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Technical Review: R. E. Fuentes, Applied Materials Research Date

FY20 Status of the Characterization of DEs from the Full Circumference Examination of the Inner Container Closure Weld Region

Summary

One of the main focus areas of the 3013 Surveillance Program is a thorough evaluation of the inner container closure weld region (ICCWR) opened for destructive examination (DE). As part of the protocol to investigate the corrosion in the ICCWR a laser confocal microscope (LCM) is used to perform close visual examination of the surface and measurements of corrosion features on the surface. DE containers from FY13 through FY16 were screened with three candidates selected for full circumference evaluation (FCE) according to the ICCWR examination protocol [1].

During the FCE, FY16 DE05 and FY15 DE07 showed suspect major corrosion events [2, 3]. FY16 DE05 Section C2 shows two crack-like features, identified as Denebola and Draco. Both features are located at the boundary of Zone 2 and Zone 3. Consequently, Section C2 of FY16 DE05 was selected for examination by serial metallography at Savannah River National Laboratory (SRNL). For FY15 DE07 the suspect corrosion events were observed on Sections C1 and C2. Section C1 shows one crack-like feature, identified as Acrux. Section C2 shows three crack-like features, identified as Bellatrix, Cursa Minor and Cursa Major. Unlike Acrux and Bellatrix, which are located at the boundary of Zone 2 and Zone 3, Cursa Minor and Cursa Major are located in Zone 2. Sections C1 and C2 of FY15 DE07 were sent to Los Alamos National Laboratory (LANL) for characterization by X-Ray Tomography (XRT).

Before preparing the sample for the metallographic analysis, the LCM images of FY16 DE05 Section C2 were analyzed using the Image Analysis Software (IAS) developed by LANL [4]. Several suspect corrosion features were identified, labeled as D1 through D6, where D1 corresponds to Denebola and D2 corresponds to Draco. Corrosion features D1 through D6 were characterized by SEM. The SEM image of Denebola (D1) show it is an apparent chipped section on the surface, but more information will be obtained in the future from the cross-section examination. Draco (D2) seems to be a crack with pits along the crack. Corrosion features D3 and D6 are large pits within the grain growth zone close to the weld. For D3, part

of the grain boundary can be observed inside the pit, but no apparent crack was identified. For D6, a large separation of the grains can be observed inside the pit which may indicate intergranular corrosion or a crack formation. Corrosion feature D4 does not show visible signs of cracks. Corrosion feature D5 is a large pit with an apparent crack inside. Intergranular and pitting corrosion was observed in most of the images. FY16 DE05 Section C2 was cut into a smaller sample, labeled as Subsection C2a, and mounted for cross-section examination. The specimen was successively ground with finer and finer abrasive media to the first position (885 μm from weld edge). SEM images from the 1st round analysis show pits of different sizes (up to 16 μm) and depths (up to 10 μm).

Initial imaging of FY15 DE07 Section C2 was obtained using XRT at LANL. However, sample geometry affected the image by showing noise and ghosting. Similar issues would be expected for Section C1. Consequently, in order to obtain a better image, LANL sent the samples back to SRNL for sectioning around the areas where the corrosion features are located. The samples were received at SRNL and cut to the specified dimensions. Both samples, subsections C1b.2 and C2b.2, show that the original corrosion features are still present and unaffected by the cutting. The cut samples were returned to LANL for further examination with the XRT.

Introduction

As part of the ICCWR examination protocol, a Keyence 3D laser confocal microscope (LCM) model VK-X110 is used to perform close visual examination of the surface at the ICCWR and surface profile measurements for pit depths or other corrosion features on the surface. Figure 1 shows the general topography of the ICCWR. In the weld fusion zone, the weld beads on the surface resemble valleys and mountains. In the heat affected zone (HAZ) the surface is mostly flat with a beveled region where the machining marks are located. The fusion zone is considered Zone 1, the region with machining marks is considered Zone 2 and the flat portion of the HAZ is considered Zone 3.

DE containers from FY13 through FY16 were evaluated to select candidates for a full circumference analysis of the ICCWR [1]. In FY17, the following DE containers were selected for full circumference analysis of the ICCWR with the following prioritization order: FY15 DE07, FY16 DE05, and FY15 DE08 [1]. In FY18 the selected DE containers for full circumference analysis were processed according to the ICCWR protocol described in reference [2] and LCM data collection was completed for the full circumference only for FY15 DE07. In FY19 the remaining DE containers were completed [3].

The three ICCWRs show general and localized corrosion on the surface. However, FY15 DE08 show more areas with agglomerated pits in Zones 2 and 3 than the other two DEs. The major suspect corrosion events were observed for FY15 DE07 on Sections C1 and C2 and for FY16 DE05 on Section C2 as shown in Figure 2. These events correspond to suspect cracks or crack-like features identified with a unique name to easily refer to each feature [2, 3]. FY15 DE07 shows a crack-like feature, identified as Acrux, in Section C1 and three features in Section C2 identified as Bellatrix, Cursa Minor and Cursa Major. Acrux and Bellatrix are located at the boundary of Zone 2 and Zone 3. Cursa Minor and Cursa Major are in Zone 2. FY15 DE08 shows two crack-like features, identified as Denebola and Draco, in Section C2. Additional characterization on the locations where the crack-like features were found started in FY20. Sections C1 and C2 of FY15 DE07 were sent to LANL for characterization by XRT. Section C2 of FY16 DE05 was kept at SRNL to perform serial metallography for characterization of the cross-sections by SEM.

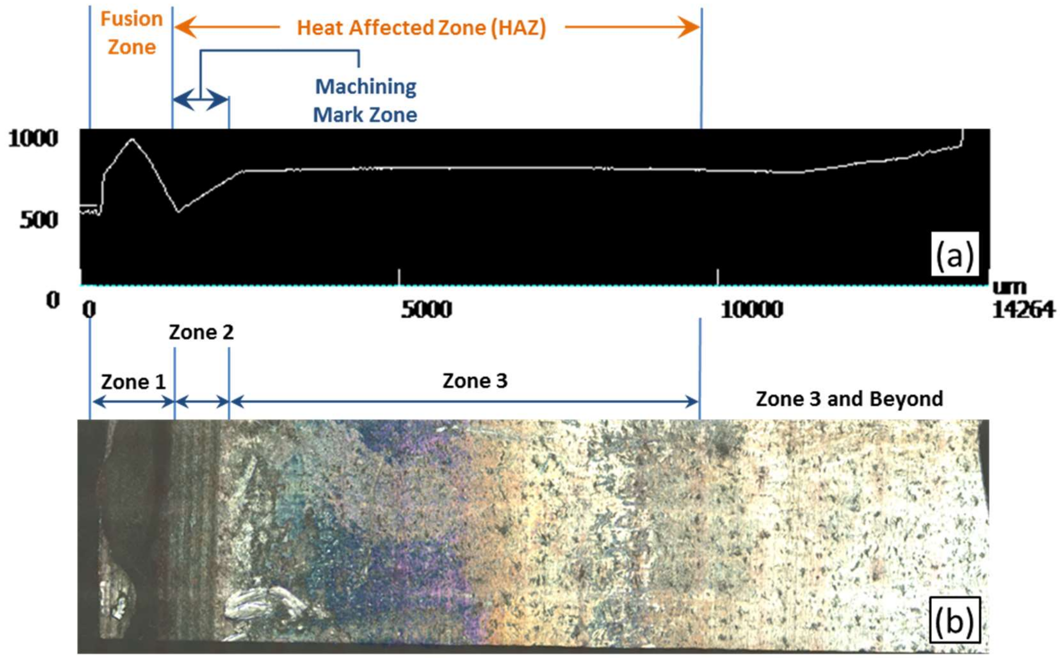

Figure 1. General topography of an ICCWR sidewall sample. Image of FY11 HHMC-90° sidewall showing (a) side view of surface profile and (b) top view optical image of surface [1, 5].

