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CORROSION OF METALS AND ALLOYS BY FLUORINE

by
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CORROSION OF METALS AND ALLOYS BY FLUORINE AT TEMPERATURES BELOW 1000°F

R. B. Jackson

SUMMARY

The corrosion of twenty-five metals and alloys by liquid and gaseous fluorine was studied. The liquid corrosion tests were performed at -320°F and exposure ranged from 5 hours to 3 days. Exposures to gaseous fluorine were made at approximately 80°, 400°, 700° and 1000°F. Exposure times were generally 5, 24 and 120 hours. Several specimens were also exposed to gaseous fluorine at elevated pressures and temperatures for 24 hours. Selected specimens were sectioned and photomicrographs taken to study the passivating fluoride film.

This work was undertaken to aid in the selection of materials of construction for handling fluorine in the Air Force's missile program. Results of the five-hour tests indicate that all tested materials, except tantalum, may be exposed to fluorine at temperatures up to 400°F without exceeding a corrosion rate of one-half thousandth of an inch (.0005") per hour. This arbitrarily chosen rate is not intended to signify acceptability. It is equivalent to 4.4 inches per year, which is an extremely high corrosion rate under normal circumstances, but not excessive when exposure for only a few hours is required.

The 24-hour and five-day tests indicate that extrapolation of the results obtained from very short exposures to long time service, such as a year, is not reliable. In general, the corrosion rates tend to decrease as the exposure period is increased. Some exceptions to this have been noted at various temperatures. Aluminum 1100 and Stainless Steel 304L both show higher rates for five-day exposures as compared to shorter exposures.

Tests of materials at elevated pressures indicate that corrosion rates are generally higher than specimens exposed to the same temperatures at atmospheric pressure. However, at 400°F this difference is not significant.

Microscopic examination of selected specimens shows that materials such as monel and nickel form a relatively uniform passivating film at elevated temperatures. Aluminum 1100, however, shows indication of deep, non-uniform penetration. These and other materials will be discussed more fully in the following pages.

A review of subject literature has been made and pertinent comparative data has been compiled. Steindler and Vogel[1] list data by several investigators which are shown in Table 10. Also shown are data from a memorandum by the Battelle Memorial Institute.

I. EXPERIMENTAL METHODS AND DATA

A. Apparatus and Procedure for the Study of the Corrosion of Materials in Liquid Fluorine

The apparatus used for testing in liquid fluorine is shown in Figure 1. Lines for the flow of fluorine were of stainless steel pipe. All fittings were back-welded to the pipe. The liquid fluorine container was constructed from 4-inch stainless steel pipe, and was fitted with a flanged cover. The Dewar flask for the liquid fluorine was supported on a lift platform. The liquid fluorine container was independently supported. After test specimens were prepared and weighed, they were placed on the test rack using monel spacers. Next, the liquid fluorine container and the transfer lines were evacuated to approximately 1-inch Hg abs.; the Dewar flask was raised into position and charged with liquid nitrogen; fluorine was fed into the test chamber and condensed during a 30-minute period. Conditions in the test chamber during the five-hour test period were -320°F and 20-inches Hg abs. In the first two runs, the high purity cylinder fluorine used was not passed through the sodium fluoride absorber. In the third run, however, fluorine from the electrolytic cell was used, and purification was accomplished by passing it through the sodium fluoride absorber. At the end of a test period, the liquid nitrogen bath was lowered and the fluorine allowed to vaporize off. This required approximately 30 minutes. Finally, the entire system was purged with gaseous nitrogen; the test specimens were removed, dried and weighed.

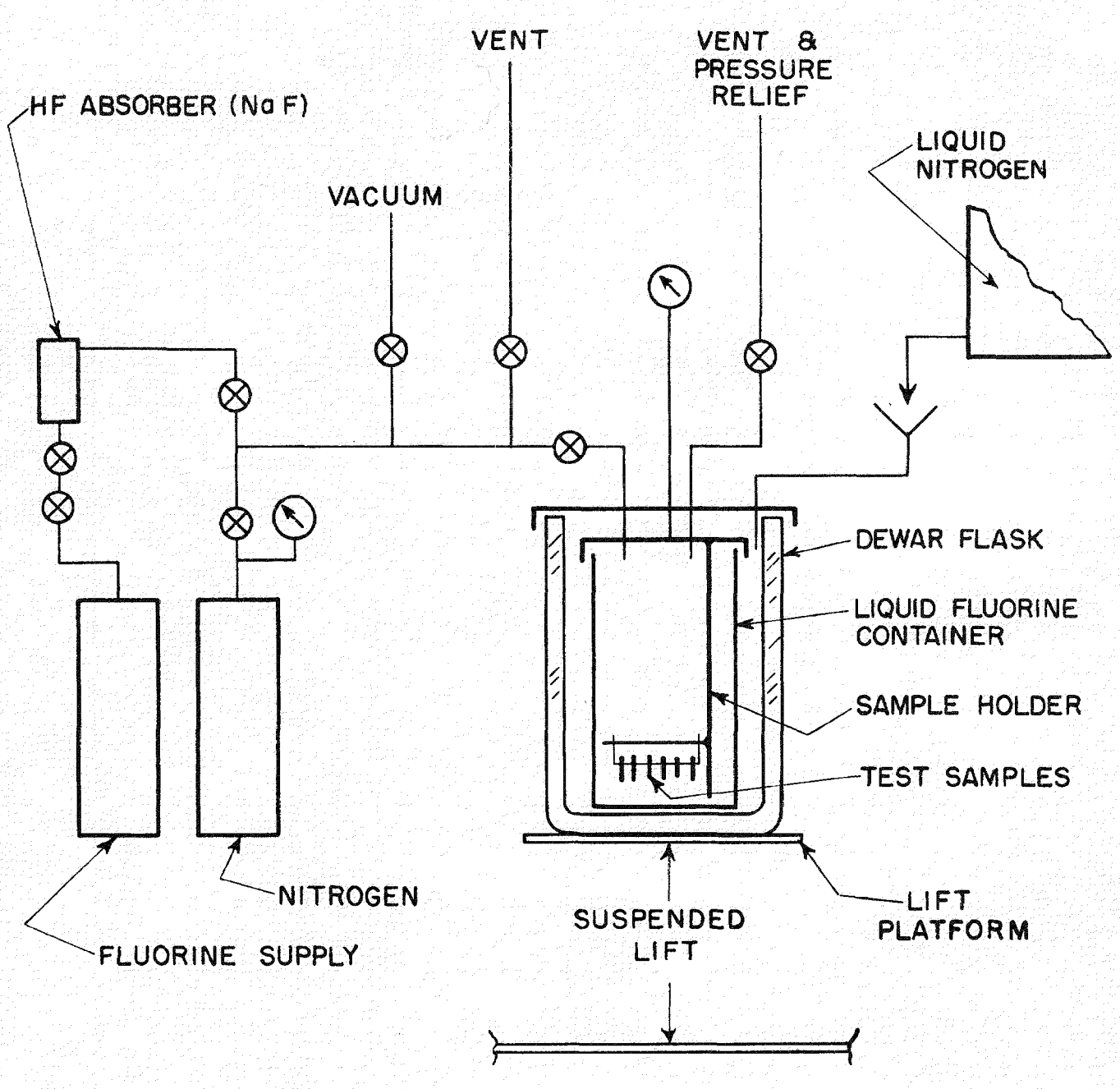

FIGURE 1.

APPARATUS FOR THE STUDY OF CORROSION OF MATERIALS IN LIQUID FLUORINE

B. Apparatus and Procedure for the Study of Corrosion of Materials in a Stream of Gaseous Fluorine

The apparatus used for this portion of the study is shown in Figure 2. Fluorine flow lines were made of copper tubing with flared connections, brass fittings and stainless steel needle valves. An absorber containing sodium fluoride was placed in the line following the fluorine cylinder to remove traces of hydrofluoric acid from the fluorine supply (General Chemical commercial fluorine, 6 lb. cylinder). The pelletized sodium fluoride used in the absorber was regenerated after each fluorine cylinder replacement. Pyrex glass apparatus was employed for the flowmeter, overpressure device, traps, fluorolube bubblers and fluorine absorber. An aluminum float was used in the flowmeter. Furnace tubes were 1-inch nickel pipe, 26 inches long, threaded at the ends, with nickel cap closures. Small diameter nickel tubing was used for the thermocouple wells, which were arranged so that they passed through the long run of brass tees which were in turn threaded into the head end nickel caps. The fluorine flow was introduced into the short stem run of the tees and entered the furnace tubes after passing through the annular spaces between the inside of the tees and the outside of the thermocouple wells. The furnace was designed and constructed to meet the requirements of this investigation. The exit fluorine stream was diluted with nitrogen (not shown) before entering the potassium hydroxide absorber, so as to prevent caking and blockage in the portion of the tube below the liquid level.

When it was desired to conduct tests at temperatures higher than room temperature, the furnace tubes were sometimes heated overnight with an inert gas purge. Prepared duplicate specimens were placed in the center of each tube by means of a small rake.

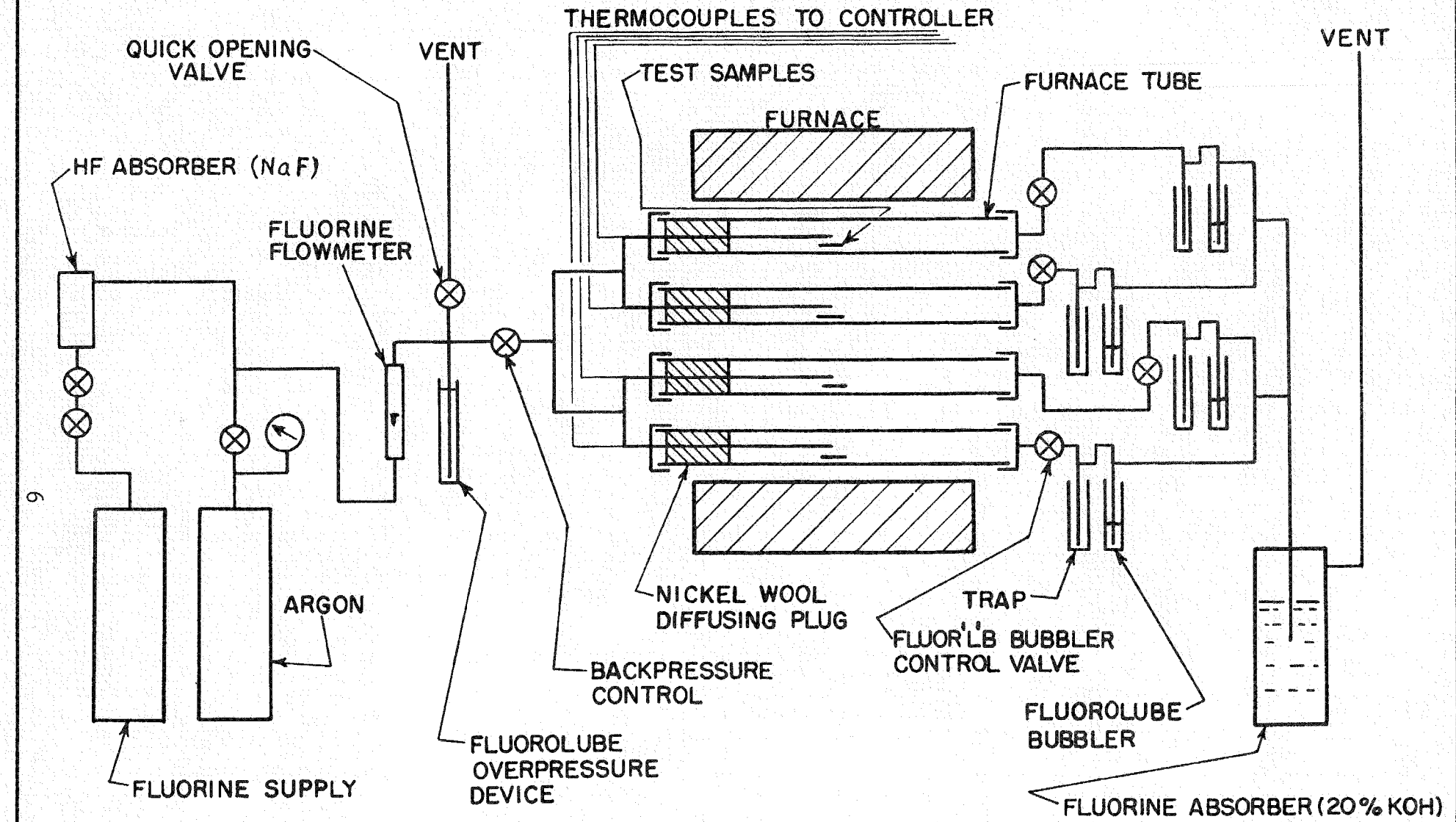

FIGURE 2.

APPARATUS FOR THE STUDY OF CORROSION OF MATERIALS IN A STREAM OF FLUORINE

The system was flushed with an inert gas, 100 cc per minute through each tube, for one half hour. Argon was used as a purge gas for the five-hour tests. Nitrogen was substituted for the other gaseous fluorine tests. A manifold system was used to obtain approximately equal flow through each tube; the equalization was controlled by means of the needle valves and the fluorolube bubblers at the furnace tube exits. Following the flushing period, the gas flow was stopped and a similar rate of fluorine passed through the tubes for the test period. At the end of the test period, the heat was turned off and the tubes were flushed with either argon or nitrogen for one hour or until the exit was free of fluorine. Finally, the specimens were removed, cooled and weighed. Some specimens from the five-hour tests were cleaned and reweighed. Cleaning became necessary when it was not possible to obtain what was considered to be an accurate weight after exposure. It consisted of washing with water, moderate brushing and loose particle removal. Specimens from the longer tests were not cleaned because of the desire to examine the film. A few additional points worthy of mention are: a vent line with quick opening needle cock was positioned before the furnace in the event that it became necessary to quickly vent the system; all unabsorbed gases were vented to the hood exhaust system; Fel-Pro high temperature thread compound was used on all threaded pipe joints; thin wall Teflon tubing was used to make connections between copper and glass tubing.

C. Apparatus and Procedure for the Study of Corrosion of Materials in Gaseous Fluorine Under Pressure

The apparatus used for this portion of the study is shown in Figure 3. The pressure vessel was a 2-inch O.D. single-ended nickel Kuentzel bomb. The 1000 lb. pressure gauge was calibrated

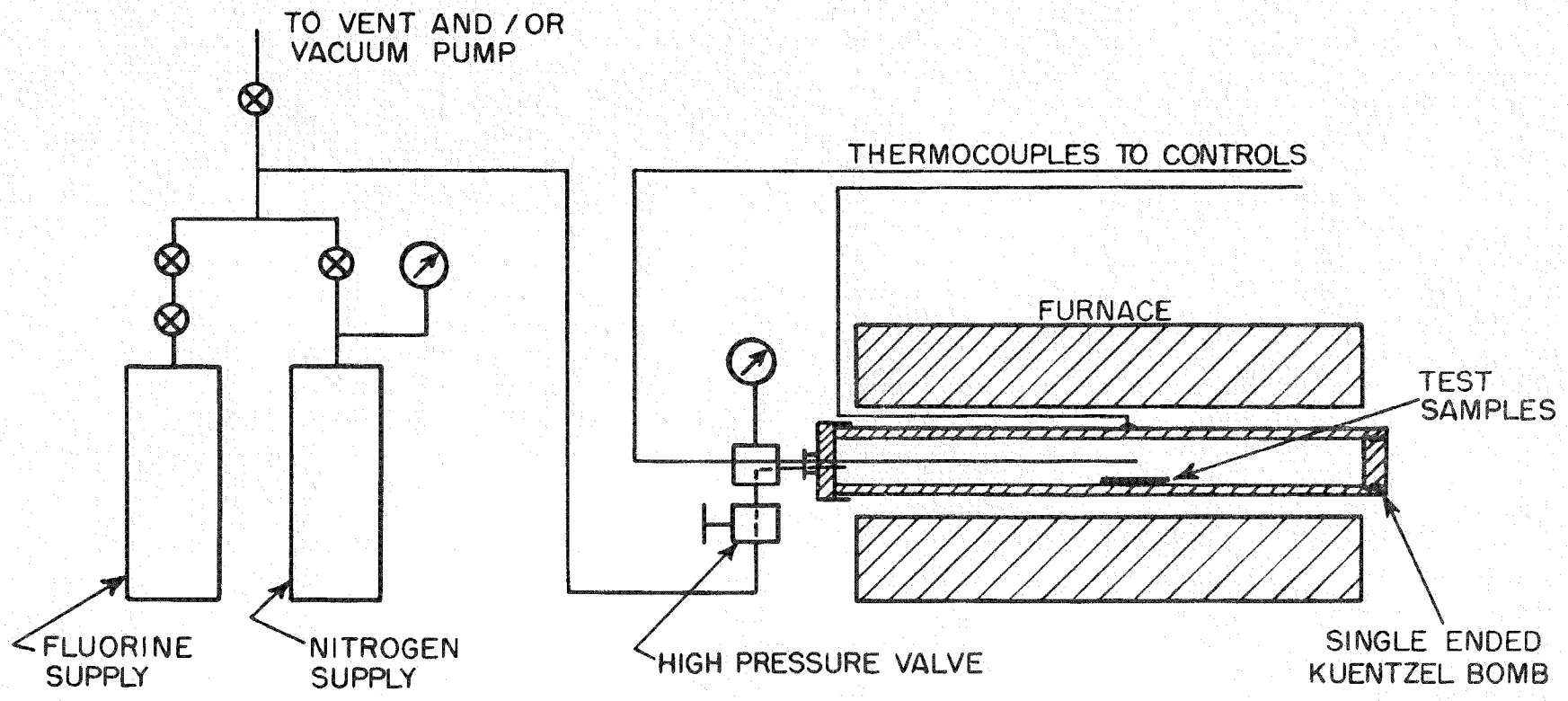

FIGURE 3.

