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Corrosion in Molten Salts

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Using Phase Change Materials (PCMs) for solar thermal energy storage will require containers made from compatible materials. Corrosion of the container material will not only limit the lifetime of the containers but may affect the thermophysical properties of the PCM. Thus the compatibility of likely PCMs with container materials must be assessed. QUT has been developing its testing and analysis capabilities for this application.

Testing Procedure

A testing methodology was developed which uses multiple samples and crucibles machined from the same alloy to prevent interactions from compounds not present in the alloy.

The crucible and salt is heated according to a desired temperature regime either isothermal or cyclic, depending upon the aims of the experiment.

Mass loss, thickness change and electrochemical methods are used to understand corrosion rate. XRD, Raman Spectroscopy, SEM-EDS and IC-OES can be used to understand corrosion products and mechanisms.

Results

A set of metals, samples were thermally cycled from 600 - 750°C for 24 hours in contact with two different salts. The SS316 coupled with the Na_2CO_3/NaCl PCM leaked out of the crucible causing the experiment to stop after 24 hours and 9 thermal cycles. Photos of the samples post-test are presented in Table 1.

TABLE 1. Alloys and salts tested

	710PCM (52wt% Na_2CO_3 : 48wt% K_2CO_3)	632PCM (60wt% Na_2CO_3 : 40wt% NaCl)
SS 316		
Inconel 601		

Corrosion Rate

The gravimetric analysis of a submerged sample was undertaken by blasting the sample with plastic beads and removing all visible corrosion product and comparing the mass to the uncorroded sample.

Thickness loss was investigated by sectioning the sample and imaging it with SEM. The thickness of the samples were measured using Image J software.

TABLE 2. Mass loss of submersed samples

Alloy	PCM	Mass Loss (g)	Mass Loss (g/cm²)	Thickness Change (µm)
Inconel 601	710	0.0185	0.00706	10
Inconel 601	632	0.0176	0.00679	0
SS316	710	0.0187	0.00724	50
SS316	632	0.0187	0.00770	10

Electrochemical Testing

Electrochemical testing of metals in molten salts is possible as molten salts are conductive. This allows the application of a raft of electrochemical testing techniques such as the impedance and potentiodynamic polarisation curves in Figure 1.

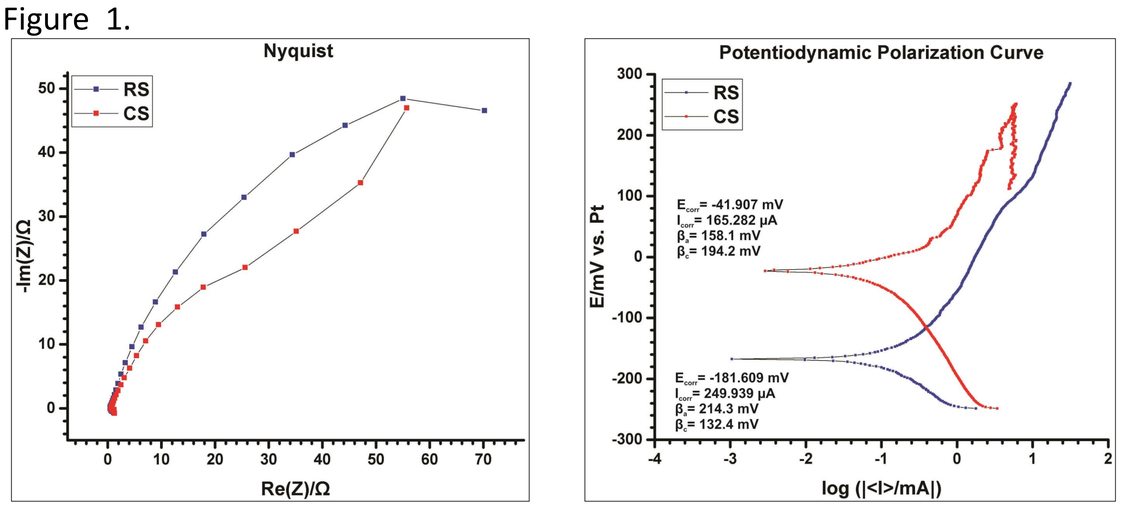

FIGURE 1. Impedance and potentiodynamic polarisation curves from electrochemical testing in molten salts (Li_2CO_3:Na_2CO_3:K_2CO_2)

IC-OES

Samples of the salts were tested with IC-OES to determine the levels of contaminants in the salt. The results for the 3 salts which did not leak are presented in Table 2.

TABLE 3. IC-OES results of salts in corroded samples by weight %

Alloy	PCM	K	Na	Cr	Fe	Ni	Mn	Mo	Si
Inc. 601	710	26.11	22.85	**0.436**	0.006	0.031	0.003	0.001	0.001
Inc. 601	632	0.028	39.96	**0.822**	0.015	0.004	0.002	0	0.002
SS316	710	29.14	19.88	0.026	**0.757**	0.007	0.028	0.032	0.004

SEM-EDS

SEM of samples was undertaken using a Zeiss Sigma field emission SEM with an Oxford XMax 50 Silicon Drift EDS detector. Figure 2 presents SEM-EDS images of the Inconel 601 samples tested with the 632PCM and depicts Chromium dissolution from the surface.

