Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

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07/25/2019

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July 25, 2019

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FY19 Status of the Full Circumference Examination of the Inner Container Closure Weld Region for Selected 3013 DE Containers

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. In FY17, DE containers from FY13 through FY16 were evaluated to select candidates for a full circumference analysis of the ICCWR. In FY18, the selected DE containers for full circumference evaluation (FCE) were processed according to the ICCWR protocol and LCM data collection was completed for the full circumference only for FY15 DE07. In FY19 the remaining DE containers were completed.

The ICCWR protocol was completed as follows: (1) the FCE containers were sectioned into quarters and the weld removed to access the ICCWR; (2) a series of images of the ICCWR full circumference were taken using a stereo microscope and assembled into panoramic views; (3) SEM/EDS analysis performed on selected sections showed what is most likely corrosion products on the surface and chlorides randomly dispersed; (4) the presence of chlorides was also confirmed with ion chromatography of citric acid washes, in which FY15 DE07 and FY16 DE05 showed a significant amount of chlorides with FY15 DE08 showing the less amount of chlorides; (5) dye penetrant examination was performed on the interior and exterior surfaces of all the sidewall sections but reveled no relevant indication of surface-breaking defects, (6) finally, as described above, the full circumference examination of the ICCWR by LCM was completed.

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 FY15 DE08 on Section C2. These events correspond to suspect cracks or crack-like features identified with a unique name to easily refer to each feature. FY15 DE07 shows a crack-like feature, identified as Acrux, in Section C1 and three features

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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. Both features are located at the boundary of Zone 2 and Zone 3. However, gas analysis and statistical evaluation of the gas compositions and pressures via GEST analyses suggest that leakage between the OI and IC volumes was unlikely. Additional characterization and analysis will be performed on the locations where the cracks were found.

Introduction

The 2014 test plan for assessing the potential of stress corrosion cracking (SCC) of the 3013 inner container was issued by the Materials Identification and Surveillance (MIS) Corrosion Working Group to determine if SCC is plausible within the 50-year design life of a 3013 storage container [1]. One of the main focus areas is a thorough evaluation of the inner container closure weld region (ICCWR) opened for destructive examination (DE), which is part of the 3013 Surveillance Program. A protocol to investigate the corrosion in the ICCWR was developed to characterize the type of corrosion (i.e., mechanisms), the extent of corrosion (percentage of area and depth of attack) and the variables impacting this corrosion (chloride concentration and metallurgical condition) [2, 3]. Figure 1 shows an overview of the protocol for the examination of the ICCWR, which includes some updated steps from the original version [4]. The steps include (1) the sectioning of the inner container lid into easily handled pieces and weld removal, (2) low-magnification imaging of the entire circumference of the ICCWR, (3) surface analysis of selected pieces using Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS), (4) chemical analysis of selected pieces using wet chemistry techniques, (5) dye penetrant examination of exterior surfaces, (6) further sectioning of selected pieces into 1/8 can sections in preparation for LCM analysis, (7) cleaning selected pieces for removing corrosion products using nitric acid, (8) surface depth profiling and high-magnification imaging of the cleaned pieces with identified corroded areas within the ICCWR using LCM, and (9) serial metallography of pieces as necessary. Results from this characterization are used to assign a corrosion categorization to the respective ICCWR.

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. Initial analysis of selected DE containers using the LCM revealed several challenges for acquiring, processing and interpreting the data [4, 5]. These challenges include topography of the ICCWR sample, surface features, and the amount of surface area for collecting data at high magnification conditions. Consequently, the LCM parameters were investigated by imaging several samples with known cracks of different sizes to identify the appropriate parameter values for data acquisition and identification of regions of interest. Using these parameter values, selected DE containers were analyzed to determine the extent of the ICCWR to be examined. These parameters and conditions have been defined and reported in FY17 [6].

DE containers from FY13 through FY16 were evaluated to select candidates for a full circumference analysis of the ICCWR. This information will be used to perform a statistical analysis with Los Alamos National Laboratory (LANL) that will help support a determination of how much of an ICCWR needs to be examined in order to make the assertion of whether or not cracking has occurred and develop an ICCWR sampling plan for analysis of subsequent containers. 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 [6].

In FY18 the selected DE containers for full circumference analysis were processed according to the ICCWR protocol described above [7]. However, only the LCM data collection was completed for FY15 DE07. In FY19 the LCM data collection of the full circumference for FY16 DE05 and FY15 DE08 were completed. This report describes the results obtained from the LCM images.



