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Protective Coatings for Alloys in Contact with Molten Drawsalt (NaNO₃-KNO₃)

R. W. Carling, R. W. Bradshaw, R. W. Mar

Prepared by
Sandia National Laboratories
Albuquerque, New Mexico 87185 and Livermore, California 94550
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PROTECTIVE COATINGS FOR ALLOYS IN CONTACT WITH MOLTEN
DRAWSALT (NaNO₃-KNO₃)

R. W. Carling, R. W. Bradshaw, and R. W. Mar
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ABSTRACT

Molten drawsalt (NaNO₃-KNO₃) is being considered as the energy transfer and storage medium for many solar central receiver applications. In an effort to reduce the cost of the containment material while maintaining corrosion resistance, alloys with aluminide coatings have been examined while in contact with molten drawsalt for more than 6000 hours at 600°C. The alloys examined were 2-1/4 Cr-1 Mo, 5 Cr-1/2 Mo, and 9 Cr-1 Mo low-alloy steels, and 316 stainless steel. The results show a steady, albeit slow, net weight loss over the course of the experiment. The weight loss has been attributed to spalling of Al₂O₃ from the surface (the occurrence of Al₂O₃ is a result of the aluminizing process) and dissolution of corrosion products NaAlO₂ and/or NaFeO₂ during post-immersion handling. Scanning electron micrographs of exposed surfaces revealed little or no corrosion of the base metal. It has been concluded that aluminide coated alloys could provide significant cost savings (~50 percent) relative to Incoloy 800, and provide at least equivalent corrosion resistance.

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PROTECTIVE COATINGS FOR ALLOYS IN CONTACT WITH MOLTEN DRAWNSALT ($\text{NaNO}_3\text{-KNO}_3$)

Introduction

Some current designs for solar central receivers propose the use of molten drawnsalt ($\text{NaNO}_3\text{-KNO}_3$) as the heat transfer and energy storage medium.¹ In such systems, receiver material containing the molten salt will be subjected to severe environments, including at least a diurnal temperature cycle of ambient to 600°C and mechanical stresses imposed by the heating of the receiver tubes. In other sections of the system (e.g., heat exchanger and salt storage tanks), temperatures, stresses, and cycling are less severe.

Early experiments screening potential alloys for molten salt containment showed that high chromium contents (~15 percent or higher) were required for adequate corrosion resistance.¹⁻³ Accordingly, high strength austenitic alloys with high chromium contents were selected for further experimentation. Currently, Incoloy 800 (I800) is proposed for virtually all solar central receiver applications (primarily receiver tubes) where high creep-fatigue strength and resistance to corrosion in 500-600°C molten salt are required.

Incoloy 800 has the disadvantage of being significantly more expensive than 300-series stainless and low-alloy steels and, because of its high chromium and nickel content, utilizes a large quantity of strategic elements. Since the United States is almost totally dependent on foreign sources for chromium and nickel supplies, future political situations may play an important role in chromium availability.^{4,5} For these reasons, other alloys and alloy treatments are being examined in an effort to reduce costs as well as the chromium and nickel content, while retaining the corrosion resistance of Incoloy 800.

An example of an alloy treatment which provides corrosion resistance in many applications is aluminide coating.⁶ The formation of this coating involves diffusing aluminum from an external source into the alloy to form a high-melting aluminum intermetallic. Recent cost analyses for alloy tubing purchased in quantities suitable for a central receiver showed that ferritic Cr-Mo steels and austenitic type 304 and 316 stainless steels are between 25 to 50 percent the cost of I800.⁷ The cost of providing an aluminide coating on the tubes would add some expense but the overall cost of an aluminide-coated alloy would be substantially less than uncoated I800. For example, the cost of 304 SS with an aluminide coating is estimated to be 50 percent the cost of uncoated I800.

