

EXPLORATORY CORROSION TESTS ON ALLOYS IN
MOLTEN SALTS AT 900°

by

R. T. COYLE, T. M. THOMAS and G. Y. LAI

Reprinted from:

High Temperature Corrosion in Energy Systems

Proceedings of the symposium co-sponsored by the Joint Corrosion and Environmental Effects Committee of The Metallurgical Society of AIME and the Materials Science Division of the American Society for Metals; the High Temperature Alloys Committee of The Metallurgical Society of AIME; The Energy and Resources Committee of the Material Science Division; and the American Society for Metals Energy Division.

Edited by
Michael F. Rothman
Cabot Corporation
Kokomo, Indiana

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MOLTEN SALTS AT 900°C

R. T. Coyle
T. M. Thomas

Materials Research Branch
Solar Energy Research Institute
Golden, CO 80401, USA

G. Y. Lai

Cabot Wrought Products Division
Cabot Corporation
Kokomo, IN 46901, USA

Exploratory corrosion tests were conducted on 16 commercial alloys in carbonate, chloride, and hydroxide molten salts at 900°C for up to three weeks. Corrosion information, including weight change, observations of the coupons, metallographic examination, and evaluation of the corrosion product by SEM, was obtained on the coupons exposed to these salts. These tests indicated that a number of the alloys showed significant resistance to metal loss in the carbonate molten salt with corrosion rates on the order of several millimeters per year. The corrosion product is an interpenetrating structure of metal from the more noble alloy ingredients and of an oxide made up of the reaction between melt components and oxidizable metals from the alloy.

INTRODUCTION

Molten salts are attractive as heat-transfer fluids and as sensible-heat storage media in high-temperature, solar-thermal energy systems. One molten salt, sodium-potassium nitrate, has recently received attention as an attractive fluid in these systems from both technical (1,2) and economic (3) viewpoints. Corrosion studies of containment materials in contact with this nitrate salt have shown that up to 600°C, Incoloy* 800, 304 stainless steel, and 316 stainless steel all corrode at a low rate, about 0.02 mm/yr (4,5).

However, because alkali nitrate salts decompose too rapidly at temperatures above 600°C, other salts have been identified as having more suitable physical properties and economic potential for use in solar thermal applications up to 900°C (6,7). These salts include molten carbonates, molten hydroxides, and molten chlorides. The purpose of our work was to assess these salts and a number of candidate alloys to identify molten-salt/alloy combinations that show promise of compatibility.

In this work we selected candidate alloys based on a survey of previous work on the corrosion of alloys in molten salts (8), documented corrosion resistance in similar applications, and good mechanical properties in the range from 700° to 900°C. The selected alloys and their nominal compositions are shown in Table I. Previous corrosion studies of alloys in chloride and hydroxide molten salts indicate that high corrosion rates might be expected in this temperature range; however, since ceramic materials were being evaluated in these salts in a related study, the alloys were exposed to these salts as well to confirm the literature assessment.

Our corrosion studies consisted of immersing coupons of the candidate alloys in the three molten salts at 900°C for up to 21 days, monitoring weight changes, and examining the coupons under a microscope after immersion. Metallographic examination and SEM examination of the metallographic sections were done using back scattered electron (BSE) imaging and chemical analysis using an energy dispersive x-ray (EDX) technique.

EXPERIMENTAL

Corrosion Apparatus

An illustration of a crucible used for exposing metals to molten salts is shown in Figure 1. The crucible was made of nominally 99.8% pure aluminum oxide and contained a volume of 300 cm³ of salt during testing; this resulted in a salt depth of about 19 cm. A number of different alloys were introduced into the crucible at the same time, and all of them were immersed in the salt. The salt temperature was maintained at 900°C, and the temperature gradient in the 19 cm depth of salt was about 4°C.

The top of the crucible was covered with a water-cooled plate, and a thermocouple and gas purge tube were suspended in the crucible from this plate. Mass flow controllers maintained a constant flow of purge gas to the crucibles.

*Incoloy is a registered trademark of the Inco family of companies.

