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Residual Stresses in 3013 Containers

By

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Abstract

The DOE Complex is packaging plutonium-bearing materials for storage and eventual disposition or disposal. The materials are handled according to the DOE-STD-3013 which outlines general requirements for stabilization, packaging and long-term storage. The storage vessels for the plutonium-bearing materials are termed 3013 containers. Stress corrosion cracking has been identified as a potential container degradation mode and this work determined that the residual stresses in the containers are sufficient to support such cracking. Sections of the 3013 outer, inner, and convenience containers, in both the as-fabricated condition and the closure welded condition, were evaluated per ASTM standard G-36. The standard requires exposure to a boiling magnesium chloride solution, which is an aggressive testing solution. Tests in a less aggressive 40% calcium chloride solution were also conducted. These tests were used to reveal the relative stress corrosion cracking susceptibility of the as fabricated 3013 containers. Significant cracking was observed in all containers in areas near welds and transitions in the container diameter. Stress corrosion cracks developed in both the lid and the body of gas tungsten arc welded and laser closure welded containers. The development of stress corrosion cracks in the as-fabricated and in the closure welded container samples demonstrates that the residual stresses in the 3013 containers are sufficient to support stress corrosion cracking if the environmental conditions inside the containers do not preclude the cracking process.

Introduction

The DOE Complex is packaging plutonium-bearing materials for storage according to the DOE-STD-3013 [1] which outlines, in part, the specification for the 3013 storage containers. The internal environment for some 3013 containers is such that chloride stress corrosion cracking (SCC) is a potential degradation mode of the stainless steel containers during storage [2]. The three factors necessary for SCC are a susceptible material, a corrosive environment, and a tensile stress. The stress can result from either an applied load or a residual stress resulting from fabrication or welding processes. For the 3013 containers, both fabrication and welding residual stresses may be present. The outer container has both a fabrication weld that joins the bottom to the sidewall and a closure weld. The convenience and inner containers are made by a flow form process, which cold works the stainless steel during fabrication. The inner containers also are sealed at the container top with a closure weld to the sealing plug. The residual stresses that are produced by fabrication and welding processes may exceed the nominal yield

strength of the stainless steel and are particularly problematic in the weld heat affect zone (HAZ) where microstructure also contributes to the potential for SCC [3].

Testing was performed under several conditions to determine if the residual stresses were sufficient for the initiation and propagation of SCC. Testing in boiling magnesium chloride is an accepted ASTM standard practice for assessing the effects of material properties (composition, surface finish, microstructure, heat treatment, etc.) and stress conditions on susceptibility to chloride SCC [4]. The boiling magnesium chloride solution is a severe environment that may be much more aggressive than the actual exposure environment, such as that encountered in 3013 containers during storage [4, 5].

Less aggressive SCC testing has been developed so that the test results correlate better with actual industrial service. Two such environments are a boiling acidified sodium chloride solution (ASTM G123 [6]) and a 40% calcium chloride solution at 100 °C. The 40% calcium chloride solution was used in this study because cracking had occurred during corrosion tests under simulated storage conditions with salts containing calcium chloride [7, 8]. For these simulated storage tests, the test coupons were teardrop-shaped coupons fabricated of Type 304L stainless steel. Similar teardrop-shaped coupons and an inner-container welded-top section were tested in the 40% calcium chloride solutions to provide comparative results for assessing the effect of residual stresses in a less aggressive environment which is more relevant to 3013 containers.

Experimental Test Setup

The SCC testing was performed with different solutions, exposure conditions, and samples to assess the effect of residual stresses on the SCC susceptibility of 3013 containers. The various test conditions are discussed herein.

Test Samples

Test samples consisted of both standard commercial teardrop coupons and specimens cut from 3013 containers. The teardrop coupons were made of 304L stainless steel (304L). Two sizes were used with dimensions of 0.16 cm (0.0625 in) thick × 1.91 cm (0.75 in) wide × 10.16 cm (4.0 in) length and 0.16 cm (0.0625 in) thick × 1.27 cm (0.5 in) wide × 6.35 cm (2.5 in) length. The different sizes were used to develop comparison data with previous testing performed in simulated 3013 environments containing plutonium and the test results with actual 3013 containers.