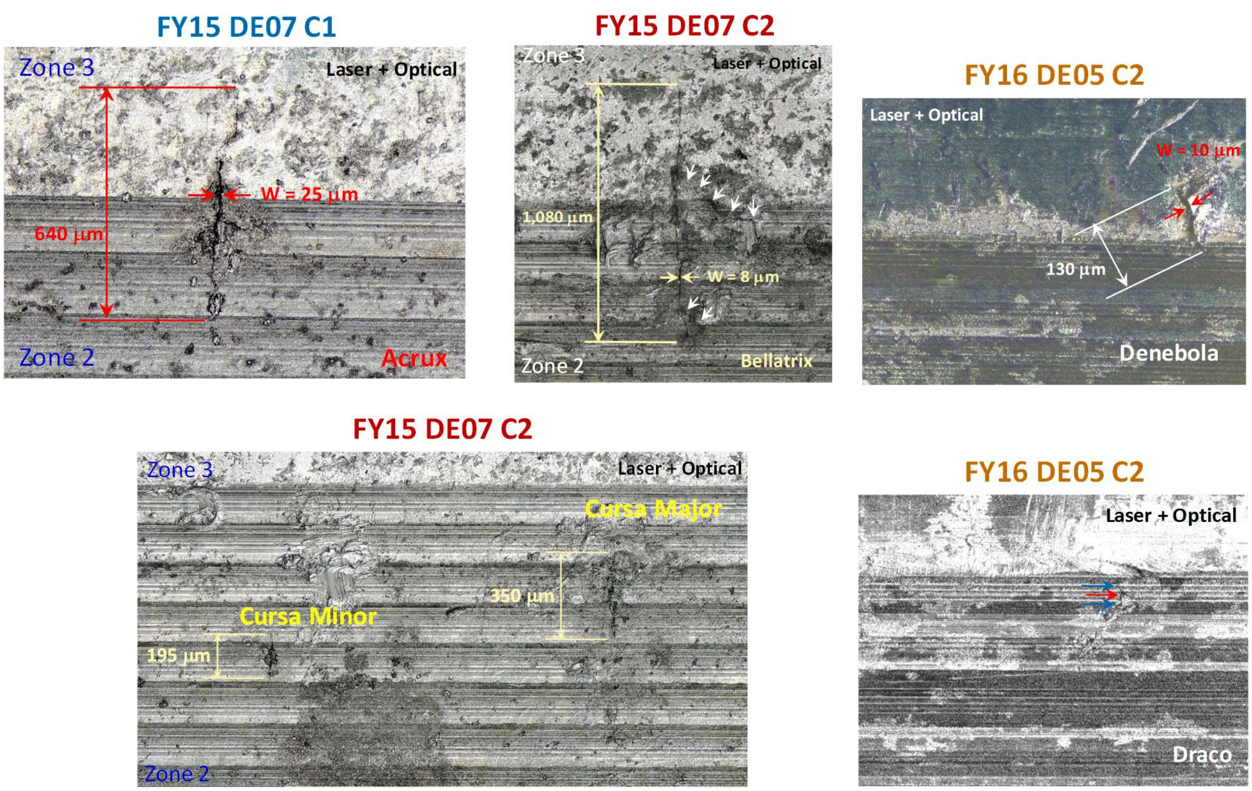

Figure 2. Major suspect corrosion events observed for FY15 DE07 on Sections C1 and C2 and for FY16 DE05 on Section C2.

Status of FY16 DE05 ICCWR Characterization

During the full circumference examination for FY16 DE05 two major suspect corrosion events were observed on Section C2 [3]. This section shows two crack-like features, identified as Denebola and Draco. Both features are located at the boundary of Zone 2 and Zone 3. Consequently, Section C2 of FY16 DE05 was selected for examination by serial metallography at SRNL. The process consists of a systematic examination of the cross section of the sidewall by SEM after the specimen is prepared by various methods of grinding and polishing. This stepwise process provides information about the corrosion features, such as pit and cracks geometry and depth.

Before preparing the sample for the metallographic analysis, the LCM images of FY16 DE05 Section C2 were analyzed using the IAS developed by LANL [4]. Figure 3 shows a grid over the LCM image of a portion of FY16 DE05 Section C2 with the suspect corrosion features locations labeled as D1 through D6, where D1 corresponds to Denebola and D2 corresponds to Draco. These locations correspond to the cells ranked using the crack strength determined by the IAS. In the IAS, a template matching method is used to match corrosion features to several crack templates. The crack templates consist of predefined crack shapes, sizes and patterns. The crack strength represents the value, determined for each cell, based on the number of matches of the corrosion features to crack templates. The higher the crack strength number indicates that more crack templates match the corrosion features for that cell. Consequently, this will indicate a higher probability of finding a corrosion event in that cell. The cells were ranked based on the crack strength. For example, the cell with the most cracks matching (highest crack strength) correspond to a rank of 1 and so on.

Corrosion features D1 through D6 were characterized by SEM as shown in Figure 4 through Figure 12. A secondary electron detector (SED) was used to produce a topographic SEM image while, in some cases, a back-scatter detector (BSD) was used to obtain an image with contrasts due to composition. The SEM image of Denebola (D1) is shown in Figure 4. Denebola is an apparent chipped section on the surface, but more information will be obtained in the future from the cross-section examination. However, pitting corrosion is observed around Denebola. Draco (D2) is shown in Figure 5, which show a crack with pits along the crack. Pitting corrosion is also observed around Draco. Corrosion feature D3, shown in Figure 6, is a large pit within the grain growth zone close to the weld. A higher magnification SEM image of the area of interest is shown in Figure 7. The large pit has a size of 27 μm. Pitting corrosion is observed around the large pit, with many found between the grains (intergranular corrosion) or within a pit cluster. Figure 8 shows a higher magnification of the large pit. Part of the grain boundary can be observed inside the pit, but no apparent crack was identified. Corrosion feature D4, shown in Figure 9, does not present visible or signs of cracks. However pitting corrosion can be observed on the image. Corrosion feature D5, shown in Figure 10, is a large pit of 38 μm with an apparent crack inside. Intergranular corrosion and coalesced pits were also observed in the image. Corrosion feature D6, shown in Figure 11, is a large pit within the grain growth zone close to the weld. A higher magnification SEM image of the area of interest is shown in Figure 12. The large pit has a size of 52 μm. A large separation of the grains can be observed inside the pit which may indicate intergranular corrosion or a crack formation. Pitting corrosion is observed around the large pit as well.

FY16 DE05 Section C2 was cut into a smaller sample, labeled as Subsection C2a, using a Buehler IsoMet Low Speed precision cutting saw in preparation for the cross-section examination. Figure 13 shows the section of the sample cut, which includes the suspect cracks or corrosion features D1 through D6. Subsection C2a was mounted using Technovit® 5000, an electroconductive polymer based on modified methyl methacrylate, which contain dendritic copper particles. After mounting and curing, the specimen was successively ground with finer and finer abrasive media to the first position (885 μm from weld edge), as shown in Figure 14. SEM images were obtained from this 1st round along the cross-section at several locations, as shown in Figure 15 through Figure 17. Although the best effort was made to stop at corrosion features D3 and D6, the corrosion feature D6 was missed due to the difficulty to stop at a very precise location. However, the main purpose of the cross-section examination is to be able to verify the larger corrosion features D1 and D2 (Denebola and Draco, respectively). Corrosion features D3 through D6 are being analyzed as best as possible while the sample is ground to the target position (D1 and D2). SEM images from the 1st round analysis show pits of different sizes (up to 16 μm) and depths (up to 10 μm). The sample has been ground to the next position and the 2nd round analysis is in progress.

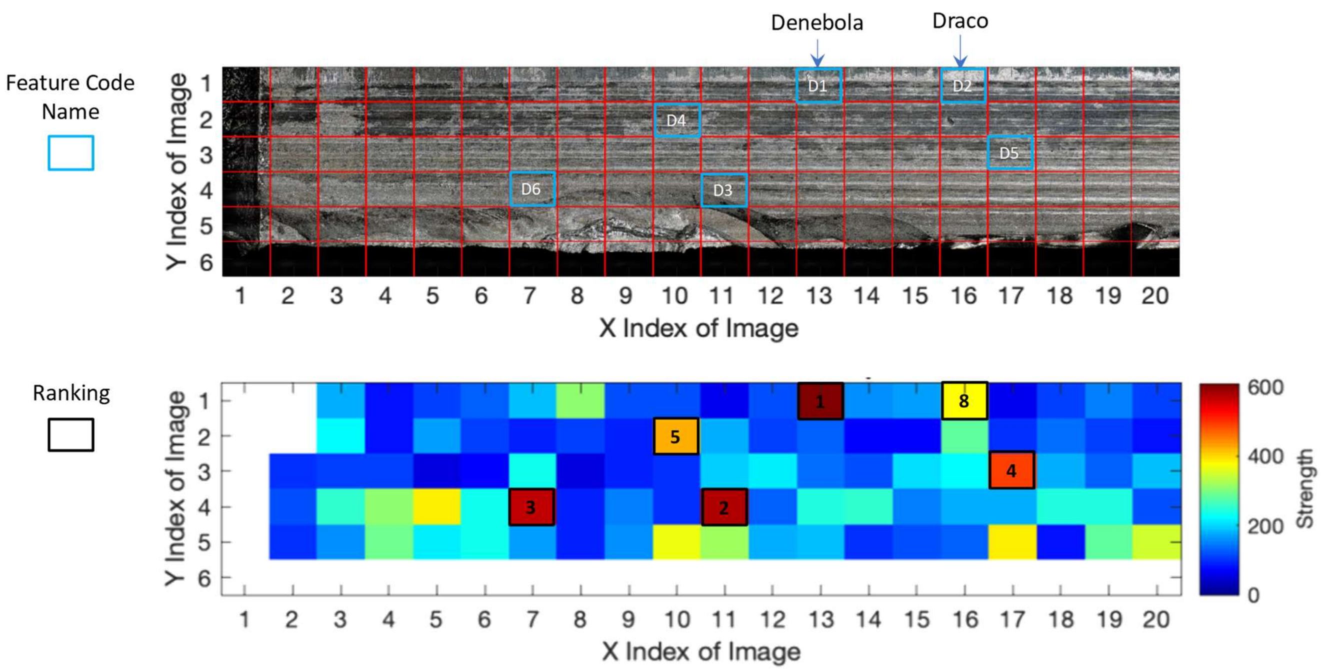

Figure 3. LCM optical image of Zones 1 and 2 for a portion of the ICCWR of FY16 DE05 Section C2 (top). The grid over the image show the suspect corrosion feature locations labeled as D1 through D6, where D1 corresponds to Denebola and D2 corresponds to Draco. The crack strength ranking for each cell was determined by the LANL Image Analysis Software (bottom).

