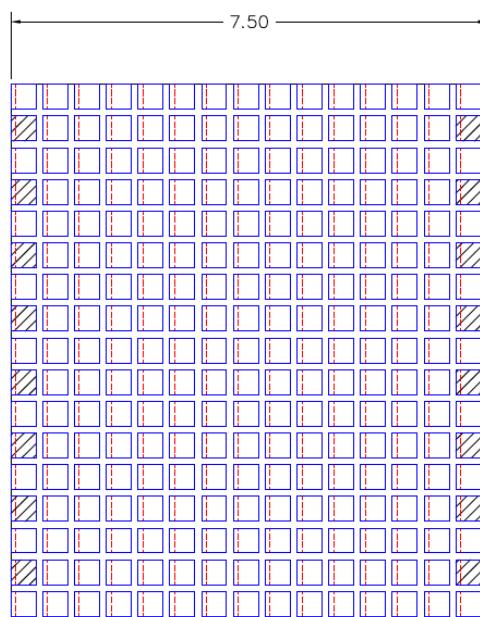
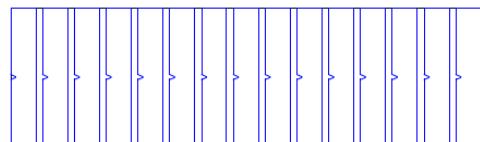
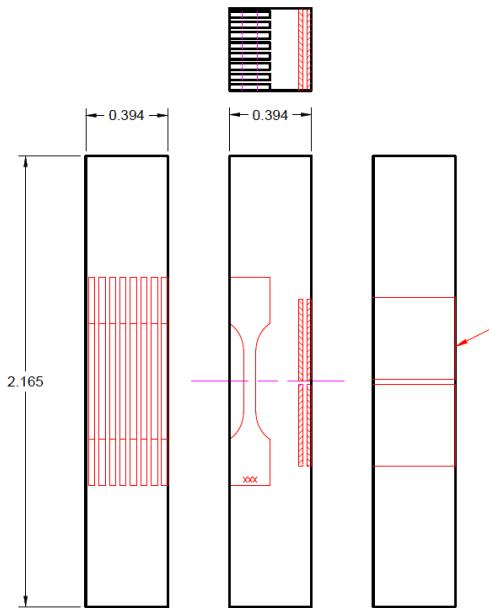


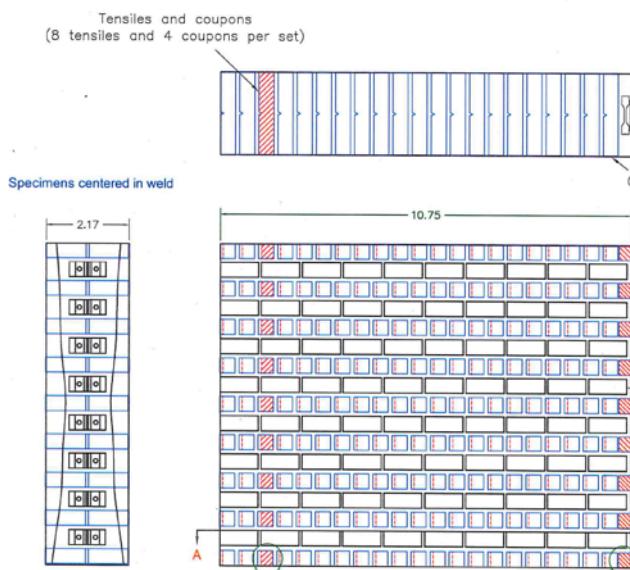
Fig. 18. Layout for machining Charpy and tensile specimens from the block C2. Hatched blocks were machined into SS3 tensile specimens and coupons.



**Fig. 19.** Charpy-sized bar machined into 8 SS-3 tensile specimens and 4 chemical and microstructural coupons/

#### MACHINING THE BELTLINE WELD CF BLOCK

Prior to machining the beltline weld CF block, chemical etching, using a 10% Nital solution, identified the centerline and cross-section of the weld. This was critical to insure that the V-notch of the Charpy specimen, the gauge section of the SS3 tensiles, and the 0.4T C(T) notches were centered on the beltline weld as shown in Figs. 20 and 21.



**Fig. 20.** Detailed CF block layout of alternating rows of 0.4T C(T) and Charpy specimen positioned over the WF-70 beltline. Hatched blocks were machined into SS3 tensile specimens and chemical and microstructural coupons.

Following the etching, twenty-two specimen bars were machined from alternating rows oriented in the circumferential direction of the segment (aligned with the beltline weld) and 9 bars were machined through the thickness of the vessel segment. From those 198 Charpy-sized bars, 180 CVN specimens (10 x 10 x 55 mm) were machined. The remaining 18 Charpy-size bars were machined into 144 SS3 tensile specimens and 72 coupons (10 x 10 x 0.5 mm) for chemical and microstructural characterization. Finally, 80 compact tension specimens were machined from the CF block. Specifically, ten 0.4T C(T) specimens were machined from alternating rows oriented in the circumferential direction of Segment 1 (aligned with the beltline weld) and 8 specimens were machined through the thickness of the vessel segment.

