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Specimen Machining for the Study of the Effect of Swelling on CGR in PWR Environment

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ABSTRACT

This report describes the preparation of ten specimens to be used for the study of the effect of swelling on the propagation of irradiation-assisted stress corrosion cracking cracks. Four compact tension specimens, four microscopy plates, and two tensile specimens were machined from an American Iron and Steel Institute (AISI) 304 material that was irradiated up to 33 dpa. The specimens had been machined to allow a study of materials with 3.7% swelling and <2% swelling.

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ACRONYMS

AISI	American Iron and Steel Institute
CGR	Crack Growth Rate
CT	compact tension
dcpd	direct current potential drop
EBR-II	Experimental Breeder Reactor
IASCC	irradiation-assisted stress corrosion cracking
TEM	transmission electron microscope

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1. INTRODUCTION

To predict the susceptibility to irradiation assisted stress corrosion cracking (IASCC) of high fluence materials, it is necessary to estimate how features appearing at high fluence may affect IASCC. One of those features is void swelling. In a material with voided microstructure, one can expect the high density of voids (both intergranular and intragranular) to affect the propagation of a stress corrosion crack. Intragranular void may affect local stress and deformation, and the presence of intergranular voids and bubbles may affect grain boundary cohesion and any diffusion process on the grain boundary. To study the effect of void swelling on IASCC crack propagation, it was decided to test a highly irradiated material whose irradiation conditions generate a swelling gradient through the component. This report describes the material selected and the machining of the various specimens to be used in thus study.

2. MATERIAL HISTORY

The material that was cut from a thick hexagonal block made of American Iron and Steel Institute (AISI) 304 stainless steel that served in one of the reflector assemblies in the Experimental Breeder Reactor (EBR-II) fast reactor. The chemical composition is Fe-12.26Cr-8.81Ni-1.57Mn-0.43Si-0.056C-0.027P-0.03S wt%. The material was not annealed before irradiation and microstructure characterization of archive material suggests 5% cold work at the center of the block. The doses received and irradiation temperatures are evaluated from the temperature and dose calculated for the encasing duct using reactor physics and heat transfer calculation (Bond 1999, Garner 2006, Garner 2007). This block was part of a series of five blocks that were in the reactor for 13 years: 4.5 years in Row 8, and 8.5 years in Row 16. However, most of the dose received (97%) was received in Row 8. The material comes from Block 3 that was in the center of the core and received the most dose (from 33 dpa for the face located toward the core center to about 22 dpa for the opposite face). More specifically the material comes from a 0.5-in.-thick coin labelled 3F3 initially located toward the center of Block 3. The time average temperature was 390°C; however, due to gradient gamma heating, there was an off-center peak in temperature inside the block as illustrated in Figure 2. The maximum average temperature inside Block 3 is estimated to be about 460°C (Garner 2014). Such temperature gradient is expected to have led to swelling gradient. Ultrasonic time-of-flight measurements performed on Coin 3F3 confirm an off-center swelling peak, with a maximum at about 3.7% swelling and a minimum <2% (Garner 2013, Garner 2014).

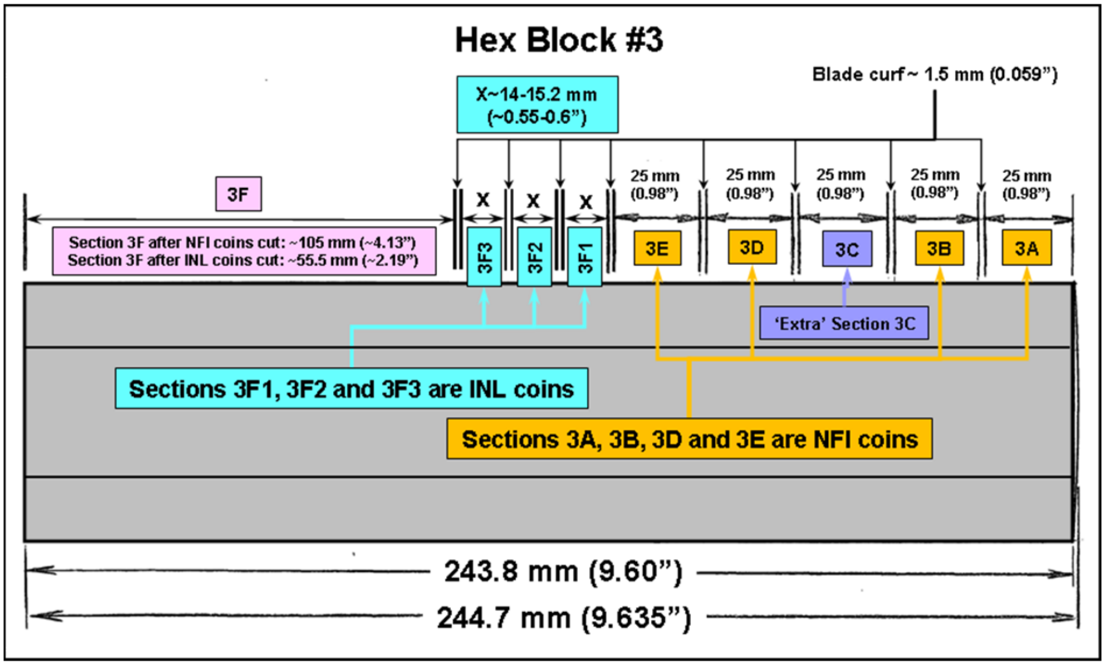


Figure 1. Schematic of Block 3 from which the material used for this study (Coin 3F3) was cut.