APPARATUS FOR THE STUDY OF CORROSION OF MATERIALS IN FLUORINE UNDER PRESSURE.

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in 10 lb. subdivisions and was equipped with a monel Bourdon tube. All high pressure fittings and connections were nickel. The lines from the cylinders to the high pressure valve were 1/4" copper with flare fittings. The other valves shown in the drawing were monel needle valves. The bomb was charged with fluorine at elevated pressures. Various units were omitted from the system to facilitate this transfer. Units removed were the scrubber, rotameter and overpressure device. Actual procedure consisted of placing the specimens in the center of the bomb before sealing. Although thread compounds (Fel-Pro and Silver Goop) were used to prevent seizing, the threads were stripped in each run at 1000°F when the bomb was opened. The sealed bomb and lines were flushed with nitrogen and then evacuated with a pump. After pumping the system down, it was charged with fluorine to 100 pounds and the fluorine was then bled out. This fluorine flush was repeated twice again and the bomb was then charged with fluorine for the run. The charging pressure was such that the bomb when heated to the desired temperature would have a pressure of approximately 250 lbs. gauge. As a precaution, two thermocouples were used. A pencil type thermocouple was inside the bomb above the center of the specimens. The second thermocouple was in contact with the outside wall of the bomb directly opposite the pencil thermocouple. The bomb was fitted inside a 2" top opening multiunit electric furnace which facilitated rapid heating and cooling. Heating time never exceeded 1 hour and cooling time was only slightly longer for the 1000°F runs. Upon completing the run (all runs were 24 hours) the furnace was shut off and the bomb removed and cooled. Generally, the fluorine was bled out when the temperature was less than 200°F When the pressure had dropped to zero on the gauge, the system was pressurized with

nitrogen. The nitrogen was then bled out. This was repeated several times until the fluorine in the exit gas was very low. The system was then evacuated using a water aspirator and then brought back to atmospheric pressure with nitrogen. The bomb was then opened and the specimens removed, cooled and weighed. As mentioned previously, severe difficulty was noted in opening bombs exposed at 1000°F. Each of the three bombs was badly damaged and the work was curtailed as a result. One bomb was actually cut open to remove the specimens. Since the bombs were made especially for this job and delivery was in excess of three months, it was felt that replacements were not warranted. Very poor life was also obtained from the nickel sheathed pencil thermocouples. The tips burned out after one run at 1000°F. The process whereby the sheath end was sealed either degraded the metal causing excessive corrosion or the lack of proper annealing was responsible.

D. Procedure for Mounting and Preparing Specimens for Microscopic Examination

Selected specimens were cut in two and mounted between two pieces of a similar material. This sandwich was then encased in Bakelite using a Fisher Press. The mounting was polished on a Buehler metallurgical polishing belt using successive belts of No. 120, 180, 240 and 320 grit. Specimens were next polished by hand on emery polishing papers ranging from No. 0 through No. 4/0. Final polishing was performed on a Fisher Metallographic Polisher using 6 micron and 1/2 micron diamond dust, successively, as an abrasive. Polished specimens were examined and film thicknesses measured and calculated. Results are listed in Table 9. Photographs of these specimens may be found at the end of this report. It should be noted that most of the specimens exposed at low temperatures have negligible films and were, therefore, not

included in this study. Specimens were examined by means of a Leitz Panphot. All photomicrographs were 1000 X magnification before reproduction.

E. Data

A summary of the data obtained in this work is presented in the following tables. Table 1 represents corrosion rates for five-hour exposures. Table 2 shows results of 24-hour exposures. Table 3 represents five-day exposures and Table 4, the 24-hour tests at elevated pressure. The appearance of the test specimens after exposure to fluorine are noted in the "A" series of tables. For example, Table 1A lists the appearance of the 5-hour exposure specimens.

II. DISCUSSION

A. Results of this Investigation

Factors used in calculating corrosion or penetration rates from weight changes, and the analysis, density and the pickling procedure for each metal or alloy, are shown in Table 11. Results of this study are shown in Tables 1 to 4. Factors used in calculating these results are shown in Tables 12 and 14, while other pertinent information will also be found in Tables 13 and 15. Corrosion results were calculated by applying the proper weight change factor to the data in Tables 1 to 4. In the majority of cases, weight changes were positive, due to the formation of a film scale or other coating. Since removal of films and coatings would, in most cases, have presented a very difficult problem, the weight gain method of calculating corrosion results was used. A problem of interpretation arises when both volatile and non-volatile fluorides are formed: such formation obviously causes simultaneous weight increases and decreases. Nevertheless, the method of calculating results is believed to

be both practical and reliable. Steindler and Vogel[1] used this method in investigating the corrosion of materials in the presence of fluorine at elevated temperatures. Additional information concerning the appearance of the test specimens after exposure to fluorine may be obtained by referring to Tables 1A to 4A. The corrosion results have not been presented in graphical form because, in most cases, there was insufficient data to allow us to draw meaningful curves. In order to simplify discussion of the results, the materials are grouped in such a way as to reflect their relative corrosion resistance to fluorine. Results on duplicate specimens were taken from Table 1, averaged and recompiled in Tables 5 and 8. Data for liquid fluorine are shown in Table 5, and for gaseous fluorine in Table 8.

1. Resistance to Liquid Fluorine

The work on liquid fluorine corrosion was carried out in two stages. Results from the two are not consistent; to avoid confusion, they are described separately.

a) First Set of Experiments

In the first stage, twenty-four materials were exposed to liquid fluorine in three batches: ten in the first, ten in the second and four in the third, each material having duplicate specimens. The greatest variation between duplicate specimens was in the first batch of materials. The second batch showed good agreement in three cases and better agreement in six others than in the first ten, one instance of galvanic corrosion being encountered. The third batch of materials showed excellent agreement. Generally speaking, the results in this part of the study are higher than was expected. For example, stainless steel No. 304 shows, on the basis of a 5-hour exposure, an average expected corrosion rate

of 0.016 mils/hour (0.14 IPY); if this rate remained constant for a year, the penetration would be a little over one-eighth of an inch, a penetration far greater than our practical experience with liquid fluorine storage leads us to believe possible. A possible explanation is that in the initial few hours a high penetration rate is experienced because of the formation of a fluoride layer; thereafter, the rate may decrease sharply because of the protective action of the surface layer. Although we have grouped the metals into three categories (Table 5), the difference between the lowest and the highest rate of corrosion is only four-hundredths of a mil per hour. We conclude, therefore, that over the period of this 5-hour test, all the metals are relatively inert to liquid fluorine. It is pertinent to note, further, that it would be interesting to study the effect of surface condition of, say, titanium on its reactivity to liquid fluorine. A freshly broken surface, for example, might react much more vigorously.

b) Second Set of Experiments

As mentioned above, the data obtained in the first two of the three series of tests showed poor agreement between duplicate specimens for any given material. Also, the results of all the liquid fluorine tests were higher than had been expected. In an effort to clarify these points, we conducted two additional series of tests on four selected materials. We were not only desirous of spot-checking the previously obtained data, but, also of developing some basic data regarding the formation of fluoride films and their ability to protect against continued corrosion. The results of this additional work,

together with comparable results taken from Table 1, are shown in Table 6. In each material series shown, the first two of the 5-hour period results are those taken from Table 1. The third 5-hour, the first 24-hour and the 97.5-hour period results are for the first group of tests conducted to determine the effect of longer periods of exposure on the fluoride film formation of the various materials. The fluorine used for this group was taken not from cylinders, but from fluorine cells. Since cell gas usually has a somewhat higher HF content, it was thought that this might account for the rather erratic weight changes observed. Therefore, still another group of tests was conducted in which cylinder fluorine was used. This group consists of the last two 5-hour, the last two 24-hour and the 102-hour period results. It should be noted that, in view of the prior erratic weight losses, extreme care was taken with the handling of this last group of specimens. While the results in Table 6 show a lessening in corrosion rate as the exposure period is increased, the erratic weight change data cannot be overlooked or explained at this time. Possible explanations are: (1) a galvanic effect, (2) a partial solution of the fluoride films in liquid fluorine, or (3) the loosening and subsequent loss of fluoride film during exposure by reason of unaccountable vibration. Since the surfaces of a given material were similarly treated prior to exposure, it is not likely that the surface condition could be a factor. In any case, our ideas concerning the formation of adherent protective fluoride films by exposure in liquid fluorine have been somewhat clouded by these data, and it would

appear that further work along this line is certainly warranted. Conclusions which may be drawn from this work are that there is little difference in the corrosion rates of aluminum 3003-H14, monel and stainless steel No. 304, and that an increase in the liquid fluorine exposure time generally results in a decrease in the corrosion rate, probably because of the formation of a fluoride coating.

2. Resistance to Gaseous Fluorine

a) Five-Hour Exposure

Except for a few cases, the agreement between duplicate specimens is considered to be very good. Numerous instances of weight losses, up to 0.8 mgm, instead of weight gains were encountered. It must be assumed that these weight losses are due to a reaction of fluorine with surface impurities, experimental error or a combination of both. In any case, these differences are not considered to be excessively high. The grouping in Table 7 is intended to show, besides the relative corrosion resistance of the materials studied, the average corrosion rate range within which a particular material falls at the maximum temperature (1000°F) at which the material was tested. With respect to Group B-1, it might be added that the very low rate of corrosion is due to a very low reaction rate between the basic or constituent elements, or to the formation of a non-volatile adherent and protective constituent fluoride layer on the surface. In instances where both volatile and non-volatile fluorides were formed, the corrosion mechanism was not completely known, and it was necessary to make certain assumptions in order to calculate results

from the data at hand. While the materials in Group B-1 are in a class by themselves, as the exposure temperature decreases, materials from the other groups are found to approach them, until, at room temperature, there is very little, if any, difference in the resistance of any of the materials studied. Although they show a low resistance to fluorine, relative to other materials, Zirconium, Zircalloy II and Titanium B120-VCA performed better than had been expected. On the other hand, the performance of the stainless steels was disappointing.

b) Twenty-four Hour and Five-Day Exposures

Materials which showed high resistance to fluorine in the five-hour tests or had desirable structural properties were subjected to longer exposures. As was expected, these materials generally showed lower rates as the exposure time increased. Data is summarized in Table 8. The longer exposures also tend to minimize any errors in weighing due to factors such as balance sensitivity. An error, for example, of -.0002 gm in the weight change of Aluminum 1100 would indicate a corrosion rate of 0.0039 IPY for a five-hour exposure and for five days, 0.0002 IPY. The significance of surface impurities on the test specimens are likewise reduced in the longer runs. Runs at room temperature and at 400°F show only small rate changes as the exposure time increases. At 700° and 1000°F, changes become more noticeable. Two materials which did not fall into the expected pattern are Stainless Steel 304L and Aluminum 1100.

Stainless Steel 304L showed a slight decrease in corrosion at room temperature as the exposure time increased. This is no doubt due to the lessening effect of error noted

above. Runs at 400°F show more than a three-fold increase in corrosion rate for the 5-day test compared to the 1-day test, respective rates being 0.0254 IPY and 0.0075 IPY. The above specimens exposed for 5 days showed a thin, lightly adherent brown scale which was removed easily. Spectrographic analysis of this scale indicates it is almost entirely iron, nickel and chromium fluorides.

Stainless Steel 304L at 700°F showed IPY's of 1.565 for 5 hours and 6.018 for 24 hours. The specimens exposed for 24 hours were very severely attacked and were reduced to about 75% of the original thickness. The residue, present as a brown powder, was analyzed to determine if the corrosion was selective. Analysis showed 48.8% fluorine, 4.9% nickel, 37.4% iron and 7.5% chromium. The ratios of Ni, Fe and Cr checked almost exactly with the composition of the Stainless Steel 304L. The above analysis indicates a powder composition as follows:

FeF_3 = 75.77%
NiF_2 = 8.07%
CrF_3 = 15.72%
99.56% total

It is believed that the chromium is initially attacked, exposing finely divided nickel and iron to fluorine, in which form they are readily corroded. As a result of these tests, it was felt that 5-day runs at 1000°F and 700°F were unwarranted.

The phenomenon of Aluminum 1100 is quite different. At both 700°F and 1000°F, the five-day runs gave higher corrosion rates than one-day tests. In this case, it is believed that the fluoride formed permits fluorine to penetrate and attack beneath the surface film.

c) Twenty-four Hour Exposure at Elevated Pressures

Materials tested in a bomb for 24 hours at 400°F and approximately 250 pounds pressure of fluorine show no significant corrosion rate changes as compared to specimens exposed at the same temperature and time in a gaseous fluorine flow. As shown in Table 8, however, pressure makes significant changes in two materials at 700°F. The corrosion rate for Aluminum 1100 is 0.1654 IPY as compared to 0.0295 IPY for the same conditions in gaseous flow environment. Aluminum 2024 also showed a rather large increase in corrosion rate at 700°F, rising from .0018 to .0314 IPY. As previously noted, only three materials were tested at 1000°F under fluorine pressure. These materials all showed higher rates as compared to the previous tests at atmospheric pressure. From this work, it was concluded that the effect of pressure is negligible at low temperatures but is significant at elevated temperatures. Each material appears to be affected differently and no general conclusion can be drawn as to what constitutes the maximum non-corrosive pressure temperature combination. In Table 4 it was noted that the pressure tended to drop in the bomb as the fluorine was consumed. It is presumed that specimen corrosion was responsible although the nickel bomb undoubtedly continued to react with fluorine to some extent during the runs. The bombs had been passivated by exposure to fluorine at elevated temperature before being used and care was taken not to disturb the fluoride films on the bombs during this study. Work done by Steunenberg, Seiden and Griffin[2] indicates fluorine consumption can be measured by pressure drop. Since

pressure drop was negligible when corrosion rates were low, the pressure drop, when it did occur, was attributed to specimen attack but no attempt to correlate this data was made. One run with Magnesium AZ91C showed a loss in pressure which could not be attributed to the specimen's corrosion. Subsequent investigation disclosed evidence of a leak through the tip of the thermocouple pencil.

3. Determination of Fluoride Film Thickness

Portions of several specimens were prepared for metallographic examination. Microscopic measurements as well as an investigation into the nature of the fluoride films were undertaken. The film thickness of these and many other specimens were also determined by calculations based on the weight gain and the surface area. The calculated and measured film thicknesses are listed in Table 9. The factors used to calculate these results are shown in Table 15. To simplify measuring film thickness, photomicrographs at 1000 X were taken (Exhibits A to M). At this magnification, one millimeter represents one micron and measurements were, therefore, made directly. This method may introduce slight errors since the appearance of the film is somewhat dependent on the photographer's technique. Table 9A shows a comparison between film thicknesses on nickel obtained by other investigators (Steindler and Vogel[1] and Jarry, Gunther and Seiden[3]) and General Chemical Research Laboratory data from this investigation. The method of calculation used in this study is similar to the work of Jarry, Gunther and Seiden[3]. The measured and calculated results for monel and nickel are generally in very close agreement. The results for nickel (Table 9A) checks very well with our results and those of Jarry, et al. In general, the films studied here fall into

three general categories: (a) uniform films, (b) dispersed fluoride and (c) irregular fluorine penetration. These categories will be discussed below.

a) Uniform Films

As can be seen in Exhibits J, K, L and M, both monel and nickel form a fairly dense, uniform film. It appears that this type of film would retard fluorine penetration and thereby passivate the metal. This is verified by the corrosion rates as shown in Tables 1 to 3 where corrosion rate decreases as exposure period increases. It should be noted too, that when exposed to fluorine under pressure, the nature of the film does not change. It is believed that exposures at higher temperatures might result in a thicker film which would tend to spall on cooling. Aluminum 5154 (Exhibit F) also falls in this uniform film category.

b) Dispersed Fluoride Formation

As can be seen in Exhibits G, H and I, magnesium alloys do not show a dense uniform fluoride film. The appearance is rather that of individual cells of MgF_2. It is probable that this is a result of the extreme grain growth of the magnesium alloys at high temperature, inducing some porosity (Exhibits O and P). Aluminum 2024, which also showed grain enlargement (Exhibit Q) shows a somewhat similar scattered fluoride formation (Exhibit B). Magnesium M1A, on the other hand, does not show this grain growth (Exhibit N), but rather shows a higher concentration of fluoride cells or crystals near the surface.

c) Irregular Fluorine Penetration

Aluminum 1100 is the most unique of all specimens examined. This material (Exhibits A to D) always shows

a relatively deep but non-uniform penetration. The fluoride appears to form in an almost straight penetration, supplemented by a relatively thin uniform film. The very nature of this fluoride formation, we feel, explains why Aluminum 1100 alone tends to show a higher corrosion rate as exposure time increases (where non-volatile fluorides are formed). Exhibit B does not show this penetration to the same extent as the others. It is considered an exception in light of prior investigations made by the author on fluoride films which have consistently shown penetration. Exhibits C and D, which show specimens exposed under pressure, are also different since they do not show uniform surface films. It is felt attack is more rapid and the phenomenon of penetration is enhanced by pressure.