Table I. Nominal Chemical Composition of Alloys Under Investigation
(The alloys are grouped by type)

| Alloy | C | Fe | Ni | Co | Cr | Mo | W | Si | Mn | Al | Ti | Others |
|-------------------------------|--------|-----|-------|-------|----|----|------|------|-------|-------|------|---|
| <u>Stainless Steel</u> | | | | | | | | | | | | |
| 304 | 0.08+* | 68 | 10 | - | 19 | - | - | 1+ | 2+ | - | - | S=0.03+, P=0.045+ |
| 316 | 0.08+ | 66 | 12 | - | 17 | 2 | - | 1+ | 2+ | - | - | S=0.03+, p=0.045+ |
| <u>Nickel</u> | | | | | | | | | | | | |
| | - | - | 99.99 | - | - | - | - | - | - | - | - | - |
| <u>Nickel-Chromium</u> | | | | | | | | | | | | |
| Cabot** alloy 600 | 0.08+ | 8 | 74 | 1+ | 15 | - | - | 0.5+ | 1+ | 0.35+ | 0.3+ | Cu=0.5+, S=0.015+ |
| Inconel*** alloy 600 | 0.15 | 8 | 75 | in Ni | 15 | - | - | 0.5+ | 1+ | - | - | Cu=0.5+, S=0.015+ |
| Inconel alloy X750 | 0.08+ | 7 | 72 | in Ni | 15 | - | - | 0.5+ | 1+ | 0.7 | 2.5 | S=0.01+, Cu=0.5+, Cb+Ta=1 |
| Cabot alloy 214 | - | 2.5 | 77 | - | 16 | - | - | - | - | 4.5 | - | Y=present |
| <u>Iron-Nickel-Chromium</u> | | | | | | | | | | | | |
| Cabot alloy 800H | 0.08 | 41 | 32 | 2+ | 21 | - | - | 1+ | 1.5+ | 0.4 | 0.4 | Cu=0.75+, S=0.015+ |
| Incoloy*** alloy 800 | 0.1+ | 43 | 32 | - | 21 | - | - | 1+ | 1.5+ | 0.4 | 0.4 | Cu=0.75+, S=0.015+ |
| Haynes** alloy 556 | 0.1 | 29 | 20 | 20 | 22 | 3 | 3 | 0.4 | 1 | 0.2 | - | Cb+Ta-1, La=0.2, N=0.2, Zr=0.02 |
| <u>High Refractory Metals</u> | | | | | | | | | | | | |
| <u>Content</u> | | | | | | | | | | | | |
| Hastelloy** alloy X | 0.1 | 18 | 47 | 1.5 | 22 | 9 | 0.6 | 1+ | 1+ | - | - | B=0.01+, P=0.04+, S=0.03+ |
| Hastelloy alloy N | 0.06 | 5+ | 71 | 0.2+ | 7 | 17 | 0.5+ | - | - | 0.3+ | 0.3+ | B=0.01+, Cu=0.035+ |
| Hastelloy alloy S | 0.02+ | 3+ | 63 | 2+ | 16 | 15 | - | 0.4 | 0.6 | 0.3 | - | La=0.06, B=0.015+ |
| Haynes alloy 230 | 0.1 | 3+ | 55 | 3+ | 22 | 2 | 14 | 0.5 | 0.7 | 0.3 | - | B=0.015+, La=0.05 |
| Cabot alloy R-41 | 0.08 | 5+ | 50 | 11 | 19 | 10 | - | 0.5+ | 0.1+ | 1.5 | 3.1 | B=0.006, S=0.015+ |
| Haynes alloy 188 | 0.1 | 3+ | 22 | 38 | 22 | - | 14 | 0.35 | 1.25+ | - | - | La=0.08, B=0.015+, P=0.02+, S=0.015+ |

** Indicates maximum.

**Cabot, Haynes, and Hastelloy are registered trademarks of the Cabot Corporation.

***Inconel and Incoloy are registered trademarks of the Inco family of companies.

Coupons

The metal coupons were 1.3×1.9 cm and were typically about 0.3 cm thick. Holes of about 0.4 cm diameter were made in the coupons so we could mount them on 99.8% pure aluminum oxide sample holders using spacers of this ceramic to separate the coupons. The coupons were cleaned, weighed, and measured before testing.

Corrosion Test Procedure

One coupon of each alloy was exposed to each molten salt. Each coupon was removed after two or three days, after about one week, and after about three weeks for weighing and examination. After cleaning salt from the coupons in distilled water, rinsing in solvent and drying (without removing the corrosion scale), we weighed the coupons and reported any weight changes as mg/cm^2 of coupon surface area.