Earlier work that investigated aluminide-coated 304 stainless steel in molten HITEC (40% NaNO_2 , 7% NaNO_3 , and 53% KNO_3 by weight) indicated that aluminide coatings improved corrosion resistance under severe conditions.⁸ The use of HITEC and the high temperature (770°C) in those experiments represented an overtest for a central receiver application, since HITEC is more corrosive than drawsalt⁹ and the maximum tube temperatures in a central receiver are expected to be about 600°C. The work reported here concerns other alloys (i.e., 316 stainless steel, 2-1/4 Cr-1 Mo, 5 Cr-1/2 Mo, and 9 Cr-1 Mo steels) that have potential applications in various components of a central receiver system using molten drawsalt.

Experimental

Coupons (approximately 7 x 25 x 2 mm) were prepared from three chromium-molybdenum alloy steels and 316 stainless steel for a corrosion experiment. The alloy compositions are presented in Table I. The coupons to be tested were coated by a pack cementation process by Alon Processing, Inc., Tarentum, PA. This treatment, commonly referred to as "alonizing," diffuses aluminum into the metal surface to form a high-melting aluminum intermetallic. The two-layer structure of the coating typically formed on the Cr-Mo steels is shown by the micrograph in Figure 1. The outer layer has a relatively high Al content (AlFe intermetallic) and tends to have inward-oriented cracks. The inner layer has a somewhat lower Al content and appears to be a solid-solution alloy of Al in Fe, and is crack free.

The drawsalt composition was prepared by mixing equimolar amounts of NaNO_3 and KNO_3 . Three coupons of each alloy were attached to aluminum rods using nickel wire and placed within their own alumina tube containing drawsalt. The alumina tubes were held in a muffle furnace and heated to 600°C. The coupons were withdrawn at periodic intervals, washed of excess salt with water, and weighed. After the final exposure of the alloy to the molten salt, the coupons were mounted and polished for metallographic examination.

Salt that had been exposed to the alloys was analyzed for metal impurities after 1000 and 4000 hours of testing using arc-emission spectroscopy. X-ray diffraction was used to identify phases on the alloy surfaces.

Results

The gravimetric results for the alloys exposed to molten drawsalt are shown in Figures 2-5. These results represent net weight changes after the samples were rinsed in water and dried. Adherent oxide corrosion products were not removed. Each figure provides a comparison of the coated and uncoated alloys. In the case of 2-1/4 Cr-1 Mo and 5 Cr-1/2 Mo, the uncoated alloys were tested at 550°C instead of 600°C since even at 550°C corrosion of the low-Cr steels was rapid.

TABLE I
ALLOY COMPOSITIONS

Alloy*	Cr	Ni	Mo	C	Mn	Si	P	S	Fe
2 1/4 Cr-1 Mo	2.13	—	1.11	0.081	0.57	0.365	—	—	balance
5 Cr-1/2 Mo	5.00	—	0.58	0.120	0.60	0.505	—	—	balance
9 Cr-1/2 Mo	8.30	—	1.08	0.113	0.67	0.533	—	—	balance
316 SS	16-18	10-14	2-3	0.08	2	1	0.045	0.030	balance

*Analyses performed by Oregon Metallurgical Corporation, Albany, Oregon, except for 316 SS which is a nominal composition.