A 3013 container system consists of up to three containers, outer, inner, and usually a convenience container, which are shown in Figure 1. The outer container is made of 316L stainless steel (316L), while the inner and convenience containers are made of 304L. The 316L outer container is fabricated from a formed bottom, a sidewall, and a top lid. The sidewall and bottom sections are butt joined together by GTA welding. The two types of closure welds approved for use on the outer container were tested, namely a laser beam weld and a gas tungsten arc (GTA) weld. The inner and convenience container bodies are fabricated by a flow form process. Flow forming is a cold metal forming process where a preform is extruded over a rotating mandrel to produce a

rotationally symmetrical hollow component. At the Savannah River Site and Hanford Site, the inner container lid is also GTA welded, while a convenience container, if used, does not have a welded top. For these tests, the containers were sectioned by electrodischarge machining (EDM) at the center height so that the residual stress states would remain as undisturbed as possible. Each container half was used as both a test specimen and a solution container.

The inner containers had been used previously for other types of testing and had several small holes in the bottom and side walls. To prevent the test solution from leaking through the container sections, these holes were filled with weld filler metal and then ground flat. The holes on the bottom were not completely filled so a small 0.64 cm (0.25 in) diameter hole partially through the bottom was present on the solution side.

Table 1 gives the test matrix for the container sections and teardrop coupons. Post-test analyses were primarily visual evaluations documented by photography. Limited metallographic examinations and dye penetrant testing were performed also.

Table 1 Test Matrix

Test Sample	Boiling MgCl₂ – 155 °C	40 % CaCl₂ – 100 °C
Outer Container – Welded Top	X	
Outer Container - Bottom	X	
Inner Container – Bottom	X	
Inner Container –Welded Top	X	X
Hanford Convenience	X	
SRS Convenience	X	
304L Teardrops	X	X

Test Solutions

Test samples were exposed to either a boiling magnesium chloride solution per ASTM G36 or a 40% calcium chloride solution at 100 °C following the guidance of ASTM G123. ASTM G36 is an accelerated, aggressive test and involves exposing test samples to a boiling magnesium chloride solution for a user-defined test period. The calcium chloride test at 100 °C was performed to determine crack initiation and propagation rates in an environment that was more similar to the small scale corrosion test environment where SCC was observed [6].

Test Setup

The magnesium chloride tests used the container section as the sample as well as the vessel to contain the chloride solutions. The test section was placed in a larger containment vessel to collect any solution that may leak through the container specimen if cracking occurred during testing. The nested assembly was then placed on a hot plate. The test solution (45% magnesium chloride) components, 600 g of MgCl₂·6H₂O and 15 mL of water, were placed into the container specimen. A glass lid was made to fit snugly

into the opening of the container specimen with ports for a thermometer and a water-cooled condenser which had a liquid trap on top. A solution of 25% magnesium chloride was placed in the liquid trap per the ASTM standard. Once the boiling temperature of the test solution was established at $155.0 \pm 1^\circ\text{C}$, any necessary temperature adjustments were made during the test by either adding additional magnesium chloride or water. The teardrop coupons were suspended in boiling (155°C) magnesium chloride solutions contained in glass vessels. Tests generally were conducted until the samples cracked.

At the conclusion of a test, the container specimens were filled with water to determine if leaking occurred from both known and unknown cracks. If leaking did not occur the container specimens were tested with dye penetrant to determine if cracking had occurred.

The potential for corrosion in the interior vapor space of 3013 containers was tested on one inner container with a welded top that was suspended over a boiling magnesium chloride solution for five days. The solution and container top section were enclosed in a glass vessel. The remainder of the set up was similar to the procedure described above.

The calcium chloride test solutions were prepared using standard reagent chemical and distilled water which was added to either the container specimen or standard laboratory glassware for the teardrop coupons. The container specimen set up was similar to that used for the magnesium chloride testing shown in Figure 2. The test solution was heated to 100°C and maintained until the container specimen or teardrop coupons cracked.