Molten Salt Chemistry

We used a binary eutectic sodium-potassium carbonate for these experiments (58 mol % Na_2CO_3). The salt was prepared using reagent grade chemicals. The space above the molten salt was purged with dry nitrogen containing CO_2 gas at $100 \text{ cm}^3/\text{min}$; however, atmospheric air also entered the crucible during the experiments. The estimated CO_2 partial pressure was 0.1%, and the estimated O_2 partial pressure was about 1% to 10% based on mass spectrometric analysis of the gas above the salt in subsequent work (9).

We used reagent grade NaOH for the hydroxide tests and purged the space above the molten salt with $100 \text{ cm}^3/\text{min}$ of N_2 . Atmospheric air also entered the crucible in this experiment, and we estimated that the partial pressure of H_2O was about 0.1-1% and that of O_2 was about 1-10%.

The chloride experiments were conducted with ternary eutectic sodium-potassium-magnesium chloride (33 mol % NaCl , 45.4 mol % MgCl_2), and the salt was prepared with reagent grade chemicals. The space above the salt was purged at $100 \text{ cm}^3/\text{min}$ with dry N_2 , and, as with the other two salts, the atmospheric air that entered the crucible resulted in an O_2 partial pressure of 1-10% and an H_2O partial pressure of 0.1-1%.

RESULTS AND DISCUSSION

In this section we discuss the results of the exploratory corrosion studies on 16 alloys in molten carbonate, molten hydroxide, and molten chloride and observations made on the handling characteristics of the three molten salts. The data presented for the corrosion of the alloys include weight change and observations on the

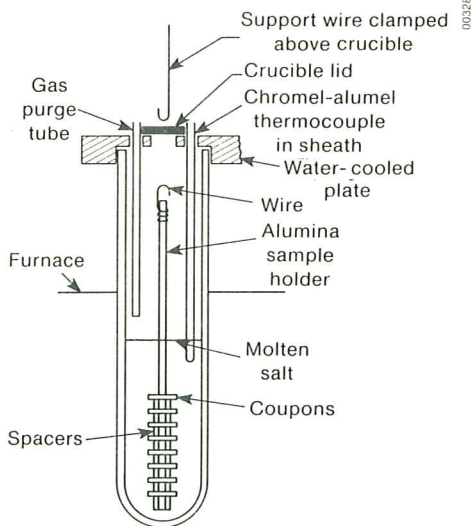


Figure 1 - Crucible used for conducting molten salt corrosion experiments.

condition of the coupons after testing. We present the loss of metal during exposure; this was determined using sectioned coupons to measure the amount of uncorroded metal. The structure of the corrosion product for the coupons tested in the carbonate is discussed on the basis of examination using back-scattered electron SEM and EDX analysis.

Exploratory Corrosion Studies of Alloys in Carbonate, Hydroxide, and Chloride Molten Salts

Table II shows the results of the exploratory corrosion experiments for the 16 commercial alloys after immersion at 900°C for 6, 3.5, and 21 days in molten eutectic sodium-potassium-magnesium chloride, molten sodium hydroxide, and molten eutectic sodium-potassium carbonate, respectively.

The results in Table II are in the form of weight change occurring per unit of coupon surface area, in mg/cm², with notes indicating the appearance of some of the coupons after exposure to the molten salt.

Carbonate Experiments. Six of the coupons tested in the carbonate salt, the ones not footnoted, were discolored because of exposure to the salt but showed no cracking and only minimal spalling of the corrosion product. These six alloys appeared to be quite resistant to attack by the salt. They were nickel, the two samples of alloy 600, Cabot 214, Haynes 556, and Hastelloy N.

These six along with the two samples of alloy 800, Hastelloy X, Inconel X750, and Haynes 188 also showed the least weight change of the coupons tested in the carbonate. The weight changes ranged from about -40 to 20 mg/cm². These weight changes were about one or two orders of magnitude higher than those reported by the General Electric Company for the corrosion of alloys at 650°C in a molten carbonate for a fuel cell application (10), where the metal loss rate was about 0.05 mm/yr. These weight changes were also about one or two orders of magnitude higher than those reported by Bradshaw (11) for the corrosion of 316 stainless steel and Incoloy 800 in molten nitrate salt at 600°C for use in central solar receiver systems, where the metal loss rate was about 0.02 mm/yr. These comparisons indicate that the metal loss rates for the alloys tested in our carbonate experiments would be one or two orders of magnitude higher than those results, or in the range from 0.2 to 5 mm/yr. Predicting corrosion rates based on comparisons from such dissimilar data is very speculative, however. The metal loss rates determined from the metallographic studies reported later in this section support the upper end of this range of corrosion rates.

