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Argonne National Laboratory

**LABORATORY INVESTIGATIONS IN SUPPORT OF
FLUID-BED FLUORIDE VOLATILITY PROCESSES**

**Part XIV. The Corrosion of Nickel and
Nickel Alloys by Fluorine, Uranium
Hexafluoride, and Selected Volatile
Fission Product Fluorides at 500°C**

by

**William H. Gunther and
Martin J. Steindler**

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Chemical Engineering Division

December 1966

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ABSTRACT

The corrosion of selected materials of construction by fluorides of interest in the Fluoride Volatility Process has been studied. The metals investigated include: nickel-200, nickel-201, Monel, Inconel, Duranickel-301, HyMu-80, INOR-8, and nickel-200 coupons containing areas of nickel-200 weld, nickel-61 weld, or silver solder. Exposures of these metals for periods up to 30 hr at 500°C were carried out in elemental fluorine, UF_6 , SF_6 , and some of the following volatile fission product fluorides, both individually and in the presence of fluorine: GeF_4 , AsF_5 , NbF_5 , SbF_5 , BrF_5 , SeF_6 , MoF_6 , and TeF_6 . Corrosion rates were calculated from both the weight gain of the coupons after exposure and the weight loss of the coupons after descaling in a KNO_3 - $NaNO_3$ bath at 500°C. All of the exposed coupons were examined microscopically for intergranular penetration of the base metal.

Nickel-200 and Monel exhibited the best overall resistance to corrosion by fluorine, UF_6 , and some of the volatile fission product fluorides individually and in the presence of fluorine at 500°C.

I. INTRODUCTION

In the fluoride volatility process for the recovery of uranium and plutonium from partially spent nuclear reactor fuels, volatile fluorides of uranium, plutonium, and selected fission products are formed by reaction with fluorine. Nickel or nickel-containing alloys such as Monel are extensively used as the material of construction in both laboratory-scale and pilot plant-scale equipment which is to be exposed to fluorine and to

corrosive fluorides at elevated temperatures. Extensive attack of these materials is generally prevented by the formation of a protective scale of nickel fluoride that limits further reaction. In order to make estimates of the expected useful lifetimes of equipment exposed to fluorine, uranium hexafluoride, and the volatile fission product fluorides at elevated temperatures, a knowledge of the rates of corrosion by these fluorides is required.

This report contains the results of a survey program (1) to determine the resistance of several commonly used materials of construction to volatile fluorides likely to be encountered in the fluoride volatility process, and (2) to identify the more-promising metals of construction.

Static experiments have been performed at 500°C in which nickel-200, nickel-201, Monel, Inconel, Duranickel alloy 301, HyMu-80, INOR-8 and nickel-200 coupons containing areas of nickel-200 weld, nickel-61 weld, or silver solder were exposed to elemental fluorine and to uranium hexafluoride (UF_6), germanium tetrafluoride (GeF_4), arsenic pentafluoride (AsF_5), niobium pentafluoride (NbF_5), antimony pentafluoride (SbF_5), bromine pentafluoride (BrF_5), selenium hexafluoride (SeF_6), molybdenum hexafluoride (MoF_6), and tellurium hexafluoride (TeF_6) individually and in the presence of fluorine for periods up to 30 hr. Corrosion rates were calculated from the weight changes of the coupons after reaction. Either the weight gains of the corroded specimens were determined and/or the specimens were descaled and their weight losses determined. All of the coupons were examined for intergranular penetration of the base metal. Most of the above-mentioned alloys were also exposed to sulfur hexafluoride (SF_6) at 500°C to permit comparison of the corrosion rates and intergranular penetrations of the Group VI (S, Se, Te) hexafluorides.

A survey of the unclassified literature on the rates of corrosion of nickel and nickel alloys by fluorine, uranium hexafluoride, and the volatile fission product fluorides is included.

II. LITERATURE SURVEY

A. Corrosion by Gaseous Fluorine

A survey of the unclassified literature on the corrosion of nickel and three nickel-containing alloys (Monel, Inconel, and Duranickel-301) by fluorine at elevated temperatures revealed corrosion rates summarized in Tables 1, 2, and 3. A wide variation in results is evident for the exposure times ranging from 4 to 960 hr and pressures at or near atmospheric. It has been shown that the kinetics of the reaction of high-purity nickel with

TABLE 1. Corrosion Rates for Nickel Exposed to Gaseous Fluorine

Material	Time (hr)	Temperature (°C)	Corrosion Rate (mil/hr)	Reference
Nickel-200	120	362	0.00004	5
Nickel-201	5	380	0.00017	5
Nickel ^a	4-15	400	0.0010	8
Nickel-200	33	400	0.0002	1
Nickel-200	50	400	0.00015	4
Nickel ^a	4-15	450	0.0026	8
Nickel-200	960	475	0.00010	11
Nickel ^a	-	500	0.0083	10
Nickel ^a	4-15	500	0.0071	8
Nickel-200	5	500	0.0024	3
Nickel-201	5	500	0.0017	3
Nickel-201	6	500	0.0015	This study
Nickel-200	9	500	0.0027	This study
Nickel-200	33	500	0.00041	1
Nickel-200	50	500	0.00037	4
Nickel-201	5	550	0.0023	5
Nickel-200	6	550	0.0005	2
Nickel-200	120	550	0.0016	5
Nickel ^a	4-15	600	0.0403	8
Nickel-200	5	600	0.0150	3
Nickel-201	5	600	0.0062	3
Nickel-200	33	600	0.0018	1
Nickel-200	50	600	0.00092	4
Nickel-201	77	600	0.0021	6
Nickel-200	96	600	0.0027	9
Nickel-201	132	600	0.0011	6
Nickel-200	132	600	0.0010	6
Nickel-200	195	600	0.00034	6
Nickel ^a	4-15	650	0.022	8
Nickel-200	5	650	0.0067	2
Nickel ^a	4-15	700	0.047	8
Nickel-201	5	700	0.011	3
Nickel-200	5	700	0.130	3
Nickel ^a	-	700	0.139	10
Nickel-200	4	750	0.040	2

^aType of nickel not specified.

TABLE 2. Corrosion Rates for Monel Exposed to Gaseous Fluorine

Time (hr)	Temperature (°C)	Corrosion Rate (mil/hr)	Reference
120	362	0.00014	5
4-15	400	0.0007	8
4-15	450	0.0021	8
-	500	0.0278	10
4-15	500	0.0028	8
9	500	0.004	This study
120	550	0.0008	5
4-15	600	0.083	8
195	600	0.0005	6
4-15	650	0.111	8
6	650	0.0046	2
-	700	0.278	10
4-15	700	0.208	8
6	750	0.33	2

TABLE 3. Corrosion Rates for Inconel and Duranickel-301
Exposed to Gaseous Fluorine

Material	Time (hr)	Temperature (°C)	Corrosion Rate (mil/hr)	Reference
Inconel	5	368	0.0088	5
Inconel	4-15	400	0.053	8
Inconel	4-15	450	0.133	8
Inconel	4-15	500	0.086	8
Inconel	9	500	0.138	This study
Duranickel-301	9	500	0.006	This study
Inconel	5	536	0.394	5
Inconel	5	550	0.37	2
Inconel	4-15	600	0.236	8
Inconel	77	600	0.0053	6
Duranickel-301	243	600	0.0029	6
Inconel	4-15	650	0.181	8
Inconel	6	650	0.81	2
Inconel	4-15	700	0.708	8
Inconel	5	750	2.83	2

fluorine initially follows a parabolic rate law from 300 to 600°C, but that after a period of time the reaction slows, conforming to a cubic or lower rate.¹ Thus, long-term corrosion rates, at least for nickel, calculated from short-term exposure data, would be erroneously high.

Jarry and co-workers¹ studied the kinetics of the nickel-fluorine reaction between 300 and 600°C using high-purity fluorine (99.93 v/o) and both nickel-200 and high-purity vacuum-melted nickel, designated Nivac (99.92%), by following the decrease in fluorine pressure as a function of time in a static system. The fluorine pressure was maintained at 700 ± 25 Torr and the internal surface of the reaction vessel served as the nickel reactant. The reaction between fluorine and nickel-200 was shown to be slower at a given temperature than that between fluorine and Nivac nickel. The difference was greatest during the initial periods of the reaction, and became less pronounced with time and at higher temperatures. No clear reason for the difference in reactivity was uncovered.

Steindler and Vogel² studied the corrosion of nickel-200, Monel, and Inconel at temperatures from 550 to 750°C in a fluorine-flow system. Corrosion rates were calculated from both the weight changes of the coupons and the thicknesses of the scale as determined by microscopic examination. The authors believed that, due to difficulties in obtaining representative scale-thickness data, the corrosion rates derived from weight changes were more reliable. Metallographic examinations of the specimens after exposure did not reveal any unusual features in the corrosion process.

Jarry and co-workers³ exposed nickel-200 and nickel-201 coupons to a stream of fluorine for 5 hr at 500, 600, and 700°C. Calculations based on weight-change data were reported as thickness of the NiF_2 scale formed on the coupon surface. The nickel-200 coupons appeared to react to a greater extent, and film spalling was more pronounced than for the nickel-201 coupons.

Steunenbergh *et al.*⁴ investigated the reaction of nickel-200 and fluorine at 300 to 600°C by observing the decrease in fluorine pressure as a function of time in a static system. The initial fluorine pressure was about 500 Torr. The specimen itself was a nickel vessel whose volume and internal surface area were known; a new vessel was used for each experiment. The data were reported as millimoles of fluorine consumed per square centimeter of nickel surface for periods up to 50 hr. At 300 and 400°C a logarithmic rate law appeared to hold, but as the temperature was raised, the tendency was toward a parabolic behavior.

