

CORROSION OF TYPE 304L STAINLESS STEEL
AND HASTELLOY N BY MIXTURES OF
BORON TRIFLUORIDE, AIR, AND ARGON

J. W. Koger

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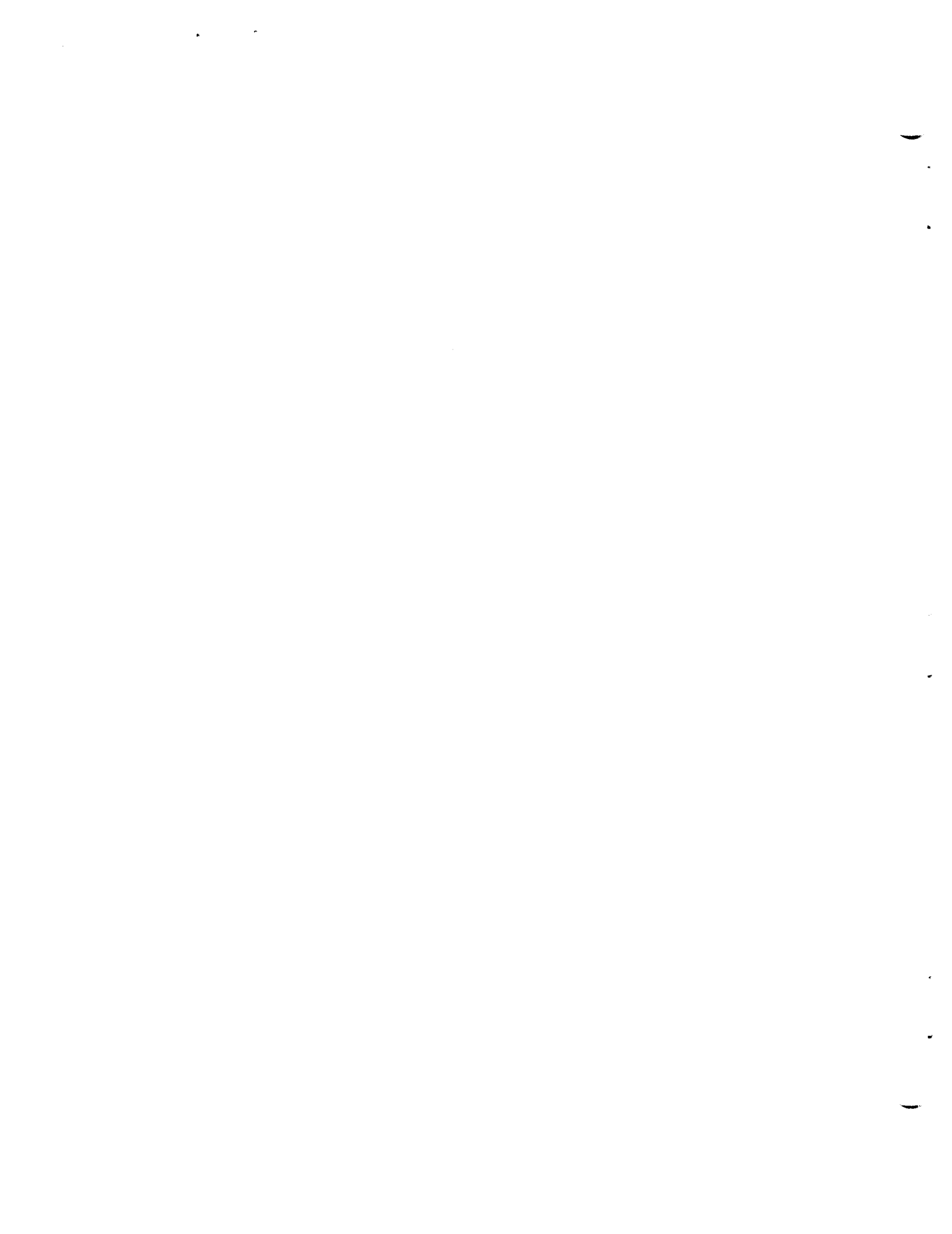
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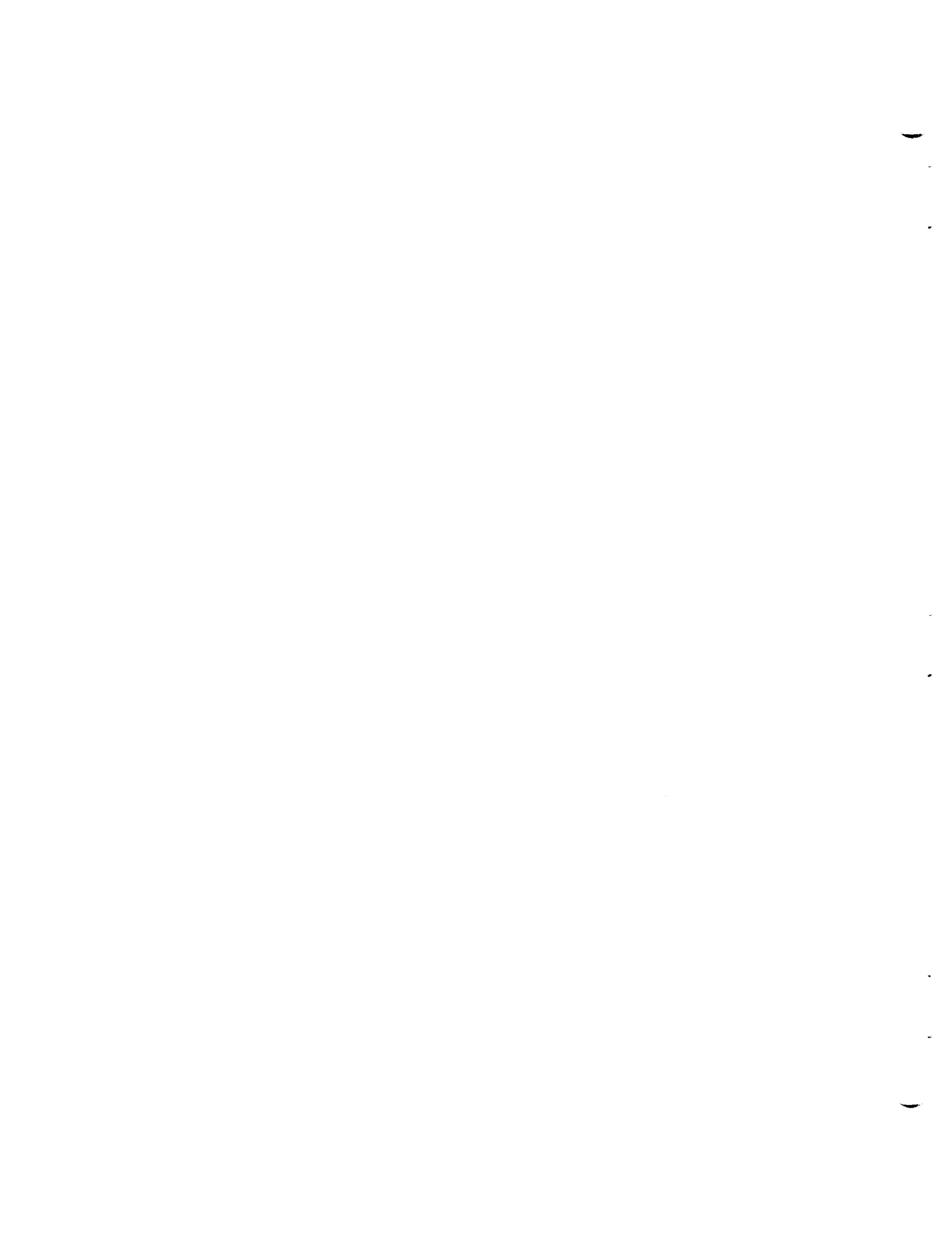
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CORROSION OF TYPE 304L STAINLESS STEEL AND HASTELLOY N BY
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ABSTRACT

