SAND2021-12282C

# Haynes 230 and Inconel 625 Corrosion Analysis within a Ternary Chloride Salt

Aaron Overacker[1, a)], Patrick Burton [2)], Dimitri Madden[1)] and Kenneth Armijo[1)]

[1]Concentrating Solar Technologies Department 8923, Sandia National Laboratories, PO 5800 MS 1127, Albuquerque NM, USA 87185
[2]WMD Threats & Aerosol Science Department 6633, Sandia National Laboratories, PO 5800 MS 0734, Albuquerque NM, USA 87185
[a)] aovera@sandia.gov

**Abstract.** The United Sates Department of Energy (DOE) Generation 3 Concentrated Solar Power (CSP) program is interested in higher efficiency power systems at lower costs, potentially with systems utilizing chloride molten salts. Ternary chloride molten salts are corrosive and need to be held at high temperatures to achieve higher power system efficiencies. However, materials and cost of manufacturing of such a facility can be very expensive, particularly using exotic materials that are not always readily available. Materials that can withstand the harsh corrosive and thermal-mechanical environments of high-temperature molten salt systems ( >700 °C) are needed. High temperature systems offer greater thermodynamic efficiency but must also make cost efficient use of corrosion-resistant alloys. To ensure reliable high-performance operation for molten salt power plant designs confidence in materials compatibility with CSP Gen 3 halide salts must be established. This paper will present an analysis of Inconel 625 as an alternative to the costly Haynes 230. Each sample was weighed pre- and post-test, with a final composition analysis using Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray Spectroscopy (EDS). Preliminary findings suggest that Haynes 230 outperformed Inconel 625, but more research at longer durations, 1,000 hours will be required for full reliable assessment.

## INTRODUCTION

A ternary chloride salt with a composition of 20%NaCl/40%$MgCl_2$/40%KCl base mole fraction has attractive thermophysical properties for employment in a Gen 3 concentrating solar power system as well as thermal energy storage. Molten salt compatibility analysis of Haynes 230 and Inconel 625 coupons was conducted to determine materials compatibility. Results from this investigation will aid in the development of thermal-hydraulic equipment, such as molten salt valves and pumps, to operate within systems >700°C. The test materials investigated here were representative of forging, wrought and casting processes. In addition, the team also investigated salt compatibility with weldments that were created within some of the coupons to assess corrosion and mass loss.

## TEST SET UP

### Molten Salt Test Pot Design

To safely conduct these tests while maintaining required temperature, pressure, salt purity, and internal atmospheric conditions, a molten salt test pot system was designed consisting of two vessels. The first vessel was a transfer vessel designed for maintaining purity of the ternary chloride salt. The transfer vessel was exchanged with the National Renewable Energy Laborites (NREL) for purification and inventory. The transfer vessel was filled with purified salt under a glove box with an argon cover gas. After filling, the various ports on the vessel were closed with Swagelok caps and the flange was torqued to the 100 ft-lbs to ensure a proper seal. A spiral wound gasket was used in the flange area. Once purified, the chloride salt was kept sealed within the vessel to avoid introducing air into the bulk volume which could decrease salt purity and facilitate $MgCl_2$ decomposition to liberate chloride ions. The decomposition products could result in formation of hydrochloric acid. To solve this issue, the vessel was constructed out of 316L stainless steel pipe, 316L plate, and a class 150 flange. The vessel has ports for supplying nitrogen cover gas, connecting a vacuum line, inserting thermocouples, and inserting a 0.25 in tube for flowing salt out of the vessel once it is sealed. At SNL, the transfer vessel was immediately connected to argon cover gas supply and pressurized to maintain positive pressure while the vessel was integrated into the larger test system. The second vessel was the salt corrosion test pot. This test pot was also constructed out of stainless steel 316L pipe, 316L plate, and a class 150 flange sealed with a spiral wound gasket. The purpose of this test vessel was to create an enclosed environment where pressure, temperature, and atmospheric conditions can be maintained. An alumina crucible was used inside this pot in order to avoid cross contamination of the stainless-steel vessel and the nickel alloys. The salt was transferred directly into the crucible at low pressure to reduce splashing. The test pot has ports for connecting a vacuum line, a cover gas line, and for connecting a 0.25 in tube which was used to flow molten salt between vessels. There is also a large port on top of the flange used for raising and lowering the metal samples into and out of the salt without exposing the salt to air.

