DYNAMIC COMPATIBILITY OF HALOGEN PROPELLANTS

E. M. Vander Wall, et al

Aerojet Liquid Rocket Company

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AEROJET LIQUID ROCKET COMPANY
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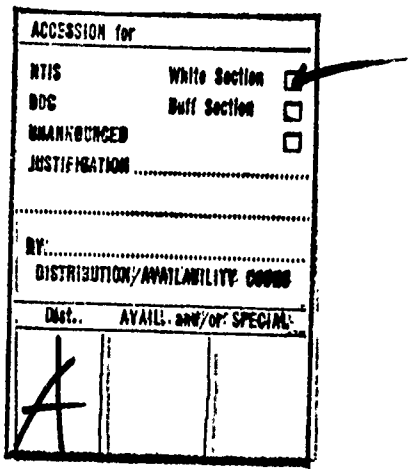

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13. ABSTRACT

The objective of this program was to establish design criteria on the dynamic compatibility of liquid and gaseous fluorine and chlorine pentafluoride with designated metals. Eleven metallic materials of construction were included in the program: 301 (cryoformed), 304L, 321 and 347 stainless steel; A-286 super alloy steel; 2219 and 6061 aluminum; OFHC copper; Monel K-500; Inconel 718; and Nickel 200. Six tests were used to obtain the data required to establish the design criteria: (1) maximum velocity, (2) adiabatic compression (U-tube), (3) liquid impact, (4) flexing/film degradation, (5) vibration/film degradation, and (6) liquid phase shock wave.

Nickel 200 was found to be suitable for use at temperatures up to 1750°F in gaseous fluorine and chlorine pentafluoride. Inconel 718, under comparable testing, was suitable to 1550°F, while Monel K-500 could withstand 1500°F except when subjected to adiabatic compression. The austenitic stainless steels were suitable at temperatures up to 1100°F except when subjected to adiabatic compression in chlorine pentafluoride. Copper and the aluminums were resistant to attack at temperatures up to 800°F.

An extensive literature survey and critical review were conducted in conjunction with the experimental portion of the program.

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Adiabatic Compression Tests						
Liquid Phase Shock Wave Tests						
Liquid Impact Tests on Heated Metals						
Flexing/Film Degradation Tests						
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FOREWORD

This report covers the work performed under Contract F04611-72-C-0031, "Dynamic Compatibility of Halogen Propellants," performed by the Aerojet Liquid Rocket Company at Sacramento, California, and conducted under Air Force Project 5730, Task 07. The performance period covered from 3 January 1972 to 31 August 1972.

The project manager was Dr. S. D. Rosenberg; the project chemist was Dr. E. M. Vander Wall. The experimental work was conducted by Dr. Vander Wall; R. L. Beegle, Jr., chemist; and J. A. Cabeal, senior laboratory technician. The literature search and review was conducted by R. E. Anderson, chemistry specialist.

The program was administered under the direction of the Air Force Rocket Propulsion Laboratory, Capt. Howard M. White, project engineer.

This report was submitted by the authors on 31 October 1972.

This report has been reviewed and is approved.

Howard M. White, Capt., USAF
Project Engineer

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SECTION I

INTRODUCTION

The use of fluorine and chlorine pentafluoride in rocket engines and laser systems has attracted considerable attention over a period of years. A large number of basic research and technology programs involving these very active oxidizers has been conducted. The inherent reactivity of these oxidizers is well known. Consequently, material compatibility problems have always been aspects of the technology programs. A large amount of compatibility data has been generated and compiled in connection with static systems; however, the compatibility in dynamic systems has received only limited attention and a comprehensive compilation of the data did not exist. The purpose of the investigation which is discussed in this report was to establish design criteria on the dynamic compatibility of liquid and gaseous fluorine and chlorine pentafluoride with metals normally encountered in halogen systems. Prior to conducting the experimental portion of the program, an extensive literature survey was conducted to define the areas in which experimental data were lacking. The results of the literature search are included as an appendix to this report.

During the course of the experimental portion of the investigation, eleven metallic materials of construction were considered:

Steels 301 (cryo-formed), 304L, 321, 347, and A-286

Aluminums 6061 and 2219

Oxygen-free high conductivity (OFHC) copper

Monel K-500

Inconel 718

Nickel 200

The materials were subjected to six tests with the oxidizers:

Maximum Velocity Test

Adiabatic Compression Test

Liquid Phase Shock Wave Test

Liquid Impact Test

Flexing/Film Degradation Test

Vibration/Film Degradation Test

The discussion of the experimental results is presented in the order in which the tests are specified above.

SECTION II

EXPERIMENTAL RESULTS AND DISCUSSIONS

PHASE I - DETAILED TEST PLAN

The work in this phase was divided into two principal tasks: (1) Literature Search and Review and (2) Compatibility Test Plan. The purpose of the literature search and review was to document the current status of dynamic compatibility data of fluorine and chlorine pentafluoride with various metallic construction materials and to establish the proper basis for further experimental work to acquire adequate design criteria data.

A. LITERATURE SEARCH AND REVIEW

Computer searches of the NASA and DDC report files were requested and obtained. Manual searches of Aerojet Liquid Rocket Company Technical Information Center Indexes, Liquid Propulsion Information Agency (LPIA) and Chemical Propulsion Information Agency (CPIA) Abstracts, Nuclear Science Abstracts, Chemical Abstracts, and Engineering Indexes were also conducted. Based on the index and/or abstract information, the pertinent reports were obtained and reviewed in detail. The search and review is presented in Appendix A of this report. The significant findings of the literature search and review and their impact on the testing in Phase II are discussed in the following paragraphs.

No data were found to be available concerning the effect of the adiabatic compression of either fluorine or chlorine pentafluoride vapors in the presence of metals. Thus, the literature had no impact on the testing insofar as adiabatic compression tests were concerned.

The literature search and review revealed four sources of data concerning the effects of fluorine flow on metals (see Appendix A, pp. A-1 to A-25) and three sources that concerned chlorine pentafluoride flow testing (see Appendix A, pp. A-58 to A-60 and A-65). The data from these sources show that the liquid phases have minimal effects on metals at velocities as high as 400 ft/sec. The data show that temperatures well above the critical temperatures are required before the flowing gases attack the metals significantly. The data pertinent to this flow/temperature are, however, extremely limited. From this it became apparent that additional flow testing as a function of velocity should be restricted to elevated temperatures where only the gas phases can exist.

The literature search and review revealed that liquid fluorine impacting on metal surfaces has little or no effect at low temperatures (see Appendix A, pp. A-19 to A-25). No comparable data were found for chlorine pentafluoride; however, liquid chlorine trifluoride was found to be destructive when impacting metals of elevated temperatures (see Appendix A, pp. A-61 to A-62). From this it was concluded that liquid impact testing should be restricted to heated metal surfaces.

II, A, Literature Search and Review (cont.)

Ample data on the effect of flexing metals in liquid and gaseous fluorine were found in the literature (see Appendix A, pp. A-37 to A-48). These data indicated that flexing below the elastic limit does not enhance the corrosion significantly. No corresponding data were found concerning chlorine pentafluoride. Therefore, flexing/film degradation testing was deemed necessary only with chlorine pentafluoride.

Limited data were found concerning the effect of ultrasonic vibration/cavitation on aluminum in liquid fluorine (Appendix A, p. A-49) and the effect of low frequency vibration on various metals in gaseous fluorine (Appendix A, p. A-50). No corresponding data were found for chlorine pentafluoride. Insufficient data were available to impact the test planning.

Data regarding the effects of shock waves promulgated through liquid fluorine were limited to two sources (see Appendix A, pp. A-56, A-57). No data were found concerning these effects in liquid chlorine pentafluoride. Based on the available data, it was concluded that further testing with liquid fluorine was justified and testing with chlorine pentafluoride was required.

Data regarding the effects of mechanical impacting in liquid and gaseous fluorine and chlorine pentafluoride were found and are presented in Appendix A, pp. A-26 to A-36, A-66 to A-70. This type of testing was beyond the scope of the planned experimental work.

A category of test data involving friction, abrasion, and fracture was also found in the literature. These data are presented in Appendix A, pp. A-51 to A-55, A-66 to A-67. This type of testing was also beyond the scope of the planned experimental program.

B. TEST PLAN

The objective of the program was to establish design criteria on the dynamic compatibility of liquid and gaseous fluorine and chlorine pentafluoride with the selected metals. Based on the preliminary literature survey and review, an experimental program was defined which consisted of six types of tests necessary to obtain the following information:

(1) Maximum allowable flow velocity and resultant failure mode above that velocity;

(2) Maximum allowable temperature, pressure, and rate of adiabatic compression of propellant vapors;

(3) Maximum allowable velocity of impact of liquid propellant on a previously unwetted surface;

(4) Degradation of the passive film as a result of cyclic loading (for ClF_5 only);

(5) Effect of sonic and ultrasonic vibration on the passive film and the test material; and

II, B, Test Plan (cont.)

(6) Effect of a shock wave promulgated through the liquid (the water hammer effect).

The final experimental program was formulated on the basis of the comprehensive literature review discussed in the preceding section. The tests and rationale for the test plan are presented below.

1. Maximum Velocity Tests

The objective of the maximum velocity test is to determine the maximum allowable flow velocity of fluorine and chlorine pentafluoride that the selected metallic materials can withstand at elevated temperatures without detrimental degradation of the materials.

Based on the literature survey and review, there are considerable data available with regard to the flow of liquid fluorine at relatively high velocities, up to 400 ft/sec, in a variety of metals with no indication of failure. Few data are available for the failure of metals at temperatures greater than 1000°F with chlorine pentafluoride. The data indicated that failure occurred only at temperatures well beyond the critical temperatures of fluorine and chlorine pentafluoride. Therefore, testing was limited to gaseous fluorine and chlorine pentafluoride at temperatures ranging from ambient to 2300°F. In order to attain these conditions, a gas flow system was designed such that the velocity of the gas could be controlled through a test specimen orifice as both the gas itself and the test specimen were heated simultaneously. The heating was accomplished by electrical resistance heating of the gas flow tube which was fabricated from Nickel 200. This selection was based on the data existing in the literature.

The tests were to be conducted at three gas velocity values through the specimen orifice and at zero velocity. In this manner, the relative effects of velocity and temperature on the failure of the metals could be ascertained.

2. Adiabatic Compression Tests

The objective of the adiabatic compression tests is to determine the maximum allowable temperature and pressure which the various metallic materials can withstand in the presence of fluorine and chlorine pentafluoride under adiabatic compression conditions. The adiabatic compression test simulates events which may occur during valve operation.

Based on the literature survey and review, there were no data available concerning this type of test. The type of apparatus selected for testing is a slight modification of the adiabatic compression test apparatus used for evaluation of storable liquid propellants. The initial temperature of the fluorine and chlorine pentafluoride vapor were to be controlled and varied to attain different resultant temperatures and gas densities from the adiabatic compression process.

II, B, Test Plan (cont.)

3. Liquid Impact Tests

The objective of the liquid impact tests is to determine the maximum temperatures to which the selected metals may be heated without detrimental effects occurring when impacted by liquid streams of fluorine and chlorine pentafluoride. The test simulates a situation which may occur during restarts during a heat-soak condition.

The literature search and review documented that liquid fluorine impacting on metal plates at ambient temperatures did not cause significant changes in the plate surfaces. In addition, data were found for the impacting of gaseous chlorine pentafluoride on heated metal plates and showed that reaction occurs at temperatures of approximately 1100°F. However, no data were found concerning the impacting of liquid fluorine and liquid chlorine pentafluoride on heated metal plates. Consequently, testing was required in order to define the design criteria.

The apparatus selected for conducting tests consists of a temperature-conditioned liquid propellant feed system which delivers a liquid stream through an orifice to impact on an electrical-resistance heated test specimen. The test specimen temperature is measured by means of a thermocouple attached to the back of the test specimen. Two liquid velocities are used in order that the effect of liquid velocity can be evaluated as well as the effect of the temperature of the metal specimens on the failure point of the selected metals.

4. Flexing/Film Degradation Test

The objective of the flexing/film degradation tests is to determine the compatibility of fluorine and chlorine pentafluoride with the selected metals under conditions in which the metals are flexed within their respective elastic limits. The tests simulate the conditions that are encountered by flexing bellows.

Based on the literature search and review, adequate data were found to be available on the flexing of metals in the presence of liquid and gaseous fluorine, but no data were available concerning the flexing of metals in the presence of liquid and gaseous chlorine pentafluoride. Therefore, it was deemed necessary to conduct flexing experiments with the selected metals in chlorine pentafluoride. Because effects of flexing metals in fluorine were slight, only slight effects of flexing were anticipated for the chlorine pentafluoride. In order to differentiate between the inherent effect of flexing and the effect of the propellant during flexing, baseline tests are conducted with the flexing metals in trichlorotrifluoroethane which is assumed to be inert with respect to the selected metals. In addition, static tests with the metals immersed in chlorine pentafluoride are conducted for comparable exposure periods in order to define the inherent effect of the chlorine pentafluoride on the metals. Flexing for 10^5 cycles

II, B, Test Plan (cont.)

in chlorine pentalfluoride provides data which can be compared to data obtained with fluorine. The metal specimens are mounted in such a manner that exposure to the liquid and vapor phase occurs simultaneously.

5. Vibration/Film Degradation Tests

The objective of the vibration film degradation tests is to determine whether the "passivating film" on the selected metals undergoes appreciable degradation when subjected to vibration. The test simulates a situation such as in pumping in which cavitation occurs.

Sufficient data were available in the literature to indicate that in the sonic frequency range, vibration of metallic materials in liquid fluorine caused no adverse effects on the materials. However, metal erosion was detected with an aluminum casting vibrated at an ultrasonic frequency in liquid fluorine and liquid nitrogen. No data were available for the metals selected for testing in the current program in either liquid fluorine or liquid chlorine pentafluoride at ultrasonic frequencies. Therefore, such testing was warranted. In order to differentiate between the effect of the propellant on the metals and the effect of the vibration on the metals, three series of tests are conducted. The first are static tests with the metals in the propellants; the second are tests in which the metals are subjected to ultrasonic vibration in the propellants, and third are tests in which the metals are vibrated in inert liquids instead of the propellants. Liquid nitrogen is the inert substitute for liquid fluorine, and trichlorotrifluoroethane is the inert substitute for chlorine pentafluoride. The tests are conducted in an ultrasonic cleaning bath which is driven at a frequency of 62.5 k Hz.

6. Liquid Phase Shock Wave Tests

The objective of the liquid shock wave test is to determine the effects of a shock wave promulgated through liquid fluorine and chlorine pentafluoride on the selected metals. The test simulates the "water hammer effect" in the metallic materials produced during valve closure.

The literature data indicated that shock waves have been generated in liquid fluorine, but the data are difficult to relate to an operational feed system. No comparable data were found for chlorine pentafluoride. The testing is therefore warranted in both liquid fluorine and chlorine pentafluoride under well-defined conditions so that the adiabatic compression effect on the vapor phase can be discriminated from the liquid shock wave effect. In order to accomplish this, the tests are both conducted in the U-tube adiabatic compression apparatus. In one test series, only vapor is present for evaluating the adiabatic compression effect; in the other test series, sufficient liquid is used in the U-tube to assure that the shock wave is promulgated through the liquid phase. By comparison of the data on the test specimens, the effect due to the water hammer can be defined.

PHASE II - EXPERIMENTAL STUDIES

In order to obtain valid data concerning the inherent compatibility of the oxidizers with the selected metals, the metal samples were thoroughly cleaned and then passivated in the test apparatus prior to conducting the tests. The as-received materials were degreased with trichlorotrifluoroethane, then washed with an aqueous detergent solution, and thoroughly rinsed with deionized water. The metal samples were then immersed in a 28 percent nitric acid/2 percent hydrofluoric acid pickling solution until the surface condition was uniform. The stainless steel and Inconel 718 samples were immersed for a period of two hours; the copper, Monel aluminum, and nickel samples were immersed repeatedly for a few seconds because of the rapid reaction which occurs upon immersion. After pickling, the samples were thoroughly rinsed with deionized water, then rinsed with anhydrous methanol, and finally rinsed with a very pure grade of trichlorotrifluoroethane and then dried under vacuum. The metal samples were stored in a dry, inert atmosphere until required for testing.

The test apparatus were subjected to the same cleaning and pickling procedures as detailed above and then passivated with either gaseous fluorine or chlorine pentafluoride prior to testing. During the course of the entire investigation, the proper precautions were taken to maintain the apparatus and samples scrupulously clean in order to circumvent problems due to contamination.

The fluorine used in the investigation was supplied by the Air Force in conformance with Military Specification MIL-P-27405. The chlorine pentafluoride was supplied by the Air Force in conformance with Military Specification MIL-P-27413.

The data that are reported in connection with this investigation are applicable to systems which are properly cleaned and passivated. The presence of contaminants in any system utilizing fluorine or chlorine pentafluoride can lead to premature failure with regard to the design limits discussed in this report.

The experimental results from the investigation are discussed under six headings and in the following order: (A) Maximum Velocity Tests, (B) Adiabatic Compression Tests, (C) Liquid Phase Shock Wave Tests, (D) Liquid Impact Tests, (E) Flexing/Film Degradation Tests, and (F) Vibration/Film Degradation Tests.

A. MAXIMUM VELOCITY TESTS

The objective of the maximum velocity tests is to determine the maximum allowable flow velocity of fluorine and chlorine pentafluoride that metallic materials can withstand at elevated temperatures without detrimental

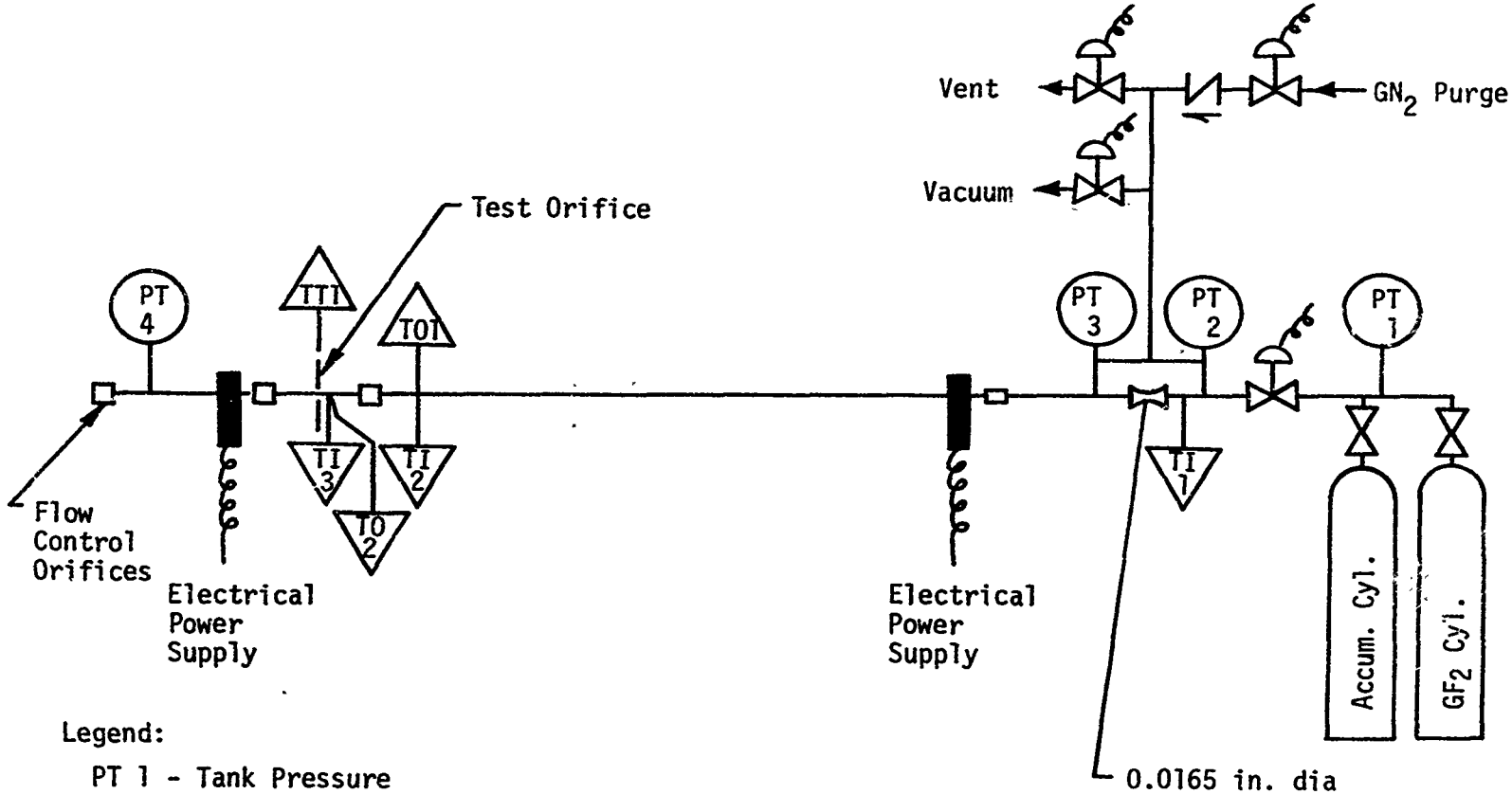
II, A, Maximum Velocity Tests (cont.)

degradation. The literature survey indicated that the metals were not susceptible to failure except at elevated temperatures such that liquid fluorine and liquid chlorine pentafluoride were precluded from consideration during the testing. Attainment of the failure points is necessary in order to define the design criteria and consequently the experimental investigation was conducted with gaseous fluorine and gaseous chlorine pentafluoride in an apparatus capable of functioning from ambient temperatures to 2500°F.

1. Apparatus and Procedures

A schematic of the apparatus in which the tests were conducted is shown in Figure 1 and a photograph of the apparatus is shown in Figure 2. The basic apparatus consists of a Nickel 200 tube (0.5 in.-diameter with 0.035-in. wall thickness) which was heated by direct electrical resistance heating. The tube is divided in three parts: A preheating section for temperature-conditioning the gas; the heated test section; and the downstream section with the flow-controlling orifices. The preheat section is five feet long; the test section is six inches long; and the downstream section is eighteen inches long. The test section was fitted with a disc of the metal of interest and it was welded in place at the midpoint of the test section. A 0.020-in. diameter hole was drilled through the disc to serve as the flow passage for the gas and the disc was approximately 0.25-in. thick. An isolated junction, chromel-alumel thermocouple sheathed in 0.020-in. diameter Inconel was inserted into a hole drilled into the side of each metal disc to within 0.010 in. of the flow passage. An assembled test section is shown in Figure 3. The copper and aluminum discs were not held in place by a weld in the test section. Rather, they were held in place with a modified Swagelock fitting at the midpoint of the test section as shown in Figure 4.

Prior to conducting the tests, the apparatus was checked out using nitrogen to insure satisfactory operation and calibration of the test apparatus. The procedure for testing under flow conditions was as follows. The test section containing the desired metal disc was placed in the apparatus and the system was purged with dry nitrogen followed by the gradual purging with either fluorine or chlorine pentafluoride. After repeated purging with halogen to remove the residual nitrogen, the heating of the apparatus was initiated. The test specimens were exposed to the passivating gas during the purging cycle for approximately five minutes. The procedure for testing under static condition was to evacuate the apparatus to 1 torr after insertion of the test section. Then the system was gradually pressurized with the desired oxidizer. After the initial charge of gas was in the system for a few minutes, the vent was opened and a fresh charge of gas was placed in the apparatus for the heating cycle. The heating rate used was approximately 10°F/sec.



Legend:

PT 1 - Tank Pressure
PT 2 - Pressure Upstream of Feed Orifice
PT 3 - Pressure Upstream Test Orifice
PT 4 - Pressure Downstream Test Orifice

TI 1 - Temperature of Gas Supply
TI 2 - Temperature of the Heated Gas
TO 1 - Temperature of Tube Surface
TI 3 - Temperature of Gas at Test Orifice Face
TO 2 - Temperature of Tube Surface at Test Orifice
TT 1 - Temperature of the Test Orifice

Figure 1. Schematic of Maximum Velocity Test Apparatus

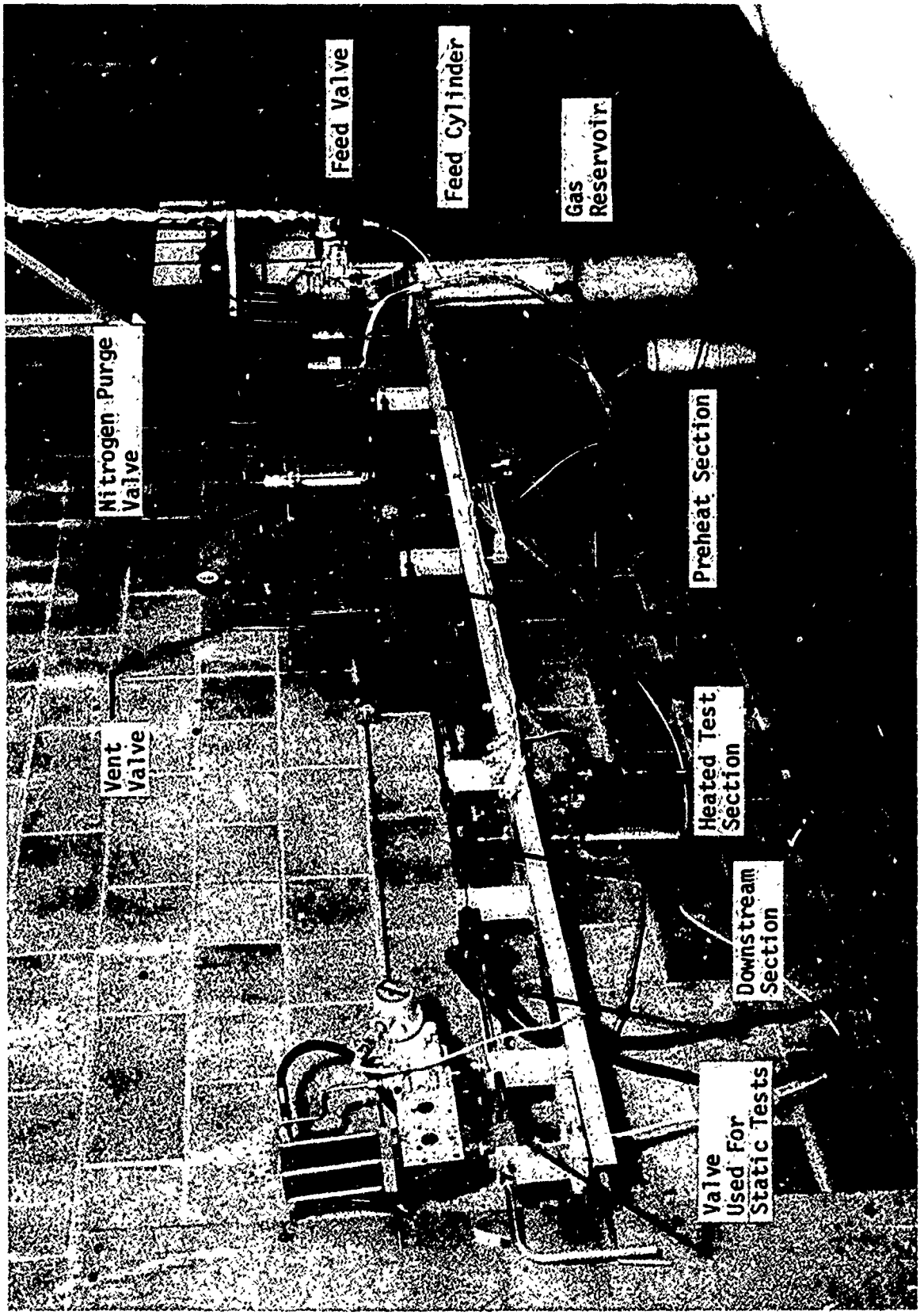


Figure 2. Maximum Velocity Test Apparatus

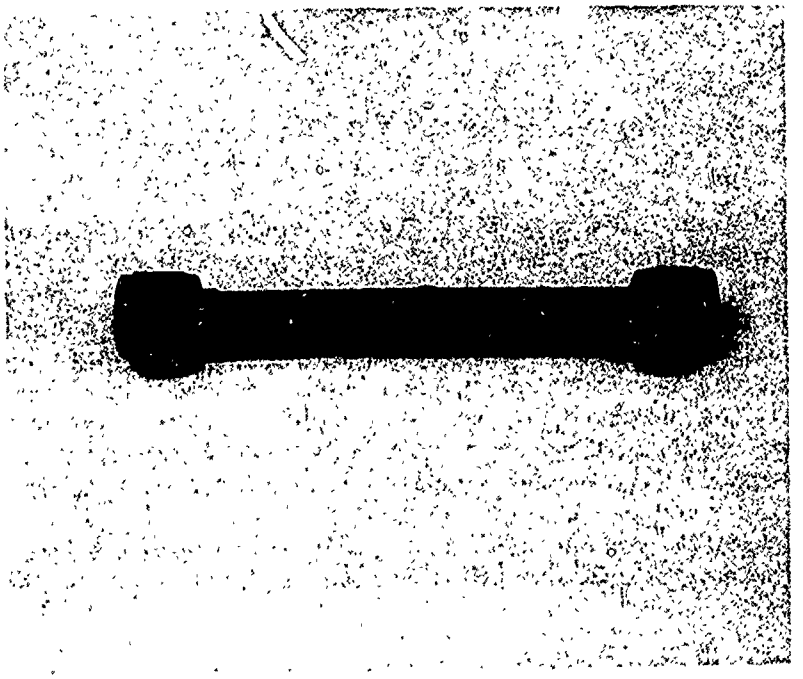


Figure 3. Photograph of an Assembled Test Section

Figure 4. Photograph of a Test Section used to Contain Copper and Aluminum Test Specimens

II, A, Maximum Velocity Tests (cont.)

The flow rate of the gas through the orifice of the test specimen was controlled by the use of orifices at the exit of the downstream section. For the static tests, the exit of the tube was capped; for the subsonic velocity tests two dishpan orifices (0.0135- and 0.0160-in. dia) were used; and for the sonic velocity tests the exit was left open. In case of burn out of the test section, the gaseous flow was limited by a 0.0165-in. dia orifice in the unheated portion of the feed system. The data were recorded with an analog-to-digital recorder with each instrumentation channel sampled 85 times per second (the system sampling rate is 3,750 channels per second) and the data were reduced using a Hewlett Packard Model 2100 electronic computer. A direct read-out system and television monitor were used during the tests as means to indicate heating rates and to indicate when the test specimen failed.

The pressure transducers used in the apparatus were accurate to $\pm$ 0.6 psi and were sensitive to 0.1 psi which is more than adequate for the testing. The thermocouples were prepared from special chromel and alumel wire and were accurate to $\pm$ 0.38% in the temperature range that was encountered. Thus, the temperature values reported are accurate to at least $\pm$ 10°F.

2. Experimental Results

The experimental results are discussed under three headings: (1) Gaseous Fluorine, (2) Gaseous Chlorine Pentafluoride, and (3) Comparison of the Behavior of Fluorine and Chlorine Pentafluoride with the Metals. The discussion follows in the preceding order.

a. Gaseous Fluorine

Flow tests with gaseous, preheated fluorine on heated specimens were conducted with nine metals. The fluorine gas pressure upstream of the test specimen was nominally 100 psia throughout the investigation. The metals investigated were:

304-L Stainless Steel	6061-T6 Aluminum
347 Stainless Steel	OFHC Copper
321 Stainless Steel	Inconel 718
A-286 Stainless Steel	Monel K-500
	Nickel 200

The copious computer reduced data obtained from the tests were analyzed using two basic graphical techniques. In addition, the specimens, when not totally consumed, were recovered from the test apparatus and submitted to visual examination to define final condition and ultimate failure mode.

II, A, Maximum Velocity Tests (cont.)

The first graphical technique of data analysis involved plotting specimen temperatures versus time. In this manner, changes in the rate of temperature rise were immediately made evident and exotherms and endotherms along with their relative magnitudes were brought out prominently. In general, the various specimens, regardless of flow regime (static, subsonic and sonic), exhibited one or more of the following types of thermal responses superimposed on a rising temperature/time profile: (1) small-scale exotherms or endotherms or small changes in slope such as shown in Figure 5 (304-L stainless steel specimen 2D) at temperatures below 1700°F, (2) moderate-scale exotherms and endotherms such as shown in Figure 7 for Nickel 200 specimens (10D, 10E, and 10F) in sonic and sub-sonic fluorine at temperatures below approximately 1900°F, and (3) large-scale exotherms such as shown in Figure 5 at approximately 1720°F and in Figure 7 at approximately 1900 and 2000°F for specimens 10E and 10D, respectively.

From this type of analysis alone, the threshold temperatures for incipient failure could be defined by the onset of the large exotherms (the third type of response mentioned above). The mechanism of failure was, however, evident only by examination of the "failed" specimen. A "failed" specimen of 304-L stainless steel is shown in Figure 6 and clearly shows the ultimate failure was brought about by burning. In contrast to this, examination of "failed" specimens of Nickel 200 showed every little change in the geometry of the flow passage but the specimen entrance and the passage itself to be coated with a yellowish-green nickel fluoride film several thousandths of an inch thick. Thus, post-test examination showed the "failure" mode for Nickel 200 was one involving significant reaction to form a substantial protective coating which prevented ignition and burning.

Threshold temperatures indicated by the small-scale and moderate-scale thermal responses (the first and second types of responses previously mentioned) were temporarily assumed to indicate possible potential failures, i.e., failures that might occur given sufficient time at that temperature and flow condition. In these cases, the validity of the assumption and the mechanism of that assumed potential failure could not be clarified by post-test examination of the specimens because they were almost always taken to "failure" by test plan and physical evidence of an earlier potential failure mode was destroyed. To clarify the validity of earlier potential failures from the data available, the second graphical technique of data analysis was employed.