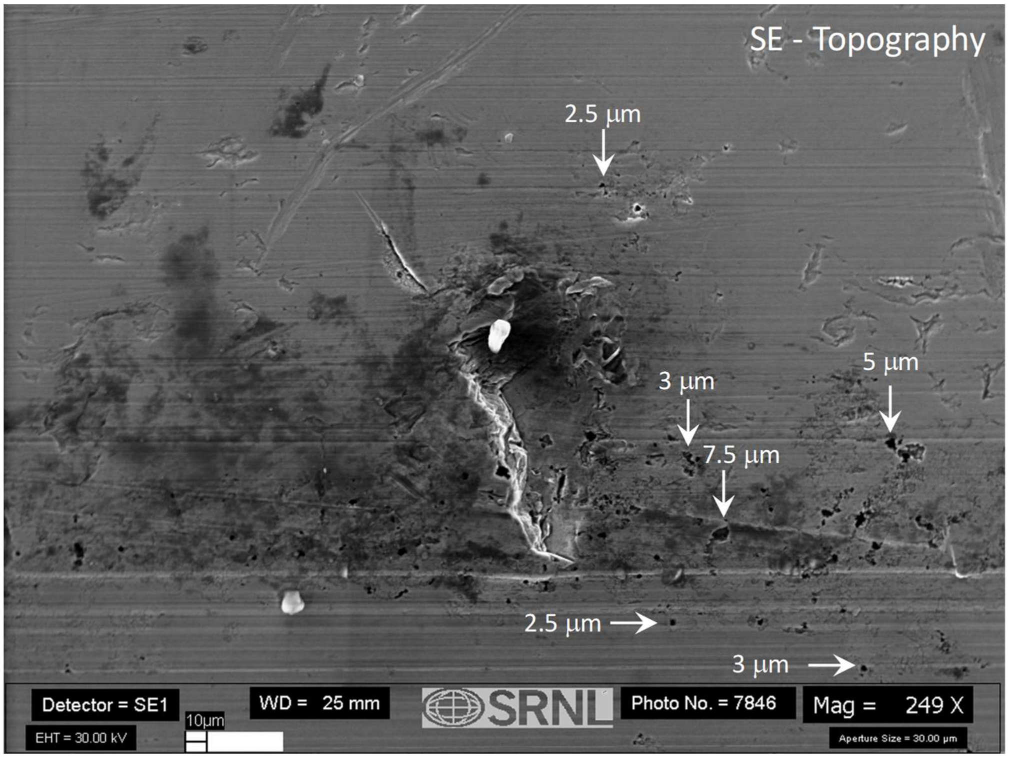

Figure 4. SEM image of Denebola (corrosion feature D1).

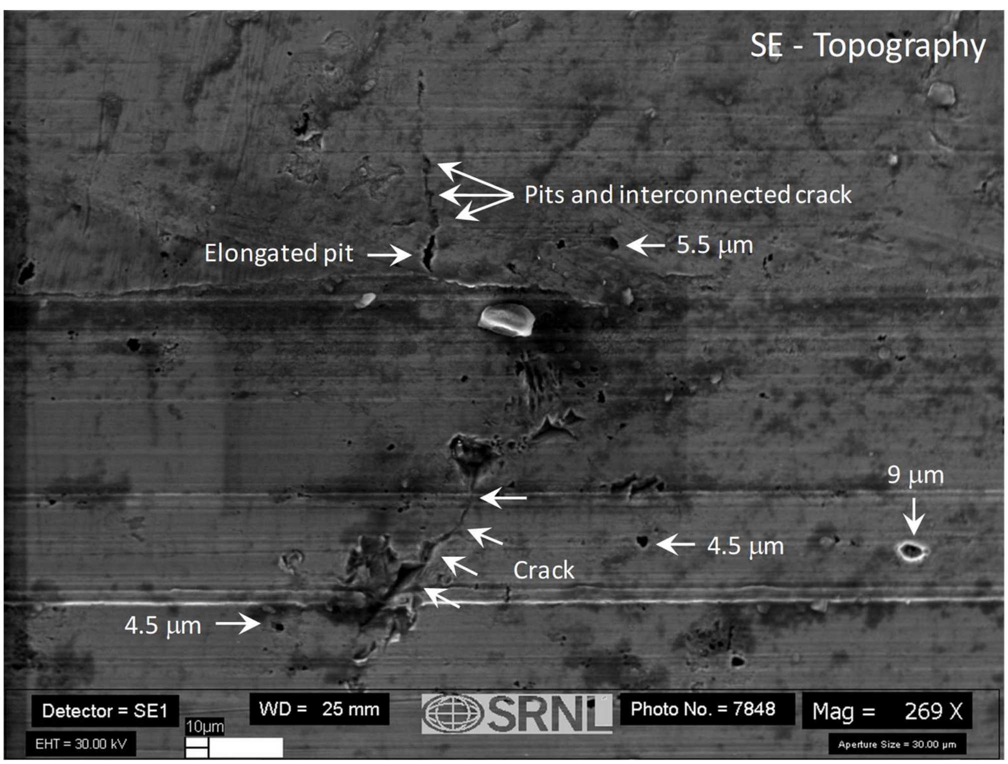

Figure 5. SEM image of Draco (corrosion feature D2).

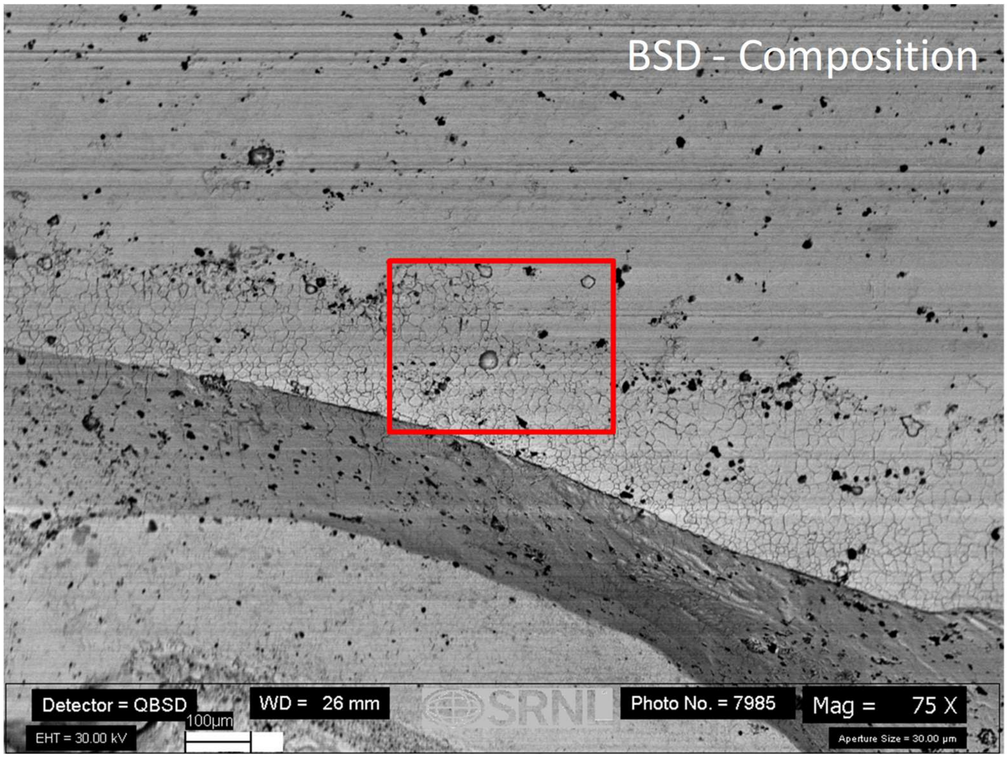

Figure 6. SEM image of corrosion feature D3. Red box corresponds to location of SEM image shown in Figure 7.