Figure 1. Protocol overview for the examination of the ICCWR.

ICCWR Examination for Selected DE Containers

The protocol as described above was completed for FY15 DE07, FY16 DE05, and FY15 DE08. For simplicity, this set of DE containers are referred as the full circumference evaluation (FCE) containers. Also, under normal circumstances, an archive piece is selected for determining surface chloride distribution within the ICCWR using SEM/EDS while the remaining quarter pieces are used to quantify the chloride concentration using wet chemistry techniques. In this case, all pieces, including the one selected as archive, were needed to make the ICCWR full circumference. Consequently, all the quarter pieces were used for chloride quantification as reported in FY18 [7]. FY15 DE07 was examined by LCM in FY18 while FY16 DE05 and FY15 DE08 were examined in FY19. Sample preparation for LCM included (1) cutting 1/4 sections (A, B, C and D) into 1/8 sections of the container lid to reduce the curvature of each sidewall piece to be scanned and to fit the sample in the sample holder box (LCM box); (2) cleaning the samples from corrosion products using 2.0 M nitric acid solution and sonication for 60 min at 60°C; (3) mounting each 1/8 section into the LCM box minimizing tilt as much as possible. Detailed sample preparation is described in previous report [4]. Analysis by serial metallography is determined on as needed basis.

The LCM parameters used to collect data for the full circumference were determined in FY17 and are discussed in details in reference [6]. The parameters set during the data collection were the image magnification, measurement area (refers to x-y directions in pixels), quality (related to the laser speed), and pitch (refers to the spacing resolution in the z-direction). These parameters were selected for the examination of the ICCWR with detection of cracks of, at least, 1 μ m. Image and height data were collected on Zone 3 of the ICCWR using 20X magnification, with the double scan function disabled, measurement area set to standard, quality set to high accuracy, and a pitch value of 0.5 μ m. The vertical extent of the data collection in Zone 3 was 6 mm. Zones 1 and 2 were examined but images or height data were suspect cracks, significant number of pits or other corrosion features are located. However, due to the difficulty for observing a curved surface in real time because most of the surface is out-of-focus while a portion is in-focus, many of the sections have data collected in the full circumference of Zones 1 and 2. Data collection for Zones 1 and 2 used the same parameters as Zone 3, except the double scan function was enabled.

The optical data obtained from the LCM for the ICCWR full circumference of FY15 DE07 was reported in FY18 [7]. The optical data of FY16 DE05 is shown in Figure A1 through A8 in Appendix A and the optical data of FY15 DE08 is shown in Figure B1 through B9 in Appendix B. Each figure shows the set of assemblies that comprise the total for 1/8 section of the container. Each 1/8 container section was generally divided into 14 sub-sections for data collection because of the curvature nature of the sample in the x-direction as described in the previous report [7]. Figures A7 and A8 show loss of part of the ICCWR due to technical issues during the weld removal of FY16 DE05 Section D as explained previously [7].

ICCWR Full Circumference Examination by LCM of FY15 DE07

The data collected for FY15 DE07 shows general and localized corrosion on the surface. The major suspect corrosion events were observed on Sections C1 and C2. These events correspond to suspect cracks or crack-like features. In order to distinguish the different major features found on the ICCWR of DEs a unique name was used to easily refer to each feature. Figure 2 shows a summary of the potential crack features found on the ICCWR of FY15 DE07. The major features named Acrux in Section C1 and Bellatrix in Section C2 were described in FY18 [7]. Two additional features, Cursa Minor and Cursa Major, were found in Section C2 are shown in Figure 3. 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. The vertical length that can be observed from the optical image for Cursa Minor and Cursa Major is approximately 195 µm and 350 μm, respectively, as shown in Figure 3(a). Height scan data shown in Figure 3(b) indicates the presence of pits scattered throughout the surface. The horizontal linear profile (A-B) in Figure 3 (c) shows the depth of Cursa Major at the center as 17 µm and the profile (C-D) in Figure 3(d) shows the depth of Cursa Minor at the center as 34 µm. Also, a pit close to Cursa Minor shows a depth of 29 µm. However, gas analysis and statistical evaluation of the gas compositions and pressures via GEST analyses suggest that leakage between the OI and IC volumes was unlikely [8]. Sections C1 and C2 of FY15 DE07 were sent to Los Alamos National Laboratory (LANL) for characterization by X-Ray Tomography (XRT).



