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Residual Stresses in SAVY 4000 and Hagan Container Bodies

Mary Ann Stroud, Mary Ann Hill, Justin Tokash, Robert Forsyth and Holden Hyer

Abstract

Chloride-induced stress corrosion cracking (SCC) has been investigated as a potential failure mechanism for the SAVY 4000 and the Hagan containers used to store plutonium-bearing materials at Los Alamos National Laboratory. This report discusses the regions of the container bodies most susceptible to SCC and the magnitude of the residual stresses in those regions. Boiling $MgCl_2$ testing indicated that for both containers the region near the top weld was most susceptible to SCC. The Hagan showed through wall cracking after 22-24 hours of exposure both parallel (axial stresses) and perpendicular (hoop stresses) to the weld. The SAVY 4000 container showed significant cracking above and below the weld after 47 hours of exposure but there was no visual evidence of a through wall crack and the cracks did not leak water. Two through wall holes formed in the bottom of the SAVY 4000 container after 44-46 hours of exposure. For both containers, average "through wall" residual stresses were determined from hole drilling data 4 mm below the weld. In the Hagan body, average tensile hoop stresses were 194 MPa and average compressive axial stresses were -120 MPa. In the SAVY 4000 body, average compressive hoop stresses were 11 MPa and average tensile axial stresses were 25 MPa. Results suggest that because the Hagan container exhibited through wall cracking in a shorter time in boiling $MgCl_2$ and had the higher average tensile stress, 194 MPa hoop stress, it is more susceptible to SCC than the SAVY 4000 container.

Introduction

The Hagan was used as the primary container for packaging of plutonium-bearing materials at Los Alamos National Laboratory since the late 1990s. The SAVY 4000 container was introduced as a replacement for the Hagan with an initial design lifetime of 5 years as of April 2014. A lifetime extension program has been initiated to determine how long a SAVY 4000 container may be used safely, potentially extending the design lifetime. A surveillance program has been initiated to observe SAVY 4000 and Hagan containers during usage. Results on SAVY 4000 containers with in-service usage up to 4.9 years and Hagan containers with usage up to 14.4 years have been obtained. Surveillance testing found corrosion in 16 of the 68 containers inspected. It was believed that hydrochloric acid gas generated inside these containers led to the observed corrosion.[1] Because of these findings, chloride-induced stress corrosion cracking (SCC) has been investigated as a potential failure mechanism for the SAVY 4000 and the Hagan containers. Stress corrosion cracking is induced from the combined influences of tensile stress, a corrosive environment and a susceptible material. This report discusses the regions of the container bodies most susceptible to SCC and the magnitude of the residual stresses in those regions. The relative susceptibility of the Hagan and SAVY 4000 containers is also discussed for possible application in the lifetime extension program.

SCC tests in boiling magnesium chloride solution were conducted on SAVY 4000 and Hagan bodies to identify the regions most susceptible to SCC. The magnitude of the residual stresses in susceptible regions was determined using hole drilling. Results of the experiments are used to provide a relative comparison of the susceptibility of the SAVY 4000 and Hagan container bodies to chloride induced corrosion.

Description of the SAVY 4000 and Hagan Container Bodies

One of the factors necessary for SCC to occur is the presence of tensile stress in the container. For the unannealed Hagan container both fabrication and welding residual stresses may be present. The Hagan body is 304L stainless steel (SS) formed from a deep drawing process. Gas tungsten arc welds (GTAW), also known as tungsten inert gas (TIG) welds, are present both near the top of the container and midway down the side of the container. The SAVY 4000 body is 316L SS and is also formed from a deep drawing process but is annealed after fabrication to relieve residual stresses. The collar material is annealed prior to machining only. The SAVY 4000 container has a Laser Beam Weld (LBW) near the top of the container that connects the body to the collar. The SAVY 4000 and Hagan container have relatively thin walls, 0.76 mm and 0.75 mm respectively. The bottom of the SAVY 4000 container is approximately 20% thinner at 0.6 mm. Container lids were not investigated in this study. The container lids are expected to be less susceptible to SCC because the walls of the container lids are thicker and do not contain welds.

Experimental Procedure for Container Sections

Note: The boiling magnesium chloride solution is a severe environment that is more aggressive than the actual exposure environment encountered in SAVY 4000 *and Hagan containers during storage. A container body that normally provides acceptable resistance in a chloride environment may crack in this test.*

A boiling magnesium chloride ($MgCl_2$) test was conducted on the bodies and body sections of Hagan and SAVY 4000 containers. The boiling $MgCl_2$ environment provides an accelerated method for ranking the relative degree of stress-corrosion cracking susceptibility of the bodies in aqueous chloride-containing environments. The location of SCC caused by the boiling $MgCl_2$ identifies of the regions of greatest susceptibility. Time to failure data provides a ranking of the relative degree of stress-corrosion cracking susceptibility for the container bodies.

Initially, sections of a one quart Hagan and SAVY 4000 containers were tested in boiling $MgCl_2$. Photographs of the Hagan section prior to exposure to the boiling $MgCl_2$ are shown in Figures 1 and 2 .

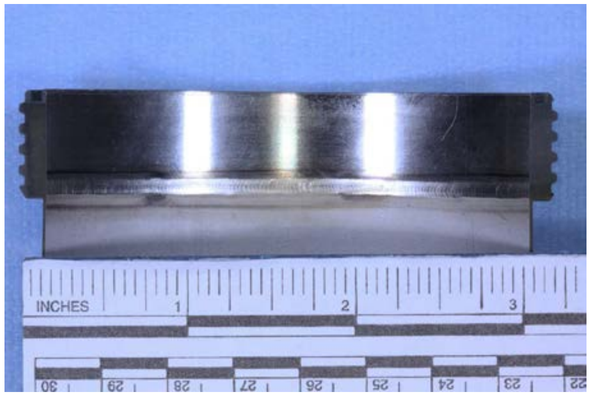


Figure 1 Photographs of the inner contour of the Hagan-1 section prior to exposure to boiling $MgCl_2$.

a) b)

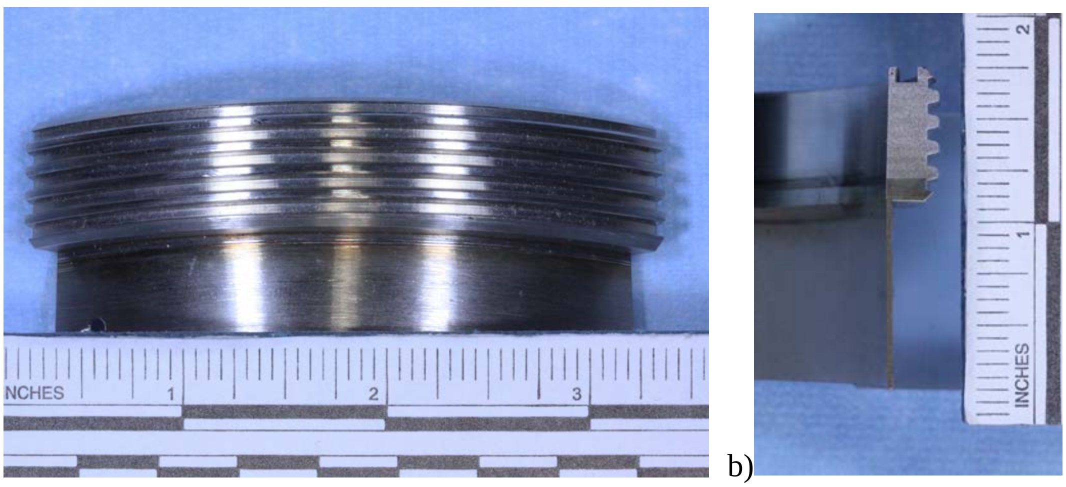


Figure 2 Photographs of the a) outer contour and b) edge of the Hagan-1 section prior to exposure to boiling $MgCl_2$

Photographs of the quarter section of the SAVY 4000 prior to exposure to boiling $MgCl_2$ are shown in Figure 3.