That second technique involved plotting subsonic flow specimen pressure drops versus time and/or temperature (such plots are meaningless under static and sonic flow conditions because the pressure drops are zero and nearly constant, respectively). In this way, small effects on flow passage geometry could be ascertained in a first approximation (upstream

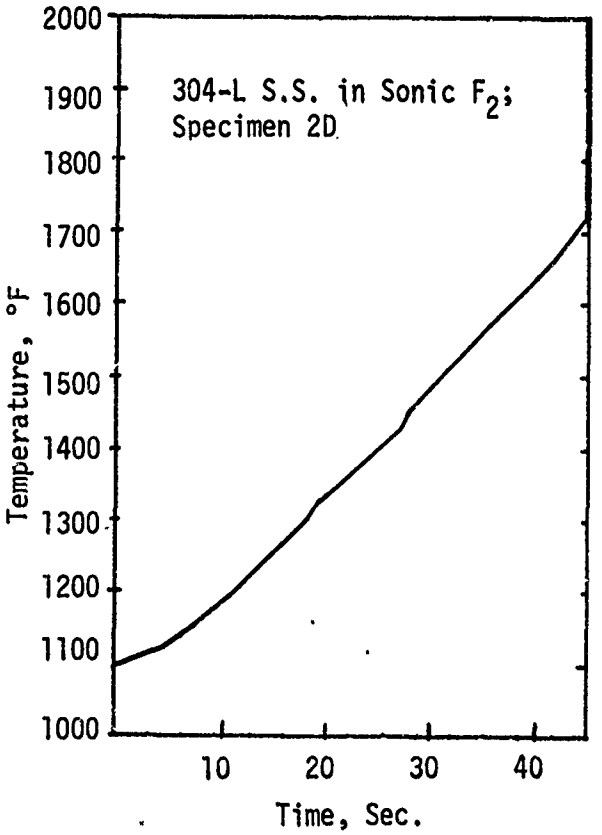


Figure 5. Exotherm Produced by the Reaction of Gaseous Fluorine with 304L Stainless Steel

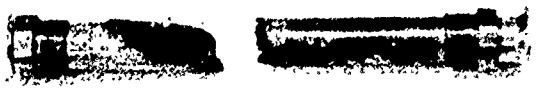


Figure 6. Test Section Remaining After Fluorine Consumed the 304L Stainless Steel Metal Specimen

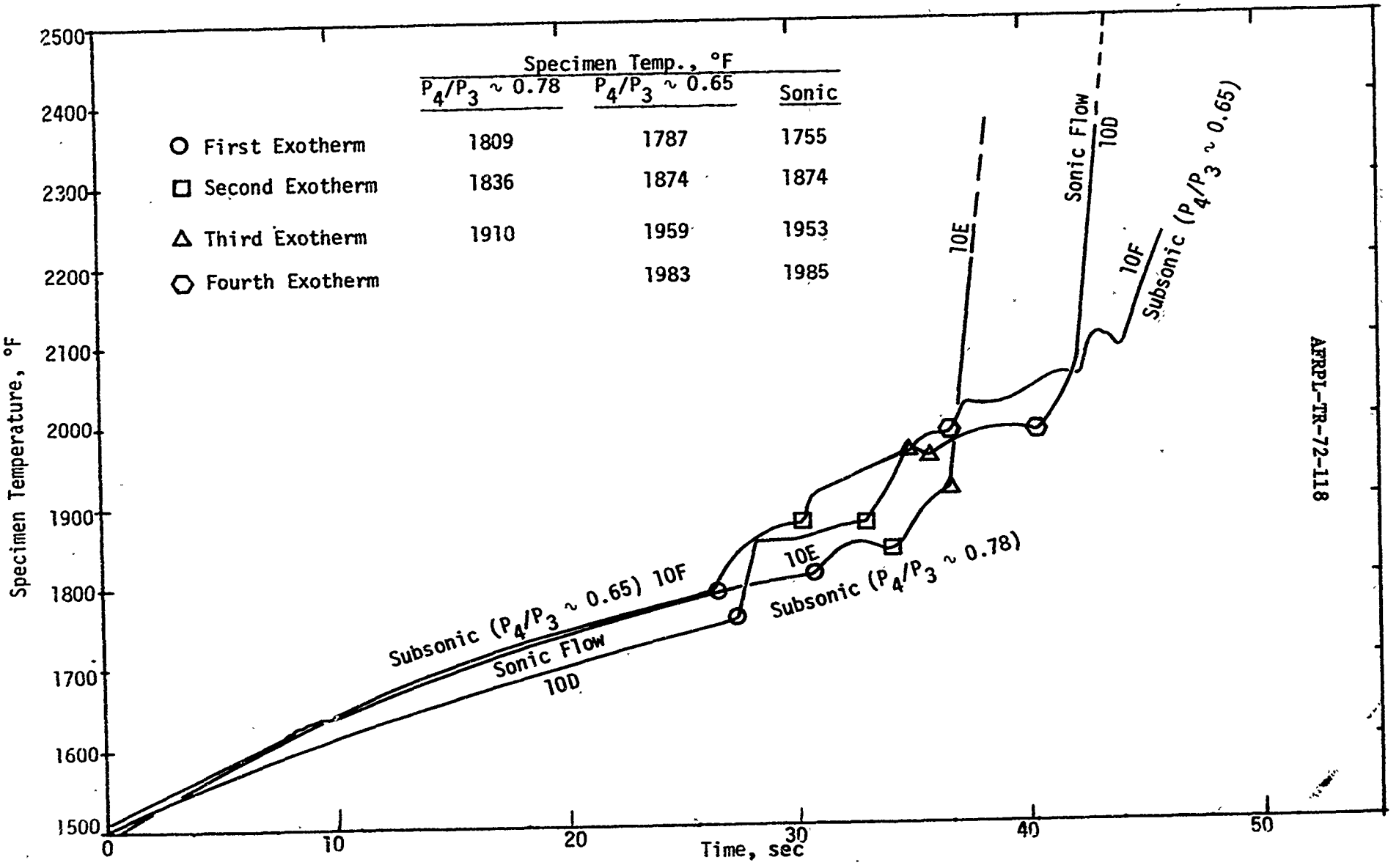


Figure 7. Exotherms Denoting Gradual Reaction of Fluorine with Nickel 200 at Sonic and Subsonic Flow Conditions

II, A, Maximum Velocity Tests (cont.)

pressure was nearly constant and the orifice coefficient is essentially constant until significant attack occurs). These analyses showed the conditions at which apparent flow passage restriction or opening was occurring and were marked by significant changes in the slope of the ΔP versus time or ΔP versus temperature curves. The onset of a rapidly increasing specimen ΔP was taken as an indication of chemical attack which resulted in "film" or corrosion product buildup while a decreasing ΔP (or ΔP slope) was taken as an indication of corrosion/erosion phenomenon or "film" degradation depending upon whether or not a preceding film buildup had occurred. Such changes in the specimens were regarded as potential failure modes, particularly when the conditions of onset were corroborated by the first data analysis technique. Almost invariably, the conditions at which a significant change in a specimen ΔP occurred were mirrored by a corresponding small-scale or moderate-scale thermal response as defined by the first data analysis technique. The correspondence was so good for the subsonic specimens that the minor thermal responses observed in the analysis of static and sonic specimens could generally be deduced to correspond to some particular geometric change and mechanism for the change. This was possible primarily because the threshold temperatures defined for the various specimens showed a relatively small dependence upon flow condition, although the physical change in a given specimen at a potential or incipient failure threshold temperature could be quite different in magnitude. That is to say, any indication of a potential failure threshold temperature for a subsonic flow condition would normally correspond to less and more pronounced indications of potential failures at about the same threshold temperature for static and sonic flow conditions, respectively.

In most tests which were initially flowing in a subsonic regime, film buildup during the course of a test was sufficient to cause the flow passage to become sonic prior to a major exotherm or change in geometry. Thus, incipient failure thresholds could not normally be ascertained at subsonic flow conditions.

The results of these data analyses and specimen examinations are summarized in Table I wherein threshold temperatures and the corresponding material responses are defined for the various materials and flow regimes. Although some of the materials tested under some of the flow conditions exhibited very little or very gradual geometric and/or thermal responses as temperature increased up to the point a very dramatic change occurred, the most common behavior was for the materials to undergo one or more "potential failure" responses prior to an "incipient failure" response. This latter common behavior makes the definition of a single threshold set of dynamic conditions for failure difficult because a particular "potential failure" material response in terms of these tests could be accentuated or damped in a related but different flow system. For example, the onset of rapid film

TABLE I

RESPONSE OF METALS TO GASEOUS FLUORINE AT VARIOUS TEMPERATURES AND FLOW STATES

Material	Static		Subsonic Flow		Sonic Flow	
	Threshold Temp., °F	Material Response	Threshold Temp., °F	Material Response	Threshold Temp., °F	Material Response
6061-T6 Al						
OFHC Cu	1090*	Probable onset of film buildup.	1000 σ = 40*	Onset of film buildup.	~1000	Probable onset of film buildup.
	1230*	Apparent exothermic reaction (slight).	1190 σ = 25*	Significant change in film buildup/thermal effects.	1150	Major exotherm and loss of specimen.
	1400*	Apparent endothermal reaction (slight).	1380 σ = 25*	Moderate exotherms and film changes.	855	Onset of a series of small exotherms and endotherms.
	1585*	Significant exotherm, modest specimen attack.	1525 ± 58*	Major exotherm and loss of specimen material.	1150	Significant exotherm.
321 S.S.	1145	Probable onset of rapid film buildup.			1250	Relative large exotherm.
	1890	Onset of a series of exotherms reaching a max. temp. of 2475°F, significant material loss.			1485	Major exotherm reaching a max. temp. of ~2100°F, specimen suffers modest attack.
304-L S.S.	1285	Probable onset of rapid film buildup.			1115	Probable onset of rapid film buildup.
	1930	Major exotherm with significant loss of specimen material.			1700	Major exotherm with significant loss of specimen material.
347 S.S.					1140	Probable onset of rapid film buildup.
	1833	Major exotherm with significant loss of specimen material.	1335	Onset of rapid film buildup.	1722	Major exotherm with significant loss of specimen material.
A-286 S.S.	~1400	Probable onset of rapid film buildup.			1315	Probable onset of rapid film buildup.
	1900	Onset of a series of exotherms reaching a max. temp. of 2320°F, significant material loss.	1350	Onset of rapid film buildup.	1723 σ=2	Major exotherm with significant loss of specimen material.
Inconel 718					1350	Probable onset of rapid film buildup.
			~1675	Onset of very rapid film buildup.	1704	Major exotherm with significant loss of specimen material.
Monel K-500	1695	Significant exotherm.			1675	Onset of a series of exotherms reaching a max. temp. of 2480°F, flow passage corroded closed.
	1905	Major exotherm reaching max. temp. of ~2400°F, flow passage corroded closed.	1575	Onset of very rapid film buildup.	1520	Probable onset of rapid film buildup.
Nickel 200					1715	Major exotherm reaching max. temp. of 2300°F, flow passage corroded and restricted.
			1785	Onset of a series of exotherms with max. temp. ≥ 2340°F, no significant change in flow passage.	1755	Onset of a series of exotherms with max. temp. >2360°F, no significant change in flow passage.
			≤2300**	No evidence of melting or ignition.		

* Temperature based on outside wall temperature.
**Information based on Reference 2.

II, A, Maximum Velocity Tests (cont.)

buildup is considered a "potential failure" mode in terms of these tests but a similar response in a close tolerance component could become an "incipient failure" or in a much larger flow system, an inconsequential response. Inspite of this limitation, the data do provide a set of "potential failure" and "incipient failure" threshold temperatures and velocity conditions for a variety of metals subjected to gaseous fluorine in a variety of flow conditions and which are applicable to all but the most critical and forgiving flow systems.

On the basis of the test results presented in Table I, the potential and incipient failure threshold temperatures and failure modes for the various flow conditions, i.e., static, subsonic, and sonic velocity, are presented in Table II and recommended for the guidance of designers. In the use of Table II, it must be recognized that the incipient failure thresholds should never be used for design purposes without specific independent test verification. Additionally, an analysis of test to test variations in repetitive samples shows standard deviations as high as 40°F, suggesting that the potential failure thresholds should be reduced approximately 3σ or 120°F when considering limiting design capabilities.

In a related series of fluorine flow tests reported by Duckering (Reference 1), a 0.050-in. wall Nickel 200 tube was electrically heated to selected temperatures and then exposed to gaseous fluorine at $\sim$260 psig and a velocity of $\sim$30 ft/sec for 1-sec periods of time. The tests were conducted at specimen temperatures of 1000 to 2000°F at 200°F increments and from 2000 to 2300°F at 100°F increments. There was no evidence of tube melting or ignition in any of the tests. A sonic orifice located downstream of the tube specimen and exposed to gaseous fluorine at $\sim$140 psia, 200-250°F, and $\sim$1000 ft/sec was similarly unaffected.

Goodwin and Lorenzo (Reference 2) determined the ignition temperatures of several metals in essentially static gaseous fluorine using two experimental techniques both of which involved electrical heating of metal wire specimens. Temperatures were calculated from current, voltage, and temperature-resistivity data. In "Technique A" an evacuated bomb was filled with gaseous fluorine at atmospheric pressure and the temperature of the specimen was gradually increased until burnout was achieved. In "Technique B" the test specimen was brought to a predetermined temperature in the evacuated bomb before the introduction of fluorine. Fluorine was admitted and the time for the reaction to go to completion was measured. The data obtained from these tests are summarized in Table III. The ignition temperatures defined by the two techniques are in reasonable agreement; however, both techniques give ignition temperatures of questionable accuracy because the effect of preignition reactions on the current, voltage, temperature-resistivity data use in calculating temperatures are not taken into account. The preignition reaction effect appears less significant in "Technique B" and, therefore, probably yields the more reliable data.

TABLE II

POTENTIAL AND INCIPIENT FAILURE THRESHOLD TEMPERATURES AND MODES OF FAILURE FOR METALS IN GASEOUS FLUORINE

POTENTIAL FAILURES

Material	Static Fluorine		Subsonic Fluorine		Sonic Fluorine	
	Threshold Temp., °F	Mode of Failure	Threshold Temp., °F	Mode of Failure	Threshold Temp., °F	Mode of Failure
6061-T6 Aluminum					1000	Corrosion product buildup
OFHC Copper	∿1090	Corrosion product buildup	∿1000	Corrosion product buildup	855	Corrosion/erosion
321 Stainless Steel	1145	Corrosion product buildup			1115	Corrosion product buildup
304-L Stainless Steel	1285	Corrosion product buildup			1140	Corrosion product buildup
347 Stainless Steel			1335	Corrosion product buildup	1315	Corrosion product buildup
A-286 Stainless Steel	∿1400	Corrosion product buildup	1350	Corrosion product buildup	1350	Corrosion product buildup
Inconel 718			∿1675	Corrosion product buildup	∿1675	Corrosion product buildup
Monel K-500	∿1695	Corrosion product buildup	1575	Corrosion product buildup	1520	Corrosion product buildup
Nickel 200			∿1785	Protective film buildup	∿1755	Protective film buildup

INCIPIENT FAILURES

6061-T6 Aluminum					1150	Melting and/or burning
OFHC Copper	1585	Corrosive burning	1525	Corrosive burning	1485	Corrosive burning
321 Stainless Steel	1890	Corrosive burning			1700	Corrosive burning
304-L Stainless Steel	1930	Corrosive burning			1720	Corrosive burning
347 Stainless Steel	1830	Corrosive burning			1720	Corrosive burning
A-286 Stainless Steel	1900	Corrosive burning			1700	Corrosive burning
Inconel 718					>1675	Gross corrosion (no burning)
Monel K-500	1905	Gross corrosion (no burning)			1715	Gross corrosion (no burning)
Nickel 200			>2300	Possible corrosive burning	>2300	Possible corrosive burning

TABLE III

IGNITION TEMPERATURES OF METALS IN STATIC GASEOUS FLUORINE

Technique A

Material	Average Ignition Temp., °F	Observed Range of Ignition Temp., °F	Standard Deviation, °F
Molybdenum	398	370 - 428	22
Tungsten	540	500 - 630	45
Monel	745	658 - 819	61
Iron	1241	1233 - 1251	9
302 Stainless Steel	1259	1058 - 1465	164
Copper	1277	1193 - 1377	64
Nickel	2123	1983 - 2226	86
Aluminum	>M.P.		

Technique B

Iron	1144 - 1191
Copper	1272 - 1294
Nickel	2287 - 2311

II, A, Maximum Velocity Tests (cont.)

Inspection of the data presented in Tables I, II and III for aluminum, shows: (1) the earliest signs of attack have been observed to occur near 1000°F under sonic flow conditions, (2) a major exotherm occurs at ∿1150°F with sonic fluorine and results in a melting- and/or burnout-type failure, and (3) ignition in static fluorine occurs at or above the melting point of aluminum. A similar inspection of the data on copper shows: (1) the earliest signs of copper attack occur in the temperature range of ∿850 to 1100°F, varying within this range more or less inversely with flow velocity, (2) a number of more significant velocity dependent exotherms, endotherms, and film changes are observed in the temperature range of ∿1150 to 1400°F which may result in ignition under certain poorly defined conditions that are not strongly dependent on velocity, and (3) a major reaction is almost certain to occur when the temperature reaches 1485 to 1585°F. The combined data from this investigation and Reference 2 on austenitic stainless steels indicate: (1) significant attack occurs within the temperature range of ∿1100 to 1400°F, varying within this range somewhat inversely with velocity and the particular stainless steel, (2) ignition may occur within this same temperature range under certain poorly defined conditions apparently not related strongly to flow velocity, and (3) a major reaction involving a large heat release and material loss is almost certain to occur upon reaching a temperature of ∿1700°F under sonic conditions and 1830 to 1930°F under static conditions.

The data on Inconel 718 are difficult to interpret but indicate an important threshold temperature near 1675°F that is quite velocity independent. Gradual film buildup appears to be the significant reaction at temperatures up to this threshold at which point the reaction rate increases rapidly. In some cases this condition appears to cause the film to immediately fail and reform resulting in significant exotherms and formation of corrosion products while in other cases, the film is not disrupted until temperatures of up to 1835°F are reached. Although appreciable quantities of corrosion products and pulse temperatures of over 2400°F have been observed following film failures on Inconel 718, true ignition with the loss of substantial base metal has not been observed.

Monel K-500 undergoes a rapid increase in the rate of film buildup in the temperature range of ∿1500 to 1600°F, apparently varying somewhat inversely with velocity. At a temperature near 1700°F the film may fail in either static or sonic fluorine and result in an exotherm as it reforms. This film may remain intact, however, up to temperatures as high as 1850°F and ∿1900°F in sonic and static fluorine, respectively. Although pulse temperatures approaching 2400°F were recorded following film failures, an appreciable loss of base material was observed in only one case, and true ignition was never observed. An ignition temperature of ∿745°F for Monel as reported in Reference 2, cannot be reconciled with the data obtained in the current investigation.

II, A, Maximum Velocity Tests (cont.)

Inspection of the data on nickel shows the earliest signs of reaction with fluorine occur at ∿1750 to 1800°F and are apparently only slightly influenced by velocity. This onset of reaction appears normally as a series of mildly exothermic film-forming reactions (see Figure 7). As temperature increases beyond ∿1900°F, the exotherms may become quite large, increasing the specimen film temperature several hundred degrees (°F) in less than a second. Peak temperatures of greater than 2360°F were recorded while the specimens underwent no significant damage other than the formation of a film. This agrees well with the data of Reference 1 wherein temperatures up to 2300°F in low velocity fluorine caused no evidence of melting or ignition. In Reference 2, the "ignition temperature" of nickel in static fluorine is indicated to be in the range of ∿1980 to 2310°F; however, the indirect method of defining temperature decreases the reliability of the data.

From all the data available, it must be concluded that nickel is the best metal known for containing hot, flowing, gaseous fluorine. In descending order of preference the other metals studied are ranked as follows: Inconel 718 and Monel K-500, austenitic stainless steels, copper, and aluminum.

b. Gaseous Chlorine Pentafluoride

Flow tests were conducted with gaseous, preheated chlorine pentafluoride on heated specimens of ten metals. The following metals were investigated:

304-L Stainless Steel	6061-T6 Aluminum
301 Cryo Stainless Steel	OFHC Copper
347 Stainless Steel	Inconel 718
321 Stainless Steel	Monel K-500
A-286 Stainless Steel	Nickel 200

The chlorine pentafluoride gas pressure upstream of the metal test specimen was nominally 65 psia throughout the test series. The test data were obtained, reduced, and analyzed as previously described for the fluorine flow tests. A photograph of a test section originally containing a A-286 metal specimen is shown in Figure 8. The heat generated by the reaction of chlorine pentafluoride with the A-286 was sufficient to cause a rupture of the Nickel 200 tube. A photograph of an Inconel 718 test specimen after reaction with chlorine pentafluoride is shown in Figure 9. An original test specimen is also shown in the figure for comparison purposes.

The results of these data analyses and specimen examinations are summarized in Table IV wherein threshold temperatures and the corresponding material responses are defined for the various materials and flow

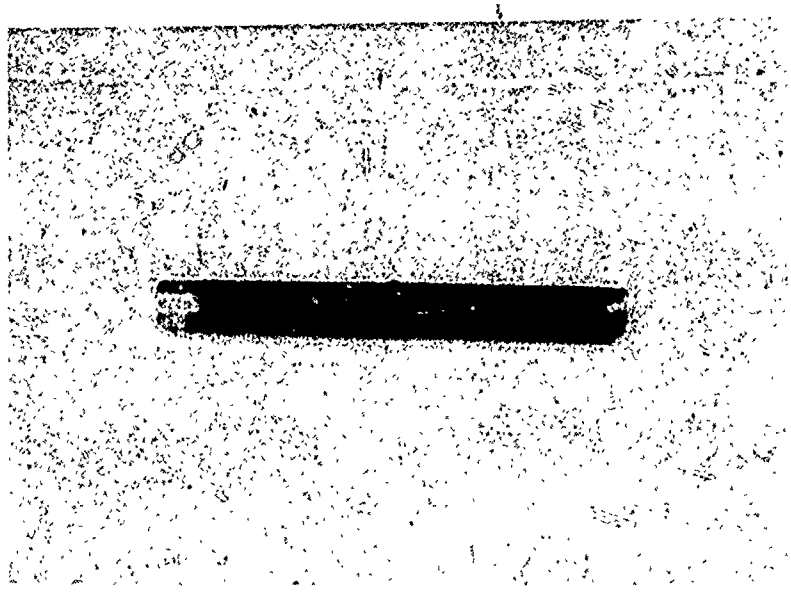


Figure 8. Test Section Remaining After Chlorine Pentafluoride Reacted with A-286 Metal Specimen

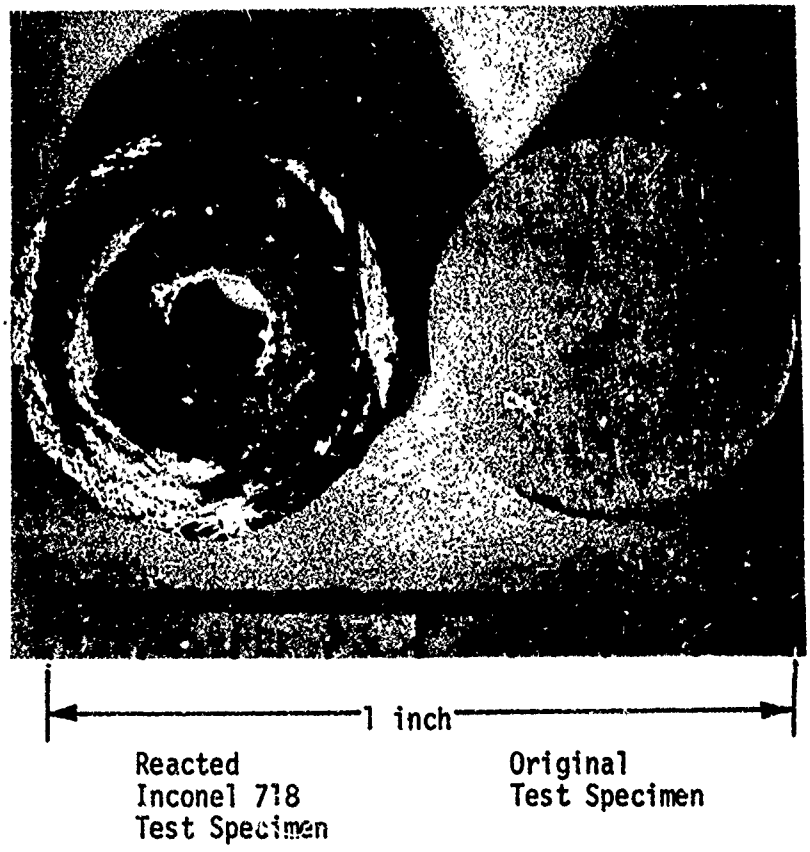


Figure 9. Inconel 718 Test Specimen After Reaction with Chlorine Pentafluoride and an Original Test Specimen

TABLE IV

RESPONSE OF METALS TO GASEOUS CHLORINE PENTAFLUORIDE AT VARIOUS TEMPERATURES AND FLOW STATES

Material	Static or Very Low Velocity Flow		Subsonic Flow		Sonic Flow	
	Threshold Temp., °F	Material Response	Threshold Temp., °F	Material Response	Threshold Temp., °F	Material Response
6061-T6 Aluminum					1155	Major exotherm and loss of specimen
OFHC Copper			865	Onset of rapid film build-up	~840	Probable onset of film build-up
			1090	Increased film build-up rate	~1000-1060	Film changes
			1250	Film disruption	1230	Additional film changes
			1430	Major film breakdown and exotherm	1360	Major exotherm and loss of specimen
			1450*	Onset of significant exotherms		
304-L S.S.	1110	Probable onset of rapid film formation			1140	Probable onset of rapid film formation
	1625	Onset of a series of minor exotherms and endotherms			1215	Onset of specimen material loss
	1930	Large exotherm to max temp of 2275°F, slight specimen attack			1560	Major exotherm and loss of specimen
301 Cryo S.S.	1133	Probable onset of rapid film formation	1125	Onset of rapid film formation	1210	Probable onset of rapid film build-up
	1590	Minor exotherm			1560	Minor exotherm, very rapid film build-up
	1675	Minor exotherm			1602 ± 7	Major exotherm and loss of specimen
	1715	Large exotherm to max temp of 2170°F, specimen corroded to blindoff opening.				
347 S.S.	1260	Onset of rapid film build-up	1206 σ = 6	Onset of rapid film build-up	~1200	Probable onset of rapid film build-up
	~1500	Minor endotherm and exotherm			1525 σ = 5	Very rapid film build-up
	~1655	Large exotherm to max temp of 2300°F, slight specimen attack			1632 σ = 20	Major exotherm with significant to major loss of specimen material
321 S.S.			1170 ± 27	Onset of rapid film build-up	~1210	Probable onset of rapid film build-up
			1605	Very rapid film build-up	1610	Minor exotherm, very rapid film build-up
					1660 σ = 20	Major exotherm and loss of specimen material
A-286 S.S.	1675	Onset of very rapid film build-up	1370 σ = 32	Onset of rapid film build-up	~1250	Probable onset of rapid film build-up
	1775	Large exotherm to max temp of 2250°F, slight specimen attack			1656 σ = 24	Very rapid film build-up
					1692 σ = 31	Major exotherm and loss of specimen material
Inconel 718	1620	Probable onset of rapid film build-up	1605	Onset of rapid film build-up	1555	Onset of rapid film build-up
	1790	Minor exotherm			1787 σ = 3	Major exotherm with significant to major loss of specimen material
	1890	Large exotherm to max temp of 2340°F, significant specimen loss				
Monel K-500			1800	Onset of rapid film build-up	1800	Onset of rapid film build-up
			1865	Major exotherm to max temp of 2370°F, build-up of corrosion products in specimen	1875	Major exotherm to max temp of 2030°F, build-up of corrosion products in specimen
Nickel 200			1860	Onset of a series of large exotherms to max temp of 2280°F, specimen covered by film, but minimal change in geometry	1788 ± 3	Onset of a series of large exotherms to max temp of 2370°F, specimen covered by film, but minimal change in geometry

*Temperature based on outside wall temperature.

II, A, Maximum Velocity Tests (cont.)

regimes. Although some of the materials tested under some of the flow conditions exhibited very little or very gradual geometric and/or thermal responses as temperature increased up to the point a very dramatic change occurred, the most common behavior was for the materials to undergo one or more "potential failure" responses prior to an "incipient failure" response. This latter common behavior makes the definition of a single threshold set of dynamic conditions for failure difficult because a particular "potential failure" material response in terms of these tests could be accentuated or damped in a related but different flow system as discussed previously for fluorine. In spite of this limitation, the data do provide a set of "potential failure" and "incipient failure" threshold temperature and velocity conditions for a variety of metals subjected to gaseous chlorine pentafluoride in a variety of flow conditions and which are applicable to all but the most critical and forgiving flow systems.

On the basis of the test results presented in Table IV, the potential and incipient failure threshold temperatures and failure modes for the various flow conditions, i.e., static, subsonic velocity, and sonic velocity, are presented in Table V and recommended for the guidance of designers. In the use of Table V, it must be recognized that the incipient failure thresholds should never be used for design purposes without specific independent test verification. Additionally, an analysis of test to test variations in repetitive samples shows standard deviations as high as 32°F, suggesting that the potential failure thresholds should be reduced approximately 3σ or 100°F when considering limiting design capabilities.

Johnson and Lawrence (Reference 3) have defined the ignition temperatures of passivated copper and 304 stainless steel in flowing gaseous chlorine pentafluoride in the course of determining the optimum passivation temperatures for preparing copper and 304 stainless steel for chlorine pentafluoride service. The apparatus used consisted of an electrically heated tube furnace containing a Vycor tube. The test section was a 27-in. long section of 3/8-in. OD tubing and passed through the Vycor tube. A thermocouple was welded to the outside wall in the center of the test section. In operation, the test section was heated to the desired passivation temperature ($\sim$20°C, 100°C, or 200°C) and gaseous chlorine pentafluoride at 20 psig was flowed through the section for 20 min. After passivation, the chlorine pentafluoride was purged from the test section with nitrogen. Power to the furnace was increased so that the temperature of the test section increased at 6 to 12°C/min. When the temperature reached 600°C, the flow of gaseous chlorine pentafluoride (velocity undefined) was resumed with the pressure being maintained as before at 20 psig. The ignition temperature was taken as the point at which burn-through was audibly apparent (and where a time-temperature plot dramatically changed slope).

TABLE V

POTENTIAL AND INCIPIENT FAILURE THRESHOLD TEMPERATURES AND FAILURE MODES FOR METALS IN GASEOUS CHLORINE PENTAFLUORIDE

POTENTIAL FAILURES

Material	Static ClF_5		Subsonic ClF_5		Sonic ClF_5	
	Threshold Temp., °F	Mode of Failure	Threshold Temp., °F	Mode of Failure	Threshold Temp., °F	Mode of Failure
OFHC Copper			865	Corrosion product buildup.	~840	Corrosion product buildup.
304-L Stainless Steel	1110	Corrosion product buildup.			1140	Corrosion product buildup.
301 Cryo. S.S.	1130	Corrosion product buildup.	1125	Corrosion product buildup.	1125	Corrosion product buildup.
347 Stainless Steel	1260	Corrosion product buildup.	1205	Corrosion product buildup.	~1200	Corrosion product buildup.
321 Stainless Steel			1170	Corrosion product buildup.	~1210	Corrosion product buildup.
A-286 Stainless Steel			1370	Corrosion product buildup.	~1250	Corrosion product buildup.
Inconel 718	1620	Corrosion product buildup.	1605	Corrosion product buildup.	1555	Corrosion product buildup.
Monel K-500			1800	Corrosion product buildup.	1800	Corrosion product buildup.
Nickel 200			1860	Protective film buildup.	1790	Protective film buildup.

INCIPIENT FAILURES

6061-T6 Aluminum					1155	Melting and burning.
OFHC Copper			1450	Corrosive burning.	1360	Corrosive burning.
304-L Stainless Steel	1930	Corrosive burning.			1560	Corrosive burning.
301 Cryo. S.S.	1715	Gross corrosion buildup.			1600	Corrosive burning.
347 Stainless Steel	~1655	Corrosive burning.			1630	Corrosive burning.
321 Stainless Steel		Corrosive burning			1660	Corrosive burning.
A-286 Stainless Steel	1775	Corrosive burning.			1690	Corrosive burning.
Inconel 718	1890	Corrosive burning.			1785	Corrosive burning
Monel K-500			1865	Gross corrosion buildup.	1875	Gross corrosion buildup.
Nickel 200			≳2300	Possible corrosive burning.	≳2300	Possible corrosive burning.

II, A, Maximum Velocity Tests (cont.)

Duplicate tests using copper tubing were completed at each passivation temperature and with 304 stainless steel using a 20°C passivation. The results are summarized in Table VI.

TABLE VI

IGNITION TEMPERATURES OF COPPER AND 304 STAINLESS STEEL IN FLOWING CHLORINE PENTAFLUORIDE

Test Material	Passivation Temp., °F	Ignition Temp., °F*
Copper (3/8-in. OD Tubing	68	1445
	212	1598
	392	1463
304 SS (3/8-in. OD Tubing)	68	1218

*Average of duplicate tests

Comparing Table VI with Table IV, it can be seen that two of the three ignition temperatures given for copper in Table VI correspond very closely to the threshold temperature for the onset of significant exotherms for subsonic flow given for copper in Table IV. Similarly, the ignition temperature for 304 stainless steel given in Table VI compares very favorably with the threshold temperature for the onset of significant specimen loss for sonic flow given for 304-L stainless steel in Table IV. Although the material responses at the given threshold temperature are not the same in the two different types of flow tests, it does appear that both types of flow tests define temperatures at which very important changes in material response occur. The fact that the material responses are not the same and are not easily correlated considering only flow velocity or Reynolds Number shows that more subtle flow system characteristics which affect heat and mass transfer rates play an important role in determining the particular way a material will respond upon reaching a given threshold temperature. This further points up the need to design flow systems based on the threshold temperatures at which the earliest significant changes in material response can be ascertained.

c. Comparison of the Behavior of Fluorine and Chlorine Pentafluoride with the Metals

For the sake of comparing the relative reactivity of chlorine pentafluoride and fluorine with the metals considered in this investigation, data obtained under comparable conditions are compiled in Table VII. The data are taken from Tables II and V from the columns listed under "Potential Failures".

II, A, Maximum Velocity Tests (cont.)

TABLE VII

THRESHOLD TEMPERATURES AT WHICH GASEOUS FLUORINE AND GASEOUS CHLORINE PENTAFLUORIDE INITIATE SIGNIFICANT ATTACK ON SELECTED METALS

	Static Condition		Subsonic Flow		Sonic Flow Conditions	
	F_2	ClF_5	F_2	ClF_5	F_2	ClF_5
OFHC Copper	∿1090		∿1000	865	855	∿840
321 Stainless Steel	1145			1170	1115	∿1210
304-L Stainless Steel	1285	1110			1140	1140
347 Stainless Steel		1260	1335	1205	1315	1200
A-286 Stainless Steel	∿1400		1350	1370	1350	∿1250
Inconel 718		1620	∿1675	1605	∿1675	1555
Monel K-500	∿1695		1575	1800	1520	1800
Nickel 200			∿1785	1860	∿1755	1790

These data clearly show that velocity has a relatively small influence on the potential failure threshold temperature of any of the metals studied in either fluorine or chlorine pentafluoride. Generally, OFHC copper, the stainless steels, and Inconel 718 have slightly lower threshold temperatures in the presence of chlorine pentafluoride than in fluorine under comparable flow conditions. The threshold temperatures of Monel K-500 and Nickel 200 are lower in the presence of fluorine than in chlorine pentafluoride.

II, Experimental Results and Discussion (cont.)

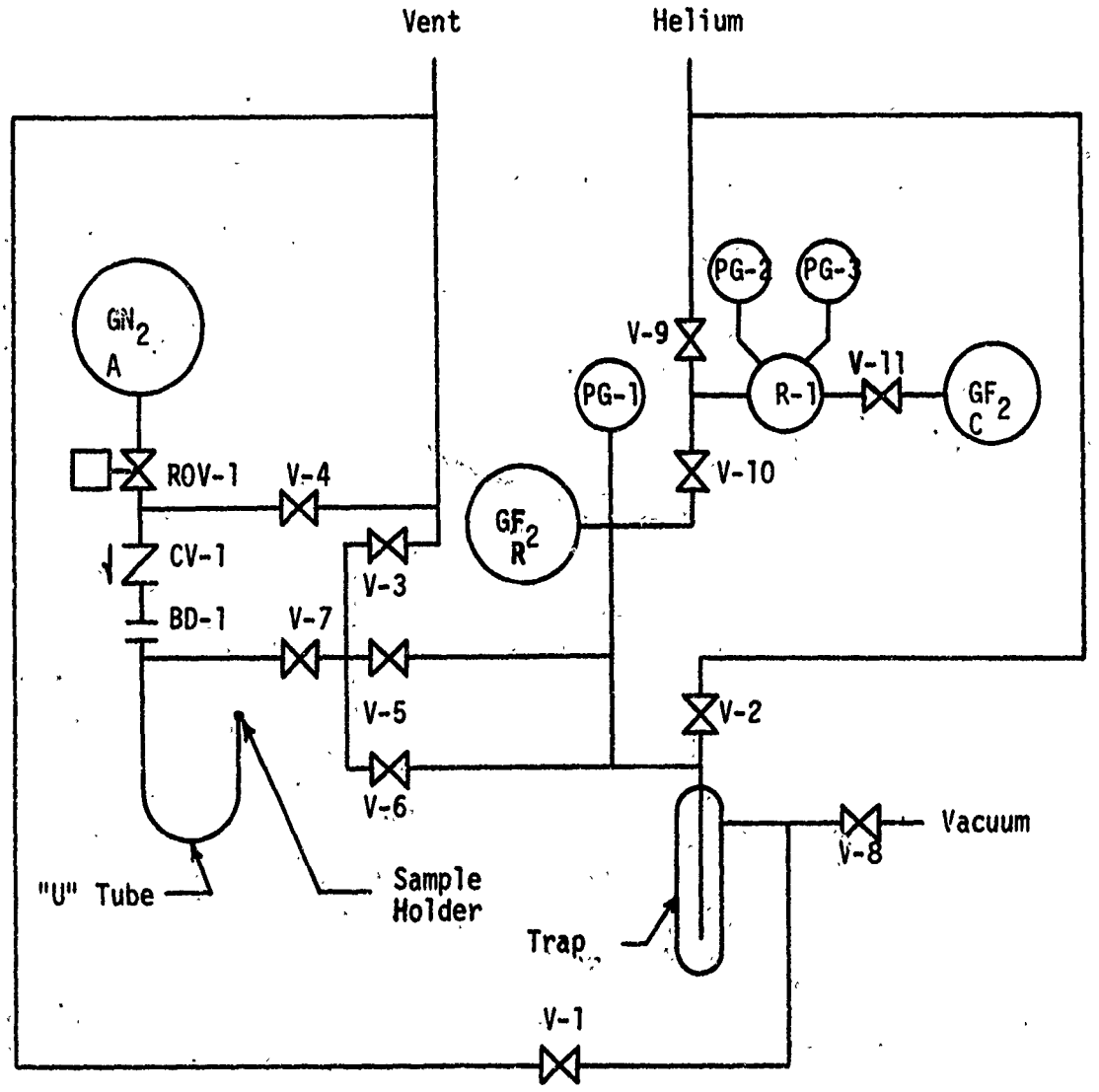
B. ADIABATIC COMPRESSION TESTS

The objective of the adiabatic compression tests was to determine the maximum allowable temperatures and pressures of gaseous fluorine and chlorine pentafluoride which can be tolerated by the metals selected for this investigation.

