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SAND2011-6579

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Stainless Steel Corrosion by Molten Nitrates: Analysis and Lessons Learned

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Stainless Steel Corrosion by Molten Nitrates

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Abstract

A secondary containment vessel, made of stainless 316, failed due to severe nitrate salt corrosion. Corrosion was in the form of pitting was observed during high temperature, chemical stability experiments. Optical microscopy, scanning electron microscopy and energy dispersive spectroscopy were all used to diagnose the cause of the failure. Failure was caused by potassium oxide that crept into the gap between the primary vessel (alumina) and the stainless steel vessel.

Contents

Abstract	3
Contents	4
List of Figures	4
Conclusions	5
Recommendations for future work	5
References	10
Distribution	11

Figures

Figure 1. Stainless steel containment vessel, corroded (left) and uncorroded (right).....	6
Figure 2. Images of corroded vessel, showing blister (top right), hole (bottom right), position of weld compared to hole (bottom left), and cross-section (top left)...	7
Figure 3. Cross-section of corroded vessel (left) and optical microscopy image of pitting corrosion in the vessel wall (right). A large blister can be seen on the left bottom corner of the cross-section.	7
Figure 4. SEM image of corroded pit and close up of oxide formation on pit surface (left).....	8
Figure 5. EDS spectrum analysis of corroded stainless steel vessel (baseline alloy analysis).....	8
Figure 6. EDS analysis of oxide layer on corrosion pit.	9
Figure 7. EDS analysis of blister formed on corroded stainless steel vessel.....	9

Conclusions

Molten nitrate solar salt (89% KNO_3 , 11% NaNO_3 by weight) was used during chemical stability experiments, with an oxygen cover gas, at a salt temperature of 350-700°C. Nitrate salt was primarily contained in an alumina vessel; however salt crept into the gap between the alumina and 316 stainless steel. Corrosion occurred over a period of approximately 2000 hours, with the end result of full wall penetration through the stainless steel vessel; see Figures 1 and 2 for images of the corrosion damage to the vessel.

Wall thickness was 0.0625", which, based on previous data, should have been adequate to avoid corrosion-induced failure while in direct contact with salt temperature at 677°C (0.081"/year). [Ref. 1] Salt temperatures exceeding 650°C lasted for approximately 14 days. However, previous corrosion data was performed with air as the cover gas. High temperature combined with an oxygen cover gas obviously drove corrosion rates to a much higher value.

Corrosion resulted in the form of uniform pitting (Figures 3 and 4). Based on SEM and EDS data, pits contained primarily potassium oxide and potassium chromate, reinforcing the link between oxides and severe corrosion (Figure 6). In addition to the pitting corrosion, a large blister formed on the side wall, which was mainly composed of potassium, chromium and oxygen (Figure 7).

All data indicated that corrosion initiated internally and moved outward. There was no evidence of intergranular corrosion nor were there any indication of fast pathways along grain boundaries. Much of the pitting occurred near welds; however this was the hottest region in the chamber. Pitting was observed up to two inches above the weld, indicating independence from weld effects.

Recommendations for future work

The primary containment vessel should be alumina that extends to the top of the secondary containment. Cooling at the top of the vessel will help mitigate creeping behavior of the salt into the gap between the stainless steel. Additionally, a nickel stainless steel, if not prohibitively expensive, should be used as the secondary vessel. Typically nickel steel has a corrosion rate that is about half of 316 stainless steel.

As higher temperatures are employed preliminary corrosion testing should be completed. As a minimum, corrosion testing of potential containment materials should be performed in a short-term test (~2-3 weeks). This will give at least one data point for design considerations.