The calculation of film thickness, based as it is on weight gain, is subject to error since non-uniformity of coatings, film density variation and other causes must be considered. Measurements are likewise subject to error since the measurements were made on what was considered to be a representative section of the specimen's surface. Obviously, there are sections where the film thickness is different due to possible mechanical abrasion of the film or non-uniformity in film formation.

4. Physical Changes After Exposure

Specimens exposed for five days to fluorine at 1000°F were not measured, but were instead subjected to a physical study. As can be seen in Exhibit R, Aluminum 1100 has become severely embrittled and cracks when bent. Aluminum 5154, on the contrary, can be bent 90° with no evidence of cracking.

Aluminum 2024 after similar exposure was so brittle it was snapped in two using finger pressure (Exhibit Q). The two magnesium alloys, AZ81 and AZ91 were also brittle and broke easily, (Exhibits O and P). Magnesium M1A (Exhibit N) showed no grain growth and broke in a manner similar to an unexposed specimen. Temperature conditions may well have been more responsible for embrittlement than the fluorine atmosphere, per se.

B. Physical Data, Factors and Calculations

The analysis of materials used in this investigation, their densities and preparation are shown in Table 11. The method of calculating corrosion results and weight change factors are described (Tables 12 to 14) along with other pertinent data. Table 15 covers factors on determination of film thickness by calculation.

1. Calculation of Corrosion Results

Exposure of a metal or alloy to liquid or gaseous fluorine will result in either gain or loss in weight depending upon the material in question. It is necessary to convert these weight changes to a corrosion or penetration rate. The corrosion rate is reported as inches penetration per year (IPY). The equation used to calculate the corrosion rates for this study follows:

$$\text{Corrosion Rate} = \frac{\Delta W}{A \times t} \times K = IPY$$

where: ΔW = weight change in grams resulting from exposure
A = total exposed surface in sq. in.
t = exposure in hours
K = weight change factor x 8760

It should be noted that K is derived as follows:

$$\frac{\Delta W}{A \times t} \times \text{actual weight change factor} = \text{inches/hour}$$

$$\frac{\Delta W}{A \times t} \times \text{actual weight change factor} \times \frac{8760 \text{ hrs.}}{\text{yr.}} = \text{IPY}$$

$$\frac{\Delta W}{A \times t} \times K = \text{IPY}$$

2. Weight Gain Factors

Factors which were used to calculate corrosion penetration from positive weight changes are shown in Table 12. It will be noted that the actual weight gain factor as shown in this table has been multiplied by 10^3 for simplicity in tabulating. The factors (Table 12) were calculated by making certain assumptions about the behavior of the constituents of the alloys under fluorination. We assumed that where a volatile fluoride or fluorides of one or more of the constituents were known to exist, for example, CrF_4 and CrF_5, these fluorides would form and vaporize at their known vaporization temperatures. On the other hand, the non-volatile fluoride would remain attached to the parent alloy. Thus, in the former case, the effect on the test specimen would be a weight loss, while in the latter, it would be a weight gain. If the major constituent of the alloy were of a kind which formed non-volatile fluorides, weight gain would predominate over all; if the major component volatilized in fluorine, then obviously weight loss would predominate. In Table 13 are presented data on metal fluorides of interest. The calculation of a weight gain factor is illustrated for Inconel. (Refer to Table 11 for the composition of this alloy.)

a) Calculation of Inconel Factors

At temperatures up to 662°F, none of the component fluorides formed are volatile. Based on 100 gms. of this alloy, the following amount of fluorine is added when components are converted to the fluorides.

		Fluorine Added Gms.
Cr ——> CrF_3:	15.23 x 3 x 19/52.01 =	16.69
Fe ——> FeF_3:	7.00 x 3 x 19/55.85 =	7.15
Ni ——> NiF_2:	77.28 x 2 x 19/58.71 =	50.00
	Total =	73.84
Fluorine added/gm. of Inconel	=	0.7384

$$\text{Wgt. gain factor} = \frac{\frac{0.06102^{a}}{\rho^{b}}}{\frac{\text{gms. fluorine added}}{\text{gms. Inconel}}} = \frac{0.06102}{0.7384\rho}$$

Wgt. gain factor = 0.009710

Wgt. gain factor x 10^3 = 9.710

[a] converts cc to cu. in.

[b] density in gms/cc

At 932°F, CrF_4 and CrF_5 are assumed to be formed and to be volatile.

	Fluorine Added Gms.
Cr ——> CrF_4 and CrF_5:	-15.23
Fe ——> FeF_3:	7.15
Ni ——> NiF_2:	50.00
Total =	41.92
Fluorine added/gm. of Inconel =	0.4192

Wgt. gain factor $= \frac{0.06102}{0.4192\rho}$

Wgt. gain factor = 0.017104

Wgt. gain factor x 10^3 = 17.104

3. Weight Loss Factors

As in the case of weight gain factors, there are here two cases: (1) the uncomplicated case of a pure metal which volatilizes in fluorine (Wgt. loss factor = $\frac{.06102}{\rho}$) and (2) the more complex case of an alloy where volatile as well as non-volatile fluorides are formed. The calculation of the former is straightforward; the procedure for the latter is the same as in the case of weight gain factors.

Weight loss factors listed in Table 14 are those required in this study, non-pertinent ones having been omitted.

4. Calculation of Film Thickness

The film thicknesses of several exposed specimens as shown in Table 9 were both measured and calculated. The following equation was used to calculate the film thickness:

$$\text{Film thickness } (\mu) = \frac{\Delta W \left(\frac{MW\ MF}{MW\ F}\right) (10^4 \mu/\text{cm.})}{A\ (\rho MF)}$$

where: ΔW = weight gain (grams)
MW = molecular weight
MF = metal fluoride
A = area (sq. cm.)
ρMF = crystal density of metal fluoride

It can be readily seen that film thickness factors or constants can be calculated for each material. Some such factors are listed in Table 15.

a) Calculation of Monel Factors

An example showing the derivation of the monel film thickness factor, follows: For simplicity, we will assume the composition of monel to be 70% Ni and 30% copper.

$$\text{Film thickness } (\mu) = \frac{\Delta W \left(\frac{MW\ \text{monel fluoride}}{MW\ \text{fluorine}}\right) (10^4 \mu/\text{cm.})}{A\ \text{cm}^2\ (\rho\ \text{monel fluoride})}$$

$$\mu = \frac{\Delta W \left[(.70)\left(\frac{MW\ NiF_2}{MW\ F_2}\right) + (.30)\left(\frac{MW\ CuF_2}{MW\ F_2}\right)\right] 10^4}{A(in^2) \times 6.4516\ cm^2/in^2\ [(.70)(\rho NiF_2) + (.30)(\rho CuF_2)]}$$

$$\mu = \frac{\Delta W\ [(.70)\left(\frac{96.69}{38}\right) + (.30)\left(\frac{101.54}{38}\right)]\ 10^4}{(A\ in^2)(6.4516)[(.70)(4.63) + (.30)(4.25)]}$$

$$\mu = \frac{\Delta W}{A\ in^2} \times 886.5$$

The film thickness factor for monel, therefore, is 886.5. It should be noted that this factor includes a conversion constant for changing square inches to square centimeters. This procedure was used since all specimen areas listed in Tables 1 to 4 are in square inches. The listed factors (Table 15) can thus be applied directly.

C. Acknowledgements

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D. References

[1] Steindler, M. J., and R. C. Vogel, ANL-5662, (Jan., 1957)

[2] Steunenberg, R. K., L. Seiden, H. E. Griffin, ANL-5924, 42, 43, (1958)

[3] Jarry, R. L., W. H. Gunther, and L. Seiden, ANL-6029, 80-85, (1959)

[4a] Landau, R., and R. Rosen, Ind. Eng. Chem., 39, 281 (1947)

[4b] Landau, R., Corrosion 8, 283 (1952)

[5] Myers, W. R., and W. B. DeLong, Chem. Eng. Progress, 44, 359-362 (May, 1948)

[6] Ericson, G. L., W. K. Boyd and P. D. Miller, Memorandum on Corrosion of Titanium and Titanium-Base Alloys in Liquid and Gaseous Fluorine, (April, 1958), Battelle Memorial Institute

[7] International Nickel Company private communication to Steindler and Vogel

TABLE 1

CORROSION OF METALS AND ALLOYS IN FLUORINE
5 HOUR EXPOSURE

Material	Type	Ave. Temp. °F	Area Sq. In.	Weight Change Gms.	Corrosion Rate IPY
Aluminum	1100-H14	-320	1.92	0.0091	.0876[z]
"	"	"	1.91	0.0091	.0876[z]
"	"	77	1.97	-0.0006	.0122[a]
"	"	"	"	-0.0002	.0039[a]
"	"	336	"	-0.0001	.0020[a]
"	"	"	"	-0.0001	.0020[a]
"	"	662	1.96	0.0007	.0066
"	"	"	1.95	0.0006	.0056
"	"	993	3.93	0.3813[b]	1.822
Aluminum	2024-T3	-320	1.66	0.0145	.1489[z]
"	"	"	1.67	0.0157	.1840[z]
"	"	77	1.71	-0.0003	.0079[a]
"	"	"	"	-0.0003	.0079[a]
"	"	342	"	-0.0001	.0020[a]
"	"	"	"	0.0000	.0000
"	"	657	"	0.0000	.0000
"	"	"	"	-0.0002	.0039[a]
"	"	977	"	0.0030	.0342
"	"	"	"	0.0021	.0245
Aluminum	3003-H14	-320	2.03	0.0114	.1051[x]
"	"	"	2.03	0.0307	.3154[x]
"	"	68	2.00	0.0002	.0019
"	"	"	1.98	0.0000	.0000
"	"	376	2.00	0.0001	.0010
"	"	"	1.99	0.0000	.0000
"	"	658	2.00	0.0000	.0000
"	"	"	1.99	-0.0001	.0020[a]
"	"	972	2.01	0.0024	.0228
"	"	"	"	0.0020	.0193
Aluminum	5154-H34	-320	1.72	0.0079	.0876[x]
"	"	"	1.71	0.0263	.2978[x]
"	"	68	"	-0.0003	.0081[a]
"	"	"	1.70	-0.0003	.0081[a]
"	"	374	"	-0.0002	.0040[a]
"	"	"	"	-0.0002	.0040[a]
"	"	662	1.71	-0.0001	.0020[a]
"	"	"	"	-0.0003	.0081[a]
"	"	999	"	0.0006	.0068
"	"	"	1.70	0.0008	.0088

TABLE 1 - continued

Material	Type	Ave. Temp. °F	Area Sq. In.	Weight Change Gms.	Corrosion Rate IPY
Brass (Amer.)	#243	-320	2.03	0.0055	.0578[y]
"	"	"	2.02	0.0050	.0534[y]
"	"	77	"	0.0010	.0105
"	"	"	"	0.0023	.0245
"	"	390	"	0.0015	.0158
"	"	"	"	0.0023	.0245
"	"	689	"	0.0374	.3942
"	"	"	"	0.0346	.3679
"	"	995	"	0.3652	3.889
"	"	"	"	0.3581	3.811
Brass	Red	-320	2.03	0.0101	.1051[x]
"	"	"	2.01	0.0202	.2102[x]
"	"	68	2.07	-0.0004	.0025[a]
"	"	"	2.03	-0.0004	.0025[a]
"	"	415	2.07	0.0005	.0051
"	"	"	2.04	0.0006	.0061
"	"	700	2.04	0.0070	.0718
"	"	"	2.05	0.0068	.0701
"	"	982	2.00	0.0384	.3942
"	"	"	1.97	0.0394	.4030
Copper	ETP	-320	2.04	0.0125	.1226[x]
"	"	"	2.03	0.0190	.1927[x]
"	"	68	2.07	-0.0003	.0018[a]
"	"	"	2.06	-0.0008	.0048[a]
"	"	414	2.07	0.0012	.0123
"	"	"	2.05	0.0002	.0020
"	"	691	2.05	0.0032	.0324
"	"	"	2.06	0.0029	.0289
"	"	984	2.00	0.0247	.2453
"	"	"	2.01	0.0222	.2190
Illium	R	-320	1.95	0.0153	.1226[x]
"	"	"	1.94	0.0062	.0517[x]
"	"	77	1.91	0.0001	.0010
"	"	"	1.92	0.0003	.0029
"	"	360	1.89	0.0002	.0019
"	"	"	1.90	0.0010	.0105
"	"	687	"	0.0011	.0114
"	"	"	1.92	0.0013	.0140
"	"	991	1.97	0.1249	4.038
"	"	"	1.96	0.1241	4.038

TABLE 1 - continued

Material	Type	Ave. Temp. °F	Area Sq. In.	Weight Change Gms.	Corrosion Rate IPY
Inconel		-320	2.00	0.0024	.0201[y]
"		"	1.99	0.0039	.0333[y]
"		77	"	0.0002	.0017
"		"	"	-0.0001	.0006[a]
"		396	2.00	-0.0002	.0012[a]
"		"	"	0.0000	.0000
"		694	"	0.0089	.0753
"		"	"	0.0094	.0797
"		997	3.99	0.4616[b]	3.4514
Magnesium	M1A	-320	1.96	0.0066	.1314[y]
"	"	"	1.99	0.0000	.0000[y]
"	"	77	"	0.0000	.0000
"	"	"	"	0.0000	.0000
"	"	405	"	0.0000	.0000
"	"	"	1.98	0.0001	.0019
"	"	712	1.97	0.0003	.0059
"	"	"	1.98	-0.0003	.0088[a]
"	"	1000	1.99	-0.0037[c]	.1139[c]
"	"	"	"	0.0016	.0315
Magnesium	AZ81C-T6	-320	2.98	0.0231	.2891[y]
"	"	"	2.91	-0.0427[d]	.8666[y]
"	"	77	2.77	0.0000	.0000
"	"	414	2.88	0.0001	.0013
"	"	721	2.80	0.0010	.0131
"	"	1006	2.81	0.0030	.0394
Magnesium	HK31A-H24	-320	1.95	0.0022	.0429[y]
"	"	"	"	0.0063	.1226[y]
"	"	77	"	0.0004	.0074
"	"	"	1.96	0.0002	.0037
"	"	372	1.94	0.0006	.0114
"	"	"	1.95	0.0001	.0018
"	"	684	"	0.0005	.0096
"	"	"	"	0.0002	.0037
"	"	986	"	0.0011	.0201
"	"	"	"	0.0011	.0201
Monel		-320	2.03	0.0250	.2365[x]
"		"	"	0.0089	.0841[x]
"		77	2.00	0.0002	.0019
"		"	"	0.0003	.0029
"		374	"	0.0001	.0010
"		"	2.01	0.0000	.0000

TABLE 1 - continued

Material	Type	Ave. Temp. °F	Area Sq. In.	Weight Change Gms.	Corrosion Rate IPY
Monel		666	2.01	-0.0002	.0012[a]
"		"	2.00	-0.0004	.0025
"		1002	"	0.0030	.0289
"		"	2.01	0.0032	.0307
Monel	Cast	-320	2.35	0.0153	.1226[z]
"	"	"	2.37	0.0150	.1139[z]
"	"	77	2.38	0.0003	.0030
"	"	356	"	0.0001	.0010
"	"	727	2.34	0.0005	.0039
"	"	981	2.35	0.0504	.0429
Nickel	A	-320	2.04	0.0038	.0342[y]
"	"	"	"	0.0015	.0140[y]
"	"	77	"	0.0001	.0010
"	"	"	"	0.0001	.0010
"	"	394	"	0.0004	.0038
"	"	"	2.03	0.0003	.0028
"	"	705	2.04	-0.0001	.0006[a]
"	"	"	"	0.0003	.0028
"	"	1008	"	0.0028	.0254
"	"	"	"	0.0025	.0237
Nickel	L	-320	2.04	0.0062	.0569[y]
"	"	"	"	0.0016	.0149[y]
"	"	77	2.05	0.0001	.0010
"	"	"	2.03	0.0004	.0038
"	"	401	"	0.0002	.0018
"	"	"	"	0.0005	.0046
"	"	716	2.05	0.0002	.0018
"	"	"	2.04	0.0001	.0010
"	"	1013	"	0.0022	.0201
"	"	"	"	0.0022	.0201
Stainless Steel	#304	-320	2.04	0.0311	.2015[x]
"	"	"	2.03	0.0113	.0736[x]
"	"	68	2.18	0.0002	.0013
"	"	"	2.08	0.0003	.0020
"	"	417	2.10	0.0011	.0067
"	"	"	2.09	0.0008	.0055
"	"	698	2.12	0.2373	1.515
"	"	"	2.01	0.2405	1.612