Figure 2. Summary of potential crack features found on the ICCWR of FY15 DE07showing (a) the picture of the inner container lid indicating the approximate location of features and (b) the corresponding panoramic assemblies of stereomicroscope images indicating the location of the features. Note that the panoramic assemblies are oriented such that the weld is above Zone 2



Figure 3. LCM optical images of the ICCWR of FY15 DE07 Section C2 showing (a) a portion of Zone 2 where the potential crack features are located, (b) a zoom of the region where Cursa Minor is located and (c) a zoom of the region where Cursa Major is located. Note that the picture is oriented such that the weld is below Zone 2.



Figure 4. LCM analysis for the area of the sidewall of FY15 DE07 Section C2 where Cursa Minor and Cursa Major are located showing (a) the optical image and (b) the height scan. Increasing height is represented by transition in color from blue (lowest) to red (highest). White lines (A-B and C-D) in the height scan show positions of (c) horizontal line profile across Cursa Major and (d) horizontal line profile across Cursa Minor.

ICCWR Full Circumference Examination by LCM of FY16 DE05

The data collected for FY16 DE05 shows general and localized corrosion on the surface. The major suspect corrosion events were observed on Section C2, which show two crack-like features. These two major features, Draco and Denebola, are indicated in Figure 5. Both features are located at the boundary of Zone 2 and Zone 3 as shown in Figure 6, While Denebola appears to be a large crack-like feature, Draco is a small feature that seems to be compose of several interconnected pits. Further examination will be required to determine if a crack has been developed. The length that can be observed from the optical image for Denebola is approximately 130 μ m with its widest opening of about 10 μ m, as shown in Figure 7(a). The height scan data is shown in Figure 7(b) with the horizontal linear profile (A-B) in Figure 7(c) and the vertical profile (C-D) in Figure 7(d) crossing Denebola around the region where it is deepest. Both profiles show the depth of Denebola at the center as 20 μ m. However, gas analysis and statistical evaluation of the gas compositions and pressures via GEST analyses suggest that leakage between the OI and IC volumes was unlikely [8]. Section C2 of FY16 DE05 will be examined by serial metallography at SRNL for characterization of the cross-sections by SEM.



Figure 5. Summary of potential crack features found on the ICCWR of FY16 DE05showing (a) the picture of the inner container lid indicating the approximate location of features and (b) the corresponding panoramic assemblies of stereomicroscope images indicating the location of the features. Note that the panoramic assemblies are oriented such that the weld is above Zone 2



Figure 6. LCM optical images of the ICCWR of FY16 DE05 Section C2 showing a portion of Zones 2 and 3 where the potential crack features are located. The insert window shows a zoom of the region where Draco is located. Note that the picture is oriented such that the weld is below Zone 2.



Figure 7. LCM analysis for the area of the sidewall of FY16 DE05 Section C2 where Denebola is located showing (a) the optical image and (b) the height scan. Increasing height is represented by transition in color from blue (lowest) to red (highest). White lines (A-B and C-D) in the height scan show positions of (c) horizontal line profile and (d) vertical line profile.

ICCWR Full Circumference Examination by LCM of FY15 DE08

The data collected for FY15 DE08 shows general and localized corrosion on the surface. Although no major suspect corrosion events were observed by LCM on this DE, the ICCWR is characterized by significant number of agglomerated pits as shown in Figure 8. Unlike, FY15 DE07 and FY16 DE05, agglomerated pits were found at a larger extent on Zones 2 and 3. Figure 9 shows the panoramic assembly of Section B1 before cleaning where it can be observer corrosion products along the weld. After the sample was cleaned for LCM analysis it can be observer the pitting agglomeration close to the weld on Zone 2. Although not a corrosion event, FY15 DE08 is also characterized, at a larger extent than the other two ICCWRs, by dent and scratches on the surface as it can be seen in Figure 8(b).



Figure 8. LCM optical images of the ICCWR of FY15 DE08 showing portions of Zones 2 and 3 with pits agglomeration as indicated by the red enclosures on (a) Section A2 and (b) Section B1.

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Figure 9. Panoramic assembly of stereomicroscope images of the ICCWR of FY15 DE08 Section B1 (top) showing corrosion products (before cleaning) along the weld. Portions of the LCM optical images (after cleaning), numbered 1-4, shows pitting just above the weld on Zone 2. Both stereomicroscope and LCM images are oriented such that the weld is below Zone 2.