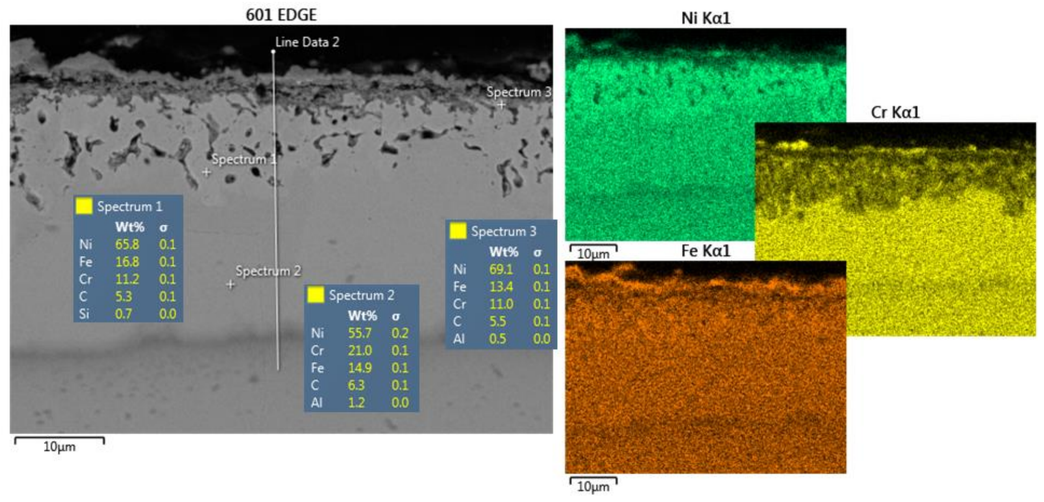

FIGURE 2. SEM-EDS and Inconel 601 after testing in 632PCM

X-Ray Diffraction

XRD was undertaken to determine the phases present on the surface of the metal samples. In addition to the salt phases, sodium ferrate ($NaFeO_2$) was the major corrosion product detected on the SS316 sample testing in 632PCM.

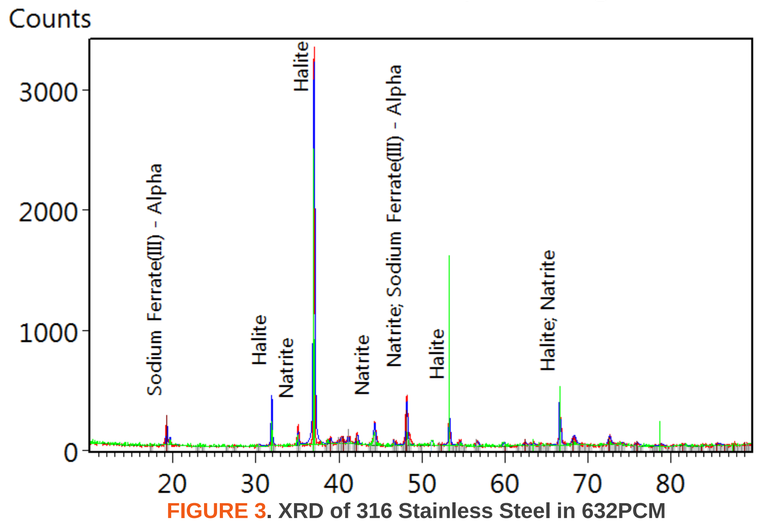

FIGURE 3. XRD of 316 Stainless Steel in 632PCM

Cyclic Testing

A System for rapid thermal cycling of a PCM at temperatures up to 700°C.

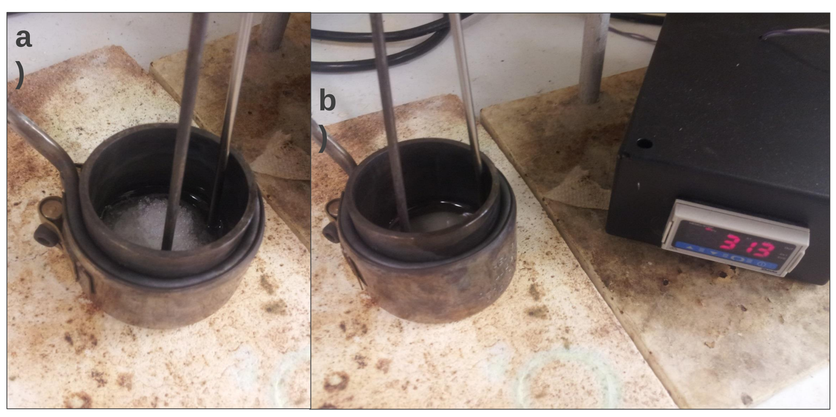

FIGURE 4: Cyclic Testing system; a) semisolid $NaNO_3$ at 305°C; b) liquid $NaNO_3$ at 313°C

Conclusion

Understanding the corrosion of alloys in molten salt media is critical for the design of thermal energy storage systems utilizing molten salts. A consistent methodology for conducting experiments and analyzing result is required meaningful comparison of results between different studies. This paper presents some corrosion testing and undertaken at QUT.

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