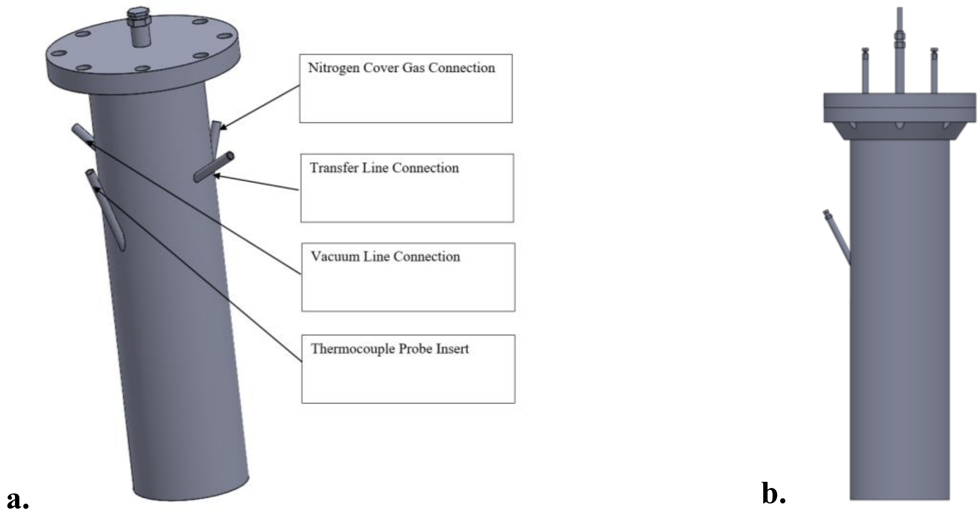


**FIGURE 1.** (a) CAD image of Transfer Vessel, and (b) CAD image of Test Pot

In order to fully submerge the test samples an alumina disk was machined to hang the 1in. x 1in. x 0.25in. samples with pure nickel wire shown in figure 2a. The samples were hung from an alumina plate to prevent galvanic coupling, which could occur if two strings of samples were in contact with the same piece of metal, allowing for ion transfer. The alumina plate was a corrosion resistant and temperature rated alternative to hanging the samples from metal. Holes were drilled in the alumina plate to allow for hanging of samples. Pure nickel wire was used to reduce the presence of galvanic coupling between dissimilar metals, however both were high in nickel content. It was later found that the liquid vapor interface of the salt proved problematic and the nickel wire was then replaced with alumina rods to survive. Figure 2b displays how the three samples are hung in series.


**FIGURE 2.** (a) Alumina Disk Machined to hold Samples, and (b) Display of how samples were hung

Each pair of holes in Fig. 2a was strung together to the samples in accordance to Fig. 2b for each even set of holes on the 60-degree lines. Each odd number set of holes held a 4 in. by 1in x 0.25 in. alumina plate to help reduce the ion mobilization from one sample to another through the salt. The alumina plate also had a 0.5 in. hole in the center. A threaded c-276 rod passed through this hole and the alumina plate was fixed in place with a c-276 nut. The threaded c-276 segment connected to a longer 0.5 in. OD rod. To raise and lower the samples out of or into the liquid salt, this rod slides through a 0.5 in. -0.75 in. bored through Swagelok reducing union on the flange of the salt pot. The fitting uses Teflon ferrules and can be tightened to lock the rod in position or loosened to allow the rod to slide freely. The Teflon ferrules survive the goal temperature of 760 ℃ due to a thermal barrier that reduces the temperature above the flange to below 100 ℃. This thermal barrier slides within the test pot above the alumina disk with a graphite seal. The barrier is a made from stainless-steel 316L and filled with mircotherm pourable insulation layers. Both vessels are supplied with cover gas via two separate but identical gas lines. Two separate lines are used so that gas supply can be separately controlled to each individual vessel.

## COMPARISON

Table 1 shows the similarity between Haynes 230 and Inconel 625. Not only are the compositions similar, but Fig. 3 provides further insight into Inconel 625's comparable strength at higher temperatures to H230.