1. Apparatus and Procedures

The apparatus used in the investigation was a U-tube adiabatic compression test apparatus which was modified to accommodate the introduction of gaseous halogen propellants and metal specimens and to incorporate a means of temperature conditioning the loaded U-tube. A schematic diagram of the entire apparatus for handling the halogens and conducting the test is shown in Figure 10. The schematic diagram of the U-tube adiabatic compression apparatus is shown in Figure 11; a photograph of the apparatus is shown in Figure 12; and the schematic of the test specimen holder with the test specimen in place is shown in Figure 13. The test specimen holder was a 1/4-in. solid AN plug used to seal the end of the U-tube. The test specimen was a strip of metal sheet 0.010-in. thick by 0.10-in. wide by 0.10-in. long which was spot welded to the end of the AN plug. The U-tube was fabricated from Inconcel X-750 1/4-in. tubing approximately 16-in. long.

The tests were conducted in the following manner. The U-tube was attached to the apparatus and the specimen holder used to seal the open-end of the U-tube. The tube was then evacuated to 1 torr or less, temperature conditioned, and then 15.7 psia of gaseous fluorine or chlorine pentafluoride was gradually introduced into the assembly. The fluorine or chlorine pentafluoride was allowed to remain in the U-tube for five minutes prior to the test to allow for initial passivation of the metal. The pneumatic line valves were then actuated and the nitrogen from the accumulator tank was used to compress the gaseous fluorine or chlorine pentafluoride. The U-tube assembly was then vented and flushed with nitrogen and the test specimen was examined visually to ascertain if any attack occurred. Microscopic examination was used to evaluate the samples which were not totally consumed in the test. A 1000 lb burst disc made of 304-L stainless was used in each test to seal the pneumatic valve and check valve assembly from the halogen atmosphere prior to the test. The driving pressure in the accumulator tank and the initial fluorine or chlorine pentafluoride temperature was varied with each material in order to define the pressures and temperatures at which the metals were susceptible to attack. The test specimen was replaced after each test to insure that comparable surfaces were being exposed to the test conditions. The pneumatic valve opened completely within 1.5 milliseconds and with an accumulator pressure of 1000 psia; the minimum pressurization rate was 6.7×10^5 psi/sec.



Legend:

BD	:	Burst Disc	PG	:	Pressure Guage
CV	:	Check Valve	R	:	Gaseous Fluorine Regulator
GN_2-A	:	Gaseous Nitrogen Accumulator	ROV	:	Remote Operation Valve (Run Valv
GF_2-C	:	Gaseous Fluorine Cylinder	Trap	:	Liquid Nitrogen Vacuum Trap
GF_2-R	:	Gaseous Fluorine Reservoir	V	:	Hand Operated Valve, 1/4-in. Control Components

Figure 10. Schematic Diagram of System for Conducting the Adiabatic Compression Testing

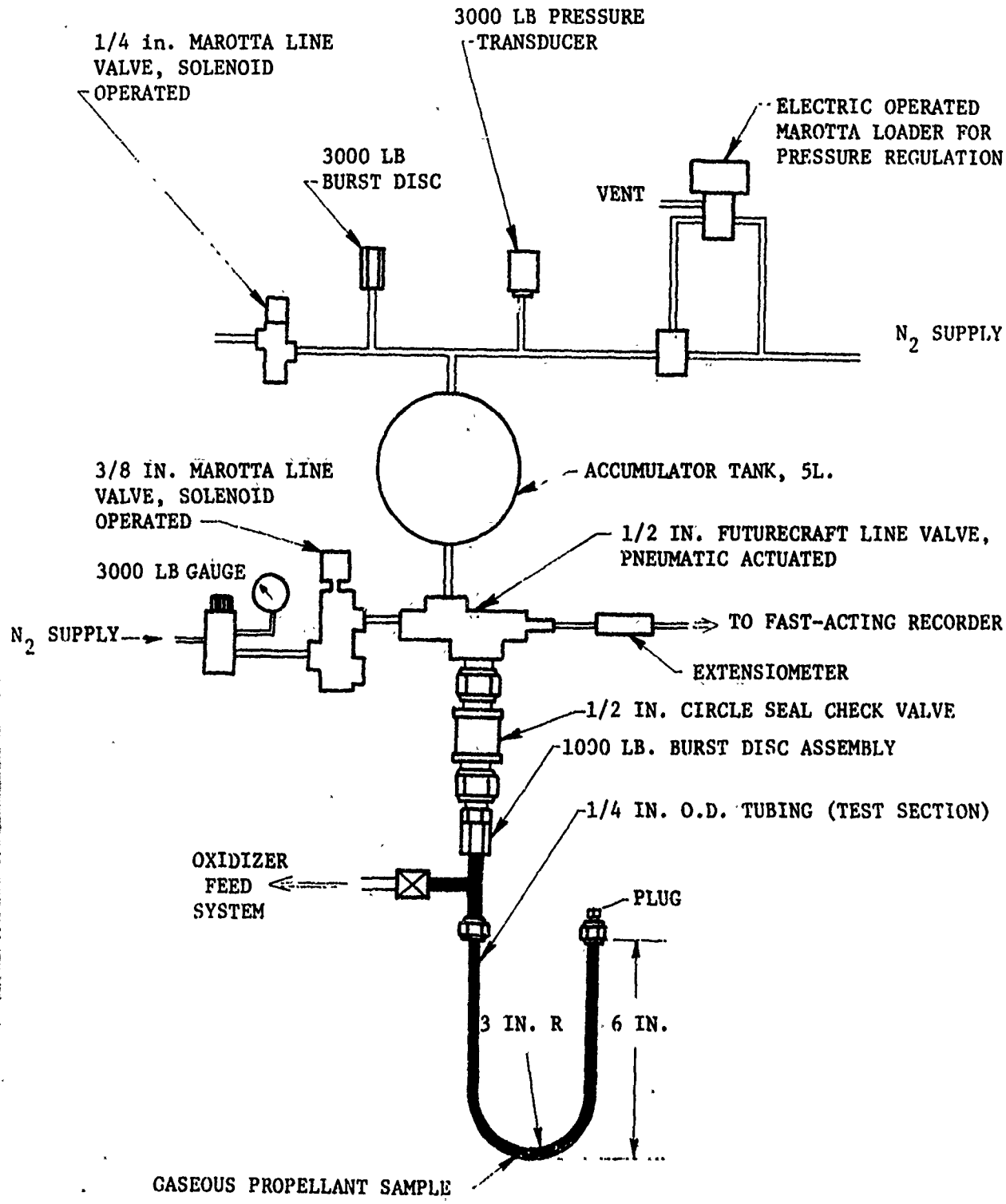


Figure 11. Schematic Diagram of U-tube Adiabatic Compression Apparatus

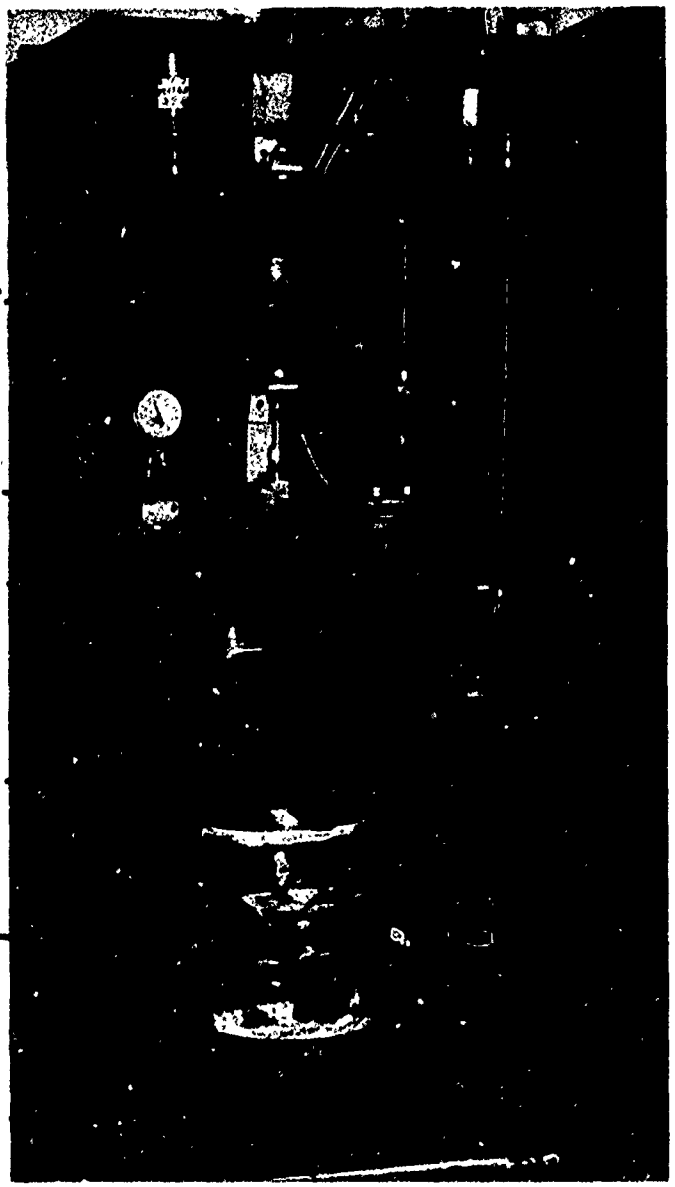


Figure 12. Adiabatic Compression Apparatus

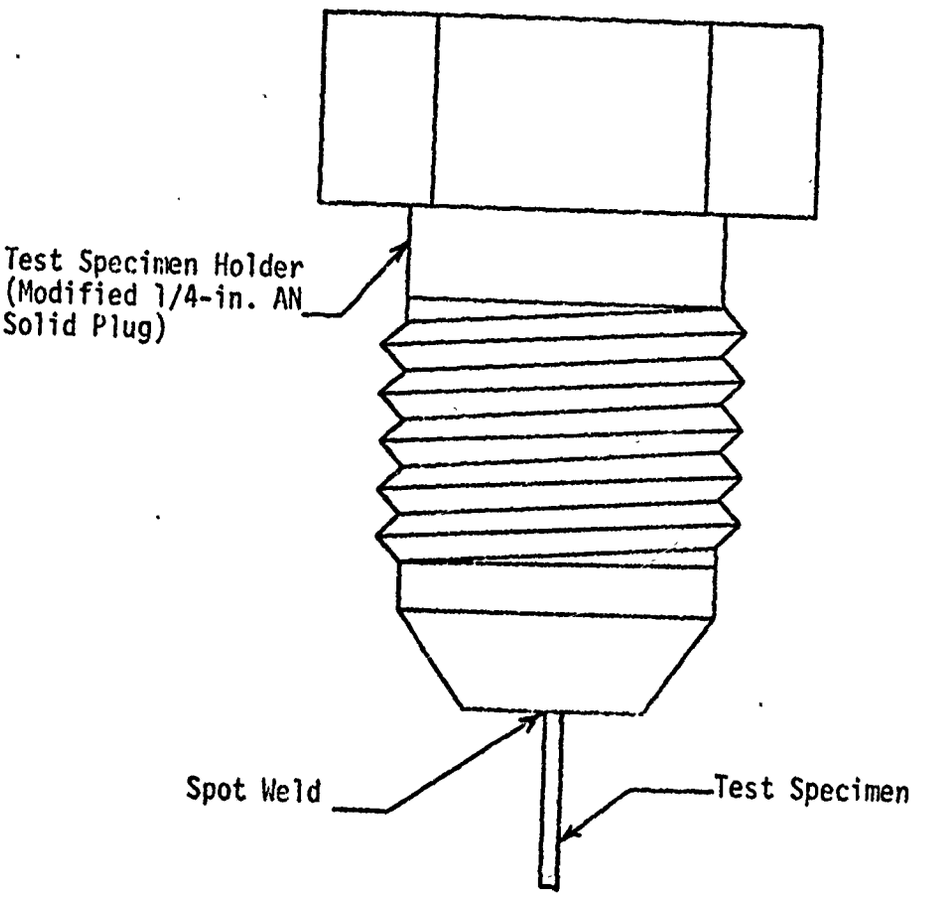


Figure 13. Schematic of Test Specimen Holder with Test Specimen in Place

II, B, Adiabatic Compression Tests (cont.)

2. Experimental Results

The experimental results are discussed under three headings: (1) Gaseous Fluorine, (2) Gaseous Chlorine Pentafluoride, and (3) Comparison of the Behavior of Fluorine and Chlorine Pentafluoride with Selected Metals. The discussion follows in the above order.

a. Gaseous Fluorine

The metal specimens used with gaseous fluorine were composed of the following alloys:

304-L Stainless Steel	6061-T6 Aluminum
347 Stainless Steel	OFHC Copper
321 Stainless Steel	Inconel 718
A-286 Stainless Steel	Monel K-500

The results of 107 tests conducted on the specimens of the nine metals are summarized in Table VIII. The initial fluorine temperature, T_o, and pressure, P_o, and the final pressure, P_f, are experimentally determined values. The final temperature, T_f, is calculated from Equation 1 assuming the fluorine to be a

$$T_f = T_o \left(\frac{P_f}{P_o}\right)^{\frac{\gamma-1}{\gamma}} \qquad (1)$$

nondissociating ideal gas and where γ is defined by Equation 2 using values of Cp for fluorine from Reference 4.

$$\gamma = \frac{\left(\frac{Cp}{Cp - R}\right)_{T_o} + \left(\frac{Cp}{Cp - R}\right)_{T_f}}{2} \qquad (2)$$

The estimated final density, ρ_f, is calculated from the perfect gas law and is evaluated at T_f and P_f.* Test results denoted by a minus sign (-) indicate that attack is not evident by microscopic examination, those denoted by a single plus sign (+) indicate attack is apparent, and those denoted by a double plus sign (++) indicate the specimen was totally destroyed.

Examples of test specimens which have been subjected to adiabatic compression are presented in Figures 14 to 19. Figure 14 is a photograph of a metal specimen which was subjected to adiabatic compression in gaseous

* Final gas densities calculated using generalized compressibility factors from Lydersen, et al. ("Generalized Thermodynamic Properties of Pure Liquids", Univ. Wisconsin Coll. Eng. Rept. 4, Oct. 1955) show maximum deviations from perfect gas behavior are $\leq$ 0.5% and justify the use of the perfect gas law.

TABLE VIII

BEHAVIOR OF VARIOUS METALS IN THE PRESENCE OF GASEOUS FLUORINE SUBJECTED TO ADIABATIC COMPRESSION

	Initial Condition		Final Condition			
Metal	Temp. T_o, °F	Press., P_o, psia	Press., P_f, psia	Calc. Temp. T_f, °F	Est. Density, ρ_f, lb/ft^3	Test Results
304-L Stainless Steel	62	15.7	1515	1085	3.48	-
	67	15.7	2515	1275	5.14	-
	68	15.7	2815	1310	5.64	-,+
	67	15.7	3015	1335	5.95	+,+
	212	15.7	1015	1310	2.03	-,-
	212	15.7	1515	1470	2.78	-,+
	212	15.7	1765	1530	3.14	-,+
	212	15.7	2015	1585	3.49	+
	369	15.7	3015	2235	3.97	-
	370	15.7	3015	2235	3.97	-
321 Stainless Steel	66	15.7	3015	1330	5.97	-
	70	15.7	3015	1345	5.92	-,-,-,-
	212	15.7	915	1270	1.87	-,-
	212	15.7	1015	1310	2.03	-,-,+
	212	15.7	1265	1395	2.42	-,++
	212	15.7	1515	1470	2.78	-
	212	15.7	2015	1585	3.49	-,++
	212	15.7	2515	1685	4.16	++,++
	212	15.7	3015	1760	4.81	+,++,+
	374	15.7	3015	2250	3.94	-
	375	15.7	3015	2250	3.94	-
347 Stainless Steel	212	15.7	2015	1585	3.49	-,-,-
	212	15.7	2265	1635	3.83	-,-
	212	15.7	2515	1685	4.16	-,++
	360	15.7	3015	2205	4.01	-
	366	15.7	3015	2225	3.98	-
A-286 Stainless Steel	62	15.7	2515	1260	5.16	-
	62	15.7	2715	1290	5.50	-
	68	15.7	2815	1310	5.64	-,+,+
	67	15.7	3015	1335	5.95	++
	212	15.7	3015	1760	4.81	-,-
	331	15.7	3015	2120	4.14	-
	332	15.7	3015	2125	4.13	-
6061-T6 Aluminum	62	15.7	2015	1180	4.36	-
	62	15.7	2515	1260	5.18	-
	66	15.7	3015	1330	5.97	-,++,-
	212	15.7	3015	1760	4.81	-,-
	369	15.7	3015	2235	3.97	-
	370	15.7	3015	2235	3.97	-

TABLE VIII (cont.)

BEHAVIOR OF VARIOUS METALS IN THE PRESENCE OF GASEOUS FLUORINE SUBJECTED TO ADIABATIC COMPRESSION (Cont.)

	Initial Condition		Final Condition			
Metal	Temp. T_o, °F	Press., P_o, psia	Press., P_f, psia	Calc. Temp. T_f, °F	Est. Density, ρ_f, lb/ft^3	Test Results
OFHC Copper	66	15.7	3015	1330	5.97	-
	70	15.7	3015	1345	5.92	-
	212	15.7	3015	1760	4.81	-,-
	367	15.7	3015	2230	3.97	-
	368	15.7	3015	2230	3.97	-
Inconel 718	62	15.7	3015	1325	5.99	-,-
	212	15.7	2265	1635	3.83	-,-
	212	15.7	2515	1685	4.16	-,+
	212	15.7	3015	1760	4.81	-,+,-,-
	358	15.7	3015	2200	4.02	-
	360	15.7	3015	2205	4.01	-
Monel K-500	66	15.7	2515	1275	5.14	-,-,+
	67	15.7	2715	1300	5.47	-
	67	15.7	2815	1310	5.64	-
	67	15.7	3015	1335	5.95	++
	212	15.7	2015	1585	3.49	-,-
	212	15.7	2515	1685	4.16	+,+
	212	15.7	3015	1760	4.81	-,-,-,-
	367	15.7	3015	2230	3.97	-
	368	15.7	3015	2230	3.97	-
Nickel 200	63	15.7	3015	1330	5.97	-,-
	212	15.7	2765	1720	4.50	-,-
	212	15.7	3015	1760	4.81	-,-
	361	15.7	3015	2210	4.00	-
	362	15.7	3015	2215	4.00	-

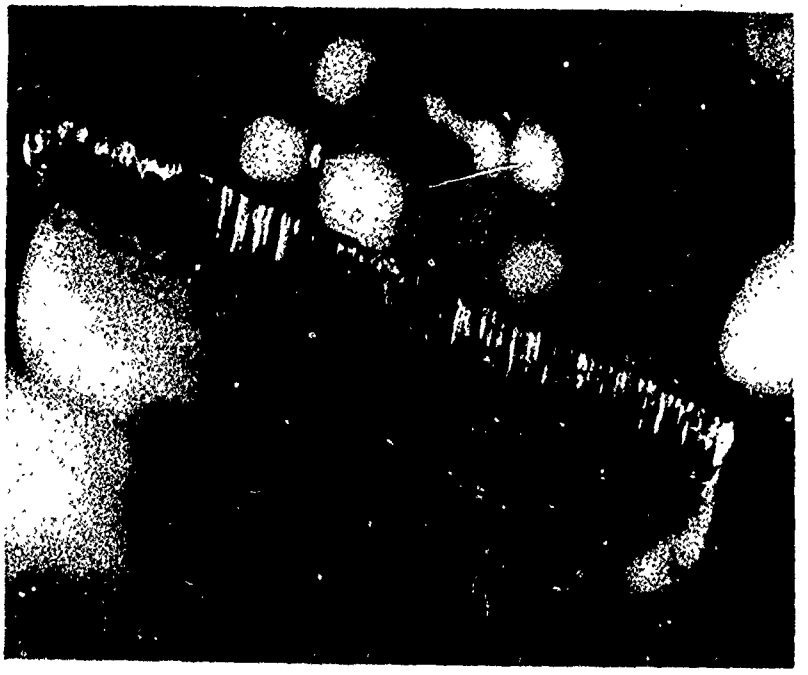


Figure 14. Photograph (40X) of the Edge of a Metal Specimen Subjected to Adiabatic Compression with Nitrogen at a Compression Ratio of 192

Figure 15. Photograph (40X) of the Edge of a Metal Specimen Subjected to 15.7 psia Fluorine Passivation

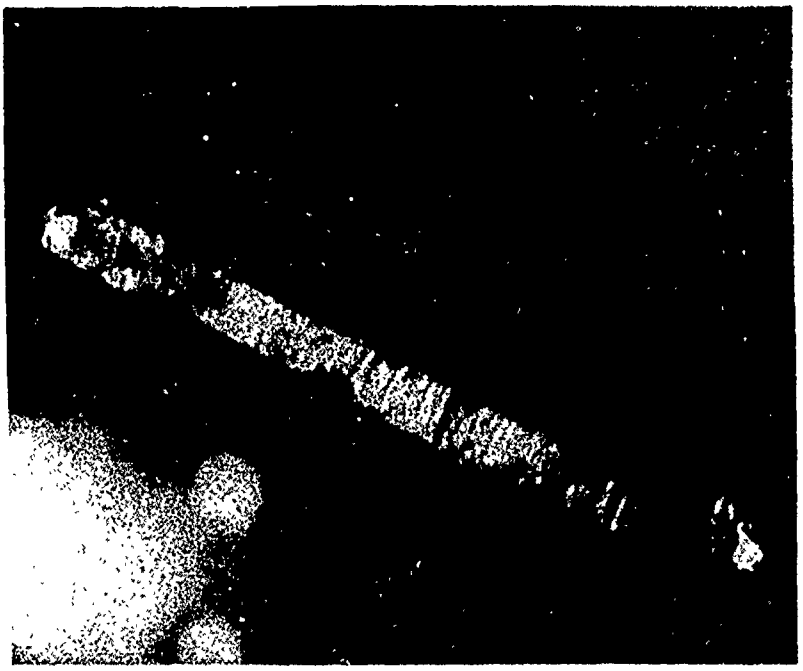


Figure 16. Photograph (40X) of the Edge of a Metal Specimen Subjected to Adiabatic Compression with Fluorine at a Compression Ratio of 197, A Negative Test Result

Figure 17. Photograph (40X) of a Monel K-500 Specimen Completely Destroyed When Subjected to Adiabatic Compression with Fluorine at a Compression Ratio of 160, A (++) Positive Test Result

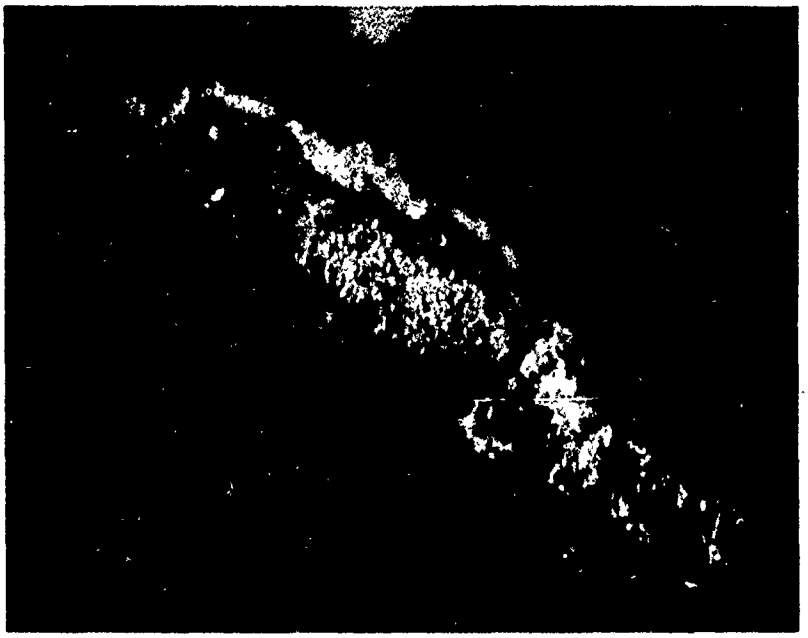


Figure 18. Photograph (40X) of a A-286 Specimen which Partially Reacted with Fluorine at a Compression Ratio of 192, A(+) Positive Result

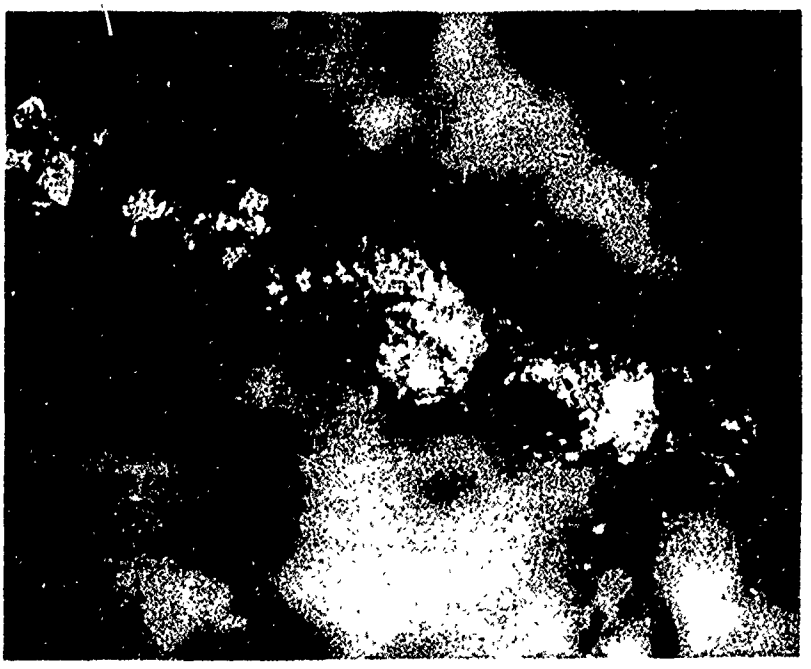


Figure 19. Photograph (40X) of a A-286 Specimen which Partially Reacted with Fluorine at a Compression Ratio of 179, A (+) Positive Result

II, B, Adiabatic Compression Tests (cont.)

nitrogen at a compression ratio of 192. This specimen serves as a reference standard for comparison purposes for the basic effect of adiabatic compression itself. In Figure 15, a photograph of a metal specimen which was subjected only to passivation with gaseous fluorine at 15.7 psia is shown. This specimen serves as a reference for the effect of fluorine passivation at low pressures. In Figure 16, a photograph is shown of a 321 SS metal specimen subjected to adiabatic compression in fluorine at a compression ratio of 192. The specimen is an example of a negative test result. In Figure 17, a photograph is shown of a Monel K-500 test specimen which was completely destroyed by adiabatic compression in fluorine. The test specimen remains are in the form of a metal globule. This is an example of a ++ positive test result. Figures 18 and 19 are photographs of A-286 specimens which partially reacted when subjected to adiabatic compression in fluorine at compression ratios of 192 and 179, respectively. The positive test results in cases such as those are denoted by a single +.

Specimens of OFHC copper and Nickel 200 were not visibly attacked under any of the test conditions. 304-L stainless steel and Inconel 718 were only slightly attacked. Monel K-500, 321 stainless steel, and A-286 stainless steel showed variable degrees of attack from superficial attack to total consumption. 347 stainless steel and 6061-T6 aluminum were totally consumed whenever attack was evident but each metal gave only one positive reaction out of 9 tests on each metal. The conditions under which the various metals can be expected to have approximately a 50% probability of reaction with gaseous fluorine subjected to adiabatic compression were derived from the data given in Table VIII. These conditions are summarized in Table IX. From the data presented in Tables VIII and IX, it is concluded that the metals can be ranked in categories in the following descending order of resistance to the effects of fluorine adiabatic compression: (1) Nickel 200 and OFHC copper, (2) Inconel 718, and (3) 304-L, 321 and A-286 stainless steels. The proper ranking of Monel K-500, 6061-T6 aluminum and 347 stainless steel is indefinite because of insufficient or irregular positive test results, but they appear to belong in the third category.

It is interesting to note that the results of these tests are in quite good agreement with gas flow and liquid impact data (see Tables II and XIII, respectively) in terms of threshold temperatures and material rankings except that the adiabatic compression tests essentially reverse the order of Monel K-500 and OFHC copper. This significant reversal in relative resistance to attack has not been explained but it is also observed in connection with ignition temperatures in static fluorine (see Table III).

It can be seen in Table VIII that no positive reactions were obtained when fluorine temperatures prior to adiabatic compression were in the temperature range of 331 to 375°F. The corresponding temperature and

TABLE IX

TERMINAL ADIABATIC COMPRESSION CONDITIONS THAT RESULT IN A 50% PROBABILITY OF FLUORINE/METAL REACTION

Metal	Conditions for a 50% Probability of Reaction: Calc. Final Temp., °F	Calc. Final Density, lb/ft^3	Magnitude of Resulting Reaction
6061-T6 Aluminum	~1330*	~5.97	Major
A-286 Stainless Steel	1300	5.57	Variable
304-L Stainless Steel	1310	5.64	Slight
	1500	2.96	Slight
321 Stainless Steel	1475	2.90	Variable
347 Stainless Steel	~1685*	~4.16*	Major
Inconel 718	~1685*	~4.16*	Slight
Monel K-500	~1305	~5.55	Variable
	~1635	~3.82	Slight
OFHC Copper	Not Defined		Insignificant
Nickel 200	Not Defined		Insignificant

*Insufficient positive reaction data points to define reaction probability accurately.

II, B, Adiabatic Compression Tests (cont.)

density conditions upon adiabatic compression were 2120 to 2250°F and approximately 4 lb/ft^3, respectively; conditions considerably more severe than those achieved in many positive tests which involved lower initial temperatures. The failure of specimens to react with fluorine under these apparently more drastic conditions has not been explained but it is postulated that passivation-type reactions at the highest initial temperatures may have resulted in the formation of "passive films" which are sufficiently protective to be affected within the time span of an adiabatic compression.

The data obtained from all the tests indicate that the threshold temperature for fluorine/metal reaction resulting from adiabatic compression varies inversely with the final fluorine densities for a given metal. However, the failure to achieve reaction under some apparently drastic conditions, indicate that some factor other than final temperature and density has an important influence on a metal's susceptibility to reaction via adiabatic compression.

b. Gaseous Chlorine Pentafluoride

The metal specimens used in the investigation were composed of the following alloys:

304-L Stainless Steel	6061-T6 Aluminum
301 Cryo Stainless Steel	OFHC Copper
347 Stainless Steel	Inconel 718
321 Stainless Steel	Monel K-500
A-286 Stainless Steel	Nickel 200

The results of sixty tests conducted on the specimens of the ten metals are summarized in Table X. The initial ClF_5 temperature, T_o, and pressure, P_o, and the final pressure, P_f, are experimentally determined values. The final temperature, T_f, is calculated from Equation 1 assuming the CLF_5 to be a nondissociating ideal gas; γ is defined by Equation 2 using values

$$T_f = T_o \left(\frac{P_f}{P_o}\right)^{\frac{\gamma-1}{\gamma}} \qquad (1)$$

of Cp for ClF_5 from Reference 5.

$$\gamma = \frac{\left(\frac{Cp}{Cp - R}\right)_{T_o} + \left(\frac{Cp}{Cp - R}\right)_{T_f}}{2} \qquad (2)$$

TABLE X

BEHAVIOR OF VARIOUS METALS IN THE PRESENCE OF GASEOUS CHLORINE PENTAFLUORIDE SUBJECTED TO ADIABATIC COMPRESSION

Metal	Initial Condition		Final Condition			Test Results
	Temp., T_o, °F	Press., P_o, psia	Press., P_f, psia	Calc. Temp., T_f, °F	Est. Density ρ_f, lb/ft^3	
301 Cryo Stainless Steel	62	15.7	3015	328	84.3	-,-
	212	15.7	2815	519	50.9	-, -
	212	15.7	3015	523	54.0	-, ++
	336	15.7	3015	688	38.4	-
	339	15.7	3015	692	38.2	-
	355	15.7	3015	714	37.0	-, ++
304-L Stainless Steel	62	15.7	3015	328	84.3	-, -
	212	15.7	2815	519	50.9	-, -
	212	15.7	3015	523	54.0	-, ++
	346	15.7	3015	702	37.6	-
	352	15.7	3015	710	37.2	-
321 Stainless Steel	62	15.7	3015	328	84.3	-, -
	350	15.7	3015	707	37.4	-
	354	15.7	3015	713	37.0	-
347 Stainless Steel	62	15.7	3015	328	84.3	-, -
A-286 Stainless Steel	63	15.7	3015	329	84.3	-, -
	612	15.7	3015	523	54.0	-, -
	346	15.7	3015	702	37.6	-
	351	15.7	3015	708	37.3	-
6061-T6 Aluminum	63	15.7	3015	329	84.3	-, -
	212	15.7	3015	523	54.0	-, -
	343	15.7	3015	698	37.8	-
	344	15.7	3015	699	37.7	-, -
OFHC Copper	63	15.7	3015	329	84.3	-, -
	212	15.7	3015	523	54.0	-, -
	341	15.7	3015	695	38.0	-
	350	15.7	3015	707	37.4	-
Inconel 718	63	15.7	3015	329	84.3	-, -
	212	15.7	3015	523	54.0	-, -
	341	15.7	3015	695	38.0	-
	346	15.7	3015	702	37.6	-
Monel K-500	63	15.7	3015	329	84.3	-, -
	212	15.7	3015	523	54.0	-, -
	345	15.7	3015	700	37.7	-
	351	15.7	3015	708	37.3	-
Nickel 200	63	15.7	3015	329	84.3	-, -
	212	15.7	3015	523	54.0	-, -
	351	15.7	3015	708	37.3	-, -

II, B, Adiabatic Compression Tests (cont.)

The estimated final density, ρ_f, is based on the density values given in Reference 6 and is evaluated at T_f and P_f. Test results denoted by a minus sign (-) indicate that attack is not evident by microscopic examination; those denoted by a double plus sign (++) indicate the specimen was totally destroyed.