Corrosion of type 304L stainless steel and Hastelloy N was studied in gaseous mixtures of BF_3 , air, and argon at 600, 300, and 200°C. In some tests the alloy specimens and gases were in contact with molten fluoride mixtures. At 600°C under air, specimens of both alloys were completely destroyed in both fuel salt and fluoroborate salt mixtures. No significant weight changes were measured for Hastelloy N immersed in salt exposed to argon or BF_3 , but type 304L stainless steel lost weight under the same conditions. This weight loss was greater under BF_3 than under argon and greater in the fluoroborate mixture than in the fuel salt. With no salt immersion, the various gases produced very small changes in the alloys at 200°C. Also with no salt, only small changes were noted at 300 and 600°C for Hastelloy N in any gas and for the stainless steel in air. At 600°C, large quantities of chromium and iron oxides were produced on the stainless steel by the air- BF_3 mixture and large amounts of unidentified corrosion products by BF_3 , argon- BF_3 , and argon-air. Drying the argon decreased the amount of corrosion. Hastelloy N was more resistant than type 304L stainless steel to corrosion by the gases tested in these experiments.

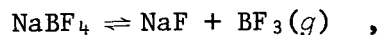
INTRODUCTION

Over the past few years, increased interest has been shown in the use of a sodium fluoroborate mixture, NaBF_4 -8 mole % NaF, as a coolant in a Molten-Salt Breeder Reactor. The physical properties of the salt mixture have been determined¹ and many experiments²⁻¹² have been undertaken to

¹S. Cantor, J. W. Cooke, A. S. Dworkin, G. O. Robbins, R. E. Thoma and G. M. Watson, *Physical Properties of Molten Salt Reactor Fuel*, ORNL-TM-2316 (August 1968).