**TABLE 1.** Alloy Composition

| ELEMENT | HAYNES 230 [1] | INCONEL 625 [2] |
|---|---|---|
| NICKEL | 57.000 | 58.000 Min |
| CHROMIUM | 22.000 | 20.000-23.000 |
| TUNGSTEN | 14.000 | |
| MOLYBDENUM | 2.000 | 8.000-10.000 |
| IRON | 3.000 Max | 5.000 Max |
| COBALT | 5.000 Max | 1.000 |
| COPPER | | |
| MANGANESE | 0.500 | 0.500 |
| SILICON | 0.400 | 0.500 |
| NIOBIUM | 0.500 | 3.150-4.150 |
| ALUMINUM | 0.300 | 0.400 |
| TITANIUM | 0.100 max | 0.400 |
| CARBON | 0.100 | 0.100 |
| LANTHANUM | 0.020 | |
| BORON | 0.015 max | |
| SULFUR | | 0.015 |
| PHOSPHORUS | | 0.015 |

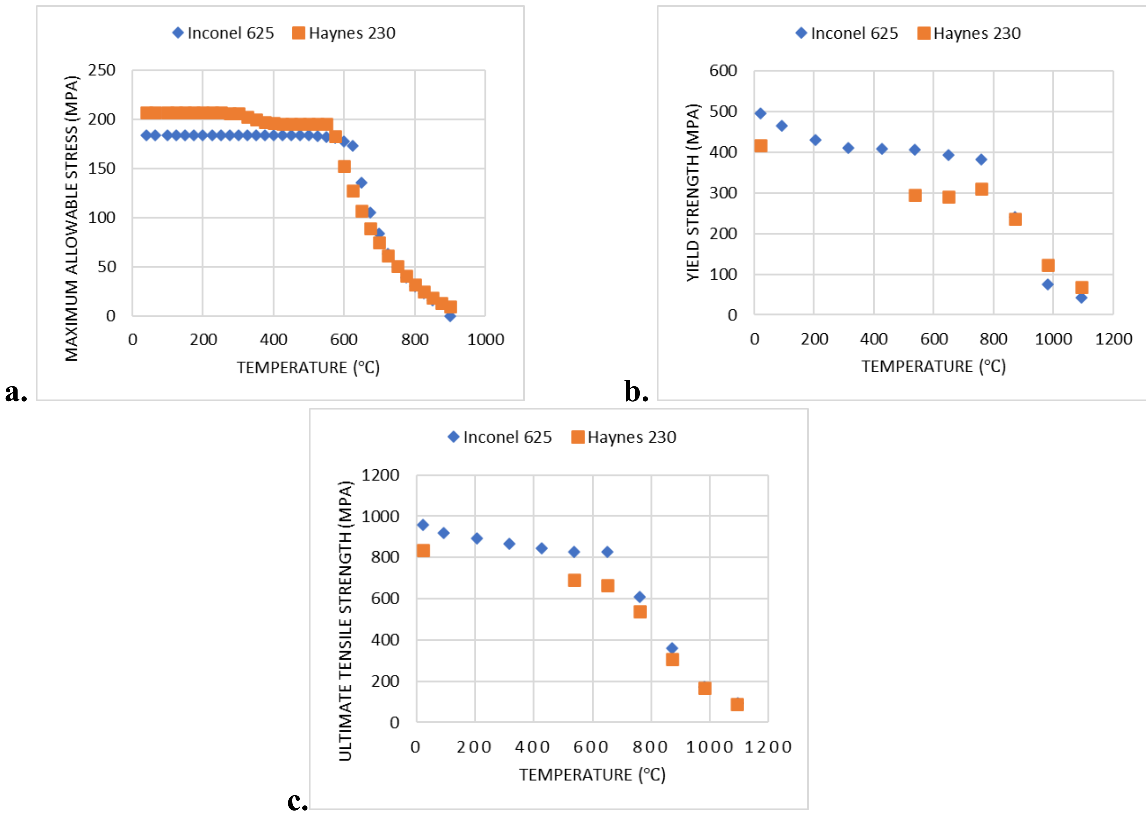


**FIGURE 3.** (a) Temperature Vs. ASME Maximum Allowable Stress, (b) Temperature Vs. Yield Strength, (c) Temperature vs. Ultimate Tensile Strength

While also showing more strength at higher temperatures, required for Gen 3 designs, Inconel 625 is more readily available and at lower cost. Multiple vendors were contacted for cost of 3in schedule 80 pipe, with a minimum order of 500 ft. Results of the vendor trade study showed that Haynes 230 had a cost of $820/ft. while Inconel 625 had a lower cost of $377.00/ft.