It should be noted that only three positive results were obtained in the sixty tests and these occurred on 301 Cryo and 304-L stainless steels at the maximum pressurization capabilities of the test apparatus (3000 psig driving pressure) and with the chlorine pentafluoride vapor at an initial temperature of 212°F or higher. Although so few positive results make it impossible to define critical adiabatic compression thresholds for most of the materials, it can be concluded that all the materials tested are highly resistant to reaction with chlorine pentafluoride undergoing an adiabatic compression more severe than could be expected in most practical rocket systems.

Based upon the data given in Table X, a safe, nonreactive region and a possibly reactive region is presented in Figure 20 in terms of final chlorine pentafluoride temperature and density coordinates. The diagram (Figure 20) is applicable to all the materials tested.

c. Comparison of the Behavior of Fluorine and Chlorine Pentafluoride with Selected Metals

The data from the adiabatic compression tests with gaseous fluorine and gaseous chlorine pentafluoride are combined and plotted as final gas density versus final gas temperature in Figure 21. The region below the lines defines the conditions under which the adiabatic compression of the gaseous propellant did not significantly attack the selected metal specimens. In the region above the lines conditions are defined under which significant reaction may occur between the metal specimens and the gaseous propellants undergoing adiabatic compression. Considerable extrapolation was necessary to generate the plot in Figure 21 and in specific design situations the original data generated in this investigation should be consulted.

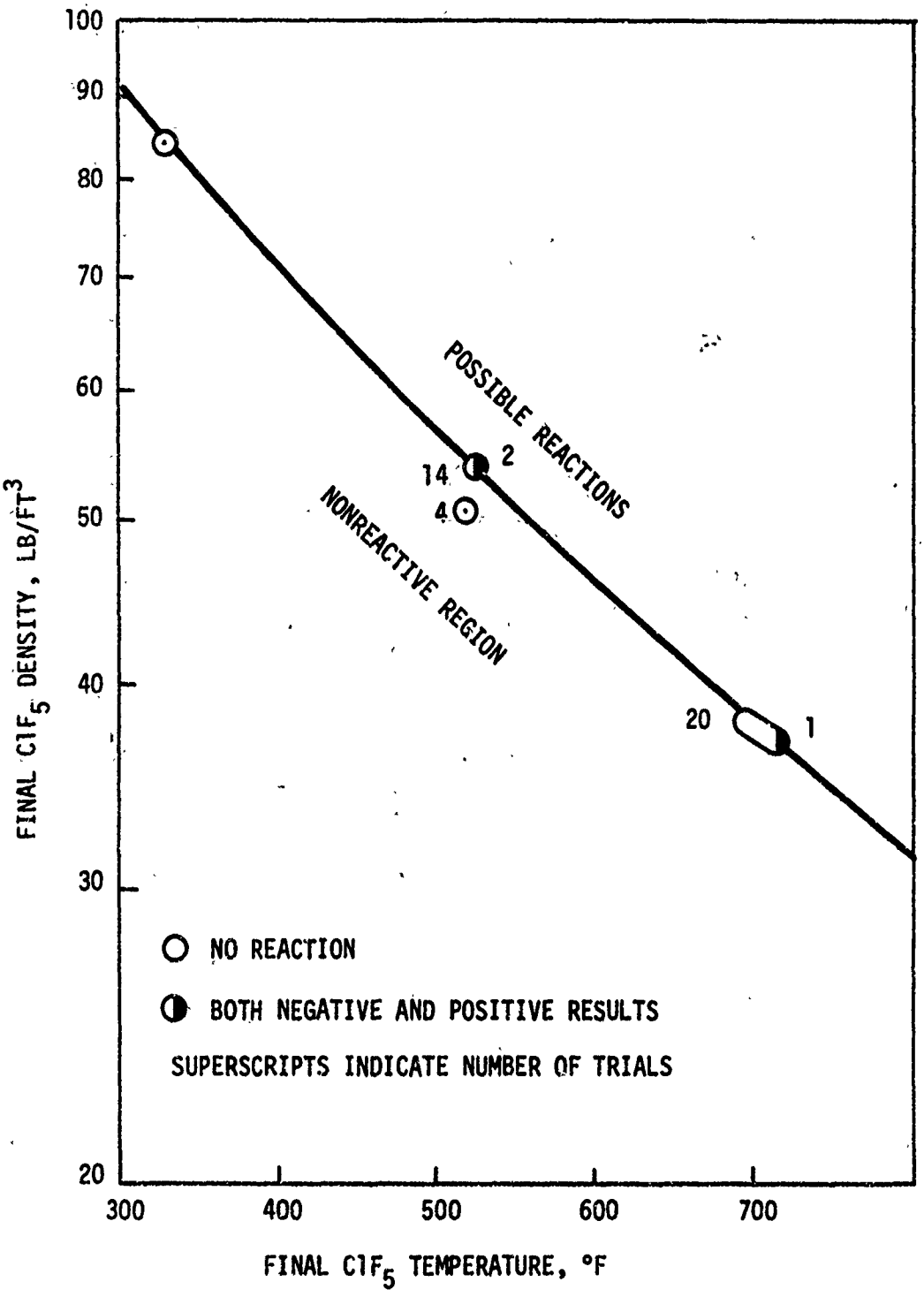


Figure 20. Reactivity Profile for Certain Metals Exposed to Gaseous Chlorine Pentafluoride Undergoing Adiabatic Compression

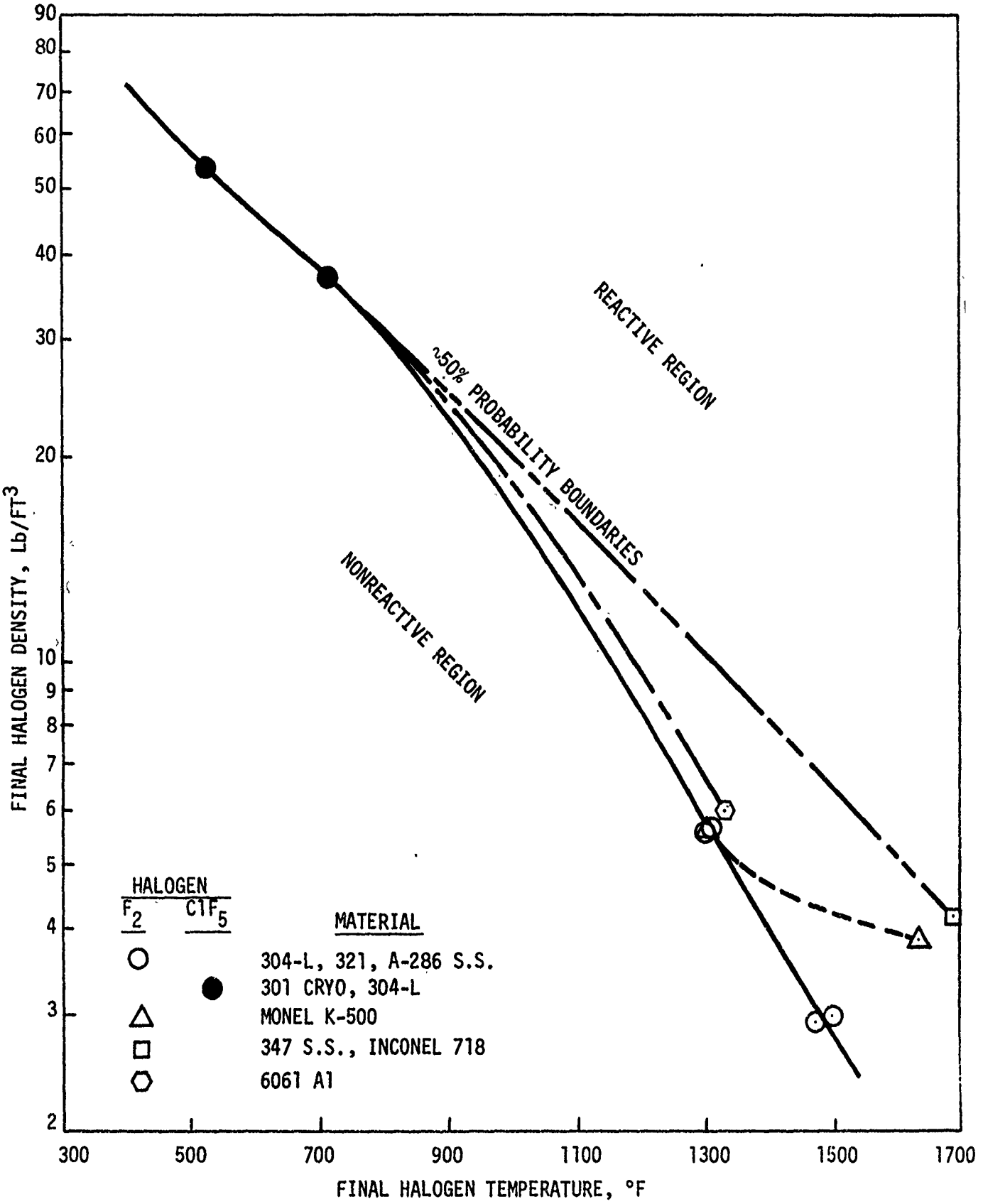


Figure 21. Reactivity Profile for Metals Exposed to Gaseous Chlorine Pentafluoride and Gaseous Fluorine Undergoing Adiabatic Compression

II, Experimental Results and Discussion (cont.)

C. LIQUID PHASE SHOCK WAVE TESTS

Tests were conducted to determine the effects of a shock wave promulgated through liquid fluorine and chlorine pentafluoride (water hammer effect) on the metals listed below.

301 Cryoformed Stainless Steel	
304-L Stainless Steel	OFHC Copper
321 Stainless Steel	Inconel 718
A-286 Stainless Steel	Monel K-500
6061-T6 Aluminum	Nickel 200

1. Apparatus and Procedures

The tests were conducted in the adiabatic compression test apparatus shown schematically in Figure 11 with 2 to 3 ml of liquid oxidizer condensed in the U-tube. The configuration of the test specimen of the metal was the same as described on page 29 which facilitated the discrimination between the results of adiabatic compression and the "water hammer effect." Calculation of the final temperature which the fluorine vapor will reach due to adiabatic compression in the "water hammer" test is 233°F which is far too low to cause any reaction with the metal specimen. Likewise a calculation demonstrates that the chlorine pentafluoride vapor will actually be condensed to the liquid phase during the compression. On these bases any attack which occurs during the tests should be due to the liquid phase shock wave. Prior to the tests with fluorine and chlorine pentafluoride, the apparatus was checked out with water in the U-tube and with various driving pressures. The resultant pressure spikes were recorded with a 6K Tabor transducer. With a 1000 psig driving pressure, a pressure spike of 8300 psig was measured showing that the pressure in the shock wave front was approximately eight times the driving pressure. The oscillographic trace is shown in Figure 22. Similar results were obtained with 2000 and 3000 psig of driving pressure; however, the transducer was mechanically stopped at 9500 psig and the absolute peak pressures were beyond the range of the transducer.

2. Experimental Results

a. Liquid Fluorine

The experimental data obtained with liquid fluorine are presented in Table XI. The significant items to be noted from the data are that: (1) 304-L stainless steel and Inconel 718 were apparently not attacked during the tests (2) Monel K-500 showed discoloration due to localized heating; and (3) 321 and A-286 stainless steels, 6061 aluminum, OFHC copper, and Nickel 200 underwent very slight attack as evidenced by the removal of the sharp edges

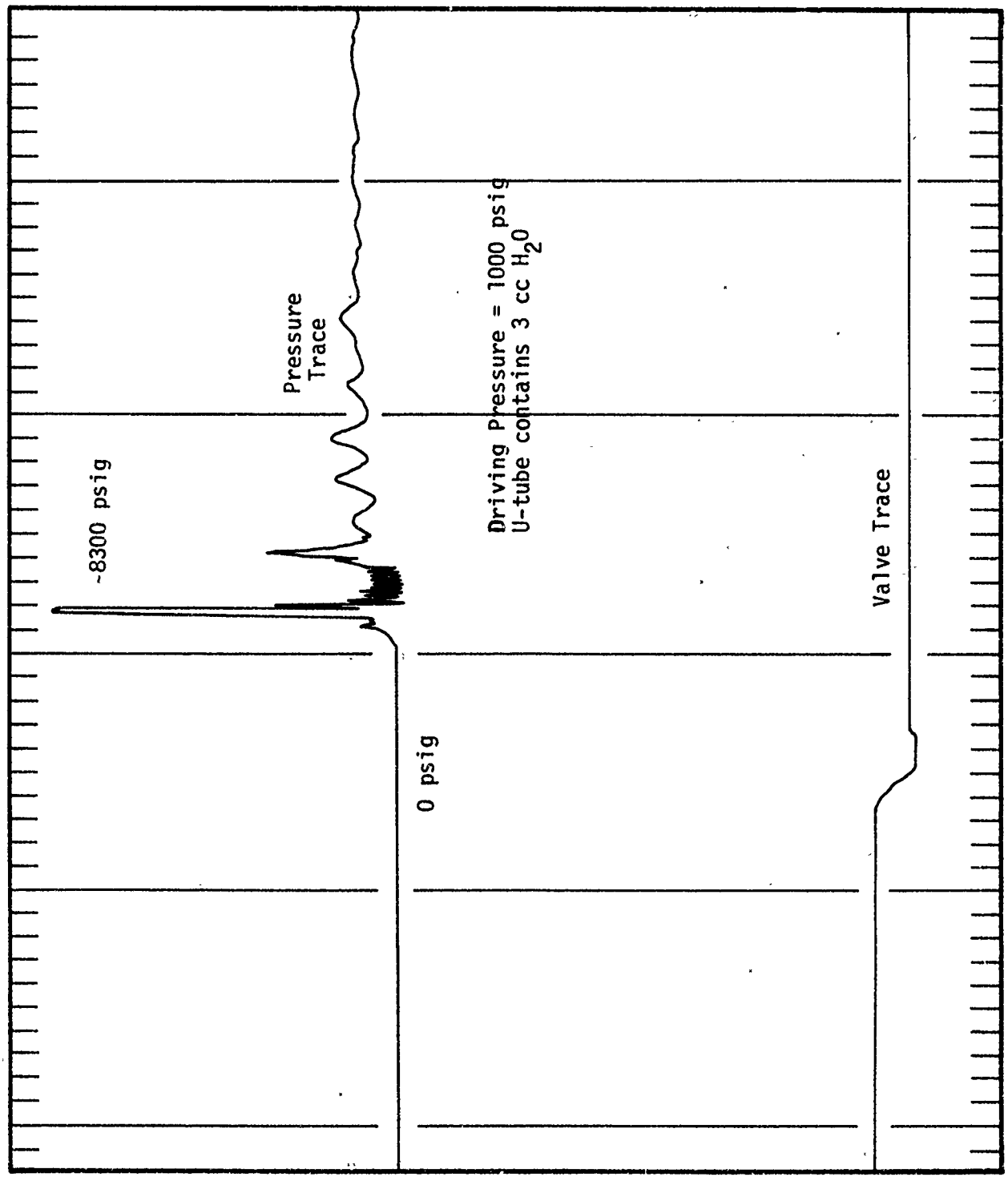


Figure 22. Water-Hammer Effect Obtained with Adiabatic Compression Test Apparatus

TABLE XI

BEHAVIOR OF VARIOUS METALS
SUBJECTED TO A SHOCK WAVE OF LIQUID FLUORINE

METAL	INITIAL LIQUID TEMPERATURE, °F	DRIVING PRESSURE, psig	RESULT
304-L Stainless Steel	-321	2000	No observable effect (2 samples)
321 Stainless Steel	-321	2000	No observable effect
321 Stainless Steel	-321	2000	Slight erosion of sample edge
A-286 Stainless Steel	-321	2000	Erosion of sample edges
A-286 Stainless Steel	-321	2000	Erosion of sample edges
6061 Aluminum	-321	2000	No observable effect
6061 Aluminum	-321	2000	Erosion of sample edges
OFHC Copper	-321	2000	No observable effect
OFHC Copper	-321	2000	Erosion of sample edge
Monel K-500	-321	2000	Discoloration due to heating
Monel K-500	-321	2000	Discoloration due to heating
Inconel 718	-321	2000	No observable effect (2 samples)
Nickel 200	-321	2000	No observable effect
Nickel 200	-321	2000	Erosion of sample edges

II, C, Liquid Phase Shock Wave Tests (cont.)

from the test specimen. It must be emphasized that none of the attack observed in these "water hammer" tests led to significant destruction of the metal. The effects were noted by microscopic examination of samples at 40X magnification. A photograph of the 321 SS specimen which experienced erosion of the sample edges is shown in Figure 23. Baseline tests, using water with 301 cryoformed stainless steel and 6061 aluminum, showed no significant attack.

The results of a related shock wave test have been reported by Douglas Aircraft Company in Reference 7. An aluminum casting alloy, A 356-T6, Grade III-F, was exposed to an explosive shock of 120,000 psi after a 24-hr soak in LF_2 (0.02% vol HF) at -320°F using a test apparatus of the card-gap type. The test resulted in a reaction with no trace of the test coupon remaining.

Generally similar tests were made by Sterner and Singleton (Reference 8) to investigate the possibility of metal ignition in liquid fluorine under conditions of explosive shock. The runs were carried out in metal tubes 2-in. long x 5/8-in. ID x 0.065-in. wall thickness, with the tubes themselves being the samples. The tubes were closed at the ends by brass plugs, silver soldered in place, and fluorine was added through 1/4-in. copper tube mounted in the sample tube wall. Two tubes of each material were tested, including Monel, nickel, brass, copper, 304 SS, 316 SS, 347 SS, and 1100 aluminum. The experimental procedure was as follows:

(1) The 1/4-in. tube was attached to the fluorine gas manifold and a No. 6 electric blasting cap was tightly fastened to the tube wall with nichrome wire.

(2) An insulated can was positioned and liquid nitrogen was added to the can, covering the cell completely.

(3) Fluorine gas was condensed into the cell, filling it completely with liquid.

(4) Ten minutes after the tube was filled, the blasting cap was set off.

The ends of the tubes were usually blown out by hydrostatic pressure generated by the deformation of the tube walls, but no evidence of fluorine reaction was observed in any run.

From the composite results of these tests, it is concluded that the shock waves generated in liquid fluorine by driving the fluorine against a dead end at a driving pressure of 2000 psig or by the close-coupled explosive shock of a No. 6 blasting cap have little or no ability to initiate a significant reaction with aluminum, brass, copper, Inconel, Monel, nickel, and austenitic stainless steels.

Figure 23. Photograph (40X) of a 321 Stainless Steel Specimen which Shows Erosion of Edges due to the "Water Hammer" Effect in Liquid Fluorine

II, C, Liquid Phase Shock Wave Tests (cont.)

b. Liquid Chlorine Pentafluoride

The experimental data obtained with liquid chlorine pentafluoride are presented in Table XII. The significant items to be noted from the data are that (1) 6061 aluminum and Monel K-500 were apparently not attacked during the tests; (2) 347 stainless steel showed slight discoloration due to localized heating; and (3) cryoformed 301, 304-L, and A-286 stainless steels, OFHC copper, Inconel 718, and Nickel 200 underwent very slight attack as evidenced by the removal of the sharp edges from the test specimen. It must be emphasized that none of the attack observed in these "water hammer" tests led to significant destruction of the metal. The effects were noted by microscopic examination of samples at 40X magnification.

Comparing the results of these tests with those obtained from the adiabatic compression tests, it can be seen that 301 cryoformed stainless steel showed evidence of attack at the lowest driving pressure (1100 psig) in these tests and is the material that gave most evidence of susceptibility to attack by gaseous chlorine pentafluoride undergoing adiabatic compression (see Table X). A photograph of the 301 specimen which experienced erosion of the sample edges is shown in Figure 24. Also, 304-L stainless steel showed evidence of attack at the next lowest driving pressure (1500 psig) in these tests and is the only other material tested that gave any evidence of attack when subjected to adiabatic compression of the vapor phase (see Table X).

TABLE XII

BEHAVIOR OF VARIOUS METALS SUBJECTED TO A SHOCK WAVE OF LIQUID CHLORINE PENTAFLUORIDE

METAL	INITIAL LIQUID TEMPERATURE, °F	DRIVING PRESSURE psig	RESULT
301 Cryo Stainless Steel	32	1100	No observable effect
301 Cryo Stainless Steel	32	1100	Erosion of sample edges
304-L Stainless Steel	32	1200	No observable effect
304-L Stainless Steel	32	1500	Erosion of sample edges
347 Stainless Steel	32	2000	Discoloration due to heating
347 Stainless Steel	32	2000	No observable effect
A-286 Stainless Steel	32	2000	Erosion of sample edges
A-286 Stainless Steel	32	2000	No observable effect
6061-T6 Aluminum	32	2000	No observable effect (2 samples)
OFHC Copper	32	2000	No observable effect
OFHC Copper	32	2000	Erosion of sample edges
Monel K-500	32	2000	No observable effect (2 samples)
Inconel 718	32	2000	No observable effect
Inconel 718	32	2000	Erosion of sample edges
Nickel 200	32	2000	Slight pitting of surface
Nickel 200	32	2000	Erosion of sample edges

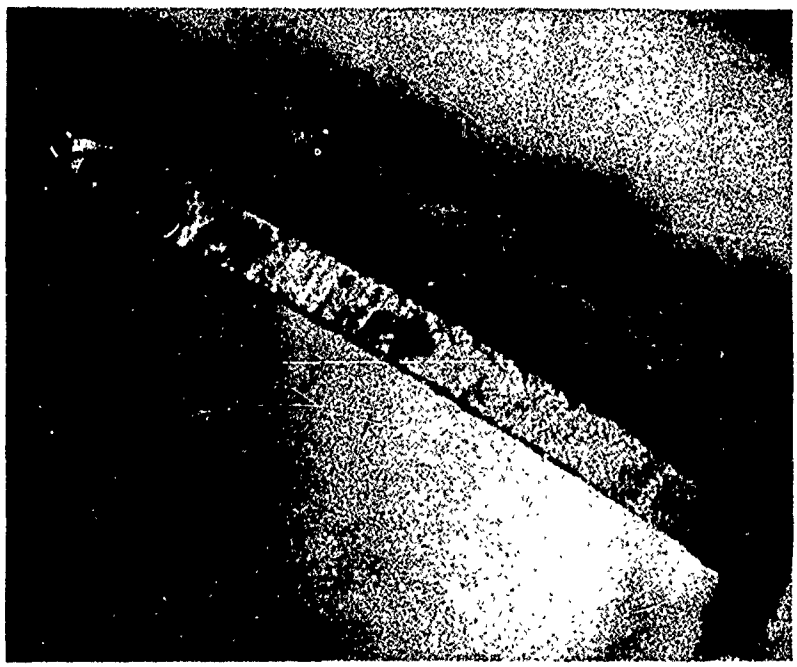


Figure 24. Photograph (40X) of the 301 Cryoformed Specimen which Shows Erosion of the Edges due to the "Water Hammer" Effect in Liquid Chlorine Pentafluoride

II, Experimental Results and Discussion (cont.)

D. LIQUID IMPACT TESTS ON HEATED METALS

The objective of the liquid impact tests on heated metal surfaces was to determine the maximum temperatures to which the selected metals could be heated without detrimental effects occurring when impacted by liquid stream of fluorine or chlorine pentafluoride. Tests were conducted with twelve metals:

304-L Stainless Steel	6061-T6 Aluminum
301 Stainless Steel	2219 Aluminum
301 Cryo Stainless Steel	OFHC Copper
347 Stainless Steel	Inconel 718
321 Stainless Steel	Monel K-500
A-286 Stainless Steel	Nickel 200

1. Apparatus and Procedures

The test apparatus consisted of two major components: (1) a liquid feed system and (2) an electrical-resistance heating system for the metal specimens. A photograph of the test apparatus is shown in Figure 25. The temperature of the metal specimens was measured by means of thermocouples attached to the back of the metal strip which was 0.010-in. thick. The metal specimens were placed within 3/16 in. of the discharge orifice of the liquid propellant feed system. The exit orifice diameter of the propellant feed system was 0.015 in. and the entire feed system was temperature conditioned to -320°F for the liquid fluorine tests and 32°F for the chlorine pentafluoride tests. For each test, a fresh, unpassivated metal specimen was used and the temperature level was increased in 100°F increments for each test until the impacting liquid propellant caused the metal specimen to burn. As soon as the metal specimen reached the desired temperature, the liquid propellant flowed for 250 milliseconds during each test and the feed lines were then immediately purged with helium in order to prevent moisture from accumulating at the exit orifice. The temperature below which significant attack occurred was determined within approximately 50°F. Until the ignition temperature was reached, there was only slight evidence of attack on the metal surfaces.

The flow rates through the exit orifice were determined as a function of the driving pressure using water, ethanol, and trichlorotrifluoroethane as the calibration fluids. The liquid velocity was calculated from the data and the value is accurate to ±2%. The temperatures were measured with chromel-alumel thermocouples which were accurate to ±0.75% in the temperature range encountered. Thus, the temperatures of the test specimens were accurate to ±15°F at the highest temperatures measured.

2. Experimental Results

The results of the liquid fluorine and chlorine pentafluoride tests are reported as being either positive or negative. A photograph containing examples of positive and negative test results is shown in Figure 26. The span between the temperature at which significant attack of the specimen first occurred and the temperature at which the specimen actually burned on impact was found to be approximately 100°F. The data from 277 tests are summarized in Table XIII.

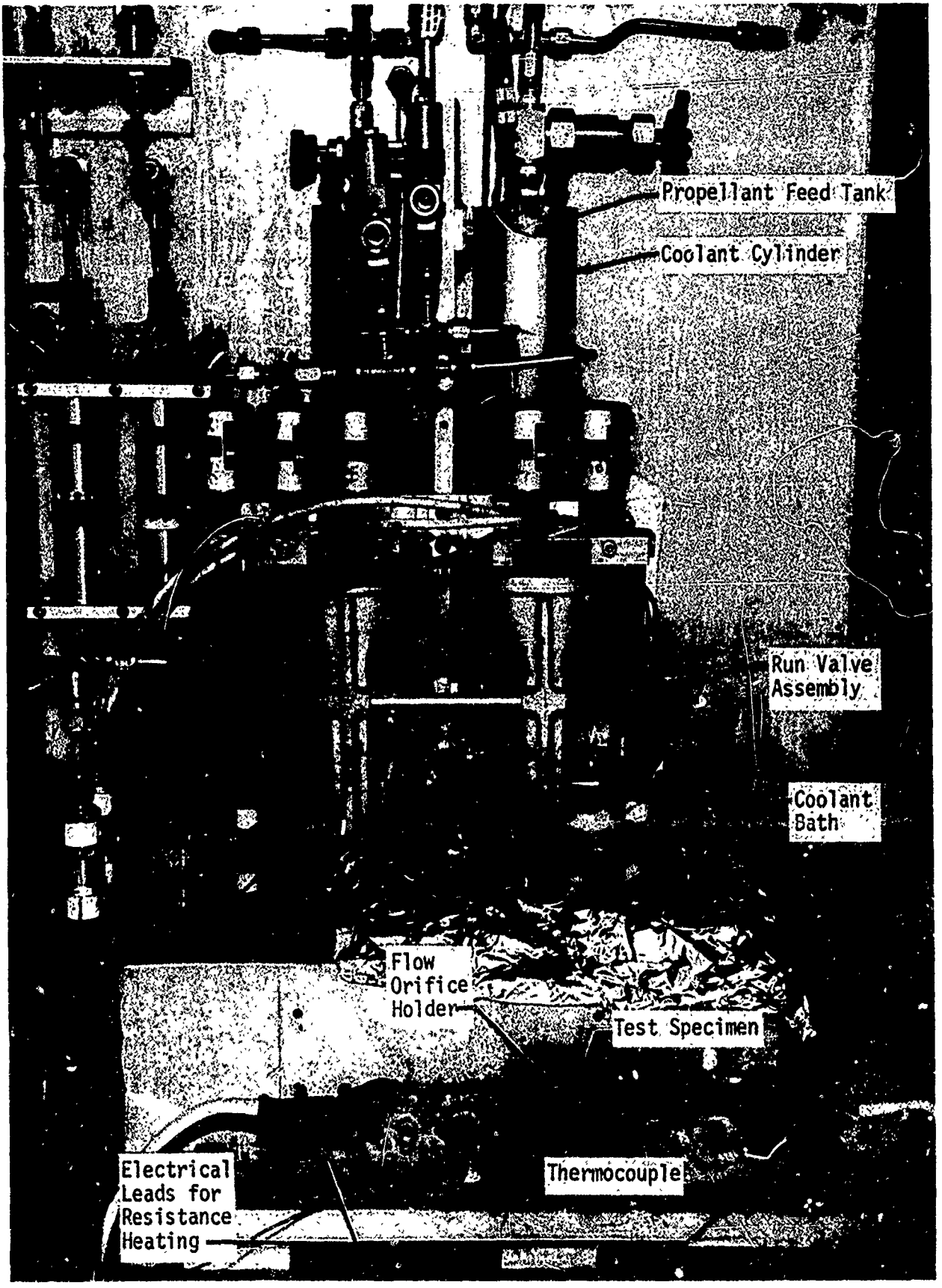
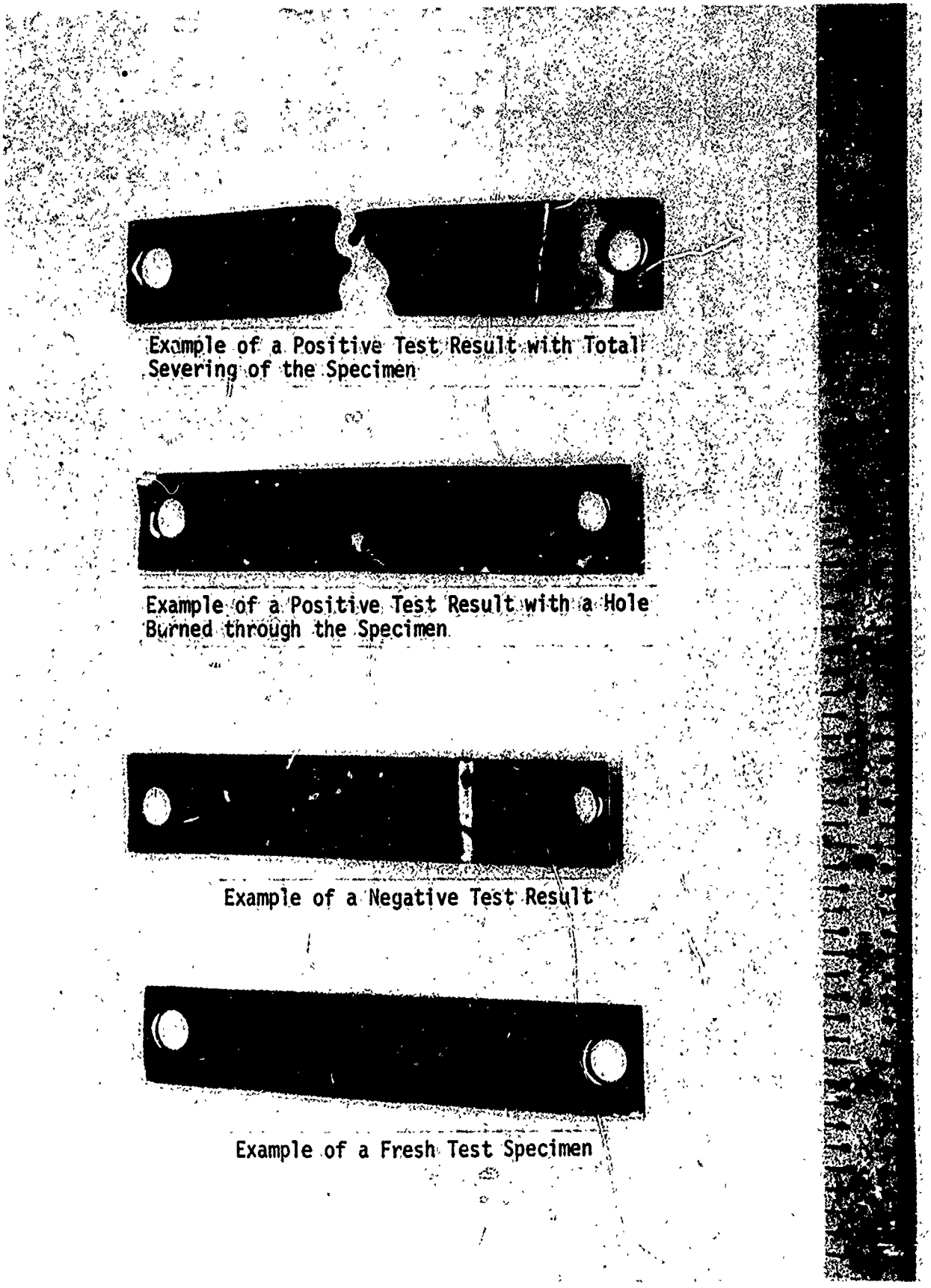


Figure 25. Apparatus Used for Liquid Impact on Heated Metal Surfaces



Example of a Positive Test Result with Total Severing of the Specimen

Example of a Positive Test Result with a Hole Burned through the Specimen

Example of a Negative Test Result

Example of a Fresh Test Specimen

Figure 26. Typical Metal Specimens from Liquid Impact Tests

TABLE XIII

TEST RESULTS OF LIQUID OXIDIZER IMPACTS ON VARIOUS HOT METALS

Specimen Material	Initial Specimen Temperature when Impacted by Liquid Fluorine, °F						Initial Specimen Temperature when Impacted by ClF_5, °F			
	LF_2 @ 295 ft/sec		LF_2 @ 240 ft/sec		LF_2 @ 130 ft/sec		ClF_5 @ 220 ft/sec		ClF_5 @ 120 ft/sec	
	Positive Reaction	No Reaction	Positive Reaction	No Reaction	Positive Reaction	No Reaction	Positive Reaction	No Reaction	Positive Reaction	No Reaction
6061-T6 Aluminum	-	-	-	-	-	≤ 750 (2)	≥1000 (2)*	≤1000 (3)	900 (1)*	≤ 850 (2)
2219 Aluminum	-	-	1050 (1)	-	-	850 (1)	≥ 950 (2)*	-	≥1050 (2)*	≤1000 (4)
OFHC Copper	-	-	1100 (1)	≤ 1050 (2)	1200 (1)	≤1150 (2)	1100 (2) 1200 (1)*	≤1050 (2)	≥1400(3) 1200 (1) 1300 (1)	≤1100 (4) 1200 (2) 1300 (1)
301 Stainless Steel	1200 (1)	1100 (1)	≥1250 (4) 1200 (1)	1100 (1) 1200 (1)	1500 (2)	≤1450 (4)	≥1550 (3)	1500 (2)	1550 (2) 1500 (1)	1450 (1) 1500 (1)
347 Stainless Steel	-	≤1300 (2)	1300 (1)	≤1200 (2)	≥1400 (2)	≤1300 (2)	1600 (2)	≤1550 (3)	≥1550 (9)	1500 (2)
301 Cryo. Stainless Steel	1500 (1)	1400 (1)	≥1450 (3)	≤1400 (4)	1700 (1) 1600 (1)	1500 (1) 1600 (1)	1550 (1)	1500 (1)	1600 (1)	1550 (1)
321 Stainless Steel	≥1450 (2)	1350 (1)	1600 (2)	≤1550 (3)	≥1600 (5)	≤1550 (3)	≥1550 (5) 1500 (1) 1450 (2)	1400 (2) 1500 (1)	≥1850 (2) 1800 (2)	1750 (1) 1800 (1)
A-286 Stainless Steel	1650 (1)	1550 (1)	1700 (1) 1650 (1)	≤1600 (2) 1650 (1)	1700 (2)	≤1650 (3)	1800 (2) 1750 (1)	≤1700 (3) 1750 (1)	≥1600 (4)	≤1550 (2)
304-L Stainless Steel	≥1450 (3)	1350 (1)	1700 (2)	1650 (2)	≥1700 (5)	≤1650 (4)	≥1550 (6) 1500 (1)	≤1450 (4) 1500 (1)	≥1950 (2)	≤1900 (4)
Inconel 718	1900 (1)	≤1850 (3)	1950 (2) 1900 (1)	1850 (1) 1900 (1)	2000 (2)	1950 (2)	≥2050 (3) 2000 (1)	1950 (2) 2000 (1)	2000 (2)	≤1950 (3)
Monel K-500	-	≤2100 (3)	2100 (1)	≤2000 (2)	≥2000 (3)	≤1950 (3)	≥2150 (3)	2100 (2)	2200 (1)*	≤2150 (7)
Nickel 200	2300 (1)	≤2200 (3)	2150 (1)	≤2100 (2)	≥2200 (3)	≤2150 (3)	2350 (2)	2300 (2)	2400 (1)* 2300 (1) 2250 (1)	2000 (1) 2200 (1) 2250 (2)

*Specimens burnt out in the ambient air at this temperature and before any impact of the liquid halogen oxidizer.