Chloride and Hydroxide Experiments. As shown in Table II, eight of the alloys tested in the chloride and six of the alloys tested in the hydroxide disintegrated after 6 and 3.5 days in the respective molten salts. None of the coupons had disintegrated after 21 days in the carbonate salt. Coupons of each of the 16 alloys tested in the chloride salt for 19 days at 900°C had disintegrated, so we included the 6-day rather than the 19-day data in the table. Besides the disintegration of six of the coupons tested in the hydroxide, each of the remaining coupons, with the exception of Hastelloy S, exhibited a larger weight change in the hydroxide after 3.5 days than was observed in the carbonate for the same alloy after 21 days. Also, we found that each coupon tested in the hydroxide that did not disintegrate had a crack in it. In contrast, most of the coupons tested in the carbonate exhibited no cracking.

Table II. Corrosion of Alloys in Molten Salts at 900°C

| Alloy | Chloride (6 Days) | Hydroxide (3.5 Days) | Carbonate (21 Days) |
|--------------------|---|---|---|
| | Coupon Weight Change mg/cm ² | Coupon Weight Change mg/cm ² | Coupon Weight Change mg/cm ² |
| Stainless 304 | d* | d | -116** |
| Stainless 316 | d | d | -108** |
| Nickel 99.99 | d | -50*** | -29 |
| Cabot alloy 600 | -260 | 32*** | -25 |
| Inconel alloy 600 | -280 | -27*** | -5 |
| Inconel alloy X750 | d | 35*** | -34** |
| Cabot alloy 214 | -120 | 160*** | -38 |
| Cabot alloy 800H | -310 | 65*** | -24** |
| Incoloy alloy 800 | d | 60*** | 10** |
| Haynes alloy 556 | -250 | d | -18 |
| Hastelloy alloy X | d | 22*** | 21** |
| Hastelloy alloy N | d | Fractured | -14 |
| Hastelloy alloy S | -400 | 4*** | -111** |
| Haynes alloy 230 | -300 | d | -62** |
| Cabot alloy R41 | -150 | d | -213** |
| Haynes alloy 188 | d | d | -34** |

*"d" indicates that the coupon disintegrated.

**Shown pronounced spalling of the corrosion product (Figure 2c), had cracks form in the corrosion product (Figures 2a and 2b), or had a combination of these.

***The coupons showed cracking during testing (Figure 2a).

Appearance of the Coupons. An example of this cracking is seen in Figure 2a for the coupon of Cabot 214 tested at 900°C in the hydroxide. The figure shows that the most severe cracking occurred in the vicinity of the hole used to mount the coupon on the alumina sample holder, which is typical for the hydroxide tests. Also, the corrosion product on all of the coupons in all of the salts had a different appearance in the area near the hole than on the rest of the coupon. This is probably caused by crevice corrosion conditions in that area, since the molten salt had limited access to the area beneath the alumina space separating the coupon from its neighbors on the sample holder (see Figure 1).



Figure 2 - Coupons after testing in molten salt at 900°C, (a) Cabot 214 in hydroxide for 3.5 days, (b) Hastelloy S in carbonate for 21 days, and (c) Hastelloy X in carbonate for 21 days.

An example of cracking that leads to delamination of the coupon is shown in Figure 2b. This was observed for a Hastelloy S coupon tested in the carbonate salt for 21 days at 900°C. Several other coupons tested in the carbonate exhibited similar cracking; however, the Hastelloy S was the most extreme example. A number of coupons--tested in the carbonate showed spalling of the corrosion product, as shown for Hastelloy X in Figure 2c. As you can see, the upper left and central portions of the coupon have experienced spalling of the corrosion product. In this coupon, as in the one in Figure 2a, the effect of the spacer on the periphery of the hole is apparent.