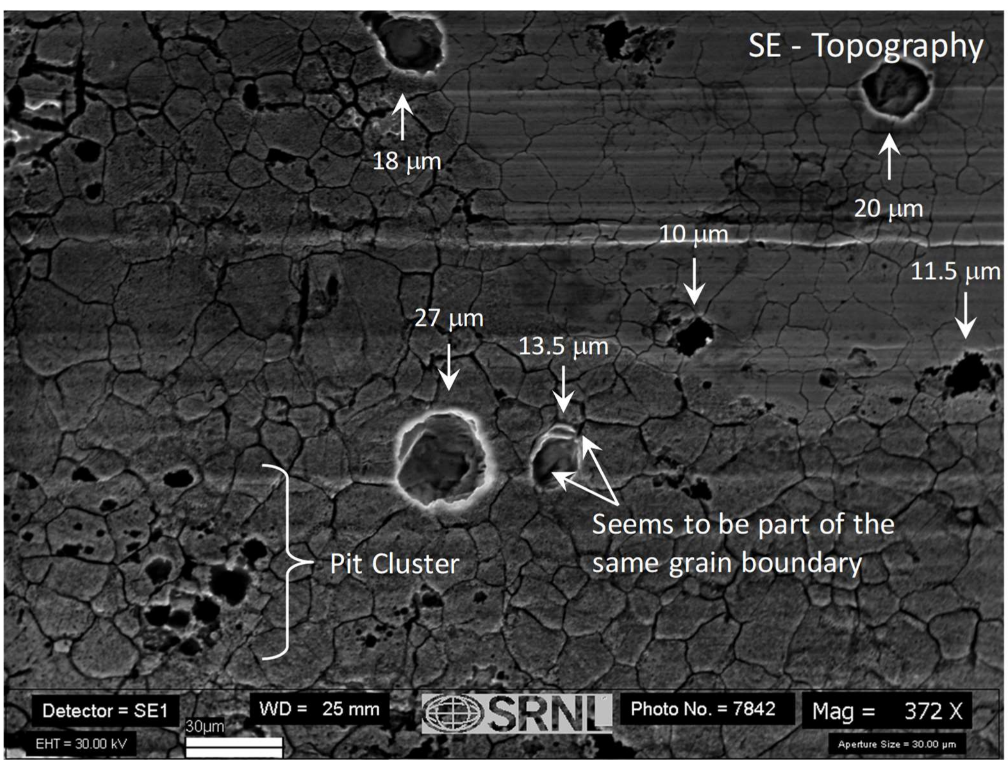

Figure 7. SEM image of corrosion feature D3 at a magnification of 372X (center pit of 27 μm).

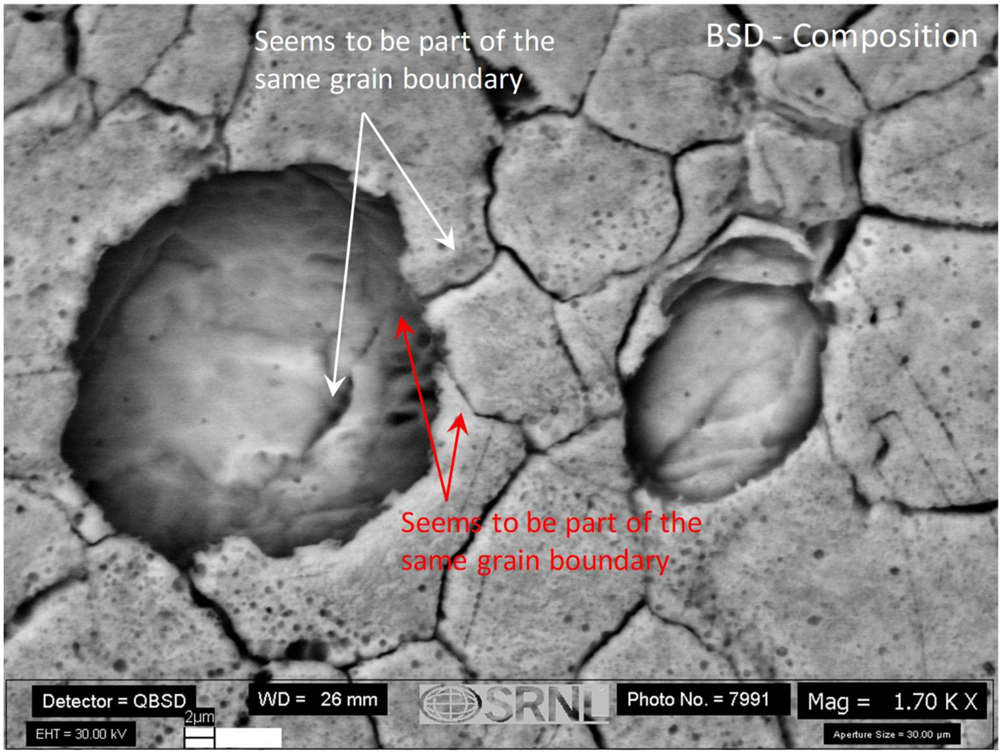

Figure 8. SEM image of corrosion feature D3 at a magnification of 1,700 X.

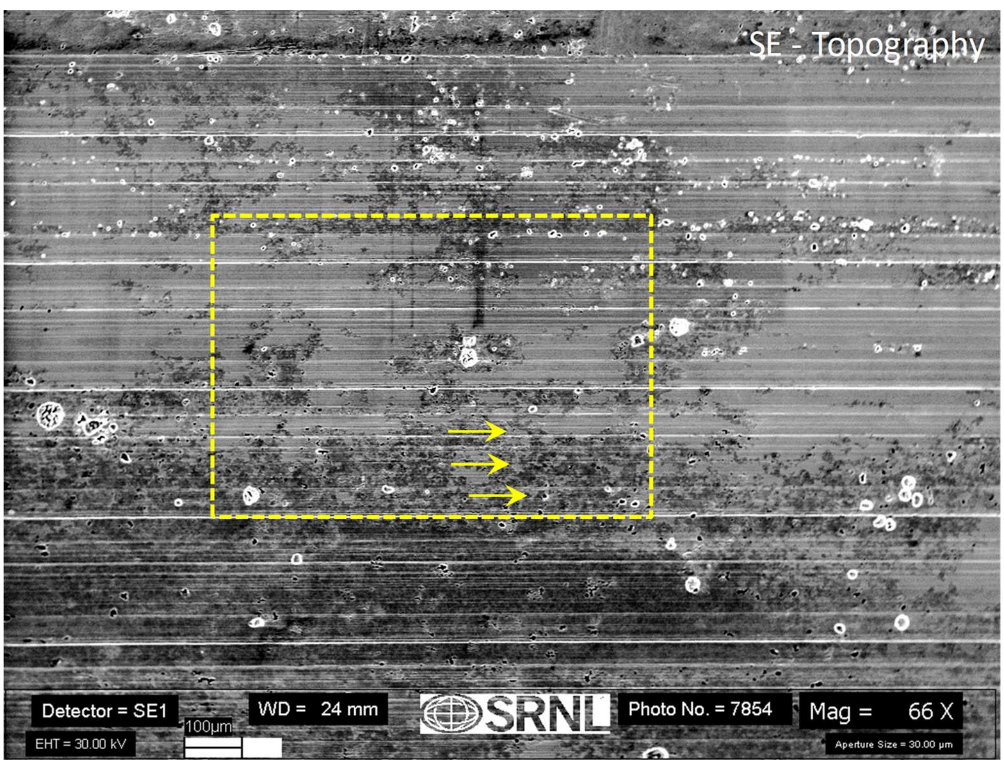

Figure 9. SEM image of corrosion feature D4 (yellow arrows).

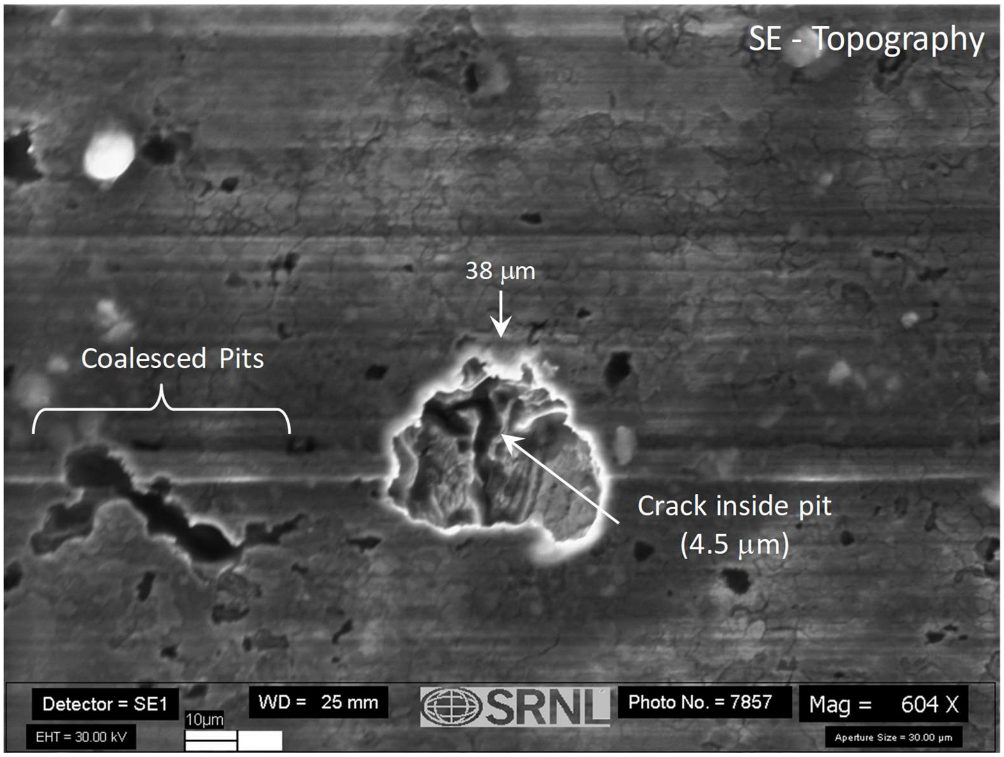

Figure 10. SEM image of corrosion feature D5 (center pit of 38 μm).