²J. 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).

ascertain the corrosion of various alloys in this salt. The use of this mixture in a high-temperature environment is somewhat complicated by the fact that the NaBF_4 dissociates by the reaction



and the effect of the BF_3 gas must be considered. The equilibrium pressure above a melt of NaBF_4 -8 mole % NaF as a function of temperature is given by

$$\log P_{\text{torr}} = 9.024 - 5920/T(^{\circ}\text{K}) \quad .$$

The maximum design temperature for the coolant salt mixture is 621°C , and this corresponds to a partial pressure of BF_3 of 252 torr. Because of the appreciable vapor pressure of BF_3 at operating temperatures, cover gases

³J. W. Koger and A. P. Litman, *Catastrophic Corrosion of Type 304 Stainless Steel in a System Circulating Fused Sodium Fluoroborate*, ORNL-TM-2741 (January 1970).

⁴J. W. Koger and A. P. Litman, *Compatibility of Fused Sodium Fluoroborates and BF_3 Gas with Hastelloy N Alloys*, ORNL-TM-2978 (June 1970).

⁵J. W. Koger and A. P. Litman, *MSR Program Semiannu. Progr. Rep. Feb. 29, 1968*, ORNL-4254, pp. 221-25.

⁶J. W. Koger and A. P. Litman, *MSR Program Semiannu. Progr. Rep. Aug. 31, 1968*, ORNL-4344, pp. 264-66 and 285-89.

⁷J. W. Koger and A. P. Litman, *MSR Program Semiannu. Progr. Rep. Feb. 28, 1969*, ORNL-4369, pp. 246-53.

⁸J. W. Koger and A. P. Litman, *MSR Program Semiannu. Progr. Rep. Aug. 31, 1969*, ORNL-4449, pp. 200-208.

⁹J. W. Koger, *MSR Program Semiannu. Progr. Rep. Feb. 28, 1970*, ORNL-4548, pp. 242-52 and 265-72.

¹⁰J. W. Koger, *MSR Program Semiannu. Progr. Rep. Aug. 31, 1970*, ORNL-4622, pp. 168-78.

¹¹J. W. Koger, *MSR Program Semiannu. Progr. Rep. Feb. 28, 1971*, ORNL-4676, pp. 192-215.

¹²J. W. Koger, *MSR Program Semiannu. Progr. Rep. Aug. 31, 1971*, ORNL-4728, pp. 138-53.

containing equivalent concentrations of BF_3 must be maintained in the free volume of the pump bowl of dynamic systems or used for gas sparge operations.

BACKGROUND DATA

An early report on corrosion experiments with gaseous boron trifluoride¹³ noted that the BF_3 reacted rapidly with traces of moisture to give hydroxyfluoboric acid (HBF_3OH) and HF. It also showed that BF_3 and glass reacted at an appreciable rate just above 200°C . Under the conditions of those experiments, there was no appreciable attack by BF_3 on any metal or alloy examined at temperatures up to 200°C .

A systematic study of the compatibility of Hastelloy N with BF_3 had not been undertaken before this study, but several observations indicated possible compatibility problems. These observations were often made in the course of experiments where compatibility was not the primary objective, and the results are difficult to interpret. However, these miscellaneous observations do contribute to our understanding of metal- BF_3 -moisture reactions.

In one experimental program¹⁴ BF_3 contacted chromium metal (a constituent in almost all alloys considered for use in a molten salt system) 60 hr at 650°C . The chromium sample gained weight (about 4%), and the weight gain showed a linear dependence on the square root of the reaction time. The surface of the chromium sample contained substantial quantities of Cr_2O_3 and minor quantities of CrF_2 - CrF_3 . Thus, commercially available BF_3 did promote the oxidation of pure chromium.

In all Hastelloy N thermal convection loop tests⁵⁻¹² that have involved the fluoroborate mixture, specimens have been placed in the vapor

¹³F. Hudswell, J. S. Nairn, and K. L. Wilkinson, "Corrosion Experiments with Gaseous Boron Trifluoride" *J. Appl. Chem.* 1: 333-36 (1951).

¹⁴J. H. Shaffer and H. F. McDuffie, *MSR Program Semiannu. Progr. Rep. Aug. 31, 1967*, ORNL-4191, pp. 163-64.

space above the hot and cold leg surge tanks to provide some data on the compatibility of BF_3 with Hastelloy N. The vapor space contained about 220 torr BF_3 and 5 psig He. Since the mass transfer rates in these systems were quite dependent on salt purity, which varied from loop to loop, the weight changes of the specimens exposed to the vapor (Table 1) are scattered. However, in none of the cases were the losses excessive. Based on uniform removal of all alloy constituents, the maximum corrosion rate was 0.04 mil/year.

In another experiment Hastelloy N specimens were exposed to static BF_3 vapor for 6800 hr at 605°C in capsules that contained the fluoroborate mixture.⁴ The BF_3 pressure was varied in each capsule. Table 2 shows the measured weight changes. The weight changes were quite small, and no composition changes were noted in any of the specimens.

