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METALS AND CERAMICS DIVISION

COMPATIBILITY OF FUSED SODIUM FLUOROBORATES AND BF_3 GAS

WITH HASTELLOY N ALLOYS

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CONTENTS

COMPATIBILITY WITH FUSED SODIUM FLUOROBORATES AND
BF$_3$ GAS OF HASTELLOY N ALLOYS

J. W. Koger and A. P. Litman[1]

ABSTRACT

Corrosion by NaBF$_4$-NaF mixtures (4–8 mole % NaF) low in
oxygen and water was studied with 6800-hr static tests at
605°C in standard Hastelloy N capsules with titanium-modified
Hastelloy N specimens. Boron trifluoride gas was added to
overpressures up to 400 psig. Specimens in the molten salts
and at the interface showed both weight gains and losses. The
gains decreased with increasing NaBF$_4$ content, and losses up
to 0.03 mg/cm^2 (< 0.01 mil/year) were observed for specimens
in 4 mole % NaF. Weight changes of specimens in vapor were
very small and independent of overpressure. The main reaction
was the oxidation of chromium in the alloy by iron fluoride in
the salt to form Na$_3$CrF$_6$ and deposit iron on both specimens
and capsule. Metallographic examination showed only minor
attack and no difference in the two Hastelloy N alloys.

The rate of chromium depletion at 605°C was consistent
with the rate of solid-state diffusion of chromium to the
alloy surface. Additional tests from 538 to 760°C showed
Arrhenius behavior that confirmed this mechanism.

Fluoroborate salt mixtures were found compatible with
Hastelloy N alloys under the conditions of these experiments,
and comparison with other experiments showed that increases
in water and oxygen content increased the chromium uptake
and corrosion.

INTRODUCTION

The successful use of molten fluoride salts in a reactor system, as
demonstrated by the Molten Salt Reactor Experiment (MSRE) at the Oak
Ridge National Laboratory, has led to development studies for a Molten
Salt Breeder Reactor (MSBR). One of the most promising features of
molten salts has been the low corrosion rates experienced with materials
of construction, principally nickel-base alloys.

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[1]Now with AEC Division of Space Nuclear Systems, Washington, D.C.

Theory and experiment[2] have shown that the corrosion resistance of metals to molten fluoride salts varies directly with the "nobility" of the metal; that is, the resistance to attack varies inversely with the magnitude of the free energy of formation of the fluorides of metals in the container material. Therefore, the container alloy for the fluoride salt must have only a small percentage of constituents that are easily oxidized by the components of the salt. Considering these facts and utilizing information gained in corrosion testing of commercial alloys, ORNL developed[3] a high-strength nickel-base alloy, Hastelloy N, containing 17% Mo, 7% Cr, and 5% Fe, for use in the MSRE. These early experiments, as well as many recent corrosion tests[4-6] in $LiF-BeF_2-ThF_4$, $LiF-BeF_2-UF_4$, and $LiF-BeF_2-UF_4-ThF_4$, have shown that Hastelloy N is very resistant to attack and corrosion rates are below 0.1 mil/year.

Recently the selection of a fluoride salt that can be used as a coolant to transfer heat from the fuel salt to a steam power conversion system has been seriously considered. Cost and melting point considerations favor the sodium fluoroborate mixture, $NaBF_4$–8 mole % NaF. However, little information exists on corrosion by fluoroborates. Initial corrosion results were obtained from an impure fluoroborate salt (high in oxygen and water) in thermal convection loop tests,[7] but these results were not considered indicative of behavior with purer salt. Since that time, new methods of preparation have greatly increased the purity of available salt with respect to oxygen and water.

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[2]J. H. DeVan and R. B. Evans III, Corrosion Behavior of Reactor Materials in Fluoride Salt Mixtures, ORNL-TM-328 (Sept. 19, 1962).

[3]W. D. Manly, J. H. Coobs, J. H. DeVan, D. A. Douglas, H. Inouye, P. Patriarca, T. K. Roche, and J. L. Scott, "Metallurgical Problems in Molten Fluoride Systems," Progr. Nucl. Energy Ser. IV 2, 164–179 (1960).

[4]J. W. Koger and A. P. Litman, MSR Program Semiann. Progr. Rept. Feb. 29, 1968, ORNL-4254, pp. 218–221.

[5]J. W. Koger and A. P. Litman, MSR Program Semiann. Progr. Rept. Aug. 31, 1968, ORNL-4344, pp. 257–264.

[6]J. W. Koger and A. P. Litman, MSR Program Semiann. Progr. Rept. Feb. 28, 1969, ORNL-4396, pp. 243–246.

[7]J. W. Koger and A. P. Litman, Compatibility of Hastelloy N and Croloy 9M with $NaBF_4-NaF-KBF_4$ (90-4-6 mole %) Fluoroborate Salt, ORNL-TM-2490 (April 1969).

The overall objective of this study was to determine the corrosion characteristics of pure (low oxygen and water) $NaBF_4$-NaF mixtures under isothermal conditions. Of particular concern was the temperature-dependent dissociation of the salt into liquid NaF and BF_3 gas. Thus, we first determined the effects of varying the concentration of our salt mixture and the BF_3 pressure over it in 6800-hr tests at 605°C. In a later series of experiments we determined the rate of chromium uptake by $NaBF_4$–8 mole % NaF between 538 and 760°C in 1200-hr tests.