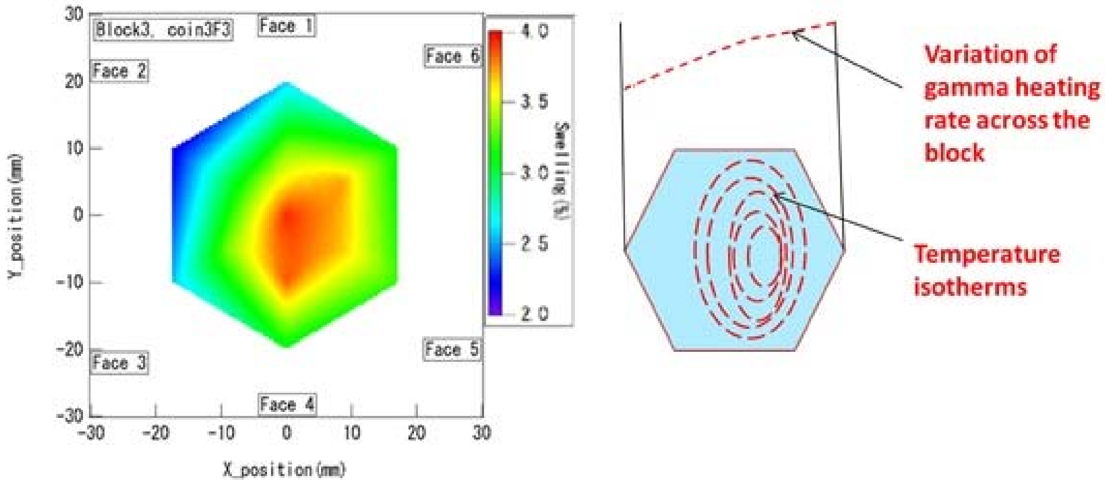


Figure 2. Temperature distribution across the block (Garner 2014).

3. MACHINING

3.1 Objectives

The machining layout was designed so that two pairs of compact tension (CT) specimens, two pairs of microscopy plates, and two tensile specimens were generated.

The location for machining was determined so, for each pair of CT specimens, the cracks will grow in a material with similar swelling. Two specimens were machined so that the cracks will grow in a material with 3.7% swelling and two specimens were machined so that the cracks will grow in less than 2% swelling. At this point the percent swelling was determined by ultrasonic technique. The orientation of the CTs with regard to the component was the same. In addition to machining the specimens, shorts leads to be used for dcpd (direct current potential drop) were connected to the specimens.

It is critical to know the microstructure (swelling) of the material in which the crack will propagate. As the microstructure of the original material is not uniform (which allows us to have a swelling gradient across the component), we relied on work previously done with nondestructive examination to determine the location of the various areas of interest. However, as such measurements were indirect and with any machining process come a level of uncertainty (possible misalignment, location of cuts…). Therefore, thin plates, initially adjacent to the material used to machine the CT specimens and with known orientation were machined. Those plates are referred to as "microscopy plates" as they will be used to generate transmission electron microscope (TEM) disks along the area directly adjacent to the crack path. Analysis of those disks will permit confirmation of the microstructure in which the cracks will grow.

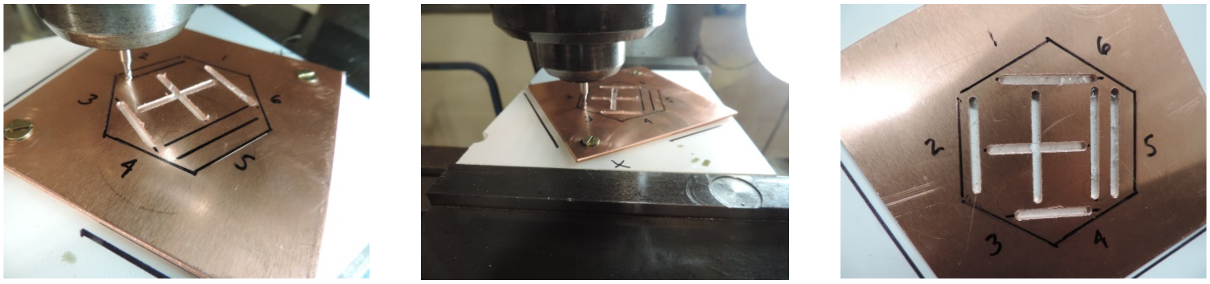
In addition, two tensile specimens were machined. Based on the mapping of swelling, the gauge area was machined in a material with an expected 2% swelling.

3.2 Preparative: Fabrication of a Template for Marking Specimen Blank Position on Hex Coin 3F3

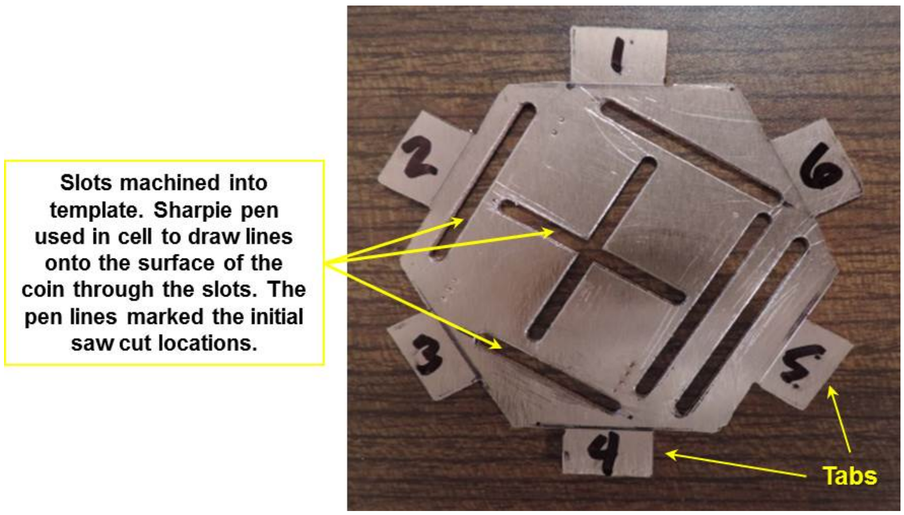
Prior to sectioning Coin 3F3, it was necessary to accurately mark the surface of the coin inside the hot cell to identify all saw cut locations. To mark the saw cut locations on the coin surface, a custom thin sheet template that fit on the top surface of the coin was fabricated outside of the cell. The template included "side tabs," which were bent down over the edges of the coin to secure the template in the correct position prior to marking the coin surfaces in-cell with a permanent pen.