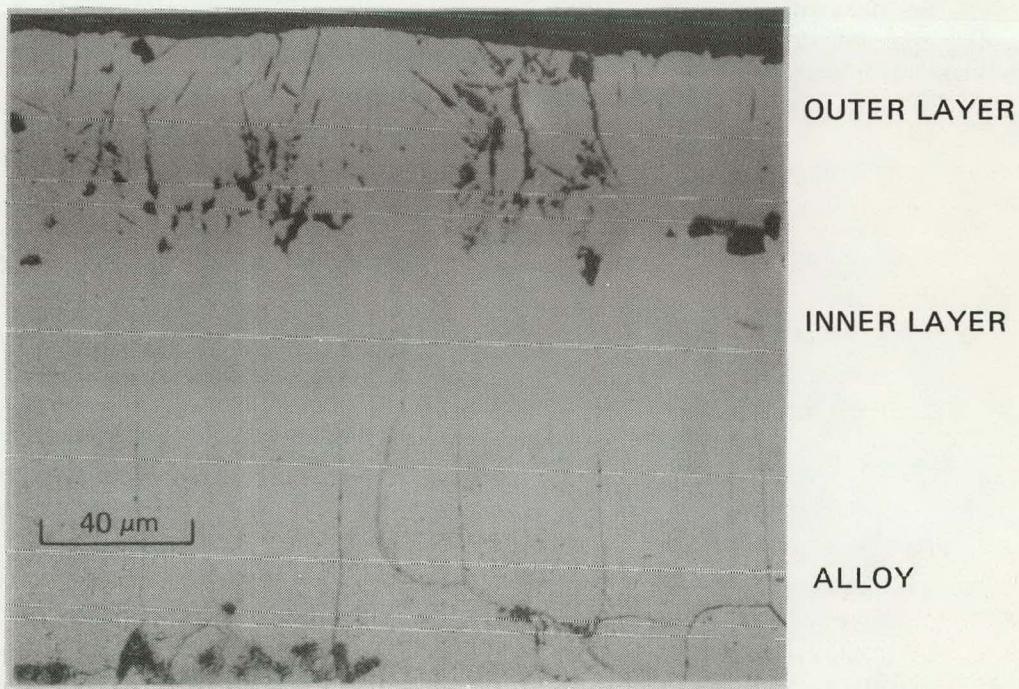


Figure 1. Coating Morphology of As-received Diffusion-Coated 2-1/4 Cr-1 Mo Steel

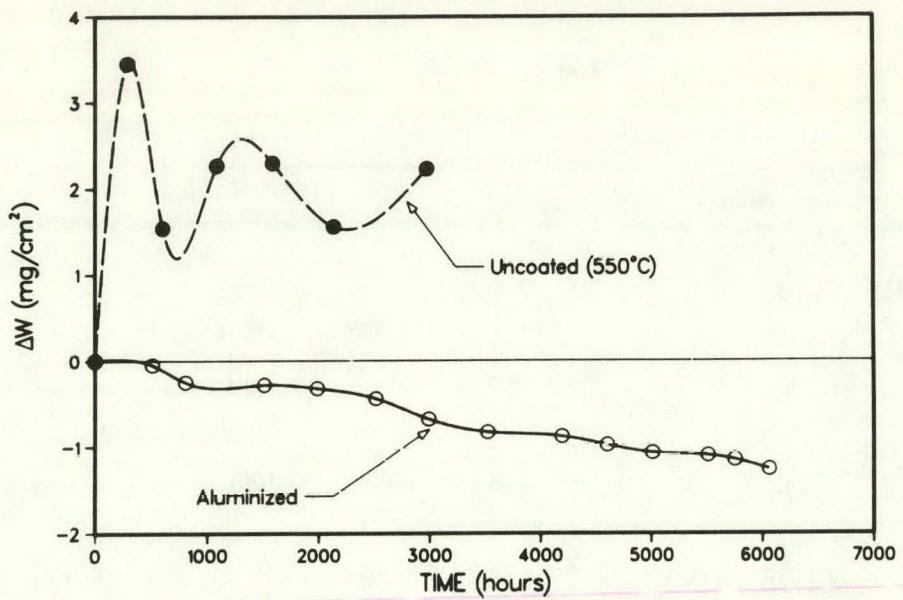


Figure 2. Weight Changes for 2-1/4 Cr-1 Mo: This Work, 600°C; Reference 10, 550°C