EXPERIMENTAL METHOD AND MATERIALS

The capsule design used in studying the effect of salt composition and BF_3 overpressure (Series I) is pictured in Fig. 1. Each capsule contained three specimens of titanium-modified Hastelloy N, one located in the vapor space, one in the salt, and one at the liquid-vapor interface. The capsule was constructed of commercial Hastelloy N and was 2 in. in diameter × 6 in. high. The titanium-modified alloy is considered because of its superior radiation-resistant properties.
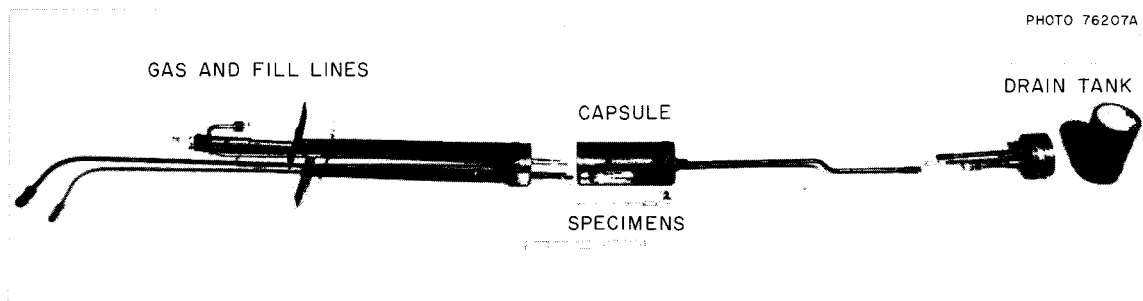
Fig. 1. Hastelloy N Capsule Assembly for Studying Effects of Salt Composition and BF_3 Overpressure.

The capsule used in studying the effect of temperature (Series II) was basically the same. However, the test specimens were commercial Hastelloy N in the form of 0.030-in.-thick strips to provide more surface area. Each capsule was 2 in. in diameter × 8.5 in. high and contained 16 specimens. The capsule is pictured in Fig. 2, and the test specimens are shown in Fig. 3. Table 1 lists the compositions of the alloys used for the capsules and specimens.

PHOTO 76436A

CAPSULE

DUMP TANK

HASTELLOY N
PLATES

GAS AND FILL LINES
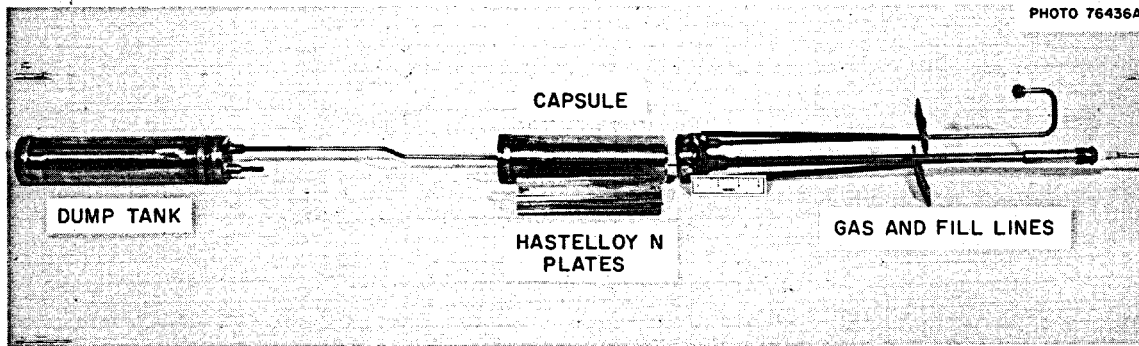
Fig. 2. Hastelloy N Capsule Assembly for Temperature Effect Study.

Photo 76435

INCHES

(a)