Another experience with BF_3 involved a pump loop (PKP-1), which was used to test a molten-salt pump with the fluoroborate mixture.^{15,16} An Inconel 600 bubbler tube, which had been used for BF_3 addition and salt level indication, had been in service 11,567 hr and was suspected to have become plugged just before the loop shutdown. During most of the test program the total gas flow rate was $370 \text{ cm}^3/\text{min}$. The gas was He-13.5 vol % BF_3 . The outside of the tube was exposed to the fluoroborate mixture at 550°C . After removal from the loop, examination disclosed a plug at the bottom of the inside of the tube. Above the plug, in order, were a thin black film, a gray thin deposit with a sprinkling of green particles, a bright green deposit, and finally green material covered with magnetic powder. The green deposit was Na_3CrF_6 , and the magnetic mixture was equivalent to Ni_3Fe . Figure 1 shows a cross section of the tubing wall near the liquid-gas interface. We saw a zone of metallic crystals on the inner surface to a thickness of approximately 20 mils. In view of the apparent loss of wall thickness in this area, these crystals probably represent vestiges of the original metal surface. Figure 2 shows a higher

¹⁵A. N. Smith, *MSR Program Semiannu. Progr. Rep. Feb. 28, 1971*, ORNL-4676, pp. 55-57.

¹⁶J. W. Koger, *ibid.*, pp. 211-15.

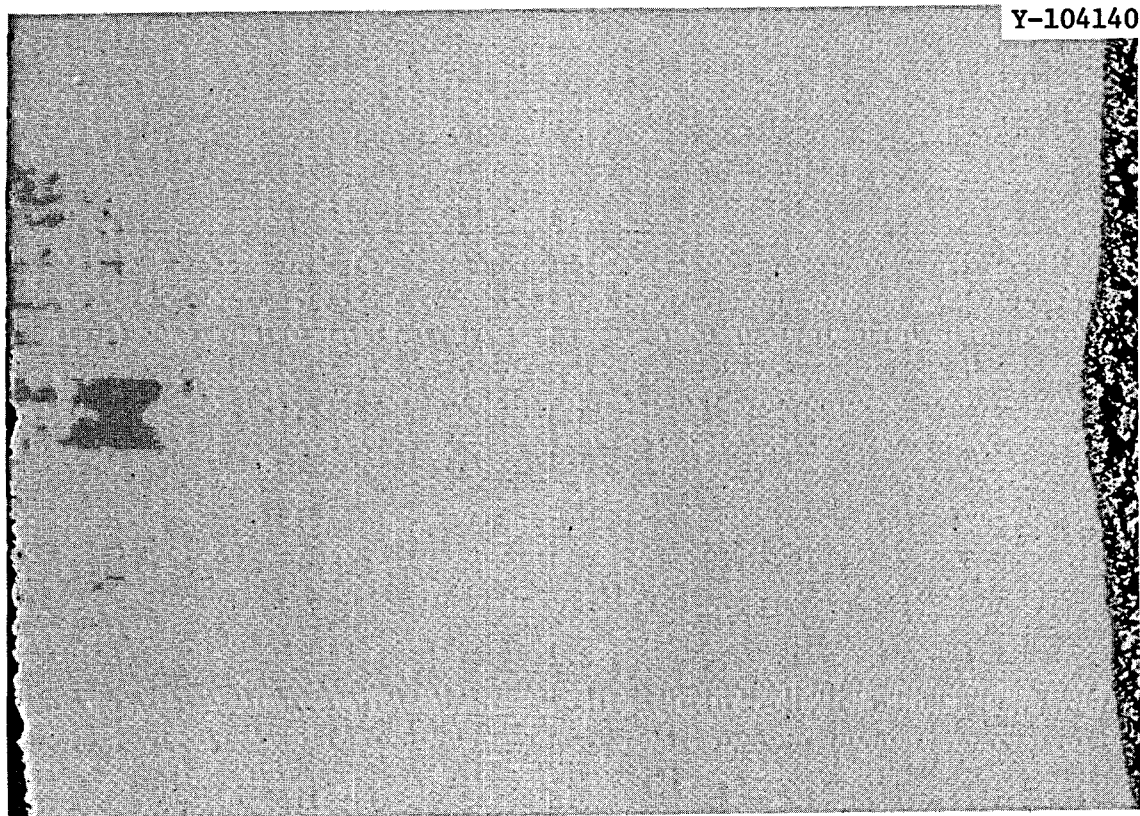
Table 1. Weight Losses of Hastelloy N Specimens Exposed to BF_3 in a Thermal Convection Loops

Temperature (°C)	Time (hr)	Weight Loss (mg/cm ²)	Rate of Weight Loss (mg cm ⁻² hr ⁻¹)
530	4,150	0	0×10^{-5}
530	21,983	0.3	1.4
530	23,427	0.3	1.3
530	32,090	0.15	0.47
593	19,433	0.4	2.1
607	4,150	0.4	9.7
607	21,983	2.2	10
607	23,427	0.3	1.3
607	32,090	2.0	6.2
687	19,433	0.3	1.5

Table 2. Weight Losses of Hastelloy N Specimens Exposed to Various Pressures of BF_3 at 605°C for 6800 hr.^a

BF_3 Pressure (psia)	Weight Loss (mg/cm ²)
4.2	0.03
64.7	0.3
114.7	0.4
414.7	0

^aJ. W. Koger and A. P. Litman, *Compatibility of Fused Sodium Fluoroborates and BF_3 Gas with Hastelloy N Alloys*, ORNL-TM-2978 (June 1970).