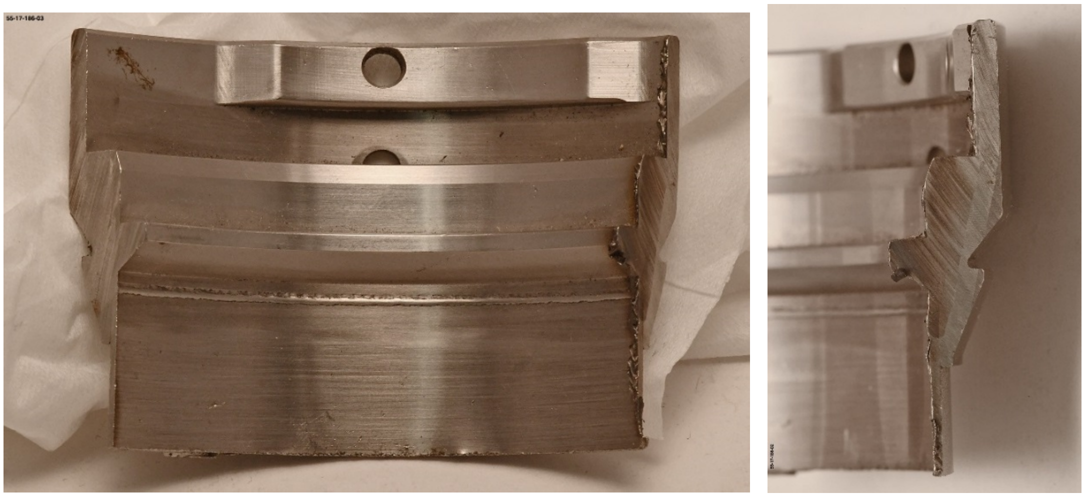


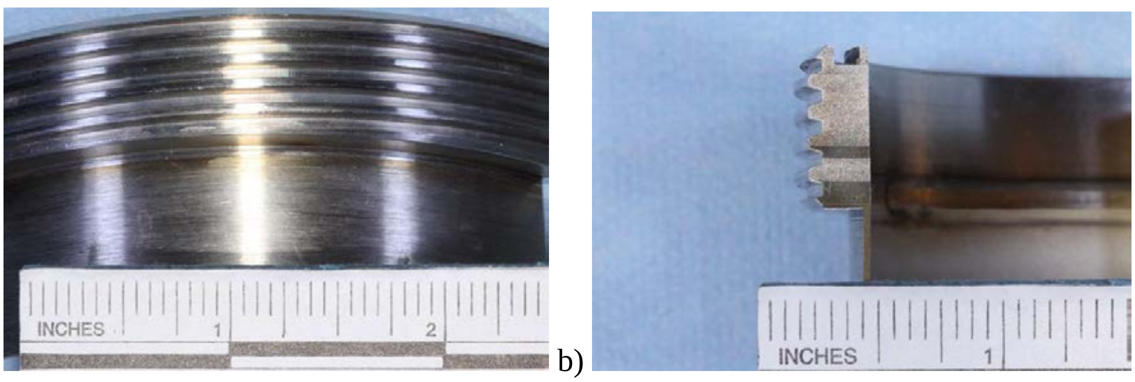
Figure 3 Photographs of the a) inner contour and b) edge of the SAVY 4000 section prior to exposure to boiling $MgCl_2$.

A quarter section of a 3013 SRS/Hanford inner container was also present in the flask during the experiment. All container sections were ultrasonically cleaned for five minutes in Alconox detergent, and subsequently rinsed with isopropyl alcohol. The test was in accordance with *ASTM standard G36: Evaluating resistance to SCC of metals and alloys in boiling $MgCl_2$* [2] except that temperatures were lower than the 155º C stated in the standard. No boiling chips were used. Glassware and set-up (Figure 4) is described in ASTM G36.

Figure 4 Experimental set-up for the boiling $MgCl_2$ experiment on container pieces.

A 1.3 kg mass of $MgCl_2 \cdot 6H_2O$ was weighed out and mixed with 11.5 mL of H_2O. The salt was placed in the flask, melted and boiled at a temperature of 145^{o}C ± 1^{o}C for the duration of the experiment. (The lower temperature is attributed to the addition of the water and the lower atmospheric pressure in Los Alamos (~ 776 mbar)). Container sections were completely submerged in the boiling $MgCl_2$ solution. The solution was initially clear and became discolored as the samples corroded in the boiling $MgCl_2$ solution. The Hagan section (Hagan-1) was removed from the boiling $MgCl_2$ after 16 hours. The SAVY 4000 section was removed from the boiling $MgCl_2$ after 43 hours.

The experiment was repeated for a Hagan section (Hagan-2) with a shorter exposure time. Photographs of the unexposed section are shown in Figure 5.



a) b)

Figure 5 Photographs of the a) outer contour and b) edge of the Hagan-2 prior to exposure to boiling $MgCl_2$.

This section, Hagan-2, was removed after one hour of exposure to boiling $MgCl_2$.

Boiling $MgCl_2$ Testing Results

Photographs of the Hagan-1 section after the 16 hour exposure are shown in Figures 6 and 7.

Figure 6 Inner contour of the Hagan-1 section after 16 hours of exposure to boiling $MgCl_2$.

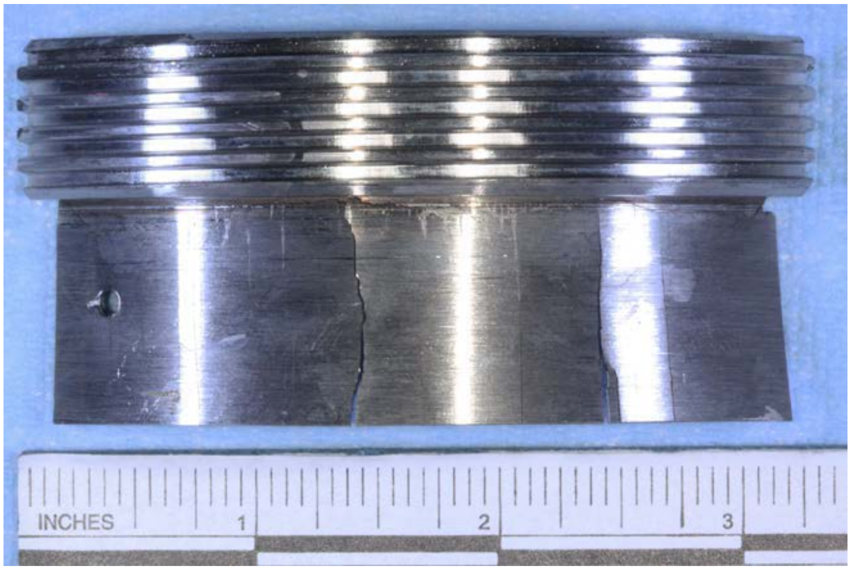


Figure 7 Outer contour of the Hagan-1 section after 16 hours of exposure to boiling $MgCl_2$

Extensive through wall cracking parallel and perpendicular to the weld was observed in the Hagan-1 section. Vertical through wall cracks propagated through the entire length of the wall piece.

Photographs of the Hagan-2 section after one hour of exposure to boiling $MgCl_2$ are shown in Figures.

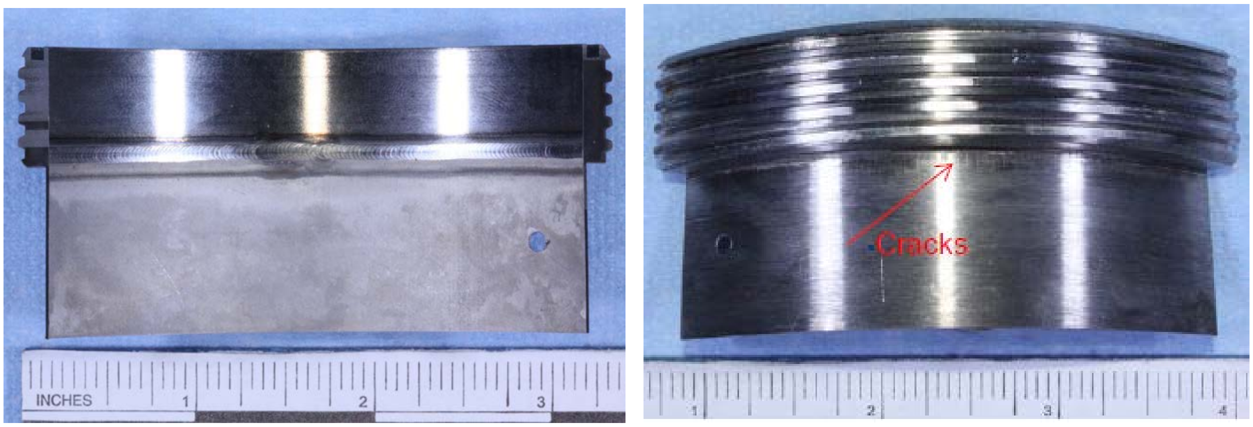


Figure 8 Photographs of the a) inner contour and b) outer contour of Hagan-2 after one hour of exposure to boiling $MgCl_2$.

Cracking was only observed on the exterior of the Hagan-2. The full submersion experiment is not representative of typical storage conditions. During storage, the exterior of the container has limited, if any, exposure to the corrosive chloride environment.

Photographs of the section of the SAVY 4000 after 43 hours of submersion in boiling $MgCl_2$ are shown in Figures 9 and 10.

a) b)

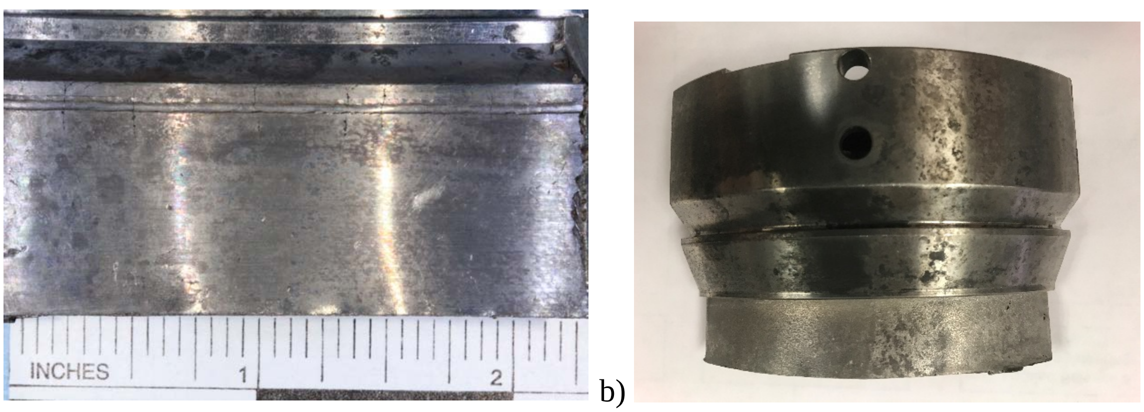


Figure 9 Photograph of the a) inner and b) outer contour of the SAVY 4000 section after submersion in boiling $MgCl_2$ for 43 hours.