Photo 76434

INCHES
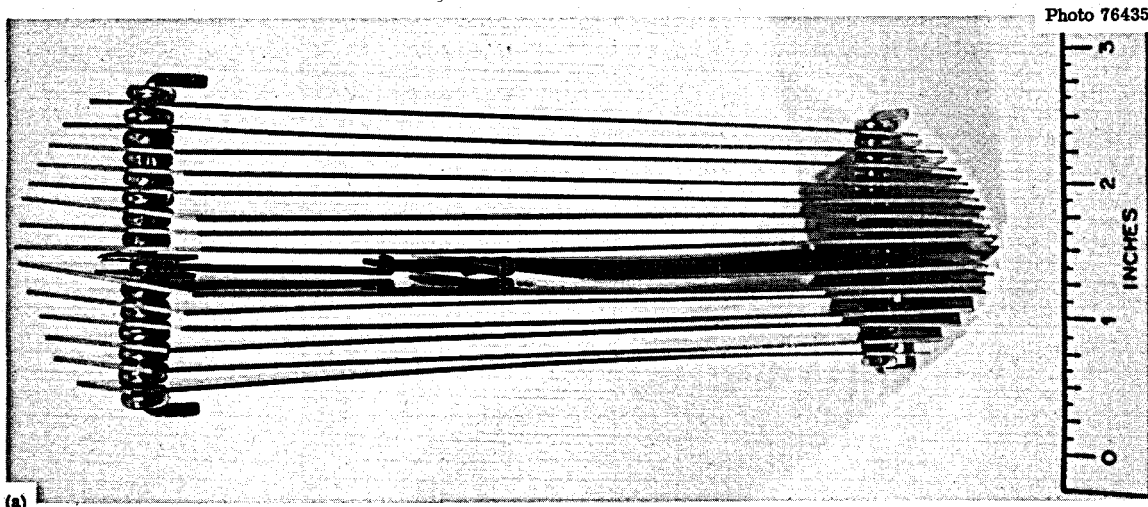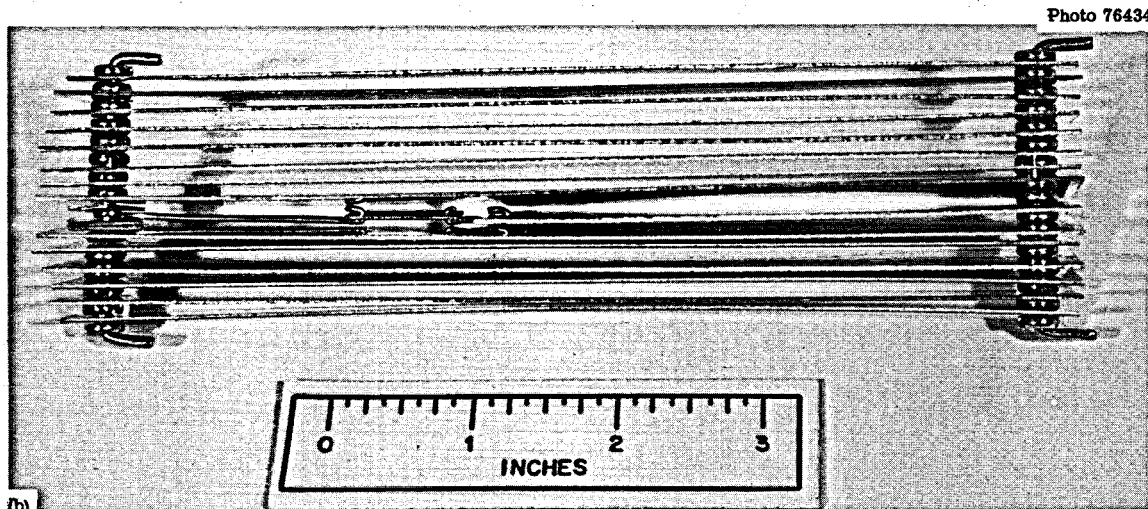
(b)

Fig. 3. Standard Hastelloy N Specimens Made to Provide Maximum
Surface Area for Study of Temperature Effects. (a) End view. (b) Side
view.

Table 1. Composition of Hastelloy N

Test Series	Component	Composition (wt %)[a]					
		Mo	Cr	Fe	Si	Mn	Ti
I	Capsules	15.8	7.4	2.4	0.2	0.3	0.02
	Specimens	12.0	7.3	< 0.1	< 0.01	0.14	0.5
II	Capsules and Specimens	17.0	7.2	4.3	0.45	0.5	0.02

[a]Balance nickel.

The salt for these tests was processed by the Fluoride Processing Group of the Reactor Chemistry Division. The very pure (> 99.9%) starting materials were evacuated to about 380 torr, heated to 150°C in a vessel lined with nickel, and then held for about 15 hr under these conditions. After the rise in vapor pressure was observed to be not excessive (indicating no volatile impurities), the salt was heated to 500°C while still under vacuum and agitated by bubbling helium through the capsule for a few hours. The salt was then transferred to the fill vessel and forced into the capsules with helium pressure. Salt chemistry is discussed under "Results" in this report.