# RESULTS & DISCUSSION

## Corrosion Rate

Prior to salt exposure the samples were weighed to find the corrosion rate using the equation below.

$$\frac{\mu m}{Y} = 87{,}600 * \left(\frac{\Delta M}{\rho A T}\right) \tag{1}$$

Following the completion of the salt compatibility experiments, samples were immediately put in an inert environment to minimize the corrosion by residual salt that dried on the surface during cooling. Samples were then cleaned in an ultrasonic bath with deionized water, rinsed in ethanol, and reweighed. Samples were not scoured and were handled as delicately as possible to avoid dislodging any material. Table 2 displays the corrosion rate, demonstrating that Haynes 230 outperforms Inconel 625 for 500-hrs. in ternary chloride salt.

**TABLE 2.** Corrosion Rate (µm/year)

| Alloy Type | Sample 1 Corrosion Rate | Sample 2 Corrosion Rate | Sample 3 Corrosion Rate |
|---|---|---|---|
| **Inconel 625** | 57.4 | 47.8 | 44.5 |
| **Welded Inconel 625** | 56.6 | 79.7 | 73.6 |
| **Haynes 230** | 12.6 | 7.08 | 18.9 |
| **Welded Haynes 230** | 8.27 | 1.18 | +5.61 |

Though this seems like a significant win for Haynes 230, Inconel 625 has no documented performance assessments in ternary chloride salts besides what is shown above.

## SEM/EDS

The outer surface and cross-sectioned material surfaces, of each coupon were examined using scanning electron microscopy (SEM) equipped with an energy dispersive spectroscopy (EDS) detector. After rinsing and drying the coupons, a brittle green deposited material remained adhered in some areas. The deposit was found to consist mostly of magnesium oxide, alumina, and occasional residual KCl crystallites. The Haynes 230 coupons tended to accumulate more residue than the Inconel 625. Long term material accretion on these surfaces may be an issue and will be investigated in longer duration tests.