II, D, Liquid Impact Tests on Heated Metals (cont.)

a. Liquid Fluorine

Based on the data summarized in Table XIII, a tabulation of the maximum temperatures to which metal surfaces can be heated and then impacted with liquid fluorine at -320°F was developed. This information is presented in Table XIV.

Recognizing that the threshold temperatures given in Table XIV are accurate to within only about ±50°F, it would appear that the velocity of impingement generally has a weak influence on threshold temperatures with few exceptions. For example, the apparent velocity effect on threshold temperatures is greater than 150°F for more than a two-fold change in velocity on only certain of the stainless steels, and specifically, on 301, 321, and 304-L.

Comparing the nonignition threshold temperatures in Table XIV for liquid impingement with the incipient failure threshold temperatures in Table II for flowing gas shows that both tests lead to practically identical material rankings. Further comparisons show that aluminum, copper, and the stainless steels appear to have appreciably lower threshold temperatures to high velocity liquid impingement than to sonic gas flow. Again comparing Table XIV and Table II data, a first glance would suggest that Inconel 718, Monel K-500, and Nickel 200 are more resistant to impinging uid fluorine than to sonic gas flow, but the thresholds in Table II for these materials refer to a potential failure mode involving the onset of strong exotherms and the buildup of significant films or corrosion products only. No evidence of ignition was obtained in flowing sonic gas through Inconel 718, Monel K-500, and Nickel 200 even though temperatures excursions near 2400°F were observed.

b. Liquid Chlorine Pentafluoride

Based on the data in Table XIII, a tabulation of the maximum temperatures to which metal surfaces can be heated and then impacted with liquid chlorine pentafluoride at 32°F is presented in Table XV. Recognizing that the threshold temperatures given in Table XV are accurate to within only about ±50°F, it would appear that the velocity of ClF_5 impingement generally has a weak influence on threshold temperatures with few exceptions. For example, the apparent velocity effect on threshold temperatures is greater than 100°F on only certain of the stainless steels, and specifically, on 321, 304-L, and A-286.

Comparing the nonignition threshold temperatures in Table XV for liquid impingement with the incipient failure threshold temperatures in Table V for flowing gas shows that both tests lead to practically identical material rankings. Further comparisons show that aluminum and copper appear to have appreciably lower threshold temperatures (~300°F) to high velocity liquid impingement than to sonic gas flow while the stainless steels are only slightly less resistant to high velocity impingement. Again comparing Table XV and Table V data, a first glance would suggest that Inconel 718, Monel K-500, and Nickel 200 are more resistant to impinging liquid ClF_5 than to sonic gas flow, but the thresholds in Table V for Monel K-500 and Nickel 200 refer to a potential failure mode involving the onset of strong exotherms and the buildup of significant films or corrosion products only. No evidence of ignition was obtained in flowing sonic gas through Monel K-500 and Nickel 200 even though temperature excursions approaching 2400°F were observed.

TABLE XIV

MAXIMUM TEMPERATURES OF METAL SURFACES ON WHICH IMPACTING STREAMS OF LIQUID FLUORINE DO NOT RESULT IN IGNITION

Material	Approximate Nonignition Threshold Temperature, °F		
	LF_2 Velocity of 130 ft/sec	LF_2 Velocity of 240 ft/sec	LF_2 Velocity of 295 ft/sec
6061-T6 Aluminum	850*		
2219 Aluminum	850*	850*	
OFHC Copper	1150	1050	
301 Stainless Steel	1450	1100	1100
347 Stainless Steel	1300	1200	1300
321 Stainless Steel	1550	1550	1350
304-L Stainless Steel	1650	1650	1350
301 Cryo Stainless Steel	1550	1400	1400
A-286 Stainless Steel	1650	1600	1550
Inconel 718	1950	1850	1850
Monel K-500	1950	2000	2100
Nickel 200	2150	2100	2200

*Temperatures at which specimen would occasionally burn in air.

TABLE XV

MAXIMUM TEMPERATURES OF METAL SURFACE ON WHICH IMPACTING STREAMS OF CHLORINE PENTAFLUORIDE DO NOT RESULT IN IGNITION

Material	Approximate Nonignition Threshold Temperatures, °F	
	Liquid ClF_5 Velocity of 120 ft/sec	Liquid ClF_5 Velocity of 220 ft/sec
6061-T6 Aluminum	850*	850*
2219 Aluminum	850*	850*
OFHC Copper	1100	1050
321 Stainless Steel	1750	1400
304-L Stainless Steel	1900	1450
301 Stainless Steel	1450	1500
301 Cryo Stainless Steel	1550	1500
347 Stainless Steel	1500	1550
A-286 Stainless Steel	1550	1700
Inconel 718	1950	1950
Monel K-500	2150	2100
Nickel 200	2200	2300

*Temperatures at which specimen would occasionally burn in air.

II, D, Liquid Impact Tests on Heated Metals (cont.)

c. Comparison of the Behavior of Liquid Fluorine and Chlorine Pentafluoride with Selected Metals

The results of the liquid impact tests on the heated metal specimens are formulated into a bar chart presentation in Figure 27. A bar represents a given velocity of the liquid stream. The full width of the bar indicates the temperature region in which the metals are not significantly attacked by the impacting liquid stream and the test result is negative. The diagonal tops of the bars indicate the temperature regions in which there is a possibility of reaction as the heated metal specimen is impacted by the liquid stream; the jagged tops of the bars indicate the temperatures above which the heated metal specimens react rapidly with air. The region above the bars indicates temperature regime in which the heated metal specimens react positively with the impacting liquid streams.

One generalization that is apparent from the chart is that impacting liquid fluorine reacts with heated metal specimens at temperatures slightly lower than the temperatures at which the metal specimens react with impacting liquid chlorine pentafluoride.

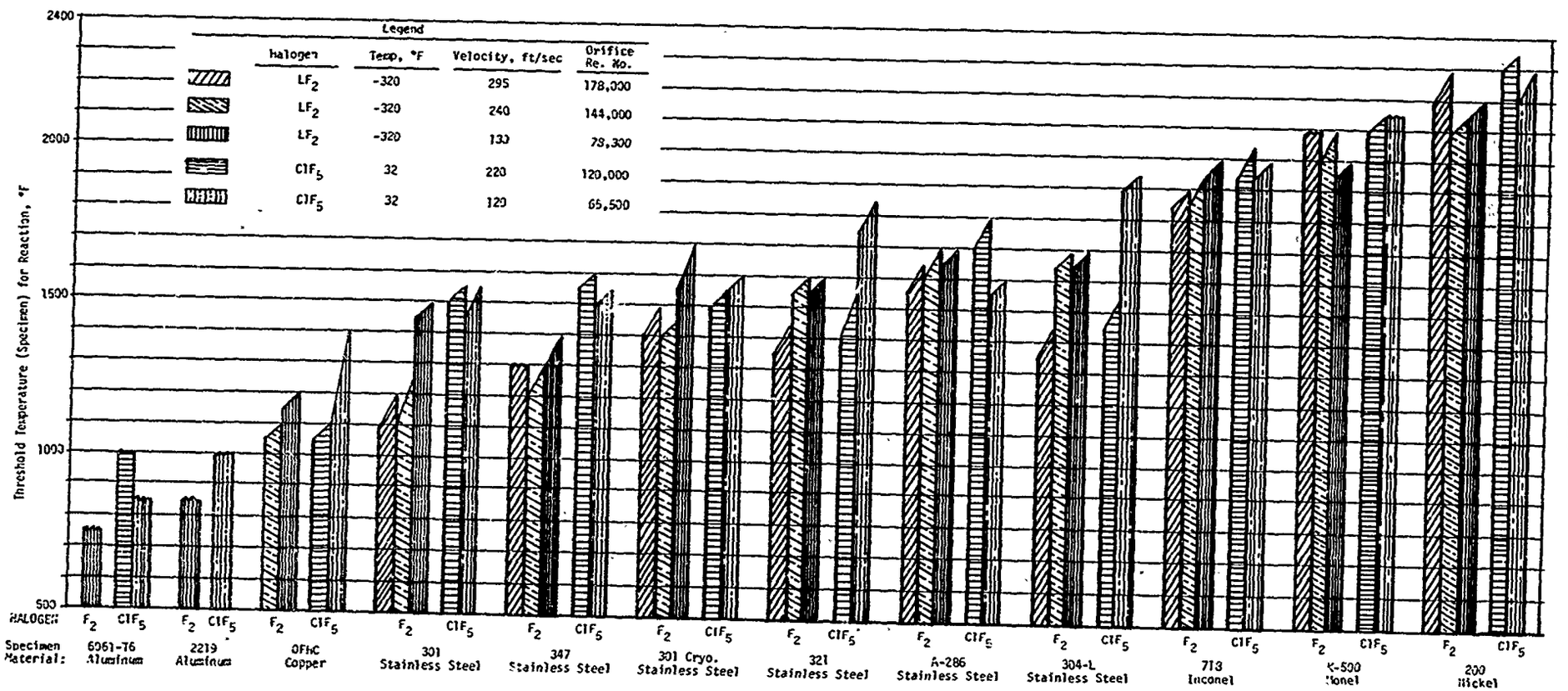


Figure 27. Threshold Temperatures for the Reaction of Liquid Halogen Oxidizers Impacting on Various Hot Metals

II, Experimental Results and Discussion (cont.)

E. FLEXING/FILM DEGRADATION TESTS

Tests were conducted to determine whether the "passive films" on various metals undergo appreciable degradation when subjected to flexing below their elastic limits in the presence of liquid/vapor ClF_5. The ten metals investigated are listed below:

301 Cryo Stainless Steel	6061-T6 Aluminum
321 Stainless Steel	OFHC Copper
347 Stainless Steel	Inconel 718
A-286 Stainless Steel	Monel K-500
2219-T37 Aluminum	Nickel 200

1. Apparatus and Procedures

The tests were conducted on specially designed specimens nominally 0.010-in. thick, except for the Nickel 200 which was nominally 0.006-in. thick. The specimen design is shown in Figure 28 and a photograph of a specimen with stainless steel buffer plates is shown in Figure 29. The flexure test fixture used in the testing is shown in an exploded view in Figure 30. The bottom of the specimens are appropriately spaced and rigidly fixed in place on the fixture by aluminum clamping blocks. The top of the specimens with attached stainless steel buffer plates, appropriate brass spacers, and stainless steel locking nuts are mounted along the horizontal reciprocating shaft which provides positive right and left specimen flexure while allowing free vertical movement of the specimens relative to the reciprocating shaft. The reciprocating shaft, fitted with a compression return spring and cam follower, passes through a brass sleeve bearing (fitted with a Teflon "O-Ring") mounted in the closure flange. It is positioned at the opposite end with a brass bearing in the shaft support member. The mounted specimens and flexure actuator are covered by an outer case which is bolted to the closure flange using a Teflon gasket as a seal. The outer case and closure flange provides a reservoir for the chlorine pentafluoride which covers the specimens to approximately their half-height. The sealed flexure fixture is mounted on a rigid plate along with the actuation motor which is fitted with a combination tachometer and cam which provides a total throw of 1 inch. The fixture and drive are adjusted so that the specimens are in a vertical (neutral) position when the cam is at midposition. This adjustment thereby gives a flexure amplitude of 1/2-in. right and left of neutral on each revolution of the cam. The cam rotates at a nominal speed of 300 RPM giving a nominal flexure frequency of five complete cycles/sec. or 10 flexures/sec.

The tests were conducted according to the following procedure. The test specimens were degreased, detergent washed, pickled, rinsed, and vacuum dried. After attachment of the specimen buffer plates, they were

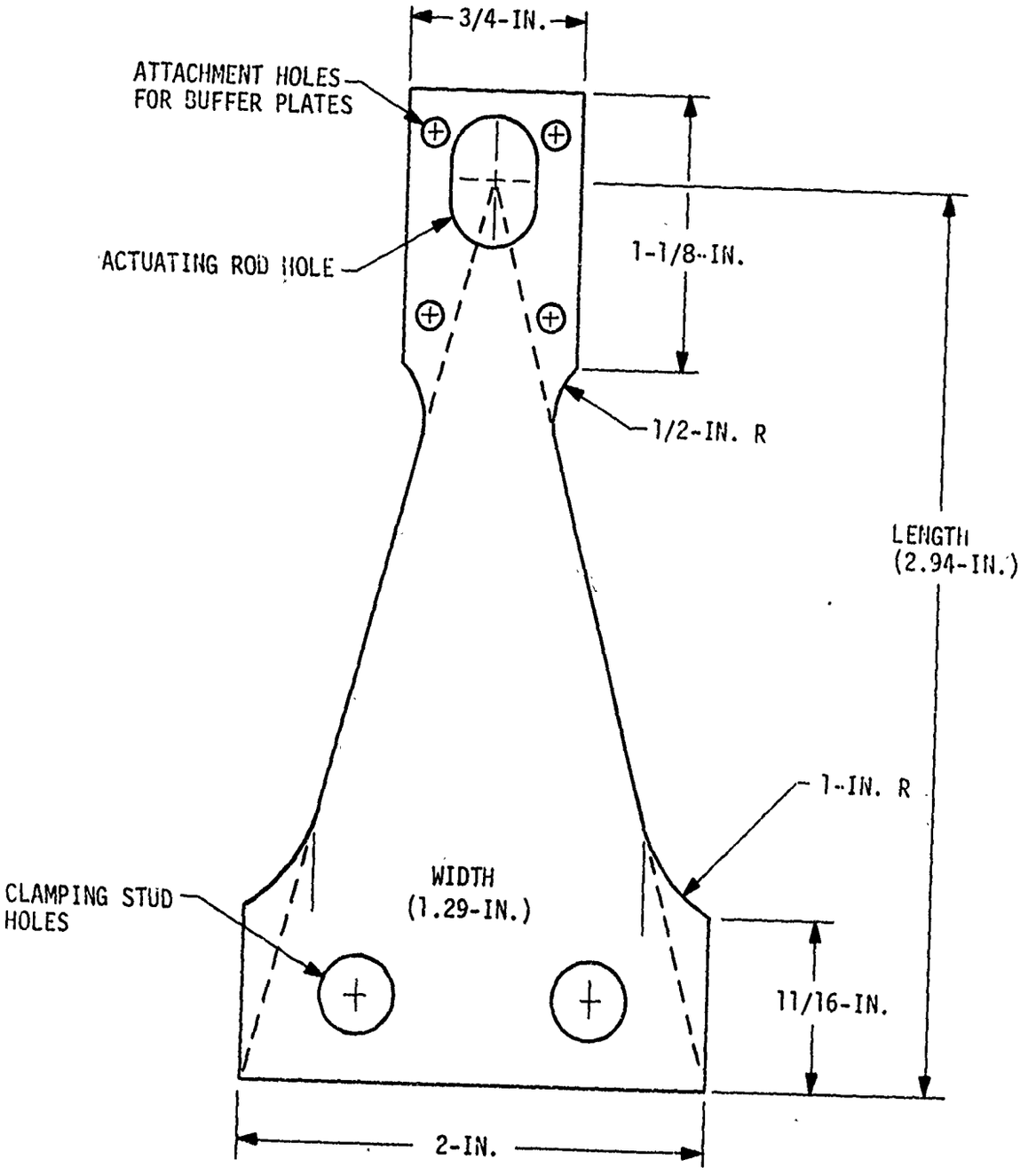


Figure 28. Flexure Test Specimen

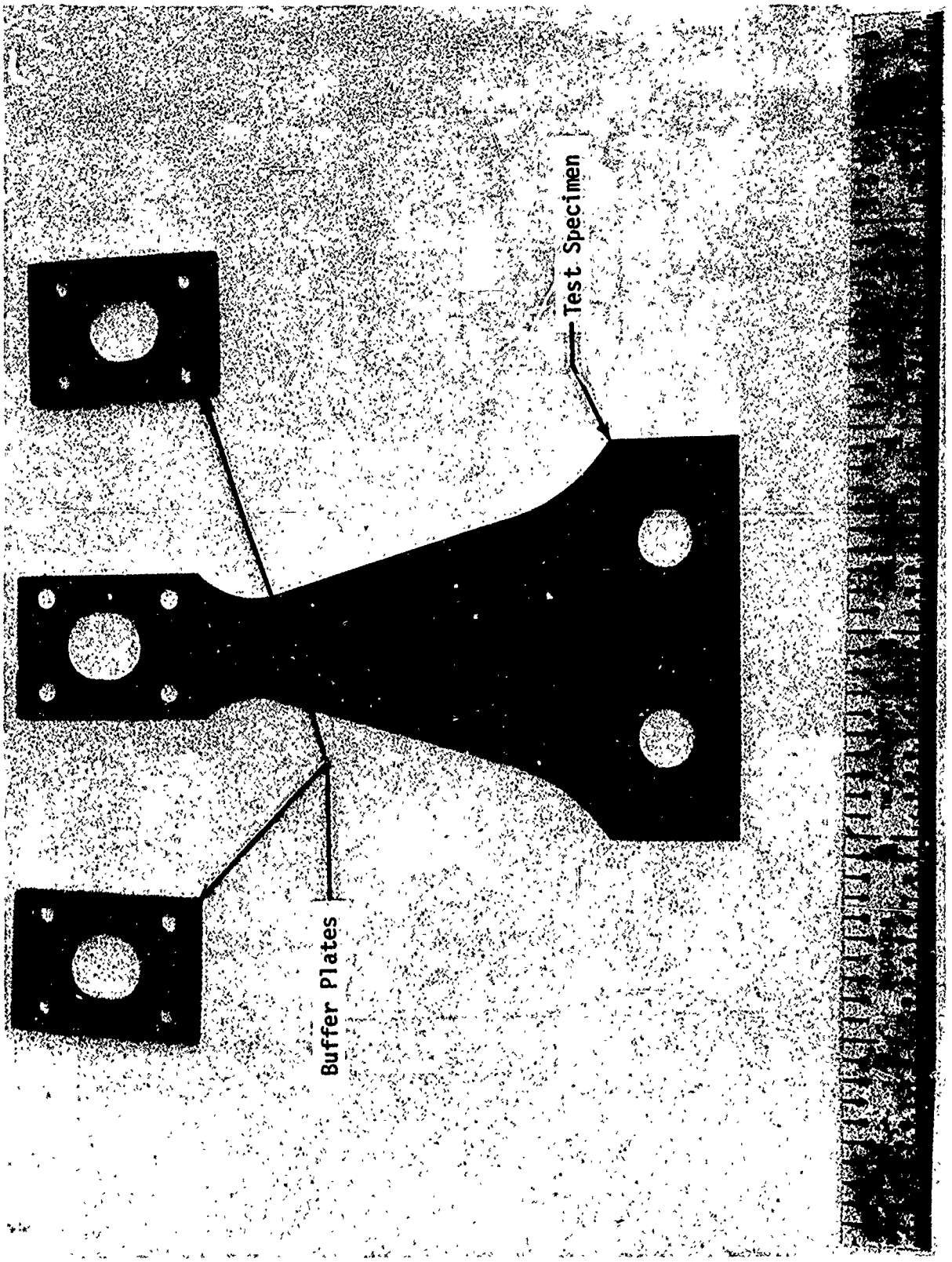


Figure 29. Flexure Test Specimen with Buffer Plates

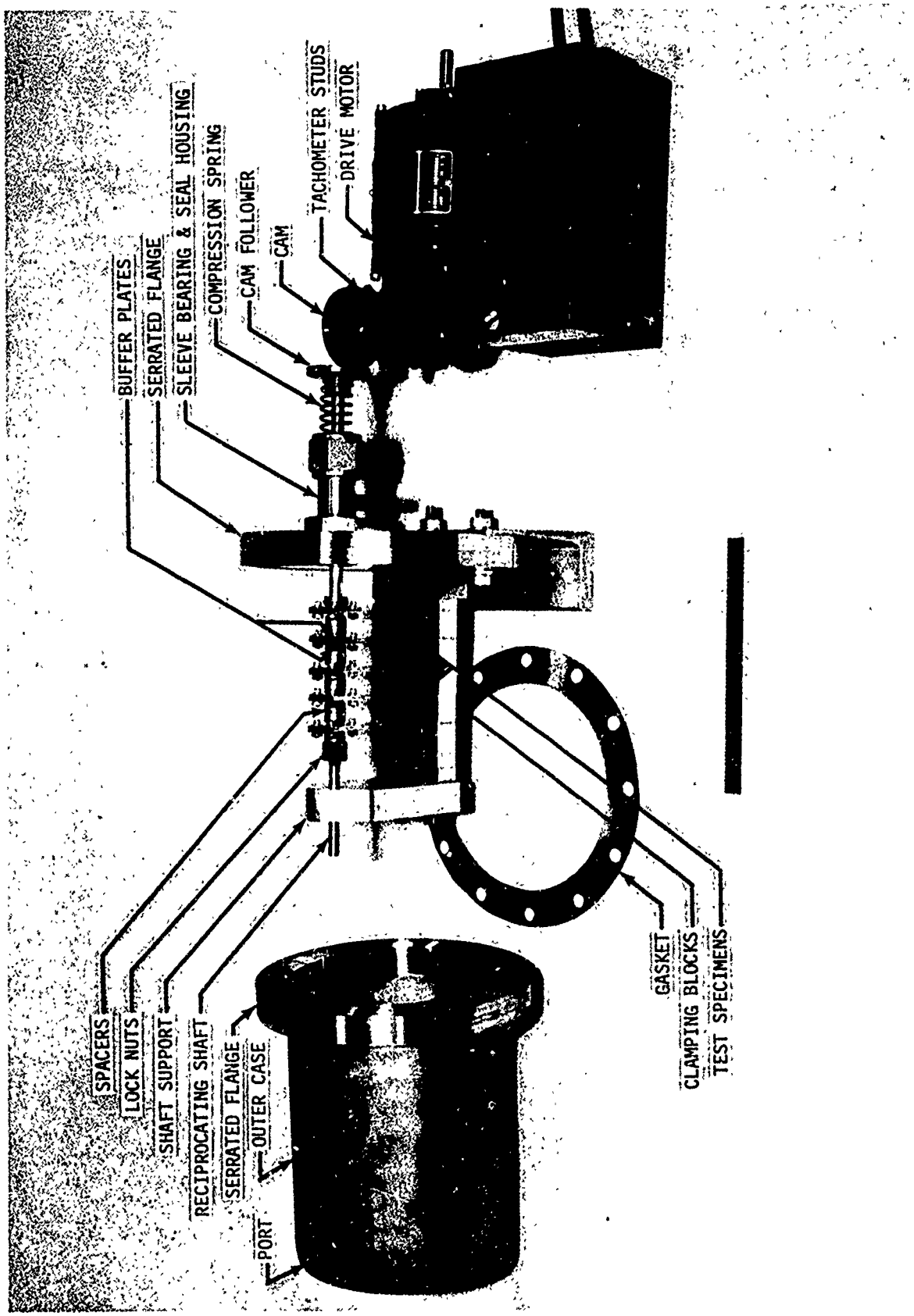


Figure 30. Flexure Test Fixture

II, E, Flexing/Film Degradation Test (cont.)

weighed to the nearest 0.1 mg and assembled into the test fixture as shown in Figure 30. The assembled fixture was then mounted in a stirred trichlorotrifluoroethane bath, connected to a chlorine pentafluoride feed system, evacuated, and filled with chlorine pentafluoride vapor for 15 min. to effect a passivation. The bath was cooled to approximately 0°F and the fixture loaded with sufficient chlorine pentafluoride (~800 cm^3) to cover the specimens to their mid-height. The bath was allowed to warm to approximately 10-20°F (chlorine pentafluoride vapor pressure of 1 to 4 psig) and maintained at that temperature for the duration of the test. The fixture was actuated for 100,000 cycles, then vented and dried. It was disassembled in a glove box under gaseous nitrogen. The test specimens, with attached buffer plates, were reweighed to the nearest 0.1 mg. The test specimens were then replaced in the test fixture, the filling and testing procedure being repeated for another 100,000 cycles. Upon termination of the test, the fixture was vented and dried as before and the specimens reweighed (both with and without buffer plates) to the nearest 0.1 mg. Finally, the specimens were stored under dry nitrogen.

Similar tests were conducted in the test fixture in chlorine pentafluoride without specimen flexure, but for an equivalent duration, and in an "inert" fluid (trichlorotrifluorethane) with specimen flexure for 200,000 cycles. These tests served as controls. All specimens were visually inspected at 40X magnification.

2. Experimental Results

The data resulting from all these tests are summarized in Table XVI. The primary conclusion reached from these tests is that the "passive films" on the materials tested are not degraded by flexure below the elastic limit as evidenced by both minimal changes in specimen weight and appearance. The control tests involving the static exposure of the specimens to liquid/vapor chlorine pentafluoride show that all the metals tested gain a slight amount of weight (average gain = 0.52 mg, σ = 0.19 mg, and only OFHC copper deviates from the average by more than 1.2σ). This small gain in weight appears mostly readily explained by the formation of "passive films". The control tests involving flexure of the specimens (unpassivated) in trichlorotrifluoroethane similarly showed slight weight gain of about the same average magnitude as in static chlorine pentafluoride, but of greater variability between specimens of different metals (average gain = 0.69 mg, σ = 0.51 mg, maximum deviation from the average = 1.8 σ). Although the cause of weight gain was not established, it appears most easily reconciled by a postulated surface absorption of trichlorotrifluoroethane. Comparing the results of the flexure tests in chlorine pentafluoride with the static control tests in chlorine pentafluoride, shows that the average weight gains of the specimens exposed to flexure in chlorine pentafluoride are about

TABLE XVI

EFFECT OF FLEXING ON "PASSIVE FILMS" ON METALS IN THE PRESENCE OF LIQUID CHLORINE PENTAFLUORIDE

Material	Static Exposure in Liquid/Vapor Chlorine Pentafluoride for 640 min: Initial Sample Wt, g	Static Exposure: Wt Change After Exposure, mg	Static Exposure: Observations	Flexing Exposure in Liquid/Vapor Freon 113 for 200,000 Cycles: Initial Sample Wt, g	Flexing Freon 113: Wt Change After Exposure, mg	Flexing Freon 113: Observations	Flexing Exposure in Liquid/Vapor Chlorine Pentafluoride: Initial Sample Wt, g	Wt Change After Exposure, mg: First 100,000 Cycles	Second 100,000 Cycles	Total	Observations
301 Cryo Stainless Steel	8.0614	+0.6	No change	7.6807	+1.4	No change	5.7478	+1.5	+2.3	+3.8	No change
321 Stainless Steel	5.5310	+0.6	No change	5.4896	+1.6	No change	5.5011	+0.5	+2.7	+3.2	No change
347 Stainless Steel	5.5443	+0.3	No change	5.4549	+0.1	No change	5.4534	+1.9	-0.9	+1.0	No change
A-286 Stainless Steel	7.0086	+0.4	No change	6.9719	+0.1	No change	6.8566	+2.1	-1.7	+0.4	No change
2219-T37 Aluminum	1.9830	+0.5	No change	1.8260	+0.5	No change	1.8388	+1.6	+3.2	+4.8	No change
6061-T6 Aluminum	1.8328	+0.4	No change	1.8035	+0.2	No change	1.8153	+1.5	+0.6	+2.1	No change
OFHC Copper	6.1765	+1.3	No change	5.7610	+0.4	Possible roughening	5.8114	+1.3	+0.2	+1.5	Possible roughening
Inconel 718	5.8902	+0.4	No change	5.7427	+0.6	No change	5.7972	+0.2	+2.2	+2.4	No change
Monel K-500	5.7920	+0.4	No change	5.4377	+0.4	No change	5.1493	+2.5	-0.7	+1.8	Very slight roughening
Nickel 200	3.6840	+0.3	Very slight roughening	3.6739	+1.6	No change	3.4638	+2.2	+2.2	+4.4	Slight roughening
Average		+0.52			+0.69			+1.53	+1.01	+2.54	
Standard Deviation, σ		0.19			0.51			0.53	1.51	1.21	

II, E, Flexing/Film Degradation Test (cont.)

three and five times greater after 100,000 and 200,000 flexures, respectively, than the gains found during static exposure. This indicates that the "passive film" buildup in chlorine pentafluoride is generally enhanced by flexure and dependent upon cycle number, but different metals exhibit considerable variability in these tendencies. Although an enhancement of surface reactions by flexure is indicated, the absolute effects on the specimens are so small that design constraints to counter this particular effect appear meaningless.

Photographs of the surface of representative metal specimens are shown in Figure 31. At 50 fold magnification, the 6061-T6 aluminum surfaces appear similar whether flexed in trichlorotrifluoroethane or chlorine pentafluoride, or exposed for a comparable period of time in a static mode in chlorine pentafluoride. At 50 fold magnification, the A-286 surface exhibits some apparent attack by the chlorine pentafluoride as compared to the surface exposed to trichlorotrifluoroethane. However, the surfaces of A-286 exposed to chlorine pentafluoride in a static manner and to the chlorine pentafluoride in the flexing mode are apparently very similar.

F. VIBRATION/FILM DEGRADATION TESTS

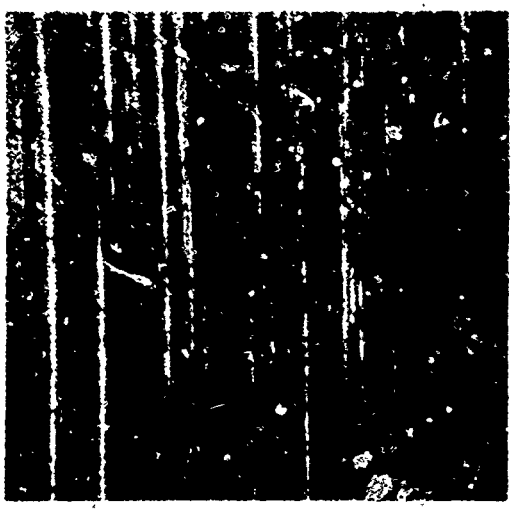
Tests were conducted to determine whether the "passive film" on the various metals undergoes appreciable degradation when subjected to ultrasonic vibration/cavitation in the presence of liquid fluorine and chlorine pentafluoride. The metals investigated are listed below.

301 Cryo Stainless Steel	6061-T6 Aluminum
304-L Stainless Steel	OFHC Copper
321 Stainless Steel	Inconel 718
347 Stainless Steel	Monel K-500
A-286 Stainless Steel	Nickel 200

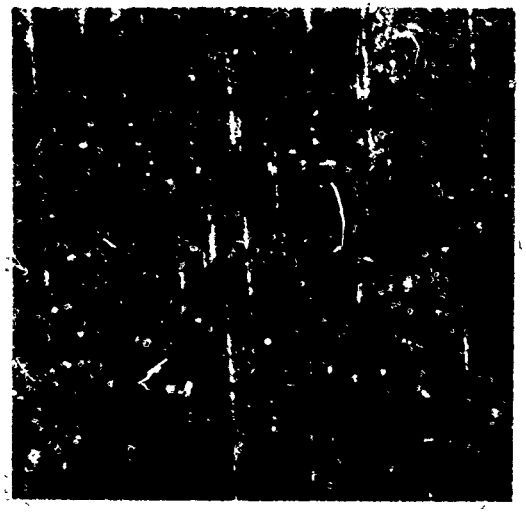
1. Apparatus and Procedures

The tests were conducted in the following manner. A stainless steel cylinder was fabricated with four vertical rows of welded-on B-nut cap assemblies. The metal specimens were mounted by strapping to AN plugs which sealed the B-nut cap assemblies (Figure 32). In this manner, eighteen metal specimens could be tested concurrently. The assembled stainless steel cylinder was immersed in an ultrasonic cleaning bath* which was driven with 400 watts at a frequency of 62.5 k Hz. A liquid nitrogen bath was used for the liquid fluorine tests and trichlorotrifluoroethane for the chlorine pentafluoride tests. The specimens were passivated in fluorine vapor or chlorine pentafluoride vapor overnight prior to exposure to the liquid. Prior to conducting the vibration tests, separate control tests were made wherein the metal specimens were immersed in static liquid oxidizer for 5-1/2 hours, then

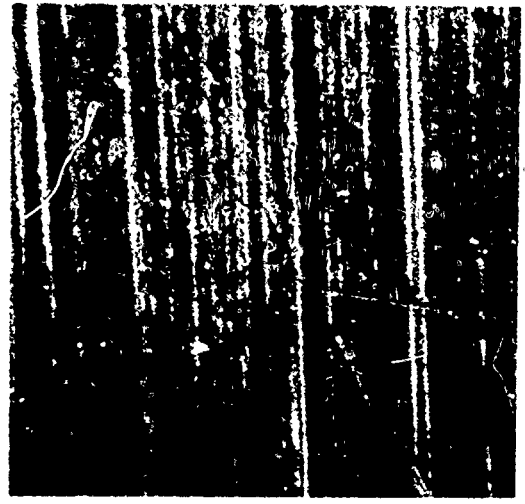
*L and R Ultrasonic Model 540, L and R Manufacturing Company, Kearny, New Jersey



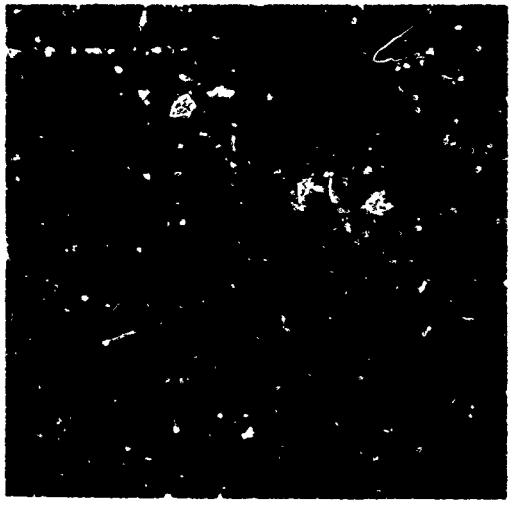
6061-T6 Al
Flexed in
Trichlorotrifluoroethane



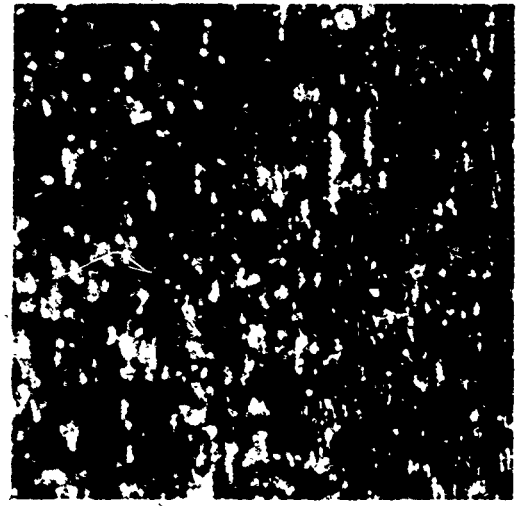
6061 T6 Al
Static Exposure in
Chlorine Pentafluoride



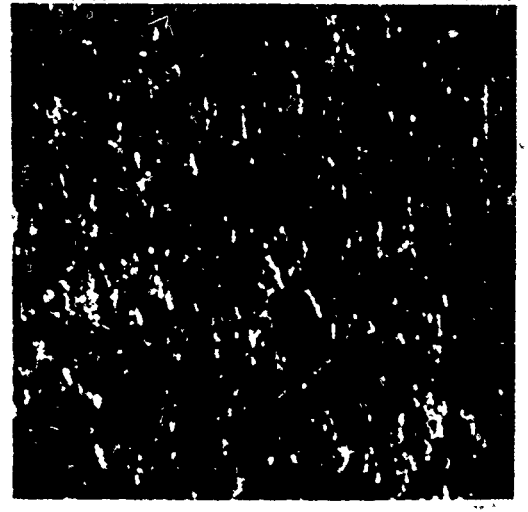
6061 T6 Al
Flexed in
Chlorine Pentafluoride



A-286 Flexed in
Trichlorotrifluoroethane



A-286 Static
Exposure in
Chlorine Pentafluoride



A-286 Flexed in
Chlorine Pentafluoride

Figure 31. Photographs of Metal Specimens Flexed in Chlorine Pentafluoride Taken at a 50-fold Magnification

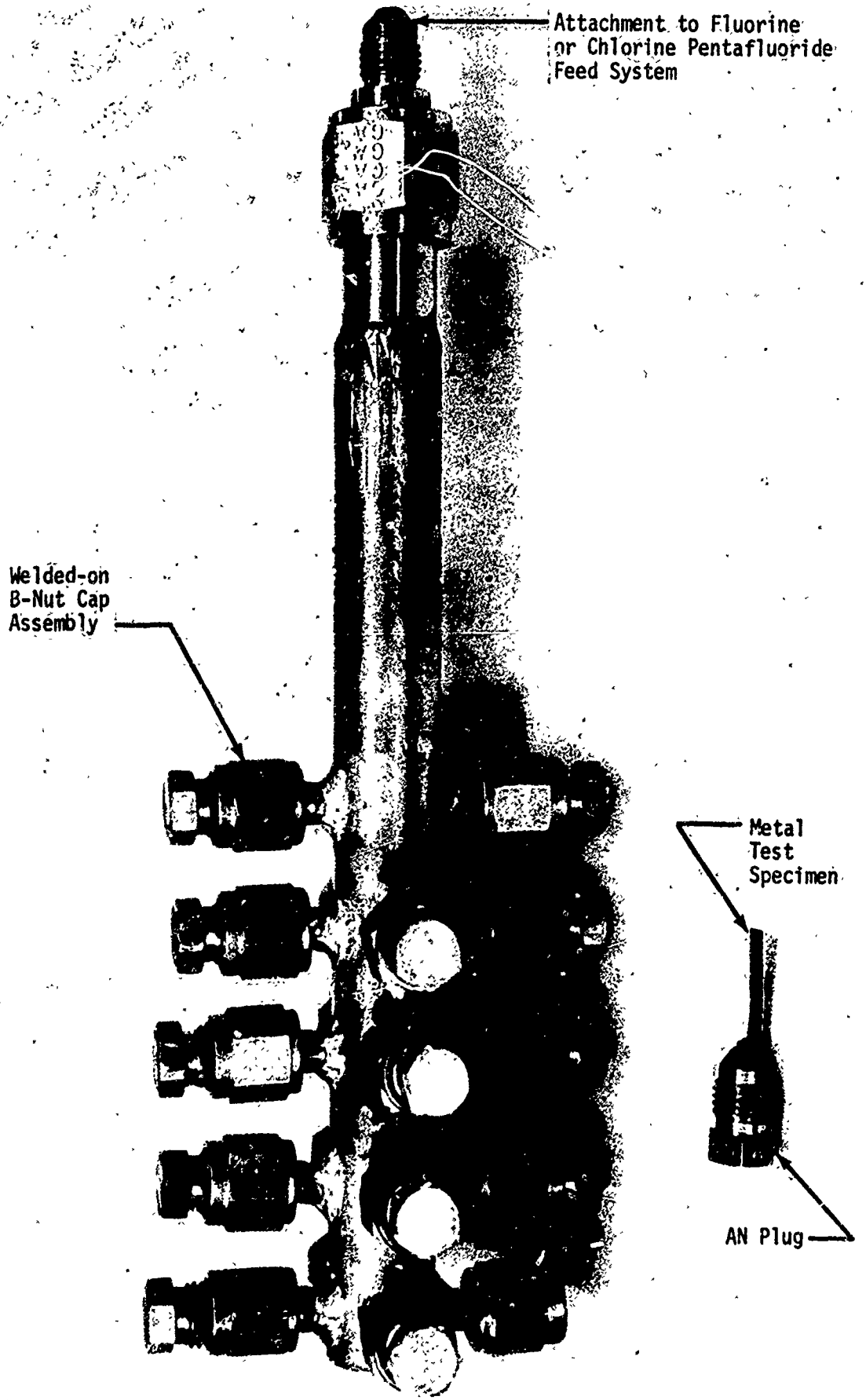


Figure 32. Metal Specimen Fixture Used for the Ultrasonic Vibration Tests with Liquid Fluorine and Chlorine Pentafluoride

II, F, Vibration/Film Degradation Tests (cont.)

weighed to determine whether weight changes occurred. New metal specimens were installed in the test fixture. The fixture with metal specimens in place was passivated overnight and then subjected to the vibration test. The vibration test cycle consisted of one-half hour of operation, one-half hour of shutdown, and this was continued for six cycles. Upon completion of the vibration test cycle, the specimens were weighed and examined visually.