All capsules were heated in vertical muffle furnaces (Fig. 4) and monitored with Chromel-P vs Alumel thermocouples, which had been spot welded to the capsule and covered with shim stock. In our first series of tests all capsules were operated at 605°C, close to the maximum temperature proposed for the coolant salt in the MSBR. Capsule 1 of this series contained a helium overpressure of 4 psig and no added BF_3. Capsules 2, 3, and 4 contained BF_3 corresponding to overpressures of 50, 100, and 400 psig, respectively. Initially the added BF_3 dissolved in the molten salt or combined with the NaF, but after a few minutes the pressure in the capsule gradually increased. However, when the capsules had been pressurized to the operating pressures, no changes in BF_3 content were necessary during the test. The compositions of the salts calculated from amounts of BF_3 added are shown in Table 2.
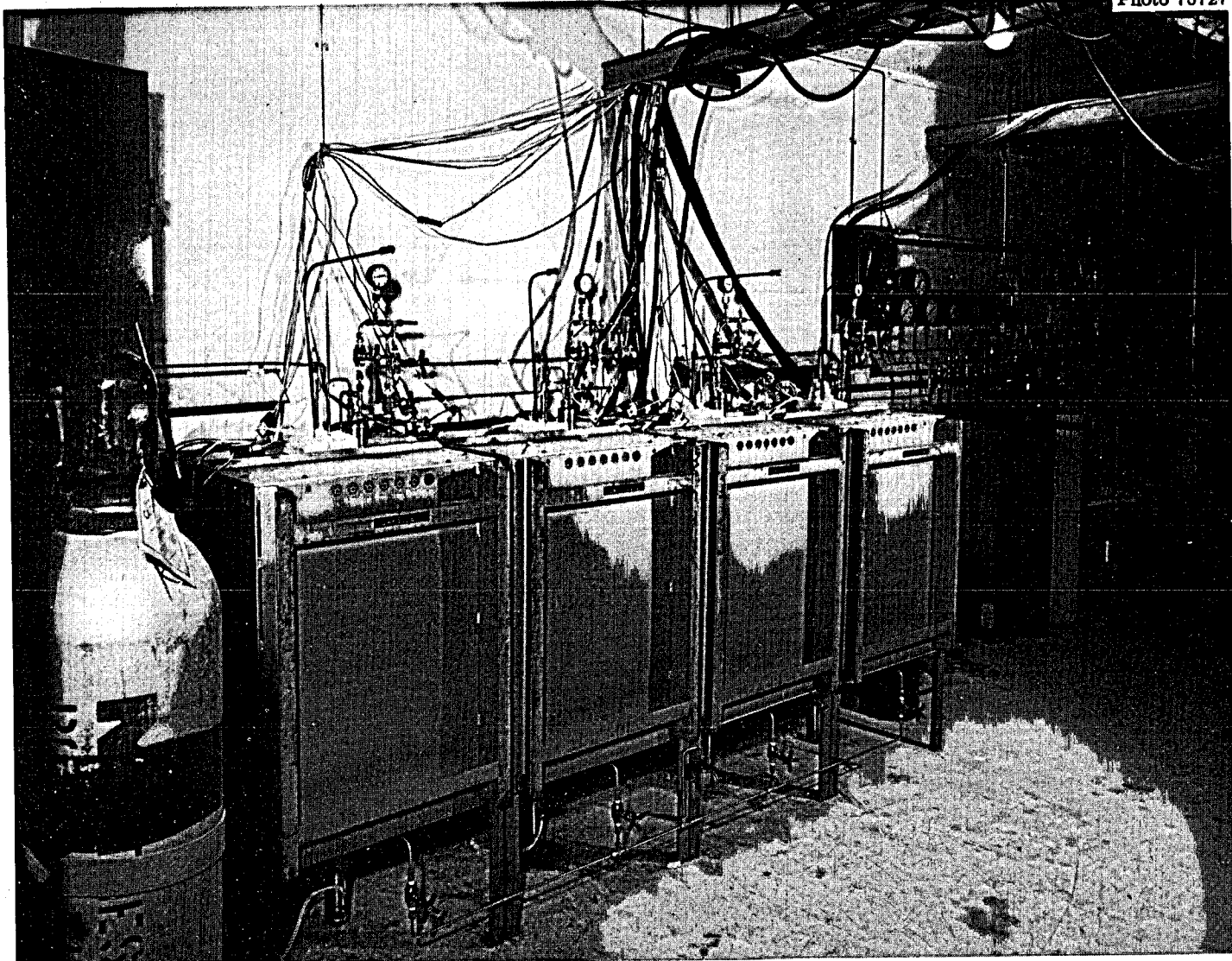
Fig. 4.   Furnace Assembly.

9

Table 2. Effect of Salt Composition and BF_3 Overpressure
on Weight Changes of Hastelloy N

| Capsule | BF_3 Overpressure (psig) | Salt Composition (mole %) | | Weight Change (mg/cm^2) | | |
		NaBF$_4$	NaF	Vapor	Interface	Liquid
1	0	92	8	−0.03	+0.5	+1.3
2	50	94	6	−0.3	+0.4	+1.2
3	100	95	5	−0.4	+0.2	+0.06
4	400	96	4	0.0	−0.03	−0.03

After 6800 hr the salt in each capsule was removed and sampled. The capsules were opened and the specimens were removed, washed in warm distilled water, dried with ethyl alcohol, and weighed. The specimens and capsules were examined metallographically and by x-ray fluorescence and microprobe analysis. The salts were analyzed before and after the tests.

In our second series of experiments, the capsules were operated at 427°C (800°F), 538°C (1000°F), 649°C (1200°F), and 760°C (1400°F) with 5 psig He overpressure and calculated equilibrium BF_3 pressures of 0.005, 0.07, 0.5, and 2.5 psia, respectively. After 1200 hr the salt in each capsule was removed and sampled for analysis. The salt had also been analyzed before test.

RESULTS

Effect of Salt Composition and BF_3 Pressure (Series I)

Weight Changes

The compositional changes in the salt effected by BF_3 gas additions in our first series of tests caused a measurable difference in the weight changes of our test specimens. As shown in Table 2, the weight changes were relatively small, and some specimens showed weight gains instead of the expected losses. There was no correlation between the pressure of BF_3 and the weight change of the specimens exposed to the vapor.

The specimens immersed in salt in capsules 1, 2, and 3 showed progressively decreasing weight gains. The salt specimen in capsule 4 showed a weight loss equivalent to a corrosion rate less than 0.01 mil/year. The same weight change pattern was observed for the specimens at the interface. Thus, the weight gain of specimens in the liquid and at the interface increased as the amount of $NaBF_4$ in the salt decreased.