Summary. The comparison of the corrosion results for the alloys in the three salts done in the preceding discussion pointed out that coupons tested in the carbonate showed less weight change, less cracking, and less disintegration than those observed in the chloride and hydroxide. For these reasons and because the carbonate salt showed fewer problems in handling, we confined our characterization of the metal loss and the structure and composition of the corrosion products to the coupons tested in the carbonate salt.

Handling of the Carbonate, Chloride, and Hydroxide Molten Salts

One goal of these exploratory experiments was to gain experience with the handling characteristics of the candidate molten salts. During the corrosion tests in the chloride salt, we observed a continual evolution of fumes, presumably HCl that was generated by the reaction between the chloride salt and moisture from the atmosphere above the salt. This fuming caused corrosion of several of the metal parts in the vicinity of the mouth of the crucible. This made it clear that one must eliminate moisture from the molten chloride or, alternatively, keep the fumes from contacting the metal structures in the vicinity of the salt.

The hydroxide salt crept out of its crucible if the lip of the crucible was higher than 150° to 200°C, even though the melting point of the salt was 318°C. Also, the salt readily wetted any high temperature surfaces that were in contact with it. In contrast, experiments on ternary eutectic lithium-sodium-potassium carbonate, which has a melting point of 397°C, showed no signs of this creeping. Also, while the carbonate salt generated a small amount of a white condensable fume, there was no observable corrosion of the metal parts in the vicinity of the mouth of the crucible. The carbonate was easier to work with than either the chloride or the hydroxide.

Metal Loss in the Carbonate Experiments

The weight changes on the coupons tested in the molten eutectic sodium-potassium carbonate at 900°C for 21 days combined with the condition of the coupons observed after testing establish, qualitatively, the extent of the interaction between the salt and the coupons. However, the weight change observed in corrosion experiments can be weight gain caused by oxidation and dissolution of alloy components by the molten salt. Thus, metal loss can be computed from the weight change information only in the case where one weight change mechanism is dominant and the weight change is not the result of several processes that make offsetting contributions. In corrosion situations where intergranular attack occurs, a considerable depth of corrosive attack can be accompanied by very little weight change.

We know that the coupons tested in our carbonate experiments experienced weight gains because of oxidation, as evidenced by the dark oxide scale observed on the coupons after testing and by the net weight gain of two of the coupons, Hastelloy X and Incoloy 800, as shown in Table II. Weight losses caused by spalling of the corrosion product were also observed in our experiments, as shown in Figure 2c for Hastelloy X and in varying degrees for the other 15 alloys evaluated. We also observed in the carbonate experiments that the molten salt took on a yellowish discoloration during testing that we interpret as evidence of weight loss caused by alloy ingredients dissolving from the coupons. Chromium as the chromate ion reportedly dissolves in the carbonate (12). Molybdenum and tungsten that were in some of the alloys are expected to behave similarly to the chromium since they are in the same group (VIB) of the periodic table. These alloy ingredients are known to form molybdates and tungstates that are analogous to the chromates (13).

Since at least three weight change mechanisms are operative in our carbonate experiments, the metal loss for the coupons cannot be determined from the weight change measurements.

Metal Loss Measurements

We determined the metal loss for the coupons in our carbonate experiments by measuring metallographic sections of the corroded coupons with an optical micrograph. The results of these measurements are shown in Table III where the alloys are listed in the order of the least metal loss for the coupon to the most. The total depth of corrosive attack is computed from half of the difference between the original thickness of the coupon before it was exposed to the molten carbonate and the thickness of the unaffected metal that was measured with the optical micrograph. Also included in Table III are the results of the weight change measurements and observations from Table II. Photographs of representative metallographic sections are shown in Figure 3.

Table III shows that the coupon of Hastelloy X had a metal loss of 0.12 mm/side when exposed to the carbonate for 21 days at 900°C. If the rate of metal loss is linear, then the alloy would undergo an attack of about 2 mm/yr per side. The spalling of the corrosion product observed for Hastelloy X and shown in Figure 2c supports the notion of a nonprotective scale that could result in a linear rate of metal loss. However, an extrapolation from this result at 21 days to the annual corrosion rate is highly speculative, and more detailed studies at longer exposure times would be required to determine a reliable value for the annual corrosion rate.

The Hastelloy S coupon was the most corroded one; the metal was completely consumed during the 21 day exposure, and the coupon was cracked along its centerline after the test, as shown in Figure 2b.