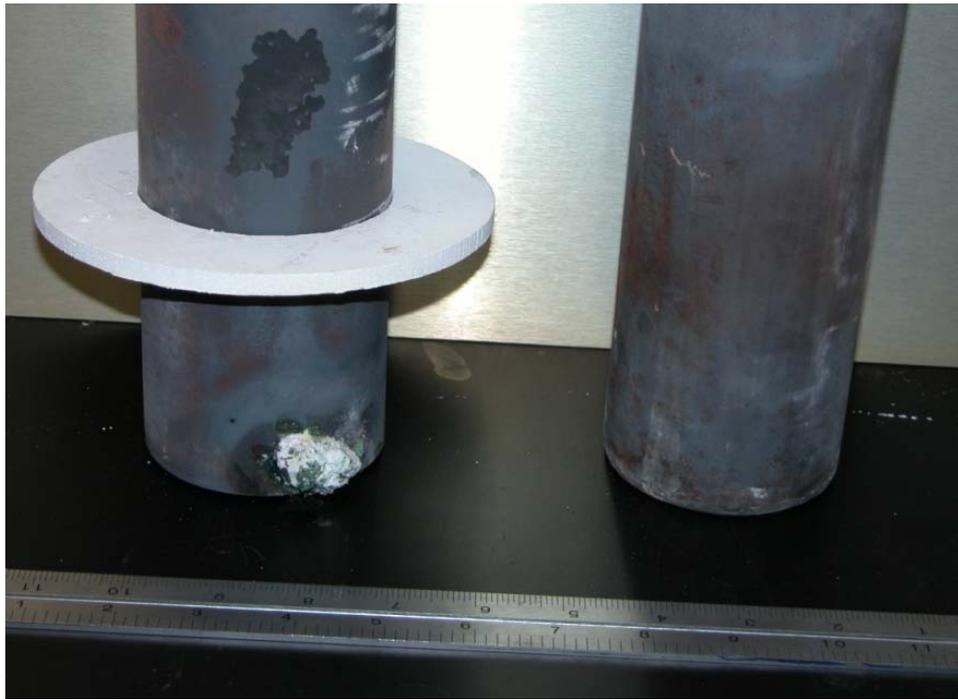


Figure 1. Stainless steel containment vessel, corroded (left) and uncorroded (right).

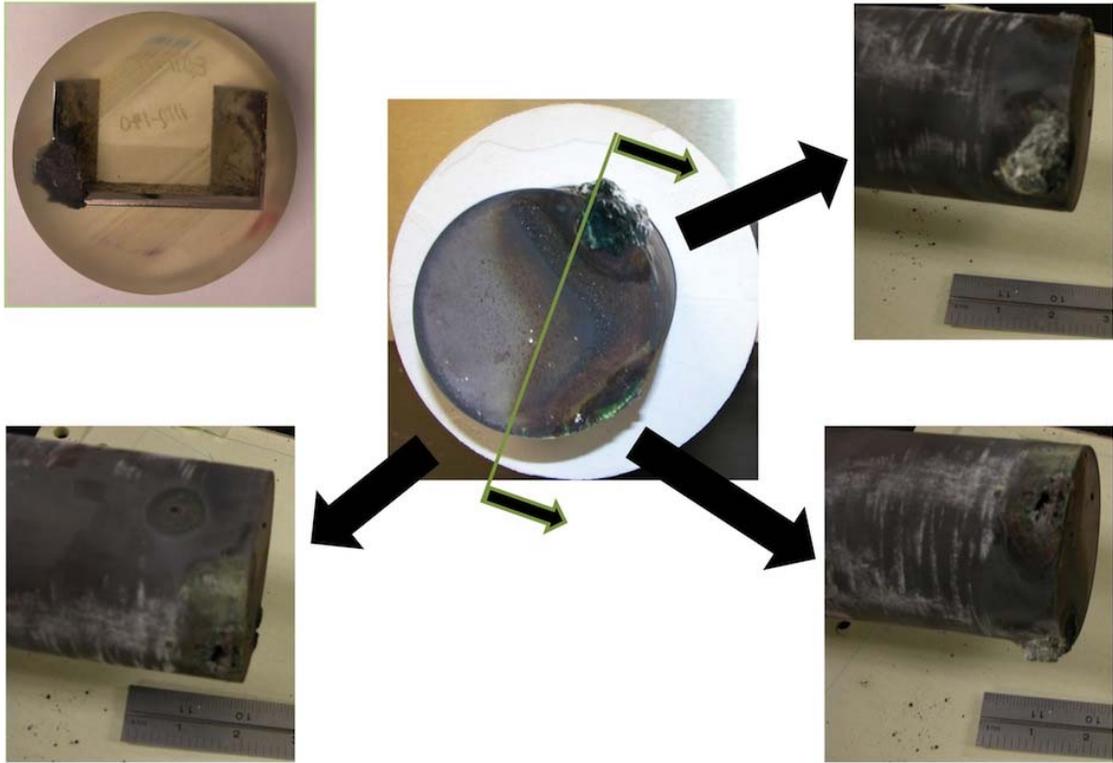


Figure 2. Images of corroded vessel, showing blister (top right), hole (bottom right), position of weld compared to hole (bottom left), and cross-section (top left).