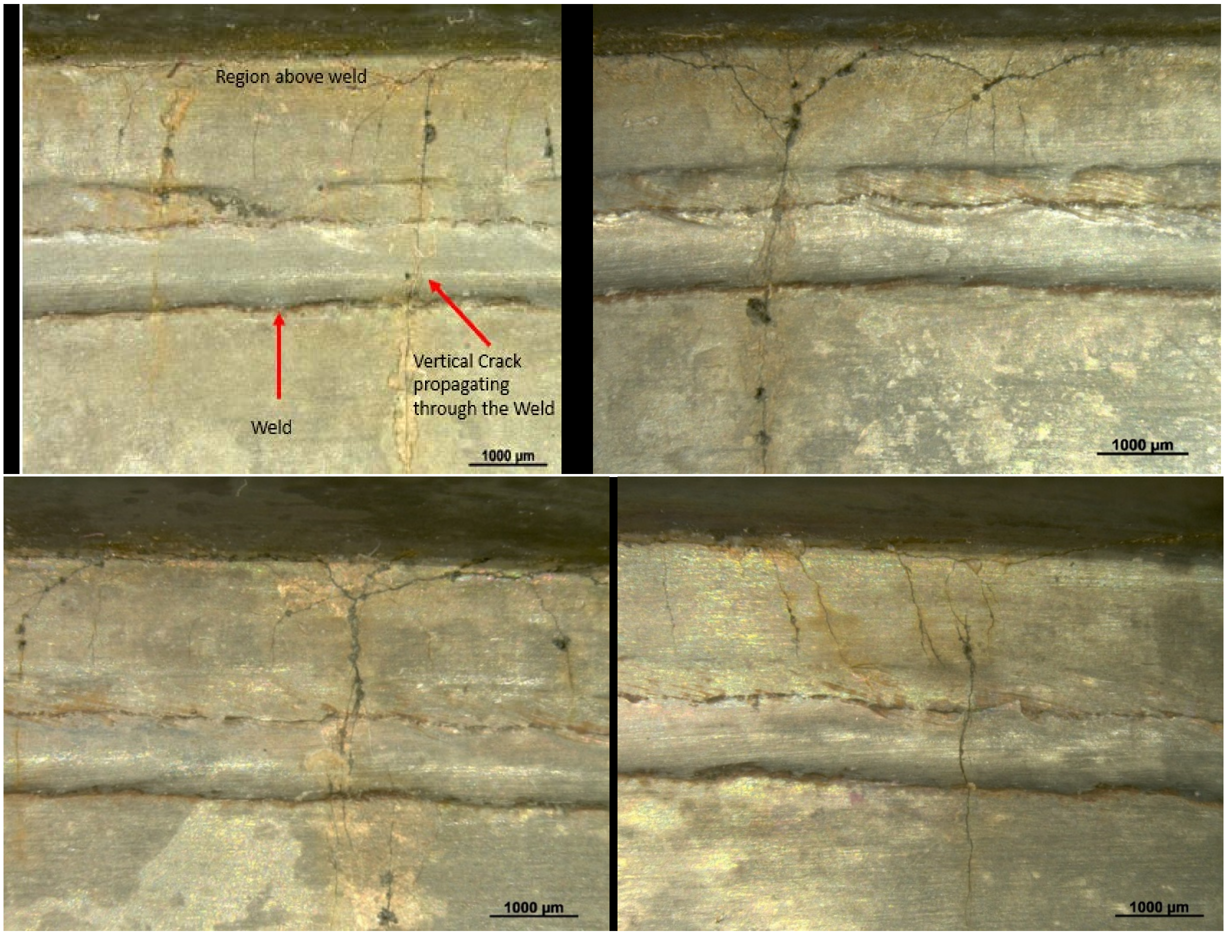


Figure 10 Close up photographs of the inner contour weld region of the SAVY 4000 section after submersion in boiling $MgCl_2$ for 43 hours.

The greatest concentration of cracks occurred in the region directly above the weld. Photographs of the section suggest vertical cracks propagate from above the weld, through the weld, to below the weld. The longest vertical "through-weld" crack observed was 6.5 mm with 4 mm below the weld. Cracks parallel to the weld were also observed.

Boiling $MgCl_2$ Testing Experimental Procedure on Containers

Boiling $MgCl_2$ tests were also conducted on the full container bodies per ASTM Standard G36. Photographs of the one quart Hagan body, S/N 08/07-01147, prior to exposure to the boiling $MgCl_2$ are shown in Figure 11.

a) b)

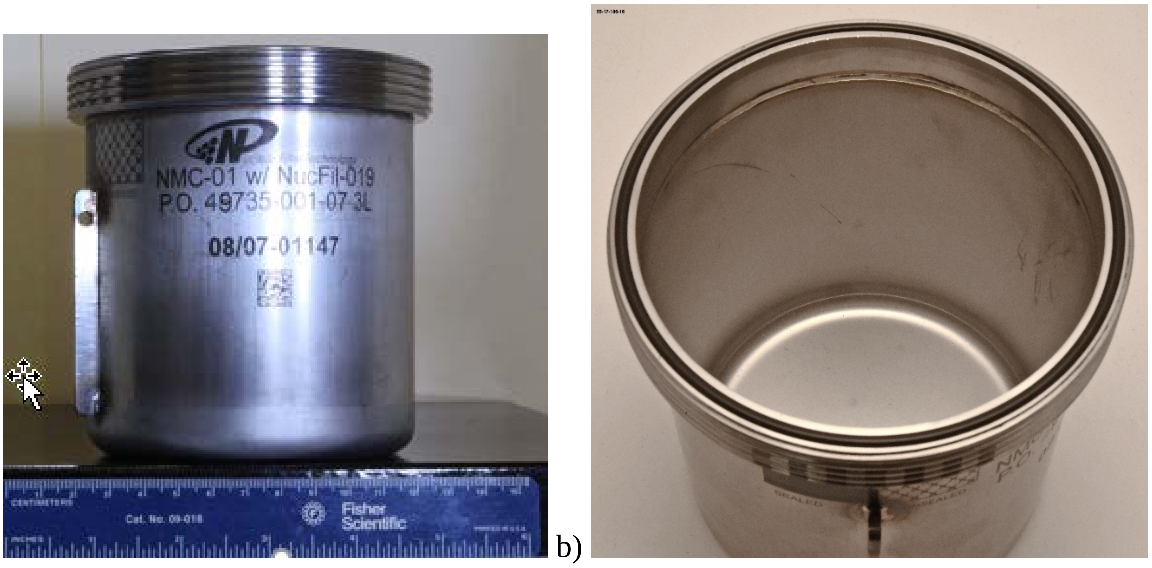


Figure 11 a) Exterior and b) interior of the one quart Hagan container prior to exposure to boiling $MgCl_2$.

The Hagan container body was gradually filled with 1466 g of reagent grade $MgCl_2.6H_20$ while being heated on a hotplate. No water or boiling chips were added to the container. Solution level was above the weld near the top of the container. A specially designed Teflon lid with condenser and thermometer affixed was screwed onto the body of the Hagan container when the salt was near the melting point. The experimental set-up is shown in Figure 12.

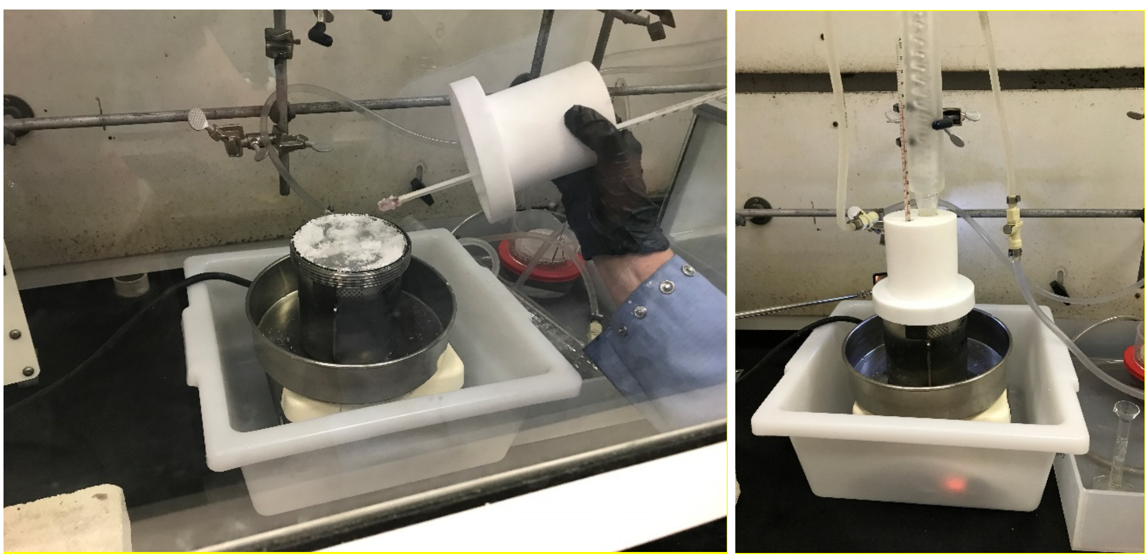


Figure 12 Photographs of the boiling $MgCl_2$ experimental set-up for the Hagan container.