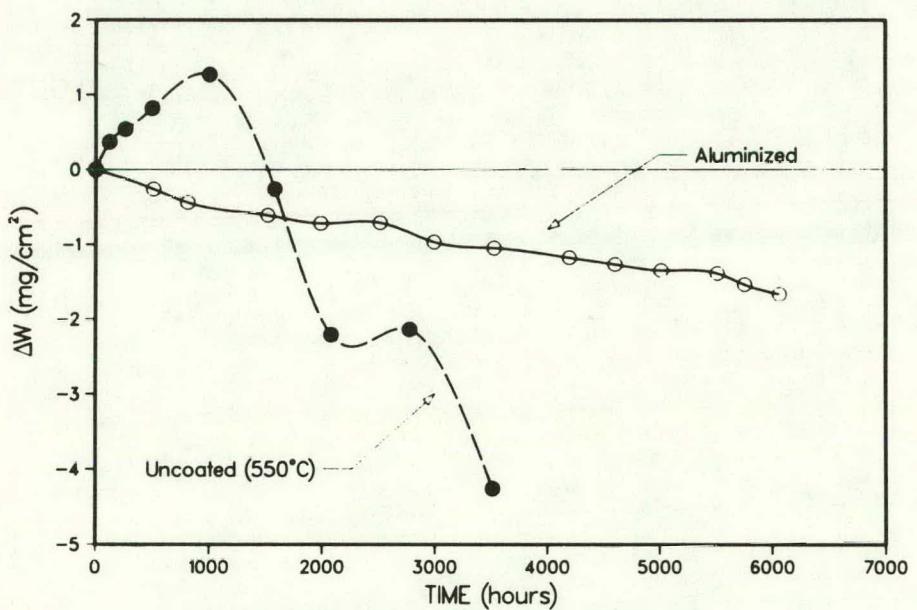


Figure 3. Weight Changes for 5 Cr-1/2 Mo: This Work 600°C; Reference 10, 550°C

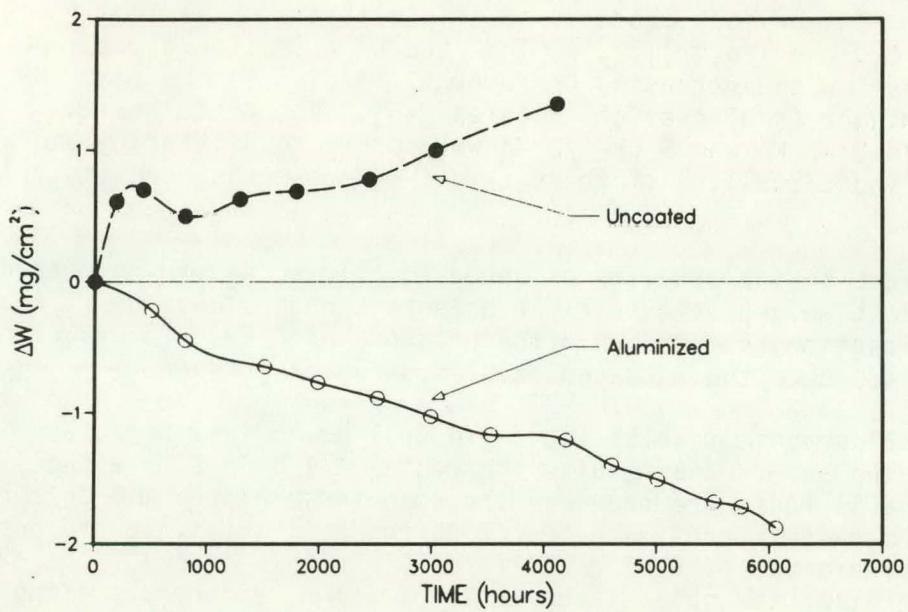


Figure 4. Weight Changes for 9 Cr-1 Mo: This Work, 600°C; Reference 10, 600°C

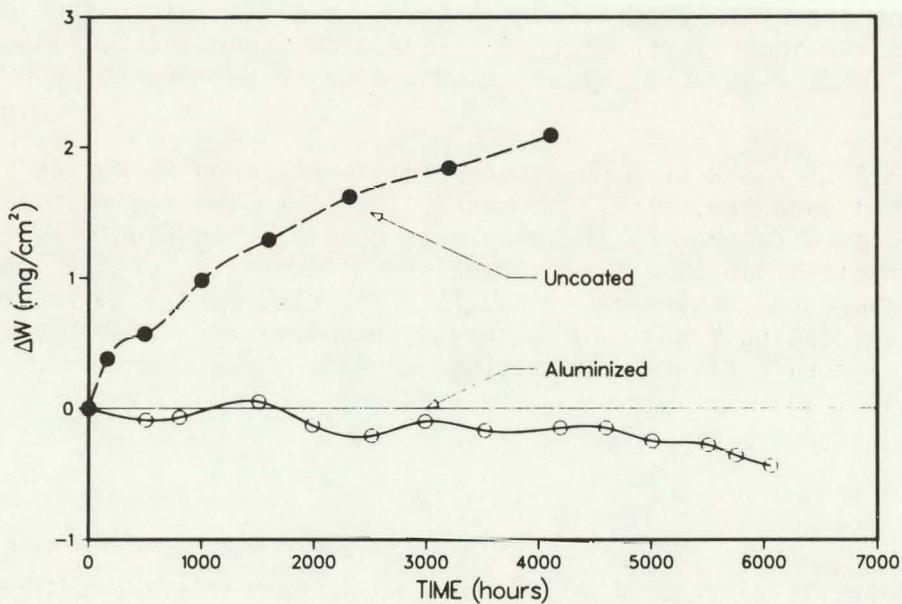


Figure 5. Weight Changes for 316 SS: This Work, 600°C; Reference 10, 600°C

The uncoated alloys initially demonstrated net weight gains as a result of oxidation. The primary products of this oxidation have been identified as Fe_3O_4 , Fe_2O_3 , and $(Fe,Cr)_3O_4$.¹⁰ The rate of oxidation of the uncoated alloys decreases with increasing Cr content, leading to the lower net weight changes for higher Cr alloys (cf. Figures 2-5). The oxide scales formed on uncoated 2-1/4 Cr-1 Mo and 5 Cr-1/2 Mo were prone to blistering and were not adherent; periodic spalling of these oxides was observed, causing a loss in weight.

In contrast to the behavior of uncoated alloys, aluminide-coated samples all lost weight at a fairly constant, though slow, rate. Analysis by X-ray diffraction revealed only the presence of AlFe. The voluminous oxide layers found on the uncoated samples were not present.

The metallographic results for 2-1/4 Cr-1 Mo, 5 Cr-1/2 Mo, and 9 Cr-1 Mo were much the same. The results for coated 2-1/4 Cr-1 Mo after immersion at 600°C for 6061 hours are shown in the scanning electron micrographs in Figure 6. The oxide corrosion products at the salt interface did not form, but there was evidence of some oxide formation within cracks. The salt can be seen penetrating the outer layer of the coating, apparently along pre-existing cracks within the coating. (Several of the cracks are visible in the optical micrograph of an as-received, coated sample shown in Figure 1.) Energy dispersive X-ray analyses showed that the oxides in the crevices (region B in Figure 1) consisted solely of iron oxides; aluminum was absent. Regions A and C in Figure 1 contain aluminum.