Results

Cracking occurred in all container specimens exposed to either the boiling magnesium chloride or the 40% calcium chloride solutions. These results demonstrate that residual stress levels were sufficient to cause SCC in numerous locations in the 3013 containers. The test results for the 3013 container specimens tested in boiling magnesium chloride solutions are summarized in Table 2. Multiple tests were performed on inner container bottom sections because of the variable results and the complications from the previous testing holes, which were located in the bottom and sidewall. Two inner container top sections were tested for reproducibility.

Table 2 Boiling Magnesium Chloride Test Results for 3013 Container Specimens

Container Section	Hours in Test	Crack Locations
Outer Container – Bottom with fabricaton weld	48	Axial cracks in sidewall Radial cracks in bottom
GTAW Closure Weld outer container	48	Axial cracks in sidewall Radial cracks in bottom Parallel crack near weld
LBC Weld outer container	48	Axial cracks in sidewall Radial cracks in bottom

		Pitting on interior container bottom
Inner Container #1 – Bottom No sidewall hole	5	EDM cut rim Bottom drilled hole Circumferential
Inner Container #4 – Bottom	24	Sidewall hole Bottom drilled hole Circumferential
Inner Container #2 – Bottom	48	Sidewall hole Bottom drilled hole Circumferential and axial in sidewall
Inner Container #3 – Bottom No sidewall hole	48	EDM cut rim Bottom drilled hole
Inner Container #3 – Top	2	Perpendicular to weld
Inner Container #1 – Top	3.5	Parallel and perpendicular to weld
Inner Container #2 - Top – Vapor	120	Small cracks in crevice perpendicular to weld
SRS Convenience Container	5.5	Axial in sidewall and radial in bottom
Hanford Convenience Container	48	Circumferential and axial in sidewall
304 L Teardrops	1.3	Through wall near weld

Outer Container – Fabrication Weld

Through wall cracks were observed in the bottom half of the as-fabricated container specimen after 48 hours of testing in boiling magnesium chloride solutions, Figure 3. Other cracks were observed on the interior of the container specimen that did not appear to propagate through the wall. Five through wall cracks which ranged in length from 0.38-2.06 cm (0.15-0.81 in) were visible. All of the cracks observed were generally perpendicular to the base-to-container fabrication weld. Metallographic examination of one crack showed that the cracking initiated in the HAZ and grew into weld and base of the container specimen, Figure 4. The cracks were typical of stress corrosion cracks with regions of branching along their length and some areas of cracking 90° from the main crack. The location and orientation of the cracks are consistent with the residual stresses expected as a result of the base-to-container weld and indicate that the residual stress state is significant in the weld region of the as-fabricated container.

Outer Container - GTAW Closure Weld

Through wall stress corrosion cracks perpendicular to the GTA closure weld and along the length of the container specimen body were observed after 48 hours of testing in boiling magnesium chloride solutions, Figure 5 (a). Through thickness radial cracks were also observed on the lid, Figure 5 (b). All of these cracks are consistent with the residual stresses expected as a result of the GTA closure weld. A crack that grew perpendicular to the weld while in the weld metal turned to be parallel to the closure weld in the heat affected zone was also observed using dye penetrant testing. The external cracks in the lid and through the weld along the length of the container specimen (Figure 5) were not

imaged using dye penetrant testing. This observation indicates that the external crack was insufficiently open to allow the dye penetrant to move into the crack even though the cracks penetrated through the container wall.

Outer Container - Laser Beam Welds

Through wall cracks perpendicular to the weld and along the length of the container specimen body were observed in a sample containing a laser closure weld. These cracks were observed after the sample was exposed to boiling magnesium chloride solution for 48 hours. Radial cracks on the lid were also observed, Figure 6. All of these cracks are consistent with the residual stresses expected as a result of the laser closure weld. Pits were also observed inside the container specimen, Figure 6. The pits may be chloride induced pits that are exacerbated by the high residual stresses developed during the closure weld. Dye penetrant testing of the exterior of the container specimen showed that the cracks in both the lid and the body were through wall cracks and ran continuously from the lid into the body

Inner Container – Bottom

The cracking in inner container bottom specimens occurred at several locations and not all container specimens cracked at each location. The container specimens all had a hole drilled in the bottom, which was sealed with weld metal as discussed previously. The container specimens cracked and generally leaked at this location. Two container specimens had a hole in the sidewall, which was formed during a container puncture test and cracking also occurred at these holes. Cracks from both these holes followed an irregular circuitous path.