With the Teflon lid in place it was difficult to see the solution. Boiling starting time was defined as the time solution reached 155ºC and condensate was visible in the condenser. The solution maintained a temperature of 155ºC ±1º C throughout the experiment. After 18 hours of boiling, a few small vertical cracks were observed near the weld on the outer contour. After 20.5 hours, many small vertical cracks near the weld were observed. After 22.5 hours many vertical cracks and a crack parallel to the weld were visible. Cracks continued to grow but no solution was observed leaking from the container. The experiment was stopped after 24 hours. Detailed information on the experimental observations as a function of time is in Appendix 1. After the container cooled and the $MgCl_2$ was removed, water was added to the container. Water immediately leaked from the cracks confirming through wall cracking. The major source of the leaks was the large crack parallel to the weld. The small cracks, perpendicular to the weld, were through wall but very small. There was minimal leaking from these cracks, if any. No cracking was observed near the attachment weld on the side of the container and no leaks were observed in the region.

Boiling $MgCl_2$ experiments were also conducted on the body of the one quart SAVY 4000 container 011701149B. Photographs of the SAVY 4000 prior to exposure are shown in Figure 13.

a) 

b) 

Figure 13 Photographs of the a) exterior and b) interior of the SAVY 4000 container prior to exposure to boiling $MgCl_2$

The SAVY 4000 container body was gradually filled with 1349 g of reagent grade $MgCl_2 .6H_20$ while being heated on a hotplate. The solution level was above the weld near the top of the container. No water or boiling chips added to the container. A specially designed Teflon lid with condenser and thermometer affixed was place onto the body of the SAVY 4000 container. Boiling starting time was defined as the time solution reached 154ºC and began to condense. After 44 hours no cracks were visible on the outer contour of the SAVY 4000 and no solution was observed in the catch pan. After 46 hours a great deal of $MgCl_2$ was observed in the catch pan at the bottom of the SAVY 4000 container (Figure 14).

Figure 14 SAVY 4000 container after failure with $MgCl_2$ in catch pan.

The solution maintained a temperature of 154ºC ±1 ºC for 46 hours until significant salt had leaked from the container. After 47 hours the temperature began to drop and the experiment was stopped.

Boiling $MgCl_2$ Testing Results

Photographs of the Hagan container after 24 hours of exposure are shown in Fig 15.

a) b)

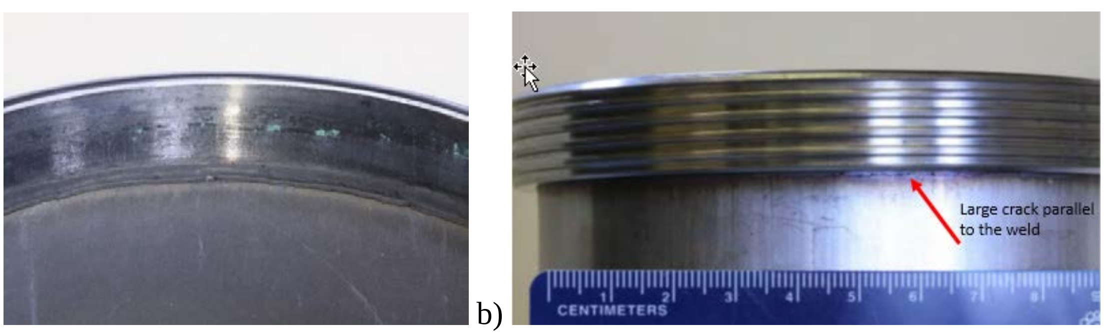


Figure 15 a) Inner contour and b) outer contour of the Hagan container after exposure to boiling $MgCl_2$ for 24 hours.

Vertical cracks approximately 5mm in length and cracks parallel to the weld were visible on the interior and exterior of the Hagan container. Water immediately leaked from the large cracks parallel to the weld when the exposed Hagan was filled with water. Little, if any water was observed leaking from the vertical cracks.

Photographs of the bottom of the SAVY 4000 container after 47 hours of exposure to boiling $MgCl_2$ are shown in Figure 16.

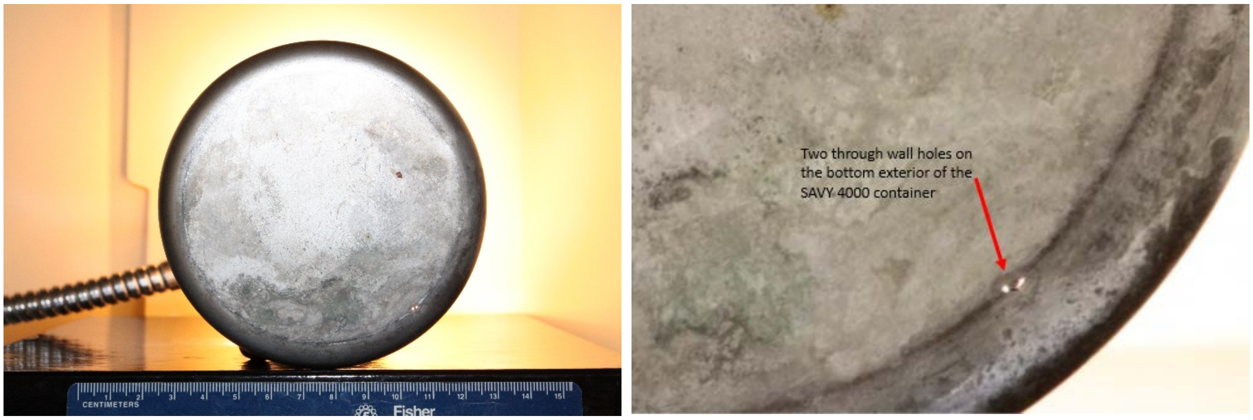


Figure 16 Photographs of the bottom of the SAVY 4000 container after 47 hours of exposure to boiling $MgCl_2$.

Two holes were clearly visible in the bottom of the container.

Photographs of the exterior of the SAVY 4000 container after 47 hours of exposure are shown in Figure 17.

Figure 17 Exterior of SAVY 4000 after 47 hours of exposure to boiling $MgCl_2$.

No cracks were visible on the exterior of the SAVY 4000 container, however, the weld region is covered by the collar.

Photographs of the inner contour weld region of the SAVY 4000 container after 47 hours of exposure are shown in Figure 18.

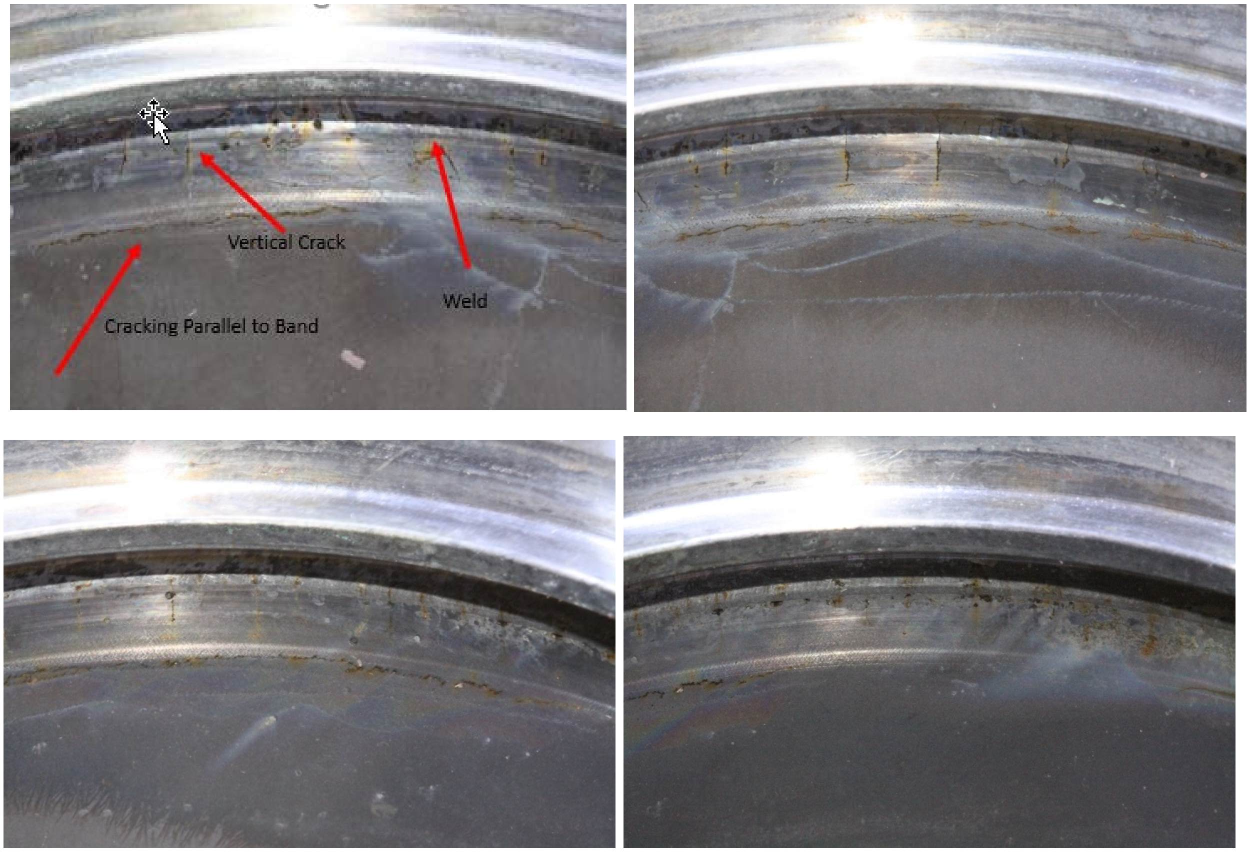


Figure 18 Photographs of the inner contour weld region of the SAVY 4000 container after 47 hours of exposure to boiling $MgCl_2$.