The behavior of the aluminide-coated sample contrasts markedly with that of the uncoated sample, as seen by comparing Figures 6 and 7. Extensive oxide buildup is observed on the uncoated coupon after only 1581 hours at 550°C. The corrosion products consisted of a mixed outer layer of Fe_2O_3 and Fe_3O_4 , and an inner layer of an iron-chromium spinel, $(Fe,Cr)_3O_4$. The blisters and voids evident in Figure 7 considerably degrade the adherence of the scale.

Steels 316 SS and 9 Cr-1 Mo display relatively good corrosion resistance even when uncoated.^{1,10,11} However, the corrosion resistance of these alloys is improved further by the aluminide coating. In Figure 8, a micrograph of aluminized 316 SS after exposure to molten salt for 6061 hours at 600°C shows that the coating is virtually unaffected by the molten draw-salt. Several coating cracks are observed, but they were probably present initially as a result of the aluminizing process.

Discussion

The beneficial effects of aluminide coating were clearly evident. Alloys that normally oxidize rapidly when immersed in drawsalt were protected. In fact, the weight changes for coated samples were negative, suggesting no oxide formation and buildup at all. Loss of spalled Al_2O_3 , a residue of the processing treatment, is a possible cause of the steady loss of weight of the coated coupons. The coupons were prepared by heating them in a bed of powdered aluminum and Al_2O_3 sealed in a retort under a reducing