Post-test water leak checks showed leaks only from the bottom and the sidewall drilled hole. Dye penetrant testing on the container interiors however showed a circumferential crack near regions of the container where the diameter changed, Figure 7. Small portions of these cracks were through wall. One inner container bottom section did not develop any cracks other than those associated with the drilled holes. This variability in cracking for the inner container bottom may be associated with differences in container fabrication as well as testing variability.

Inner Container – Welded Top

The welded tops on the inner container specimens cracked after 2 and 3.5 hours exposure in the boiling magnesium chloride. Testing was stopped because large amounts of the test solution were leaking from the cracks. The cracks were along the weld, perpendicular to the weld along the container specimen axis, and radial from the plug perimeter, Figure 8. The crack along the weld occurred in the container specimen which was exposed for the longer time and covered approximately 60% of the perimeter. The cracks in the sidewall were greater in number than those in the plug. Additionally, the cracks were longer (0.5-0.9 cm (0.2-0.35 in) length) and a greater number of the cracks had propagated through wall. The radial and perpendicular cracks were straight and unlike the cracks typically generated at the drilled holes.

The welded top of inner container #1 was sectioned to look for evidence of corrosion within the crevice formed by the top plug and sidewall. Although large pits were found in the plug and sidewall there was no evidence of crevice corrosion. In many cases the pits which were observed were associated with the cracks as shown in Figure 9. The small cracks had multiple orientations relative to the pits. The growth patterns for these cracks indicated a complex stress state near the weld.

The welded tops of the inner container specimen exposed to the vapors of the boiling magnesium chloride solution had small cracks in both the sidewall and top after five days of exposure. The cracks were not through wall. The container specimen interior showed only a small degree of corrosion although the surface was wetted during testing. The container specimen was sectioned by EDM so that the interior surface of the crevice could be inspected for pitting and cracking since the use of dye penetrant testing was unsuccessful. Several pie-shaped sections were cut from the container specimen. The crevice was revealed by flattening. Large pits and small axial cracks were found inside the crevice and when the sample sections were flattened to open the crevice, the cracks also opened up, Figure 10. Additional cracks were found on the side of plug.

Convenience Container

The two convenience container specimens both cracked in the boiling magnesium chloride test. One container did not leak after 48 hours while the other leaked to such an extent that the test had to be stopped after only 5.5 hours of exposure. Most of the cracks were in the region of the diameter change of the container specimen, Figure 11. No cracks were found on the bottom of one convenience container specimen, while the other had radial cracks. The convenience container specimens that lasted for 48 hours showed only minimal leaking during the post-test leak check, but leaked severely after tapping to loosen the cracks.

304L Teardrops Coupons

304L teardrop coupons with autogenous welds were exposed to plutonium-oxide-bearing material with 0.2 % calcium chloride in simulated 3013 corrosion tests performed at room temperature. These specimens cracked after 166 hours of exposure [6]. The relationship of corrosion susceptibility of these teardrop coupons to flow formed 304L containers has not been established. As a measure of this relationship, teardrop coupons identical to those used in the small scale test were tested in boiling magnesium chloride solution. The teardrop coupons failed after approximately 1.3 hours of exposure with a full-width through wall crack, Figure 12. The crack was near the weld but outside the HAZ. The small-sized teardrop coupon failed in approximately 2 hours with a through wall crack at a similar location to the cracks in the large teardrop coupons.

Inner Container – Welded Top – Calcium Chloride Test

The test with the inner container welded-top specimen exposed to calcium chloride solutions at 100 °C was performed for approximately 84 hours. Multiple cracks grew perpendicular to the weld in the sidewall, similar to those in the boiling magnesium chloride tests. The cracks were approximately 0.5-0.7 cm (0.2-0.27 in) long as measured

macroscopically on the exterior sidewall. Many of these cracks were through wall. Cracks were also found in the top plug but only two were suspected to be through wall.