Outside the cell, slots were machined in the thin sheet at locations corresponding to the saw cut locations. The template was then transferred to the inside of the hot cell and fit on the top surface of the coin. The template side tabs were bent down over the edges of the coin to secure the template in the proper position on top of the coin. A permanent pen was then used in the cell with the tip of the pen placed inside the machined template slots. Lines were draw onto the surface of the coin corresponding to the initial cut locations. After the permanent pen marks were drawn on the coin, the template was removed from the coin. The coin was then punch marked using a Westinghouse modified automatic spring-loaded center punch to document the CT blanks that were subsequently cut from the coin. In particular, the location of each CT specimen was punched with 1, 2, 3, or 4 punch marks corresponding to CT blanks CT1, CT2, CT3, and CT4. This punch marking was necessary to track the location and orientation of each CT specimen, which would subsequently be machined from the coin. Figure 3 provides photographs taken during various stages of fabrication of the marking template and the template after fabrication was completed.

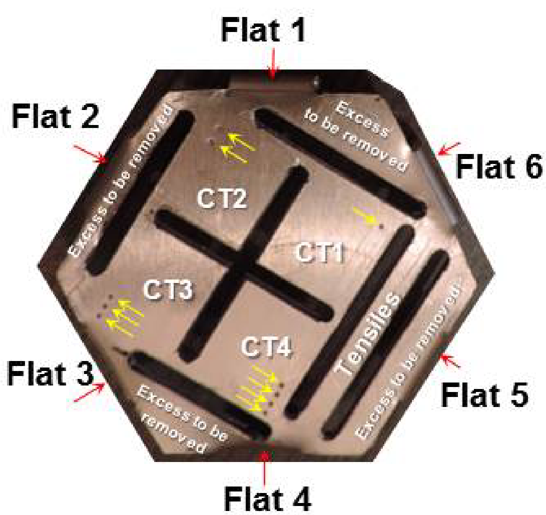
The blanks used to later machine the 0.25-T CT specimens and the underlying corresponding microscopy plates were cut together from the 0.5-in.-thick coin. The blanks were cut from the coin using the same Westinghouse custom-modified portaband horizontal/vertical bandsaw using a staggered-tooth bimetal bandsaw blade as that used to blank the tensile specimens. The rough-cut blanks were approximately 20% oversized from the final machined specimen dimensions. The location/orientation of each blank was maintained after separation from the coin.



(a) Fabrication of the thin sheet template.



(b) Completed template with side tabs.



(c) Completed template with side tabs folded under (marked with red arrows) and punch marks (marked with yellow arrows) documenting the position and orientation of each compact tension specimen.

Figure 3. Fabrication of thin sheet template with side tans for marking the cut locations onto the surface of the Hex Coin 3F3.

3.3 Description of the Machining Steps for Machining of the CT Specimens

There are two pairs of identical specimens. The first pair, composed of specimens CT1 and CT4, was machined to grow the crack in a material with 3.7% swelling. The second, composed of specimens CT2 and CT3, was machined to grow a crack in less than 2% swelling. Each specimen, marked for identification, is a 0.25-T CT specimen machined according to the schematic presented in Figure 4. The location of the specimen with respect to the initial coins is show in Figure 5. The process was as follow:

- Locating the blanks in the initial coin
- Cutting of each blank
- Machining the blank to dimension
- Machining of the notch
- Drilling of the loading pin holes
- Machining of the grove
- Installing the connecting plugs (described in more details later).

The results of the machining test various steps are presented Figure 6. In this figure the material used was an unirradiated material used for practice. Considering the value of the material and the difficulty to machine a 0.25-T CT specimen, extensive practice trials were performed on an unirradiated standard 304 stainless steel material. The trials focused on the operation of the newly acquired and recently custom-modified Inteletek CNC milling machine. This milling machine had not yet been installed in the cell so it could be used for practice trails outside of the cell. Once practice trials were completed, the milling machine was installed into the hot cell and the same machine was used for machining the CT specimens from Hex Coin 3F3.

In the hot cell the machining of the CT specimens started after the blank was sectioned, such as the top portion will be used to machine the specimen and the bottom to machine the microscopy plates. The B dimension (thickness=0.25 in.) was machine finished using a custom-modified Inteletek CNC milling machine. The milling machine is modified with pneumatic vices for gripping specimens. The jaws of the vices were modified to accommodate the small size of the .25-T CT specimens. In addition, three different-sized flat and square spacers were fabricated to raise the blanks up to the appropriate location for milling and drilling. New 5-flute .75-in.-diameter solid carbide milling tools were used to mill the blank surfaces. Multiple passes were used on the two opposing blank faces to produce flat and parallel surfaces and to reduce the B dimension to 0.25 in. Each pass removed approximately 5 to 10 mils from the surface. The face that contained the punch marks was immediately repunched after the punch marks were removed by the milling operation. The other sides of the specimen (corresponding to the 0.6 in. and 0.625 in. in Figure 4) were then milled to final specimen dimensions using the same process as described above for the B dimension.

The notch depth on each specimen was then partially cut using a 0.025-in.-thick CBN cutting blade, which cuts approximately 80% of the notch depth. This CBN cutting blade was used to initially remove a significant amount of material from the notch area. The final notch depth and notch radius were cut using a solid carbide, 0.025-in.-thick, 32-teeth blade, which had been ground to a 30 degrees, including an angle and a maximum tip radius of 0.003 in. The specimens were then side grooved with a full-radius custom-ground 0.025-in.-thick solid carbide blade to a depth of 0.0125 in.

The pin holes were then located, center drilled, and final drilled using a Number 29 (0.136 in.) diameter drill. The machine-finished specimens (without dcpd leads and before final cleaning) are presented Figure 7. The respective dose rate of each specimen is presented in Table 1.