Jackson⁵ studied the corrosion of twenty-five metals and alloys by both liquid and gaseous fluorine. Several specimens were exposed to

gaseous fluorine at elevated pressures and temperatures. The data summarized in this report cover the corrosion of nickel-200, nickel-201, Monel, and Inconel in a stream of gaseous fluorine at temperatures to 538°C and exposure times up to 120 hr. Rates were usually calculated from the weight increase of the specimens after exposure. Only the corrosion rates calculated from the longest exposure times are listed in Tables 1, 2, and 3.

Miles *et al.*⁶ exposed a variety of metals to gaseous fluorine at 600°C. The progress of the corrosion was observed by interrupting the exposure at intervals, weighing the samples, and then re-exposing for additional periods up to 243 hr. The results were reported as milligrams of fluorine consumed per square centimeter of metal exposed. The relatively low values obtained by these workers may be due to the cyclic exposure of the samples to fluorine and the laboratory atmosphere. The effect of cycling of fluorinated nickel has been described in the literature.⁷ A rather remarkable degree of protection was obtained for nickel tubes by allowing them to stand for some time at room temperature open to the atmosphere after an initial prefluorination.

Myers and DeLong,⁸ using a flow system, studied the corrosion of various metals including nickel, Monel, and Inconel by fluorine in the temperature range 400-700°C. The corrosion scale was removed by both mechanical polishing and electrolytic methods. Corrosion rates were obtained from both weight-gain and weight-loss data, with generally good agreement for results from both methods. The durations of exposure varied from 4 to 15 hr. No significant decrease of corrosion rate with time (up to 15 hr) was noted, and it was concluded that the scales of corrosion products formed on the samples were not protective. This non-protectiveness of the corrosion scales is at variance with most other corrosion data in which with increasing exposure of nickel and nickel alloys to fluorine, a decrease in rate was observed.

Trevorrow and Gunther⁹ exposed nickel-200 discs (1/16 in. thick and 1/2 in. in diameter) to fluorine in a static system for 4 days at 600°C. The fluorine pressure was maintained at 700 ± 25 Torr. Corrosion rates were calculated from the weight increases of the discs, since the NiF_2 scale which formed on the disc surface was uniform and adherent. The extent of fluorination was recorded as milligrams of fluorine consumed per square centimeter of disc surface.

Landau and Rosen¹⁰ present data for the rates of attack of nickel and Monel sheet by fluorine at 500 and 700°C. No mention was made of the type of nickel used or the exposure times. Rates were calculated from increases in weight of the samples and were tabulated as penetration in inches per month.

Chilenskas and Gunderson¹¹ exposed nickel-200 plate to an atmosphere of 50 v/o fluorine in nitrogen at a total pressure of about 1000 Torr at 475°C for periods up to 960 hr. The corrosion rates were calculated from the weight losses of the specimens after descaling. Microscopic examination of the specimens after exposure disclosed no evidence of intergranular penetration. These authors also studied the effect of thermal cycling upon the corrosion rates of nickel-200 and nickel-200 weld in a fluorine environment. In their experiments, a reactor containing the corrosion specimens was maintained continuously at 550°C and was fed a mixture of 50 v/o fluorine in nitrogen at about 1000 Torr for 8 hr each day. For the remainder of each day, a purge of only nitrogen was maintained. A second reactor in series with the first received the same reactants for the same times, but the temperature was maintained at 550°C only for the 8 hr of fluorine exposure, following which it was cooled to room temperature and reheated to 550°C before the next cycle. This cycle continued for 10 days (total exposure of 80 hr to fluorine), after which the specimens were descaled and weighed. It was concluded that under these conditions the effect of thermal cycling was negligible. No intergranular penetration of the cycled specimens was observed.

Hale and co-workers¹² exposed discs of nickel-200 and Monel to fluorine at pressures up to one atmosphere and at each of the temperatures 593, 704, and 816°C for a total of 36 hr. Since the discs were exposed at more than one temperature, corrosion rates were not calculated. However, from measurements of the thickness of the corrosion scale, Monel was estimated to have consumed at least forty times as much fluorine as nickel-200.

B. Corrosion by Uranium Hexafluoride

The unclassified literature contains little information concerning the corrosion of nickel and nickel alloys in the presence of UF_6 . Heymann and Kelling¹³ contacted liquid UF_6 at 80°C with Monel coupons for periods up to two months, obtaining a rate of corrosion of less than 2×10^{-7} mil/hr. The authors believed that there were definite indications that the presence of hydrogen fluoride in the UF_6 substantially increased the corrosion rate.

Langlois¹⁴ conducted a laboratory study on the effect of temperature on the corrosion by UF_6 of electrically heated wires at temperatures to 1000°C. Corrosion rates of 0.5-mm-diameter nickel, Monel, and Inconel wires were obtained. This technique made it possible to determine continuously the corrosion rate by measuring the change in the electrical resistance of the wire as a function of time and temperature. Anomalous results were obtained for the corrosion of nickel by UF_6 in the temperature range 550-700°C, in which rates increased sharply, reached a maximum at about 640°C, and then rapidly decreased. With increasing temperature

the rate again increased above 700°C. Below 700°C, localized corrosion of the nickel was visually observed. However, above 700°C the corrosion appeared quite uniform. Intergranular penetration of up to 8 mils was observed in samples exposed in the 500-700°C temperature range. Metallographic studies indicated that at these temperatures impurities precipitated at the grain boundaries and were most pronounced at about 650°C, which is midway in the temperature range necessary to recrystallize nickel.¹⁵

Prefluorinated nickel-200 discs covered with a NiF_2 scale averaging 37000 Å in thickness were exposed by Hale *et al.*¹² for 24 hr at 816°C. Based on the weight loss of the UF_6 source pot, a corrosion rate of 0.0004 mil/hr was obtained. X-ray diffraction patterns of the corrosion scales indicated the presence of UF_4 and NiF_2 .

C. Corrosion by Fission Product Fluorides

Little data are available on the corrosion of nickel or nickel alloys by the volatile fission product fluorides or on the thermal stability of the fission product fluorides at elevated temperatures. Miller and co-workers¹⁶ exposed nickel-201, Monel, and INOR-8 to boiling NbF_5 (b.p. 235°C) at vapor, interface, and liquid areas for 405 hr. A fluorine over-pressure was maintained at all times. The rate of corrosion at the interface was 2-4 times higher than the rate in either the vapor phase or the liquid phase. Corrosion rates in the vapor phase for nickel-201, Monel, and INOR-8 were calculated to be 0.00012, 0.00014 and 0.00011 mil/hr, respectively. Intergranular penetration of nickel-201 and Monel totaled 2.5 and 0.6 mils, respectively. The INOR-8 samples showed no evidence of intergranular penetration.

Oak Ridge¹⁷ has reported that tellurium fluorides are quite easily reduced by metallic nickel at intermediate temperatures (up to 300°C). It was also shown¹⁸ that when TeF_6 gas, diluted with helium, was passed through a column of nickel mesh (residence time—0.5 min) at 400, 500, and 600°C, tellurium retentions of up to 88.5% were obtained. Junkins *et al.*¹⁹ observed that at about 200°C TeF_4 disproportionated into TeF_6 plus a lower fluoride of tellurium. Above 333°C the products of the first phase of the disproportionation themselves disproportionated to TeF_6 plus either a still lower tellurium fluoride or elemental tellurium.

D. Corrosion of Various Types of Nickel Welds

Data reported in the literature concerning the corrosion of various types of nickel welds by fluorinating agents are somewhat conflicting. Chilenskas and Gunderson¹¹ studied the fluorination of nickel-200 plates containing areas of nickel-200 weld and nickel-61 weld. After an exposure of 240 hr at 475°C in an atmosphere of 50 v/o fluorine in nitrogen and at a total pressure of about 1000 Torr, the corrosion rates of specimens

containing nickel-200 and nickel-61 welds were 0.0002 and 0.150 mil/hr, respectively. The rates were calculated from the weight losses of the specimens after descaling. For those specimens containing areas of nickel-200 weld, rates were calculated assuming uniform corrosion of the entire specimen. Corrosion rates for the specimens containing areas of nickel-61 weld were calculated assuming corrosion of only the welded areas.

The authors attributed the high corrosion rate exhibited by the nickel-61 welded areas to the presence of titanium in the nickel-61 stock weld. Titanium forms a volatile fluoride which, if present under the scale of NiF_2 that ordinarily forms a protective layer on the nickel, could result in a pressure buildup under the scale. Subsequent release of pressure would result in a rupture and loss of integrity of the scale. X-ray diffraction analysis showed TiOF_2 to be the major constituent of a condensed phase in the gas stream exhausting from the furnace in which the specimens were exposed.

A comparison of the corrosion rates for nickel-200 plate and nickel-200 welded with nickel-200 welding rod showed that the welded specimens exhibit only slightly higher corrosion rates than the plate specimens. No evidence of intergranular penetration of either the welded or nonwelded specimens was found.

Litman and Goldman²⁰ exposed a variety of materials including nickel-201 welding rod and nickel-61 welding rod to a molten fluoride salt ($\text{NaF-ZrF}_4\text{-UF}_4$) for about 900 hr at temperatures from 540 to 730°C. About 10% of the time a fluorine sparge was employed. Corrosion rates of the two specimens were both about 0.09 mil/hr. The nickel-61 weld specimens showed generally analogous bulk metal loss behavior when compared to the nickel-201 specimens. Microscopic examination showed the nickel-201 to have intergranular penetration to a depth of about 5 mils, whereas the nickel-61 welding rods showed no intergranular penetration.