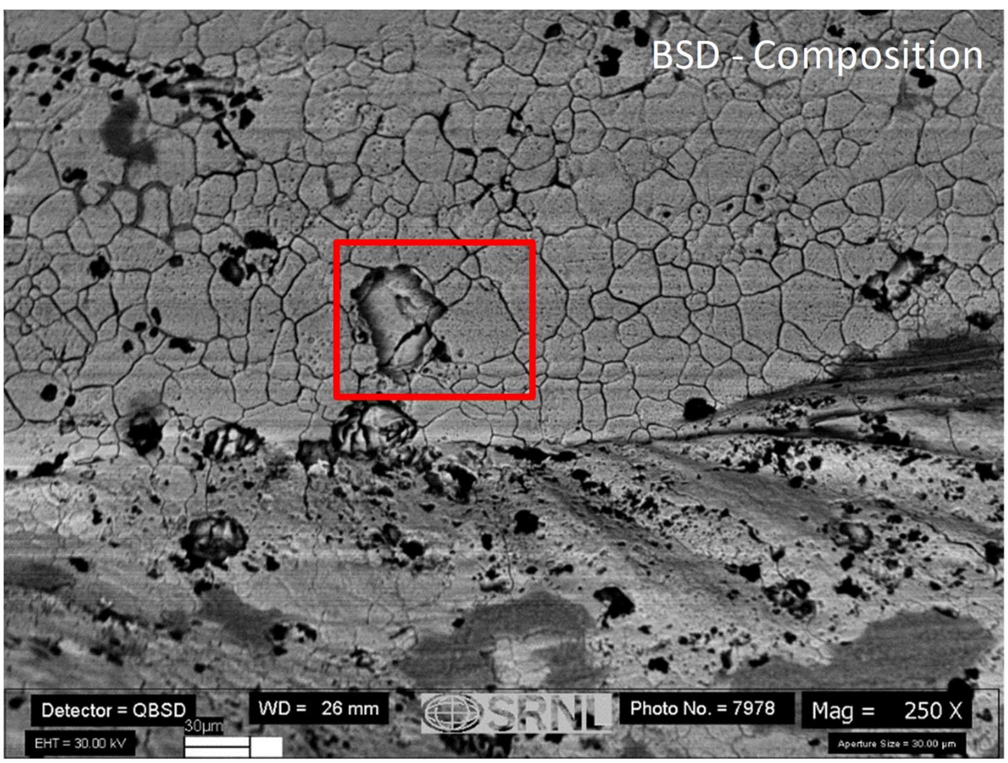

Figure 11. SEM image of corrosion feature D6. Red box corresponds to location of SEM image shown in Figure 12.

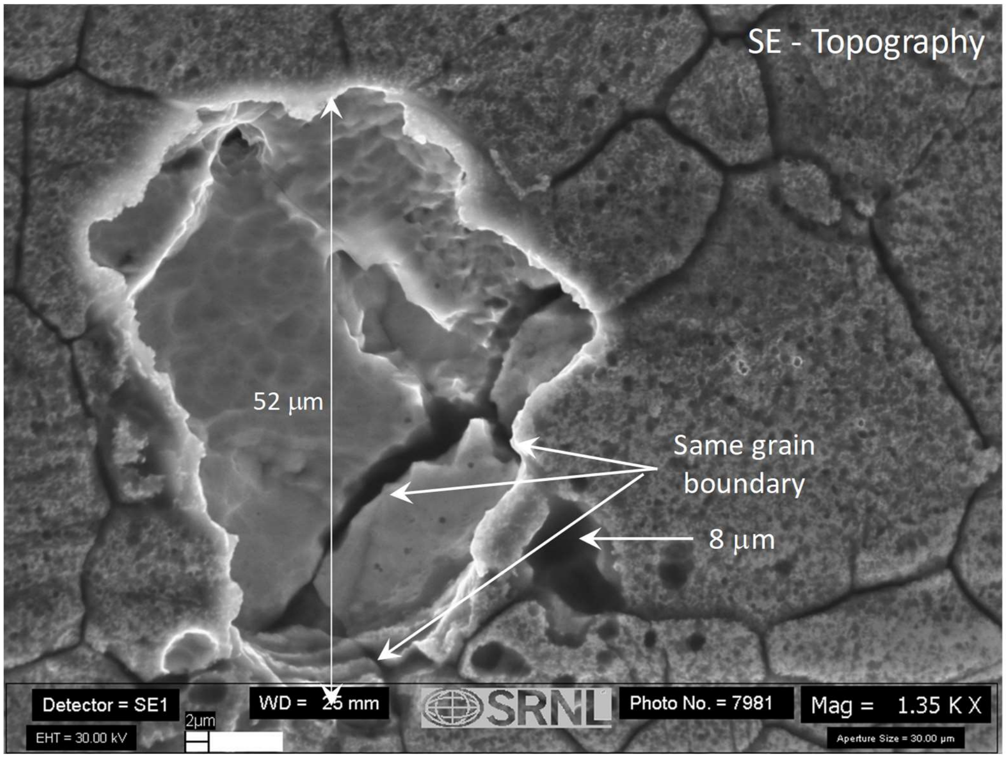

Figure 12. SEM image of corrosion feature D6 at a magnification of 1,350X.

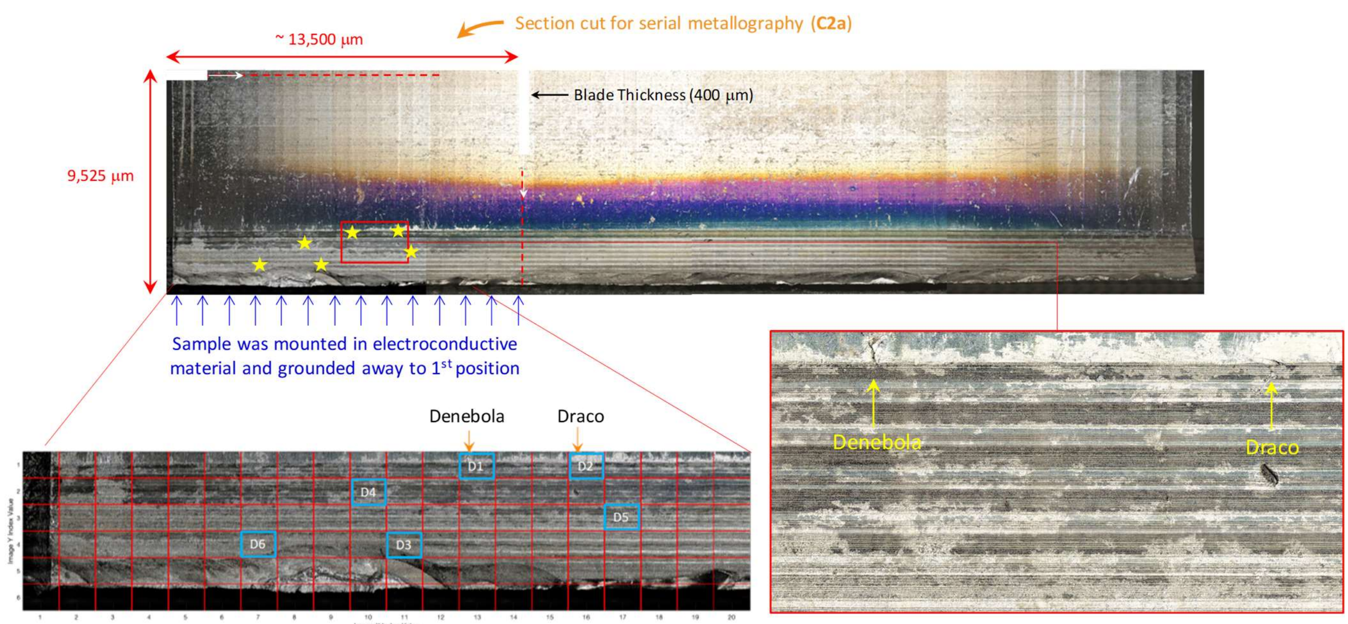

Figure 13. LCM optical image of the ICCWR of FY16 DE05 Section C2 showing subsection C2a with locations, marked with yellow starts, of suspect cracks or corrosion features. Grid over a portion of Zone 2 show the locations labeled as D1 through D6, where D1 corresponds to Denebola and D2 corresponds to Draco.