Outside

Inside

Fig. 1. As-Polished Cross Section from Near Liquid-Gas Interface of Inconel 600 Bubbler Tube from PKP-1 Pump Loop, 100 \times .

magnification of the inside surface, both as polished and etched, and tends to corroborate that the crystals are a result of material leaving the surface rather than material deposition. The material on the inside of the tube above the liquid level appeared to be the result of attack by water-derived impurities in the BF_3 .

On removing the pump rotary element from the loop and dismantling the various pump components, Smith¹⁷ found that the inner heat baffle plates had been severely attacked, as evidenced by holes in some places and by severe pitting in other places. The attack was not homogeneous on any one plate or constant from one plate to another. The top surface of the top

¹⁷A. N. Smith, *MSR Program Semiannu. Progr. Rep. Aug. 31, 1971*, ORNL-4728, pp. 31-34.

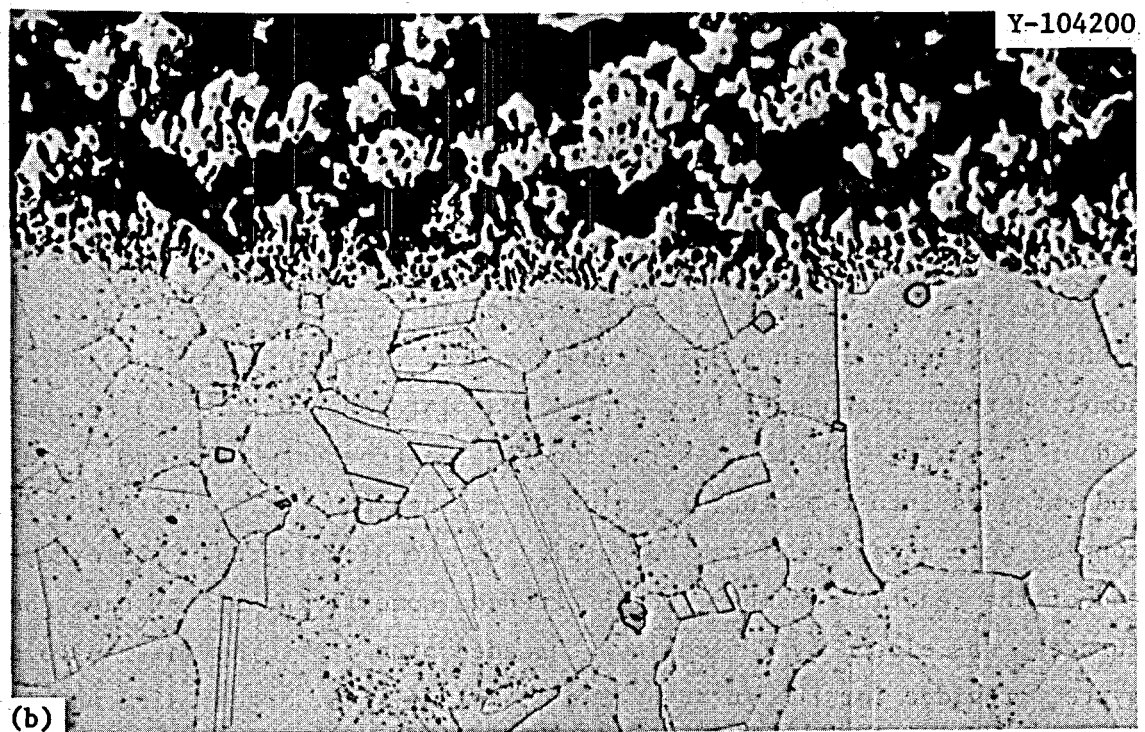
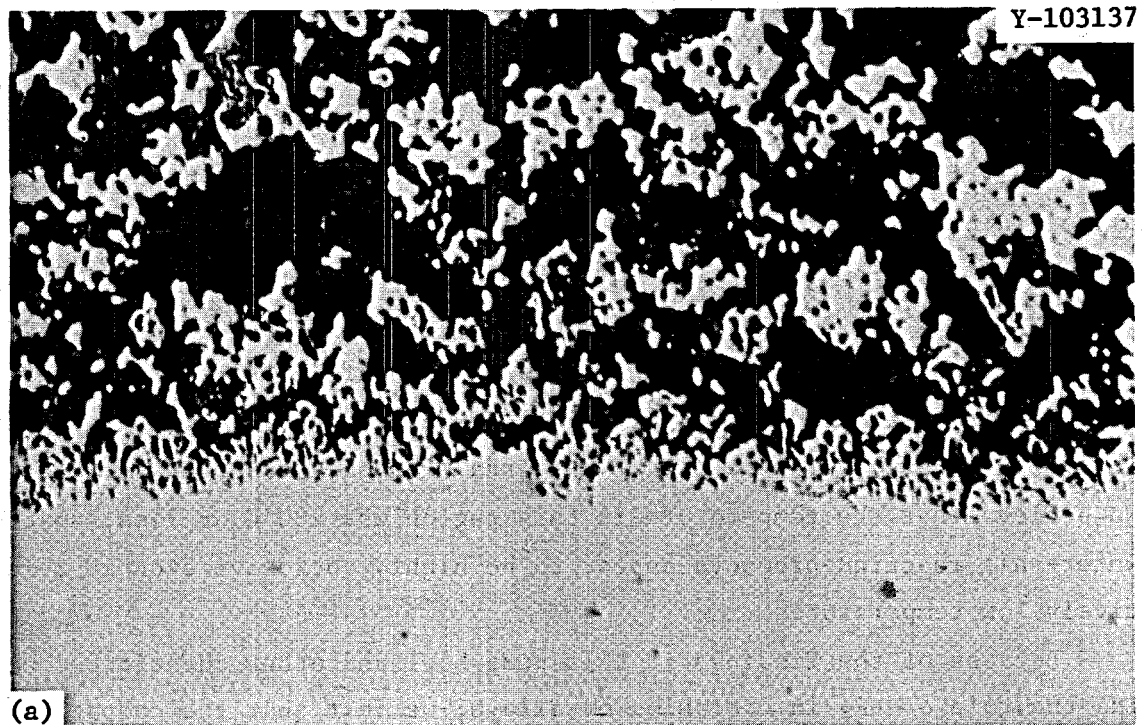