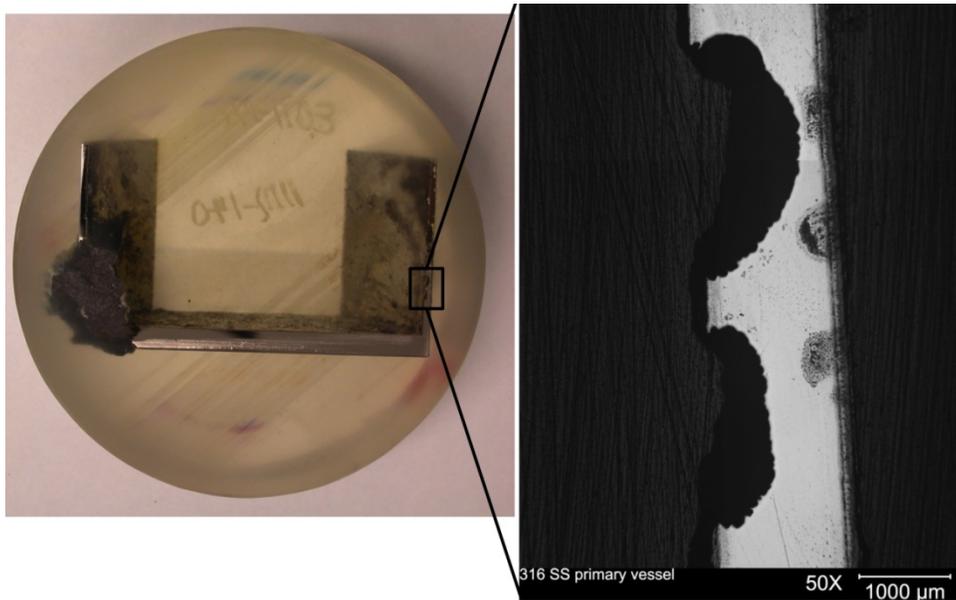


Figure 3. Cross-section of corroded vessel (left) and optical microscopy image of pitting corrosion in the vessel wall (right). A large blister can be seen on the left bottom corner of the cross-section.