2. Experimental Results

a. Liquid Fluorine

The results of the static exposure and vibration exposure tests with liquid fluorine are presented in Table XVII. In addition, the results of a second control test obtained with trichlorotrifluoroethane under the vibrating conditions are included in the table for comparison. The specimens were examined at 60X magnification to determine if significant attack had occurred. Weight changes of $\pm$ 0.1 mg are considered to be the approximate sensitivity limits of the weighing procedure.

The significant items to be noted from the data on the control tests given in Table XVII are: (1) ultrasonic vibration of the metal specimens in the "inert," trichlorotrifluoroethane, results in no significant weight change (average weight change is +0.036 mg, σ = 0.073 mg, and the maximum weight change lies within 2σ of the average) and only slight superficial visual effects on the surfaces, (2) static exposure of the metals, except for 347 stainless steel, to liquid fluorine results in no significant weight change (average weight change is 0.000 mg, σ = 0.056 mg, and the maximum weight change, excluding 347 S.S., is within 2σ of the average), and (3) 347 stainless steel appears to suffer very slight attack by static fluorine as evidenced by a weight change that differs from the average of the other metals by 3.6σ. Using the above baseline statistical information, the following conclusions are reached regarding the effect of ultrasonic vibration on metals in the presence of liquid fluorine.

(1) None of the metals tested exhibit any appreciable change in weight or visual appearance other than staining or discoloration (average weight change = -0.011 mg, σ = 0.060 mg, maximum deviation = 3.2σ), and

(2) The weight change observed with Inconel 718 is very slight, but is statistically significant in view of the facts that the weight change of the Inconel specimen was different from the average weight change of all materials tested by 3.2σ and the observed weight change was twice the estimated sensitivity of the weighing procedure.

In related tests at Douglas Aircraft Company at much higher energy inputs (Reference 7), the aluminum casting alloy, A-356-T6, was exposed to liquid fluorine (0.02% vol HF) at -320°F and subjected to vibration at 27.8 k Hz in an

TABLE XVII

EFFECT OF ULTRASONIC VIBRATION ON "PASSIVE FILMS" ON VARIOUS METALS IN THE PRESENCE OF LIQUID FLUORINE

Metal	Static Exposure in Liquid Fluorine			Vibration Exposure in Liquid Fluorine			Vibration Exposure in Trichlorotrifluoroethane		
	Initial Sample Wt., mg	Wt Chnge After Exposure mg	Observations	Initial Sample Wt., mg	Wt Chnge After Exposure mg	Observations	Initial Sample Wt., mg	Wt Chnge After Exposure mg	Observations
301 Cryo Stainless Steel	249.2	-0.1	No Change	253.9	0.0	No Change	-	-	-
304-L Stainless Steel	129.6	-0.1	Staining	123.8	0.0	No Change	-	-	-
304-L Stainless Steel	125.1	0.0	Staining	141.1	-0.1	No Change	-	-	-
321 Stainless Steel	130.9	0.0	No Change	133.7	0.0	No Change	-	-	-
347 Stainless Steel	141.8	+0.2	Staining	143.9	0.0	No Change	130.2	+0.1	Staining
347 Stainless Steel	152.3	+0.1	Staining	135.1	0.0	No Change	134.6	+0.1	Staining
A-286 Stainless Steel	181.9	0.0	Staining	189.0	0.0	No Change	184.0	+0.1	No Change
A-286 Stainless Steel	189.0	0.0	Staining	166.3	0.0	No Change	176.9	+0.1	No Change
6061-T6 Aluminum	47.9	0.0	No Change	48.9	0.0	No Change	44.0	-0.1	No Change
6061-T6 Aluminum	50.2	+0.1	No Change	53.2	+0.1	No Change	43.9	+0.1	No Change
OFHC Copper	143.7	0.0	Discolored	151.2	0.0	Discolored	153.1	0.0	Discolored
OFHC Copper	153.9	-0.1	Discolored	164.5	-0.1	Discolored	154.0	0.0	Discolored
Inconel 718	121.3	0.0	No Change	123.1	+0.1	Staining	137.4	0.0	No Change
Inconel 718	128.1	+0.1	No Change	126.4	-0.2	Staining	120.9	-0.1	No Change
Monel K-500	128.5	0.0	Staining	144.9	+0.1	No Change	158.5	+0.1	No Change
Monel K-500	132.6	0.0	Staining	142.3	+0.1	No Change	149.8	-0.1	No Change
Nickel 200	150.5	-0.1	No Change	147.0	-0.1	Staining	127.1	+0.1	No Change
Nickel 200	155.0	-0.1	No Change	127.8	-0.1	Staining	148.9	+0.1	No Change
Average		0.000			-0.011			+0.036	
Standard Deviation, σ		0.056			0.060			0.073	

II, F, Vibration/Film Degradation Tests (cont.)

ultrasonic mechanical stepped horn apparatus in repeated cycles of 1/2 hr on and 3 hr off. Similar tests were conducted with water and liquid nitrogen for comparison. The average sample weight changes in water, LN_2, and LF_2 were 0.01, 0.02, and 0.04 mg/cm^2-min, respectively. Average corrosion pit depths in LN_2 and LF_2 were 18 and 40 microns, respectively.

The ultrasonic vibration involved in the Douglas Aircraft tests was sufficiently powerful to disrupt the surface very significantly as evidenced by weight loss and pitting when the specimen was exposed to the inert liquid nitrogen. The fact that the presence of liquid fluorine in that same highly disruptive environment does not lead to gross fluorine/metal reaction corroborates the present findings and indicates that the "passive film" or loss of such is not an item of great importance in liquid fluorine/metal compatibility.

b. Liquid Chlorine Pentafluoride

The results of the static exposure and vibration exposure tests with liquid chlorine pentafluoride are presented in Table XVIII. In addition, the results of a second control test obtained with trichlorotrifluoroethane under the vibrating conditions are included in the table for comparison. The specimens were examined at 60X magnification to determine if significant attack had occurred. Weight changes of $\pm$ 0.1 mg are considered to be the approximate sensitivity limits of the weighing procedure.

The significant items to be noted from the data on the control tests given in Table XVIII are: (1) ultrasonic vibration of the metal specimens in the "inert", trichlorotrifluoroethane, results in no significicant weight change (average weight change is +0.036 mg, σ = 0.073 mg and the maximum weight change lies within 2σ of the average) and only slight superficial visual effects on the surfaces, (2) static exposure of the metals, except for 301 cryoformed stainless steel, to liquid chlorine pentafluoride results in no significant weight change (average weight change is -0.006 mg, σ = 0.047 mg and the maximum weight change, excluding 301 S.S., is within $\sim$2σ of the average), and (3) 301 cryoformed stainless steel appears to suffer slight attack by static chlorine pentafluoride as evidenced by weight changes that differ positively and negatively from the average of the other metals by +4 and -10σ, respectively. Using the above baseline statistical information, the following conclusions are reached regarding the effect of ultrasonic vibration on metals in the presence of liquid chlorine pentafluoride:

(1) 301 cryoformed stainless steel, 6061-T6 aluminum, and Nickel 200 are uneffected except for possible staining (average weight change = -0.033 mg, σ = 0.067 mg, maximum deviation = 2σ),

(2) 347 and A-286 stainless steels and OHHC copper suffer slight weight losses and staining or discoloration (average weight change = -0.250 mg, σ = 0.050 mg, maximum deviation = 1σ),

TABLE XVIII

EFFECT OF ULTRASONIC VIBRATION ON "PASSIVE FILMS" ON METALS IN THE PRESENCE OF LIQUID CHLORINE PENTAFLUORIDE

Metallic Material	Static Exposure in Liquid Chlorine Pentafluoride			Vibration Exposure in Liquid Chlorine Pentafluoride			Vibration Exposure in Trichlorotrifluoroethane		
	Initial Sample Weight, mg	Weight Change After Exposure, mg	Observations	Initial Sample Weight, mg	Weight Change After Exposure, mg	Observations	Initial Sample Weight, mg	Weight Change After Exposure, mg	Observations
321 SS	131.0	0.0	No Change	127.3	-0.2	Discolored	-	-	-
321 SS	131.8	0.0	No Change	127.4	-1.0	Discolored	-	-	-
347 SS	135.7	+0.1	No Change	131.8	-0.2	Staining	130.2	+0.1	Staining
347 SS	138.0	0.0	No Change	138.1	-0.3	Staining	134.6	+0.1	Staining
A-286	183.6	0.0	No Change	172.4	-0.2	Staining	184.0	+0.1	No Change
A-286	179.1	-0.1	No Change	153.8	-0.3	Staining	176.9	+0.1	No Change
6061 Al	45.9	-0.1	No Change	47.4	-0.1	No Change	44.0	-0.1	No Change
6061 Al	45.3	0.0	No Change	45.2	-0.1	No Change	43.9	+0.1	No Change
OFHC Copper	149.8	-0.1	Discolored	181.2	-0.3	Discolored	153.1	0.0	Discolored
OFHC Copper	163.7	0.0	Discolored	170.2	-0.2	Discolored	154.0	0.0	Discolored
Monel K-500	133.5	0.0	No Change	144.0	-0.5	Staining	158.5	+0.1	No Change
Monel K-500	142.5	-0.1	No Change	141.2	-0.2	Staining	149.8	-0.1	No Change
Inconel-718	149.0	0.0	No Change	129.7	-0.5	Staining	137.4	0.0	No Change
Inconel-718	133.7	0.0	No Change	144.4	-0.4	Staining	120.9	-0.1	No Change
Nickel 200	146.7	+0.1	No Change	142.4	0.0	Staining	127.1	+0.1	No Change
Nickel 200	128.1	+0.1	No Change	131.0	+0.1	Staining	148.9	+0.1	No Change
301 cryoformed	259.7	-0.5	No Change	238.0	-0.1	No Change	-	-	-
301 cryoformed	258.8	+0.2	No Change	209.0	0.0	No Change	-	-	-

II, F, Vibration/Film Degradation Tests (cont.)

(3) Monel K-500 experiences a small, quite variable weight loss and staining (average weight change = -0.35 $\pm$ 0.15 mg),

(4) Inconel 718 experiences a small, quite reproducible weight loss and staining (average weight change = -0.45 $\pm$ 0.05 mg), and

(5) 321 stainless steel suffers a modest weight loss of relatively poor reproducibility and discoloration (average weight change = 0.6 $\pm$ 0.4 mg).

SECTION III

CONCLUSIONS AND RECOMMENDATION

A. CONCLUSIONS

During the course of the experimental program, strict cleaning and passivation procedures of the metals were utilized and, based on the experimental program, the following conclusion may be drawn.

1. In hot, flowing gaseous fluorine at sonic conditions at 100 psig, or gaseous chlorine pentafluoride at 50 psig, the maximum temperatures (°F) which the selected metals can tolerate without significant attack occurring are as follows:

	Fluorine	Chlorine Pentafluoride
Nickel 200	1755	1790
Inconel 718	1675	1555
Monel K-500	1520	1800
A-286	1350	1250
347 SS	1315	1200
304L	1140	1110
321 SS	1115	1170
301 SS (Cryoformed)	-	1125
6061T6 Aluminum	1000	1150
OFHC Copper	855	840

The data are based on exposure periods of seconds and, as the gaseous velocity decreases, the temperature values increase only slightly. Nickel 200 is the most compatible material for use with fluorine and chlorine pentafluoride.

2. Generally, the metals can withstand adiabatic compression in the presence of gaseous fluorine to the same resultant temperature limits that can be tolerated in the sonic flow conditions. The only apparent exceptions are Monel K-500 and A-286 which can tolerate resultant temperature limits of 1275 and 1290°F, respectively. Nickel 200 is the preferred material under these conditions. Because the self-heating during adiabatic compression in chlorine pentafluoride is much less pronounced than with fluorine at comparable compression ratios, only two materials were found to react at compression ratios up to 192, 301 Cryoformed SS and 304L SS at resultant temperatures as low as 523°F. The final density value of the chlorine pentafluoride is two orders of magnitude greater in the adiabatic compression tests than the density value in the gas-flow tests and can account for the dramatic lowering of the tolerable temperature limits.

3. Although no gross failures were noted, some minor attack on metal specimens occurred as they were subjected to a liquid shock wave (water hammer effect). With liquid fluorine, 304L SS and Inconel 718 were unchanged, Monel K-500 discolored, and 321 SS, A-286, 6061-Al, OFHC Copper, and Nickel 200

A, Conclusions (cont.)

underwent slight attack. With liquid chlorine pentafluoride, 6061-Al and Monel K-500 were unchanged, 347 SS discolored, Cryoformed 301 SS, 304L SS, A-286, OFHC Copper, Inconel 718, and Nickel 200 underwent slight attack. The slight attack which was noted occurred on the sharp edges of the test specimens and not on the major surfaces, and at pressure spikes greater than 8500 psig.

4. The maximum temperatures (°F) to which the metals may be heated while impacted by liquid fluorine or chlorine pentafluoride without significant reaction occurring are as follows:

	Fluorine	Chlorine Pentafluoride
Nickel 200	2100	2000
Monel K-500	1950	2100
Inconel 718	1850	1950
304L SS	1350	1450
A-286	1550	1550
321 SS	1350	1400
301 SS (Cryo Formed)	1400	1500
301 SS	1100	1450
347 SS	1200	1500
OFHC Copper	1050	1050
2219 Aluminum	850*	950*
6061T6 Aluminum	750*	850*

*Reaction with air becomes appreciable at these temperatures.

Nickel 200, Monel K-500, and Inconel 718 are the materials most compatible with the fluorine and chlorine pentafluoride under these conditions.

5. The flexing of metal specimens in liquid chlorine pentafluoride did not significantly disrupt the passivation film.

6. Ultrasonic vibration of the metal specimens in liquid fluorine or chlorine pentafluoride produces minimal changes on the metal surface.

B. RECOMMENDATION

The current investigation was limited to gas pressures of 100 psig in flow tests and 3000 psig peak pressure in the adiabatic compression tests. There is indication in the data that the density of the gaseous phase does affect the temperature limits which the metals can tolerate. In order to complete the compatibility evaluation of fluorine and chlorine pentafluoride with the metals, it is recommended that metal compatibility at high temperatures, with gaseous fluorine and chlorine pentafluoride at pressures up to 5000 psi, be investigated.

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APPENDIX A

A LITERATURE SEARCH AND REVIEW OF THE DYNAMIC COMPATIBILITY OF FLUORINE AND CHLORINE PENTAFLUORIDE WITH METALS

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SECTION I

FLUORINE DYNAMIC COMPATIBILITY

A. FLOW-TYPE TESTS

1. Dynamic Corrosion Rates of Refractory Metals in Flowing High Temperature Fluorine-Argon Gas Mixtures (References 1, 2, and 3)

IIT Research Institute has conducted an extensive investigation of the dynamic corrosion rates of various refractory metals in flowing, high-temperature, fluorine-argon gas mixtures. The test facility was capable of producing variable gas flow conditions and maintaining specimens at temperatures up to 5800°F. A schematic diagram of the test chamber is shown in Figure 1.

Test samples 0.5 x 0.5 x 0.125-in. thick are heated by self-inductance in a thick-walled stainless steel chamber using a 2.5 or 5 kW induction unit. Thus, the maximum temperature limit is defined by the material under test and the input power. Temperature measurements are made optically through a top sight glass which permits visual observation of specimen deterioration during exposure. Argon and the corrosive gases are metered in precision flow meters and premixed before entering the nozzle assembly. Fluorine is passed through a sodium bifluoride trap to remove residual hydrogen fluoride prior to entering the fluorine flow meter. The nozzle assembly consists of thick-walled stainless tubing swaged to obtain a 0.036-in. exit nozzle. During testing, the nozzle exit is maintained at a fixed distance (usually 1 in.) from the test specimen. Test specimens are supported in the induction coil by a 0.125-in. tungsten rod, which is split to minimize the contact area and to stabilize the test specimen. Exiting reaction products are passed through an activated charcoal absorption column prior to exhausting through a laboratory hood.

The following conditions for corrosion tests were constant during most of the tests: (1) nozzle distance, 1 in.; (2) total gas flow rate, 10 ft^3/hr; (3) exposure time, 5 min; and (4) nozzle exit velocity, 400 ft/sec. Deviations were made only in specific cases where the effects of variations were examined. The test procedure was constant for the various corrosion tests. Test samples

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Ref. 1 "Protective Coatings for Refractory Metal in Rocket Engines", IIT Research Institute, Chicago, Illinois, Contract NAS7-431, Interim Report IITR-B6058-13, January 15, 1967.

Ref. 2 "Protective Coatings for Refractory Metal in Rocket Engines", IIT Research Institute, Chicago, Illinois, Contract NAS7-431, Interim Report IITR-B6058-26, March 15, 1968.

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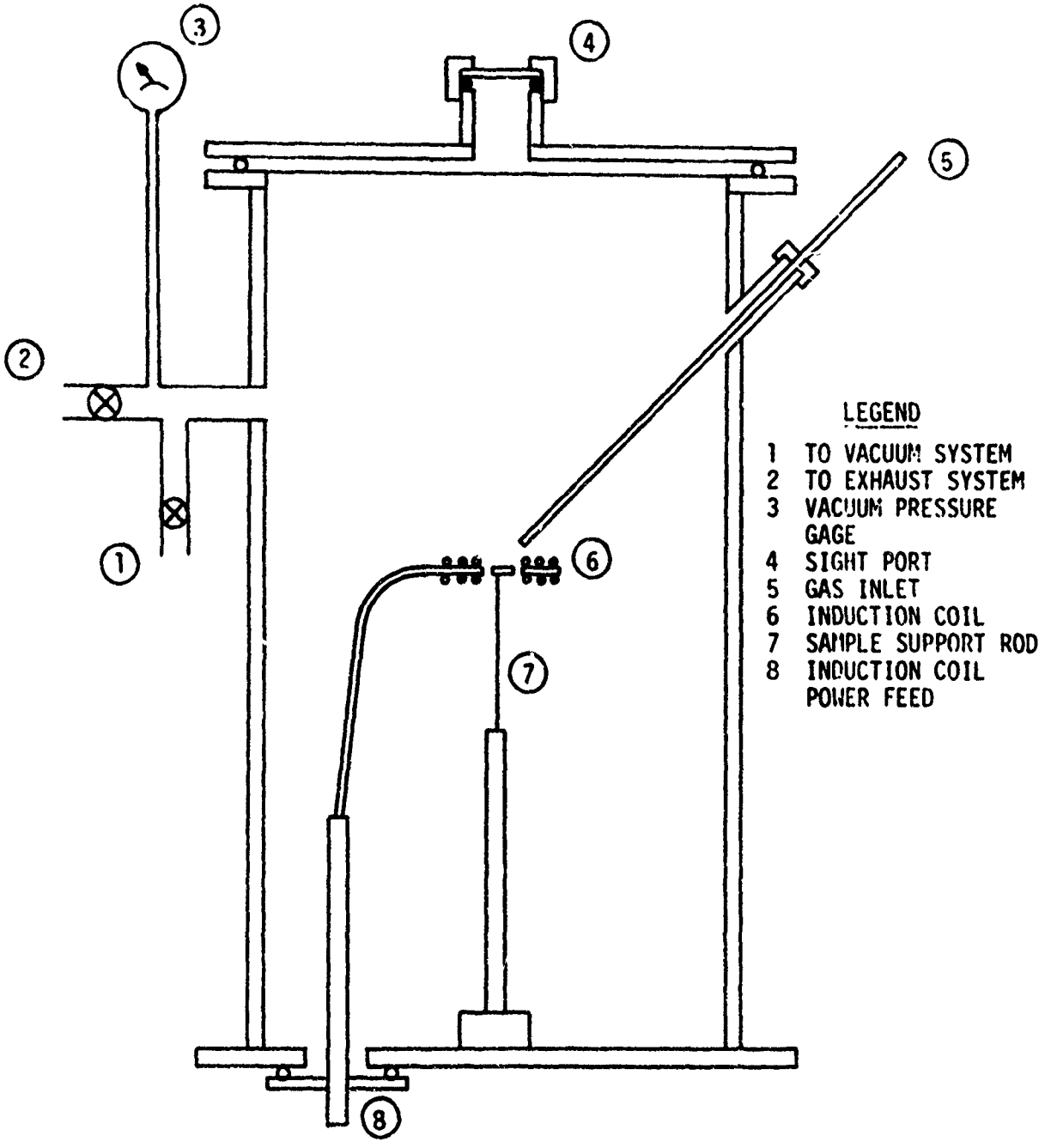

Figure 1. Schematic Diagram of Refractory Metals/Fluorine Reaction Chamber

1, A, Flow-Type Tests (cont.)

were heated to the exposure temperature with only the argon flowing. After stabilization of the sample temperature, the fluorine was introduced. Normally, this did not result in significant variation of the sample temperature as argon represented the major portion of the gas stream. In some cases, it was necessary to reduce the exposure time to 3 min or increase exposure time up to 10 min to obtain total weight losses of an amount required for recession rate calculations.

The surface recession rates were calculated from weight loss of the 0.5 x 0.5 x 0.125-in. (nominal) thick test samples, assuming a linear time dependence of the weight loss. The effective surface area was obtained using one of the 0.5 x 0.5-in. surfaces (location of direct gas impingement) and the four 0.5 x 0.125-in. edges of the test sample. This is consistent with the observed location of attack on all test samples. Attack of the corrodent gases was usually not uniform over the test sample; material removed was maximum at the center of the 0.5 x 0.5-in. surface where direct gas impingement occurred. Thus, test samples tended to have a concave top surface after exposure. This concave top surface is described as "cratering".

The effect or nozzle distance on the surface recession of tungsten in argon-6.5 vol % fluorine at 4000 °F is summarized in Table I and plotted in Figure 2. These data show that corrosion rate increases with decreasing nozzle distance but varies only a minor amount for nozzle distances of around 1 in. At the 1 in. nozzle distance the gas velocity across the sample surface is considered to be only slightly less than the nozzle exit velocity (400 ft/sec). Also plotted in Figure 2 are the maximum recession rates obtained by micrometer measurements of the minimum thickness of the test samples after exposure. Note that the "cratering" increases rapidly at distances less than 1.5 in.

The effects of fluorine flow rate, velocity, and concentration on the corrosion rate of tungsten in fluorine-argon mixtures were determined at 4000°F. The pertinent data are summarized in Table II and plotted in Figures 3, 4, and 5. These data show that the corrosion rate is dominantly dependent upon the fluorine flow rate rather than either velocity or concentration, although the latter effects may not be negligible. This suggests that the corrosion rate is not controlled by reaction rates but rather by mass transport conditions at this high temperature.

The effect of temperature on the corrosion rate of tungsten under various flow conditions was studied to obtain insight into the possible corrosion mechanism. The pertinent data are summarized in Table III and shown graphically in Figure 6. It can be seen in Figure 6 that the curves all have the same general shape and that corrosion rates decrease quite rapidly at approximately 4700 to 5000°F. This decrease is attributed to the thermal instability of the tungsten/fluorine reaction products of temperatures within that range. That apparent basis for decreased corrosion rates is strengthened by the observation

TABLE I

EFFECT OF NOZZLE DISTANCE ON THE CORROSION RATE OF TUNGSTEN IN A FLUORINE-ARGON MIXTURE (6.5/93.5 VOL) AT 4000°F* (Ref. 2)

Nozzle Distance, in.	Average Specific Weight Loss, $mg/cm^2/min$	Average Surface Recession Rate, mils/min	Maximum Measured Surface Recession, mils/min
0.75	140.0	2.86	9.3
1.0	128.6	2.62	6.8
1.25	130.5	2.66	6.2
1.25	137.0	2.79	6.3
1.5	119.5	2.44	4.6
1.75	109.0	2.22	3.8
1.75	120.5	2.46	4.3
2.5	109.0	2.22	3.0
3.0	90.5	1.83	2.5
3.0	94.5	1.93	2.4

*Total gas flow rate, 10 ft^3/hr; fluorine flow rate, 0.65 ft^3/hr; nozzle exit velocity ~400 ft/sec. Specimen oriented ~45° to gas stream.

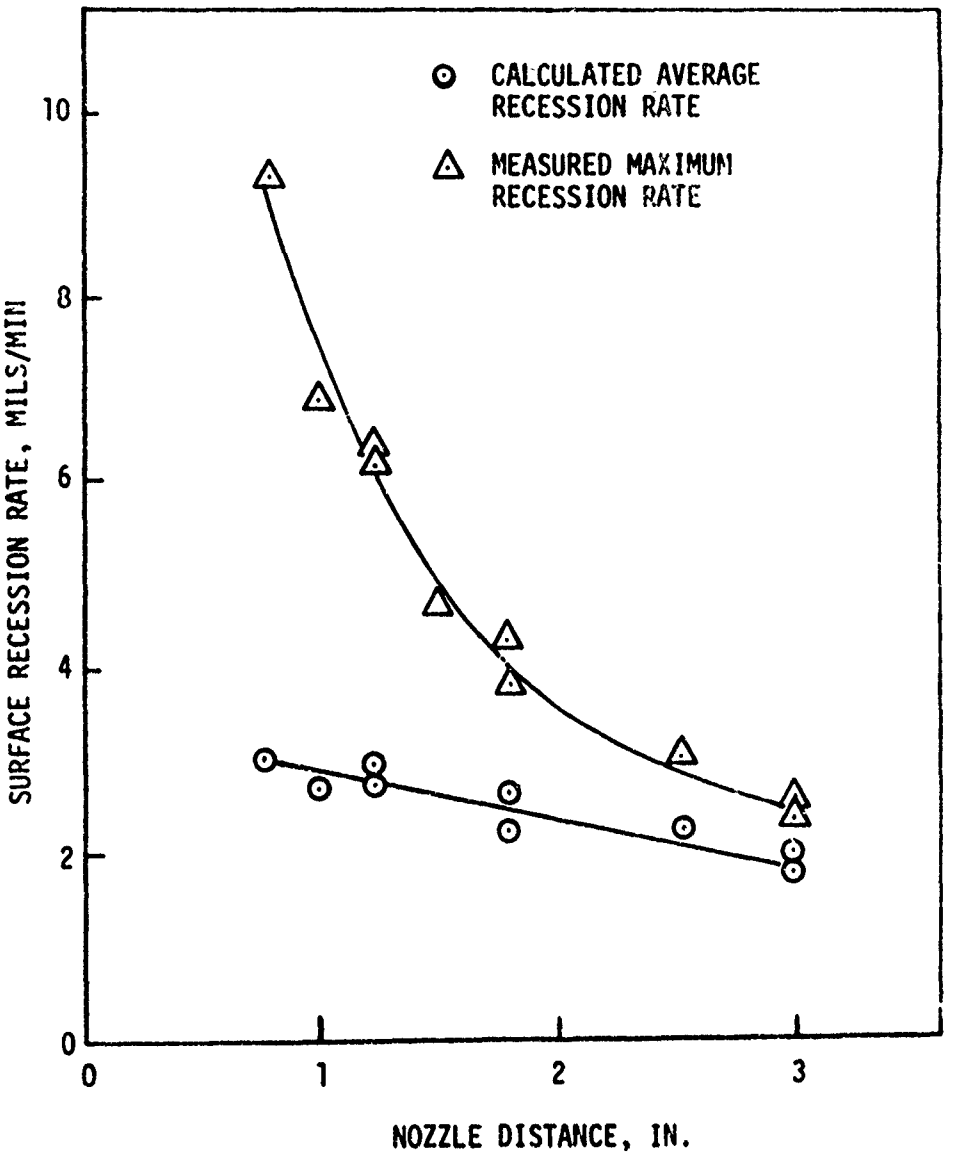

Figure 2. Effect of Nozzle Distance on the Corrosion Rate of Tungsten at 4000°F in Fluorine-Argon Mixture (6.5/93.5 vol)

TABLE II

EFFECT OF FLUORINE FLOW RATE, VELOCITY, AND CONCENTRATION ON THE CORROSION RATE OF TUNGSTEN IN FLUORINE-ARGON MIXTURES AT 4000°F (Ref. 2)

Total Flow Rate, ft^3/hr	Fluorine Flow Rate, ft^3/hr	Fluorine Content, % vol	Nozzle Exit Velocity, ft/sec	Average Specific Weight Loss, $mg/cm^2/min$	Average Surface Recession Rate, mils/min
2.5	0.65	26	98	115.0	2.34
4	0.26	6.5	157	50.6	1.03
5	0.65	13.0	196	120.0	2.45
8	0.52	6.5	314	101.0	2.06
10	0.25	2.5	393	47.6	0.97
10	0.65	6.5	393	128.6	2.62
10	1.0	10.0	393	186.5	3.80
12.5	0.65	5.2	491	139.0	2.83
12.5	0.25	2.0	491	51.5	1.05
12.5	0.81	6.5	491	152.0	3.10
12.5	1.0	8.0	491	191.0	3.90

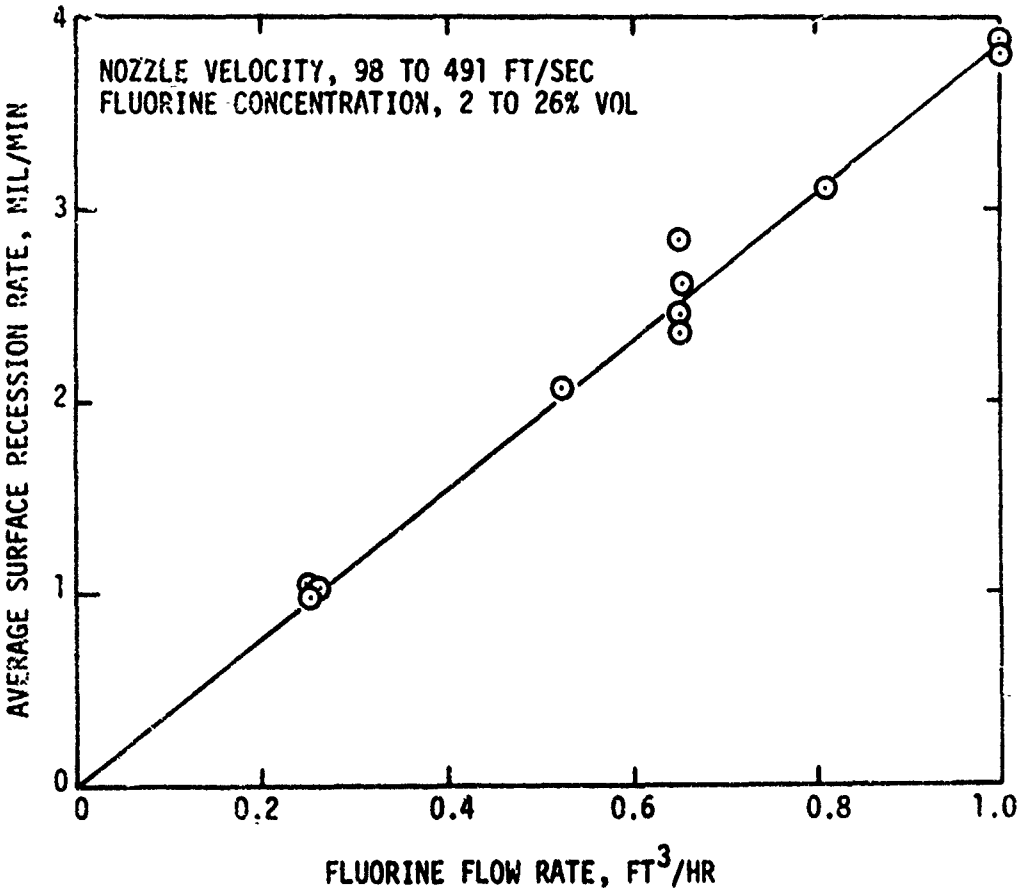

Figure 3. Effect of Fluorine Flow Rate on the Corrosion Rate of Tungsten at 4000°F and a Nozzle Distance of 1 in.