Miller and co-workers¹⁶ exposed nickel-200 coupons containing areas of nickel-201 weld to boiling NbF_5 (b.p. 235°C) for 405 hr. Microscopic examination showed no preferential attack occurring at the welds of any of the coupons. No intergranular penetration was observed.

Smiley et al.^{21a} subjected samples of nickel-200, nickel-200 containing areas of nickel-61 weld, and pieces of nickel-61 welding rod to 20 cycles of alternating exposure to hydrogen chloride-hydrogen at 400°C and to dilute fluorine at 540°C for a total of 313 hr. Corrosion rates were calculated from the weight changes of the samples. Nickel-200 exhibited the best corrosion resistance. The weight losses for the welded samples were greater than those for unwelded samples. The corrosion rates for nickel-200, nickel-200 containing areas of nickel-61 weld, and nickel-61 welding rod were 0.0006, 0.0008, and 0.021 mil/hr, respectively.

Microscopic examination of the welded specimens showed intergranular penetration of the weld to be about 1 mil or several times that of the base metal. Samples^{21b} of nickel-200 and nickel-201 containing areas of nickel-200 weld, nickel-201 weld, and nickel-61 weld were subjected to successive exposures to hydrogen chloride-hydrogen at 400°C, oxygen-nitrogen at 500-540°C, and dilute fluorine at 540°C for periods of 111 to 161 hr. The corrosion rates obtained ranged from 0.003 to 0.006 mil/hr for nickel-200 weld; from 0.002 to 0.066 mil/hr for nickel-201 weld; from 0.001 to 0.072 mil/hr for nickel-61 weld. No attempt was made to explain the wide variation in the rates of corrosion of nickel-201 and nickel-61 welds.

III. CORROSION OF NICKEL AND NICKEL ALLOYS BY FLUORINE AND SELECTED VOLATILE FLUORIDES

A. Experimental

1. Materials

Two types of nickel were used in this study; nickel-200 and nickel-201. The major difference between the two metals is the carbon content, which was shown by spectrographic means to be 0.12 and 0.02 w/o for nickel-200 and nickel-201, respectively. The dimensions of a typical nickel coupon were 1.0 by 1.4 by 0.17 cm.

To identify the particular alloys of Monel and Inconel used in these experiments, representative samples of the two alloys were analyzed by X-ray spectral techniques. These analyses showed that the Monel contained approximately 0.5% titanium, and that the Inconel contained little or no niobium or titanium. The only Monel and Inconel alloys meeting these specifications and commercially available in sheet form are Monel alloy K-500 and Inconel alloy 600.²² Thus the Monel and Inconel used in these experiments are regarded as such. The dimensions of typical Monel and Inconel coupons were 0.33 by 1.0 by 0.14 cm, and 1.0 by 1.4 by 0.17 cm, respectively.

The Duranickel alloy 301 was obtained from the International Nickel Company, Inc., and was designated cold-rolled annealed, 1/2 hard. The metal was coated with a very hard, thin, shiny black scale which could not be removed by scraping or by immersion in either concentrated nitric acid or aqua regia. The dimensions of a typical Duranickel coupon were 1.0 by 1.4 by 0.14 cm.

HyMu-80 plate and INOR-8 plate were obtained from L. Anderson, Oak Ridge Gaseous Diffusion Plant, and were subsequently cut into coupons whose approximate dimensions were 0.90 by 1.9 by 0.67 cm.

The welded coupons were prepared by joining two pieces of nickel-200, using either nickel-200 filler metal or nickel-61 filler metal, and employing the Tungsten Arc Inert Gas (TIG) process. These filler metals were manufactured by the International Nickel Company, Inc.

Silver-soldered coupons were prepared by joining two pieces of nickel-200 using silver solder designated No. 355 and obtained from the American Platinum Works Division of Engelhard Industries, Inc., Newark, N. J. The nominal composition of the solder is 56% silver, 22% copper, 17% zinc, 5% tin, and no cadmium. X-ray spectral measurements verified that the solder was cadmium-free. The average dimensions of the welded and soldered coupons were 1.0 by 2.0 by 0.17 cm.

All of the above-mentioned alloys were used as received after drilling 3/32-in. support holes in each end, polishing the edges, and degreasing the coupons in methylethylketone. The nominal chemical compositions, in weight percent, for all of the metals used in this study including nickel-200 and nickel-61 welding rods are tabulated in Table 4.

TABLE 4. Nominal Chemical Compositions, in Weight Percent, of Nickel and Nickel Alloys

Alloy	Ni	C	Mn	Fe	S	Si	Cu	Cr	Ti	Al	Mo	Co
Nickel-200	99.5	0.06	0.25	0.15	0.005	0.05	0.05	-	-	-	-	-
Nickel-201	99.5	0.01	0.20	0.15	0.005	0.05	0.05	-	-	-	-	-
Monel K-500	65.0	0.15	0.60	1.00	0.005	0.15	29.5	-	0.50	2.80	-	-
Inconel-600	76.0	0.04	0.20	7.20	0.007	0.20	0.10	15.8	-	-	-	-
HyMu-80	79	0.05	0.5	16	-	0.15	-	-	-	-	4	-
INOR-8	70	0.06	0.8	5	0.01	0.3	0.35	7	-	-	16	0.2
Duranickel-301	94.0	0.15	0.25	0.15	0.005	0.55	0.05	-	0.50	4.50	-	-
Nickel-61												
welding rod	93.0	0.15	1.0	1.0	0.01	0.75	0.25	-	2.0-3.5	1.50	-	-
Nickel-200												
welding rod	99.5	0.06	0.25	0.15	0.005	0.05	0.05	-	-	-	-	-

The fluorine used in these studies was purchased from the Allied Chemical Company and was passed through a heated sodium fluoride trap for the removal of hydrogen fluoride. GeF_4 , AsF_5 , and SeF_6 obtained from Ozark-Mahoning Company, Tulsa, Oklahoma; MoF_6 and TeF_6 obtained from the Allied Chemical Company; SF_6 and BrF_5 obtained from the Matheson Company; and UF_6 obtained from Oak Ridge National Laboratory, were all purified by vacuum distillation. Their purities were ascertained by vapor density, vapor pressure, and/or infrared spectral measurements. NbF_5 and SbF_5 were obtained from the Allied Chemical Company. Because of their low vapor pressures at room temperature, 0.01 and 4.3 Torr for NbF_5 and SbF_5 , respectively, it was not practical to use vapor density or infrared spectral measurements to determine their purity. However, these pentafluorides were cooled to -195°C , all of the volatile gases were removed by directly pumping on the fluorides, and their purities were ascertained by chemical analysis.

2. Apparatus

The apparatus for this work was contained in a vacuum frame hood and included a nickel manifold employing 3/8-in. Hoke 413A diaphragm valves, distillation traps, storage vessels, a mechanical forepump, an oil diffusion pump, an activated alumina tower for disposal of the corrosive fluorides, a Booth-Cromer pressure transmitter and self-balancing relay²³ connected to a mercury manometer, and helicoid, thermocouple, and ionization gages to measure the system pressure. The components of the system were joined by either silver-soldered connections or flare fittings sealed with Teflon or copper gaskets. Cylinders containing the fission product fluorides were each attached to the manifold by a 3/8-in. Teflon-gasketed flare fitting. The reactions were carried out in a 3/4-in. by 12-in. nickel test tube. Temperature control was maintained at $500 \pm 10^\circ\text{C}$ in a vertical 1 1/4-in.-diameter resistance tube furnace by means of a proportionally circuited Variac controlled by a Brown Pyr-O-Vane pyrometer controller. The pyrometer was activated by a chromel-alumel thermocouple attached to the reaction vessel wall.

Normally, the parts of the system outside the furnace were operated at ambient temperatures. However, when the less volatile fluorides, namely, NbF_5 or SbF_5 , were present in the system, it was necessary to heat the lines, including the Booth-Cromer pressure transmitter, to about 180°C to prevent condensation of the fluorides. A storage vessel containing NbF_5 or SbF_5 was heated to the appropriate temperature necessary to maintain a pressure of about 100 Torr. Prior to use, the reaction vessel was always prefluorinated with fluorine at 550°C overnight.

Descaling of the coupons after exposure was carried out in a 1 7/8-in. by 10-in. nickel vessel which was positioned in a vertical 2 1/2-in.-diameter resistance tube furnace. A chromel-alumel thermocouple, which was located inside a 1/4-in. nickel tube sealed at the bottom, was immersed in the molten salt bath and controlled the temperature in a manner similar to that described above for the corrosion apparatus. The descaling vessel was open to the hood atmosphere during the descaling process. This process involved immersion of the coupons in an equimolar sodium nitrate-potassium nitrate molten salt bath (about 375 g total) at 500°C for 0.5 hr. A holder, made of 1/4-in. nickel tubing, was used to suspend up to 10 coupons from nickel hooks which were silver-soldered in place on the holder. The descaling bath was periodically replaced with a fresh bath.

3. Procedure

Prior to the start of an experiment, the specimen coupons were polished on the edges to remove burrs, measured to the nearest 0.1 mm, degreased in methylethylketone, weighed to the nearest 0.1 mg, and connected together

with nickel wire. The uppermost coupon was wired to a hook welded onto a 3/4-in. male flare fitting. Usually, ten coupons were interconnected (see Fig. 3, p. 32). The loaded vessel was sealed to the flare fitting with a copper gasket, leak-tested, and attached to the manifold.