Cracks perpendicular to the weld were observed on the inner contour up to 2 mm above the weld and cracks 1 cm in length were present below the weld. A band was visible approximately 6 mm below the weld, roughly corresponding to the bottom of the collar. Cracks propagated through the weld and the band. Cracks parallel to the band were also observed. General corrosion was also observed near the weld. (Figure 19).

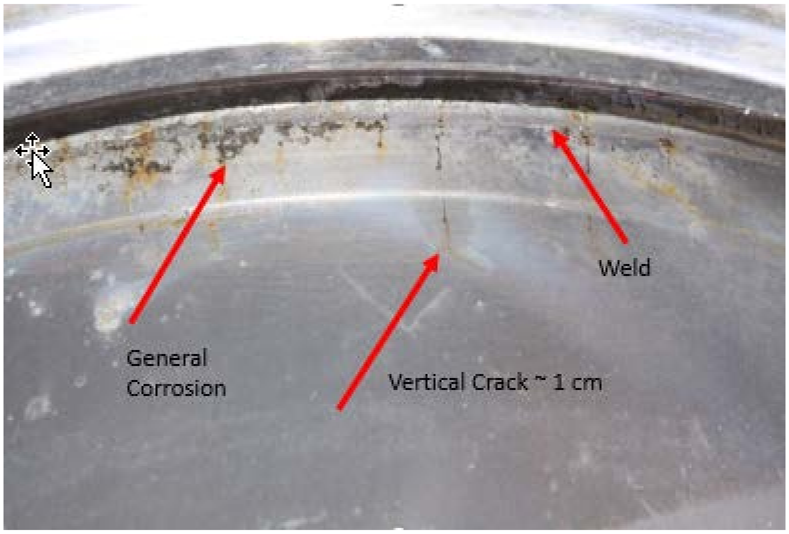


Figure 19 SAVY 4000 inner wall weld region after exposure to boiling $MgCl_2$ showing vertical cracks and general corrosion.

It was not possible to visually determine if cracks were present on the outer wall near the weld without removing the collar. However, the holes on the bottom of the container were plugged and the container was filled water and observed for approximately 15 minutes. No solution was observed leaking from the container however small through wall cracks could still be present in the weld region of the container. A helium leak check of the container body is planned to confirm the weld region will still pass the helium leak test criterion.

Laser etching was not visible on the inside of the contour, unlike SAVY 4000 containers exposed to HCl vapor for 14 months.[3]

Results of the boiling $MgCl_2$ experiments on the one quart SAVY 4000 and Hagan container bodies are summarized in Table 1.

Container	SAVY 4000 Container	Hagan Container
Time to Failure	44-46 hrs (holes in bottom)	22-24 hrs (cracks clearly visible on outside of container)
Hours in Test	47 hrs	24 hrs
Crack Location	Many perpendicular to weld extending through the weld Parallel cracks to band at end of collar	Below Weld: Parallel to weld Perpendicular to weld
Maximum Vertical Crack Length	~12 mm (9 mm below the weld)	5 mm
Water leak	No flow of water from cracks after 15 minutes	Immediate large flow of water from cracks parallel to the weld

Boiling $MgCl_2$ Testing Discussion

Data from boiling $MgCl_2$ testing provided an accelerated method of ranking the relative degree of stress-corrosion cracking susceptibility in aqueous chloride container environments for Hagan and SAVY 4000 containers. Time-to-failure (through wall holes in bottom) for the SAVY 4000 body was approximately twice as long (44-46 hours) as the Hagan body (22-24 hours), indicating that the SAVY 4000 is less susceptible to a container breech due to chloride induced corrosion. Small through wall cracks may have formed in the SAVY 4000 container near the weld in a shorter time period. Helium leaking is necessary to confirm.

The boiling $MgCl_2$ testing on the Hagan body indicated that the most susceptible region to chloride induced SCC is the weld near the top of the container. Both vertical cracking caused by tensile hoop stresses as well as cracks parallel to the weld caused by tensile axial stresses are a potential concern. Surveillance monitoring should ensure a careful examination of the Hagan top weld region.

The boiling $MgCl_2$ testing on the SAVY 4000 body suggest that through wall pitting near the container bottom was the primary failure mechanism. The bottom is the thinnest region (0.6 mm) of the SAVY 4000 container. Pits, with depths up to 40 microns, have been observed in an In-Service Hagan container.[4] To date, no pitting has been observed in an In-Service SAVY 4000 container.

Both vertical cracking caused by tensile hoop stresses as well as cracks parallel to the weld caused by axial tensile stresses were also present on the inside of the exposed SAVY 4000 container. The largest number of cracks was located directly above the weld in the collar, suggesting this was the region of highest tensile stress. Because of the increased thickness of the container in this region, through wall SSC is less likely. (Figure 20)

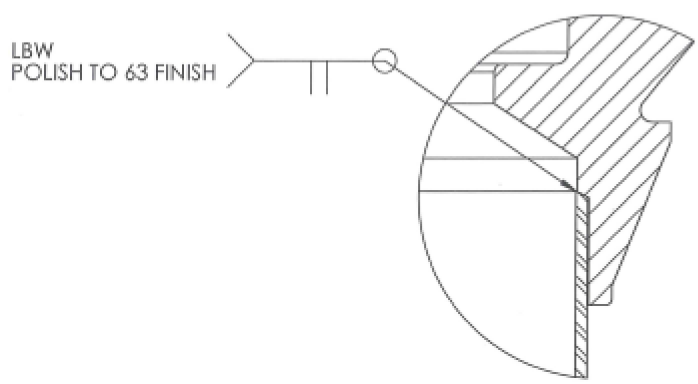


Figure 20 Drawing of the weld and collar region of the SAVY 4000 container. Arrow indicated the weld location.

However, many cracks that continued through the weld to the thinner walled region below the weld were also observed. Based on these results surveillance monitoring should focus on the bottom of the container and the weld region.

Residual Stress Measurements Experimental Procedure

Introduction

Residual stress measurements were performed on the Hagan and SAVY 4000 containers to determine the magnitude of axial and hoop stresses in the containers. Measurements were obtained near the top welds of the containers, based on results of the boiling MgCl2 testing. Hole drilling was used to determine the residual stress in the container bodies. Hole drilling is standardized by ASTM under E837. The method can be applied to quantify the average residual stress over the depth of the hole or incremental hole drilling can be performed to determine the distribution of residual stress versus depth from the surface.

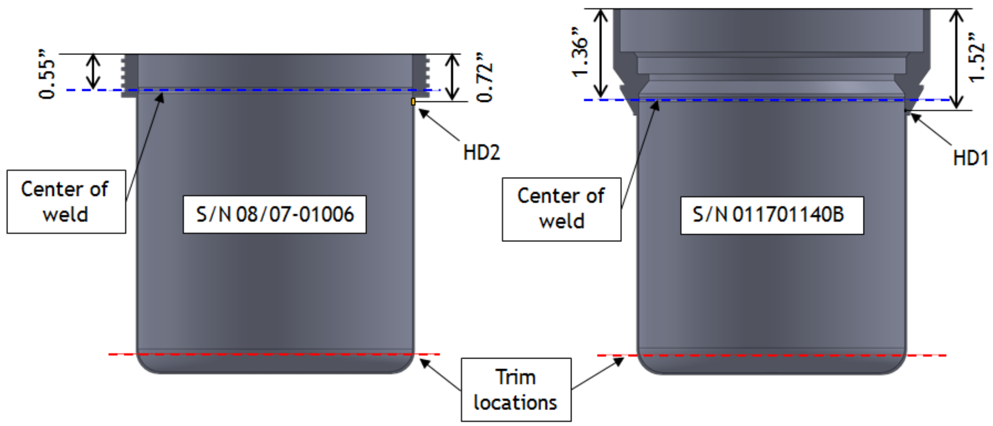
Experimental Procedure

Hole drilling measurements were obtained on a 1 quart SAVY 4000 container and a 1 quart Hagan container shown in Figure 21.[5] Residual stress was determined from the measured strain versus depth data through an elastic inverse solution based on the principles of elasticity.

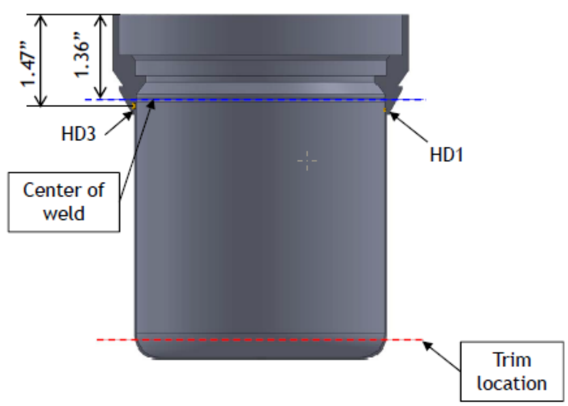
Figure 21 Photograph of the SVY and Hagan containers used for hole drilling measurements

Container Sectioning

Prior to performing the hole drilling measurements, the bottoms of the canisters were cut off to allow for equipment access. The cutting planes (trim location) are shown in Figure 22 below.



a)



b)

Figure 22 Sectioning and hole drilling locations on a) the Hagan (S/N 08/07-01006)-HD2 and SAVY 4000 (S/N 0117011408) – HD1 and b) SAVY 4000-HD3.