Fig. 2. Inside Surface of Inconel 600 Bubbler Tube from PKP-1 Pump Exposed to He-13.5 vol % BF_3 for 11,500 hr. (a) As Polished, 500 \times . (b) Etched with Aqua Regia, 500 \times .

plate was severely pitted, but the other surfaces that combined to form the chamber above the top plate (shaft, inner surface of impeller housing support cylinder, and lower surface of the cooling oil chamber) were relatively free of attack. All baffle plates except the one below the top plate had holes corroded completely through. The inner heat baffles fitted very tightly against the inner surface of the upper impeller housing support cylinder, so that probably most of the gas flow was past the inner annulus between the baffle plate and the pump shaft. The attack was attributed to the intimate contact of purge gas (or more correctly, the moisture in the purge gas) with puddles of salt that lay on the baffle surfaces. We believe that the temperature in this region was 280°C or less, so the water-salt reaction products appear to be highly corrosive even at relatively low temperatures.

Because of the findings in this pump bowl and other questions concerning the role of BF_3 in corrosion of alloys, we felt that the compatibility of BF_3 and various gas mixtures with candidate alloys for molten salt use should be studied systematically. As a first test, we exposed type 304L stainless steel and Hastelloy N to argon, air, BF_3 , and mixtures of these for 100 hr at temperatures from 200 to 600°C.

RESULTS OF THE CURRENT EXPERIMENTS

The reaction chamber was a 30-in. length of 2-in. IPS nickel pipe, mounted horizontally in a 3-in. tube furnace. The reacting gases were admitted through a penetration in the end plate that was welded to one end of the pipe. Sheathed thermocouples also penetrated the end plate and extended into the central region on the heated zone. Several thermocouples were attached along the outside of the nickel pipe. The other end of the reaction chamber, which extended some 10 in. out of the tube furnace, was closed by Teflon in a threaded pipe cap. The gas manifold system provided for the introduction of helium, BF_3 , argon, air, or mixtures of these gases at known flow rates into the reaction chamber. The system was sealed from the atmosphere by bubbling the gas effluent through a fluorocarbon oil. Metal samples were carried in nickel boats inserted

through the threaded access port. In several runs, metal specimens were placed with a fuel salt, LiF-20 mole % BeF₂-11.7 mole % ThF₄-0.3 mole % UF₄, or a coolant salt, NaBF₄-8 mole % NaF, in the nickel boats. The tests lasted 100 hr, and the gas flow rate was 100 cm³/min. The metal specimens were approximately 0.75 × 0.375 × 0.030 in. and weighed about 1 g. Specimens were weighed before and after each test, and reaction products were analyzed whenever possible. The nominal amounts of the major alloying constituents of Hastelloy N and type 304L stainless steel are given in Table 3.

Table 3. Nominal Compositions of Alloys

Alloy	Content, wt %			
	Ni	Cr	Fe	Mo
Hastelloy N	70	7	5	17
Type 304L stainless steel	10	18	69	0

Table 4 gives the results for the metal-salt combinations exposed to various gases at 600°C for 100 hr.

As was expected, air in combination with the fluoride salts produced highly corrosive conditions, which destroyed not only the specimens but the nickel boats. None of the weight changes measured for the Hastelloy N immersed in salt and exposed to any of the gas mixtures other than air were significant. For the stainless steel specimens immersed in salt, BF₃ gas caused more attack than argon. A larger moisture content in the BF₃ probably caused the greater attack. The fluoroborate mixture was more aggressive toward the stainless steel than the fuel salt. Weight losses rather than weight gains occurred because the corrosion products were dissolved from the specimens by the salt.

Table 5 gives the results for alloys exposed to the various gas mixtures without salt at temperatures from 200 to 600°C for 100 hr. In most cases, weight gains were found, since the corrosion products remained on the specimens and were not carried away.