TABLE 1 - continued

Material	Type	Ave. Temp. °F	Area Sq. In.	Weight Change Gms.	Corrosion Rate IPY
Stainless Steel	#347	-320	1.99	0.0161	.1051[x]
" "	"	"	2.00	0.0320	.2102[x]
" "	"	68	2.21	0.0004	.0027
" "	"	"	2.06	0.0004	.0027
" "	"	421	2.07	0.0006	.0042
" "	"	"	2.13	0.0005	.0035
" "	"	711	2.10	-0.6725[e]	4.266
" "	"	"	2.12	-0.6736[e]	4.231
Steel	LC	-320	2.00	0.0221	.0333[x]
"	"	"	2.01	0.0337	.0201[x]
"	"	77	1.99	0.0005	.0033
"	"	"	2.02	0.0009	.0060
"	"	360	1.99	-0.0007	.0047[c]
"	"	"	2.00	0.0007	.0047
"	"	682	1.98	0.0576	.3942
"	"	"	1.99	0.0591	.4030
"	"	(>980)[e]	-	-	-[f]
Tantalum		-320	2.00	-0.0215[g]	.0368[y]
"		"	1.99	-0.0216[g]	.0280[y]
"		77	"	0.0020	.0123
"		"	2.01	0.0021	.0131
"		(>154)[e]	-	-	-[f]
Titanium	A-55	-320	2.09	0.0440	.3154[x]
"	"	"	"	0.0203	.1489[x]
"	"	77	2.08	0.0006	.0045
"	"	"	2.04	0.0009	.0067
"	"	302	2.05	-0.0063	.0736
"	"	"	2.07	-0.0065	.0745
"	"	385	"	-0.1551	1.787
"	"	"	"	-0.1650	1.892
Titanium	B120-VCA	-320	1.81	0.0205	.1577[z]
"	"	"	1.83	0.0208	.1577[z]
"	"	77	1.90	-0.0002	.0022[a]
"	"	"	1.86	0.0000	.0000
"	"	309	1.89	0.0000	.0000
"	"	"	1.84	0.0000	.0000
"	"	361	"	-0.0038	.0456
"	"	"	1.86	-0.0037	.0447
"	"	390	1.84	-0.0071	.0850
"	"	"	1.85	-0.0071	.0850
"	"	426	"	-0.0305	.3329
"	"	475	1.86	-0.0962	1.148

TABLE 1 - continued

Material	Type	Ave. Temp. °F	Area Sq. In.	Weight Change Gms	Corrosion Rate IPY
Zirconium		-320	2.00	0.0129	.1314[y]
"		"	1.99	0.0140	.1402[y]
"		77	"	0.0002	.0020
"		"	1.98	0.0001	.0010
"		351	1.99	0.0025	.0245
"		"	2.00	0.0029	.0289
"		662	1.99	-0.9936[e]	8.217
"		"	"	-1.0113[e]	8.375
Zircalloy	II	-320	1.89	0.0058	.0604[y]
"	"	"	1.88	0.0061	.0648[y]
"	"	77	"	0.0003	.0030
"	"	"	1.91	0.0006	.0060
"	"	351	1.89	0.0016	.0166
"	"	"	1.88	0.0023	.0245
"	"	(642-752)[h]	1.87	-0.8705[e]	7.656

[a] Weight loss possibly due to reaction of fluorine with surface impurities, or to experimental error.

[b] Material scaled making it necessary to combine the results for two specimens.

[c] This result is questionable.

[d] Specimen showed definite evidence of galvanic corrosion.

[e] Specimens were water washed and brushed, it being impossible to obtain accurate weight change after exposure.

[f] Completely reacted, temp. increased during reaction.

[g] After water washing and brushing, weight changes after exposure (-0.0114 and -0.0086) were not considered reliable.

[h] Temperature increase during run.

[x] Result was obtained in the first series of tests run.

[y] " " " " " second " " " "

[z] " " " " " third " " " "

TABLE 1A

CORROSION OF METALS AND ALLOYS IN FLUORINE
APPEARANCE AFTER 5 HOUR EXPOSURE

Material	Type	Approximate Temperature of Test °F				
		-320	75	385	690	1000
Aluminum	1100-H14	mod. duller	no change	no change	no change	gray
"	2024-T3	" "	" "	" "	sltly duller	dull gray
"	3003-H14	" "	" "	" "	" "	much duller
"	5154-H34	" "	" "	" "	mod. "	" "
Brass	red	dull dk.yellow	" "	dull yellow-red	dull brown	gray[b]
"	Amer.#243	much duller[c]	" "	duller[d]	" "	blue[e]
Copper	ETP	much duller	" "	sltly duller	dull orange	dull yellow-pink
Illium	R	sltly "	" "	" "	much duller	dull brown
Inconel	-	mod. "	" "	no change	dull yellow-green	yellow film
Magnesium	M1A	" "	" "	sltly duller	mod. duller	much duller
"	HK31A-H24	" "	" "	no change	" "	dull gray
"	AZ81C-T6	" "	" "	mod. duller	much "	" "
Monel	-	" "	" "	no change	sltly "	" "
"	cast	much "	sltly duller	sltly duller	dull gray	dull yellow-pink
Nickel	A	" "	no change	no change	light blue hue	dull gray
"	L	" "	" "	" "	blue-purple hue	" "
Stainless Steel	#304	" "	" "	duller[g]	thick film[h]	(no test)
" "	#347	" "	" "	"	thick film[i]	" "
Steel	LC	" "	" "	blue-purple hue	powder[j]	" "
Tantalum	-	no change	" "	(no test)	(no test)	" "
Titanium	A-55	much duller	" "	darker,rough	" "	" "
"	B120-VCA	no change	" "	no change	" "	" "
Zirconium	-	much duller	" "	dull gray	dull gray[k]	" "
Zircalloy	II	dull gray	" "	" "	white film[l]	" "

[a] light and dark gray, scaled
[b] dull bluish gray over copper color
[c] dark yellow and slight copper color
[d] multicolor(yellow,rust red,brn,blk,lt. blue)film
[e] dull light blue on maroon, roughened surface
[f] thick, adherent
[g] red, green and violet film
[h] loose, yellow green
[i] very loose, yellow green
[j] dense, light green
[k] light, white powder on surface
[l] 1/16" thick, adherent but brittle

TABLE 2

CORROSION OF METALS AND ALLOYS IN GASEOUS FLUORINE
24 HOUR EXPOSURE

Material	Type	Ave. Temp. °F	Area Sq. In.	Weight Change Gms	Corrosion Rate IPY
Aluminum	1100 H-14	82	2.079	-0.0005	.0020
"	"	82	2.090	-0.0007	.0027
"	"	414	2.083	-0.0001	.0004
"	"	385	2.086	-0.0008	.0031
"	"	702	2.089	0.0169	.0314
"	"	702	2.136	0.0152	.0277
"	"	1020	2.110	0.1066	.1960
"	"	1020	2.097	0.1062	.1970
Aluminum	2024 T-3	82	2.012	-0.0001	.0004
"	"	82	2.001	0.0003	.0006
"	"	423	2.012	0.0014	.0028
"	"	385	2.007	-0.0002	.0008
"	"	707	2.008	0.0005	.0011
"	"	707	2.018	0.0007	.0014
"	"	1004	2.014	0.0031	.0063
"	"	1004	2.011	0.0031	.0063
Aluminum	5154 H-34	82	1.610	-0.0006	.0032
"	"	82	1.613	0.0003	.0008
"	"	428	1.614	-0.0004	.0021
"	"	388	1.611	-0.0003	.0016
"	"	682	1.510	0.0006	.0016
"	"	682	1.613	0.0006	.0015
"	"	1004	1.610	0.0022	.0055
"	"	1004	1.610	0.0026	.0066
Magnesium	M1A	82	1.989	0.0010	.0041
"	"	82	1.993	-0.0001	.0006
"	"	403	1.981	0.0004	.0017
"	"	374	1.983	0.0004	.0017
"	"	678	1.942	0.0007	.0030
"	"	678	2.006	0.0004	.0017
"	"	1017	1.992	0.0041	.0168
"	"	1017	1.975	0.0035	.0145
Magnesium	AZ81A-T6	82	2.787	-0.0003	.0013
"	"	82	2.748	-0.0006	.0027
"	"	408	2.770	-0.0001	.0004
"	"	370	2.782	0.0004	.0011
"	"	705	2.790	0.0014	.0039

TABLE 2 - continued

Material	Type	Ave. Temp. °F	Area Sq. In.	Weight Change Gms	Corrosion Rate IPY
Magnesium	AZ81A-T6	676	2.787	0.0015	.0042
"	"	1024	2.781	0.0038	.0106[a]
"	"	1018	2.752	0.0077	.0216[a]
"	"	941	2.744	0.0029	.0082
"	"	927	2.802	0.0037	.0102
Magnesium	AZ91C-T6	82	2.550	-0.0005	.0024
"	"	82	2.537	0.0003	.0009
"	"	421	2.558	0.0001	.0003
"	"	390	2.524	0.0001	.0003
"	"	676	2.567	0.0009	.0027
"	"	676	2.570	0.0008	.0024
"	"	1026	2.564	0.0765	.2313[a]
"	"	1026	2.544	---	--- [b]
"	"	941	2.565	0.0092	.0278
"	"	941	2.538	-0.0062	.0300
Monel		82	2.012	0.0004	.0008
"		82	2.005	0.0001	.0002
"		410	2.006	0.0000	.0000
"		381	2.006	-0.0007	.0009
"		705	1.999	0.0010	.0020
"		705	1.999	0.0007	.0014
"		1015	2.003	0.0061	.0121
"		1015	2.003	0.0053	.0105
Nickel	A	82	1.992	0.0005	.0010
"	"	82	1.994	0.0004	.0008
"	"	421	1.997	0.0005	.0010
"	"	381	1.994	0.0000	.0000
"	"	702	1.999	0.0008	.0016
"	"	702	1.991	0.0004	.0008
"	"	997	1.992	0.0088	.0172
"	"	997	1.993	0.0076	.0149
Stainless Steel	304L	82	2.094	0.0004	.0005
" "	"	82	2.087	0.0005	.0007
" "	"	405	2.082	0.0043	.0058
" "	"	412	2.084	0.0068	.0092
" "	"	718	2.069	-4.4528	5.976
" "	"	718	2.066	-4.5081	6.059

[a] Specimens had softened and deformed slightly at indicated temperature. Fresh specimens were tested at a slightly lower temperature.

[b] Specimen had fused to the thermocouple well and could not be accurately weighed after test.

TABLE 2A

CORROSION OF METALS AND ALLOYS IN GASEOUS FLUORINE
APPEARANCE AFTER 24 HOUR EXPOSURE

Material	Type	Approximate Temperature of Test °F			
		75	400	700	1000
Aluminum	1100 H-14	Unchanged	Unchanged	Dk. Gray	V. Dk. Gray
"	2024 T-3	"	"	Mod. Gray	Mod. Gray
"	5154 H-34	"	"	Lt. to Mod. Gray	Mod. Gray
Magnesium	M1A	V.Lt. to Mod.Gray	Mtl.Lt.Gray	V. Lt. Gray	Whitish Gray
"	AZ81AT6	Mtl.	" " "	Lt. Gray	Mtl. Gray[a]
"	AZ91CT6	"	Lt.to Mod. Gray	" "	" " [b]
Monel		Slt. Dulling	Mod. Dulling	Mod. Disc.	Dk. Gray
Nickel	A	Unchanged	Unchanged	V. Slt. Disc.	Mod. Gray
Stainless Steel	304L	"	Rust Like	Rust Color[c]	Not tested

Abbreviations:
Disc. - Discoloration
Dk. - Dark
Lt. - Light
Mod. - Moderate
Mtl. - Mottled
Slt. - Slight
V. - Very

[a] Additional specimens run at 941°F showed slight discoloration.

[b] " " " " 941°F were very light gray.

[c] Specimen surrounded by a mound of brown powder.

TABLE 3

CORROSION OF METALS AND ALLOYS IN FLUORINE
5 DAY EXPOSURE

Material	Type	Ave. Temp. °F	Area Sq. In.	Weight Change Gms	Corrosion Rate IPY
Aluminum	1100 H-14	79	2.082	0.0004	.0002
"	"	79	2.088	0.0002	.0001
"	"	394	2.079	0.0001	.0000
"	"	394	2.069	0.0001	.0000
"	"	673	1.990	0.1871	.0731
"	"	673	1.998	0.1976	.0769
"	"	1009	2.021	0.7470	.2875
"	"	1009	1.989	0.7308	.2858
Aluminum	2024 T-3	79	2.020	0.0001	.0001
"	"	79	2.019	0.0000	.0000
"	"	394	2.014	0.0001	.0000
"	"	394	2.016	0.0003	.0001
"	"	673	1.740	0.0113	.0053
"	"	673	1.733	0.0171	.0080
"	"	1020	1.725	0.0028	.0013
"	"	1020	1.740	0.0033	.0015
Aluminum	5154 H-34	79	1.608	0.0000	.0000
"	"	79	1.612	-0.0003	.0004
"	"	356	1.601	-0.0004	.0004
"	"	356	1.612	-0.0003	.0003
"	5154-0	640	1.989	0.0012	.0005
"	"	640	2.007	0.0012	.0005
"	"	1017	1.991	0.0031	.0013
"	"	1017	1.999	0.0030	.0012
Magnesium	M1A	79	2.002	0.0003	.0003
"	"	79	2.002	0.0001	.0001
"	"	358	1.981	0.0002	.0002
"	"	358	2.002	0.0001	.0001
"	"	653	1.926	0.0010	.0008
"	"	653	1.968	0.0011	.0009
"	"	1006	1.972	0.0128	.0106
"	"	1006	1.954	0.0121	.0101

TABLE 3 - continued

Material	Type	Ave. Temp. °F	Area Sq. In.	Weight Change Gms	Corrosion Rate IPY
Magnesium	AZ81A-T6	79	2.735	0.0003	.0002
"	"	79	2.806	0.0000	.0000
"	"	363	2.762	-0.0002	.0002
"	"	363	2.772	-0.0002	.0002
"	"	691	2.762	0.0003	.0002
"	"	691	2.737	0.0005	.0003
"	"	1000	2.780	0.0060	.0033
"	"	1000	2.783	0.0039	.0022
Magnesium	AZ91C-T6	79	2.558	-0.0002	.0002
"	"	79	2.552	0.0002	.0002
"	"	406	2.640	0.0000	.0000
"	"	406	2.664	-0.0002	.0002
"	"	691	2.656	0.0020	.0012
"	"	691	2.629	0.0015	.0009
"	"	1002	2.642	0.0012	.0007
"	"	1002	2.644	0.0026	.0015
Monel		79	2.002	0.0005	.0002
"		79	2.003	0.0003	.0001
"		396	1.996	0.0004	.0002
"		396	2.003	0.0001	.0000
"		684	2.009	0.0027	.0011
"		684	1.951	0.0031	.0013
"		1020	1.991	0.0179	.0071
"		1020	1.984	0.0180	.0072
Nickel	A	79	1.988	0.0000	.0000
"	"	79	1.997	0.0000	.0000
"	"	396	2.002	0.0002	.0001
"	"	396	2.004	0.0000	.0000
"	"	684	1.997	0.0010	.0004
"	"	684	2.007	0.0008	.0003
"	"	1026	1.982	0.0350	.0138
"	"	1026	1.984	0.0349	.0137
Stainless Steel	304L	79	2.086	0.0000	.0000
" "	"	79	2.093	0.0001	.0000
" "	"	406	2.088	0.0942	.0254
" "	"	406	2.094	0.0891	.0240

TABLE 3A

CORROSION OF METALS AND ALLOYS IN GASEOUS FLUORINE
APPEARANCE AFTER 5 DAY EXPOSURE

Material	Type	Approximate Temperature of Test °F			
		75[c]	400	700	1000
Aluminum	1100 H-14	Unchanged	Unchanged	V. Dk. Gray	V. Dk. Gray[a]
"	2024 T-3	"	"	Mtl. V. Dk. Gray	Dk. Gray[b]
"	5154 H-34	"	"	Mod. Gray	Dk. Gray
Magnesium	M1A	Mtl. Gray	Lt. Gray	Whitish Gray	Lt. to Mod. Gray
"	AZ81AT6	" "	Mtl. Mod. Gray	Mod. Gray	Mod. Gray[b]
"	AZ91CT6	" "	Mod. Gray	" "	" " [b]
Monel		Mod. Gray	Mod. to Dk. Gray	Sev. Disc.	Dk. Gray
Nickel	A	Unchanged	Slt. Iridescence	Slt. Iridescence	Green-Gray
Stainless Steel	304L	"	Rust Brown	Not Tested	Not Tested

Abbreviations:

Disc.	- Discoloration	Mtl.	- Mottled
Dk.	- Dark	Sev.	- Severe
Lt.	- Light	Slt.	- Slight
Mod.	- Moderate	V.	- Very

[a] Fluoride film shows cracks along all the edges.

[b] Surface appears to be alligatored indicative of extreme grain growth. Specimens showed severe embrittlement.

[c] Runs at this temperature were 4 days.