The volatile reactant gas or gases were added to the reactor while the reactor was at room temperature, except that when NbF_5 or SbF_5 was the reactant gas the reaction vessel and all lines were heated to a temperature higher than that of the storage vessel. The pressures of fluorine and fluorides (including UF_6 and SF_6) were arbitrarily maintained at 575 ± 100 and 100 ± 20 Torr, respectively. When fluorine was not present, the fluorides were periodically added to the reaction vessel when the pressure dropped to 80 Torr. However, when fluorine was present with a fission product fluoride, the reactor was evacuated and a fresh mixture was added each time it was necessary to add additional reactant gas.

When the reaction temperature of 500°C was reached, the pressure was recorded and the experiment commenced. Approximately 0.5 hr. was required to heat the reaction vessel to 500°C . At the conclusion of an experiment, the reactor was evacuated and allowed to cool slowly to room temperature. The coupons which did not show appreciable spalling of the fluoride scale were reweighed, and corrosion rate was calculated from the weight gain of the coupon, assuming that only NiF_2 had formed on the surface of the coupon.

It is recognized that corrosion rates based on specimen weight gains may be inaccurate for a variety of reasons, some of which are: (1) the fission product fluorides may thermally decompose, resulting in deposition on the surface of the coupon of a lower fluoride of the fission product; (2) compounds other than NiF_2 may form on the coupons during the reaction; and (3) spalling of the fluoride scale during the initial part of the reaction may occur. Thermal decomposition of the fission product fluoride and deposition of the lower fluoride on the corrosion coupon would result in apparent rates of corrosion greater than those actually occurring. Formation of compounds other than NiF_2 in the corrosion scale would result in errors in the same direction. Spalling of the scale at any time during the exposure would result in apparent rates of corrosion lower than those actually occurring.

To overcome these difficulties, the coupons were descaled after exposure to the corrosive environment. The descaling method is identical to one used by Chilenskas and Gunderson¹¹ in similar corrosion studies, and consisted of immersion of the specimens in an equimolar salt bath of potassium nitrate-sodium nitrate at 500°C for 30 min. To determine the effect of this salt bath on the metals, fresh coupons of all the metals used in this study were placed in the bath at 500°C for $1\frac{1}{2}$ hr, or three times as

long as the normal descaling period. This longer descaling period for the coupons resulted in apparent corrosion rates of less than 0.001 mil/hr for all of the metals tested, assuming a typical corrosion experiment to be 8 hr in length. The descaling procedure allowed corrosion rates to be calculated from the weight losses of the descaled coupons. No appreciable intergranular penetration was observed in coupons exposed to the descaling bath at 500°C.

For measurement of any intergranular attack, exposed coupons were mounted in plastic, polished with 0.3-micron alumina, etched, and microscopically examined. The following etchants used at ambient temperature gave the best results:

Nickel-200, Nickel-201, Nickel-200 Weld, Nickel-61 Weld, and Monel. An aqueous solution of 10% NaCN-10% $(\text{NH}_4)_2\text{S}_2\text{O}_8$ for 30 to 60 sec.

Inconel, Duranickel-301, HyMu-80, and INOR-8. A CuCl_2 -saturated aqua regia solution for 10 to 60 sec.

None of the etchants tried brought out a crystalline structure for silver solder.

B. Results

The results of the microscopic examination are presented in Table 5. Total penetration of the base metal is in mils.

The corrosion rates obtained in this study are tabulated in Tables 6, 7, 8, and 9 as mils of metal reacted per hour of exposure, and are averages of values from two to five coupons. The rates were calculated from the weight losses of the coupons following immersion of the exposed coupons in the descaling bath, assuming complete removal of all scale in the bath. The numbers in parentheses are rates calculated from the weight gains of the coupons, assuming that only NiF_2 had formed uniformly on the coupon surface. The corrosion rates for the coupons containing areas of silver solder were calculated by deducting the average corrosion rate of the base metal (nickel-200) by the corrosive environment and assuming that only AgF had formed on the silver-soldered surface. Rates reported as nil are less than 0.001 mil/hr.

1. Corrosion by F_2 , UF_6 , and BrF_5

Fluorine. All of the alloys were exposed to fluorine for 6.3 to 26.6 hr at 500°C. Coupons of nickel-200, nickel-201, Monel, and the two types of nickel weld were only slightly dulled as a result of the exposure.

TABLE 5. Microscopic Examination of Corrosion Specimens Exposed to Fluorine and Selected Volatile Fluorides at 500°C

Corrosive Environment	Reaction Time (hr)	Nickel-200	Nickel-201	Monel	Inconel	Duranickel-301	HyMu-80	INOR-8	Nickel-20J Weld	Nickel-61 Weld
F_2	6.3-26.6	Uniform I.G. ^a attack to 1.0 mil.	No I.G. ^a attack.	Isolated areas of I.G. ^a attack to 0.4 mil.	No I.G. ^a attack.	No I.G. ^a attack.	No I.G. ^a attack.	No I.G. ^a attack.	No I.G. ^a attack of weld or at junction of weld and base metal.	No I.G. ^a attack of weld or at junction of weld and base metal.
UF_6	7.2-28.8	About 50% of surface has I.G. attack to 0.5 mil; remaining 50% has no I.G. attack; isolated areas to 2.5 mils.	About 25% of surface has I.G. attack to 0.4 mil.	Isolated areas of I.G. attack to 0.5 mil.	No I.G. attack.	No I.G. attack.	About 50% of surface has I.G. attack of 2-7 mils; remaining 50% has no I.G. attack.	No I.G. attack.	No I.G. attack of weld or at junction of weld and base metal.	No I.G. attack of weld or at junction of weld and base metal.
$UF_6 + F_2$	7.0-7.2	Uniform I.G. attack to 0.8 mil, isolated areas to 1.5 mils.	No I.G. attack.	About 25% of surface has I.G. attack to 0.4 mil, isolated areas to 0.8 mil.	No I.G. attack.	No I.G. attack.	About 50% of surface has I.G. attack of 2-4 mils; remaining 50% has no I.G. attack.	No I.G. attack.	No I.G. attack of weld or at junction of weld and base metal.	No I.G. attack of weld or at junction of weld and base metal.
BrF_5	27.8	Uniform I.G. attack to 0.8 mil, isolated areas to 1.0 mil.	-	About 50% of surface has I.G. attack to 0.3 mil.	-	Isolated areas of I.G. attack to 0.4 mil.	-	-	-	No I.G. attack of weld or at junction of weld and base metal.
$BrF_5 + F_2$	7.8	Uniform I.G. attack to 0.8 mil, isolated areas to 1.6 mils.	-	About 10% of surface has I.G. attack to 0.3 mil.	-	Isolated areas of I.G. attack to 0.4 mil.	-	-	-	No I.G. attack of weld or at junction of weld and base metal.
GeF_4	8.0-28.2	No I.G. attack.	About 25% of surface has I.G. attack to 0.8 mil.	No I.G. attack.	No I.G. attack.	Isolated areas of I.G. attack to 0.4 mil.	Isolated areas of attack to 0.5 mil.	Isolated areas of attack to 0.4 mil.	No I.G. attack of weld or at junction of weld and base metal.	No I.G. attack of weld or at junction of weld and base metal.
$GeF_4 + F_2$	7.0-30.0	Uniform I.G. attack to 0.4 mil, isolated areas to 0.8 mil.	About 50% of surface has I.G. attack to 0.8 mil, isolated areas to 1.2 mils.	Isolated areas of I.G. attack to 0.4 mil.	No I.G. attack.	No I.G. attack.	No I.G. attack.	No I.G. attack.	No I.G. attack of weld or at junction of weld and base metal.	No I.G. attack of weld or at junction of weld and base metal.
AsF_5	7.0-7.3	I.G. attack on about 60% of surface to 0.4 mil.	Isolated areas of I.G. attack to 0.4 mil.	Isolated areas of I.G. attack to 0.3 mil.	No I.G. attack.	No I.G. attack.	No I.G. attack.	No I.G. attack.	No I.G. attack of weld or at junction of weld and base metal.	No I.G. attack of weld or at junction of weld and base metal.

TABLE 5 (Contd.)

Corrosive Environment	Reaction Time (hr)	Nickel-200	Nickel-201	Monel	Inconel	Duranickel-301	HyMu-80	INOR-8	Nickel-200 Weld	Nickel-61 Weld
$\text{AsF}_5 + \text{F}_2$	6.9-7.4	Uniform I.G. attack to 0.4 mil, isolated areas to 0.8 mil.	No I.G. attack.	Isolated areas of I.G. attack to 0.4 mil.	No I.G. attack.	No I.G. attack.	No I.G. attack.	No I.G. attack.	No I.G. attack of weld or at junction of weld and base metal.	No I.G. attack of weld or at junction of weld and base metal.
NbF_5	21.8	50% of surface has I.G. attack to 0.3 mil, isolated areas to 1.0 mil.	-	Isolated areas of I.G. attack to 0.3 mil.	-	No I.G. attack.	-	-	-	No I.G. attack of weld or at junction of weld and base metal.
$\text{NbF}_5 + \text{F}_2$	5.1	Uniform I.G. attack to 0.6 mil, isolated areas to 1.2 mils.	-	25% of surface has very broad I.G. attack to 0.4 mil, isolated areas to 0.8 mil.	-	25% of surface has I.G. attack to 0.3 mil.	-	-	-	No I.G. attack of weld or at junction of weld and base metal.
SbF_5	7.2	Very irregular surface; 50% has I.G. attack to 0.8 mil, isolated areas to 1.2 mils.	-	Very broad but fairly uniform I.G. attack to 0.6 mil, isolated areas to 1.5 mils.	-	Isolated areas of I.G. attack to 0.3 mil.	-	-	-	No I.G. attack of weld or at junction of weld and base metal.
$\text{SbF}_5 + \text{F}_2$	7.0	25% of surface has I.G. attack to 0.5 mil, isolated areas to 1.0 mil.	-	Very broad but fairly uniform I.G. attack to 0.6 mil, isolated areas to 1.2 mils.	-	Isolated areas of I.G. attack to 0.4 mil.	-	-	-	No I.G. attack of weld or at junction, but areas of up to 20 mils of weld removed by reaction.
SF_6	28.7-29.2	Uniform I.G. attack from 2.0-4.0 mils, isolated areas to 8.0 mils.	-	50% of surface has I.G. attack to 0.3 mil, isolated areas to 0.7 mil.	10% of surface has I.G. attack to 0.2 mil.	Isolated areas of I.G. attack to 0.3 mil.	Uniform I.G. attack 3.0 to 5.0 mils, isolated areas to 7.0 mils.	No I.G. attack.	-	-
SeF_6	7.0-29.5	Uniform I.G. attack 0.5 to 2.0 mils, isolated areas to 2.5 mils.	-	25% of surface has I.G. attack to 0.2 mil.	No I.G. attack.	No I.G. attack.	No I.G. attack.	No I.G. attack.	-	-
$\text{SeF}_6 + \text{F}_2$	6.6-7.6	Uniform I.G. attack to 0.4 mil, isolated areas to 0.8 mil.	-	No I.G. attack.	No I.G. attack.	Less than 10% of surface has I.G. attack to 0.4 mil.	No I.G. attack.	No I.G. attack.	-	-