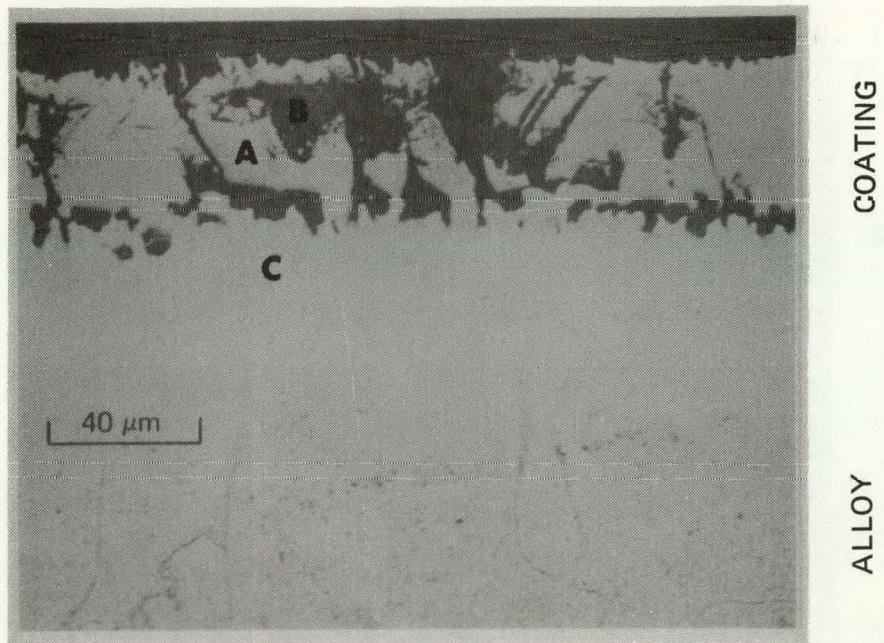


Figure 6. Micrograph of Aluminized 2-1/4 Cr-1 Mo Steel After 6061 Hours in Molten Drawsalt at 600°C

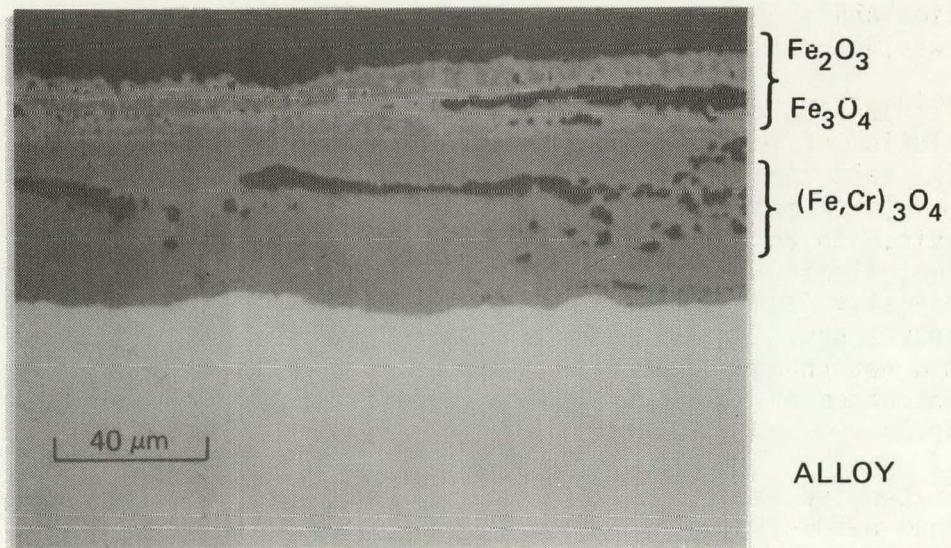


Figure 7. Micrograph of Uncoated 2-1/4 Cr-1 Mo Steel After 1581 Hours in Molten Drawsalt at 550°C