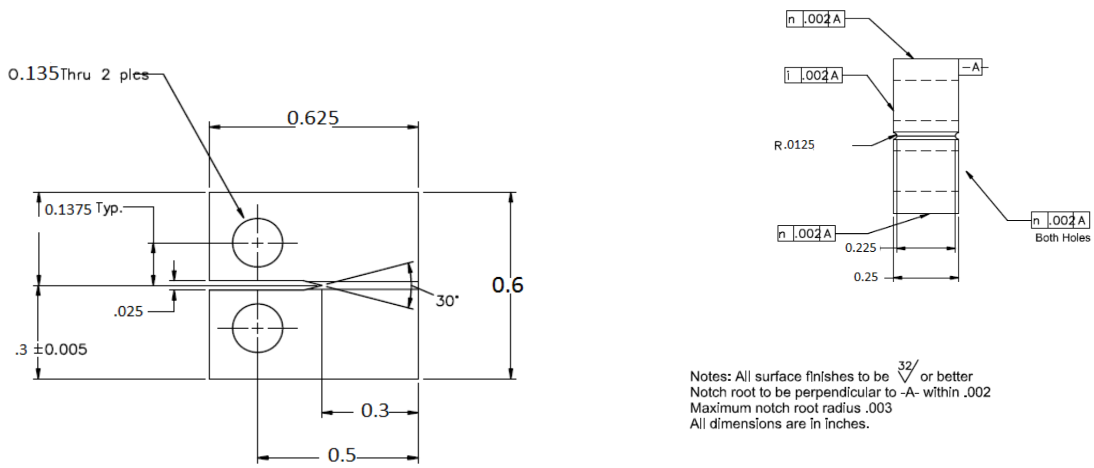


Figure 4. Schematic of the 0.25-T CT specimens machined.

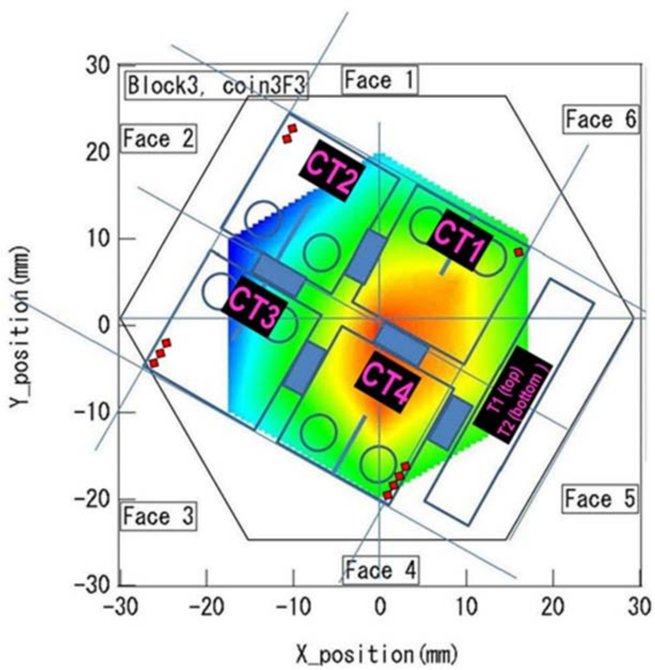


Figure 5. Location and orientation of the four CT specimens (CT1, CT2, CT3, and CT4) as they were cut in the hexagonal coin. T1 and T2 show the location of the tensile specimens.

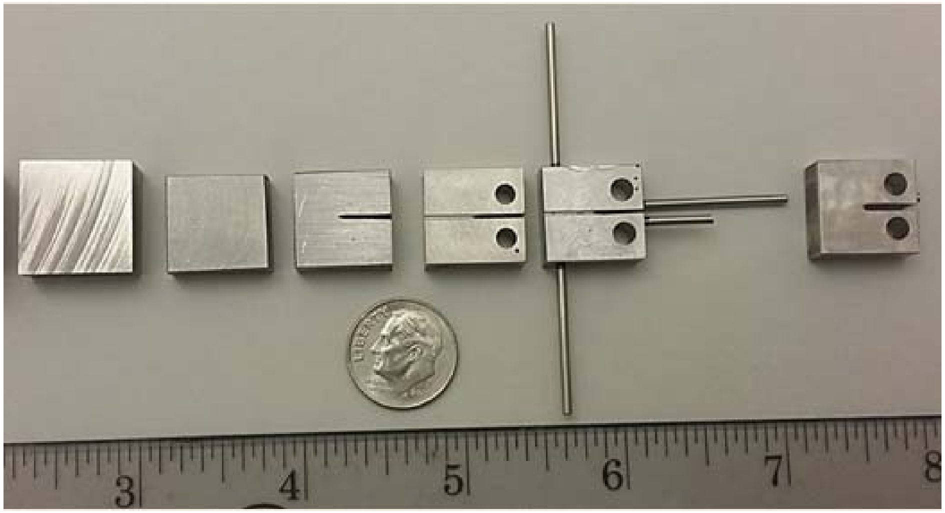


Figure 6. Progression of the machining of the CT specimen and installation of the connecting plugs. The material in this picture is the unirradiated materials used for practice run.

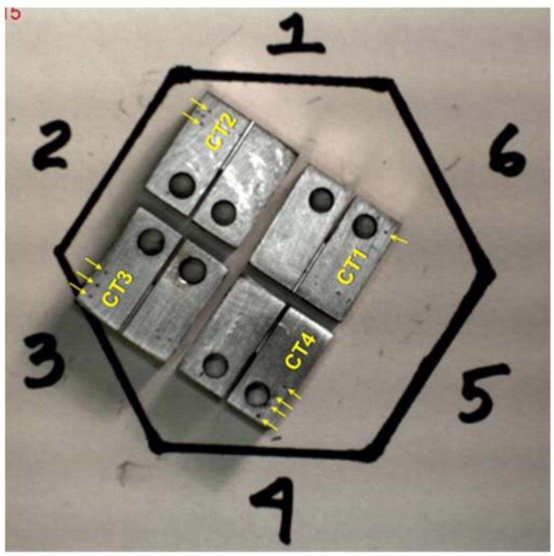


Figure 7. Machine-finished CT specimens prior to final cleaning and connection of the dcpd leads (ID/punch marks are highlighted with yellow arrows).