Figure 5 shows the specimens after test. Very little corrosion was noted at any position on the specimens or capsules.
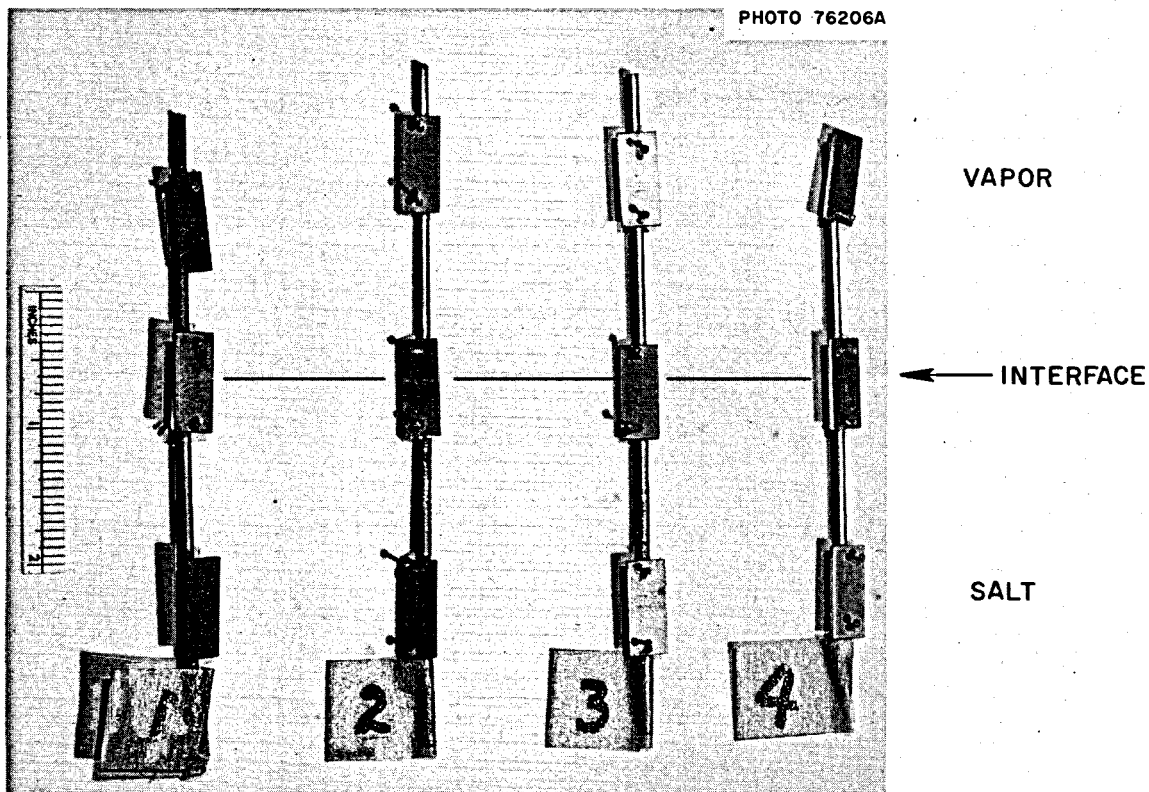
Fig. 5. Titanium-Modified Hastelloy N Specimens After Exposure to $NaBF_4$-8 mole % NaF at 605°C for 6800 hr. Numbers identify capsules.

Salt Chemistry

Chemical analyses of the salt before and after test for each capsule are given in Table 3. The most significant changes are an increase in chromium concentration and a decrease in iron concentration. No titanium concentrations are included in Table 3 because, as expected,

Table 3. Salt Analyses Before and After Test (Series I)

Element	Concentration				
	As Received	Capsule 1	Capsule 2	Capsule 3	Capsule 4
Weight Percent					
Na	21.9	21.4	21.0	21.8	21.0
B	9.57	9.54	9.48	9.48	9.49
F	68.2	69.2	68.5	68.7	68.8
Parts Per Million					
Cr	19	75	75	73	72
Ni	28	< 10	< 10	< 10	< 10
Fe	223	24	28	29	22
Mo	< 10	< 5	< 5	< 5	< 5
O	459	194	576	372	1420[a]
H_2O	400	400	460	350	460

[a]Questionable result.

no change was noted; only the test specimens contained titanium, and they constituted only a small part of the total system. The only change in the water and oxygen concentrations occurred in capsule 4. The high oxygen concentration reported for this capsule would normally have produced highly oxidizing conditions[8] and high corrosion rates. The reported value is believed to be in error because close examination of the system disclosed no leaks or signs of oxidation. Apparently some BF_3 escaped from the capsules after cooling and during opening, since the concentrations of sodium, boron, and fluorine in the salt were about the same in the final analysis of each capsule.

The increase in chromium and the decrease in iron in the salt suggest a reaction of the type

[8]H. E. McCoy, J. R. Weir, Jr., R. L. Beatty, W. H. Cook, C. R. Kennedy, A. P. Litman, R. E. Gehlbach, C. E. Sessions, and J. W. Koger, Materials for Molten-Salt Reactors, ORNL-TM-2511 (May 1969).

$$Cr(s) + FeF_2(d) \rightleftarrows CrF_2(d) + Fe(\text{deposited}) , \qquad (1)$$
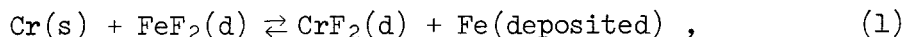
where (s) indicates solid solution in Hastelloy N and (d) indicates dissolved in salt. Any water or oxygen-type impurity in the salt would also have caused some oxidation of the container materials, but these impurities appear to have been negligible in this series of tests. Because the salt in these experiments contained NaF, the chromium fluoride in the corrosion product was found as Na_3CrF_6. This material has also been identified in other corrosion experiments[9] and its crystal structure has been determined by x-ray diffraction analysis.[10] Also, the iron fluoride in these experiments may have existed as Na_3FeF_6, but this compound was not identified.