TABLE 5 (Contd.)

Corrosive Environment	Reaction Time (hr)	Nickel-200	Nickel-201	Monel	Inconel	Duranickel-301	HyMu-80	INOR-8	Nickel-201 Weld	Nickel-61 Weld
MoF ₆	7.0-9.2	Isolated areas of I.G. attack to 0.4 mil.	-	Isolated areas of attack to 0.4 mil.	No I.G. attack.	Isolated areas of I.G. attack to 0.4 mil.	No I.G. attack.	No I.G. attack.	-	-
MoF ₆ + F ₂	6.0-7.8	Uniform I.G. attack to 0.4 mil, isolated areas to 0.8 mil.	-	Isolated areas of I.G. attack to 0.5 mil.	No I.G. attack.	No I.G. attack.	No I.G. attack.	No I.G. attack.	-	-
TeF ₆	5.5-18.9	Some coupons have no I.G. attack. Others have uniform I.G. attack 0.4 to 1.0 mil with isolated areas to 1.5 mils.	-	No I.G. attack.	No I.G. attack.	No I.G. attack.	No I.G. attack.	No I.G. attack.	-	-
TeF ₆ + F ₂	2.9-7.8	Uniform I.G. attack to 0.4 mil, isolated areas to 1.2 mils.	-	Isolated areas of I.G. attack to 0.4 mil.	No I.G. attack.	No I.G. attack.	Surface very irregular. Uniform I.G. attack of 3 to 6 mils except at areas of high corrosion, where I.G. attack was negligible.	-	-	-

^aI.G. = intergranular.

TABLE 6. Corrosion of Nickel and Nickel Alloys by Fluorine, Uranium Hexafluoride, and Bromine Pentafluoride

Temperature: $500 \pm 10^\circ\text{C}$
 UF_6 and BrF_5 Pressure: 100 ± 20 Torr
 Fluorine Pressure: 575 ± 100 Torr

Corrosion Environment	Time (hr)	Corrosion Rate ^a (mil/hr)																			
		Nickel-200		Nickel-201		Monel		Inconel		Duranickel-301		HyMu-80		INOR-8		Nickel-200 Weld		Nickel-61 Weld		Silver Solder	
F ₂	6.3	-	-	0.001	(0.002)	-	-	-	-	-	-	-	-	-	-	0.002	(0.003)	nil	(nil)	-	-
F ₂	7.0	0.003	(0.002)	-	-	0.001	(nil)	0.044	b	-	-	-	-	-	-	-	-	-	-	-	-
F ₂	7.0	-	-	-	-	-	-	-	-	0.008	(0.006)	0.034	b	0.067	(0.044)	-	-	-	-	-	-
F ₂	7.4	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.064	(0.153)
F ₂	8.6	c	(0.003)	-	-	c	(0.004)	0.138	b	-	-	-	-	-	-	-	-	-	-	-	-
F ₂	26.6	-	-	-	-	-	-	-	-	-	-	-	-	-	-	nil	(0.001)	0.001	(0.001)	-	-
UF ₆	7.2	-	-	0.010	b	-	-	-	-	-	-	-	-	-	-	0.052	b	0.067	b	0.393	b
UF ₆	7.6	-	-	-	-	-	-	-	-	0.008	(0.007)	0.023	(0.036)	0.021	(0.017)	-	-	-	-	-	-
UF ₆	8.0	-	b	-	-	-	(0.008)	-	(0.010)	-	-	-	-	-	-	-	-	-	-	-	-
UF ₆	28.8	0.001	(0.004)	nil	(0.004)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
UF ₆ + F ₂	7.0	0.010	(0.006)	-	-	0.007	(0.008)	0.196	b	-	-	-	-	-	-	-	-	-	-	-	-
UF ₆ + F ₂	7.0	-	-	0.004	(0.004)	-	-	-	-	-	-	-	-	-	-	0.012	b	0.071	b	0.693	b
UF ₆ + F ₂	7.2	-	-	-	-	-	-	-	-	0.017	(0.012)	0.046	b	0.238	(0.157)	-	-	-	-	-	-
BrF ₅	27.8	0.001	(0.001)	-	-	0.001	(0.002)	-	-	0.003	(0.003)	-	-	-	-	-	-	0.002	(0.001)	-	-
BrF ₅ + F ₂	7.8	0.012	(0.005)	-	-	0.001	(0.003)	-	-	0.007	(0.011)	-	-	-	-	-	-	0.024	(0.015)	-	-

^aCorrosion rates were calculated from the weight losses of the coupons following descaling, assuming complete removal of all scale. The numbers in parentheses are rates calculated from the weight gains of the coupons, assuming that only NiF_2 formed uniformly on the surface of the coupon. Rates reported as nil are less than 0.001 mil/hr.

^bExcessive spalling of the corrosion scale; weight-gain data unreliable.

^cCorrosion scale not completely removed by descaling bath.

TABLE 7. Corrosion of Nickel and Nickel Alloys by Germanium Tetrafluoride and by Germanium Tetrafluoride-Fluorine Mixture

Temperature: $500 \pm 10^\circ\text{C}$
 Fission Product Fluoride Pressure: 100 ± 20 Torr
 Fluorine Pressure: 575 ± 100 Torr

Corrosive Environment	Time (hr)	Corrosion Rate ^a (mil/hr)																			
		Nickel-200		Nickel-201		Monel		Inconel		Duranickel-301		HyMu-80		INOR-8		Nickel-200 Weld		Nickel-61 Weld		Silver Solder	
GeF ₄	8.0	-	(0.002)	-	-	-	(0.004)	-	(0.002)	-	-	-	-	-	-	-	-	-	-	-	-
GeF ₄	28.0	-	-	0.001	(0.002)	-	-	-	-	-	-	-	-	-	-	0.002	(0.001)	0.001	(0.002)	0.010	(0.011)
GeF ₄	28.2	-	-	-	-	-	-	-	-	nil	(0.001)	0.001	(0.001)	nil	(nil)	-	-	-	-	-	-
GeF ₄ + F ₂	7.0	-	-	-	-	-	-	-	-	0.007	(0.004)	0.031	b	0.073	(0.045)	-	-	-	-	-	-
GeF ₄ + F ₂	8.4	nil	(0.002)	-	-	0.001	(0.002)	0.130	b	-	-	-	-	-	-	-	-	-	-	-	-
GeF ₄ + F ₂	30.0	-	-	nil	(0.001)	-	-	-	-	-	-	-	-	-	-	nil	(0.001)	0.001	(0.001)	0.017	(0.052)

^aCorrosion rates were calculated from the weight losses of the coupons following descaling, assuming complete removal of all scale. The numbers in parentheses are rates calculated from the weight gains of the coupons assuming that only NiF₂ formed uniformly on the surface of the coupon. Rates reported as nil are less than 0.001 mil/hr.

^bExcessive spalling of the corrosion scale; weight-gain data unreliable.