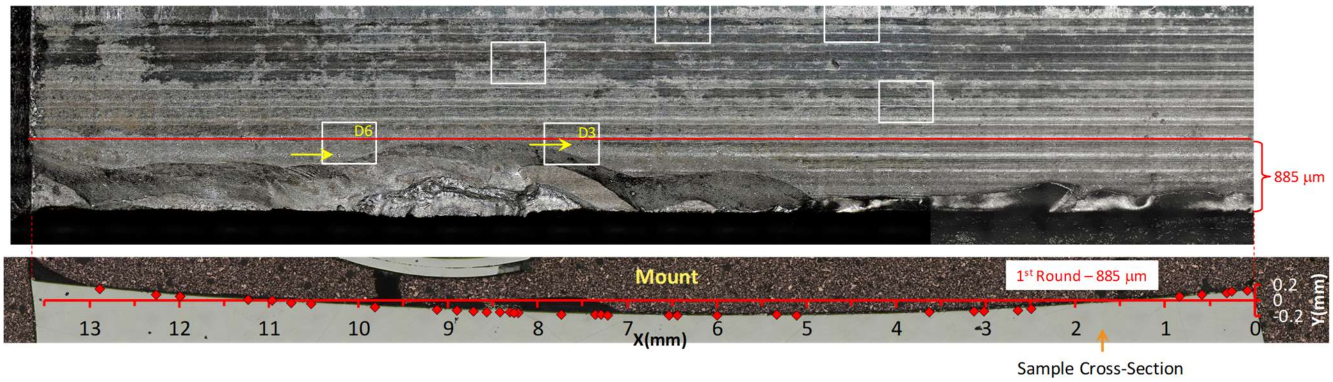

Figure 14. The 1st round on the serial metallography process for the ICCWR of FY16 DE05 Section C2a. LCM optical image of the surface (top) and LCM optical image of the mounted cross-section (bottom) ground and polished 885 µm, from the bottom edge, corresponding to the red line located on top LCM image. X-axis on cross-section image show positions, marked with red diamonds, where SEM images were obtained.

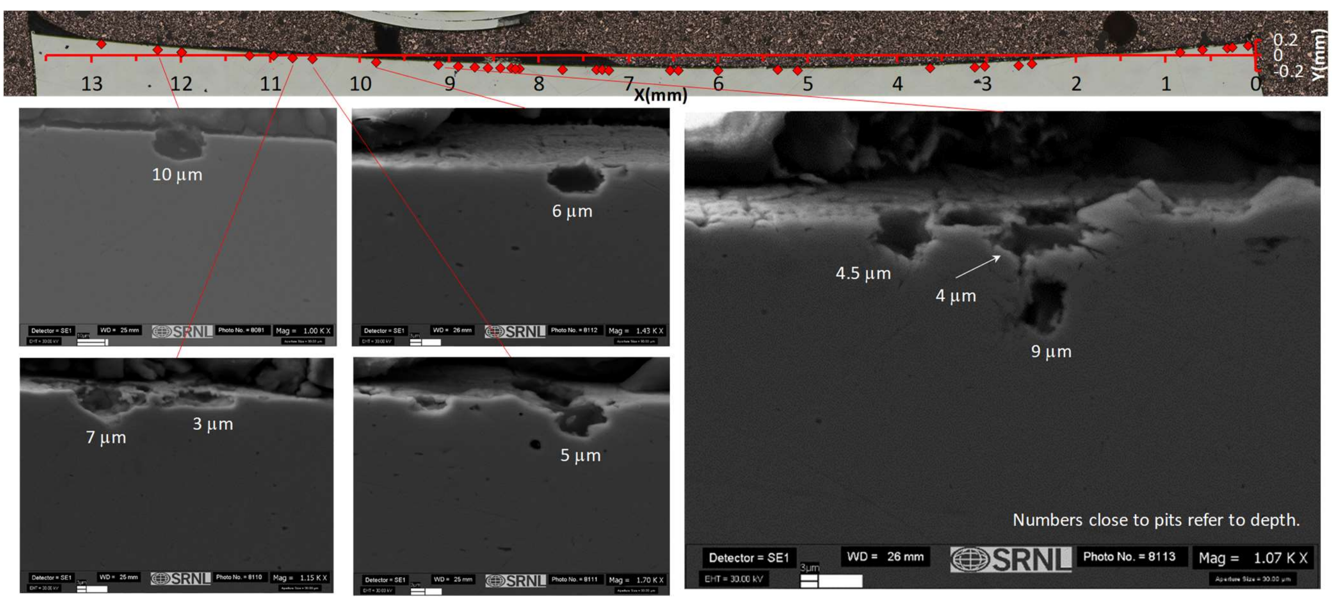

Figure 15. SEM images from the 1st round of the cross-section of FY16 DE05 Section C2a for some positions between 9 – 11 mm from the right edge of the sample.

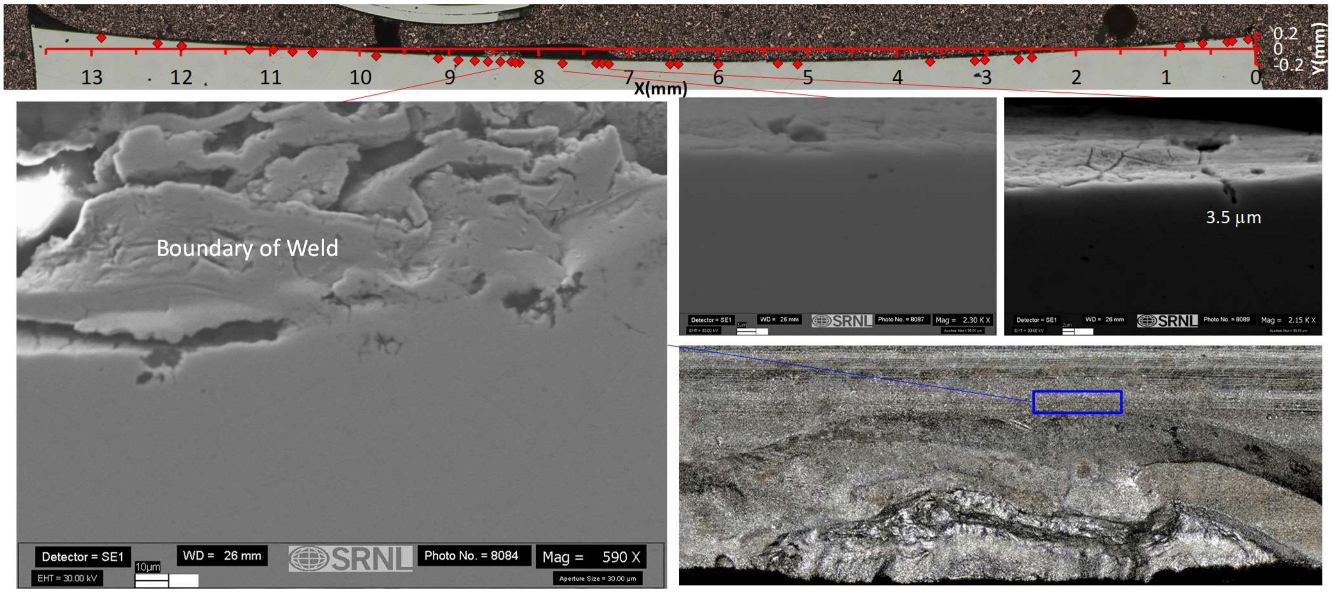

Figure 16. SEM images from the 1st round of the cross-section of FY16 DE05 Section C2a for some positions between 7 – 9 mm from the right edge of the sample.