During cutting, strain gages were placed near the measurement locations. The released strain during sectioning is summarized in Table 5, which is relatively small. The approximate released stress due to sectioning, assuming a Young’s Modulus of 196 GPa, is also reported in Table 1.

Table 1 Measured strain and calculated stress release during sectioning

Direction	Hagan		SAVY 4000	
	Microstrain ([με])	Stress (MPa)	Microstrain [με]	Stress (MPa)
Hoop	-18	-4	-68	-13
Axial	-25	-5	-67	-13

The sign convention is that tensile stress is positive and compressive stress is negative. The negative values for the observed changes indicate sectioning decreased the magnitude of the tensile stresses and increased in the magnitude of compressive stresses. The released strain is reported for information only and was not included in the computed residual stress results.

Hole drilling measurements

The hole drilling measurements were located as close to the weld as possible and ensure compliance with ASTM E837. The holes were located just below the weld based on the results of the boiling $MgCl_2$ experiments. Though the boiling $MgCl_2$ SAVY 4000 experiments suggested residual stresses were greater above the weld in the collar, it was not possible to obtain hole drilling data above the weld due to the limited space. For the Hagan container, the hole HD2 was centered 4.3 mm below the weld center (4.1 mm below the weld edge). This is near the bottom of the longest vertical cracks observed in the 16 hour boiling $MgCl_2$ test on the full body of the Hagan. For the SAVY 4000 container, the hole HD1 was centered 4.1 mm below the weld center (3.9 mm below the weld edge) and HD3 was centered 2.1 mm below the weld center (1.9 mm below the weld edge). This was approximately 5 mm above the bottom of the longest crack observed below the weld in the 47 hours boiling $MgCl_2$ test on the full body of the container. The strain gauges were located to allow calculation of residual stress in the axial and hoop directions. Hole locations are shown in Figure 22 b) above and Figure 23 below.

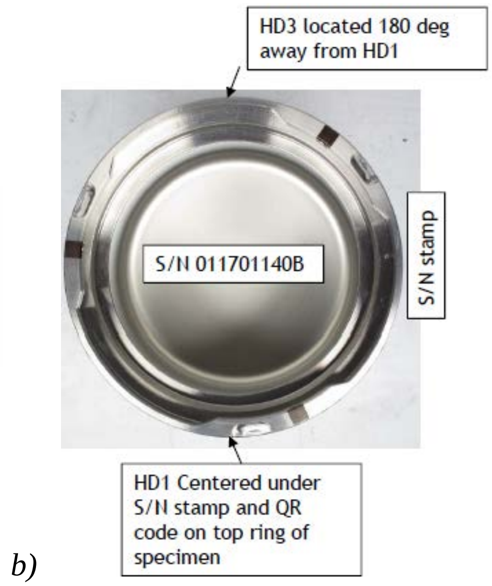


Figure 23 Illustration of hole drilling measurement locations: a) Hagan (HD2) and b) SAVY 4000 (HD1 and HD3).

For the Hagan container, a 2 mm diameter hole (HD2) was drilled through the entire wall and the average residual stress was computed based on the assumption that it was uniform through the thickness. The Hagan wall was too thin (0.76 mm thick, with no collar at the measurement location) to appropriately apply incremental hole drilling. For the SAVY 4000, a 1 mm diameter hole (HD1) was drilled in increments of 0.0254 mm to a final depth of 0.5 mm. At each incremental hole depth, the strain change was monitored using a commercial Wheatstone Bridge instrument and recorded. The second SAVY 4000 measurement (HD3) was a 2 mm diameter hole drilled in increments of 0.0254 mm to a final depth of 1.0 mm. Results of HD3 are not yet complete.

Results

The computed residual stress for the measurement on the Hagan container, based on the uniform stress assumption, is shown in Table 2.

Table 2 Average Stresses in the Hagan container wall

Axial Direction Stress (MPa)	Hoop Direction Stress (MPa)	Shear Stress (MPa)
-120	194	-45

Stress gradients present in the unannealed Hagan wall would result in regions of stress higher than the averages reported here. The compressive axial stress in the Hagan was not expected. The Hagan failed in the boiling $MgCl_2$ experiment when a large crack parallel to the weld formed. The crack was closer to the weld than the location of the measured average compressive stress. Axial stresses could be significantly higher near the weld and/or axial stresses could be tensile on the inner wall even though the average stress through the wall was compressive. Shear stress may have contributed.

A line plot of the measured residual stress versus depth for the SAVY 4000 container, HD1, is shown in Figure 24.

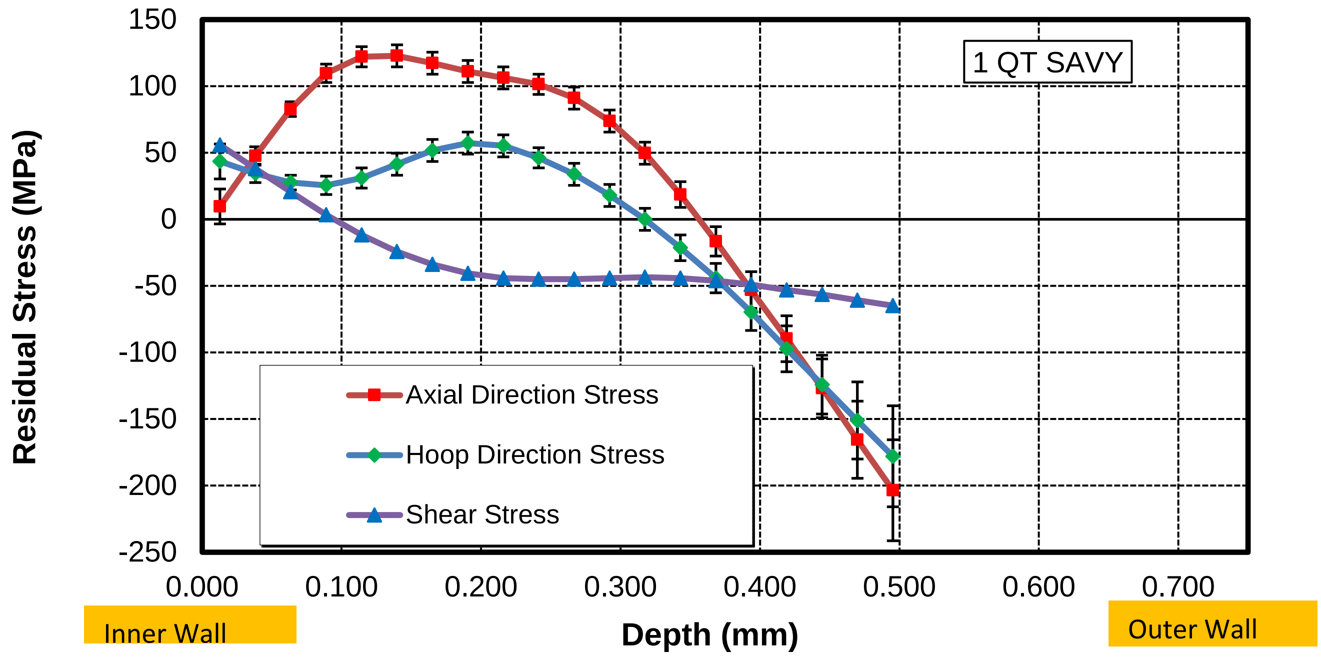


Figure 24 Line Plot of the residual stress versus distance from the inner wall for HD1.

At HD1, axial stresses were the largest magnitude stresses present in the SAVY 4000 container. Axial stresses of 10 MPa was present at the inner wall and increased to a maximum value of 123 MPa at 0.14 mm from the inner wall before steadily decreasing with increasing distance into the container. The hoop stress of 43 MPa present at the inner wall gradually increased to a maximum value of 57 MPa at 0.2 mm from the inner wall before gradually decreasing with increasing distance into the container. Shear stress at the inner wall was 56 MPa and gradually decreased with increasing distance into the container. For all incremental hole drilling results, uncertainty increased with increasing hole depth.

Discussion

To allow for relative comparison of stresses in the Hagan and SAVY 4000 container, the average stresses through the wall was calculated for the SAVY 4000. For HD1, data was only available to 0.5 mm through the container wall. Given the downward trend of the HD1, calculated HD1 stresses are probably higher than the average stress through the entire wall. Average stresses are reported in Table 3.

Table 3 Calculated Average Stresses in the SAVY 4000 container

Container	Hole Number	Distance below the weld edge (mm)	Data Summary	Average Axial Stress (MPa)	Average Hoop Stress (MPa)
HAGAN	HD2	4.1	Average of data through the entire wall thickness	-120*	194
SAVY 4000	HD1	3.9	Average of data from the inner wall to 0.5 mm (66% of wall)	25 (123 MPa max)	-11* (57 MPa max)

*The negative value denotes a compressive stress.