Specimen Chemistry

The proposed corrosion reaction was verified further by x-ray fluorescence analysis of the specimens. Table 4 gives the composition of the near-surface region of a specimen from capsule 4. The composition was determined by comparing x-ray intensities with those of a specimen not exposed to salt or vapor. For any given region in the capsule, the qualitative results from all the specimens were quite similar. These results show a depletion of chromium and enrichment of iron in the near-surface region. All specimens also showed a loss of titanium. Microprobe analysis showed essentially the same surface changes. A chromium gradient was seen but was so small that we could not distinguish between edge effects and an actual gradient.

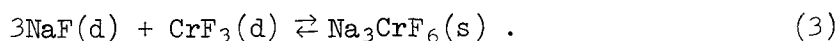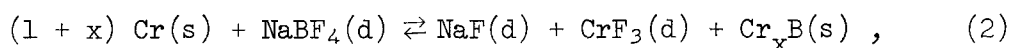According to Cantor,[11] $NaBF_4$–8 mole % NaF may also oxidize chromium by the reactions

[9]J. W. Koger and A. P. Litman, Compatibility of Hastelloy N and Croloy 9M with $NaBF_4$-NaF-KBF_4 (90-4-6 mole %) Fluoroborate Salt, ORNL-TM-2490 (April 1969).

[10]G. Brunton, "The Crystal Structure of Na_3CrF_6," Mater. Res. Bull. 4, 621–626 (1969).

[11]S. Cantor, MSR Program Semiann. Progr. Rept. Aug. 31, 1968, ORNL-4344, p. 160.

Table 4. Composition of Near-Surface Regions of Test Specimens
as Determined by X-Ray Fluorescent Analysis

Exposure Conditions	Concentration (wt %)				
	Mo	Ni	Fe	Cr	Ti
Unexposed	12.0	79.9	0.1	7.5	0.5
Vapor	12.3	79.2	0.13	7.1	0.19
Interface	13.7	69.6	13.4	3.0	0.2
Liquid	18.0	22.7	56.4	2.7	0.16

$$(1 + x) \ Cr(s) + NaBF_4(d) \rightleftarrows NaF(d) + CrF_3(d) + Cr_xB(s) \ , \qquad (2)$$

$$3NaF(d) + CrF_3(d) \rightleftarrows Na_3CrF_6(s) \ . \qquad (3)$$

However, no borides were identified either on our specimens or in the
salt, and the experimental evidence that iron replaced the chromium
indicates that reaction (1) rather than (2) was the predominant mode of
chromium oxidation. If essentially no iron fluoride compound were
present in the melt, reaction (2) could well control the corrosion
process.

Metallography

Figure 6 shows the microstructure of the specimen from the vapor
region of capsule 1. There was some indication of a very thin, discon-
tinuous deposit at the surface in the as-polished condition. After
etching, this deposit was no longer visible. Figure 7 shows the speci-
men that was exposed to salt in capsule 1. A deposit is visible on the
as-polished specimen. Etching again removed the deposit but in this
case produced considerable attack at the exposed surface. Metallographic
observation of the capsule wall showed similar behavior. This deposit is
apparently iron rich, and the area revealed by the etchant is chromium-
depleted Hastelloy N. Thus, no difference in the behavior of titanium-
modified and standard Hastelloy N was noted in this test. The interface
between the salt and gas could be seen on the capsule, but no attack was
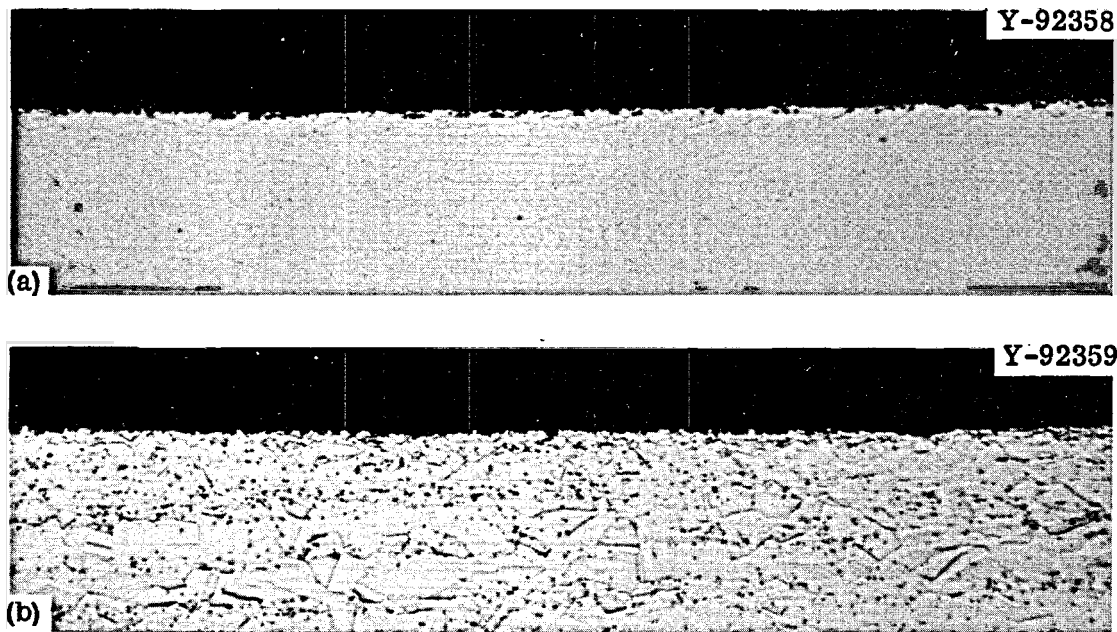noted there.