Discussion

The boiling (155 °C) magnesium chloride test is designed to indicate the susceptibility of stainless steels and related alloys to SCC and is not meant to be a quantitative test. The test was used in this study to identify locations of high residual stresses associated with fabrication and welding of the stainless steel 3013 container. Time to failure in these solutions is inversely related to the level of residual stress [9].

The 3013 outer, inner, and convenience containers all had sufficient residual stresses to crack in the boiling magnesium chloride solution, although in some tests where leaking did not occur the time to failure is not known. For the 316L outer containers, leaking did not occur during the test, but cracks were evident in the as-fabricated container specimen after 48 hours. The closure welded regions of the outer container specimens also developed similar cracks. The cracks observed in the as-fabricated outer container specimen, however, were not as numerous as those observed in the closure welded container specimens. Cracks observed in the laser closure welded container specimens were numerous and appeared to be more severe when compared to those observed in the GTA closure welded specimens.

The 304L teardrop coupons had a very short time to failure (approximately 1.3 hours) and clearly provide a conservative estimate for the behavior of the 3013 containers. The stress levels in a teardrop coupon have been shown to exceed the yield stress and approach levels equivalent to the ultimate tensile strength [8]. By comparison a welded top of an inner container specimen failed in 2 hours, which suggests that the residual stresses in the weld may be lower than the stresses in the teardrop coupons. The other welded top of an inner container specimen was tested for 3.5 hours and had a more significant degree of cracking with a circumferential crack running parallel to the weld. The perpendicular and parallel cracking near the weld indicate a complex stress state in the weld region. The inner container bottom section appears to have lower stress levels than those in the seal welds since the degree of cracking was less (i.e. smaller number of through wall cracks) and the time to cracking is greater (i.e. one container tested for 48 hours had no cracks in the sidewall).

The times to cracking for the convenience container specimens were approximately 5.5 hours. Fabrication stresses for the convenience container may thus approach those resulting from welding since times to failure were similar to the welded tops of the inner container specimens.

Additionally the inner container welded-top specimen cracked in the vapors of the boiling magnesium chloride solution. The cracks were small and not through wall, but extended exposure beyond the five-day test period could have resulted in similar cracking to those inner container welded-top specimens exposed directly to the boiling magnesium chloride

solution. These results show that the residual stresses in a weld are sufficient to cause cracking when chloride-containing vapors condense on the stainless steel surface.

The results from the calcium chloride test showed that cracking can occur in the 3013 containers in environments less severe than a boiling magnesium chloride solution, and as expected, the time to cracking was significantly longer. Testing was conducted with only an inner container welded-top specimen. Similar results would be expected for the other containers but only at longer times since they failed at longer times in the magnesium chloride tests. The cracking morphology was similar to that observed in the magnesium chloride tests, i.e. short perpendicular cracks to the weld. Cracking also is possible in the vapor space of such environments since cracking occurred in the 304L teardrop coupons, which was exposed to the vapor of a 40 % calcium chloride solution. The vapor space cracking was significantly less than that observed on teardrop coupons immersed in the solution. Crack growth rates were not determined for the immersed teardrop coupons because multiple cracks initiated.

Conclusion

The 3013 container is a nested set of three low-carbon grade 300 series stainless steel containers and is robust for packaging plutonium-bearing materials and eventual storage at various sites in the DOE Complex. Corrosion associated with impurities (specifically chlorides and fluorides) in the plutonium-bearing materials may lead to SCC of the containers. ASTM standard G-36, an aggressive testing procedure with boiling magnesium chloride solution, was used to identify the regions of the 3013 container that are most susceptible to potential degradation associated with SCC. For the 3013 outer container, significantly large residual stresses were in the bottom half of the as-fabricated container, which resulted from the base to container fabrication weld. This observation indicated that regardless of the closure weld technique, sufficient residual stresses exist to provide the stress component necessary for SCC. Cracks observed in both the lid and the body of the GTA and the laser closure welded containers indicates that significant residual stresses are inherent in the closure welding process regardless of the closure weld method.