The average hoop stress of 194 MPa in the Hagan container measured at 4 mm below the weld was the highest measured tensile residual stress in either the Hagan or the SAVY 4000. The Hagan average hoop stresses were 137 MPa greater than the SAVY 4000 MAXIMUM stress. The average SAVY 4000 hoop stress was compressive. The average axial stress in the Hagan was compressive while the average axial stresses in the SAVY 4000 was small be tensile..

Results suggest that because the Hagan container had the highest average tensile stress, 194 MPa hoop stress, it is more susceptible to SCC than the SAVY 4000 container. Reported hole drilling results represent the stresses at a single point 4 mm below the weld. Stresses are expected to change as a function of distance from the weld. Additional measurements at varying distances from the weld are necessary to better understand the residual stresses in both containers.

Application to Life Extension Program

Results from boiling $MgCl_2$ testing and residual stress measurements suggest that the Hagan container is more susceptible to SCC than the SAVY 4000 container under comparable storage conditions. Moreover, 316L SS is known to have a higher pitting resistance (pits are a precursor to SCC and can also lead to extensive failure of the material), than 304L SS.[6] Chloride-induced corrosion data is available for Hagan containers that have been in-service for over a decade longer than the SAVY 4000 container. These results provide evidence to support using the

Hagan corrosion data as a bounding case for potential SAVY 4000 corrosion, assuming similar storage conditions.

Comparison with other 3013 Storage Containers

3013 Inner and Outer containers have been used to store plutonium-bearing materials produced at several Department of Energy (DOE) laboratories. Boiling $MgCl_2$ tests were conducted on a 3013 outer container, which are the same design for all sites in the DOE complex that package 3013 containers, and Savannah River Site (SRS)/Hanford inner container.[7], [8] Table 4 compares previous results with the results obtaining in this study. Table 4 also compares materials of construction, wall thickness and weld type.

Table 4 Comparison of Time to Failure, Materials of Construction, Wall Thickness and Weld type in containers

Container Type	3013 Inner	3013 Outer	Hagan	SAVY 4000
				
Time to Failure (crack or hole) (hrs)	2-3.5	$<$ 48	22-24	44-46*
Material of Construction	304L SS	316L SS	304L SS	316L SS
Thickness (mm)	1.4	3	0.76	0.75 Wall 0.6 Bottom
Weld Type	GTAW	GTAW or LBW	GTAW	LBW
Annealing	No Annealing		No Annealing	Body Annealed Collar Annealed prior to machining

*Pending confirmation with a helium leak check

The SAVY 4000 time to failure (holes in the container bottom) was similar to the maximum possible time to failure reported for the 3013 outer container. (The 3013 container total time of exposure was 48 hours. The container had through wall cracks when it was checked after 48 hours of exposure. Failure could have occurred at a significantly shorter time.) Both containers are made of 316L SS. The Hagan container had a GTAW and the SAVY 4000 had a LBW. The SAVY 4000 failed due to holes in the container bottom while the 3013 outer failed due to SCC. This was probably due to the fact that the bottom of the SAVY 4000 is less than half as thick as the 3013 outer container walls. The unannealed 304L SS 3013 inner container time to failure was an order of magnitude less than the other containers. This container has large stresses present due to formation process as well as stresses from the weld.[9]

Residual stresses have also been measured for several 3013 containers.[9] Table 5 compares the hoop and axial stresses measured in these containers with those obtained for the Hagan and SAVY 4000 containers. Caution must be taken in comparing results directly since measurement techniques varied between containers.

Table 5 Comparison of Residual Stresses in Containers

Container Section/Sample	Hoop Maximum Residual Stress (MPa)	Axial Maximum Residual Stress (MPa)	Location	Technique
SAVY 4000*	53	123	3.9 mm below the closure weld from the inner wall to 50% of the distance through the wall	Incremental Hole Drilling
Hagan	194 (average through wall)	-120 (average through wall) (compressive)	4.1 mm below the closure weld	Hole Drilling
SRS/Hanford 3013 Outer	360	200	~ 5mm below the top of the lid ~2 mm from the outer wall	Neutron Scattering
	260		6.7 mm below top of lid 3.2 mm from outer wall	Contour (~average)
SRS/Hanford 3013 Inner	480		2.4 mm below the weld ~ 5mm from the top of the lid	Contour (~ average)
SRS/Hanford 3013 Inner	800-1100	200-400	2.4 mm below the weld 0.4-0.8 mm from outer wall	Incremental Hole Drilling
SRS/Hanford 3013 Inner	710 (no release correction)	160 (no release correction)	2.4 mm below the weld Inner Wall	Incremental Hole Drilling
RFETS/LLNL 3013 Inner	180		Closure Weld	Contour

** SAVY 4000 and Hagan hole drilling measurements were obtain at ~ 4 mm below the weld while hole drilling measurements in the SRS/Hanford container were ~ 2.5 mm below the weld. Higher residual hoop stresses are expected nearer to the weld. For the SAVY 4000 container, higher stresses are expected above the weld in the collar, based on boiling MgCl2 results, but it was not possible to obtain reliable hole drilling data in this region.*

The smallest "maximum" residual hoop stresses were measured in the SAVY 4000 container. Maximum measured hoop stress reported for the SAVY 4000 were approximately six times smaller than those reported in the 3013 outer container. Maximum measured hoop stresses reported for the SAVY 4000 were approximately 10 times smaller than those reported in the SRS/Hanford inner container. Maximum axial stresses in the SAVY 4000 are approximately 60% less than those in the 3013 outer container. Maximum axial stresses are approximately 25% or more less than those in the SRS/Hanford inner container. Figure 26 and 27 compare the hoop and axial stress versus distance from the inner wall as a % of the total wall thickness for the SAVY 4000, Hagan and 3013 inner container, obtained from incremental hole drilling.

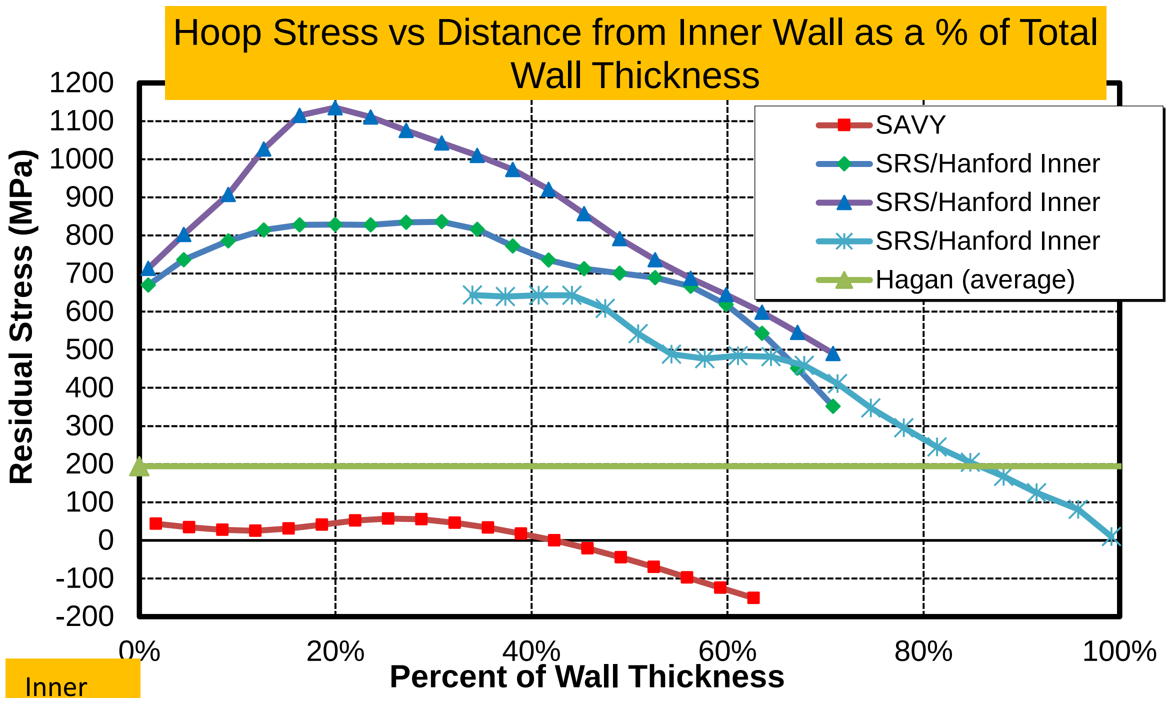


Figure 25 Comparison hole drilling hoop stresses near the weld in SAVY 4000, Hagan and SRS/Hanford Inner containers