Table 1: Compact Tension specimen IDs and dose rate information

Specimen ID	Dose Rate at 24 inch [a]	Dose Rate at 12 inch [b]	Dose Rate at 1 inch [b]
	mR/hr	mR/hr	mR/hr
CT1	40[a]	160[c]	23,040
CT2	45[a]	180[c]	25,920
CT3	48[a]	192[c]	27,648
CT4	42[a]	168[c]	24,192
Note a: Measured at 24" using Bicron Surveyor 2000 Meter Note b: Calculated from 24” value using http://www.radprocalculator.com/InverseSquare.aspx			

3.4 Description of the Installation of the Leads for dcpd Reading

One of the challenges associated with performing a crack growth rate test using dcpd technique to measure crack length is the connection of the dcpd leads. A poor connection will lead to poor data quality. Connecting the leads to the specimens when the specimen is installed in the testing jigs is challenging (limited access, difficulty to locate repetitively the exact location for connecting the leads) and an error (in the location, poor connection) may lead to complications (removal of the specimens, sanding of the area to be use for connecting the leads, etc.). For this project, it was decided that short leads will be connected to the specimens as part of the specimen machining. This was done in three steps. First, shallow holes were drilled at the required location at a total length of 0.062 in.; the flat parallel section to be filled with the plug is 0.05 in. deep and a diameter of 0.0625 in. Then, a piece of unirradiated steel (316 type) of a diameter of 0.0625 in. was press fit in the hole and cut such as 0.005 to 0.015 in. of this material stick out of the surface of the specimen. The plug extending out of the surface is required to get the best contact between the edge of the plug and the base metal as during spot welding of the lead onto the plug, very small portions of the edge of the plug will melt and flow down onto the surrounding base metal surface, which will provide excellent electrical contact between the plug and the base metal. Sketches showing the installation of those plugs are shown in Figure 8.

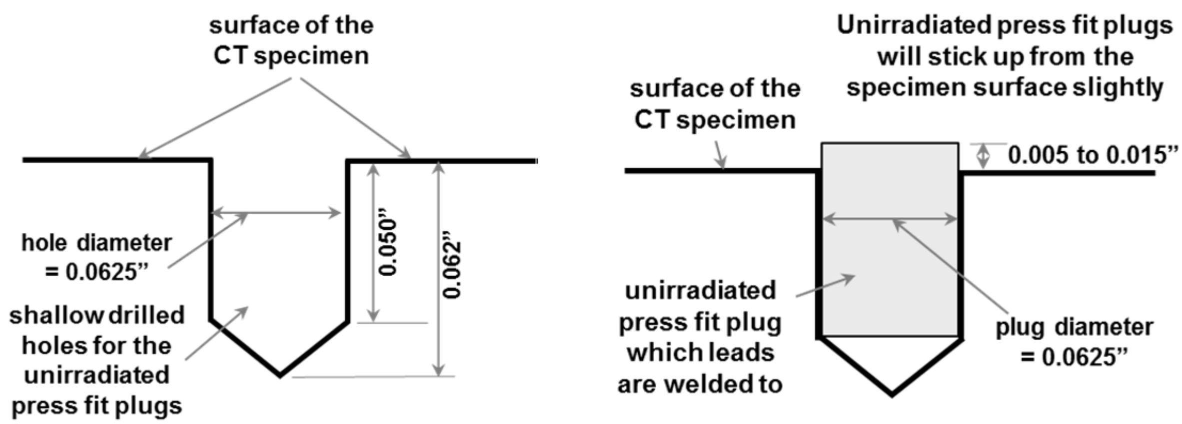


Figure 8. Sketches showing the installation of the plug for a dcpd lead connection.

The leads connected to those plugs are 32/1000-in. platinum leads for the current and 20/1000-in. platinum lead for the potentials. Two leads were used for current and two leads for potential measurements. The location of the leads can be been in the schematic in Figure 9. Although the schematic describes two pairs of potential lead, only one pair was installed to those specimens. Figure 10 shows the four CT specimens with the leads connected.

With such approach, the connection in the test cell will be done by connecting the specimen leads to the leads installed in the autoclave.

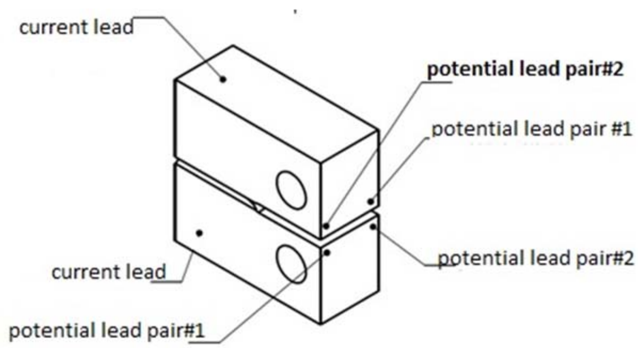


Figure 9. Schematic describing the position of the leads.