Metallographic Sections. The metallographic section for the Hastelloy S coupon is shown in Figure 3a, where the crack along the centerline in Figure 2b appears along the bottom of this figure. In addition to this center crack several other cracks are seen, suggesting that cracks are readily formed in the corrosion product. Cracks were frequently seen in the metallographic sections of the other alloys tested in the carbonate, and these cracks were in the corrosion product.

One coupon, nickel, has intergranular oxidation in the metal. The

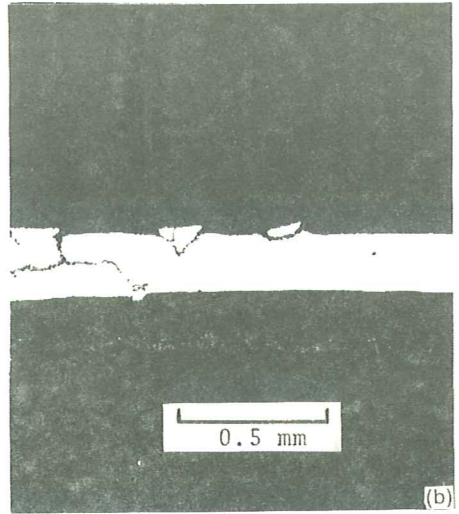
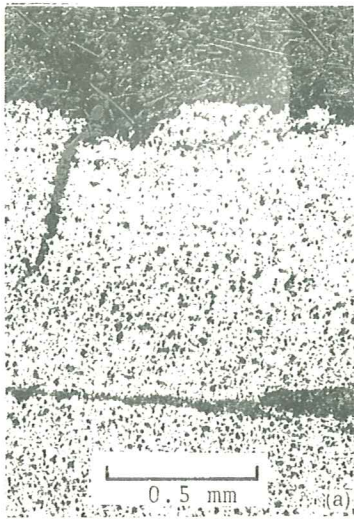


Figure 3 - Metallographic sections of coupons after 21 days at 900°C in molten carbonate, (a) Hastelloy S and (b) nickel.

900°C is shown in Figure 3b. The corrosion product on this coupon is very dark, presumably NiO, and the metal phase in the center of the coupon has intergranular oxidation. As seen in Table III, if the intergranular oxidation is not considered a part of the depth of corrosion, the nickel would have a total depth of corrosive attack of 0.19 mm and would be one of the best performers in this test.

Corrosion Rates. If the corrosion rate observed for the 21 days in this test is linearly extrapolated to a year, then a 6-mm starting thickness of Hastelloy X corroding at 2 mm/yr would be reduced to 2 mm of unaffected metal after a year. The other alloys evaluated in this test showed more attack after 21 days than the Hastelloy X. If the depth of attack for Cabot 800H, for example, is linearly extrapolated to a year and the same criterion is used (2 mm of unaffected metal remaining and 6 mm starting thickness) the projected service life would be half that for the Hastelloy X or about six months. Stated as an annual corrosion rate, the value for Cabot 800H would be about 4 mm/yr.

Using linear extrapolation may be unjustified, especially for the alloys where there was only minimal spalling and cracking of the corrosion product. As seen in Table III, Cabot 214, Haynes 556, nickel, alloy 600, and Hastelloy N all were relatively free of cracking and spalling and would be more likely than the other alloys to have longer service lives than predicted on the basis of linear extrapolation from the 21-day data. These alloys along with Hastelloy X, Inconel X750, Haynes 188, and the two samples of alloy 800 all show promise in our tests of service lives ranging from a few months to a year at 900°C in the carbonate salt. As an annual corrosion rate, this translates to several millimeters per year; however, a reliable value would require more detailed studies using longer exposure times.

Table III. Results of Corrosion Tests Performed in Eutectic Sodium-Potassium Carbonate at 900°C for 21 Days