TABLE 8. Corrosion of Nickel and Nickel Alloys by Selected Group V Fluorides, Individually and with Fluorine

Temperature: $500 \pm 10^\circ\text{C}$
 Fission Product Fluoride Pressure: 100 ± 20 Torr
 Fluorine Pressure: 575 ± 100 Torr

Corrosive Environment	Time (hr)	Corrosion Rate ^a (mil/hr)																			
		Nickel-200		Nickel-201		Monel		Inconel		Duranickel-301		HyMu-80		INOR-8		Nickel-200 Weld		Nickel-61 Weld		Silver Solder	
AsF ₅	7.0	0.001	(0.006)	-	-	nil	(0.004)	nil	(0.002)	-	-	-	-	-	-	-	-	-	-	-	-
AsF ₅	7.2	-	-	b	(nil)	-	-	-	-	-	-	-	-	-	-	0.001	(nil)	0.001	(0.001)	0.212	c
AsF ₅	7.3	-	-	-	-	-	-	-	-	b	(0.002)	0.004	(0.005)	0.001	(0.002)	-	-	-	-	-	-
AsF ₅ + F ₂	6.9	-	-	0.002	(0.002)	-	-	-	-	-	-	-	-	-	-	0.003	(0.002)	0.014	(0.009)	0.104	(0.265)
AsF ₅ + F ₂	7.2	0.002	d	-	-	nil	d	0.202	c	-	-	-	-	-	-	-	-	-	-	-	-
AsF ₅ + F ₂	7.4	-	-	-	-	-	-	-	-	0.010	(0.006)	0.027	c	0.050	(0.031)	-	-	-	-	-	-
NbF ₅	21.8	0.006	c	-	-	0.002	c	-	-	0.001	c	-	-	-	-	-	-	0.007	c	-	-
NbF ₅ + F ₂	5.1	0.004	(0.003)	-	-	b	(0.002)	-	-	0.012	(0.003)	-	-	-	-	-	-	0.004	(0.003)	-	-
SbF ₅	7.2	0.064	c	-	-	0.032	c	-	-	0.007	(0.006)	-	-	-	-	-	-	0.118	c	-	-
SbF ₅ + F ₂	7.0	0.075	(0.076)	-	-	0.021	c	-	-	0.009	(0.015)	-	-	-	-	-	-	0.356	(0.531)	-	-

^aCorrosion rates were calculated from the weight losses of the coupons following descaling, assuming complete removal of all scale. The numbers in parentheses are rates calculated from the weight gains of the coupons, assuming that only NiF₂ formed uniformly on the surface of the coupon. Rates reported as nil are less than 0.001 mil/hr.

^bCorrosion scale not completely removed by descaling bath.

^cExcessive spalling of the corrosion scale; weight-gain data unreliable.

^dWeight-gain data not obtained; all coupons showed a weight loss before descaling.

TABLE 9. Corrosion of Nickel and Nickel Alloys by Selected Group VI Fluorides, Individually and with Fluorine

Temperature: $500 \pm 10^\circ\text{C}$ Fission Product Fluoride Pressure: 100 ± 20 TorrFluorine Pressure: 575 ± 100 Torr

Corrosive Environment	Time (hr)	Corrosion Rate ^a (mil/hr)											
		Nickel-200		Monel		Inconel		Duranickel-301		HyMu-80		INOR-8	
SF ₆	28.7	nil	(nil)	nil	(nil)	nil	(nil)	-	-	-	-	-	-
SF ₆	29.2	-	-	-	-	-	-	b	(nil)	0.001	(0.001)	0.002	(nil)
SeF ₆	7.0	0.001	(nil)	0.001	(0.001)	0.002	(0.002)	-	-	-	-	-	-
SeF ₆	7.2	-	-	-	-	-	-	0.007	(0.007)	0.014	(0.016)	0.012	(0.009)
SeF ₆	29.5	b	(nil)	b	(nil)	b	(0.001)	-	-	-	-	-	-
SeF ₆ + F ₂	6.6	0.001	(0.003)	0.001	(0.005)	0.097	c	-	-	-	-	-	-
SeF ₆ + F ₂	7.6	-	-	-	-	-	-	0.006	(0.004)	0.036	c	0.047	(0.028)
MoF ₆	7.0	-	(0.002)	-	(0.008)	-	(0.005)	-	-	-	-	-	-
MoF ₆	7.0	-	-	-	-	-	-	0.008	(0.010)	0.014	(0.030)	0.009	(0.032)
MoF ₆	9.2	0.001	(0.003)	nil	(0.003)	0.001	(0.002)	-	-	-	-	-	-
MoF ₆ + F ₂	6.0	0.001	(0.001)	nil	(0.002)	0.197	(0.063)	-	-	-	-	-	-
MoF ₆ + F ₂	7.0	b	(0.001)	b	(0.002)	0.284	c	-	-	-	-	-	-
MoF ₆ + F ₂	7.8	-	-	-	-	-	-	0.006	(0.004)	0.022	c	0.033	(0.021)
TeF ₆	5.5	-	-	-	-	-	-	0.269	c	0.542	c	0.201	c
TeF ₆	5.7	0.608	c	0.250	c	0.025	c	-	-	-	-	-	-
TeF ₆	6.9	0.283	c	0.175	c	0.033	c	-	-	-	-	-	-
TeF ₆	18.9	0.040	c	0.008	c	0.011	c	-	-	-	-	-	-
TeF ₆ + F ₂	2.9	-	-	-	-	-	-	0.207	c	0.942	c	0.155	c
TeF ₆ + F ₂	5.3	nil	(0.002)	nil	(0.001)	0.058	c	-	-	-	-	-	-
TeF ₆ + F ₂	7.8	0.001	(0.001)	0.001	(nil)	0.179	(0.095)	-	-	-	-	-	-

^aCorrosion rates were calculated from the weight losses of the coupons following descaling, assuming complete removal of all scale. The numbers in parentheses are rates calculated from the weight gains of the coupons, assuming that only NiF₂ formed uniformly on the surface of the coupon. Rates reported as nil are less than 0.001 mil/hr.

^bCorrosion scale not completely removed by descaling bath.

^cExcessive spalling of the corrosion scale; weight-gain data unreliable.

The welded areas showed no visible evidence of greater corrosion than the nickel-200 portions of the coupons. The Inconel, Duranickel-301, and HyMu-80 coupons were covered with varying amounts of a green-colored scale, whereas the INOR-8 coupons were coated with a very adherent, uniform, gold-colored scale. A thin pink scale had formed on the silver-soldered portions of the coupons. The silver-soldered coupons were the most severely corroded. X-ray diffraction analyses of the scales from Duranickel-301, HyMu-80, and INOR-8 coupons indicated the presence of only NiF_2 . However, X-ray spectral analyses revealed that the elements listed in Table 10 were present in the scales removed from HyMu-80, INOR-8, and silver-soldered coupons.

TABLE 10. X-Ray Spectral Analyses of Scales from HyMu-80, INOR-8, and Silver-soldered Coupons Exposed to Fluorine at 500°C

	HyMu-80	INOR-8	Silver Solder
Major Phase	Ni	Ni	Ag, Cu, Zn
Intermediate Phase	Fe	-	-
Minor Phase	-	Fe, Cr, Mn, Mo (?)	Ni, Fe

Uranium Hexafluoride. All of the alloys were exposed at 500°C to UF_6 individually for 7.2 to 28.8 hr, and in the presence of fluorine for about 7 hr. Although the mechanism of the reaction between UF_6 and nickel is not completely understood, it is believed that thermal decomposition is not a factor, since UF_6 is thermodynamically stable at temperatures well above 1000°C.

In general, when the coupons were exposed to UF_6 alone, the corrosion was quite localized; areas of scale were observed adjacent to relatively unreacted areas. This localized corrosion was substantiated by the fact that about one-half of the surface of some coupons was shown by microscopic examination to contain intergranular penetration, while the remainder of the coupon surface was free of any penetration. Localized corrosion of nickel by UF_6 in the temperature range 500-700°C was also observed by Langlois.¹⁴ X-ray diffraction analysis of the bright green scale from a nickel-200 coupon showed it to be: major, UF_4 ; very minor, possibly NiF_2 . Hale *et al.*¹² found that in the corrosion scales of nickel coupons exposed to UF_6 at temperatures to 982°C the amount of UF_4 present ranged from 10 to 85%, with the remainder being NiF_2 . When fluorine was present with UF_6 the corrosion of the coupons was much more uniform. All of the coupons were coated with a green scale, and only slight spalling was visible on some of the coupons.

Bromine Pentafluoride. The interest in the corrosion of nickel and nickel alloys by BrF_5 is twofold: (1) BrF_5 is a volatile fluoride of fission product bromine and (2) BrF_5 is being considered for use in the fluid-bed fluoride volatility process as a selective fluorinating agent for uranium to separate uranium and plutonium. Nickel-200, Monel, Duranickel-301, and nickel-200 coupons containing areas of nickel-61 weld were exposed to BrF_5 individually and in the presence of fluorine at 500°C for 27.8 and 7.8 hr, respectively. In both experiments the coupons were coated with a dark scale. The scale from a Monel coupon which had been exposed to BrF_5 alone was submitted for X-ray diffraction analysis. It was found that NiF_2 was the major phase present, with possible minor phases of AlF_3 and CuF_2 also present. Both aluminum and copper are constituents of the Monel used in these studies. X-ray spectral analysis of this same scale showed nickel and copper to be present and bromine and iron to be absent.

2. Corrosion by a Group IV Fluoride

Germanium Tetrafluoride. All of the alloys were exposed to GeF_4 individually and in the presence of fluorine at 500°C for 7.0 to 30.0 hr. When GeF_4 alone was the reactant gas, the coupons were coated with a uniform gray-black scale identified by X-ray diffraction analysis as GeO_2 and NiF_2 . The presence of GeO_2 in the corrosion scale is presumed to be due to secondary reaction of the corrosion scale with the moist atmosphere, since it has been shown²⁴ that GeF_4 is readily hydrolyzed at room temperature, producing GeO_2 . When GeF_4 plus fluorine were the reactant gases, the resultant corrosion scales were typical of scales formed in the presence of fluorine alone.

3. Corrosion by Selected Group V Fluorides

Arsenic Pentafluoride. All of the alloys were exposed to AsF_5 individually and in the presence of fluorine at 500°C for 6.9 to 7.4 hr. In the presence of AsF_5 alone, nickel-200, nickel-201, Duranickel-301, INOR-8, and both types of nickel weld were only darkened by the exposure; however, dark-colored corrosion scales were formed on the Inconel, HyMu-80, and silver-soldered coupons. The scale from a HyMu-80 coupon was shown by X-ray diffraction analysis to be mainly NiF_2 , with an unidentified minor phase. When fluorine was present with AsF_5 , the resultant corrosion scales were typical of scales formed in the presence of fluorine alone.