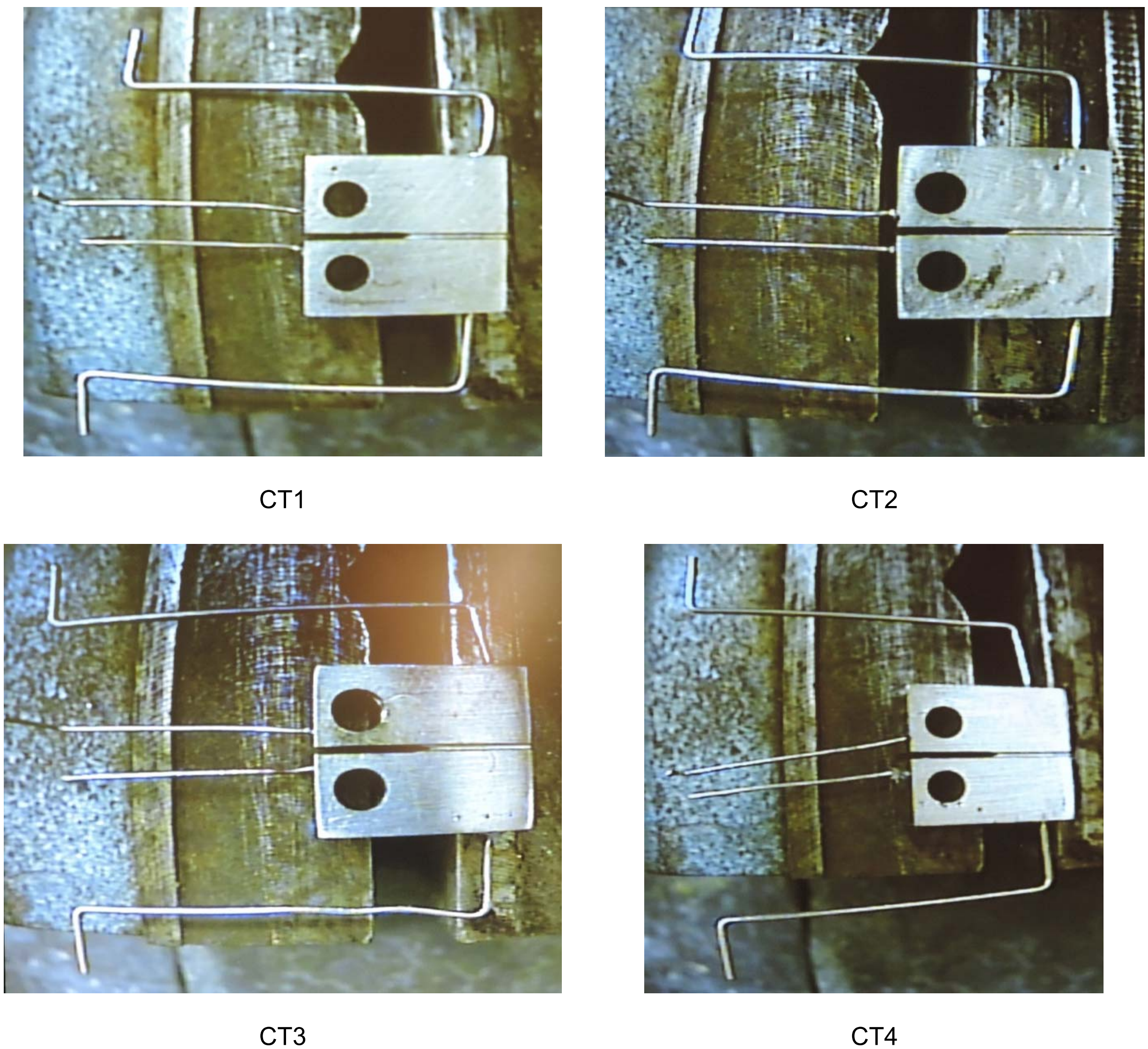


Figure 10. The four CT specimens after current and potential lead welding completed. Dimensions of photographs may appear distorted because photographs were taken from a video monitor.

3.5 Machining of the Microscopy Plates

Each microscopy plate was then separated from the four corresponding CT specimen blanks by cutting using a slow speed 5-in.-diameter wafering saw with a 0.020-in.-thick silicon carbide wafering blade. The location/orientation of each microscopy plate was maintained after separation from the larger blank. Each plate was punch marked using a Westinghouse-modified automatic spring-loaded center punch. Each plate was punched with 1, 2, 3 or 4 punch marks representing the specimen identification number (i.e., number 3F3-1B, 3F3-2B, 3F3-3B, or 3F3-4B). Each plate was stored in an individually labeled small screw cap aluminum container.

The thickness of the specimens, slightly higher than expected, is not expected to impact the preparation of the specimens given the low activity of the materials. The dimensions of the microscopy plates and respective dose rates are presented in Table 2.

Table 2. Specimen IDs of the microscopy plates with dimensions and dose rate information.

		Actual Completed Machining Dimensions						Dose Rate Information	
Specimen	Specimen ID	Measured Side Length, in.	Measured Side Length, in.	Measured Thickness, in.	Actual Volume, in.^{3}	Approximate Dimensions Defined in Scope of Work (in. x in. x in.)	Approximate Volume Based on Approximate Dimensions Defined in Scope of Work (in.^{3})	Measured Dose Rate at 12 in. (mR/hr)[a] (measured on 3/30/2015)	Calculated Estimated Dose Rate at 1 in. (mR/hr)[b]
1	3F3-1B	0.692	0.703	0.212	0.10	~0.6 x ~0.6 x ~(0.125 to 0.150)	~0.05	203	29,232
2	3F3-2B	0.712	0.677	0.189	0.09			200	28,800
3	3F3-3B	0.789	0.710	0.202	0.11			247	35,568
4	3F3-4B	0.656	0.796	0.201	0.10			197	28,368

a. Measured at 12 in. using Thermo Scientific Teleprobe FH40
b. Calculated from 12 in. measured using http://www.radprocalculator.com/InverseSquare.aspx.

3.6 Machining of the Tensile Specimens

The tensile blanks were rough cut from the hex coin in-cell using a Westinghouse custom-modified portaband horizontal/vertical bandsaw using a new staggered tooth bimetal bandsaw blade. The rough-cut blanks were approximately 20% oversized from the final machined specimen dimensions (Figure 11). The location/orientation of each blank was maintained after separation from the coin.