Fig. 6. Titanium-Modified Hastelloy N from Capsule 1 Exposed to BF$_3$ Vapor for 6800 hr at 605°C. Weight change, +0.03 mg/cm^2. 500×. (a) As polished. (b) Etched with glyceria regia.
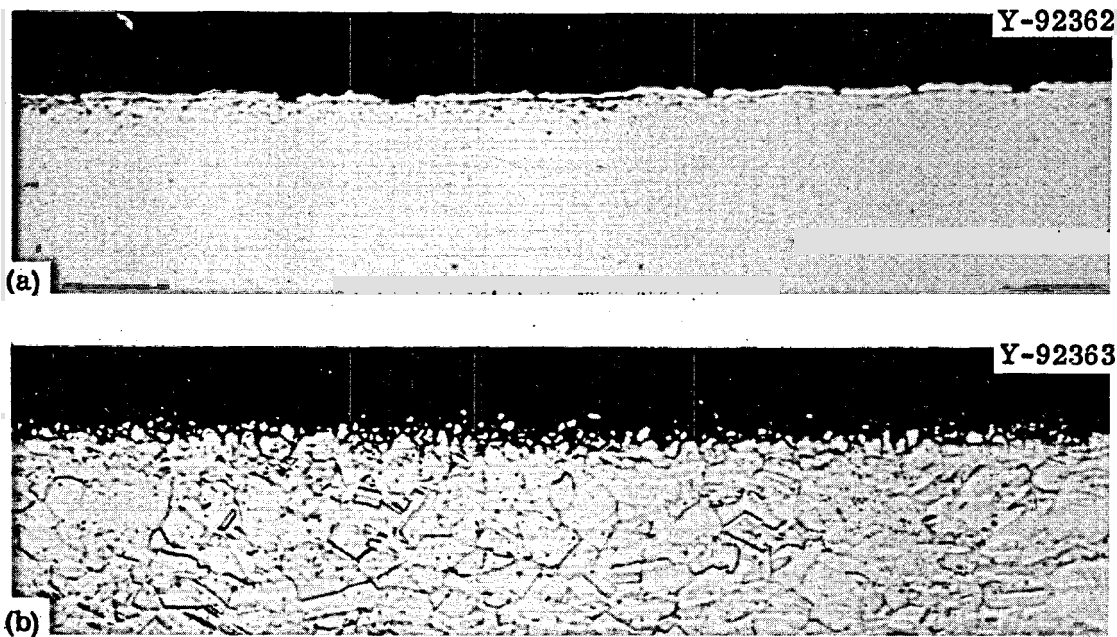

Fig. 7. Titanium-Modified Hastelloy N from Capsule 1 Exposed to NaBF$_4$-8 mole % NaF for 6800 hr at 605°C. Weight change, +1.3 mg/cm^2. 500×. (a) As polished. (b) Etched with glyceria regia.

Effect of Temperature (Series II)

Our second series of tests was designed to determine the effects of temperature on chromium mass transfer from the Hastelloy N to the salt. The salt was analyzed before and after test to determine impurity pickup. The NaBF$_4$–8 mole % NaF contained approximately 400 ppm each of oxygen and water before and after test. The chromium concentration in the salt increased while the iron decreased, as seen in Table 5. No changes in the concentration of nickel and molybdenum were noted. Again, the results suggest chromium oxidation by reduction of an iron fluoride compound. Metallography of Hastelloy N from this test showed, after etching, no attack of the specimen (Fig. 8).

Table 5. Analysis of Salt for Impurities Before and After Test (Series II)

Impurity	Average Concentration[a] (ppm)				
	As Received	Capsule 1	Capsule 2	Capsule 3	Capsule 4
Cr	15	45	87	225	575
Fe	200	190	180	150	40
O	460	430	450	412	550
H$_2$O	320	400	530	480	490

[a]Nickel and molybdenum were not detected (< 10 ppm) in any samples.

Fig. 8. Hastelloy N Exposed to NaBF$_4$–8 mole % NaF for 1200 hr at 760°C. Etched with glyceria regia, 500x.

INTERPRETATION OF CORROSION RATES

If the chromium surface concentration remains constant (only true if the salt contains an excess of iron) at any given point in the capsule, the amount of chromium leaving a capsule in Series I is given by the equation[12]

$$\Delta W = 2\rho(C_0 - C_s)\sqrt{Dt/\pi} \, , \tag{4}$$

where

ΔW = integral flux of Cr leaving metal, g/cm^2

ρ = density of metal, g/cm^3

C_0 = initial weight fraction of Cr

C_s = surface weight fraction of Cr

D = diffusion coefficient of Cr in alloy, cm^2/sec

t = exposure time, sec.

Using the following information

Salt in capsule = 282 g

Cr increase in salt = 55 ppm (by weight)

Capsule surface area = 142 cm^2

to obtain ΔW and then using

C_0 = 7.5%

C_s = 0 (consistent with excess of Fe)

ρ = 8.8 g/cm^3

t = 6800 hr,

we obtain

D_{calcd} = 3.3 × 10^{-15} cm^2/sec at 605°C.

This compares favorably with 3 × 10^{-15} cm^2/sec at 605°C extrapolated from the range 700 to 850°C from data[13] of DeVan and Evans for chromium

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[12]L. S. Darken and R. W. Gurry, Physical Chemistry of Metals, McGraw-Hill, New York, 1953.