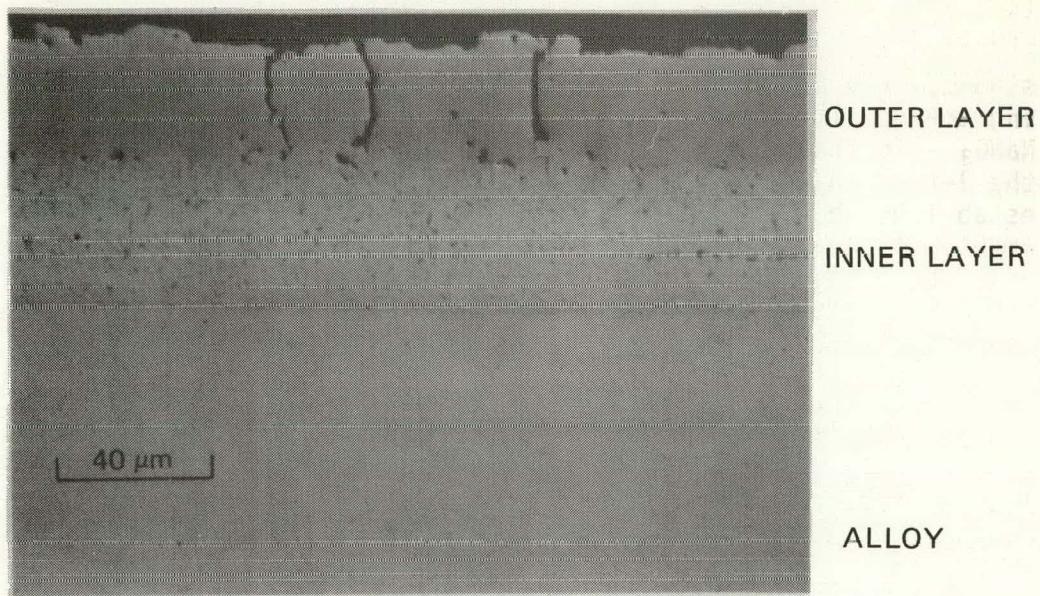


Figure 8. Micrograph of Aluminized 316 SS After 6061 Hours in Molten Drawsalt at 600°C

atmosphere. X-ray diffraction analysis showed the sample surfaces of the as-received coated coupons to be Al_2O_3 and AlFe. Optical microscopy also revealed granules of Al_2O_3 adhering tightly to the coupon surface. At the conclusion of the experiments, the surfaces were reanalyzed by X-ray diffraction and showed only AlFe. Therefore, it is concluded that the weight loss was, at least in part, due to the loss of Al_2O_3 .

It is also possible that the loss of weight was contributed to by the dissolution of metallic elements into the salt melt. To address this possibility, salt samples were taken after 1000 and 4000 hours of exposure and the levels of metal impurities were determined by arc-emission spectroscopic analysis. In addition to sodium and potassium (components of the salt), calcium, aluminum, and chromium were also identified. Calcium is an impurity normally found in the salt and was found to be present in concentrations of about 1 ppm. Al and Cr were found at the detection limit (i.e., ~1 ppm) and did not change between 1000 and 4000 hours of exposure. The invariable concentration of Al and Cr indicates metal dissolution is not a significant factor.

Extensive oxide formation is observed on the uncoated alloys. Since aluminum oxide is more stable than iron oxide, one might expect to see significant oxide formation when aluminide coatings are exposed to nitrate salts. However, the results discussed above showed no evidence of an oxide corrosion product; AlFe appears to be the stable phase in contact with the salt. A thermodynamic analysis of the probable corrosion reactions leads to a reasonable explanation of these observations.

Figure 9 shows the phases in the Na-N-O system that are stable at 600°C (outlined by heavy lines). It can be seen that under the conditions of this study, i.e., $P_{N_2} = 0.8$ atm and $P_{O_2} = 0.2$ atm, the nitrate phase is stable, as expected. Superimposed in the figure is a dotted line representing the equilibrium between $NaAlO_2$ and Al_2O_3 in the presence of excess $NaNO_3$. To the right of the dotted line, Al_2O_3 is the stable phase and to the left, $NaAlO_2$ is stable. If we assume that equilibrium conditions are established by air and are maintained, $NaAlO_2$ is the thermodynamically stable phase and one would not expect to form Al_2O_3 . The fate of Fe can be analyzed in an analogous fashion, and the $NaFeO_2/Fe_2O_3$ equilibrium line is also shown in Figure 9 (dashed line). The stable Fe containing phase in contact with $NaNO_3$ is $NaFeO_2$.