Table 4. Weight Changes of Hastelloy N and Type 304L Stainless Steel Exposed to Various Gases while Immersed in Fluoride Salts at 600°C for 100 hr. Gas Flow 100 cm³/min

Alloy	Salt	Weight Change, ^a mg/cm ²		
		Argon ^b	BF ₃ ^c	Ar-BF ₃
Hastelloy N	Fuel ^d	-0.03	+0.03	0
	Coolant ^e	+0.06	0	-0.06
Type 304L stainless steel	Fuel ^d	-0.3	-1.4	-1.6
	Coolant ^e	-1.2	-5.2	-2.4

^aSpecimens exposed to air containing 7.5 ppm moisture were completely destroyed.

^b24 ppm moisture.

^c50 ppm moisture.

^dLiF-20 mole % BeF₂-11.7 mole % ThF₄-0.3 mole % UF₄.

^eNaBF₄-8 mole % NaF.

For Hastelloy N, the only significant changes measured at 600°C were in the mixtures containing air. This effect can be attributed to too small an amount of chromium in the Hastelloy to provide good resistance to air oxidation. At 200 and 300°C, all changes were rather small.

For the stainless steel at 600°C, the air-BF₃ mixture produced a large amount of chromium and iron oxides, which is shown in Fig. 3(b). The stainless steel specimen from which the oxides in Fig. 3(b) had been removed is shown in Fig. 3(c), and a relatively unaffected stainless steel specimen that had been exposed to air at 200°C is shown in Fig 3(a). By itself, BF₃ at 600°C also produced a large amount of corrosion product on the stainless steel and was more aggressive than argon. Significant weight gains of the stainless steel specimens were produced at 300°C and 600°C by all gas mixtures except air. At 200°C, most changes were rather small. Reaction of the stainless steel with argon, air, and an argon-BF₃ mixture resulted in a red corrosion product on the surface, whereas reaction with BF₃ by itself resulted in the formation of white material on the surface. These red and white corrosion products could not be

Table 5. Weight Changes of Hastelloy N and Type 304L Stainless Steel Exposed to Various Gases at 200 to 600°C for 100 hr. Gas Flow 100 cm³/min

Gas	Moisture (ppm)	Temp. (°C)	Weight Change, mg/cm ²	
			Hastelloy N	Stainless Steel
Argon	24	600	+0.06	+0.1
		300 ^a	0	+0.3 ^b
		200	+0.03	+0.05
Argon ^c	2	600	+0.03	+0.05
		300 ^a	0	+0.17
		200	-0.03	+0.03
BF ₃	<50	600	0	+5.2
		300 ^a	+0.2	+3.3 ^d
		200	-0.03	0
Air	7.5	600	+0.2	+0.03
		300 ^a	+0.03	-0.03 ^b
		200	+0.03	0
Argon-BF ₃	24 ^e	600	-0.03	+0.22
		300 ^a	+0.03	+5.85 ^f
		200	0	0
Argon ^c -BF ₃	2 ^e	600	+0.03	+0.05
		300 ^a	+0.3	+2.0
		200	0	0
Air-BF ₃		600	+0.2	+119.2 ^g
		300 ^a	0	+25.4
		200	0	-1.2
Argon-air	24 ^e	600	-0.1	+0.8
		300 ^a	0	+10.0 ^h
		200	-0.03	+0.2

^aNear gas entrance.

^bAdherent red layer on surface.

^cDried by passing through heated titanium sponge.

^dLarge amount of nonadherent white material on surface; 0.0290 g removed.

^eIn the argon.

^fLarge amount of nonadherent red material on surface; 0.0745 g removed.

^gLarge amount of oxide on surface, identified by x-ray diffraction as 25 mole % Cr₂O₃ and 75 mole % Fe₂O₃.

^hLarge amount of material flaked off.