Conclusions and Future Work

Corrosion evaluation of three prioritized DE containers, selected from candidates between FY13 and FY16, was performed for the ICCWR full circumference. The selected DE containers for full circumference evaluation (FCE) correspond to the following prioritization order: FY15 DE07, FY16 DE05, and FY15 DE08. In FY18 these DE containers were processed according to the ICCWR protocol and LCM data collection was completed for the full circumference only for FY15 DE07. In FY19 the remaining DE containers were completed.

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 FY15 DE08 on Section C2. These events correspond to suspect cracks or crack-like features identified with a unique name to easily refer to each feature. 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. Both features are located at the boundary of Zone 2 and Zone 3. However, gas analysis and statistical evaluation of the gas compositions and pressures via GEST analyses suggest that leakage between the OI and IC volumes was unlikely.

Additional characterization will be performed on the locations where the crack-like features were found. Sections C1 and C2 of FY15 DE07 were sent to LANL for characterization by XRT. Section C2 of FY16 DE05 will be examined by serial metallography at SRNL for characterization of the cross-sections by SEM. LANL and the University of South Carolina (USC) are working on methods for the analysis of the individual LCM images.

Acknowledgements

The assistance and efforts of Gregg Creech, Vickie Timmerman, and Kellie Holland are acknowledged in the execution of this work.

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Appendix A

Assemblies of Laser Confocal Microscope Images of ICCWR Sidewall Sections of FY16 DE05



Figure A.1. Side by side LCM assemblies of Zone 3 (top) and Zones 1 and 2 (bottom) of ICCWR for FY16 DE05 Section A1. Zone 1 corresponds to the weld beads, Zone 2 corresponds to the machining marks region (above weld beads), and Zone 3 is the region above the machining marks. Each image represents an assembly of individual images. Some overlap exits between assemblies to ensure data collection of 100% of full circumference.



Figure A.2. Side by side LCM assemblies of Zone 3 (top) and Zones 1 and 2 (bottom) of ICCWR for FY16 DE05 Section A2. Zone 1 corresponds to the weld beads, Zone 2 corresponds to the machining marks region (above weld beads), and Zone 3 is the region above the machining marks. Each image represents an assembly of individual images. Some overlap exits between assemblies to ensure data collection of 100% of full circumference.



Figure A.3. Side by side LCM assemblies of Zone 3 (top) and Zones 1 and 2 (bottom) of ICCWR for FY16 DE05 Section B1. Zone 1 corresponds to the weld beads, Zone 2 corresponds to the machining marks region (above weld beads), and Zone 3 is the region above the machining marks. Each image represents an assembly of individual images. Some overlap exits between assemblies to ensure data collection of 100% of full circumference.



Figure A.4. Side by side LCM assemblies of Zone 3 (top) and Zones 1 and 2 (bottom) of ICCWR for FY16 DE05 Section B2. Zone 1 corresponds to the weld beads, Zone 2 corresponds to the machining marks region (above weld beads), and Zone 3 is the region above the machining marks. Each image represents an assembly of individual images. Some overlap exits between assemblies to ensure data collection of 100% of full circumference.



Figure A.5. Side by side LCM assemblies of Zone 3 (top) and Zones 1 and 2 (bottom) of ICCWR for FY16 DE05 Section C1. Zone 1 corresponds to the weld beads, Zone 2 corresponds to the machining marks region (above weld beads), and Zone 3 is the region above the machining marks. Each image represents an assembly of individual images. Some overlap exits between assemblies to ensure data collection of 100% of full circumference.



Figure A.6. Side by side LCM assemblies of Zone 3 (top) and Zones 1 and 2 (bottom) of ICCWR for FY16 DE05 Section C2. Zone 1 corresponds to the weld beads, Zone 2 corresponds to the machining marks region (above weld beads), and Zone 3 is the region above the machining marks. Each image represents an assembly of individual images. Some overlap exits between assemblies to ensure data collection of 100% of full circumference.

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Figure A.7. Side by side LCM assemblies of Zone 3 (top) and Zones 1 and 2 (bottom) of ICCWR for FY16 DE05 Section D1. Zone 1 corresponds to the weld beads, Zone 2 corresponds to the machining marks region (above weld beads), and Zone 3 is the region above the machining marks. A section containing Zones 1 - 3 was lost during the weld removal. Each image represents an assembly of individual images. Some overlap exits between assemblies to ensure data collection of 100% of full circumference.