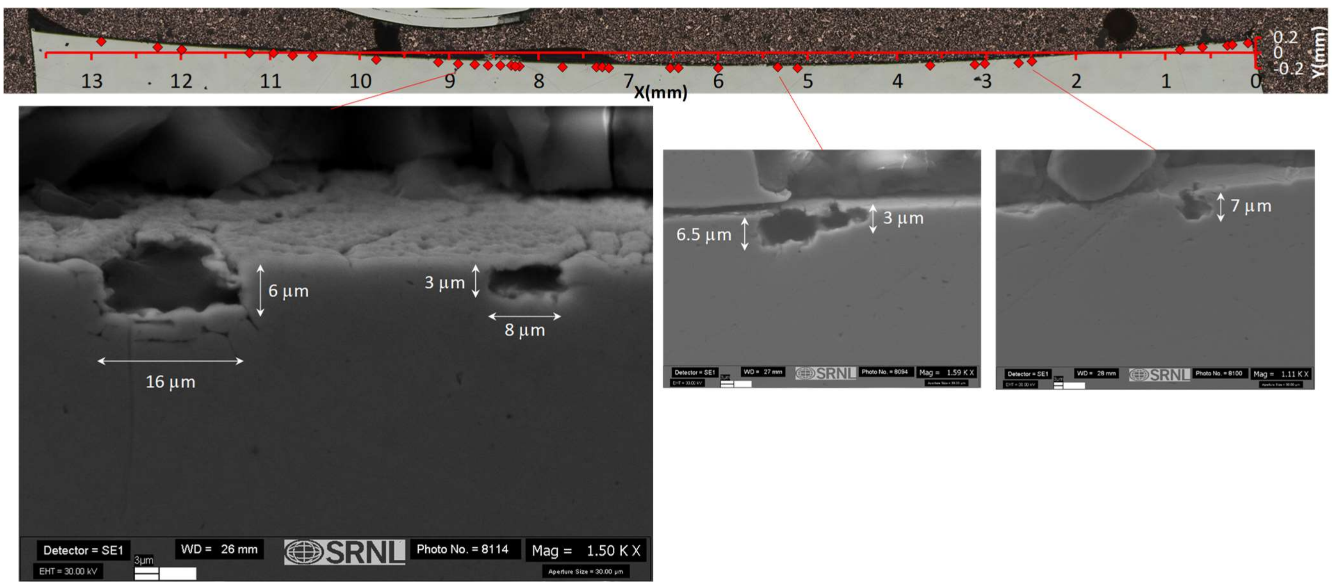

Figure 17. SEM images from the 1st round of the cross-section of FY16 DE05 Section C2a for some positions between 2 – 9 mm from the right edge of the sample.

Status of FY15 DE07 ICCWR Characterization

During the full circumference examination for FY15 DE07 major suspect corrosion events were observed on Sections C1 and C2. [2, 3]. Section C1 shows one crack-like feature, identified as Acrux. Section C2 shows three crack-like features, identified as Bellatrix, Cursa Minor and Cursa Major. Unlike Acrux and Bellatrix, which are located at the boundary of Zone 2 and Zone 3, Cursa Minor and Cursa Major are located in Zone 2. Sections C1 and C2 of FY15 DE07 were sent to LANL for characterization by XRT.

Initial imaging of FY15 DE07 Section C2 was obtained using X-ray Computed Tomography, a nondestructive technique for visualizing the interior features of the sample and obtaining digital information on their 3-D geometries. A cross-section view image of Section C2 is shown in Figure 18 [6]. The image at maximum resolution took 2 days due to sample geometry. Sample geometry also affected the image by showing noise and ghosting. Similar issues would be expected for Section C1. consequently, in order to obtain a better image, LANL sent the samples back to SRNL for sectioning around the areas where the corrosion features are located. The samples were sent back because SRNL has the capabilities for performing cuts on contaminated samples and the SRNL personnel working on these samples is familiarized with the samples and corrosion features location.

FY15 DE07 Sections C1 and C2 were received at SRNL. Planned cut locations for each sample are shown in Figure 19 and Figure 20, respectively. The samples were cut using a Buehler IsoMet Low Speed precision cutting saw. To make sure that corrosion features in the samples are not affected by the cut, extra distance was used to position the cutting blade. The final dimensions of the cut samples are shown in Figure 21 and Figure 22 for subsections C1b.2 and C2b.2, respectively. Both samples show that the original corrosion features are still present and unaffected by the cutting. The cut samples were returned to LANL for further examination with the XRT.

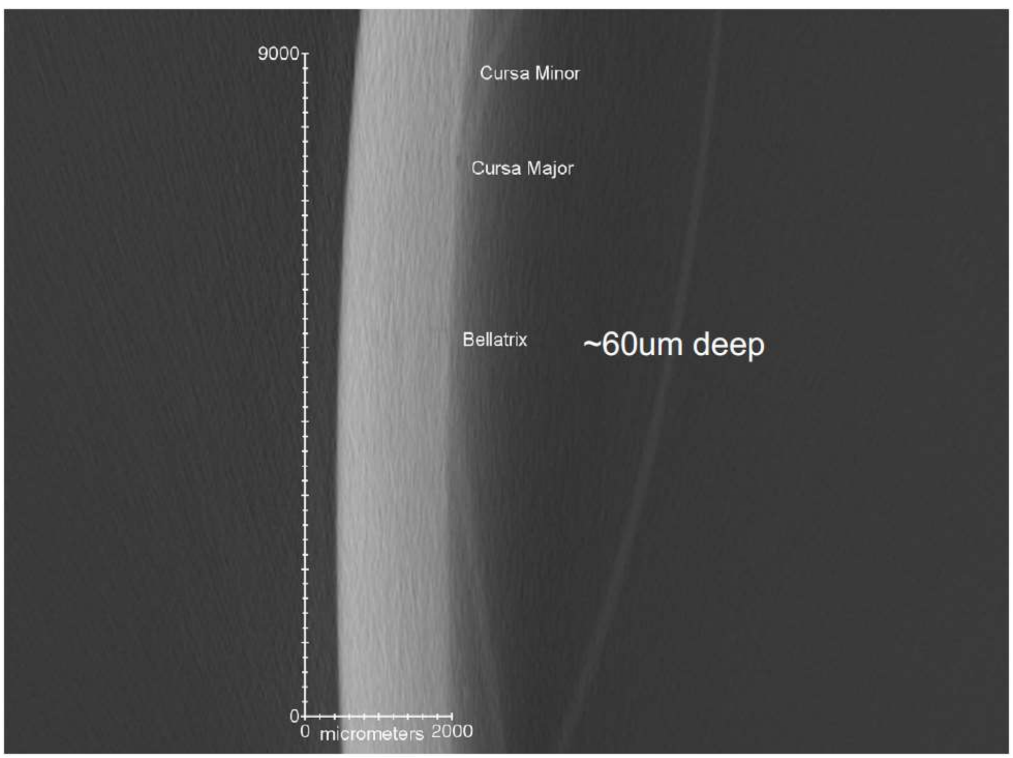

Figure 18. XRT image (cross-section view) of FY15 DE07 Section C2 obtained at LANL [6].

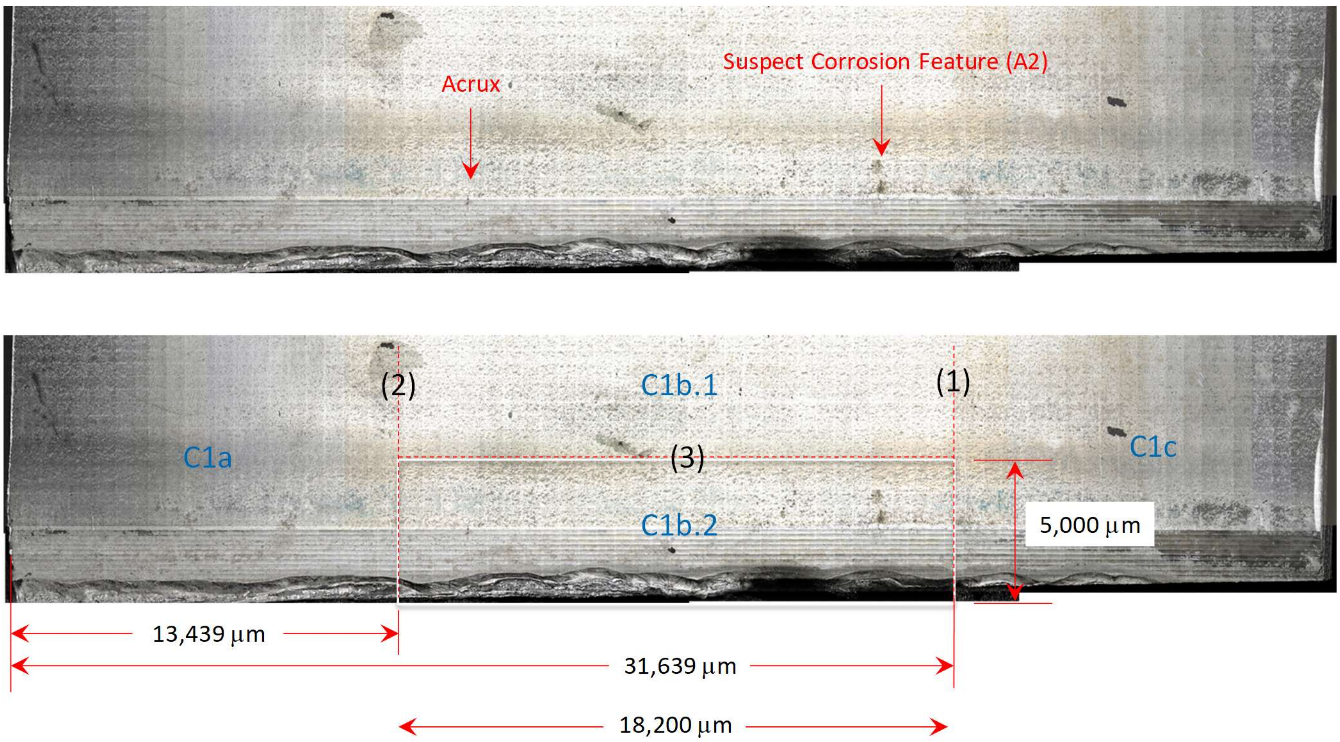

Figure 19. Planned cut locations for FY15 DE07 Section C1.