TABLE 4

CORROSION OF METALS AND ALLOYS IN FLUORINE
24 HOUR EXPOSURE AT ELEVATED PRESSURE

Material	Type	Ave. Temp. °F	Initial Press. psi	Final Press. psi	Area Sq. In.	Weight Change Gms	Corrosion Rate IPY
Aluminum	1100 H-14	401	230	230	2.030	-0.0002	.0008
"	"	401	230	230	1.994	0.0000	.0000
"	"	689	250	170[a]	1.994	0.0818	.1636
"	"	689	250	170[a]	1.927	0.0828	.1671
"	"	1004	270	180[a]	1.974	0.1004	.1978
"	"	1004	270	180[a]	1.999	0.1038	.2020
Aluminum	2024 T-3	401	230	230	1.877	-0.0003	.0013
"	"	401	230	230	1.898	-0.0003	.0013
"	"	671	260	260	1.899	0.0267	.0573
"	"	671	260	260	1.801	0.0024	.0054
Aluminum	5154-0	401	230	230	1.985	-0.0003	.0013
"	"	401	230	230	2.025	-0.0012	.0050
"	"	689	265	265	2.038	0.0003	.0006
"	"	689	265	265	1.999	0.0002	.0004
Magnesium	M1A	401	240	240	1.943	-0.0003	.0020
"	"	401	240	240	1.854	-0.0002	.0014
"	"	680	260	260	2.009	0.0002	.0008
"	"	680	260	260	2.031	0.0003	.0012
Magnesium	AZ81A-T6	392	235	235	2.780	-0.0010	.0044
"	"	392	235	235	2.755	-0.0018	.0080
"	"	698	260	260	2.768	0.0014	.0039
"	"	698	260	260	2.759	0.0015	.0042
Magnesium	AZ91C-T6	392	235	235	2.652	-0.0016	.0074
"	"	392	235	235	2.667	-0.0015	.0069
"	"	689	215	150[b]	2.828	0.0017	.0047
"	"	689	215	150[b]	2.796	0.0015	.0042
Monel		410	170	170	1.977	0.0005	.0010
"		410	170	170	1.959	0.0002	.0004
"		698	260	260	2.004	0.0011	.0022
"		698	260	260	2.002	0.0013	.0026
"		1040	255	160[a]	1.997	0.0101	.0201
"		1040	255	160[a]	1.982	0.0112	.0224

TABLE 4 - continued

Material	Type	Ave. Temp. °F	Initial Press. psi	Final Press. psi	Area Sq. In.	Weight Change Gms	Corrosion Rate IPY
Nickel	A	428	175	175	1.987	0.0002	.0004
"	"	428	175	175	2.025	0.0001	.0002
"	"	689	255	255	1.979	0.0001	.0002
"	"	689	255	255	2.005	0.0004	.0008
"	"	1040	260	200[a]	1.996	0.0231	.0451
"	"	1040	260	200[a]	1.997	0.0225	.0439

[a] Decrease in pressure attributed to partial consumption of fluorine by the specimens and equipment.

[b] Evidence of a slight leak in system was noted.

TABLE 4A

CORROSION OF METALS AND ALLOYS IN FLUORINE
APPEARANCE AFTER 24 HOUR EXPOSURE AT ELEVATED PRESSURE

Material	Type	Approximate Temperature of Test °F		
		400	700	1000
Aluminum	1100 H-14	Unchanged	Gray-Black	Lt. to V. Dk. Gray[a]
"	2024 T-3	Slt. Duller	Lt. to V. Dk Gray	Not tested
"	5154 - 0	" "	Lt. Gray	" "
Magnesium	M1A	" "	Slt. Duller	" "
"	AZ81AT6	Mod. "	Slt. Irid.	" "
"	AZ91CT6	" "	Lt. Gray	" "
Monel		Unchanged	Reddish-Brown	Reddish-Brown
Nickel	A	Slt. Duller	Ext. Irid.	Greenish-Gray

Abbreviations: Dk. - Dark; Ext. - Extreme; Irid. - Iridescence; Lt. - Light; Mod. - Moderately; Slt. - Slightly; V. - Very

[a] Film is non-uniform. The very dark area shows what appears to be very minute blistering. The light area may be due to some of the dark film flaking off. Electrical resistance is low to nil in the light area but very high in the dark area. This would indicate that the corrosion rate is actually higher than calculated.

TABLE 5

RESISTANCE OF METALS AND ALLOYS TO LIQUID FLUORINE
5 HOUR EXPOSURE

	Group A-1	Group A-2	Group A-3
Ave. Corr. Rate mils/hour	0.003-0.008	0.010-0.019	0.022-0.036
	Amer. Brass #243	Aluminum 1100-H14	Aluminum 3003-H14
	Inconel	Aluminum 2024-T3	Aluminum 5154-H34
	Magnesium M1A	Brass (red)	Magnesium AZ81C-T6
	Nickel A	Copper	Steel (low carbon)
	Nickel L	Illium R	Titanium A-55
	Tantalum	Magnesium HK31A-H24	
	Zircalloy II	Monel	
		Monel (cast)	
		Stainless Steel #304	
		Stainless Steel #347	
		Titanium B120-VCA	
		Zirconium	

Note: 0.001 mil/hr. is equivalent to 0.0088 inches/year.

TABLE 6

CORROSION OF MATERIALS BY LIQUID FLUORINE

Material	Type	Exposure Time Hours[a]	Weight Change Gms.	Area Sq. In.	Corrosion Rate IPY
Aluminum	3003-H14	5	0.0114	2.03	.1051
"	"	5	0.0307	2.03	.3154
"	"	5	0.0031	2.00	.0298
"	"	5	0.0186	2.06	.1752
"	"	5	0.0188	2.06	.1752
"	"	24	0.0000	2.00	.0000
"	"	24	0.0051	2.06	.0088
"	"	24	0.0041	2.06	.0076
"	"	97.5	0.0012	2.00	.0005[b]
"	"	102	0.0148	2.06	.0067
"	"	102	-0.0026	2.06	.0024
Monel		5	0.0250	2.03	.2365
"		5	0.0089	2.03	.0841
"		5	-0.0005	1.98	.0031[b]
"		5	0.0131	2.01	.1226
"		5	0.0125	2.02	.1139
"		24	0.0056	1.99	.0114
"		24	0.0093	2.03	.0184
"		24	0.0073	2.02	.0140
"		97.5	-0.0007	2.00	.0002
"		102	0.0115	2.02	.0057
"		102	0.0084	2.02	.0047
Stainless Steel	#304	5	0.0311	2.04	.2015
" "	"	5	0.0113	2.03	.0736
" "	"	5	-0.0004	2.02	.0026[b]
" "	"	5	0.0048	2.02	.0315
" "	"	5	0.0074	2.02	.0482
" "	"	24	0.0035	2.01	.0046
" "	"	24	0.0044	2.02	.0060
" "	"	24	0.0043	2.02	.0060
" "	"	97.5	-0.0008	2.02	.0003[b]
" "	"	102	0.0151	2.02	.0046
" "	"	102	0.0202	2.01	.0066

TABLE 6 - continued

Material	Type	Exposure Time Hours[a]	Weight Change Gms.	Area Sq. In.	Corrosion Rate IPY
Titanium	A-55	5	0.0440	2.09	.3154
"	"	5	0.0203	"	.1489
"	"	5	-0.0017	2.06	.0196[b]
"	A-70	5	0.0386	1.98	.2891
"	"	5	0.0283	1.99	.2102
"	A-55	24	0.0321	2.04	.0491
"	A-70	24	0.0064	1.99	.0096
"	"	24	0.0153	2.00	.0237
"	A-55	97.5	-0.0671	2.05	.0399[b]
"	A-70	102	0.3366	2.00	.1226
"	"	102	0.2110	1.99	.0780

[a] In liquid fluorine at -320°F.

[b] The negative or small weight change indicates probable partial loss of the fluoride film or layer.

TABLE 7

RESISTANCE OF METALS AND ALLOYS TO GASEOUS FLUORINE
5 HOUR EXPOSURE

	Group B-1	Group B-2	Group B-3	Group B-4	Group B-5
Max.Test °F[a]	1000	1000	1000	700	below 475
Ave.Corr.Rate[b]	less than 0.005	0.027-0.046	0.20-0.46	0.046-0.95	more than 0.13
	Aluminum 2024-T3	Brass(red)	Aluminum 1100-H14	Stainless Steel #304	Tantalum[c]
	Aluminum 3003-H14	Copper	Amer.Brass #243	Stainless Steel #347	Titanium A-55
	Aluminum 5154-H34		Illium R	Steel(low carbon)	Titanium B120-VCA
	Magnesium M1A		Inconel	Zirconium	
	Magnesium AZ81C-T6			Zircalloy II	
	Magnesium HK31A-H24				
	Monel				
	Monel (cast)				
	Nickel A				
	Nickel L				

[a] Approximate

[b] Mils/hr.

[c] Reacted completely at 154°F

Note: 1. .001 mil/hr. is equivalent to a corrosion rate of approximately .0088 inches/year.

2. Materials tested for longer exposure show lower corrosion rates which could possibly place them in a higher group. Strong exception to this is S.S. 304L which showed four times the rate for 24 hours as compared to the 5 hour corrosion rate at 700°F.

TABLE 8

CORROSION RATES[c] OF MATERIALS IN GASEOUS FLUORINE

Material	Exposure Time Hrs.	Temperature [a] 80°F	400°F	700°F	1000°F	Temperature [b] 400°F	700°F	1000°F
Aluminum 1100	5	.0080	.0020	.0061	1.822			
" "	24	.0024	.0018	.0295	.1965	.0004	.1654	.1999
" "	120	.0002	.0000	.0750	.2867			
Aluminum 2024	5	.0079	.0010	.0020	.0293			
" "	24	.0005	.0018	.0013	.0063	.0013	.0314	
" "	120	.0001	.0001	.0067	.0014			
Aluminum 5154	5	.0081	.0040	.0050	.0078			
" "	24	.0020	.0019	.0016	.0061	.0032	.0005	
" "	120	.0002	.0004	.0005	.0012			
Magnesium M1A	5	.0000	.0010	.0074	.0727			
" "	24	.0024	.0017	.0024	.0157	.0017	.0010	
" "	120	.0002	.0002	.0009	.0104			
Magnesium AZ81	5	.0000	.0013	.0131	.0394			
" "	24	.0020	.0008	.0041	.0092	.0062	.0041	
" "	120	.0001	.0002	.0003	.0028			
Magnesium AZ91	5	--	--	--	--			
" "	24	.0017	.0003	.0026	.0289	.0072	.0045	
" "	120	.0002	.0001	.0011	.0011			
Monel	5	.0024	.0005	.0019	.0298			
"	24	.0005	.0005	.0017	.0113	.0007	.0024	.0213
"	120	.0002	.0001	.0012	.0072			
Nickel A	5	.0010	.0033	.0017	.0245			
" "	24	.0009	.0005	.0012	.0161	.0003	.0005	.0445
" "	120	.0000	.0001	.0004	.0138			
Stainless Steel 304	5	.0017	.0061	1.565				
" " 304L	24	.0006	.0075	6.018				
" " 304L	120	.0000	.0254					

[a] Corrosion rates in a stream of gaseous fluorine 100 ml/min.

[b] Corrosion rates in gaseous fluorine under initial pressures of approximately 250 pounds gauge.

[c] Inches per year. Rates given are the average of duplicate specimens.

TABLE 9

CORROSION OF METALS BY FLUORINE - FLUORIDE FILM THICKNESS

Material	Type	Exposure Time Hrs.	Exposure Temp. °F	Area Sq. In.	Weight Change Gms.	Film Thickness (Å) Calc'd	Film Thickness (Å) Found	Exhibit
Aluminum	1100	5	993	3.93	0.3813	72.2	50[a]	A
"	"	24	702	2.089	0.0169	6.0	N.M.	
"	"	24	1020	2.110	0.1066	37.6	35[b]	B
"	"	24[d]	689	1.994	0.0818	30.5	55[c]	C
"	"	24[d]	1004	1.999	0.1038	38.6	120[c]	D
"	"	120	673	1.990	0.1871	69.9	N.M.	
"	"	120	1009	1.989	0.7308	273.0	N.M.	R
Aluminum	2024	5	977	1.710	0.0030	1.3	N.M.	
"	"	24	1004	2.014	0.0031	1.1	--[e]	E
"	"	24[d]	671	1.801	0.0024	1.0	N.M.	
"	"	120	1020	1.740	0.0033	1.4	N.M.	Q
Aluminum	5154	5	999	1.710	0.0006	0.3	N.M.	
"	"	24	1004	1.610	0.0022	1.0	4	F
"	"	24[d]	689	1.999	0.0002	0.1	N.M.	
"	"	120	1017	1.999	0.0030	1.1	N.M.	R
Magnesium	M1A	24	1017	1.992	0.0041	1.7	--[f]	G
"	"	24[d]	680	2.009	0.0002	0.08	N.M.	
"	"	120	1006	1.972	0.0128	5.4	N.M.	N
Magnesium	AZ81	5	1006	2.810	0.0030	0.9	N.M.	
"	"	24	941	2.744	0.0029	0.9	--[g]	H
"	"	24[d]	698	2.768	0.0014	0.4	N.M.	
"	"	120	1000	2.780	0.0060	1.8	N.M.	O
Magnesium	AZ91	24	941	2.565	0.0092	2.9	--[g]	I
"	"	24[d]	689	2.828	0.0017	0.5	N.M.	
"	"	120	1002	2.642	0.0026	0.8	N.M.	P
Monel		5	1002	2.010	0.0032	1.4	--[h]	
"		24	1015	2.003	0.0061	2.7	5	J
"		24[d]	1040	1.997	0.0101	4.5	6	K
"		120	1020	1.984	0.0180	8.0	N.M.	
Nickel	A	5	1008	2.040	0.0025	1.0	--[h]	
"	"	24	997	1.992	0.0088	3.8	4	L
"	"	24[d]	1040	1.996	0.0231	9.9	10	M
"	"	120	1026	1.984	0.0349	15.0	N.M.	

TABLE 9 - continued

N.M. Not measured.

[a] Average penetration. Uniform film is 10μ. The calculated measurement is based on the combined weight gain and total area of two specimens.

[b] Average penetration. Uniform film 17μ.

[c] Average penetration. No uniform film seen.

[d] Test made under pressure (approximately 250 lbs.)

[e] No measureable coating seen. Evidence of scattered fluorine penetration.

[f] No uniform coating seen but scattered penetration to 25μ.

[g] No uniform coating seen.

[h] No coating seen.

TABLE 9A

CORROSION OF NICKEL BY FLUORINE
FLUORIDE FILM THICKNESS

Material	Type	Reference	Temp. °F	Time Hours	Film Thickness (μ) Calc.	Found
Nickel	A	1	1022	6.20	.25[a]	- [b]
"	A	1	1202	5.28	2.8 [a]	15.2
"	A[c]	3	932	5	.99	N.M.
"	A[c]	3	1112	5	6.1	N.M.
"	A	GCRL	1008	5	1.0	- [b]
"	A	"	997	24	3.8	4
"	A	"	1040	24[e]	9.9	10
"	A	"	1026	120	15.0	N.M.
Nickel	L[c]	3	932	5	.69	N.M.
"	L[d]	3	932	5	.78	N.M.
"	L[c]	3	1112	5	2.5	N.M.
"	L[d]	3	1112	5	1.9	N.M.
"	L[d]	3	1292	64.5	31.9	40.4
"	L[d]	3	1292	64.5	29.8	27.0
"	L	GCRL	1013	5	.92	N.M.

[a] Calculated at GCRL from reference data.

[b] Too thin to determine or not visible.

[c] Specimen preparation - acid dipped.

[d] Specimen preparation - polished

[e] Specimen exposed at 250 lbs pressure. Note: All other runs in gaseous fluorine flow from 25 to 100 cc/minute.

N.M. Not measured.

TABLE 10

CORROSION OF METALS AND ALLOYS BY FLUORINE

Material	Type	Reference	Temp. °F	Corrosion Rate IPY
Aluminum	1100-H14	GCRL	662	.0066[a]
"	"	"	"	.0056[a]
"	2S	5	752	nil
"	"	5	842	nil
"	"	5	932	.1577
"	1100-H14	GCRL	993	1.8221[a]
"	2S	5	1112	.2190[a]
Copper	ETP	GCRL	691	.0324[a]
"	"	"	"	.0289[a]
"	- [b]	5	752	1.927
"	- [b]	4	932	2.365
"	- [b]	5	"	1.402
"	ETP	GCRL	984	.2453[a]
"	"	"	"	.2190[a]
"	- [b]	1	1022	.1139[a]
Inconel	-	GCRL	694	.0753[a]
"	-	"	"	.0797[a]
"	-	5	752	.4555
"	-	5	842	1.139
"	-	5	932	.7446
"	-	GCRL	997	3.451 [a]
"	-	1	1022	3.241 [a]
Monel	-	GCRL	666	.0012[d]
"	-	"	"	.0025[d]
"	-	5	752	.0061[a]
"	-	5	842	.0219[a]
"	-	4	932	.0237
"	-	5	"	.0237[a]
"	-	GCRL	1002	.0289[a]
"	-	"	"	.0307[a]
Nickel	A	GCRL	705	.0006[d]
"	"	"	"	.0028[a]
"	- [b]	5	752	.0841
"	- [b]	5	842	.0228
"	- [b]	4	932	.0718
"	- [b]	5	"	.0613
"	A	GCRL	1008	.0245[a]
"	"	"	"	.0219[a]
"	"	1	1022	.0438[a]

TABLE 10 - continued

Material	Type	Reference	Temp. °F	Corrosion Rate IPY
Stainless Steel	#347	5	392	nil
" "	"	GCRL	421	.0042[a]
" "	"	"	"	.0035[a]
" "	"	5	482	1.734
" "	"	5	572	2.558
" "	"	5	662	6.220
" "	"	GCRL	711	4.266 [d]
" "	"	"	"	4.231 [d]
" "	"	5	752	8.839
Steel	LC	GCRL	682	.3942[a]
"	"	"	"	.4030[a]
"	(0.007% Si)	5	752	.1402
"	"	5	842	3.592
"	"	5	932	87.6
"	LC	GCRL	>980	-- [c]
Titanium	A-75	6	-320	.0002[d]
"	A-55	GCRL	"	.3154[a]
"	"	"	"	.1489[a]
"	A-75	6	70	nil [a]
"	"	6	"	nil [a]
"	A-55	GCRL	77	.0045[a]
"	"	"	"	.0067[a]

[a] From weight gain.