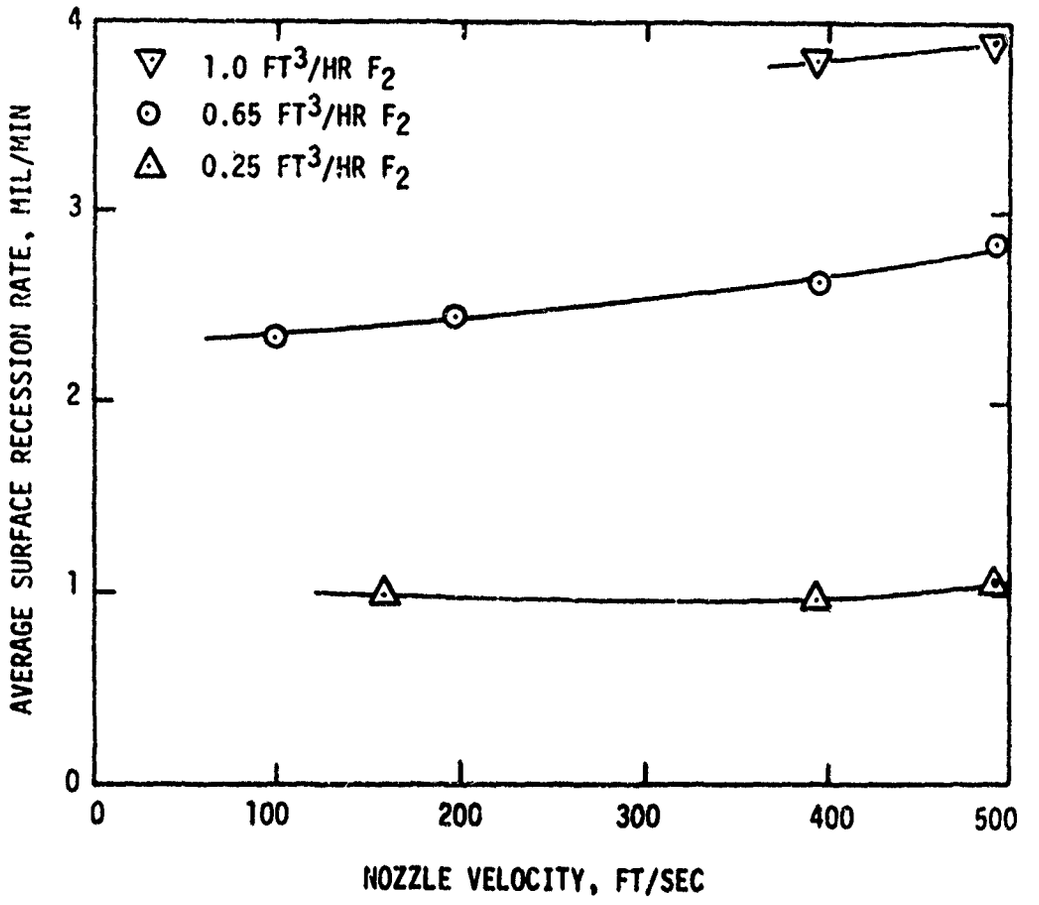

Figure 4. Effect of Nozzle Velocity on the Corrosion Rate of Tungsten at 4000°F and a Nozzle Distance of 1 in.

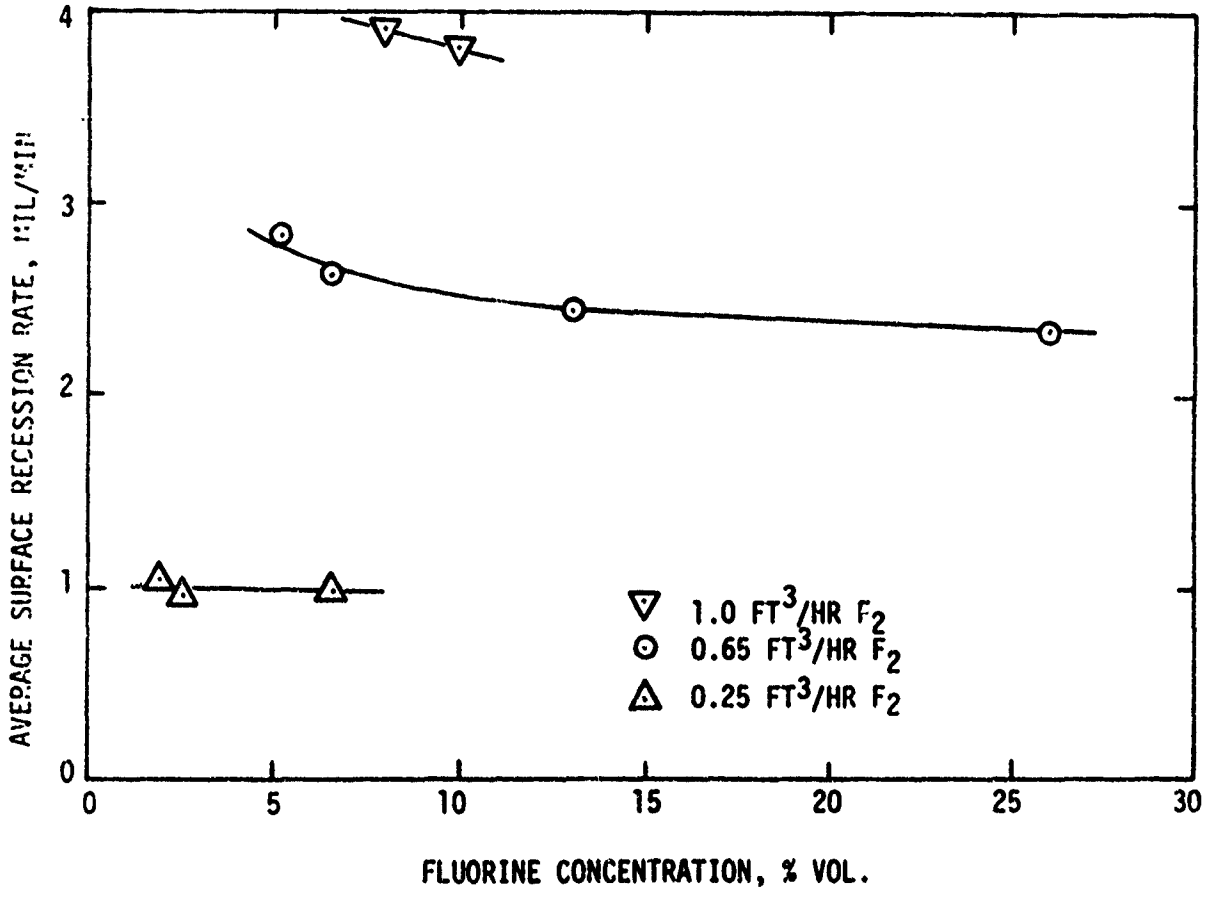

Figure 5. Effect of Fluorine Concentration on the Corrosion Rate of Tungsten at 4000°F and a Nozzle Distance of 1 in.

TABLE III

EFFECT OF NOZZLE-SPECIMEN DISTANCE AND TEMPERATURE ON THE CORROSION RATE OF TUNGSTEN IN A FLUORINE-ARGON MIXTURE (6.5/93.5 VOL)

Nozzle Distance, in.	Specimen Temperature, °F	Average Specific Weight Loss, $mg/cm^2/min$	Average Surface Recession Rate, mils/min
1	3000	121.5	2.48
1	3500	126.4	2.58
1	3500	128.6	2.63
1	4000	128.8	2.62
1	4580	124.8	2.54
1	5110	92.0	1.88
1	5500	55.2	1.13
1	5800	30.6	0.62
3	4000	90.3	1.84
3	5000	71.0	1.45
*	4000	16.8	0.34
*	5000	10.8	0.22
*	5280	4.5	0.09

*Nozzle baffled to avoid direct impingement of gas mixture on the sample.

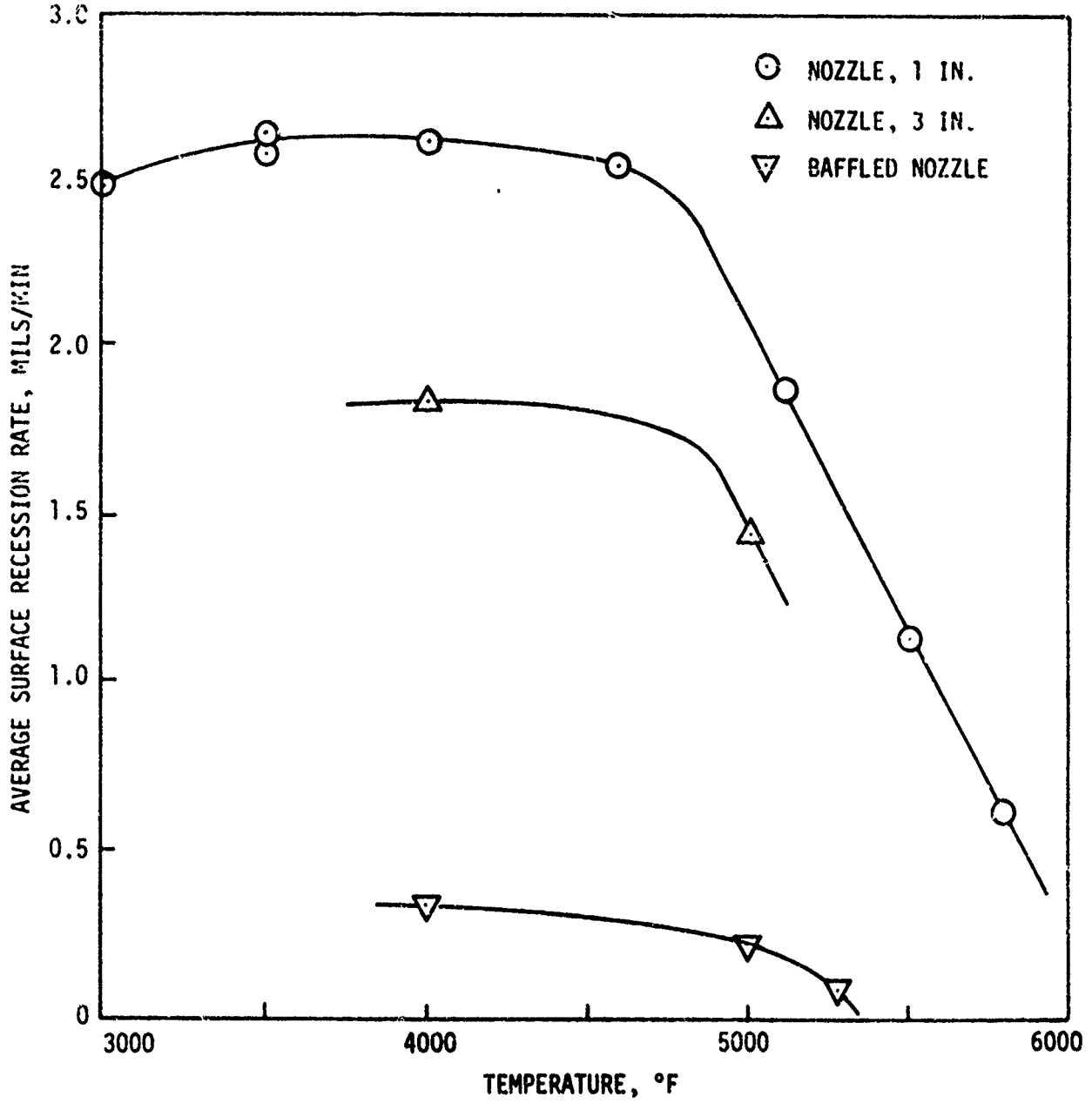

Figure 6. Effect of Temperature and Flow Conditions on the Corrosion Rates of Tungsten in a Fluorine-Argon Gas Mixture (6.5/93.5 vol)

I, A, Flow-Type Tests (cont.)

that tungsten hexafluoride, WF_6, in contact with tungsten shows markedly decreasing tungsten surface recession rates at about 4500°F. This is most directly reconciled by the dissociation of WF_6 (or a lower fluoride) to solid tungsten and fluorine.

A summary of the corrosion rate data obtained with fluorine-argon gas mixtures and tantalum, tungsten, tungsten-base alloys, rhenium-base alloys, and iridium and iridium-base alloys is given in Tables IV, V, VI, VII, and VIII, respectively. Representative data from these tables are graphically displayed in Figure 7 for the 6.5/93.5 vol fluorine-argon gas mixture. It is interesting to note that the metals appear to be corroded at about the same rates at 2500°F but differ very substantially at higher temperatures. Each material begins to show a rapidly decreasing corrosion at some high temperature and that temperature would appear to represent the temperature at which the metal fluoride shows an appreciable tendency to dissociate.

TABLE IV

CORROSION RATE OF TANTALUM IN FLOWING FLUORINE-ARGON MIXTURES(*)

Specimen Temp., °F	F_2 Conc., % vol.	Average Specific Weight Loss, $mg/cm^2/min.$	Average Specific Surface Recission, mils/min.	Ref.
4000	2.5	53.2	1.26	1
5000	2.5	58.4	1.38	1
4000	6.5	144.8	3.44	1
5000	6.5	137.0	3.25	1

*Total gas flow rate, 10 ft^3/hr. Nozzle exit velocity ~400 ft/sec. Specimen 1 in. from nozzle at ~45° to gas stream.

TABLE V

CORROSION RATE OF TUNGSTEN IN FLOWING FLUORINE-ARGON MIXTURES*

Specimen Temp., °F**	F_2 Conc., % vol	Average Specific Weight Loss, $mg/cm^2/min$	Average Surface Recession Rate, mils/min	Ref.
5110	0.4	5.6	0.12	1
3550	2.5	52.8	1.07	1
4000	2.5	47.6	0.97	1
4580	2.5	42.5	0.87	1
5110	2.5	27.1	0.55	1
5110	2.5	28.8	0.55	1
2225	6.5	106.5	2.17	2
2380	6.5	103.2	2.11	2
2390	6.5	100.0	2.04	2
2830	6.5	121.2	2.47	2
3000	6.5	121.5	2.48	2,3
3500	6.5	126.4	2.58	1,2,3
3500	6.5	128.6	2.63	1
4000	6.5	128.8	2.62	2,3
4580	6.5	124.8	2.54	2,3
4580	6.5	124.8	2.54	1
5110	6.5	92.0	1.88	1,2
5500	6.5	55.2	1.13	3
5800	6.5	30.6	0.62	3
3500	10.0	195.0	4.00	1
4000	10.0	186.5	3.80	2
4580	10.0	169.0	3.45	1
5110	10.0	142.4	2.90	1

*Total gas flow rate, 10 ft^3/hr. Nozzle exit velocity ~400 ft/sec. Specimen 1 in. from nozzle at ~45° to gas stream.

**Emittance of 0.35 used in optical temperature correction.

TABLE VI

CORROSION RATES OF TUNGSTEN-BASE ALLOYS IN FLOWING FLUORINE-ARGON MIXTURES*

I. Tungsten-Rhenium (74/26 wt)

Specimen Temp., °F	F_2 Conc., % vol	Average Specific Weight Loss, $mg/cm^2/min$	Average Surface Recession Rate, mil/min	Ref.
4000	2.5	47.2	0.94	1
4500	2.5	40.0	0.80	1
5000	2.5	40.2	0.80	1
4000	6.5	113.2	2.25	1
4500	6.5	102.0	2.03	1
5000	6.5	91.0	1.81	1
4000	10.0	163.2	3.25	1
4500	10.0	151.0	3.01	1
5000	10.0	132.6	2.63	1

II. Tungsten-Iridium (95/5 wt)

3500	6.5	104.5	2.12	2
4000	6.5	109.0	2.22	2
4500	6.5	103.5	2.11	2
4660	6.5	110.0	2.23	2
5000	6.5	106.0	2.15	2

III. Tungsten-Iridium (90/10 wt)

3500	6.5	103.8	2.09	2
4000	6.5	98.5	1.98	2
4500	6.5	96.0	1.93	2
4660	6.5	97.5	1.96	2
5000	6.5	89.5	1.80	2

*Total gas flow rate, 10 ft^3/hr. Nozzle exit velocity ~400 ft/sec. Specimen 1 in. from nozzle at ~45° to gas stream.

TABLE VII

CORROSION RATES OF RHENIUM AND RHENIUM-BASE ALLOYS IN FLOWING FLUORINE-ARGON MIXTURES*

Specimen Temp., °F	F_2 Conc., % vol	Average Specific Weight Loss, mg/cm^2/min	Average Surface Recession Rate, mil/min	Ref.
I. Rhenium				
5000	2.5	4.0	0.07	1
2500	6.5	119.5	2.24	3
2500	6.5	119.0	2.24	3
3000	6.5	119.0	2.22	2,3
3500	6.5	123.0	2.30	2,3
4000	6.5	87.6	1.64	1,3
4000	6.5	73.2	1.37	1,2,3
4500	6.5	61.9	1.16	1,2,3
4500	6.5	45.2	0.85	1,2,3
5000	6.5	23.0	0.43	1,2,3
5000	6.5	18.0	0.33	2,3
5200	6.5	28.3	0.53	3
4000	10.0	150.0	2.72	1
5000	10.0	42.2	0.79	1
II. Rhenium-Iridium (85/15 wt)				
4000	6.5	90.0	1.67	2
4500	6.5	69.2	1.27	2
4840	6.5	43.3	0.81	2
4500	10.0	75.6	1.4	2
5000	10.0	59.5	1.10	2
III. Rhenium-Iridium (75/25 wt)				
4000	6.5	81.5	1.50	2
4500	6.5	60.6	1.11	2
5000	6.5	30.3	0.56	2
4500	10.0	69.4	1.28	2
5000	10.0	43.8	0.81	2
IV. Rhenium-Iridium (65/35 wt)				
4000	6.5	71.0	1.30	2
4500	6.5	52.9	0.96	2
5000	6.5	26.8	0.47	2
4500	10.0	58.2	1.06	2
5000	10.0	41.5	0.75	2

*Total gas flow rate, 10 ft^3/hr. Nozzle exit velocity ~400 ft/sec. Specimen 1 in. from nozzle at ~45° to gas stream.

TABLE VIII

CORROSION RATES OF IRIDIUM AND IRIDIUM BASE ALLOYS IN FLOWING FLUORINE-ARGON MIXTURES*

I. Iridium

Specimen Temp., °F	F_2 Conc., % vol	Average Specific Weight Loss, mg/cm^2/min	Average Surface Recession Rate, mil/min	Ref.
2500	6.5	131.0	2.29	2,3
2730	6.5	105.0	1.84	2,3
3000	6.5	78.0	1.36	2,3
3000	6.5	68.0	1.19	3
3000	6.5	65.5	1.15	2,3
3500	6.5	29.4	0.51	1,3
3500	6.5	22.4	0.39	1
3500	6.5	18.7	0.33	2,3
4000	6.5	15.3	0.27	2,3
4000	6.5	11.2	0.20	1
4370	6.5	12.8	0.22	1
4400	6.5	10.2	0.18	1,3
4000	10.0	21.7	0.38	1

II. Iridium-Rhenium (67/33 wt)

4000	2.5	13.1	0.23	1
4425	2.5	11.2	0.20	1
2680	6.5	105.6	1.89	3
3060	6.5	70.6	1.27	3
3500	6.5	47.0	0.84	1
3500	6.5	46.0	0.82	3
3500	6.5	43.0	0.77	2,3
4000	6.5	29.3	0.53	1,3
4000	6.5	18.9	0.34	2,3
4420	6.5	16.0	0.29	3
4425	6.5	16.0	0.29	1
4500	6.5	13.3	0.24	2,3
4700	6.5	13.2	0.23	1
4000	10.0	46.0	0.82	1
4425	10.0	37.0	0.66	1

*Total gas flow rate, 10 ft^3/hr. Nozzle exit velocity ~400 ft/sec. Specimen 1 in. from nozzle at ~45° to gas stream.

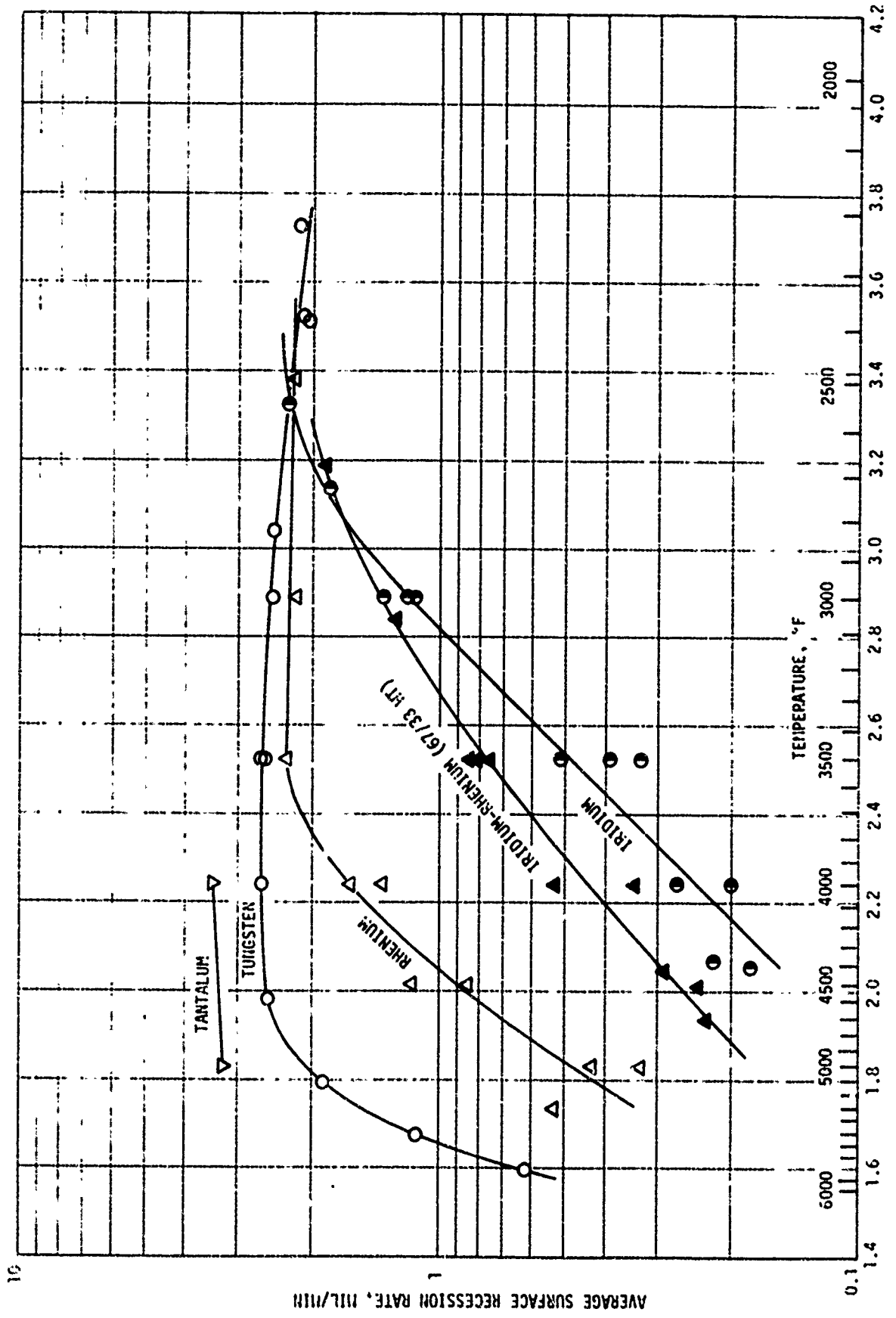

Figure 7. Corrosion Rates of Refractory Metals in Flowing Fluorine-Argon Gas Mixtures (6.5/93.5 Vol.)

I,A,Flow-Type Tests (cont.)

2. Dynamic Compatibility of Gaseous Fluorine with Nickel (Reference 4)

A 0.050-in. wall Ni 200 tube was electrically heated to selected temperatures and then exposed to gaseous fluorine at ~260 psia and a velocity of ~30 ft/sec for 1-sec periods of time. Tests were conducted at specimen temperatures of 1000 to 2000°F at 200°F increments and from 2000 to 2300°F at 100°F increments. There was no evidence of tube melting or ignition in any of the tests. A sonic orifice located downstream of the tube specimen and exposed to gaseous fluorine at ~140 psia, 200-250°F, and ~1000 ft/sec was similarly unaffected.

Ref. 4 Duckering, R. E., "Monopropellant/Bipropellant TCA", Report 6483-Q-1, Aerojet Liquid Rocket Company, Sacramento, Calif., Contract NAS2-6483, 15 July 1971.

I, A, Flow-Type Tests (cont.)

3. Compatibility of Metals with Liquid Fluorine at High Flow Velocities, Pressures and Liquid Impact Conditions (Reference 5)

Specimens of various metals in selected geometric configurations were exposed to liquid fluorine under controlled conditions of flow and pressure. None of the metal samples eroded, decomposed, or exhibited any measurable physical or chemical changes. In a run made with a Teflon sample, instantaneous chemical reaction and decomposition occurred.

Fluorine was forced through 0.0135 in. ID metal orifices with pressures up to 1500 psi and velocities up to 376 ft/sec; the maximum cumulative flow time per specimen was 1 hr and the minimum was 22 min. Larger orifices were subjected to even higher velocities (over 400 ft/sec) for periods up to 60 sec. Only a slight yellowish color appeared on the upstream side of nickel and aluminum orifices. The brass orifice appeared slightly darker, and the stainless steel appeared etched.

Impact plates of stainless-steel weld slag and aluminum sustained fluorine environments with pressures from 100 to 1350 psi and velocities from 136 to 355 ft/sec for nearly 60 sec without effects other than slight discoloration. No reaction was produced by sharpedged turbulence test wedges of stainless steel, aluminum, and brass at velocities up to 169 ft/sec.

The results of this investigation show that turbulence, fluid friction, and impact effects resulting from high-pressure, high-velocity liquid-fluorine flow through clean tubing or past irregularly shaped or sharp-edged objects are not likely to initiate fluorine-system failures. The successful operations achieved in this series of compatibility tests can be attributed to the meticulous care that was taken in the assembly, cleaning, and passivation techniques used before exposure of the system to severe dynamic fluorine service. Therefore, improper choice of hardware, poor assembly techniques, and inadequate cleaning and pickling procedures are considered to be the primary cause of fluorine-system failures.

The results of these tests are summarized in Table IX.

Ref. 5 Schmidt, H. W., "Compatibility of Metals with Liquid Fluorine at High Pressures and Flow Velocities", RM E58D11, Lewis Flight Propulsion Center (NACA), Cleveland, Ohio, July 15, 1958.

TABLE IX

COMPATIBILITY OF METALS WITH LIQUID FLUORINE UNDER HIGH VELOCITY FLOW AND IMPACT CONDITIONS

Configuration	Material	Experimental Conditions: Initial Pressure, psig	P, psi	Fluorine, lb	Time, sec	Flow Rate, lb/sec	Velocity, ft/sec	Remarks
Leak-simulator orifice 0.0135-in. ID, L/D = 27.75	Aluminum (24 ST)	525	377	9.64	528	0.0183	184	Slight yellowish discoloration on upstream side of orifice
		1012	664	9.64	340	0.0283	293	
		1500	1432	12.43	436*	0.0285	287	
		1460	1436	15.25	424	0.0360	372	
		1339	1339	15.25	432	0.0353	365	
				62.21	2160 (36 min)			
	Stainless steel (347)	1386	1226	17.7	616*	0.0287	297	Slight frosted or etched effect on upstream side. Less affected on downstream side.
		1390	1206	17.7	578	0.0306	317	
		1500	1375	13.85	518*	0.0267	276	
		1510	1350	13.85	488	0.0284	293	
		1505	1443	13.85	472	0.0294	303	
		1512	1408	13.85	480	0.0289	299	
		1500	1398	13.85	500	0.0277	286	
				104.65	3652 (60.9 min)			
	Brass (64 SE)	1495	1395	12.43	344	0.036	376	
		1500	1390	15.25	670*	0.023	235	Slightly darker after exposure
		1465	1424	15.25	440	0.035	358	
				42.93	1454 (21.9 min)			
	Nickel	1100	1015	11.92	696*	0.01713	177	Slight yellowish discoloration on upstream side
		1413	1336	11.92	616*	0.01935	200	
				23.84	1312 (21.9 min)			

*Orifice clogged

Table IX (cont.)

Configuration	Material	Experimental Conditions: Initial Pressure, psig	P, psi	Fluorine, lb	Time, sec	Flow Rate, lb/sec	Velocity, ft/sec	Remarks
Leak-simulator orifice; 0.025-in. ID, L/D = 5	Aluminum (soft)	1453	1315	17.7	132	0.134	417	
		1465	1360	17.7	130	0.136	410	
		1486	1302	13.85	110	0.126	380	Slight frosted appearance on both sides
		1500	1282	13.85	110	0.126	379	
		1495	1326	13.85	107	0.130	390	
		1492	1391	13.85	108	0.128	386	
		1505	1375	13.85	106	0.131	394	
				104.65	803 (13.4 min)			
Sharp-edged orifice; 1/8-in. ID	Aluminum (soft)	785	505	10.45	6.8	1.54	185	
		1480	1203	10.45	4.4	2.38	286	
		1465	1169	10.45	4.6	2.27	273	Slightly coated on downstream side; frosty appearance.
		1440	1144	10.45	4.6	2.27	273	
		1452	1159	10.45	4.0	2.61	315	
		1465	1196	10.45	4.6	2.27	273	Slightly etched on upstream side
		1470	1192	10.45	4.0	2.61	315	
				73.15	33.0			
	Brass (64 SE)	1008	773	9.74	5.7	1.71	206	
		1482	1206	9.74	4.9	1.99	240	
		1505	1249	9.74	4.2	2.32	279	
		1498	1226	9.74	4.5	2.16	261	
		1504	1223	9.74	4.8	2.03	245	
		1494	1232	9.74	4.7	2.07	249	Purple discoloration on downstream side of orifice
		1500	1223	9.74	4.8	2.03	244	
		1493	1233	9.74	4.4	2.21	267	
		1450	1190	9.74	4.7	2.07	249	
		1485	1334	9.74	4.4	2.21	266	
		1500	1358	9.74	4.0	2.43	266	
				107.14	51.1			

Table IX (cont.)

Configuration	Material	Experimental Conditions: Initial Pressure, psig	P, psi	Fluorine, lb	Time, sec	Flow Rate, lb/sec	Velocity, ft/sec	Remarks
Sharp-edged orifice; 1/8-in. ID (cont.)	Stainless steel (304)	85	85	9.97	20.5	0.485	58.4	
		121	114	9.97	16.0	0.622	74.9	
		300	278	9.97	9.0	1.108	133.3	
		408	391	9.97	9.1	1.095	131.8	
		1005	811	9.97	6.0	1.66	200.0	
		1202	990	9.97	5.44	1.83	220.0	
				59.82	66.04 (1.1 min)			
Impact Plate with Round-edged orifice; 1/8-in. ID	Stainless steel weld slag (304)	1344	1206	11.92	3.78	3.17	355	
		1500	1362	11.92	4.5	2.65	349	
		102	85	9.74	8.6	1.13	136	Very slight etched effect but no apparent attack on impact sample. Slight yellowish discoloration on upstream side of orifice.
		1000	704	9.74	5.8	2.12	201	
		1475	1170	9.74	4.2	2.32	279	
		1485	1173	9.74	4.4	2.21	280	
		1480	1219	9.74	3.3	2.95	350	
		1471	1168	9.74	4.6	2.12	280	
		1495	1196	9.74	4.5	2.16	282	
		1480	1176	9.74	4.0	2.43	293	
		1495	1183	9.74	4.2	2.32	284	
		1490	1337	9.74	3.9	2.50	301	
		1500	1349	9.74	4.0	2.43	293	
				130.98	57.8			
	Aluminum (soft)	800	568	10.45	5.88	1.78	214	
		1470	1195	10.45	4.2	2.49	299	Slight etched effect on impact sample
		1475	1172	10.45	4.8	2.18	262	
		1470	1180	10.45	4.6	2.27	273	
	Stainless steel (304)	1465	1165	10.45	4.4	2.38	286	No effect on orifice except slight bluish cast on downstream side
		1455	1169	10.45	4.3	2.43	293	
		1460	1167	10.45	4.6	2.27	273	
				73.15	32.8			

Table IX (cont.)

Configuration	Material	Experimental Conditions: Initial Pressure, psig	P, psi	Fluorine, lb	Time, sec	Flow Rate, lb/sec	Velocity, ft/sec	Remarks
							Cycles per second*	
Rotating-vane flowmeter, ball-bearing (stainless steel rotor)	Stainless steel (316, 420, 430, 440)	85	--	9.97	20.5	0.465	46.5	Rotor-mount retainer rings slightly eroded and loose; would have affected running on continued operation.
		121	--	9.97	16.0	0.622	61.5	
		300	--	9.97	9.0	1.108	93.0	
		408	--	9.97	9.1	1.095	109.5	
		1005	--	9.97	6.0	1.66	165.0	
		1202		9.97	5.44	1.83	183.0	
				59.82	66.04 (1.1 min)			
	Aluminum (soft)	1443	1200	16.35	0.7	23.4	155.0	All four sides of sample had sooty film or coating. Not adherent; rubbed off easily. Did not seem to be in metal surface but on it; metal underneath was unchanged.
		1260	761	16.35	3.14	5.2	34.4	
		1370	872	16.35	3.10	5.3	39.4	
		1295	921	16.35	1.96	8.3	54.0	
		1360	873	16.35	2.75	5.9	39.5	
		1295	1057	10.20	0.58	17.6	117.0	
		1357	1067	10.20	0.67	17.9	118.0	
		1391	1103	10.20	0.40	25.5	169.0	
				112.35	13.20			
	Teflon	50	50	--	--	--	--	Immediate ignition on contact.

*100 cycles · 1 lb liquid fluorine.

Table IX (cont.)