Figure A.8. Side by side LCM assemblies of Zone 3 of ICCWR for FY16 DE05 Section D2. Zones 1-2 and a section containing Zone 3 were lost during the weld removal. Each image represents an assembly of individual images. Some overlap exits between assemblies to ensure data collection of 100% of full circumference.

Appendix B

Assemblies of Laser Confocal Microscope Images of ICCWR Sidewall Sections of FY15 DE08



Figure B.1 Side by side LCM assemblies of Zone 3 (top) and Zones 1 and 2 (bottom) of ICCWR for FY15 DE08 Section A1. Zone 1 corresponds to the weld beads, Zone 2 corresponds to the machining marks region (above weld beads), and Zone 3 is the region above the machining marks. Each image represents an assembly of individual images. Some overlap exits between assemblies to ensure data collection of 100% of full circumference.



Figure B.2 Side by side LCM assemblies of Zone 3 (top) and Zones 1 and 2 (bottom) of ICCWR for FY15 DE08 Section A2a. Zone 1 corresponds to the weld beads, Zone 2 corresponds to the machining marks region (above weld beads), and Zone 3 is the region above the machining marks. Each image represents an assembly of individual images. Some overlap exits between assemblies to ensure data collection of 100% of full circumference.



Figure B.3 Side by side LCM assemblies of Zone 3 (top) and Zones 1 and 2 (bottom) of ICCWR for FY15 DE08 Section A2b. Zone 1 corresponds to the weld beads, Zone 2 corresponds to the machining marks region (above weld beads), and Zone 3 is the region above the machining marks. Each image represents an assembly of individual images. Some overlap exits between assemblies to ensure data collection of 100% of full circumference.



Figure B.4 Side by side LCM assemblies of Zone 3 (top) and Zones 1 and 2 (bottom) of ICCWR for FY15 DE08 Section B1. Zone 1 corresponds to the weld beads, Zone 2 corresponds to the machining marks region (above weld beads), and Zone 3 is the region above the machining marks. Each image represents an assembly of individual images. Some overlap exits between assemblies to ensure data collection of 100% of full circumference.



Figure B.5 Side by side LCM assemblies of Zone 3 (top) and Zones 1 and 2 (bottom) of ICCWR for FY15 DE08 Section B2. Zone 1 corresponds to the weld beads, Zone 2 corresponds to the machining marks region (above weld beads), and Zone 3 is the region above the machining marks. Each image represents an assembly of individual images. Some overlap exits between assemblies to ensure data collection of 100% of full circumference.



Figure B.6 Side by side LCM assemblies of Zone 3 (top) and Zones 1 and 2 (bottom) of ICCWR for FY15 DE08 Section C1. Zone 1 corresponds to the weld beads, Zone 2 corresponds to the machining marks region (above weld beads), and Zone 3 is the region above the machining marks. Each image represents an assembly of individual images. Some overlap exits between assemblies to ensure data collection of 100% of full circumference.

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Figure B.7 Side by side LCM assemblies of Zone 3 (top) and Zones 1 and 2 (bottom) of ICCWR for FY15 DE08 Section C2. Zone 1 corresponds to the weld beads, Zone 2 corresponds to the machining marks region (above weld beads), and Zone 3 is the region above the machining marks. Each image represents an assembly of individual images. Some overlap exits between assemblies to ensure data collection of 100% of full circumference.



Figure B.8 Side by side LCM assemblies of Zone 3 (top) and Zones 1 and 2 (bottom) of ICCWR for FY15 DE08 Section D1. Zone 1 corresponds to the weld beads, Zone 2 corresponds to the machining marks region (above weld beads), and Zone 3 is the region above the machining marks. Each image represents an assembly of individual images. Some overlap exits between assemblies to ensure data collection of 100% of full circumference.

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Figure B.9 Side by side LCM assemblies of Zone 3 (top) and Zones 1 and 2 (bottom) of ICCWR for FY15 DE08 Section D2. Zone 1 corresponds to the weld beads, Zone 2 corresponds to the machining marks region (above weld beads), and Zone 3 is the region above the machining marks. Each image represents an assembly of individual images. Some overlap exits between assemblies to ensure data collection of 100% of full circumference.