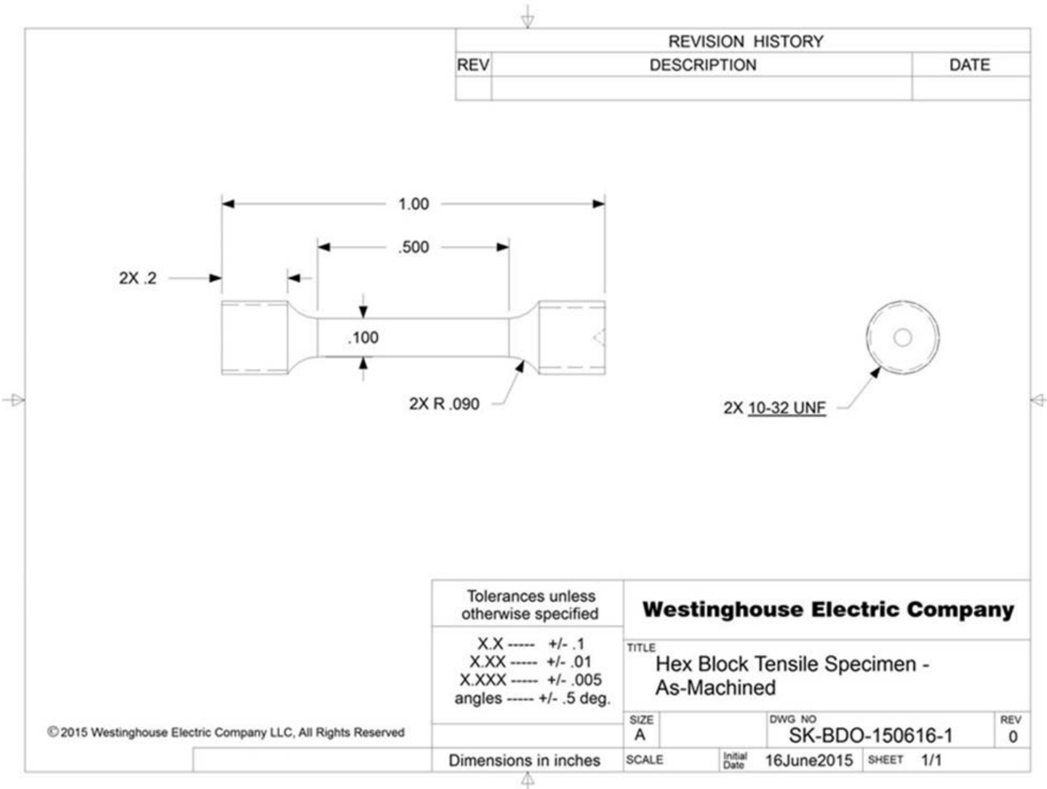


Figure 11. Tensile specimen machining drawing.

The two blanks were then turned in a Westinghouse custom-modified in-cell EMCO CNC lathe using a new turning tool with replaceable carbide inserts. The blanks were turned down to a rod with a diameter of 0.190 in. The 10-32 threads were cut into the grip section of each end of the two blanks using the in-cell lathe and a threading tool bit while the 0.190-in.-diameter rod was turning in the lathe.

The tensile reduced section was then machined using a custom-designed and fabricated Westinghouse plunge carbide tool with a 0.090-in. radius. This plunge tool allowed for the machining of the reduced section and both shoulder radii with one equipment/fixturing/tooling set-up. Multiple passes were used and the plunge tool was moved in both the x and y direction as the rod was turning in the lathe. Westinghouse has previously used this plunge carbide tool method for successful in-cell machining of tensile specimens.

The reduced section of each tensile specimen was subsequently polished using 320-grit strips of silicon carbide grinding paper. The strip width was approximately 0.5 in. and two manipulators were used to hold each end of the strip. The strips were lightly touched against the reduced section of the rotating specimen to obtain an acceptable surface condition.

The specimens were measured to confirm final machined dimensions and stored in individually labeled small aluminum screw cap vials. The specimen identifications are 3F3–T and 3F3–B where T represents the specimen from the top surface of the coin and B represents the specimen from the bottom surface of the coin. The specimen final dimensions were checked (Figure 12). The length of the specimens is 1.19 in., a little higher than the target length but, given that the extra length is in the threaded area, it will not affect any test. The specimens final dimensions and dose rate are presented in Table 3.

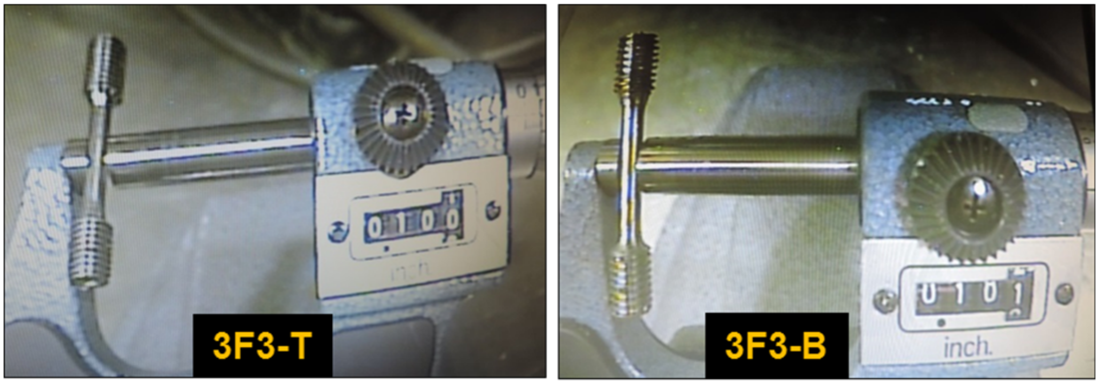


Figure 12. Gauge diameter measurement of completes tensile specimens.

Table 3. Dimensions and dose rate of the two tensile specimens.

	Actual Completed Machining Dimensions				**Dose Rate Information**
Specimen ID	Total Specimen Length, in.	Specimen Gage Length, in.	Specimen Gage Diameter, in.	Length as Defined in Scope of Work, in.	Measured [a] Dose Rate at 12 in. (mR/hr)
	(measured to 2 decimal places)		(measured to 3 decimal places)		(measured on 3/30/2015)
3F3-T	1.19	0.50	0.100	1.125	63
3F3-B	1.19	0.50	0.101		61

a. Measured at 12 in. using Thermo Scientific Teleprobe FH40.

The specimens, labeled 3F3-T and 3F3-B are shown next to their shipping vials in Figure 13.

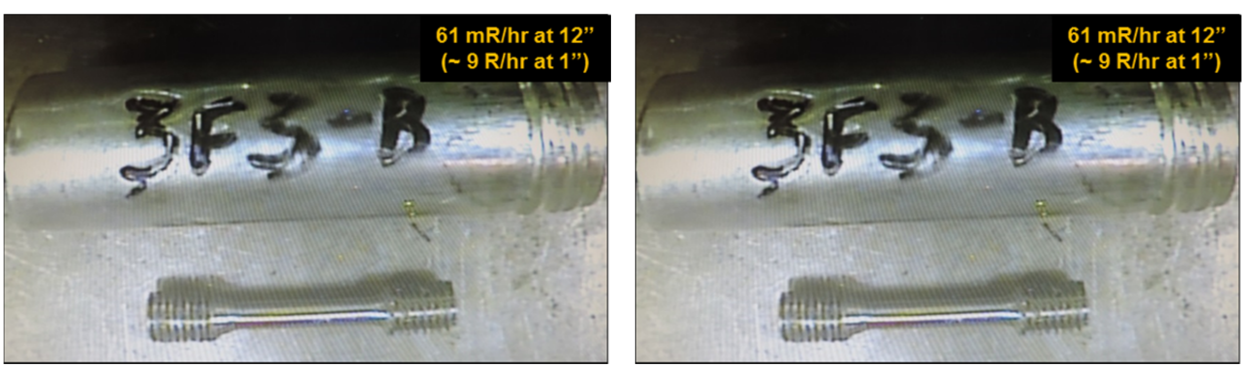


Figure 13. Completed specimens with their labeled screw-cap aluminum shipping vials.