| Alloy | Original Sample Thickness (mm) | Thickness of Unaffected Metal (mm) | Total Depth of Corrosive Attack (mm/side)* | Weight Change (mg/cm ²) |
|-----------------------|--------------------------------|------------------------------------|--|-------------------------------------|
| Hastelloy alloy X | 1.22 | 0.97 | 0.12 | 21 [¶] |
| Cabot alloy 214 | 1.98 | 1.60 | 0.19 | -38 |
| Haynes alloy 188 | 3.18 | 2.74 | 0.22 | -34 [¶] |
| Cabot alloy 800H | 3.15 | 2.64 | 0.25 | -24 [¶] |
| Haynes alloy 556 | 1.09 | 0.56 | 0.26 | -18 |
| Inconel alloy X750 | 1.12 | 0.58 | 0.27 | -34 [¶] |
| Nickel | 0.61 | (0.23)** | >0.30(0.19)*** | -29 |
| Cabot alloy 600 | 3.51 | 2.82 | 0.34 | -25 |
| Cabot alloy R-41 | 1.27 | 0.43 | 0.42 | -213 [¶] |
| Inconel alloy 600 | 1.17 | 0.28 | 0.44 | -5 |
| Hastelloy alloy N | 1.30 | 0.28 | 0.51 | -14 |
| Type 304SS | 1.50 | 0.41 | 0.54 | -116 [¶] |
| Type 316SS | 1.55 | 0.28 | 0.63 | -108 [¶] |
| Haynes Dev. alloy 230 | 4.65 | 3.10 | 0.77 | -62 [¶] |
| Incoloy alloy 800 | 1.60 | Completely Consumed | >0.8 | 10 [¶] |
| Hastelloy alloy S | 2.87 | Completely Consumed | >1.43 | -111 [¶] |

*All alloys except nickel mainly suffered metal wastage; nickel suffered metal wastage and intergranular attack.

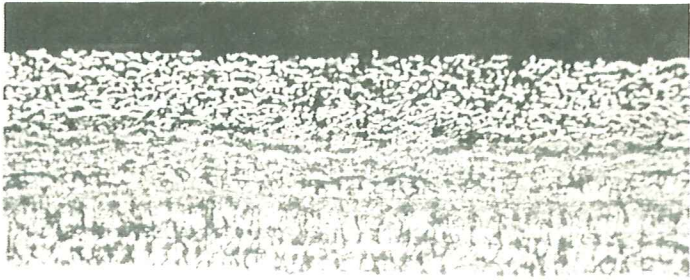
**Sample was completely penetrated by metal wastage and intergranular attack. The unaffected metal thickness was 0.23 mm when metal wastage only is considered.

***Metal wastage was 0.20 mm/side; intergranular attack was 0.11 mm/side.

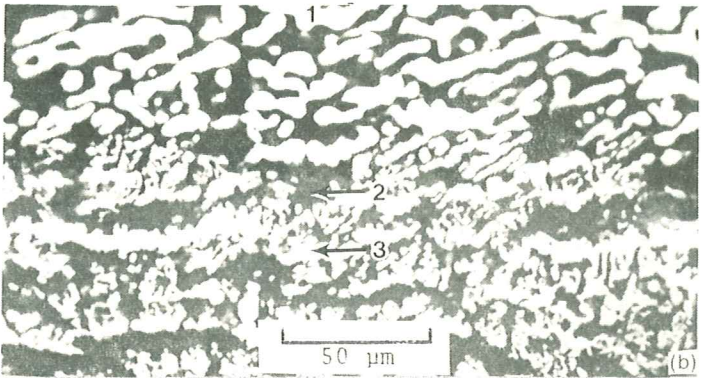
¶Showed pronounced spalling of the corrosion product (Figure 2c), had cracks form in the corrosion product (Figures 2a and 2b), or had a combination of these.

Examination of the Corrosion Product

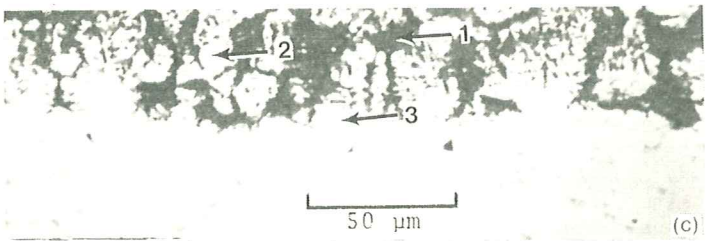
We studied the corrosion product using the metallographic section of typical alloy after exposure to the carbonate at 900°C for 21 days. We chose Inconel X750 using back-scattered electron SEM and EDX analysis to study it. The results are shown in Figure 4, where Figure 4a shows an overall view of the corrosion product above the unaffected metal and Figures 4b and 4c show the corrosion product near the surface of the coupon.