Testing of 3013 inner and convenience containers has shown that residual stresses sufficient to support SCC exist at both the inner container closure weld and in sections of the containers where the container diameter changes, i.e. near the container bottom. Cracking was found to occur sooner in the inner container welded-top specimen than the container bottom specimen of either the inner or convenience container. This shorter time is indicative of higher residual stresses from welding than fabrication. The microstructure in the HAZ is likely to provide an added component for pitting corrosion and SCC.

Teardrop coupons are a conservative marker for the containers since failure in the boiling magnesium chloride test occurred in even shorter times than the welded top. Cracking from the condensed vapors of both the hot calcium chloride and boiling magnesium chloride solutions occurred, so SCC may be a plausible mechanism for the 3013

containers if a thin chloride-bearing film forms above the oxide/salt mixture. The chloride concentrations in the 3013 containers are expected to be a small fraction of those used in this testing.

The net result of these studies is that the microstructure of the austenitic stainless steel and the residual stresses present in the steel combine to create 3013 containers (convenience, inner and outer) that are susceptible to chloride induced stress corrosion cracking. Therefore, assurance against such cracking must be provided by environmental controls during the packaging and storage processes. The 0.5 wt% moisture limit is the primary control designed to provide this assurance.

Acknowledgements

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Figure 1. 3013 containers (outer, inner, convenience) used by Savannah River Site for oxides

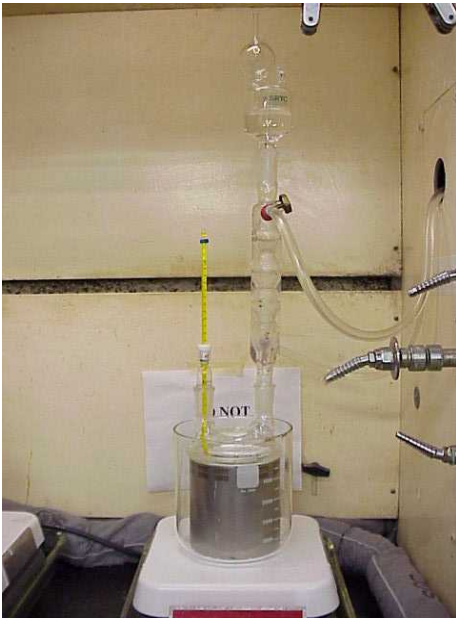


Figure 2. Test setup for boiling magnesium chloride testing



Figure 3. Bottom of as-fabricated 3013 outer container after boiling Magnesium Chloride test: a) Exterior through wall cracks; b) Interior cracks, arrows indicate cracks