The thermodynamic arguments above indicate that the oxides of Fe and Al are not stable in a nitrate melt, and this is consistent with experimental observations. However, it is surprising that experimental results indicate intermetallic AlFe to be the phase in contact with the salt, and not $NaFeO_2$ and/or $NaAlO_2$. As seen in Figure 9, there are no regions of stability for either Al or Fe. Speculating, the aluminide coating probably undergoes a slow and continual reaction with the nitrate salt, forming $NaAlO_2$ and/or $NaFeO_2$ in the process. Both of these corrosion products are soluble in water and were probably washed off prior to analysis. Thus, the continual weight loss behavior observed in Figures 2-5 is attributed in part to the interaction between the nitrate salt and AlFe, forming $NaAlO_2$ and/or $NaFeO_2$, and the subsequent dissolution of these products during post-immersion handling.

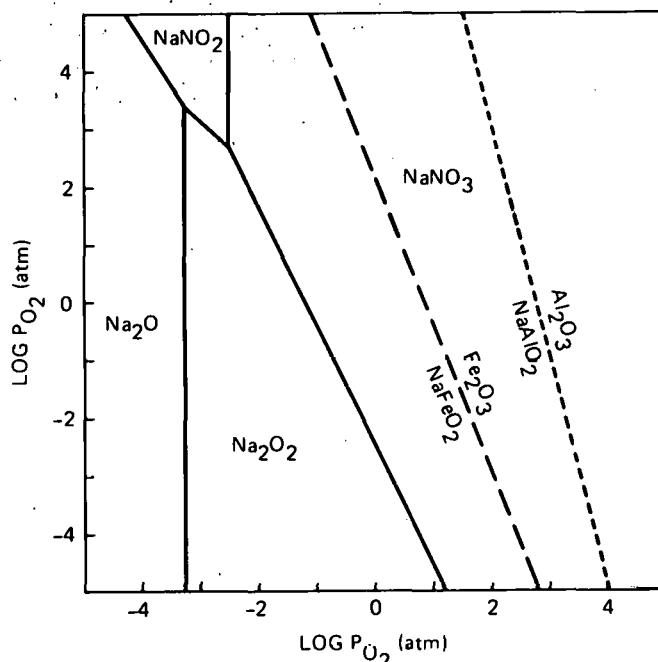


Figure 9. Phase Stability Diagram for Na-N-O at 600°C. The Stable Na-N-O Phases are Outlined by Solid Lines. Stability Regions for Al_2O_3 vs. $NaAlO_2$ and Fe_2O_3 vs. $NaFeO_2$ are Shown by the Dotted and Dashed Lines, Respectively.

It is usually informative to express corrosion results in terms of metal loss due to corrosion in mils per year. Oxide descaling experiments were carried out for the uncoated alloys, and the results indicated losses of 50-75 microns/year (2-3 mils/year) for 2-1/4 Cr-1 Mo and 5 Cr-1/2 Mo at 550°C¹⁰, and approximately 25 microns/year (1 mil/year) for 316 SS¹¹ and 9 Cr-1 Mo¹¹ at 600°C. The maximum metal loss for the aluminized samples did not exceed four microns/year. For comparison, I800 exhibits losses of 12-25 microns/year (0.5-1 mil/year).

Conclusion

Several alloys with aluminide coatings have been tested in molten draw-salt for 6061 hours at 600°C. The corrosion resistance of two low-Cr alloys, 2-1/4 Cr-1 Mo and 5 Cr-1/2 Mo, was substantially improved by the coatings to the extent that these low-alloy steels alloys are compatible with drawsalt at temperatures anticipated throughout a central receiver system. Provided that the high-temperature mechanical design considerations are satisfied, these alloys could be alternatives to higher-alloy I800 and austenitic stainless steels. Although the corrosion resistance of uncoated alloys having higher Cr contents (9 Cr-1 Mo and 316 SS) is probably adequate for some solar applications, aluminide coating provides an improvement in corrosion behavior. Coated alloys are equivalent to I800 with regard to corrosion resistance, but can offer significant cost savings.

The results described in this paper show aluminide coatings to be a promising alternative for use in molten nitrate salt containment systems. However, numerous other issues (e.g. thermal cycling, mechanical stress, thermal fatigue, and cyclic fatigue) must be evaluated before coatings can be considered for use in solar thermal systems.

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