Niobium Pentafluoride. Nickel-200, Monel, and Duranickel-301, as well as nickel-200 coupons containing areas of nickel-61 weld, were exposed at 500°C to NbF_5 individually for 21.8 hr and in the presence of fluorine for 5.1 hr. When NbF_5 was the reactant gas, no visible evidence of reaction was observed. However, when fluorine was present with NbF_5 , the resultant corrosion scales were typical of scales formed in the presence of fluorine alone.

Antimony Pentafluoride. Nickel-200, Monel, and Duranickel-301, as well as nickel-200 coupons containing areas of nickel-61 weld, were exposed to SbF_5 individually and in the presence of fluorine at 500°C for 7.2 and 7.0 hr, respectively. The nickel-200, Monel, and nickel-61 welded coupons were quite severely corroded by SbF_5 , and were uniformly coated with a scale that varied in color from green to black for nickel and green to violet for Monel. X-ray diffraction analyses indicated that only NiF_2 was present in the green scale from the nickel-200 coupon and that NiF_2 , plus an unidentified minor phase was present in the violet scale from the Monel coupon. X-ray spectral analysis of the violet scale showed nickel to be the major element present, with copper present in smaller amounts. No antimony was detected in the violet scale. The Duranickel-301 coupons exposed to SbF_5 were the least corroded of any specimens exposed to SbF_5 and were uniformly covered with a dark scale.

When fluorine was present with SbF_5 , the coupons were generally coated with a green scale. Figure 1 is a photomicrograph of a Monel coupon after exposure to SbF_5 plus fluorine for 7 hr at 500°C , showing the typically broad and uniform intergranular penetration observed; in some areas at the coupon surface, the breadth measured 0.4 mil. The welded portions of coupons containing areas of nickel-61 weld after exposure to SbF_5 plus fluorine were very severely corroded, with a lateral outgrowth of scale evident. Microscopic examination of the welds after descaling revealed that in some areas the weld had reacted to a depth of 20 mils. Figure 2 shows a nickel-61 welded coupon before and after exposure to this environment.

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Fig. 1. Photomicrograph of a Monel Coupon Exposed to SbF_5 plus Fluorine for 7 hr at 500°C

Fig. 2. Photomicrograph of a Nickel-200 Coupon Containing Nickel-61 Weld before and after Exposure to SbF_5 plus Fluorine for 7 hr at 500°C

4. Corrosion by Selected Group VI Fluorides

Sulfur Hexafluoride. Nickel-200, Monel, Inconel, Duranickel-301, HyMu-80, and INOR-8 coupons were exposed to SF_6 at 500°C for 28.7 and 29.2 hr. Although sulfur is not a fission product, exposure of metals to SF_6 permits comparison of the rates of corrosion and intergranular penetration of nickel alloys by some of the Group VIA (S, Se, Te) hexafluorides. The surfaces of all of the coupons were only slightly dulled as a result of the exposure to SF_6 , and negligible corrosion rates were obtained. Intergranular penetration of many of the alloys was appreciable, however.

Selenium Hexafluoride. Nickel-200, Monel, Inconel, Duranickel-301, HyMu-80, and INOR-8 coupons were exposed at 500°C to SeF_6 alone for 7.0 to 29.5 hr, and to SeF_6 in the presence of fluorine for 6.6 and 7.6 hr. When SeF_6 alone was the reactant gas, all of the coupons were coated with a thin black scale, with a few areas of white scale present mainly around the edges of the coupons. X-ray spectral analysis of this scale from a

nickel-200 coupon showed no detectable concentrations of selenium. When SeF_6 plus fluorine were the reactant gases, the resultant corrosion scales were typical of scales formed in the presence of fluorine alone.

Molybdenum Hexafluoride. Nickel-200, Monel, Inconel, Duranickel-301, HyMu-80, and INOR-8 coupons were exposed to MoF_6 individually and in the presence of fluorine at 500°C for 6.0 to 9.2 hr. When the coupons were exposed to MoF_6 , a thin gray-black scale formed on the surface of all the coupons. A few of the coupons had areas where a black crystalline solid had formed, which was shown by X-ray diffraction analysis to be probably a rhombohedral form of MoF_3 . When the coupons were exposed to MoF_6 plus fluorine, the resultant corrosion scales were typical of scales formed in the presence of fluorine alone.

Tellurium Hexafluoride. Nickel-200, Monel, Inconel, Duranickel-301, HyMu-80, and INOR-8 coupons were exposed at 500°C to TeF_6 individually for 5.5 to 18.9 hr, and in the presence of fluorine for 2.9 to 7.8 hr. When TeF_6 alone was the reactant gas, all of the coupons were severely corroded and covered with scales varying in color from gray to black. At the conclusion of the 18.9-hr experiment (see Table 9), it was observed that the coupons were held in place in the heated zone by a light gray solid which had completely filled the nickel reaction vessel. The uppermost coupon (nickel) was free of any scale but had lost nearly half of its weight. The next lower coupon (Monel) showed extreme lateral outgrowth of a layered black scale from all six surfaces. Small, shiny, metallic-appearing crystals were evident on all surfaces. The remaining eight coupons showed lesser degrees of corrosion, including many areas where spalling of the scale was evident. The corrosion rates for this experiment listed in Table 9 exclude data from the top two coupons. X-ray diffraction analyses of the solid reaction products are listed in Table 11.

The buildup of solid reaction products in the vessel, which eventually engulfed the coupons, may have contributed to the decrease in corrosion rates with distance from the top of the reactor. For this reason a modified nickel reactor was constructed which contained a significant volume at the bottom, located outside the heated zone; the remaining experiments involving TeF_6 were carried out in this vessel. During the experiment, the solids which formed collected in this reservoir, allowing the reactant gases to reach the coupons more easily. Nickel-200, Monel, and Inconel coupons were exposed to TeF_6 at 500°C for 5.7 hr. Figure 3 is a composite photograph showing the coupons before and after exposure. Although the coupons nearest the top of the reactor again showed the greatest amount of corrosion, the difference in rates from top to bottom was not nearly as marked as previously observed. The nickel-200 support wire was free of scale but had obviously reacted, as evidenced by the irregular surface and the decrease in diameter of the wire. The nickel-200 wires interconnecting the coupons were badly corroded and covered with a

TABLE 11. X-Ray Diffraction Analyses of Solids from the Reaction of TeF_6 with Nickel-200, Monel, and Inconel

Temperature: $500 \pm 10^\circ\text{C}$
 TeF_6 Pressure: 100 ± 20 Torr
 Duration of Experiment: As indicated

Sample Identification	Analyses of Solids	
	18.9-hr Experiment	5.7-hr Experiment
Gray scale from surface of nickel coupon	NiF_2 , NiTe_2 , and NiTe	NiF_2 and NiTe_2
Black scale from surface of Monel coupon	Major: Cu_{2-x}Te Minor: possibly NiTe_2 , CuTe (36.1-36.4% Te), and NiF_2	Major: NiTe_2 and Cu_{2-x}Te Minor: probably NiF_2 and CuTe
Black scale from surface of Inconel coupon	Major: NiF_2 Minor: NiTe_2 and possibly CrF_3	Major: NiF_2 and NiTe_2 Minor: possibly CrF_3
Metallic-appearing solid found on surface of Monel coupon	CuTe , Te , and possibly NiF_2	-
Metallic-appearing solid found on nickel support wire	-	Tellurium metal only

Fig. 3. Corrosive Effect of TeF_6 on Nickel, Monel, and Inconel Coupons-- 500°C for 5.7 hr

gray scale. A metallic-appearing substance had condensed onto the nickel support wire just outside the hot zone. X-ray diffraction analysis showed this substance to be tellurium metal. The source of this metal has not been established, but possibly it is a product of the disproportionation of TeF_4 and vaporized out of the hot zone. The vapor pressure of tellurium at 500°C is 0.9 Torr.²⁵ TeF_4 has been shown¹⁹ to be unstable above 200°C , which is substantiated by the fact that no tellurium fluoride has been found in this study in any of the corrosion scales of coupons exposed to TeF_6 at 500°C .

That Monel coupon from the 18.9-hr experiment which showed the greatest amount of corrosion was mounted in copper-impregnated Bakelite and submitted for electron-probe analysis. This analysis consisted of both continuous traces from the Monel matrix across the corrosion layer, and fixed-point measurements at various areas along the first trace. The two types of analyses were combined to identify the various layers. A photomicrograph of this scale is shown in Fig. 4. The

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Fig. 4. Photomicrograph of a Monel Coupon Exposed to TeF₆ for 18.9 hr at 500°C Showing Various Corrosion Layers as Identified by Electron-probe Analysis

extensive black scale, approximately 1200 microns thick, was identified as mostly NiF_2 . Interspersed in this scale were numerous violet-colored layers tentatively identified as Cu_4Te_3 with a 0.95 w/o nickel impurity. The copper-tellurium phase diagram shows anomalous behavior and is not clearly defined.²⁶ A white band located about 100 microns from the coupon surface was identified as a mixture of copper and nickel tellurides. A 50-micron-wide, metallic-lustered band adjacent to the base metal was shown to be NiTe_2 with traces of copper and iron impurities. No tellurium metal was observed to have diffused into the Monel coupon. The metallic-appearing crystals imbedded in the surface of the black scale were colored rose to blue-gray and were identified as CuTe . The copper and tellurium concentrations varied by less than 0.5 w/o from one color region to another. The colors may have been due to trace impurities of manganese, iron, or nickel which were not detected. A gray coating surrounding the CuTe crystals was shown to be metallic tellurium.