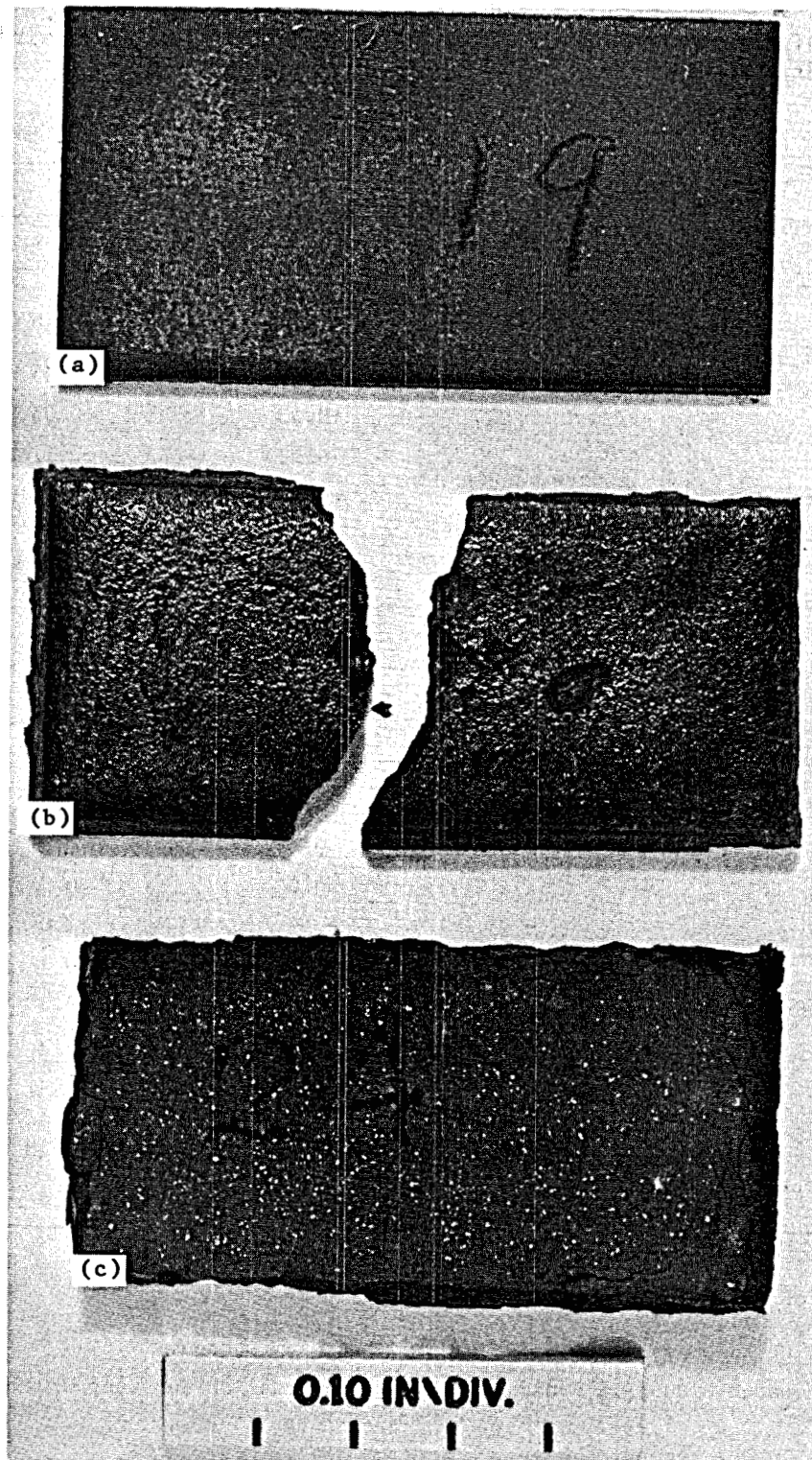


Fig. 3. (a) Type 304L Stainless Steel Specimen Exposed to Air at 200°C for 100 hr. (b) Chromium and iron oxides removed from a similar specimen after exposure to mixed air and BF_3 for 100 hr at 600°C. (c) Specimen from which oxide in (b) had been removed.

identified by x-ray analysis but contained iron, chromium, oxygen, and, where BF_3 was involved, fluorine. Thus, the corrosion products were probably complex mixtures of metal oxides and perhaps fluorides.

The use of drier argon (2 ppm moisture as opposed to 24 ppm) lowered the weight gain of the stainless steel specimens by about one-half. The effect of moisture was more dramatic when the gas was an argon- BF_3 mixture (Table 5).

Even though air had almost no effect on the stainless steel, the air- BF_3 mixture had the worst effect of any of the gases, much worse than just BF_3 . The air in combination with argon also produced more corrosion products than either of the two gases by themselves. In combination with salt, BF_3 had the worst effect. These results underline the problems that can result in a system that allows air and/or moisture to come in contact with molten fluoride salts or with the BF_3 vapor.

Except in air at 600°C , Hastelloy N was much more resistant to corrosion than the type 304L stainless steel.

CONCLUSIONS

1. At 600°C , air completely destroyed Hastelloy N and type 304L stainless steel specimens that were immersed in either a fuel salt or the fluoroborate coolant mixture.
2. No significant weight changes were measured for Hastelloy N immersed in salt exposed to argon or BF_3 at 600°C , but type 304L stainless steel lost weight.
3. For the stainless steel immersed in salt, the BF_3 gas was more corrosive than the argon.
4. Stainless steel weight losses were larger in the fluoroborate mixture than in the fuel salt.
5. At 200°C , the various gases produced very small changes in the alloys.
6. At 300 and 600°C , only small changes were noted in the Hastelloy N specimens.
7. Small changes were noted in the stainless steel exposed to air at 300°C , but large amounts of corrosion products were produced by BF_3 , argon- BF_3 , air- BF_3 , and argon-air.

8. At 600°C, the air-BF₃ mixture produced large quantities of chromium oxide and iron oxide on the stainless steel; also large amounts of corrosion products were produced by BF₃, argon-BF₃, and argon-air.

9. Drying the argon decreased the amount of corrosion.

10. Hastelloy N is more resistant to corrosion by the gases tested in this experiment than type 304L stainless steel.

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