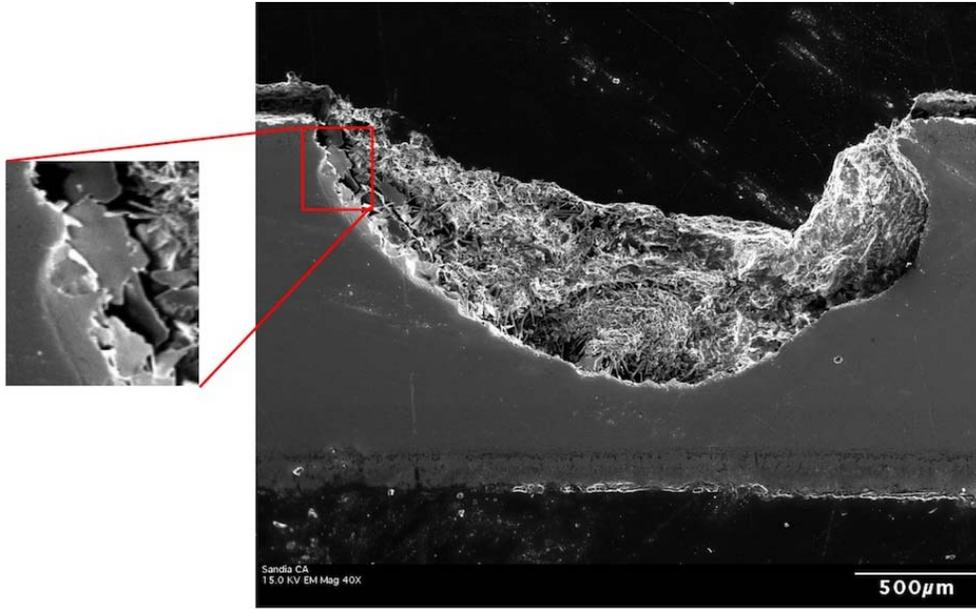
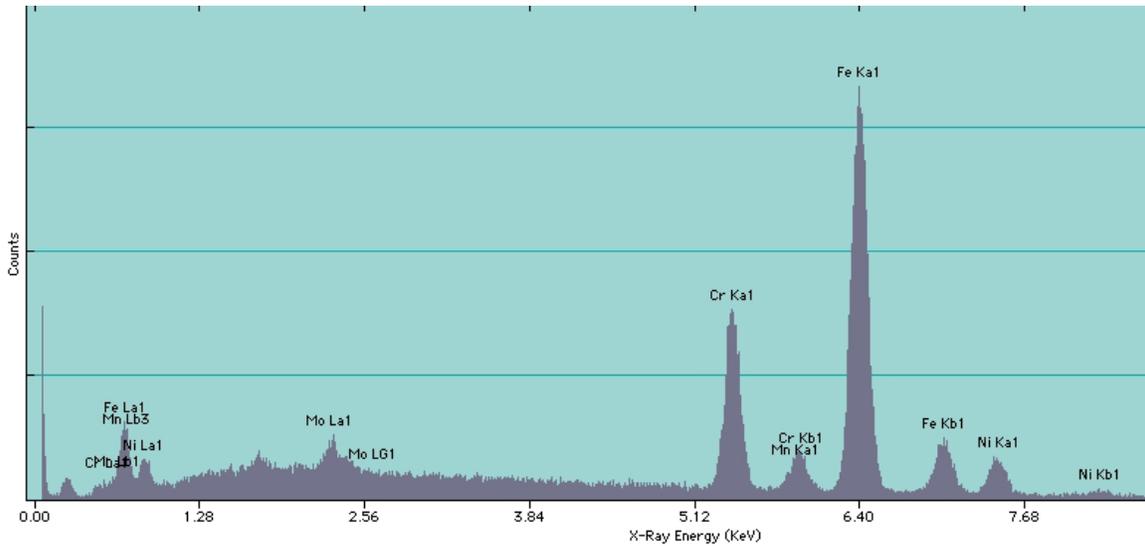


Figure 4. SEM image of corroded pit and close up of oxide formation on pit surface (left).



Quantitative Results for Spectrum1
 Analysis: Bulk Method: Standardless
 Acquired 19-Aug-2011, 15.0 KeV @10 eV/channel

Element	Weight %	Std. Dev.	MDL	Atomic %	k-Ratio	Intensities	FWHM (eV)	ROI (gross)
Mo ?	1.64	0.54	1.90	0.96	0.0131	1040.3	113.8	1208.94
Mn	2.23	0.71	1.53	2.27	0.0225	827.6	146.8	1928.34
Ni	11.04	0.79	1.20	10.51	0.1049	1935.9	158.8	2050.16
Cr	15.89	0.87	0.38	17.07	0.1803	8774.6	142.6	9414.63
Fe	69.19	1.86	0.33	69.20	0.6856	21523.9	150.6	21462.22
Total	100.00							

Figure 5. EDS spectrum analysis of corroded stainless steel vessel (baseline alloy analysis).