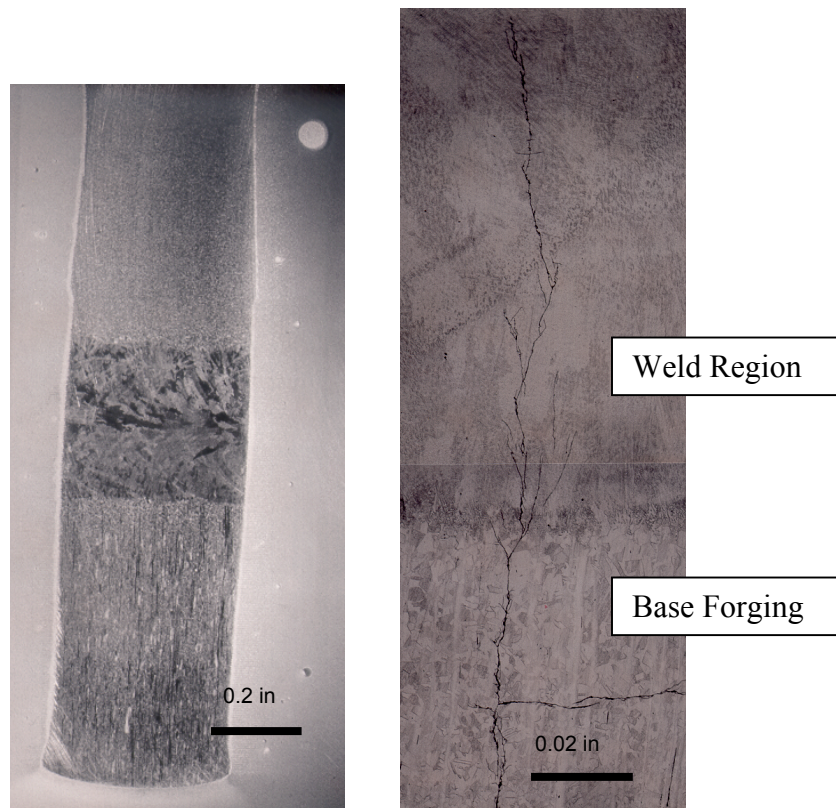


Figure 4. Metallographic cross section of through wall crack at the container to base fabrication weld in 3013 outer container after boiling magnesium chloride test: a) Overall macrograph showing base of container through fabrication weld and container body; b) Stress corrosion crack from sample shown in (a). Note the crack branching and the position of the crack, beginning in the weld region and propagating into the base of the container

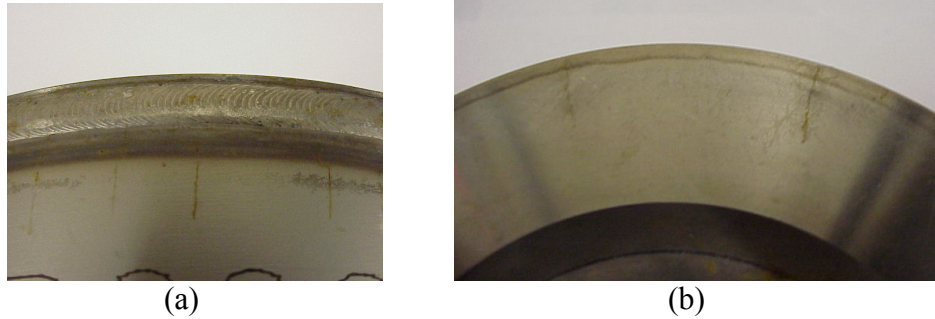


Figure 5. Top half of GTAW closure welded 3013 outer container section with through wall cracks after boiling magnesium chloride test: a) Exterior of container with cracks perpendicular to GTAW weld and running into container body (b) Exterior of lid showing appearance of cracks perpendicular to GTAW weld in lid

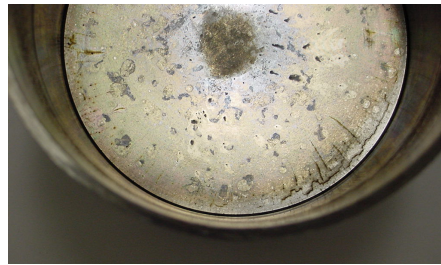


Figure 6. Laser beam closure welded 3013 outer container after boiling magnesium chloride test with interior pitting on lid as well as crack indications

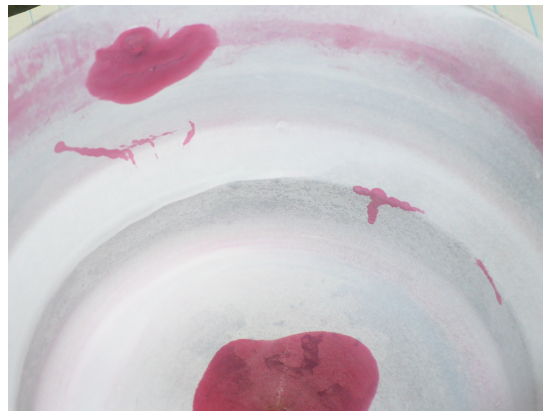


Figure 7. Dye penetrant testing of 3013 inner container bottom section; pink lines indicate crack locations and pink spots are the drilled hole locations



Figure 8. 3013 inner-container welded-top section after exposure to boiling magnesium chloride showing container interior with sidewall and plug cracks



Figure 9. 3013 inner-container welded-top section after exposure to boiling magnesium chloride solution showing pit associated with cracking in sidewall



Figure 10. 3013 inner-container welded-top section exposed to vapors of boiling magnesium chloride solution: interior container crevice between the side wall and seal plug spread open showing small cracks



Figure 11. Convenience container section after exposure to boiling magnesium chloride solution: container exterior with axial cracks in sidewall



Figure 12. 304L teardrop coupons after exposure to a boiling magnesium chloride solution