#### ZION RPV PHASE 3 MACHINING SUMMARY:

A summary of the revised machining plan for one (1) base metal "C" block, one (1) beltline weld "CF" block, and two (2) base metal "F" blocks is listed below.

##### Summary of samples machined from base metal block C2:

**239** = [(17x15)-16] Charpy specimens

**128** = (16 x 8) SS3 tensile specimens from Charpy-sized bars

**64** = (2 x 2 x 16) coupons (chemical and microstructural characterization)

##### Summary of samples machined from weld block CF:

**180** = (22 x 9 -18) Charpy specimens

**144** = (2 x 9 x 8) SS3 tensile specimens from Charpy-sized bars

**72** = (2 x 2 x 18) coupons (chemical and microstructural characterization)

**80** = (10 x 8) 0.4T C(T)

##### Summary of samples machined from base metal F blocks:

**112** = 56 (4 x 14) 0.5T C(T) x 2 "F" blocks

##### Summary of samples machined by type:

**419** Charpy specimens (239 base metal + 180 weld)

**272** SS3 tensile (128 base metal + 144 weld)

**136** Coupons (64 base metal + 72 weld)

**192** Fracture toughness (112, 0.5T base metal & 80, 0.4T weld)

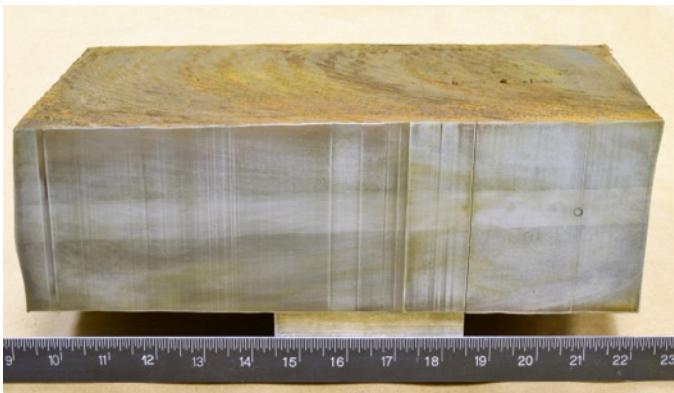


Fig. 21. WF-70 Beltline weld revealed after removing the cladding and etching block CF [18].

#### THROUGH WALL ATTENUATION STUDY OF WELDS AND BASE METAL TEST PLAN:

The through-wall mechanical test specimens, including Charpy V-Notch (CVN), tensile, and fracture toughness, machined from the beltline WF-70 weld and based metal heat B7835-1, having a peak fluence  $< 1 \times 10^{19} \text{ n/cm}^2$  will be tested to evaluate the change in mechanical properties as a function of depth (neutron fluence attenuation).

In addition to specimens for mechanical testing, through-thickness chemical characterization (Cu, Ni, Mn, Mo, P, Si, Cr), hardness distribution, and various microstructural characterization techniques such as Atom Probe Tomography (APT), Small Angle Neutron Scattering (SANS), and Positron Annihilation Spectroscopy (PAS) will be performed.

The test plans are as follows:

1. Determine the through-thickness variation in chemical composition of the weld (especially Cu).
2. If the chemical composition, especially the Cu content, is relatively uniform, perform CVN and tensile tests and compare results with surveillance results.
3. Perform CVN, tensile, hardness, and  $K_{Ic}$  testing through thickness to evaluate attenuation effects.
4. Microstructural characterization (Atom probe, SANS, SEM, TEM, and hardness) will be performed through thickness to evaluate attenuation effects using specimens obtained from  $10 \times 10 \times 0.5 \text{ mm}$  coupons.
5. Similar testing (3 and 4) through the thickness of base metal will also be performed in collaboration with the Central Research Institute for Electric Power Industry (CRIEPI) as part of the Civilian Nuclear Working Group (CNWG) program with Japan.

6. Thermal annealing of these RPV materials may also be performed to compare with the same weld metal (WF-70) previously irradiated in test reactors & annealed.

#### SUMMARY

This report documents the acquisition and shipment, by rail, of four segments of the Zion Unit 1 RPV to the EnergySolutions Memphis Processing Facility (MPF) for eventual laboratory testing. Specifically, only Segment 1 of the Zion Unit 1 RPV containing well-characterized base metal B7835-1 heat and a section of the well-characterized WF-70 beltline weld (between the lower and the intermediate shells) was harvested and shipped for cutting into blocks for machining into mechanical and microstructural characterization samples at a third vendor. Data from RPV surveillance specimens containing similar WF-70 weld materials are available in the literature for a comparison of hardening and changes in fracture toughness and microstructure [11-13]. Similarly, data from surveillance specimens containing B7835-1 plate material are available for a comparison of hardening and changes in fracture toughness [14]. Access to service-irradiated RPV welds and plate sections will allow through-wall attenuation studies to be performed, which will be used to assess current radiation damage models [2, 3, 6] and ASTM E900-15 [7].

#### NOMENCLATURE

Reactor pressure vessel (RPV); Oak Ridge National Laboratory (ORNL); Atom Probe Tomography (APT); Small Angle Neutron Scattering (SANS); Charpy V-notch (CVN); Memphis Processing Facility (MPF); Light Water Reactor Sustainability (LWRS).

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