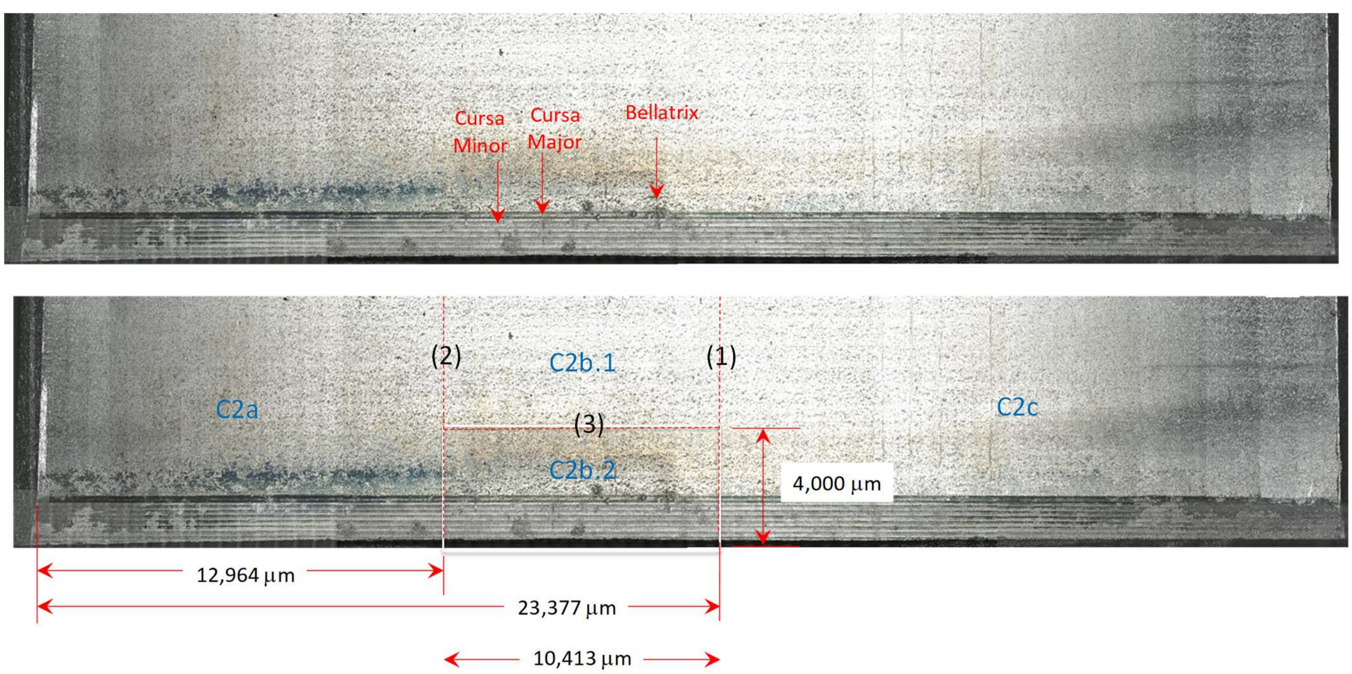

Figure 20. Planned cut locations for FY15 DE07 Section C2.

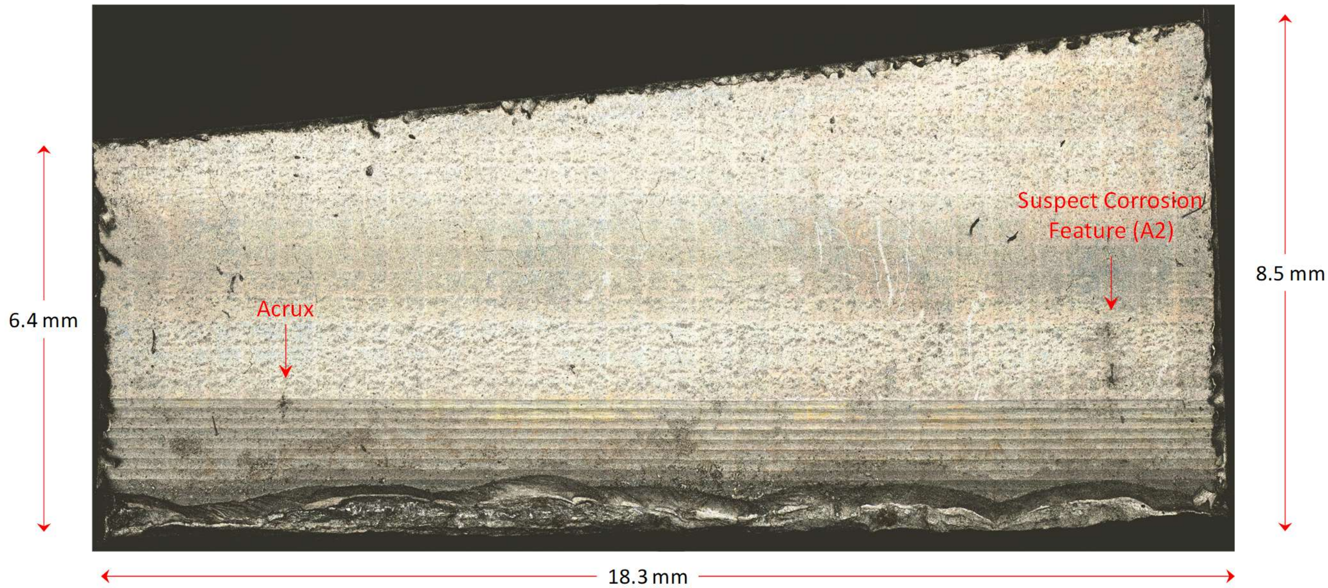

Figure 21. FY15 DE07 Section C1b.2 obtained from cutting main Section C1.

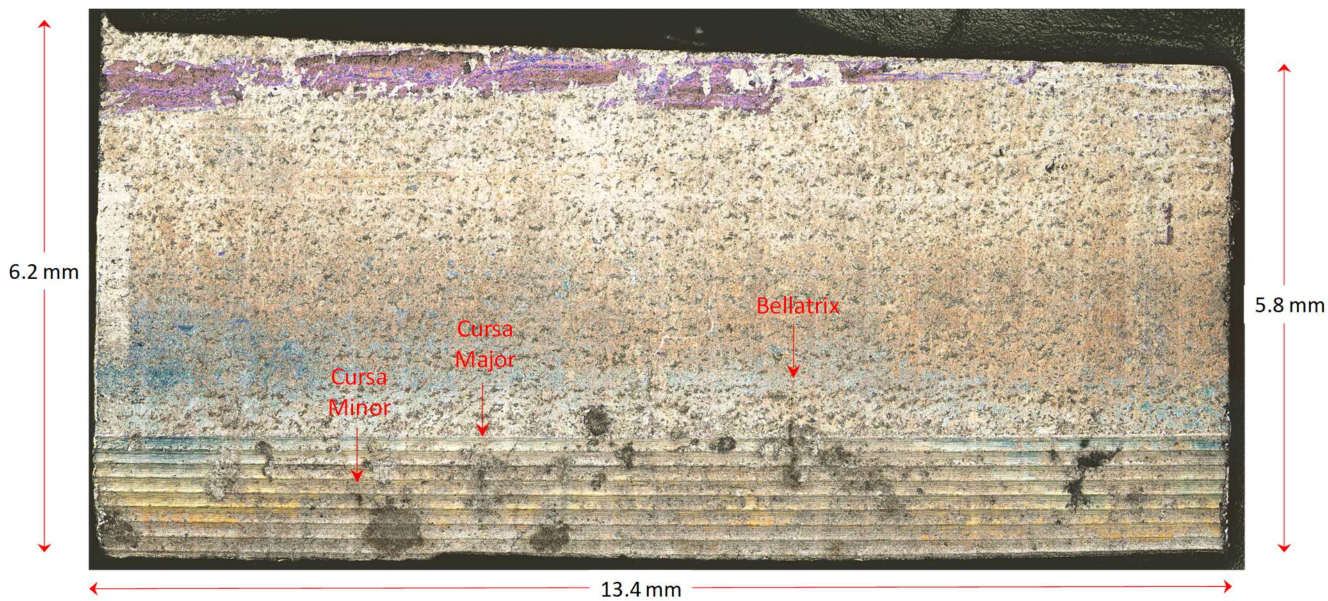

Figure 22. FY15 DE07 Section C2b.2 obtained from cutting main Section C2.

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References

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