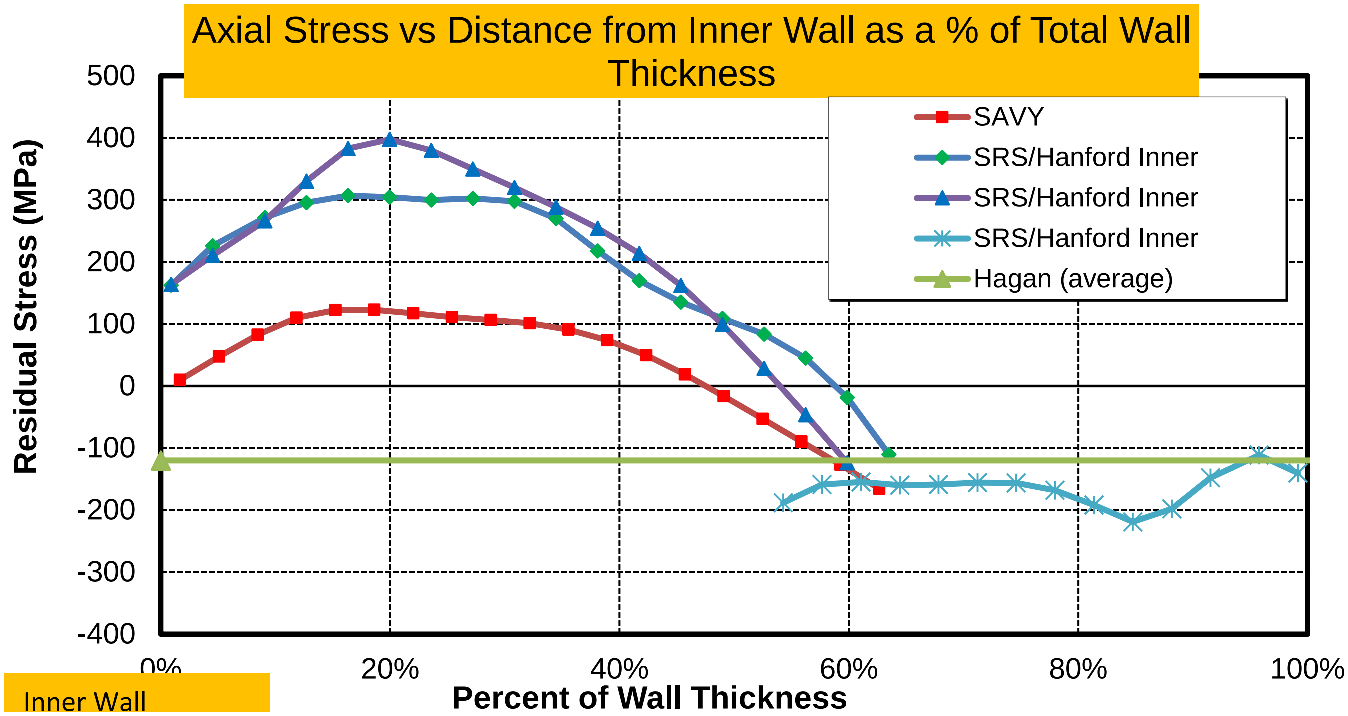


Figure 26 Comparison of hole drilling axial stresses near the weld in SAVY 4000, Hagan and SRS/Hanford Inner containers

Surplus plutonium-bearing materials have been stored in nested 3013 containers at the Savannah River Site (SRS) for five to ten years. Figure 28 illustrates the container configuration, which includes a convenience can which is in direct contact with the material.

Figure 27. 3013 (BNFL designed) outer, inner, and convenience containers.

The packaging, stabilization and surveillance of the materials, including destructive examination (DE) has been conducted in accordance with a DOE standard DOE-STD-3013 [10] developed to assure safe, prolonged storage of plutonium-bearing materials. Despite the high stresses in the inner container, Duffey concluded that it is unlikely that a corrosion-induced breach of the inner container occurred prior to DE for any of the 82 examined. [11]

Recommendations for Surveillance

Based on results of the boiling $MgCl_2$ testing, detailed corrosion inspection on Hagan and SAVY 4000 containers should include looking signs of SSC near the welds on the top of the containers. Examination of the thinner bottom of the SAVY 4000 container for pitting is also recommended.

Conclusion

Chloride-induced stress corrosion cracking (SCC) has been investigated as a potential failure mechanism for the SAVY 4000 and the Hagan containers. Regions of the container bodies most susceptible to SCC were identified and the magnitude of the residual stresses in those regions quantified. Results are applicable to both the SAVY 4000 surveillance program and the life extension program. Boiling $MgCl_2$ testing indicated that for both container bodies the region near the top weld was most susceptible to SSC. The Hagan showed through wall cracking after 22-24 hours of exposure both parallel (axial stresses) and perpendicular (hoop stresses) to the weld. The SAVY 4000 container showed significant cracking near the after 47 hours of exposure but there was no visual evidence of a through wall crack and no water leaked from the container. The confirmed failure mechanism in the SAVY was two through wall holes in the bottom of the container after 44-46 hours of exposure. For both containers, average "through wall" residual stresses were determined from hole drilling data 4 mm below the weld. In the Hagan body, average tensile hoop stresses were 194 MPa and average compressive axial stresses were -120 MPa. In the SAVY 4000 body, average compressive hoop stresses were -11 MPa and average tensile axial stresses were 25 MPa. Results suggest that the Hagan container is more susceptible to SCC than the SAVY 4000 container under comparable storage conditions.

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References

1. Reeves, K. et al. *LANL Storage Container Surveillance Report*; LA-UR-16-27427; Los Alamos, NM, 2016.
2. ASTM, ASTM G36 - Standard Practice forEvaluating Stress-Corrosion-Cracking Resistance of Metals and Alloys in a Boiling Magnesium Chloride Solution. ASTM International: West Conshohocken, PA, 19428, 2013; Vol. G 26.
3. Hyer, H. et. al. *Corrosion Potential of Laser Etching on SAVY 4000 and Hagan Containers*; LA-UR-17-28647; Los Alamos National Laboratory: 2017.
4. Narlesky, J. and Romero, Edward *Microscopic Examination of the Corroded Hagan Container Used to Store Molten Salt Extraction Residue XBPS333*; LA-UR-17-28355; Los Alamos National Laboratory: 2017.
5. Schajer, G. S., *Practical Residual Stress Measurement Methods*. John Wiley & Sons: Chicester, UK, 2013.
6. Jones, D., *Principles and Prevention of Corrosion, 2nd ed.* Prentice Hall: Upper Saddle River, NJ, 07458, 1996.
7. Dunn, K. A., Residual Stress Testing of Outer 3013 Containers (U). *SRNL* 2003, *WSRC-TR-2002-00396*.
8. Mickalonis, J., and Dunn, K.A., Residual Stresses in 3013 Containers. *Journal of Nuclear Materials Management* 2010, *38*, 31-38.
9. Stroud, M. A.; Prime, M. B.; Veirs, D. K.; Berg, J. M.; Clausen, B.; Worl, L. A.; DeWald, A. T., Assessment of Residual Stresses in 3013 Inner and Outer Containers and Teardrop Samples. *LANL* 2015, *LA-UR-15-29376*.
10. U. S. Department of Energy, Stabilization, Packaging, and Storage of Plutonium-Bearing Materials. U.S. Department of Energy: Washingon, D.C., 2012.
11. Duffey, J. M. *A Comparison of Gas Composition and Pressure of Inner and Outer 3013 DE Containers*; SRNL-STI-2014-00439, Revision 0; Savannah River National Laboratory: 2014.

Appendix 1

Experimental Data for the boiling $MgCl_2$ test on Hagan Container

Date	Time	Temperature ˚C	Observational Notes
6/27/2017	12:15	142	Chiller cooling
6/27/2017	12:19	144	Salt still getting to boil
6/27/2017	12:21	146	Salt still getting to boil
6/27/2017	12:27	151	Salt still getting to boil
6/27/2017	12:30	152	Salt still getting to boil
6/27/2017	12:33	153	Salt still getting to boil
6/27/2017	12:39	154	Salt still getting to boil
6/27/2017	12:49	154.5	Chiller cooled to 10^{o}C
6/27/2017	12:51	155	Condensing on back of canister
6/27/2017	1:00	155	Boiling $MgCl_2$ confirmed
6/27/2017	1:10	155	Chiller about 5^{o}C
6/27/2017	1:23	155	No Cracks
6/27/2017	1:40	155	No Cracks
6/27/2017	1:50	155	No Cracks
6/27/2017	2:00	155	No Cracks
6/27/2017	2:15	155	No Cracks
6/27/2017	2:25	155	No Cracks
6/27/2017	2:35	155	No Cracks
6/27/2017	2:50	155	No Cracks
6/27/2017	3:10	155	No Cracks
6/27/2017	3:25	156	No Cracks
6/27/2017	3:35	156	No Cracks
6/27/2017	3:45	156	No Cracks
6/27/2017	4:00	156	No Cracks
6/27/2017	4:10	156	No Cracks
6/27/2017	4:20	156	No Cracks
6/27/2017	4:30	156	No Cracks
6/27/2017	4:40	156	No Cracks
6/27/2017	5:00	156	Lines and top of bottom
6/27/2017	5:40	156	No cracks, but lines still there
6/28/2017	7:25	155	Few small cracks visible
6/28/2017	7:35	155	Few small cracks visible
6/28/2017	7:55	155	Few small cracks visible
6/28/2017	8:10	155	Second set of eyes confirms cracks
6/28/2017	8:30	155	Many micro cracks formed
6/28/2017	9:15	156	Many micro cracks formed/ing

Appendix 1 (continued)

Experimental Data for the boiling $MgCl_2$ test on Hagan Container

6/28/2017	9:35	155	Many micro cracks formed/ing
6/28/2017	10:00	155	Many micro cracks formed/ing
6/28/2017	10:15	155	Many micro cracks formed/ing
6/28/2017	10:55	156	Many micro cracks formed/ing
6/28/2017	11:10	156	Cracks growing in size
6/28/2017	11:20	156	Lots of vertical cracks, circumferential crack
6/28/2017	11:30	156	Cracks growing in size
6/28/2017	11:50	156	Larger vertical cracks
6/28/2017	12:10	156	Circumferential crack more obvious
6/28/2017	12:20	156	Cracks
6/28/2017	12:50	156	Test Stopped