3.7 Standard Cleaning and Contamination Measurements

The four CT specimens were ultrasonically cleaned in cell. The four specimens were first placed together into a clean glass beaker with the deionized water. Ultrasonic cleaning was then performed for 20 minutes. The deionized water was replaced with fresh deionized water and the specimens were ultrasonically cleaned for an additional 20 minutes. Following this 40 minute total ultrasonic cleaning, the specimens were then handled in such a way in-cell so that they did not directly touch any contaminated surfaces. For example, the specimens were placed into new radiologically clean beakers and/or placed onto new radiologically clean paper towels for all subsequent handling processes.

After ultrasonic cleaning, a cotton Q-tip was used to smear as much surface area as possible on each CT specimen. The Q-tip was then radiologically counted to document the amount of loose contamination on a per sample basis (not on a 100 cm^2 basis because the entire sample surface area is significantly less than 100 cm^2). Note that specimen CT3 was the first specimen to be smeared and this smear was performed after welding of the lead wires onto the specimen. Smears were taken on specimens CT1, CT2 and CT4 were taken before welding of the lead wires onto the specimens. Alpha and beta readings were obtained using a Tennelec Low Background Counting System and the gamma value was obtained using a high purity germanium detector

The results from these smears are provided in Table 4. In this table, the contamination measured on specimen CT3 is shown to be significantly less than the other three CT specimens – this is due to the smear taken from specimen CT3 not being as thorough compared to the smears obtained from the other specimens. There is still a significant amount of loose contamination that will need to be removed before shipping the specimens.

Table 4: Loose contamination levels from Q-Tip smears on specimens after initial standard ultrasonic cleanings

	Alpha dpm per 100 cm^2	**Beta** dpm per 100 cm^2	**Gamma** dpm per 100 cm^2
INL requested levels	7	70	70
CT Specimen ID	**Alpha** dpm per specimen	**Beta** dpm per specimen	**Gamma** dpm per specimen
Industry (and Westinghouse) accepted levels to be 'free releasable'	20	200	200
CT1	0	5,400	11,800
CT2	0	5,100	10,800
CT3 *	0	400	2,900
CT4	0	5,200	13,700

4. SHIPPING

To minimize the number of manipulation (cask loading, unloading, storage, etc.), it was decided to ship the specimens as a demand basis. This means that the completed ten specimens (four CT specimens, four microscopy plates, and two tensile specimens) will not be shipped to INL at once. It was decided that two CT specimens will be shipped to the building where the testing will be performed, the remaining specimens being stored in Westinghouse facility.

The tensile specimens will be shipped using screw cap aluminum vials with a label corresponding to the specimens ID. The only challenge is the shipment of the CT specimens with leads attached. Each specimen will be shipped in an individual aluminum container, maintaining the specimen in place between foam inserts, as shown in Figure 14.

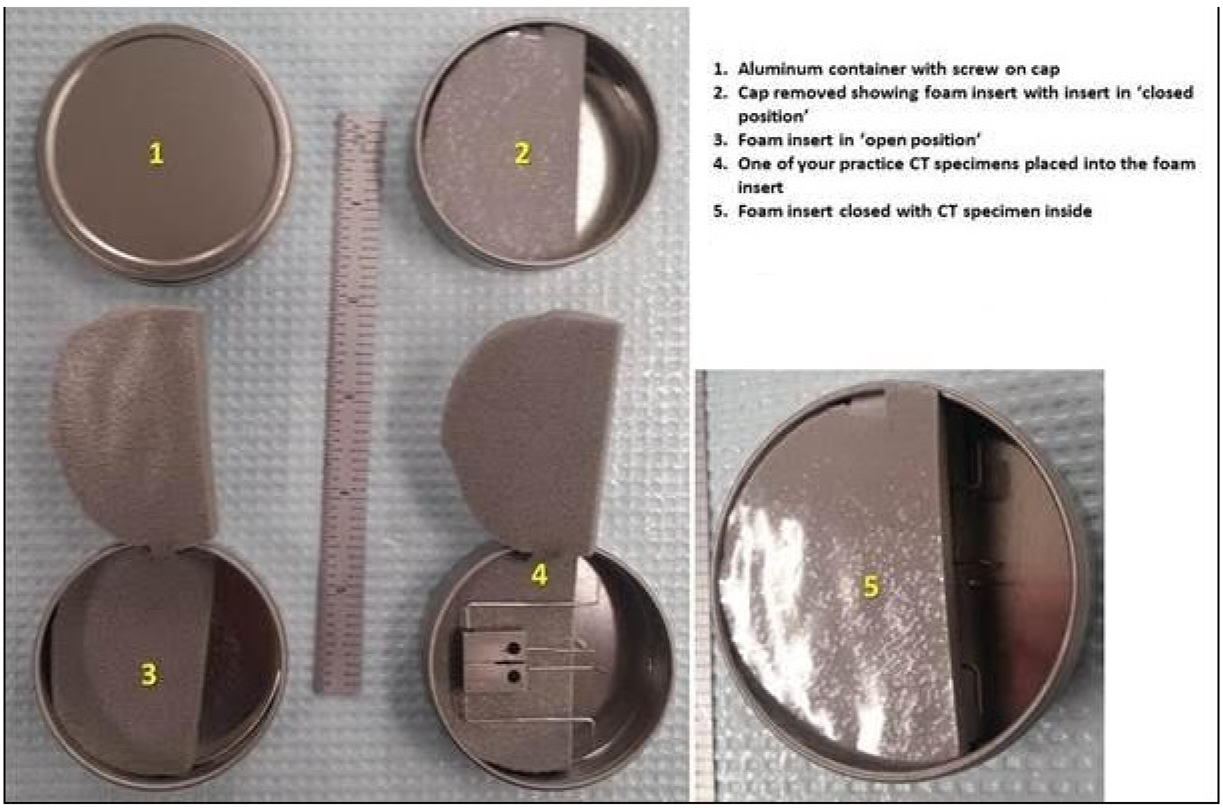


Figure 14. Packaging of CT specimens with attached leads.

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