[b] Not specified.

[c] Reacted completely.

[d] From weight loss.

NOTE: GCRL data was from 5 hour runs since data from other sources in this table covered short exposure periods.

TABLE 11

ANALYSES[a] AND DENSITIES OF METALS AND ALLOYS STUDIED
(SPECIMEN PREPARATION)[b]

Material	Type	Analysis	% Composition												Density	Treatment[c]
			Al	Cr	Cu	Fe	Mg	Mn	Mo	Ni	Si	Sn	Th	Zn	Zr	g/cc	
Aluminum	1100 H-14	Nominal	>99.0								<1.0					2.71	A
"	2024 T-3	Actual	93.0		4.41	0.36	1.41	0.63								2.71	A
"	3003 H-14	"	98.0			0.51		1.11								2.74	A
"	5154 H-34	"	95.8				3.45									2.65	A
Brass	Red	"			85.66									14.34		8.75	B
"	Amer.#243	"			61.50							1.80		36.70		8.4	B
Copper	ETP	"			100											8.92	B
Illium	R	"		21.70	2.52	4.18		0.72	5.70	64.57	0.45					8.31	C
Inconel	-	"		15.23		7.00				77.28						8.51	D
Magnesium	M1A	Nominal					98.5	1.5								1.76	E
"	HK31A	Actual					96.0						3.3		0.6	1.9	E
"	AZ81 A&C	"	7.6				91.0	0.34						0.76		1.81	E
"	AZ91C	Nominal	8.4-9.3				Bal.				<.3			.4-1.0		1.81	E
Monel	Rolled	Actual			29.7	1.3		0.9		67.7						8.84	F
"	Cast	Nominal			29.5	1.5				67.5	1.25					8.63	F

			Al	Cr	Fe	Hf	Mn	Nb	Ni	Si	Sn	Ta	Ti	V	Zr		
Nickel	A&L	Nominal							99.45							8.89	G
Stainless Steel	304	Actual		18.53	71.3		0.61		8.88	0.62						8.02	H
" "	304L	"		18.41	70.38		1.12		9.49	0.37						8.02	H
" "	347	"		18.53	67.4		1.56	0.76	11.10	0.57						8.02	H
Steel	Low C	Nominal			99+											7.86	I
Tantalum	-	Actual										99.95				16.6	J
Titanium	A-55	"											99.94			4.5	K
"	B120-VCA	"	3.7	10.7									73.0	12.5		4.82	K
Zirconium	-	"				2.0									98.0	6.5	H
Zircalloy	II	"									1.65				98.0	6.5	H

TABLE 11 - continued

[a] Components of less than 0.25% are omitted.

[b] Edge surface of all materials except tantalum and titanium B120-VCA were milled and finished with #240 grit emery, in the latter cases they were abrasive ground and finished with #240 grit emery; face surfaces of all materials except cast monel were the original mill surfaces (as received), those of cast monel being milled and finished with #240 grit emery; detailed pickling procedures follow.

[c] Pickling procedure.

DETAILED PICKLING PROCEDURES

A. 100 ml H_2O
4-8 gms NaOH
pickle for 10-30 sec. at 180°-200°F

B. Step I: 5-10% H_2SO_4 (Sp.Gr. 1.83) by volume
0.5% $CuSO_4$
Balance H_2O
pickle for 1 minute at 125°-150°F

Step II: 7% H_2SO_4 (Sp.Gr. 1.83) by volume
6 oz. $Na_2Cr_2O_7 \cdot 2H_2O$
dilute to 1 gallon with water
pickle for 15-60 sec. at 80°-120°F

C. 100 ml H_2O
100 ml 38°Be HCl
15 ml 48% HF
pickle for 15 minutes at 70°-100°F

D. 700 ml H_2O
250 ml 38°Be HNO_3
50 ml 40% HF
pickle for 5-10 minutes at 120°-140°F

E. 270 gms Chromic Acid
4 gms MgF_2
60 gms $Ca(NO_3)_2 \cdot 4H_2O$
1166 ml H_2O
pickle for 1 minute at room temperature. Neutralize in 5% Na_2CO_3

F. Step I: 1000 ml H_2O
415 ml 38% HCl
30 gms $CuCl_2 \cdot 2H_2O$
pickle for 5-10 minutes at 180°F, rinse in hot water

DETAILED PICKLING PROCEDURES - continued

Step II: 1000 ml H_2O
100 ml 66°Be H_2SO_4
132 gms $Na_2Cr_2O_7 \cdot 2H_2O$

pickle for 3-5 minutes at 70°-100°F, rinse in cold water and neutralize in 1-2% NH_4OH

G. Step I: 100 ml H_2O
50 ml 20°Be HCl
30 gms $CuCl_2 \cdot 2H_2O$

pickle for 10-20 minutes at 180°F, rinse in hot water

Step II: 100 ml H_2O
150 ml 66°Be H_2SO_4
225 ml 38°Be HNO_3

pickle at room temperature for 5-20 seconds

H. 13 ml 70% HNO_3
2 ml 48% HF
85 ml H_2O

pickle for 5 minutes at 140°-160°F

I. Pickle in 31% HCl at 140°-160°F for 10 minutes, then neutralize in a 5% Na_2CO_3 solution.

J. 30 ml 48% HF
170 ml H_2O

pickle for 10-20 minutes at 140°F

TABLE 12

WEIGHT GAIN FACTORS

Material	Type	Wgt. Gain Factor x 10^3
Aluminum	1100	10.656
"	2024	11.159
"	3003	10.907
"	5154	11.080
Brass	red	11.711
"	Amer. #243	12.260
Copper	ETP	11.440
Illium	R	9.198[a]
"	"	11.178[d]
"	"	36.333[f]
Inconel		9.710[b]
"		17.104[f]
Magnesium	M1A	22.368
"	HK31A	21.181
"	AZ81C	21.207
"	AZ91C	21.207
Monel	Rolled	10.869
"	Cast	10.678[a]
"	"	11.481[e]
Nickel	A&L	10.668
Stainless Steel	#304	7.536[a]
" "	"	7.711[e]
" "	304L	7.711[e]
" "	#347	7.630[a]
" "	"	7.795[d]
Steel	LC	7.680
Tantalum		7.006
Titanium	A-55	8.550[c]
"	B120-VCA	7.978[c]
Zirconium		11.382
Zircalloy	II	11.353

[a] For tests at approximately -320°F.

[b] For tests from -320°F to 662°F (approximately).

[c] For tests up to room temperature.

[d] For tests from room temperature to 662°F(approximately).

[e] For tests at room temperature and above.

[f] For tests above 932°F.

TABLE 13

METAL FLUORIDE DATA

Metal	Metal Fluoride Formed	Melting Pt. °F	Boiling Pt. °F
Aluminum	AlF_3	1904	
Chromium	CrF_3	>1832	
"	CrF_4[a]		
"	CrF_5[a]		
Copper	CuF_2	1742	
Hafnium	HfF_4[b]		
Iron	FeF_3[b]		
Magnesium	MgF_2	2545	
Manganese	MnF_2	1573	
"	MnF_3[c]		
Molybdenum	MoF_6	63	95
Nickel	NiF_2[b]		
Niobium	NbF_5	168	457
Silicon	SiF_4	-107	-85
Tantalum	TaF_5	206	445
Thorium	ThF_4[b]	red heat	
Tin	SnF_4		1301
Titanium	TiF_4		543
Vanadium	VF_5[d]		119
Zinc	ZnF_2	1602	
Zirconium	ZrF_4[e]		

[a] CrF_4 and CrF_5 are assumed to be formed between 662 and 932°F, and to be volatile above 662°F.

[b] It is assumed that this compound is not volatile below 1000°F.

[c] MnF_3 dissociates at moderate temperatures; therefore, formation of only MnF_2 is assumed.

[d] VF_5 boils at 119°F.

[e] ZrF_4 volatilizes at 1472-1832°F.

TABLE 14

WEIGHT LOSS FACTORS

Material	Type	Wgt. Loss Factor x 10^3 [a]
Aluminum	1100	-22.517
"	2024	-22.517
"	3003	-22.700
"	5154	-23.026
Brass	Red	- 7.264
Copper	ETP	- 6.841
Inconel	-	- 7.170
Magnesium	M1A	-34.670
"	AZ81A	-33.713
"	AZ91C	-33.713
Monel	-	- 6.903
Nickel	A	- 6.864
Stainless Steel	304	- 7.608[b]
" "	304L	- 7.608[b]
" "	347	- 7.608[b]
Steel	LC	- 7.763
Tantalum	-	- 3.676[c]
Titanium	A-55	- 9.780[d]
"	B120-VCA	-19.342[d]
Zirconium	-	- 9.780[b]
Zircalloy	II	- 9.683[b]

[a] Actual factor has been multiplied by 10^3; negative values indicate weight loss factors, as opposed to positive weight gain factors, and when applied to negative weight changes will result in positive penetration rates.

[b] When the specimen can be cleaned and film or scale is assumed to be completely removed.

[c] For tests at -320°F.

[d] For tests above 300°F; while TiF_4 has a boiling point of 543°F, it is assumed that, at moderately high temperatures, the TiF_4 formed will have an appreciable vapor pressure and will, therefore, volatilize fairly rapidly in a stream of gas.

TABLE 15

FILM THICKNESS FACTORS

Material	Type	Film Thickness Factor
Aluminum	1100	743.8
"	2024	745.0
"	5154	747.1
Magnesium	M1A	835.7
"	AZ81	815.6
"	AZ91	815.6
Monel	--	886.5
Nickel	A & L	851.8

NOTES ON EXHIBITS

Exhibits A to M are photomicrographs reduced in reproduction from 1000 x to 500 x magnification. In these exhibits, the fluorinated specimen is the bottom-most specimen in the picture. The upper metallic specimen was not exposed to fluorine. The black band seen between these specimens is the Bakelite molding compound. The fluoride film generally appears as a light gray in these reproductions as for example, in Exhibits A and B. The fluoride formation in Exhibit C, however, reproduced as a dark gray penetration into the lighter aluminum. Exhibit F is different in that we show a corner of the exposed specimen rather than the straight edge. Exhibits N, O, P and Q are approximately twice actual size. Exhibit Q does not show too clearly the alligatored surface, (Re: Exhibits O and P), in this printing. Some areas of porosity (black) can still be seen on the broken edges. Exhibit R shows approximately the actual size of the test specimens.

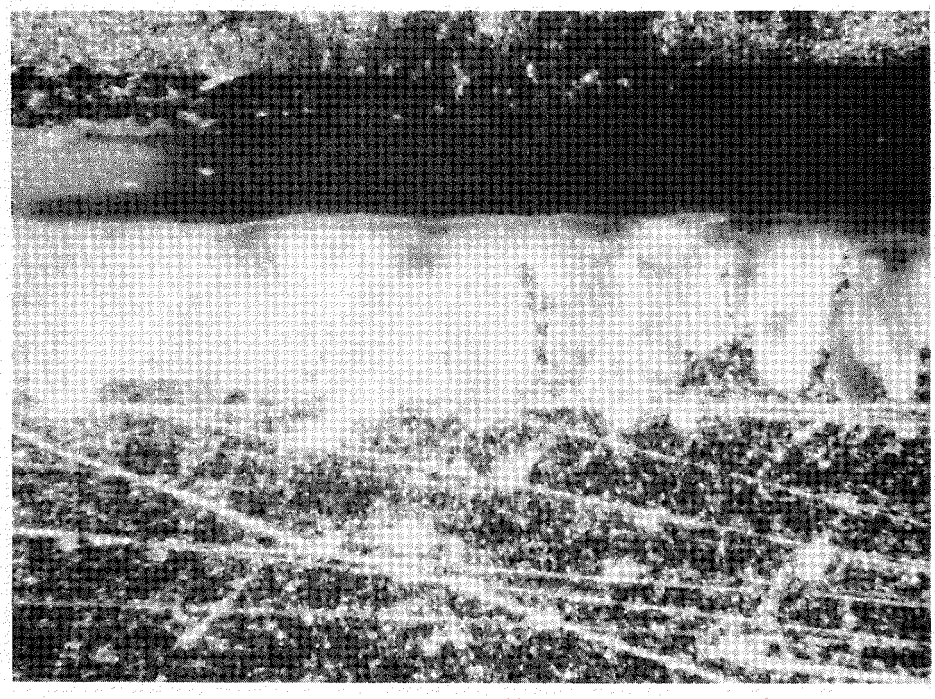

Exhibit A - Aluminum 1100 exposed to fluorine at 993°F for 5 hours.

Exhibit B - Aluminum 1100 exposed to fluorine at 1020°F for 24 hours.

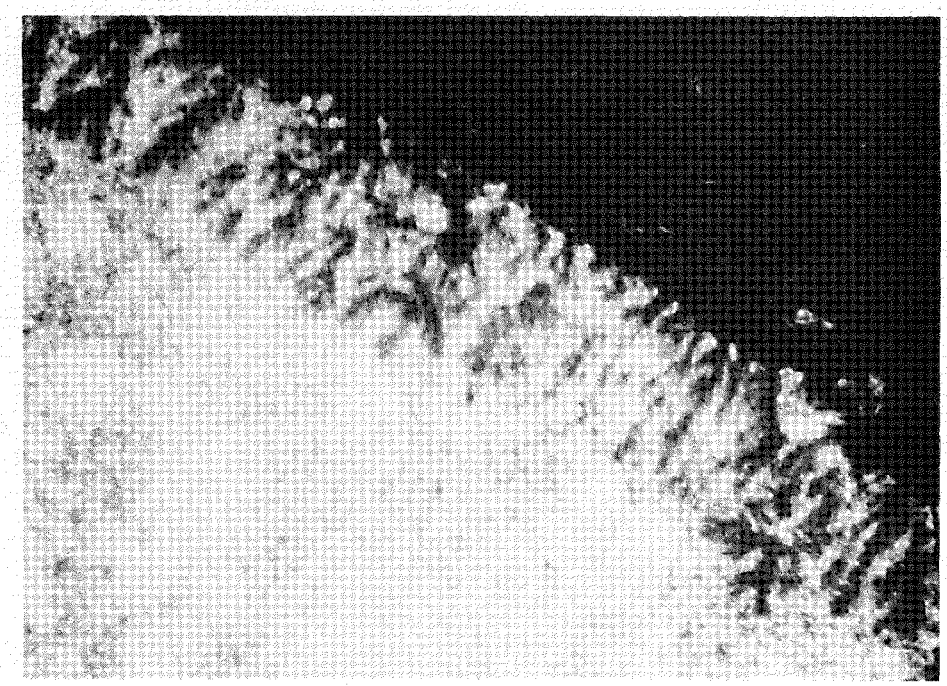

Exhibit C - Aluminum 1100 exposed to fluorine at 689°F at an initial pressure of 250 lbs. for 24 hours.

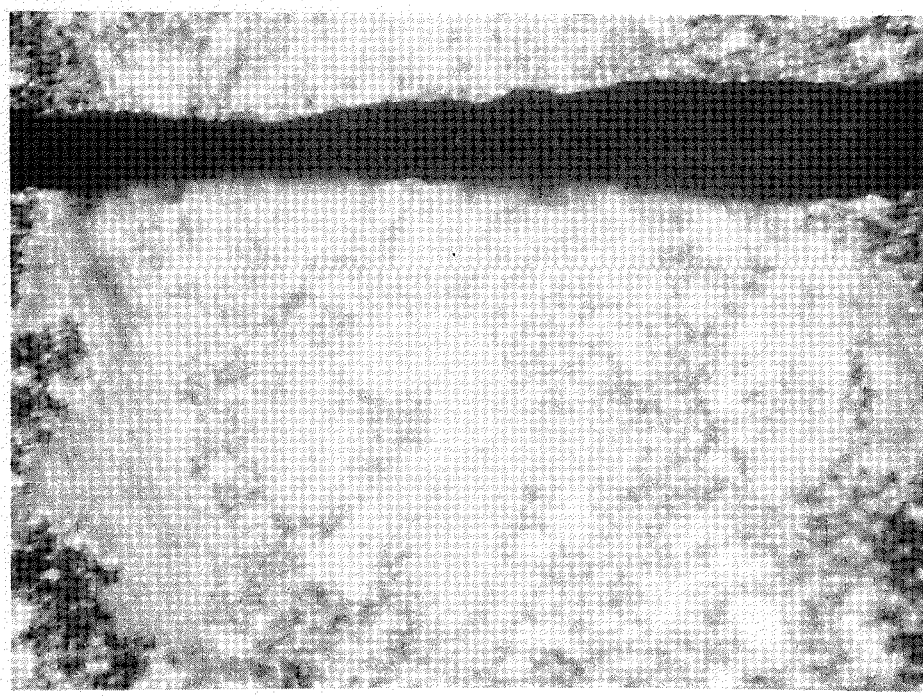

Exhibit D - Aluminum 1100 exposed to fluorine at 1004°F at an initial pressure of 270 lbs. for 24 hours.