0.1 mm (a)



| Area | Ni | Fe | Cr | Ti | Ca | Na | K |
|------|------|------|------|-----|------|----|-----|
| 1 | 98.0 | 2.0 | -- | -- | -- | -- | -- |
| 2 | 15.6 | 21.6 | 42.5 | 8.4 | 10.9 | -- | 1.0 |
| 3 | 90.5 | 3.1 | 3.7 | 0.4 | 2.3 | -- | -- |



| Area | Ni | Fe | Cr | Ti | Al | Si | Ca | Na | K |
|------|------|-----|------|-----|-----|-----|----|----|-----|
| 1 | 7.7 | 0.5 | 71.8 | 9.5 | 1.1 | 2.9 | -- | -- | 6.5 |
| 2 | 89.5 | 3.4 | 5.7 | 1.4 | -- | -- | -- | -- | -- |
| 3 | 85.2 | 4.6 | 8.5 | 1.7 | -- | -- | -- | -- | -- |

Figure 4 - Back-scattered electron SEM micrographs of the corrosion product on a coupon of Inconel X750 after 21 days in molten carbonate at 900°C. (a) overview of the corrosion product, (b) close-up of the corrosion product near the surface, and (c) close-up of the corrosion product adjacent to the unaffected metal. Analysis of the locations marked in the figures are given beneath (b) and (c) in relative intensity by EDX.

and adjacent to the unaffected metal, respectively. Several areas shown in Figures 4b and 4c were analyzed by EDX to give their elemental composition; the results are included in the figures.

Figure 4b shows that the corrosion product near the surface of the coupon at the top has light and dark areas. The elemental analysis of the light area marked "1" shows that it is primarily nickel with some iron; these light areas are metallic. The dark adjacent areas are voids where salt was leached from the coupon during an aqueous cleaning procedure. An evaluation of the composition of the metallic areas in Figures 4b and 4c shows that the intensity of nickel is a minimum at area 3 in Figure 4c, which is at the start of the unaffected metal. Metallic areas that are successively closer to the surface of the coupon have continuously increasing nickel intensity. In contrast, the intensities of chromium, iron, and titanium, the other major ingredients of the Inconel X750, continuously decrease in the metallic areas that are successively closer to the surface of the coupon. This suggests that these elements are diffusing out of the metallic areas.

At a depth of a little over 0.1 mm from the surface of the coupon the voids adjacent to the metallic areas give way to grey areas, as seen in Figures 4a and 4b. These grey areas appear to be oxide, and we interpret the elemental analysis of these areas as showing a mixture of residual carbonate salt and oxidized elements from the Inconel X750. These oxide areas show large amounts of elements from the alloy. The oxide areas also contain potassium from the salt (although sodium from the salt was not detected), silicon, and aluminum, which are present in small quantities in the alloy. Calcium was detected in one metallic area and in one oxide area. It may have come from the salt where it was present as an impurity at a concentration of 0.01 wt %.

These results indicate that the corrosion product is an interpenetrating structure, consisting of metal from which chromium, titanium, and iron are diffusing and oxide made up of the reaction product of salt components and oxidized elements from the alloy. The porous metallic portion of the corrosion product presumably allows the oxides to leach and salt components to reach the unaffected metal.

CONCLUSIONS

These exploratory corrosion experiments show that many of the commercially available alloys, such as Hastelloy X, Cabot 214, Haynes 188, alloys 600 and 800, Haynes 556, Inconel X750, and nickel, significantly resist corrosion in molten eutectic sodium-potassium carbonate at 900°C. Although more extensive and detailed studies should be done in the carbonate to qualify any of these alloys for a specific application, these results indicate that some of them might corrode at a rate of a few millimeters per year.

Corrosion rates in molten eutectic sodium-potassium-magnesium chloride and sodium hydroxide at 900°C were much higher than in the carbonate for all of the alloys investigated. These salts also presented more handling problems than the carbonate.

The mechanism of attack by the carbonate in all of the alloys included oxidation/dissolution of some alloy ingredients by the salt, particularly

chromium and titanium, leaving behind more noble ingredients, primarily nickel, in a porous structure that allowed salt to penetrate to the interior of the test coupon. The notable exception was that nickel, in addition to a porous corrosion product, also exhibited intergranular attack.

ACKNOWLEDGMENT

This work was partially funded by the Energy Storage Division of the U.S. Department of Energy under contract no. DE-AC02-83CH10093.

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