[13]W. R. Grimes, G. M. Watson, J. H. DeVan, and R. B. Evans, "Radio-Tracer Techniques in the Study of Corrosion by Molten Fluorides," pp. 559–574 in Conference on the Use of Radioisotopes in the Physical Sciences and Industry, September 6–17, 1960, Proceedings, Vol. III, International Atomic Energy Agency, Vienna, 1962.

diffusion in Hastelloy N. From this comparison, we conclude that the controlling rate mechanism in this experiment was the solid-state diffusion of chromium in the alloy and that the oxidation potential of the salt was established by the presence of iron fluoride.

Figure 9 shows the variation of chromium content with test temperature for the experiments of Series II. Included for comparison are chromium concentrations measured in loop tests after 1200 hr at various H_2O impurity levels.[14-17] An Arrhenius-type relationship appears to

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[14]J. W. Koger and A. P. Litman, MSR Program Semiann. Progr. Rept. Feb. 29, 1968, ORNL-4254, pp. 218–221.

[15]J. W. Koger and A. P. Litman, MSR Program Semiann. Progr. Rept. Aug. 31, 1968, ORNL-4344, pp. 257–264.

[16]J. W. Koger and A. P. Litman, MSR Program Semiann. Progr. Rept. Feb. 28, 1969, ORNL-4396, pp. 243–246.

[17]J. W. Koger and A. P. Litman, Compatibility of Hastelloy N and Croloy 9M with $NaBF_4$-NaF-KBF_4 (90-4-6 mole %) Fluoroborate Salt, ORNL-TM-2490 (April 1969).
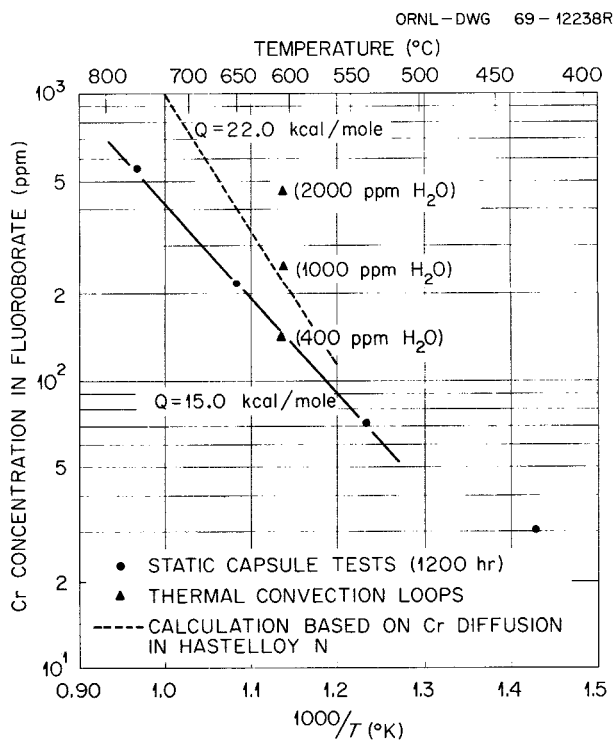

Fig. 9. Temperature Dependence of Chromium Concentration in Sodium Fluoroborate Salt.

hold between 538 and 760°C. This would be expected if the rate of chromium buildup in the salt were controlled by solid-state diffusion of chromium to the capsule wall. Using diffusion data for chromium in Hastelloy N obtained by DeVan and Evans[13] and assuming the chromium surface concentration to have been reduced to zero by the salt, we predict a chromium buildup shown by the dashed line in Fig. 9. The agreement between the slopes (activation energy) of the predicted and experimental curves, although not exact, gives credence to the assumption that chromium mass transfer to the salt is limited by solid-state diffusion of chromium to the Hastelloy N surface.

CONCLUSIONS

1. Titanium-modified Hastelloy N specimens were exposed for 6800 hr to isothermal $NaBF_4$-NaF salt mixtures containing only a few hundred parts per million oxygen and water and varying in composition from 4 to 8 mole % NaF. Specimens in the vapor region lost weight in all but the 96 mole % $NaBF_4$ mixture. Specimens fully immersed in the salt gained weight in all but the 96 mole % $NaBF_4$ mixture. The weight gains decreased as the $NaBF_4$ content of the salt increased.

2. No differences in the corrosion of specimens in the vapor region were noted as a function of BF_3 overpressure to 400 psig.

3. In the main corrosion reaction, chromium from the alloy was oxidized by iron fluoride impurities initially in the salt. The iron that was reduced deposited on the specimens and capsule.

4. Metallographic observation of both standard Hastelloy N capsules and titanium-modified Hastelloy N specimens showed very little attack, although a thin discontinuous deposit rich in iron was observed at the surface of specimens exposed to vapor.

5. A diffusion coefficient for chromium in Hastelloy N calculated from the rate of chromium removal from standard Hastelloy N by $NaBF_4$—8 mole % NaF held between 538 and 760°C agreed well with an extrapolation of published results. Thus, solid-state diffusion of chromium appeared to be the rate-controlling mechanism for corrosion in these tests.

6. Under the condition of these experiments, the fluoroborate
salt mixtures were compatible with the Hastelloy N alloys. By virtue
of other experiments we conclude that increases in water as impurity
increase the chromium uptake and the corrosion.

ACKNOWLEDGMENT

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