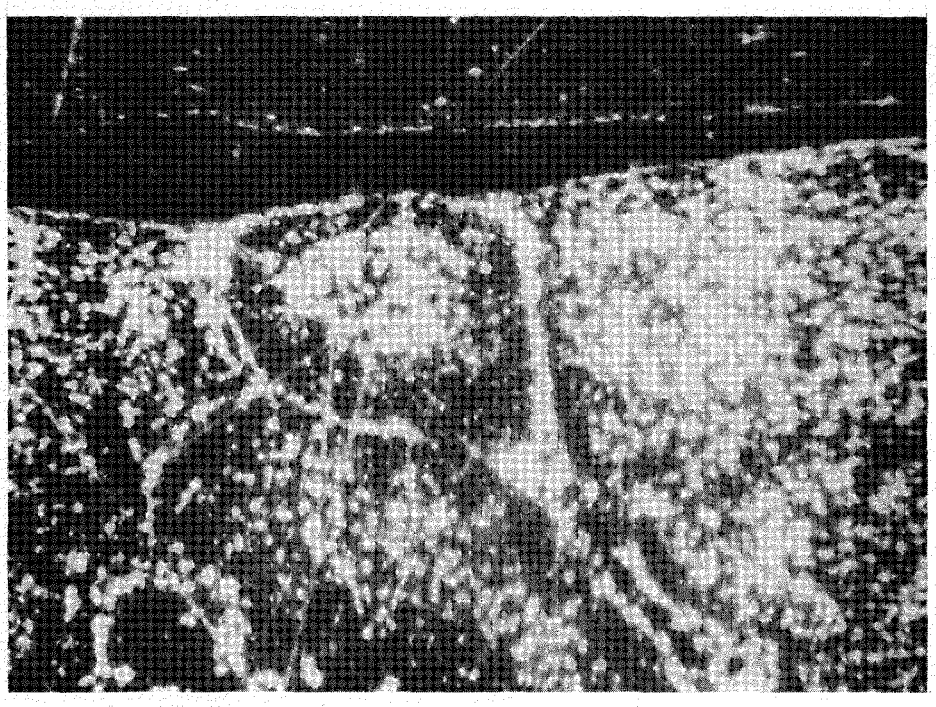

Exhibit E - Aluminum 2024 exposed to fluorine at 1004°F for 24 hours.

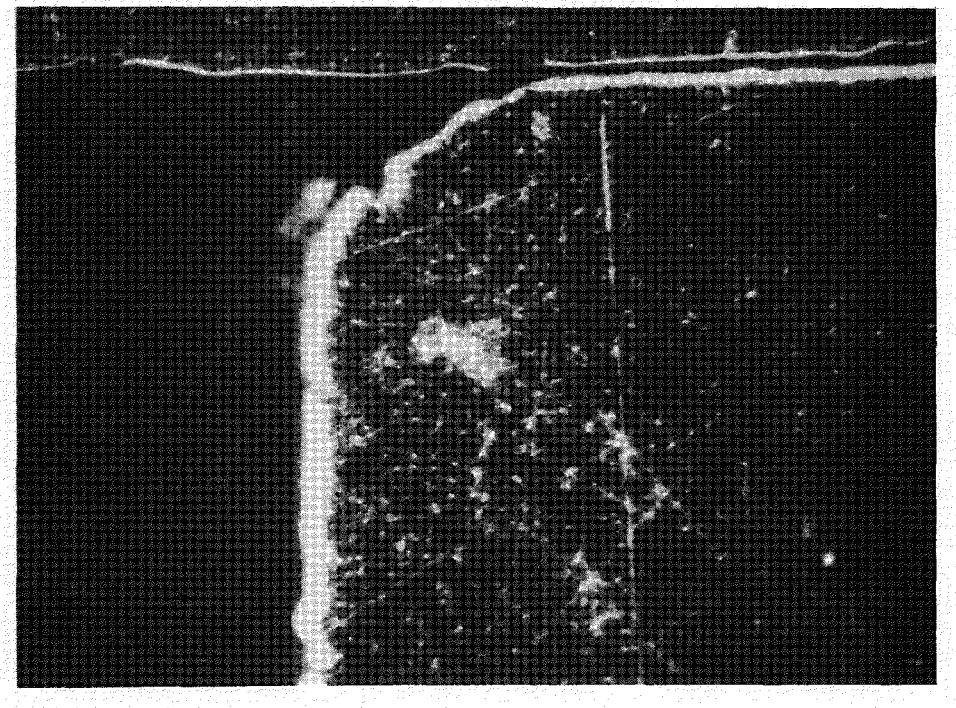

Exhibit F - Aluminum 5154 exposed to fluorine at 1004°F for 24 hours.

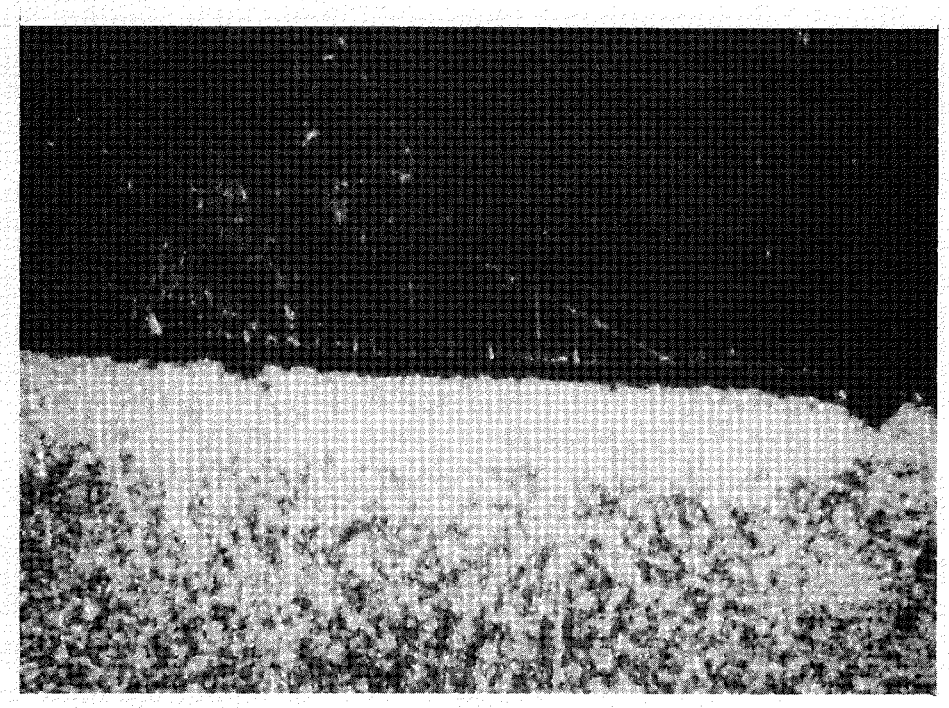

Exhibit G - Magnesium M1A exposed to fluorine at 1017°F for 24 hours.

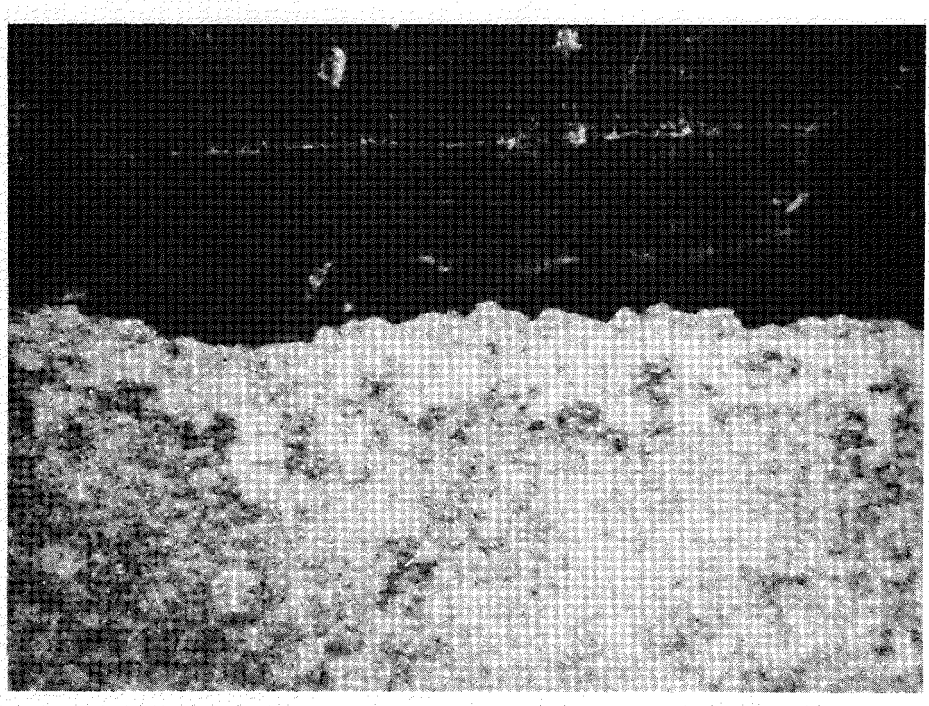

Exhibit H - Magnesium AZ81 exposed to fluorine at 941°F for 24 hours.

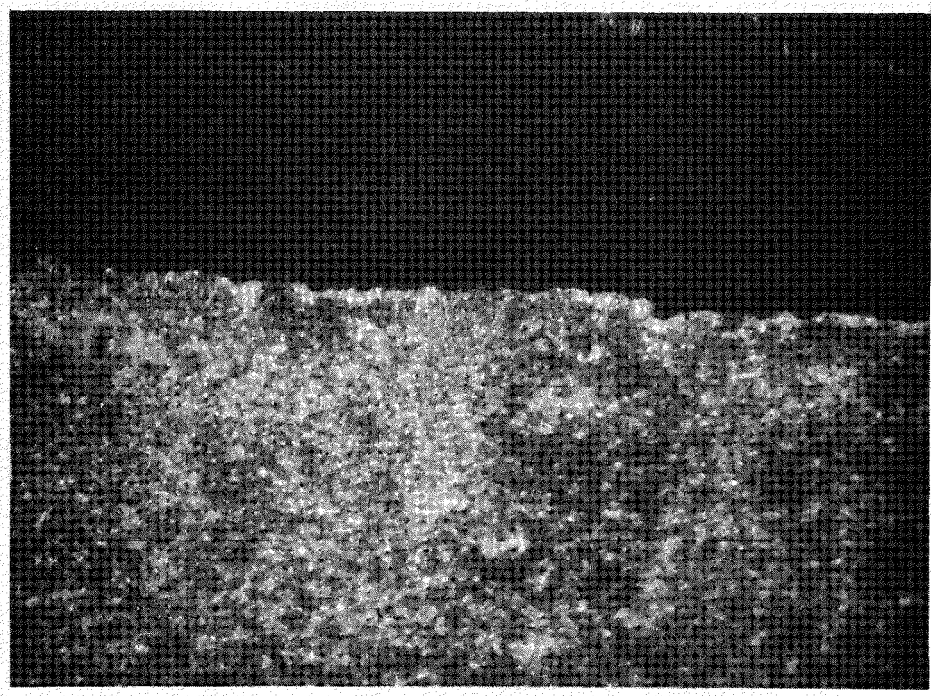

Exhibit I - Magnesium AZ91 exposed to fluorine at 941°F for 24 hours.

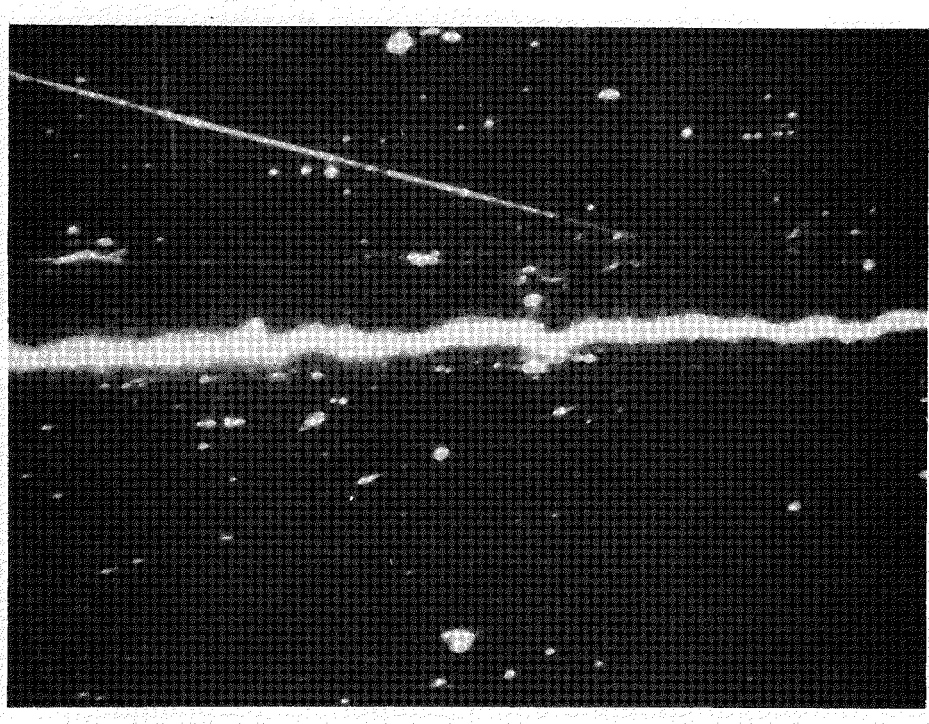

Exhibit J - Monel exposed to fluorine at 1015°F for 24 hours.

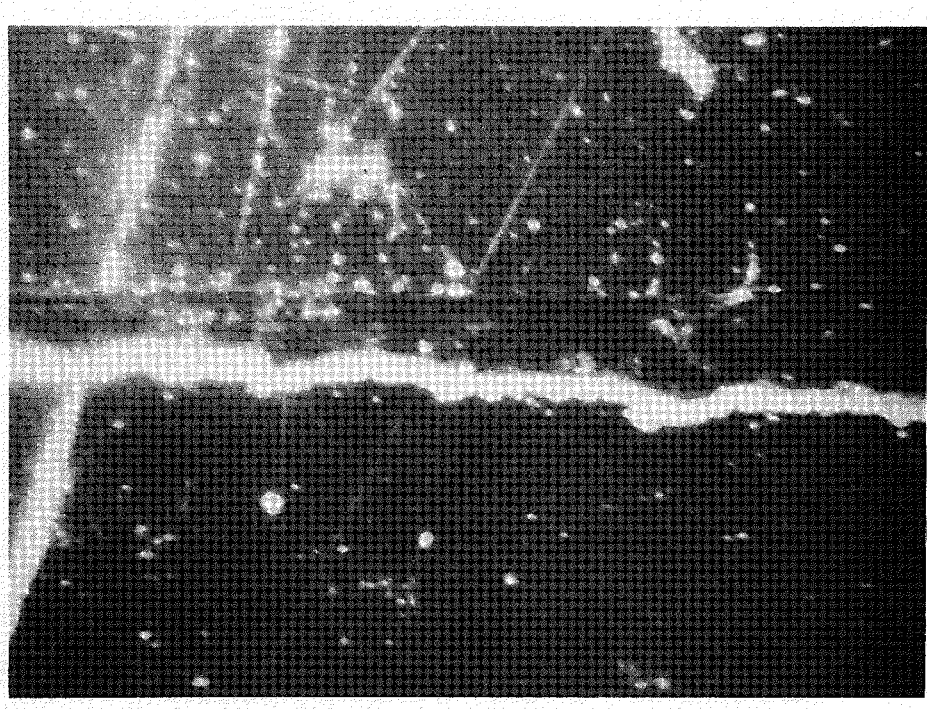

Exhibit K - Monel exposed to fluorine at 1040°F at an initial pressure of 255 lbs. for 24 hours.