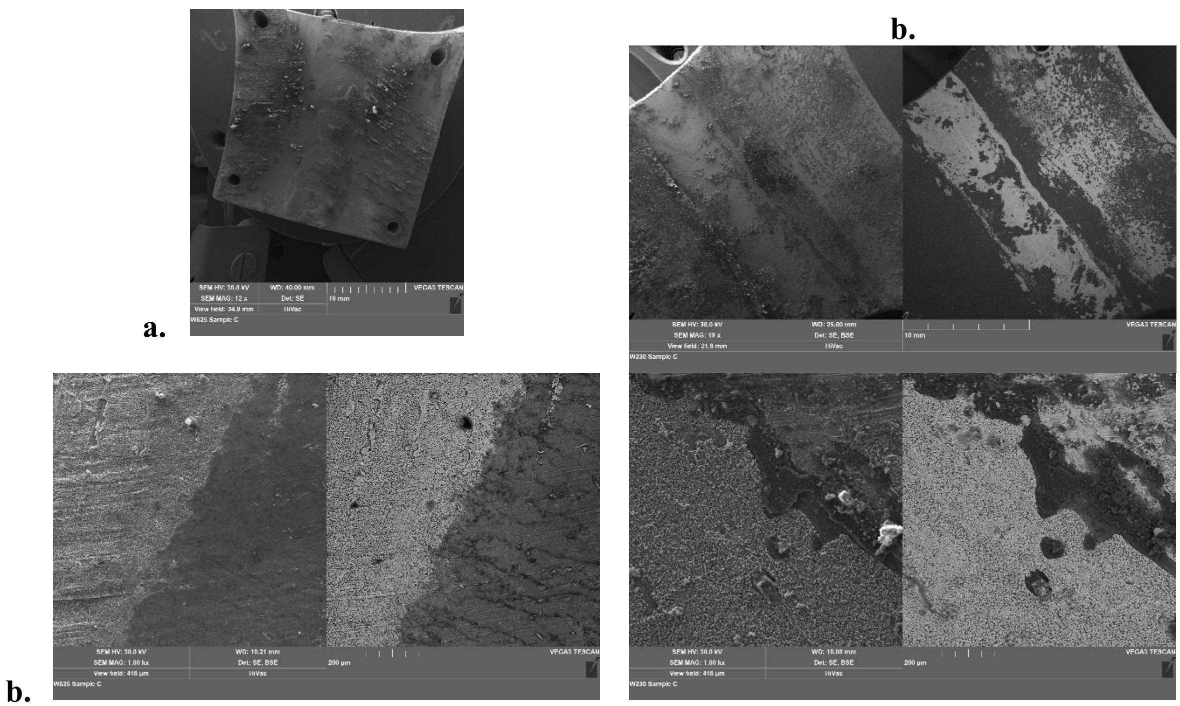

**FIGURE 4.** (a) Wide field SEM micrograph of welded Inconel 625 coupon and (b) higher magnification area near weld seam. (c) Haynes 230 coupon and (d) higher magnification showing exposed metal and residue (secondary and backscatter views).

The depth of corrosion was determined by measuring a chromium depletion boundary layer relative to the bulk composition. A line scan was preformed across multiple regions on each sectioned coupon, with at least 50 µm between each probed region. The welded materials were sectioned perpendicular to the welds so the weld bead could be seen in its entirety.

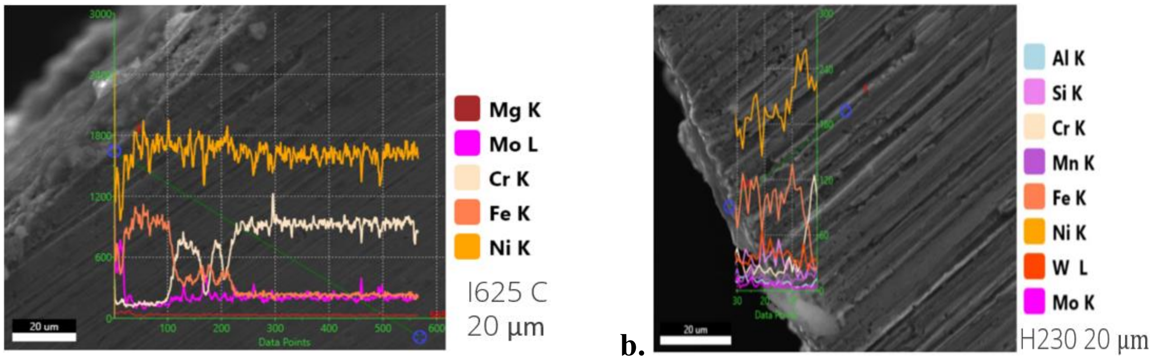


**FIGURE 5.** (a) Inconel 625 display of chromium depletion, and (b) Haynes 230 display of chromium depletion

Fig. 4 a and b illustrate the chromium depletion near the surface. As the line scan from the EDS continues further into the sample, the chromium level should surpass the iron level due the percentage of chromium for both Inconel 625 and Haynes 230. On average both metals have comparable chromium depletion with Inconel 625 at 35 µm and Haynes 230 at 33 µm

## CONCLUSION

Since neither material exhibited a substantially superior performance, Inconel 625 will be continued to be tested at longer durations as well as tested for strength post exposure. Haynes 230 demonstrated a slight edge in performance, but at nearly four times the cost. Inconel 625 could drive down the cost of multiple components in next generation solar thermal facilities, but continued research needs to be done in order to determine if Inconel 625 would be sufficient in such a harsh environment.

## ACKNOWLEDGMENTS

Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

## REFERENCES

1. Haynes International, *Haynes 230 alloy*, http://haynesintl.com/docs/default-source/pdfs/new-alloy-brochures/high-temperature-alloys/brochures/230-brochure.pdf
2. Special Metals, *Inconel alloy 625*, https://www.specialmetals.com/assets/smc/documents/alloys/inconel/inconel-alloy-625.pdf
3. High Temp Metals, *Inconel 625 Technical Data*, http://www.hightempmetals.com/techdata/hitempInconel625data.php
4. W. Yu, D. Singh, D.M. France, *Investigation of Corrosion of 304 Stainless, Inconel 625, and Haynes 230 in a Chloride-Salt-Based Thermal Storage Medium* (Journal of Materials Engineering and Performance, 2019) 7379–7389. https://doi.org/10.1007/s11665-019-04508-y