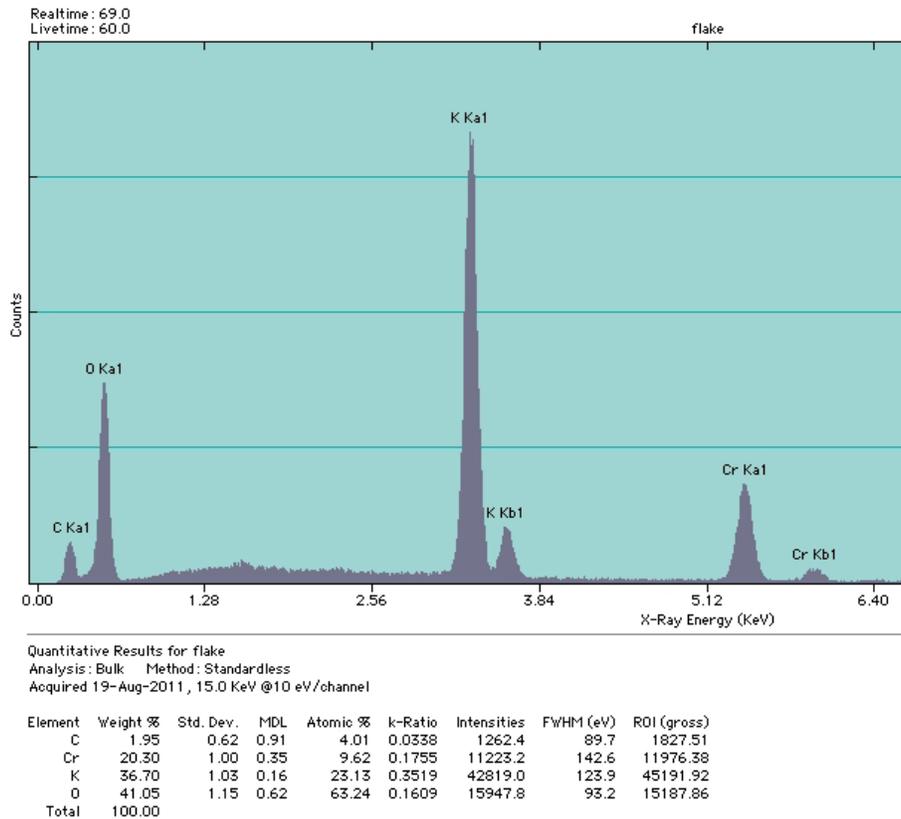


Figure 6. EDS analysis of oxide layer on corrosion pit.

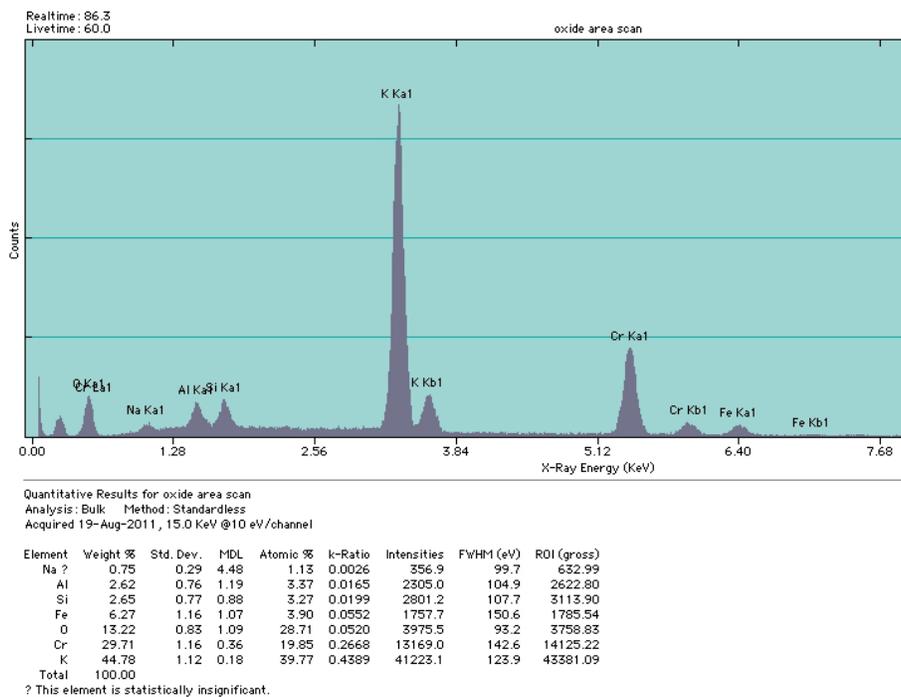


Figure 7. EDS analysis of blister formed on corroded stainless steel vessel.

References

1. R. Bradshaw, *Oxidation and Chromium Depletion of Alloy 800 and 316SS by Molten NaNO₃-KNO₃ at Temperatures Above 600 Degrees Centigrade*, SAND86-9009. Sandia National Laboratories, Livermore, CA, January 1987.

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