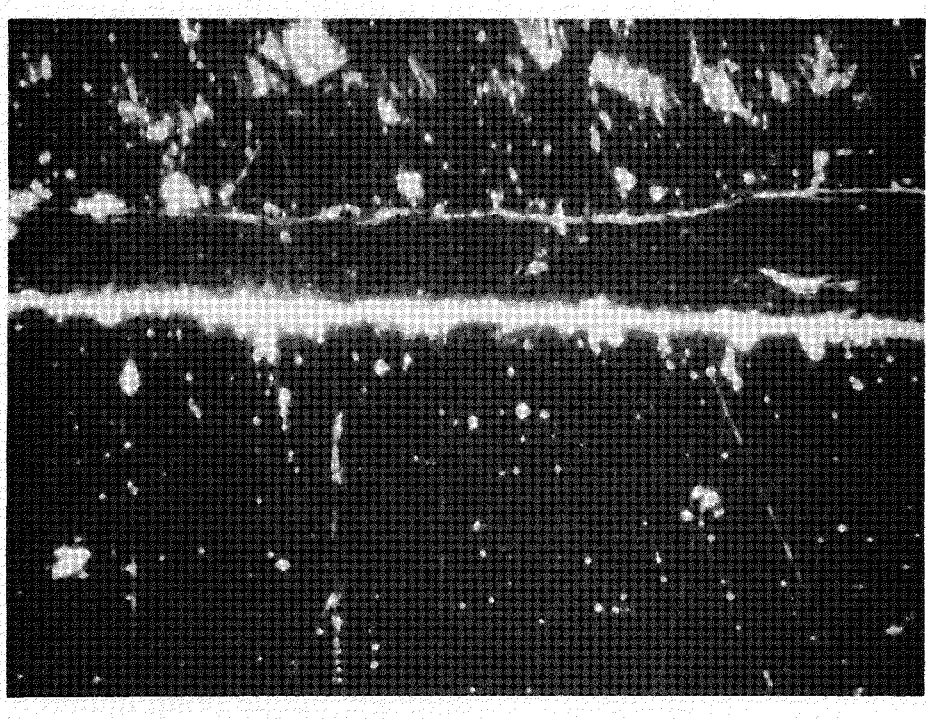

Exhibit L - Nickel exposed to fluorine at 997°F for 24 hours.

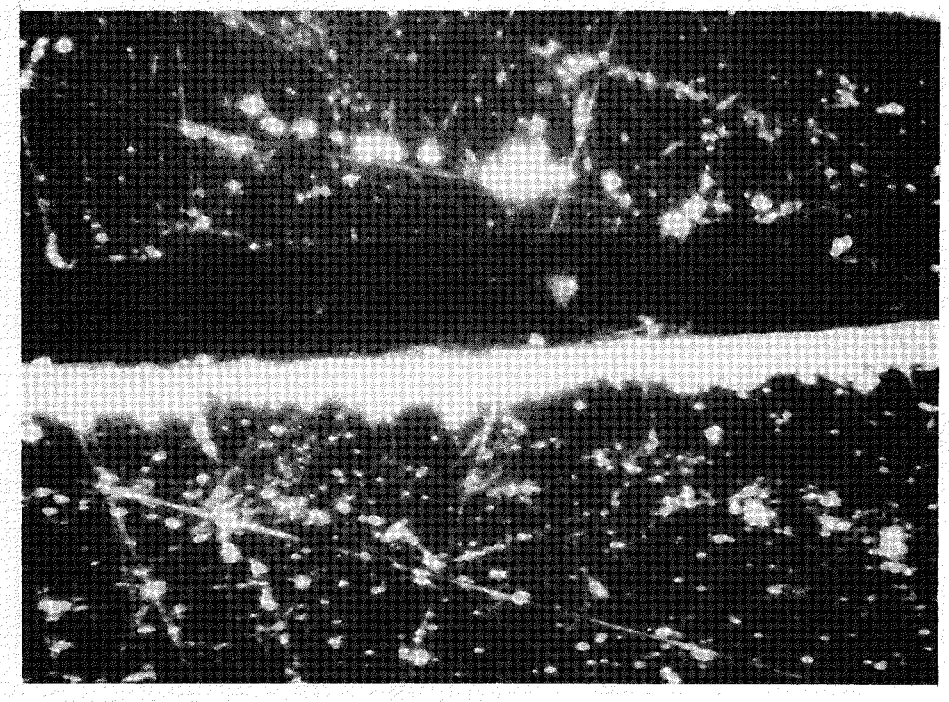

Exhibit M - Nickel exposed to fluorine at 1040°F at an initial pressure of 260 lbs. for 24 hours.

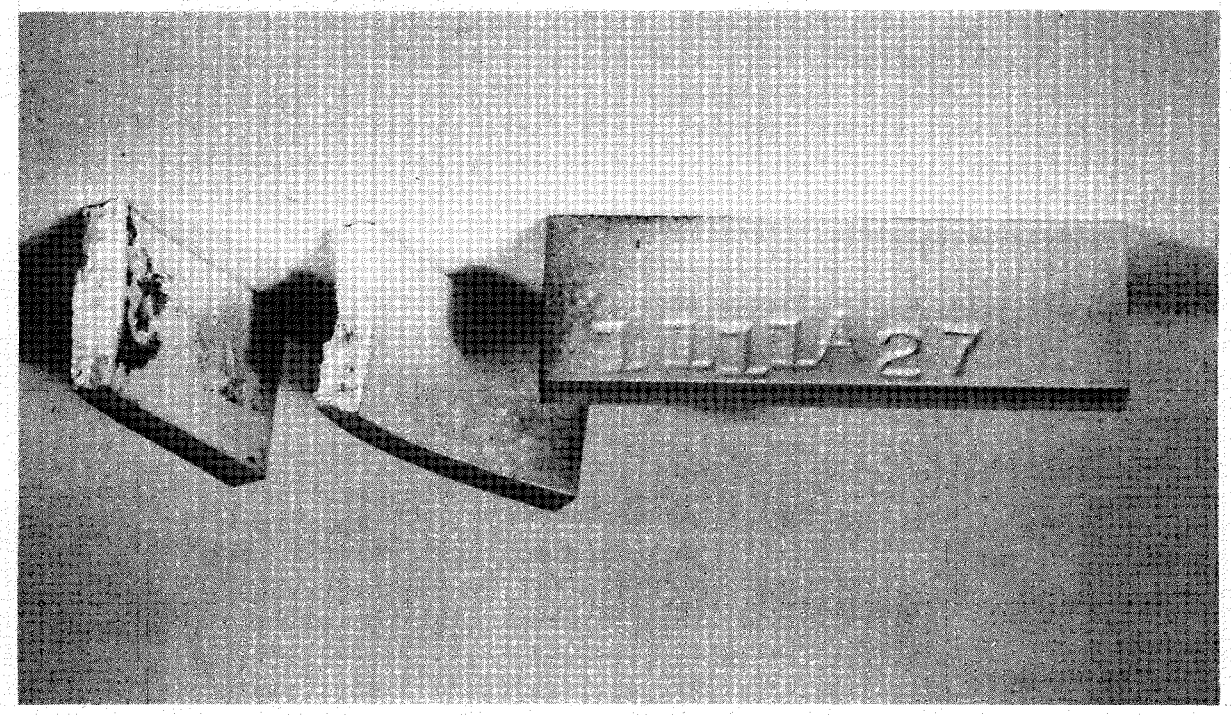

Exhibit N - Magnesium M1A exposed to fluorine at 1006°F for 5 days.

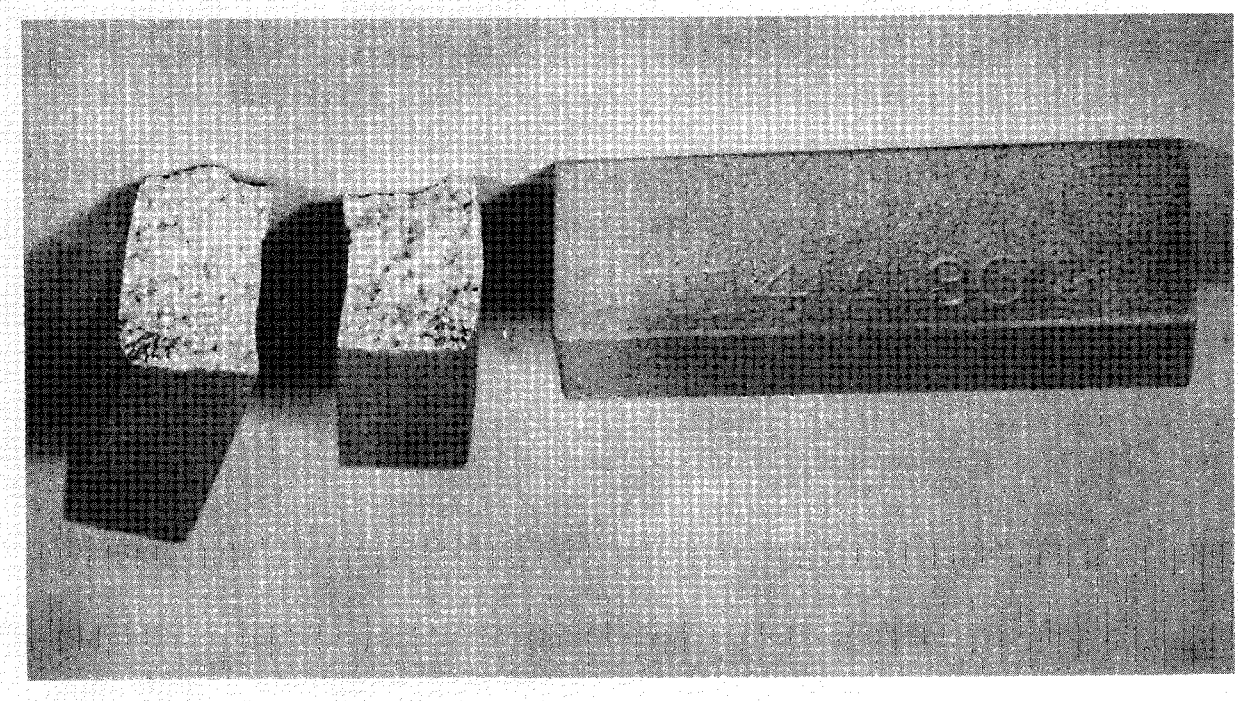

Exhibit O - Magnesium AZ81 exposed to fluorine at 1000°F for 5 days.

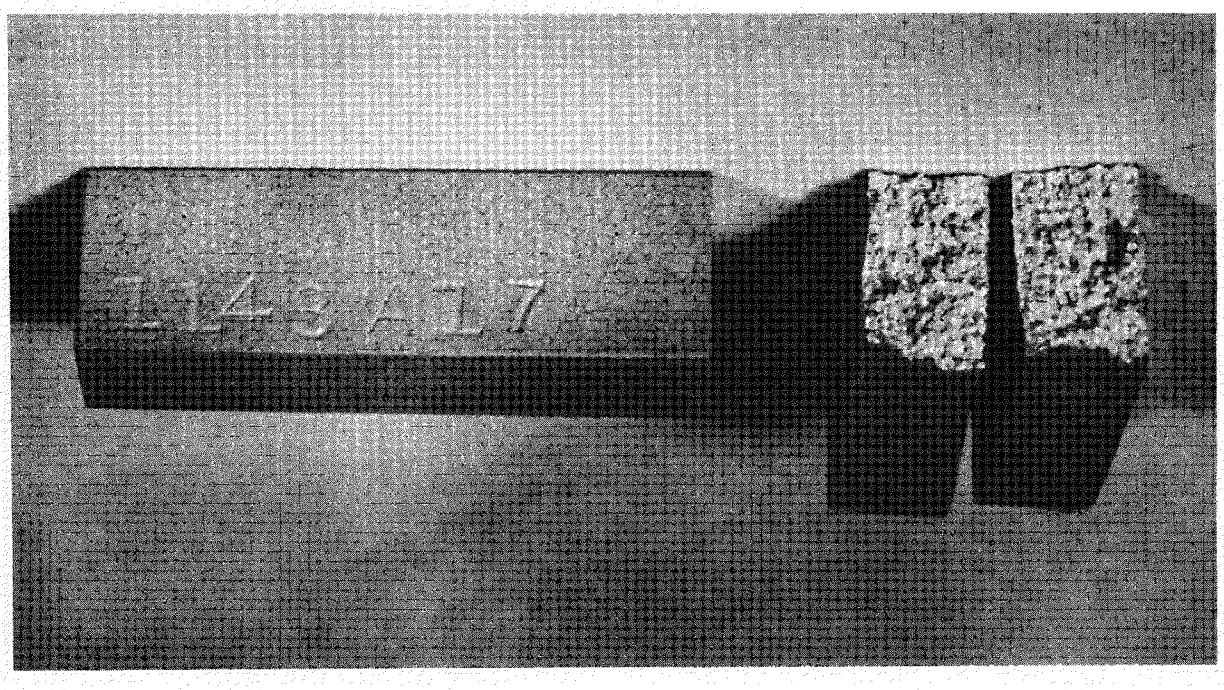

Exhibit P - Magnesium AZ91 exposed to fluorine at 1002°F for 5 days.

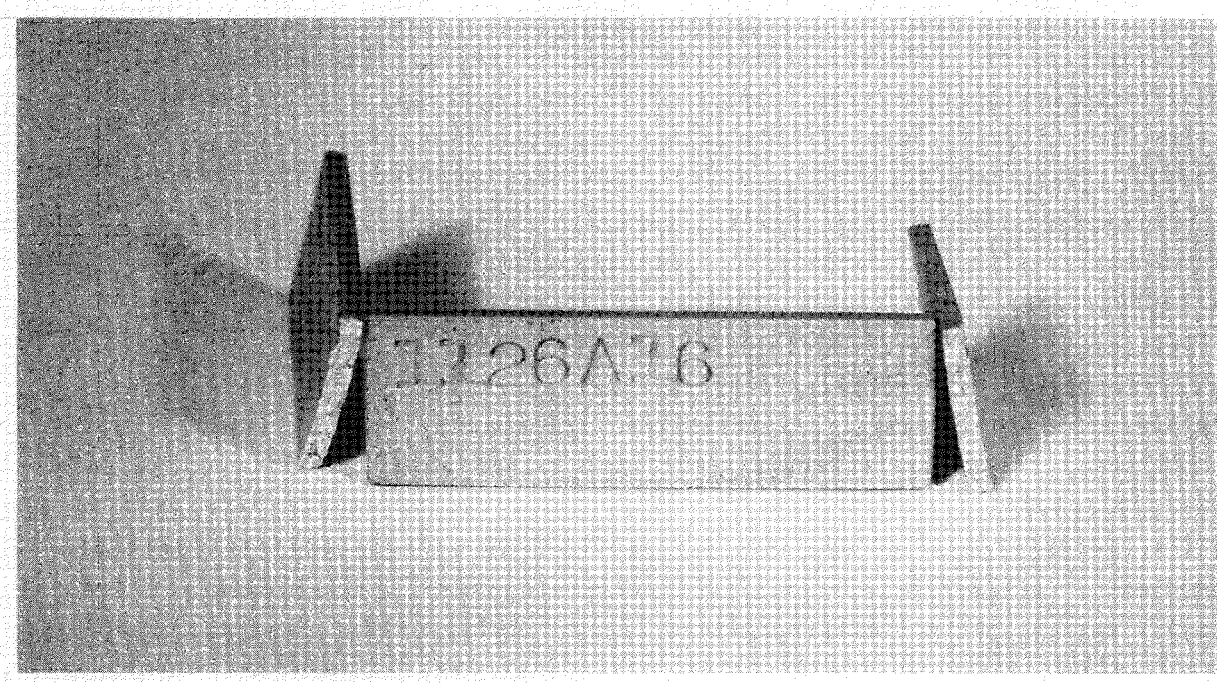

Exhibit Q - Aluminum 2024 exposed to fluorine at 1020°F for 5 days.

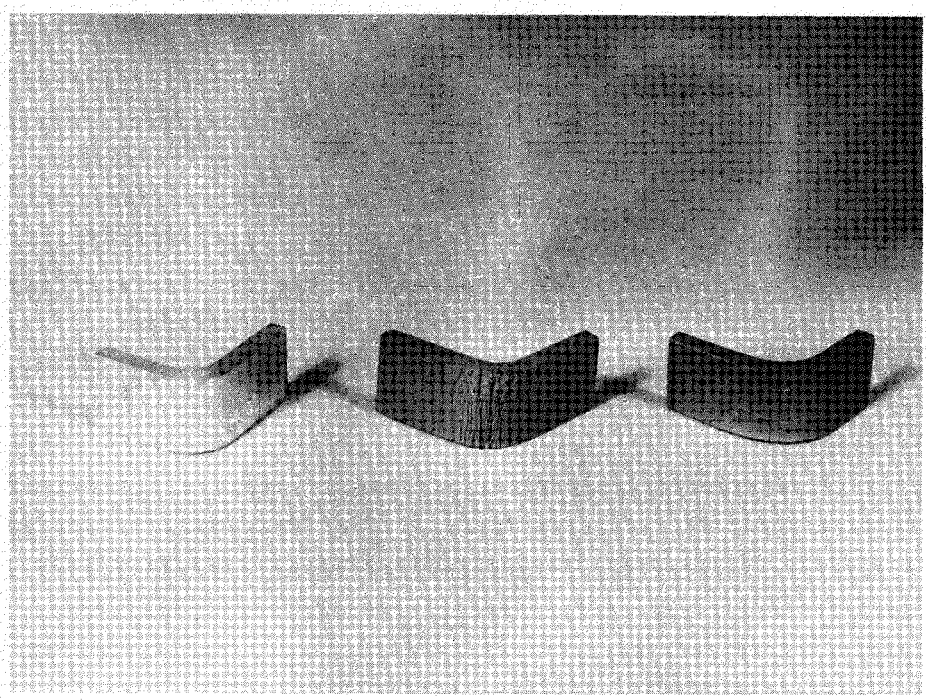

Exhibit R - (L. to R.) Unexposed Aluminum 1100, Aluminum 1100 exposed to fluorine at 1009°F for 5 days and Aluminum 5154 exposed to fluorine at 1017°F for 5 days.