When the coupons were exposed to TeF_6 plus fluorine, the nickel-200 and Monel coupons, which had shown the greatest amount of corrosion when exposed to TeF_6 alone, were only slightly discolored and showed very little visible evidence of reaction. Similar reduction in rates was not observed with Inconel, Duranickel-301, HyMu-80, and INOR-8 coupons, which were severely corroded and coated with gray scales. The Inconel and HyMu-80 coupons were uniformly corroded, whereas the Duranickel-301 and INOR-8 coupons showed evidence of localized corrosion.

C. Discussion and Conclusions

If the behavior of nickel-rich alloys is similar to the behavior of nickel,^{1,4} it can be assumed that rates of corrosion derived from short-term experiments with nickel-rich alloys are considerably greater than rates that would be derived from long-term experiments. For this reason, data such as those listed in Tables 6, 7, 8, and 9 would represent maximum corrosion rates for the indicated metals. The course of intergranular corrosion as a function of time is not as well defined, and the data of Table 5 may correspond to rates that would be observed in long-term experiments.

The average rates of corrosion of nickel-200 and nickel-201 by fluorine at 500°C obtained in this study are 0.0027 and 0.0015 mil/hr, respectively, values in excellent agreement with data obtained by Jarry and co-workers,³ namely, 0.0024 and 0.0017 mil/hr for nickel-200 and nickel-201, respectively, for equal-length exposures at 500°C. Jarry *et al.*¹ and Steunenberget *et al.*⁴ exposed nickel-200 to fluorine at 500°C for 33 and 50 hr, respectively. If corrosion rates after 8-hr exposures are calculated from the kinetic data of these longer-term experiments, a rate of 0.0011 mil/hr is obtained for both sets of experiments.

Myers and DeLong⁸ exposed nickel, Monel, and Inconel specimens to fluorine at 4-15 hr at 500°C and obtained corrosion rates of 0.007, 0.003, and 0.086 mil/hr, respectively. These values compare to the results obtained in this study of 0.0027 mil/hr for nickel-200, 0.004 mil/hr for Monel, and 0.138 mil/hr for Inconel.

Langlois¹⁴ exposed nickel, Monel, and Inconel to UF_6 at pressures of 240 Torr and obtained the following corrosion rates: nickel, 0.00008 mil/hr at 425°C and 0.002 mil/hr at 535°C; Monel, an extrapolated rate of 0.001 mil/hr at 500°C; and Inconel, 0.067 mil/hr at 500°C. The rates obtained in the present work for the corrosion of nickel-200, Monel, and Inconel by UF_6 at 100 Torr and at 500°C are 0.004, 0.008, and 0.010 mil/hr, respectively.

A comparison of the results obtained in this study for nickel-200 and nickel-201 shows very little, if any, difference in corrosion rates. However, microscopic examination reveals that nickel-201 shows considerably less intergranular penetration than does nickel-200 when exposed to similar environments.

Nickel-200 and Monel exhibit the best overall resistance to corrosion by the volatile fission product fluorides individually and in the presence of fluorine at 500°C. Although the rates of corrosion of nickel-200 and Monel by TeF_6 alone at 500°C are relatively high, the presence of fluorine with TeF_6 significantly reduces these rates. This rate reduction in the presence of fluorine may be the result of the refluorination of decomposition products by fluorine back to TeF_6 . It should be noted that the pressure of TeF_6 used in this study was about 100 Torr, whereas the TeF_6 pressure expected in the fluid-bed reactor under process conditions is considerably less than 1 Torr. SbF_5 alone and with fluorine cause moderate corrosion of nickel-200 and Monel at 500°C. The rate of corrosion of Monel, however, is lower by about a factor of three than that of nickel-200. Of the metals examined, Duranickel-301 has the best resistance to SbF_5 plus fluorine at 500°C, the corrosion being lower by a factor of two than that of Monel. Nevertheless, the high rate of corrosion of Duranickel-301 in the presence of TeF_6 individually or with fluorine, plus the fact that the resistance of Duranickel-301 to fluorine alone is lower by at least a factor of two than that of either nickel-200 or Monel will limit the usefulness of Duranickel-301. Inconel, HyMu-80, and INOR-8 must be considered of questionable value for process considerations at this time because of their high rates of corrosion by fluorine and by TeF_6 . Nickel-200, Monel, and Duranickel-301 show excellent resistance to BrF_5 at 500°C. However, when fluorine is present with BrF_5 , Monel corrodes at a rate which is two to three times lower than either nickel-200 or Duranickel-301, and Monel is therefore considered to be superior under these conditions.

The corrosion rates of the two types of nickel weld, namely, nickel-200 and nickel-61 weld, in the presence of fluorine at 500°C are low. This low rate is contrary to data obtained by Chilenskas and Gunderson¹¹ who

observed that in fluorine at 475°C, nickel-61 weld corroded at a rate about 750 times faster than nickel-200 weld. In an attempt to resolve this discrepancy in rates, various reacted specimens containing areas of nickel-61 weld were obtained from Chilenskas. These samples, whose welded portions had shown a high rate of corrosion when exposed to fluorine/nitrogen at 500°C for 480 hr, along with coupons containing areas of nickel-61 weld from this study which had been exposed to fluorine at 500°C for about 27 hr, were submitted for electron-probe analysis. Unexposed coupons from the two studies were also included for analysis to be used as a reference. The samples were scanned for titanium, aluminum, iron, and manganese, which are the major constituents of a nickel-61 welding rod other than nickel. The results of the analysis showed that there was no detectable difference in the elemental concentration of the exposed and unexposed coupons from both studies either (1) across the cross section of the weld, or (2) immediately into the base metal adjacent to the weld. In the present experiments, nickel-200 weld corroded at a lesser rate than nickel-61 weld when exposed to AsF_5 or UF_6 in the presence of fluorine. Although the corrosion resistance of the two weld materials generally appears comparable, the data from UF_6 and AsF_5 experiments suggest that welds made from nickel-200 filler metal may be considered superior to those made from nickel-61 filler metal. The corrosion resistance of silver solder to fluorine and most fluorides at elevated temperatures is extremely poor, and equipment which

will see service in the presence of corrosive fluorides at high temperatures should be of welded construction.

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Fig. 5. Comparison of Intergranular-penetration Rates of Nickel-200, Monel, and Inconel at 500°C by SF_6 , SeF_6 , and TeF_6

A comparison of the intergranular penetration caused by SF_6 , SeF_6 , and TeF_6 on nickel-200, Monel, and Inconel at 500°C is shown in Fig. 5. The effect of sulfur on nickel has long been recognized to be severe although the chemistry of the intergranular attack is undefined. The data illustrated in Fig. 5 would lead to the conclusion that, although the effect of SF_6 is significant, TeF_6 attacks nickel-200, Monel, and Inconel to a lesser degree (based on rates of intergranular penetration) than does either SeF_6 or SF_6 . Since TeF_6 , unlike SF_6 and SeF_6 , causes corrosion of nickel-200, Monel, and Inconel by surface reaction to form NiF_2 and tellurides of nickel and copper, concern about intergranular penetration seems unwarranted.

Generally, the corrosion rates of nickel and nickel alloys by fluorine at 500°C were not increased when a fission product fluoride was also present. However, a decrease in the rate of corrosion of nickel-200 and Monel by TeF_6 was observed when fluorine was present with TeF_6 at 500°C.

IV. SUMMARY

Rates of corrosion of nickel and nickel alloys by fluorine, UF_6 , and selected volatile fission product fluorides at 500°C have been obtained. The method employed consisted of exposing representative coupons, in a static system, to the fluorides either individually or in the presence of fluorine. Corrosion rates were calculated in two manners: (1) from the weight losses of the coupons after descaling in an equimolar KNO_3 - NaNO_3 bath at 500°C for 0.5 hr, assuming complete removal of all corrosion scale, and (2) from the weight gains of the coupons after exposure to the corrosive environment, assuming that only NiF_2 formed uniformly on the coupon surface. Intergranular penetration was determined microscopically.

Nickel-200 and nickel-201 showed similar corrosion rates; however, in most instances, nickel-200 exhibited considerably more intergranular penetration than nickel-201. Nickel-200 and Monel exhibited the best overall resistance to corrosion by the volatile fission product fluorides, individually and in the presence of fluorine at 500°C. Although TeF_6 caused fairly extensive corrosion of these two metals, the rate of corrosion by TeF_6 was significantly reduced when fluorine was present. SbF_5 alone and with fluorine caused quite severe corrosion of nickel-200 and Monel at 500°C. The rate for Monel, however, was lower by a factor of two to three than that for nickel-200. Duranickel-301 had the best resistance to SbF_5 plus fluorine, the corrosion rate being lower by a factor of two than that for Monel. The high rate of corrosion of Duranickel-301 in the presence of TeF_6 , individually or with fluorine, plus the fact that the resistance of Duranickel-301 to fluorine alone was lower by at least a factor of two than that of either nickel-200 or Monel, will limit the usefulness of this alloy. Inconel, HyMu-80, and INOR-8 must be considered of questionable value for process considerations at this time because of their high rates of corrosion by fluorine and by TeF_6 .

In the present work, nickel-200 coupons containing areas of either nickel-200 weld or nickel-61 weld showed similar low corrosion rates at 500°C, which should make either type weld acceptable for use under fluorinating conditions at temperatures up to 500°C. Silver solder has shown extremely poor resistance to fluorine and to most of the fluorides examined at 500°C, and should not be used in equipment which will be exposed to these

corrosive fluorides at elevated temperatures. Generally, no enhancement of the corrosion rates of nickel and nickel alloys at 500°C was observed when fluorine was present with the fission product fluorides during exposure.

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