Configuration	Material	Initial Pressure, psig	P, psi	Fluorine, lb	Time, sec	Flow Rate, lb/sec	Velocity, ft/sec	Reynolds Number, Re	Remarks
1/4-in. tubing; 0.1285-in. ID, length = 11.3 ft	Stainless steel (304)	0-120*	115	11.0	68.8	0.16	17.4	88,000	All runs with 1/4-in. tubing made without liquid-nitrogen bath. Initial temperature, 75°F.
		0-420*	397	11.0	26.4	0.417	45.4	232,000	
		0-1017*	837	11.0	18.24	0.603	65.7	337,000	
		0-1505*	1297	11.0	14.64	0.751	81.7	543,000	
				44.0	128.08 (2.13 min)				
1/4-in. tubing; 0.182-in. ID, length = 12.2 ft	Aluminum (5052 SO)	0-133*	118	11.0	25.6	0.43	24.4	176,000	All runs with 1/4-in. tubing made without liquid-nitrogen bath. Initial temperature, 75°F.
		0-410*	385	11.0	22.0	0.50	28.4	205,000	
		0-1010*	768	11.0	12.0	0.917	52.0	377,000	
		0-1500*	1275	11.0	7.36	1.495	84.8	614,000	
		0-1485*	1312	11.0	7.16	1.536	87.3	632,000	
				55.0	74.12 (1.24 min)				
3/4-in. tubing	Stainless steel (304)	1260	--	16.35	3.14	5.2	17.8	526,000	The 3/4-in. tubing was part of the test apparatus
		1370	--	16.35	3.10	5.3	20.3	660,000	
		1360	--	16.35	2.75	5.9	20.4	602,000	
		1340	--	16.35	1.96	8.3	28.5	843,000	
		1340	--	16.35	1.26	13.0	44.5	1,315,000	
		1330	--	16.35	1.14	14.3	46.3	1,370,000	
		1319	--	16.35	1.0	16.4	55.8	1,650,000	
		1391	--	10.2	0.9	18.2	62.1	1,835,000	
			--	16.35	0.7	23.4	71.4	2,110,000	
			--	10.2	0.4	25.5	87.3	2,580,000	
Turbulence test wedge (sharp-edged)	Stainless steel (347)	1340	862	16.35	1.14	14.34	90.0		No effect
		1340	740	16.35	1.26	12.97	86.4		
		1330	886	16.35	1.0	16.35	108.5		
		1404	978	16.35	0.9	18.16	120.5		
				65.4	4.3				
	Brass (64 SE)	1319	800	10.2	0.9	11.3	70		Very slightly frosted appearance; slightly lighter in color than parent metal
		1383	1036	10.2	0.5	20.4	135		
		1286	1028	10.2	0.57	17.9	118		
		1346	1100	10.2	0.66	15.5	102		
				40.8	2.63				

*Instantaneous pressure release.

I, A, Flow-Type Tests (cont.)

4. Compatibility of an Aluminum-Silicon Alloy with Liquid Fluorine at High Flow Velocities and Liquid Impact Conditions (Reference 6)

Experiments were conducted to evaluate the erosion and impingement resistance of aluminum-silicon alloy A356-T6 in LF_2 (0.02 vol % HF). This was accomplished by flowing LF_2 through an orifice test coupon and impinging on another test coupon 3/16 in. downstream.

Orifice and impingmenet specimens were exposed to F_2 at -235°F and 400 psig for 30 min at velocities of 150 and 50 ft/sec. No orifice change was observed but superficial impingement attack was observed at both velocities.

The test conditions and the results of the individual runs are summarized in Table X.

TABLE X

COMPATIBILITY OF AN ALUMINUM-SILICON ALLOY WITH LIQUID FLUORINE UNDER HIGH VELOCITY FLOW AND IMPACT CONDITIONS*

Material	Velocity, ft/sec	Orifice Diameter, in. Initial	Final	Final Condition of Impingement Coupon
Al A356-T6 NASA Grade IIIF	50	0.065	0.065	Superficial attack
Al A356-T6 NASA Grade IIIF	150	0.039	0.039	Superficial attack

*LF_2 temperature, -235°F: LF_2 static pressure, 400 psig; flow rate, 0.50 gal/min; test time, 30 min.

Ref. 6 "Development and Demonstration of Criteria for Liquid Fluorine Feed System Components", Final Report NASA CR-72063, Douglas Aircraft Co., Santa Monica, Calif., Contract NASw 1351, October 1967.

I, Fluorine Dynamic Compatibility (cont.)

B. IMPACT-TYPE TESTS

1. Impact Testing of a Titanium Alloy in Liquid and Gaseous Fluorine (Reference 7)

Open cup impact tests with liquid fluorine (-320°F) and gaseous fluorine (65°F, ambient pressure) and titanium alloy, Ti-5Al-2.5Sn (ELI), were performed with a modified ABMA impact test apparatus at an impact energy of 72 ft-lb. With liquid fluorine two reactions were observed in two tests and with gaseous fluorine no reactions were observed in 20 tests.

Ref. 7 Endicott, D. L. and L. N. Donahue, "Development and Demonstration of Criteria for Liquid Fluorine Feed System Components", NASA CR-72543, McDonnell Douglas Astronautics Co., Huntington Beach, Calif., Contract NAS 3-11195, June 1969.

I, B, Impact-Type Tests (cont.)

2. Impact Tests of Aluminum and Titanium Alloys in Liquid Fluorine (Reference 8)

Modified ABMA open cup impact tests were conducted on specimens of Al 2014-T6 at an impact energy of 70 ft-lb in LF_2 (-320°F) using 17-4 PH stainless steel strikers and Al 1100 sample cups. No reactions were observed in 20 tests.

Modified ABMA open cup impact tests on Ti-6Al-4V (ELI) in liquid fluorine indicated it was impact sensitive when the following test combinations were used: (1) Ti-6Al-4V (ELI) striker pin, disc, and cup; and (2) Ti-6Al-4V (ELI) striker pin and disc and Al 1100 cup. The energy for a 50% probability of initiating a reaction with test combination (1) in LF_2 at -320°F was calculated to be 48 ft-lb. The test combination (2) was found impact sensitive at 70 ft-lb. The test combination Ti-6Al-4V (ELI) disc, cup, and 17-4 PH striker pin was not impact sensitive in LF_2 at 75 ft-lb.

Three drop-weight closed cup impact tests were conducted with LF_2 at -240 $\pm$ 10°F (485 $\pm$ 15 psig) in the following manner: 86 cc of gaseous fluorine was condensed in the test unit at -320°F; the test assembly was allowed to heat up by letting the LN_2 evaporate from the cryogenic bath; temperature and pressure were recorded and the drop-weight released when the pressure reached 500 psig. For these reactive impact tests, a 10 to 30 psi pressure drop in the test bomb was observed when impact occurred. Examination of the Ti-6Al-4V (ELI), disc, striker pin and cup revealed numerous reaction sites. This impact energy value was deliberately chosen to be less than the open cup E_{50} of 48 ft-lb required for a 50% probability of initiating a reaction at -320°F in LF_2. Qualitatively, the reaction found at 40 ft-lb impact energy level indicates that the titanium alloy is more reactive than previously believed. This is borne out by the numerous reaction sites observed on the disc, striker pin and cup. These test results are summarized in Table XI.

Ref. 8 Toy, S. M., L. E. Bell, W. D. English, and N. A. Tiner, "Tankage Materials in Liquid Propellants", AFML-TR-68-204, McDonnell Douglas Corp., Contract F33615-67-C-1718, July 1968.

TABLE XI

IMPACT TEST RESULTS ON 2014-T6 ALUMINUM AND Ti-6Al-4V(ELI) TITANIUM IN LIQUID FLUORINE

Metal Specimen	Striker Material	Sample Cup Material	Liquid Fluorine Temp., °F	Impact Energy, ft-lb	Number of Tests	
					Positive Reaction	No Reaction
Al 2014-T6	17-4 PH	Al 1100	-320	70	0	20
Ti-6Al-4V	Ti-6Al-4V	Ti-6Al-4V	-320	≤43	0	2
				47	1	7
				47.5	0	1
				48	1	1
				48.5	1	0
				49	1	0
				50	0	1
Ti-6Al-4V	Ti-6Al-4V	Al 1100	-320	≥51.5	2	0
				47	0	1
				58.5	1*	0
				70	1	0
Ti-6Al-4V	17-4 PH	Ti-6Al-4V	-320	≤72	0	5
Ti-6Al-4V	Ti-6Al-4V	Ti-6Al-4V	-240±10	≥40	3	0

* Reaction on rebound only

I, B, Impact-Type Tests (cont.)

3. Impact Testing of an Aluminum Casting Alloy in Liquid Fluorine (Reference 6)

Open cup impact tests with liquid fluorine at -320°F and aluminum-silicon casting alloy, A356, (Types IV, III, II, and I, in order of increasing quality) were performed with a modified ABMA impact test apparatus at an impact energy of 72 ft-lb. Many of the test samples cracked upon testing (the frequency of cracking decreasing as the quality of the alloy increased) but even with the formation of new metal surfaces from sample cracking, reactions were not initiated. Samples which had previously been dye-penetrant inspected using both organic and inorganic dye penetrants and then thoroughly cleaned and baked in a vacuum prior to impact testing showed that the penetrants lead to increased impact sensitivity and, thus, are not safe quality inspection tests of cast materials for fluorine service.

A closed-cup impact test at the 72 ft-lb impact level on aluminum A356-T6, Grade III-F, which had been immersed in LF_2 at -320°F for 21 days showed no sign of reaction initiation but cracked similar to specimens used in open-cup tests.

Ref. 6 "Development and Demonstration of Criteria for Liquid Fluorine Feed System Components", Final Report NASA CR-72063, Douglas Aircraft Co., Santa Monica, Calif., Contract NASw-1351, October 1967.

I, B, Impact-Type Tests (cont.)

4. Impact Ignition Tests in Liquid Fluorine (References 9 and 10)

It had been shown that titanium is liable to ignite with liquid oxygen upon suitable impact.* On the basis of this evidence, it was suspected that titanium and possibly some of the other metals would be sensitive to impact with liquid fluorine. Therefore, tests were performed to establish the energy level required to ignite titanium in liquid fluorine. In general, the tests were accomplished by dropping weights of various sizes from various heights or by releasing a compressed spring to obtain suitable impact energies. These energies ranged from 2.6 to 65 ft-lb. The samples and strikers were examined microscopically for evidence of ignition and the results reported in a qualitative manner.

The samples were polished 1/2-or 5/8-in. squares of metal and 0.050 in. thick. The sample and all of the components of the impact cell were scrupulously cleaned and dried before being placed into the cell.

Several striker materials and a variety of striker faces were used in the tests. The preliminary work was performed using Monel strikers with titanium samples. Later work used titanium strikers with titanium samples. A few tests were conducted with aluminum and stainless steel strikers with aluminum samples. The types of strikers which were used are as follows:

Flat smooth	-	polished circular striking face, dia from 1/8 to 3/8-in.
Flat rough	-	3/8-in. dia circular striking face scarred with file marks.
Flat grooved	-	1/4-in. dia circular striking face grooved similarly to phonograph record.
Chisel pointed	-	striking face was 1/4-in. knife edge.
Conical pointed	-	sharply pointed with 60° included angle.
Hollow pointed	-	small flat tip with 1/6-in. hole drilled into it.
Round	-	hemispherical striking face, 1/8 in. radius.

*Preliminary Data on the Reaction Sensitivity of Titanium and Oxygen, Defense Metals Information Center, Battelle Memorial Institute, August 1959.

Ref. 9 Sterner, C. and Singleton, A. H.,"The Compatibility of Various Metals and Carbon with Liquid Fluorine", WADD-TR-60-436, Air Products, Inc., Contract AF 33(616)-6515, August 1960.

Ref. 10 Sterner, C. and Singleton, A. H.,"The Compatibility of Various Metals and Carbon with Liquid Fluorine", WADD-TR-60-819, Air Products, Inc., Contract AF 33(616)-6515, March 1961.

I, B, Impact-Type Tests (cont.)

Table XII presents a summary of the impact test results. The impact levels listed were calculated from the weight and distance dropped for the dropweight tests, and in the case of the spring-driven plunger, the energy level was calibrated by cocking the spring against a calibrated spring-scale. Ignition, when it was observed, was indicated by the formation of small craters and gullies together with droplets of melted metal on the sample or on the striker face. Usually the crater or gully formation on the sample was mirrored almost exactly on the face of the striker. The direction of ignition was generally away from the center of impact.

The preliminary tests with Monel metal strikers and titanium samples provided the surprising result that the striker tip reacted with the fluorine even at very low impact levels, but the sample did not. This indicated that the configuration of the striker face played as large a part in inducing ignition as did the impact energy level. This impression was strengthened in later tests as higher impact levels were used.

Various strikers were used in the hope of obtaining reasonably reproducible ignitions. The flat, smooth strikers were intended to provide a standard for comparison. The conical and chisel point strikers were intended to deliver all of the impact energy at one spot. The hollow, pointed and flat, grooved strikers were intended to trap some of the liquid and vaporized fluorine under the striker to provide high temperature by adiabatic compression. One problem with the conical pointed strikers at the higher energy levels was the welding of the tip to the sample surface. This made it difficult to detect whether ignition had occurred.

As can be seen in Table XII, the results were not very reproducible and in no case did ignition become general. There was no appa difference between the two titanium alloys tested. With aluminum, one was obtained out of 24 tests. With titanium, ignition was initiated by a types of strikers at different impact levels. The effectiveness of ignitic by the various striker configurations in increasing order was: flat, smooth, pointed, and grooved.

TABLE XII

IMPACT TEST RESULTS ON ALUMINUM AND TITANIUM ALLOYS IN LIQUID FLUORINE AT -320°F

Striker		Impact Energy,	Calc. Max. Energy Flux,	Number of Tests	
Material	Configuration	ft-lb	ft-lb/in.^{2}-sec	Positive Reaction	No Reaction
	Titanium Samples (A 110 AT and C 120 AT)				
Monel	Flat, smooth	2.6	--	0	5
	Flat, rough	2.6	--	1	1
	Hemispherical	2.6	--	1	1
	Chisel-pointed	2.6	--	2	0
	Conical, pointed	2.6	--	3	0
				7	7
Titanium	Flat, smooth	40	--	2	0
		45	1.61×10^7	3	13
		52	1.86×10^7	5	11
		55	--	2	0
		58	--	1	3
		60	2.15×10^7	10	6
	Flat, grooved	5	--	2	0
		15	--	2	0
		30	--	2	0
		55	--	1	1
		58	--	2	0
		60	--	10	2
	Conical, pointed	2.6	--	1	2
		6	--	3	0
		9	--	2	0
		35	--	1	0
		50	--	2	0
		55	--	2	0
		58	--	1	1
		60	--	8	6
		61	--	0	1
	Hollow, pointed	6	--	1	3
		10	--	2	2
		38	--	1	0
	Aluminum Samples (Al 6061)				
Stainless Steel	Flat, grooved	58	--	1	11
	Conical, pointed	58	--	0	2
Aluminum	Conical, pointed	30	--	0	10

I, B, Impact-Type Tests (cont.)

5. Tube Impact Tests with Liquid Fluorine (Reference 9)

Tubes of the various sample materials were cut to 2-in. lengths and thoroughly cleaned. Brass plugs were made up to fit the bore of the tube and they were silver soldered into the ends of the tube. A 1/4-in. copper tube was silver soldered into a hole drilled into the side of the tube. Tubes were cleaned after this with dilute hydrochloric acid, neutralized, rinsed and thoroughly dried. The tube was supported in the test cabinet in a horizontal position by a suitable mounting and a sharp conical stainless striker was positioned on the upper surface of the tube. The tube was evacuated, immersed in liquid nitrogen and completely filled with liquid fluorine. Impact was provided by dropping either a 4- or a 6-lb weight onto the striker. Impact energies of approximately 1 to 10 ft-lb were used, depending on the thickness of the tube wall. In most cases, the impact was just below that necessary to rupture the tube wall, while in a few cases the wall was actually ruptured.

Tubes of this same type filled with liquid fluorine were also exposed to explosive shock from the ignition of dynamite caps attached to the outside of the tube. Some of these explosive shock tests were performed with metal filings of the sample material placed inside the tube.

The tube impact tests were intended to test the hypothesis that a fluoride film protects the metal surface at low temperatures. It was reasoned that a severe blow on the tube wall would stretch the metal sufficiently to break the film and expose bare metal to the liquid fluorine. In many cases, the impact was severe enough to produce cracks on the inside wall of the tube and in 5 cases out of 20, the tube was ruptured. No apparent ignition or increased corrosion was detected upon examining the impacted area microscopically.

After obtaining no positive test results with the impact produced by the pointed striker, it was believed that explosive impact would provide a more severe test. Impact was supplied by firing an electric detonator cap wired to the outside of the tube. In several cases, the hydraulic pressure of the liquid fluorine drove the ends out of the tube at the time of impact. No apparent ignition or increased corrosion was detected upon microscopic examination. The tube impact test results are summarized in Table XIII.

Ref. 9 Sterner, C. J., and Singleton, A. H., "The Compatibility of Various Metals and Carbon with Liquid Fluorine", WADD-TR-60-436, Air Products Inc., Contract AF 33(616)-6515, August 1960.

TABLE XIII

TUBE IMPACT TESTS WITH LIQUID FLUORINE

Tube Material	Wall Thickness	Nominal Impact Energy	Remarks
Cu	1/16"	1 ft-lb	Energy was about 25% of amount required to rupture tube. No unusual corrosion of signs of ignition observed on inside of tube. A sharp pointed conical striker was used in all tube impact tests.
Cu	1/16"	2 ft-lb	No unusual corrosion or signs of ignition observed on inside of tube.
Cu	1/32"	3 ft-lb	Energy was about 95% of amount required to rupture tube. No unusual corrosion or signs of ignition observed on inside of tube.
Cu	1/32"	3 ft-lb	Fairly heavy corrosion on the inside of the tube, but nothing unusual in the area of impact. No signs of ignition.
Brass	1/16"	1 ft-lb	Energy was about 5% of amount required to rupture tube. No unusual corrosion or signs of ignition observed on inside of tube.
Brass	1/16"	2 ft-lb	No unusual corrosion or signs of ignition observed on inside of tube.
Brass	1/32"	3 ft-lb	Energy was about 90% of amount required to rupture tube. No unusual corrosion or signs of ignition observed on inside of tube.

TABLE XIII (cont.)

Tube Material	Wall Thickness	Nominal Impact Energy	Remarks
Brass	1/32"	3 ft-lb	Some cracks showed on the inside of the tube, but tube was still vacuum tight. Uniform light corrosion observed inside tube, but not apparently related to the impact.
SS-304	.010"	1 ft-lb	Energy was about 95% of amount required to rupture tube. No unusual corrosion or signs of ignition observed on inside of tube.
SS-304	.010"	1 ft-lb	Tube was ruptured, leaving a jagged hole approximately 1/16" in diameter. No unusual corrosion or signs of ignition observed on inside of tube, or around holes.
SS-316	.010"	1 ft-lb	Energy was about 95% of amount required to rupture tube. No unusual corrosion or signs of ignition observed on inside of tube.
SS-316	.010"	1 ft-lb	Tube was ruptured, leaving a jagged hole approximately 1/16" in diameter. No unusual corrosion or signs of ignition observed on inside of tube, or around holes.
SS-321	.010"	1 ft-lb	Tube was ruptured, leaving a jagged hole approximately 1/16" in diameter. No unusual corrosion or signs of ignition observed on inside of tube, or around holes.
SS-321	.010"	1 ft-lb	Tube was ruptured, leaving a jagged hole approximately 1/16" in diameter. No unusual corrosion or signs of ignition observed on inside of tube.

TABLE XIII (cont.)

Tube Material	Wall Thickness	Nominal Impact Energy	Remarks
SS-347	1/32"	5.0 ft-lb	Energy was about 90% of amount required to rupture tube. No unusual corrosion or signs of ignition observed on inside of tube.
SS-347	1/32"	5.5 ft-lb	Energy was about 95% of amount required to rupture tube. Some small cracks observed on inside of tube in impact area, but tube was still vacuum tight. No unusual corrosion or signs of ignition observed on inside of tube.
Nickel	0.059"	7.8 ft-lb	Energy was about 20% of amount necessary to rupture tube. Uniform heavy corrosion inside tube was probably due to contamination from the soldering operation, and did not appear to be related to impact. Striker did not hit squarely, and did not make a very big indentation.
Nickel	0.059"	5.3 ft-lb	Energy was about 50% of amount necessary to rupture tube. Some small cracks were observed on inside of tube in impact area, but tube was still vacuum tight. No unusual corrosion or signs of ignition observed on inside of tube.
Monel	1/32"	8.3 ft-lb	Tube was ruptured leaving a jagged hole approximately 1/32" in diameter. No unusual corrosion or signs of ignition observed on inside of tube, or around holes.
Monel	1/32"	5 ft-lb	No unusual corrosion or signs of ignition observed on inside of tube.

I, Fluorine Dynamic Compatibility (cont.)

C. FLEXURE-TYPE TESTS

1. Mechanical Stability of Metal Fluoride Films to Flexing and Thermal Shock in Gaseous and Liquid Fluorine (Reference 11)

Mechanical stability tests were designed to evaluate the metal fluoride stability on metal bellows of 316 stainless steel and copper when exposed to various combinations of flexing, cryoshock, gaseous fluorine and liquid fluorine conditions. The test data seem to indicate that the mechanical stability of metal fluorides formed excellent and that for all practical purposes very little (max. 0.007%) of the exposed metal fluoride surface is depleted.

a. Test Apparatus and Materials

The basic test apparatus consists of a double walled stainless steel test container, a specimen mount attached to the lid (to allow translational reciprocating motion of the specimens), a metal bellows seal on the reciprocating shaft, a variable eccentric cam drive to change the axial displacement of the shaft, and an electrically driven, variable speed rotary motion power unit. The jacket of the test container contains no direct plumbing connections with the interior of the container. It is used as a temperature control bath by flowing H_2O or LN_2 through it during different tests. A particle collector is connected at the bottom of the test chamber and is immersed in LN_2 to maintain it at -320°F when necessary.

Materials tested were in the form of bellows specimens. They were formed from cold roller mill annealed (dead soft) sheet. The sheets were rolled into a tube, butt welded and hydroformed to contain three typical bellows outside convolutions. During fabrication of the bellows, the weld is work hardened (1/8 to 1/4 full hard).

During testing the axial displacement of bellows from the rest position is maintained such that the maximum stresses at the root of the convolutions is equal to 75% yield stress. This stress level is within the elastic region.

b. Test Procedures

The mechanical stability of fluoride film was evaluated by the low cycle fatigue test, 5 cycles/sec, in the test apparatus according to the following four test procedures:

Ref. 11 Asunmea, S. K. et al, "Halogen Passivation Procedural Guide", AFRPL-TR-67-309, Astropower Laboratory, A Division of McDonnell Douglas Corporation, Contract F04611-67-C-0033, December 1967.

I,C, Flexure-Type Tests (cont.)

Test 1 - The metal bellows is fluorinated in gaseous fluorine, GF_2, at 1 atm for 1 hr, flexed for 500 cycles, and then thermally shocked by flowing liquid nitrogen during an additional 1500 cycles. The LN_2 containing any spalled-off metal fluoride particles is drained into the liquid-nitrogen-cooled particle collector below the test chamber. The test chamber is reheated to room temperature and the low cycle fatigue and thermal shock treatment is repeated four more times on the metal bellows. The particles collector is warmed up after the last test cycle. The test chamber and particle collector are evacuated. The test chamber is flushed with 500 cc of filtered Freon TF which is drained into the particle collector. The contents of the particle collector are discharged through a 0.45-micron millipore filter unit. The filtrate is collected for reuse. The millipore filter unit is capped-off and the 0.45-micron filter paper is subsequently removed in a dry box. The sample is subjected to metallurgical examination and analyses.

Test 2 - Test 1 procedure is repeated with same metal bellows specimen without flexing. Metal bellows and filter paper samples are subjected to metallurgical examination and analysis.

Test 3 - A new metal bellows is passivated, tested for 2000 cycles in 2 atm GF_2. The fluorine is flushed out with GN_2, and particulate matter is collected into particle collector with Freon TF. The filter paper and metal bellows are subjected to metallurgical examination and analysis.

Test 4 - A new metal bellows specimen is cooled to -320°F. LF_2 is condensed into the test chamber. The immersed bellows is flexed for 2000 cycles and then is drained into particle collector. The test apparatus is purged with GN_2 and flushed with Freon TF into particle collector. The filter paper sample and metal bellows are subjected to metallurgical examination and analysis.

c. Test Results

(1) Stainless Steel Specimens

After repeated test apparatus cleaning, a 316 stainless steel bellows was run through Test 1. An examination of the particulate collected for this test revealed: (1) three mica type flakes, (2) very small fragments of polymer, and (3) "structureless" coating covering the entire millipore surface. Specimens (1) and (3) were examined by selected area electron diffraction. Specimen (1) appeared to be a muscovite type material. The origin of this material cannot be explained, unless material of this type was used for packing of Freon containers or was introduced from the liquid nitrogen system. Iron fluoride in addition to iron oxides were identified in Specimen (3). The major fraction appeared to consist of iron oxides rather than iron fluorides, $Fe_3F_5 \cdot 3H_2O$, $Fe_3F_5 \cdot 7H_2O$, and FeF_2 were indicated.

I, C, Flexure-Type Tests (cont.)

The same 316 stainless steel bellows was subsequently run through Test 2. The particulate matter from this test revealed more polymer flakes than in Test 1. The interference colors noted indicated that the flake thicknesses were approximately 600 to 1000 Å. Gray and dark material which adhered to the polymer flakes appeared to be the main particulate matter collected. Electron diffraction indicated mainly Fe_2O_3 and NiO with minor amount of iron and nickel fluorides. FeF_2 and Fe_3F_5 with 7 and $3H_2O$ were also indicated. The total amount of fluorides appears to be very low and represents a thin surface layer.

The fluorinated bellows surfaces were examined under a binocular microscope, at a magnification of about 30X and under a metallographic microscope at a magnification of about 100X. Local removal of the surface layer due to mechanical damage was observed close to the regions exposed to handling. Minor areas of attack were noted on the bellows edges under maximum tension.

(2) Copper Specimens

The second series of low cycle fatigue tests was conducted on copper bellows. Cu bellows No. 4 was run through Test 1. Copper bellows No. 3 was run through Test 3. Copper bellows No. 5 was run through Test 4. No visual damage of any of these bellows was seen.

Electron diffraction analyses of filter residues indicated the presence of copper, copper oxide (CuO) and copper hydroxide ($Cu(OH)_2$). Other observations suggest that a minimum amount of the passivating CuF_2 coating was removed from the bellows under flexing. The trace amount found was detected at the edges of a copper flake which may have originated from the machined parts where apparently some mechanical damage resulted from handling. It should be noted that the copper fluoride was identified as CuF_2 rather than as $CuF_2 \cdot nH_2O$ or CuOHF. It is concluded that contact with atmospheric moisture and its decomposing action was prevented in handling operations. Virtually no parts of the passivating coating were detached due to failing adhesion at the copper-CuF_2 interface. Other crystalline fractions were identified among the particulates, e.g., Fe_2O_3, FeO(OH) and calcium aluminate hydrate approximating $Ca_3Al_2O_6 \cdot nH_2O$.

It is concluded that only trace amounts of the passivating coating are susceptible to the flexing attack. A great majority of the particulate matter collected appears to originate from other parts of the reaction chamber than the passivated copper bellow surfaces. Visual observations of samples from copper bellows Test 3 were similar to those from Test 1 procedure.

I, C, Flexure-Type Tests (cont.)

2. Flexure/Vibration Tests of Metals in Gaseous and Liquid Fluorine at Stress Levels in the Plastic Range (Reference 6)

The effects of the possible loss of the passivation film from the surface of flexing/vibrating parts was evaluated. The general concern was the reduction in cycle life of feed system components caused by possible flaking-off of the passivation film. A preliminary analysis indicated that no loss in cycle life would be expected for feed system components. The limited test results of other investigations agreed with that analysis. To verify the validity of this analysis, a test plan was designed to cycle test specimens to failure in gaseous and liquid F_2 environments. Because absolute fiber stress data were not available on potential test specimens, control runs were made with each specimen configuration, using suitable non-oxidizing media. This provided a form of comparative data.

The convoluted bellows consistute the most common form of flexible joint for state-of-the-art feed systems and was selected for testing. The test plan was designed to evaluate three potential bellows materials (Al 6061-T6, Inconel 625, and Armco 21-6-9) and to test each material at three stress levels. A wide range of stress levels are used in designing conventional convoluted bellows. Commercial grade bellows are normally designed for a cycle life of 10^5 to 10^6 cycles, which result in fiber stresses that are in the elastic range for the material. Aerospace bellows, which are flight-weight feed systems, are normally designed for a cycle life of 10^3 to 10^4 cycles and, therefore, the fiber stress may be in either the elastic or in the plastic range of the material. Stress loadings that loaded the test specimens into the plastic range were selected for these tests. This decision was based on the three following factors: (1) aerospace bellows may be loaded in the plastic range, (2) adverse effects from exposure to F_2 would normally be more pronounced when the specimen is stressed in the plastic range, and (3) test time would be minimized.

The apparatus used for bellows testing consisted primarily of a specimen actuation shaft, which was actuated by an eccentric cam driven by a variable-speed electric motor. The complete drive mechanism was mounted on an unjacketed lid assembly, which was interchangeable with the tank assemblies used for both flow and nonflow type tests.

The original testing concept of using the bellows specimens as the return mechanism for the eccentric drive became invalid when the decision was made to load the specimens into the plastic range. Auxiliary return springs

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Ref. 6 "Development and Demonstration of Criteria for Liquid Fluorine Feed System Components", NASA CR-72063, Douglas Aircraft Co., Santa Monica, Calif., Contract NASw-1351, October 1967.

I, C, Flexure-Type Tests (cont.)

were added to the drive assembly to ensure proper return motion. With the addition of the helper springs it was no longer possible to determine failure of a single test specimen by the loss of proper cam follwer return action. Therefore, it became necessary to incorporate a different test technique to determine the first specimen failure. This alternate method consisted of operating the test assembly for a fixed number of cycles and then stopping the device, while the specimens were examined. This method resulted in test data points that had some scatter because the exact cycle of failure was not detectable.

Testing was conducted in four media: (1) ordinary air at ambient temprature and pressure; (2) LN_2 at atmospheric pressure and normal boiling point (-320°F); (3) GF_2 at ambient temperature and 0.5 psig pressure; and (4) LF_2 at -320°F, pressurized with GHe to 0.5 psig. The data show a consistent reduction in cycle life during the F_2 tests of approximately 30%, as compared to the control runs. This percentage holds true for both ambient and cryogenic testing. The exact cause of the cycle life reductions is not known.

The test results are summarized for each of the test environments (ambient air, liquid nitrogen, gaseous fluorine, and liquid fluorine) and aluminum 6061-T6, Armco 21-6-9, and Inconel 625 hydroformed bellows specimens in Tables XIV, XV, and XVI, respectively. The corresponding data correlations of endurance cycles versus stress level are graphically displayed in Figures 8, 9, and 10, respectively.

TABLE XIV

FATIGUE LIFE OF FLEXED HYDROFORMED ALUMINUM 6061-T6 BELLOWS

Nominal Root Stress Level, psi	Ambient Air	LN_2	GF_2	LF_2
69,900	3 tests, mean value 9 specimens - 4,000	4/4 between 35,000 and 40,000	1/2 between 2,500 and 3,000	2/2 below 25,000
90,000	4/4 between 2,000 and 2,500	---	1/2 between 1,000 and 1,500	2/2 below 15,000

TABLE XV

FATIGUE LIFE OF FLEXED HYDROFORMED ARMCO 21-6-9 BELLOWS

Nominal Root Stress Level	Ambient Air	LN_2	GF_2	LF_2
99,500	2/2 failed between 40,000 and 60,000	---	2/2 failed below 20,000	---
125,000	10,000 cycles, 2 tests, no effect.	100,000 cycles, no effect.	---	---
175,000	2/2 failed between 10,000 and 20,000	2/2 failed below 48,000	2/2 failed between 12,000 and 17,000	1/2 failed below 21,000 1/2 unchanged at 48,000
202,000	---	1/2 failed between 50,000 and 60,000	---	---
268,000	---	2/2 failed between 20,000 and 30,000	1/2 failed between 2,000 and 4,000	2/2 failed between 25,000 and 30,000

TABLE XVI

FATIGUE LIFE OF FLEXED HYDROFORMED INCONEL 625 BELLOWS

Nominal Root Stress Level	Ambient Air	LN_2	GF_2	LF_2
91,000	1/2 between 6,000 and 20,000 1/2 no change, 80,000	--	--	--
147,000	No change 10,000	1/4 between 45,000 and 50,000 3/4 no change, 100,000	--	--
200,000	1/2 failed between 10,000 and 20,000	2/2 below 48,000	1/2 between 12,000 and 15,000	1/2 below 21,000 1/2 no change 48,000
290,000	--	1/2 between 15,000 and 20,000 1/2 between 20,000 and 30,000	--	--
294,000	2/4 failed below 10,000	--	--	--
396,000	--	2/2 between 7,000 and 10,000	2/2 between 2,000 and 4,000	2/2 between 8,000 and 12,000

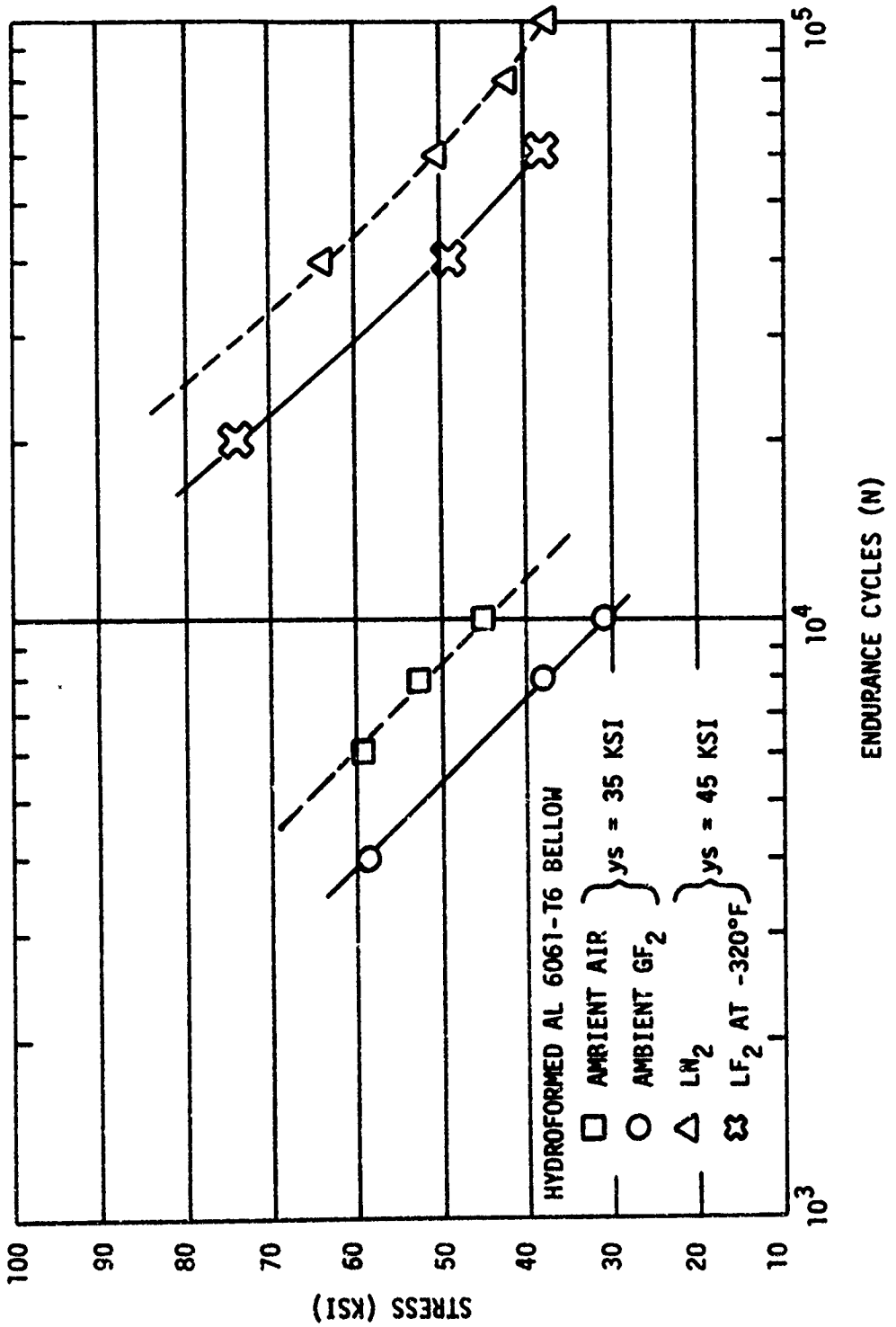

Figure 8. Fatigue Strength of Hydroformed 6061-T6 Aluminum Bellows in Fluorine Environments

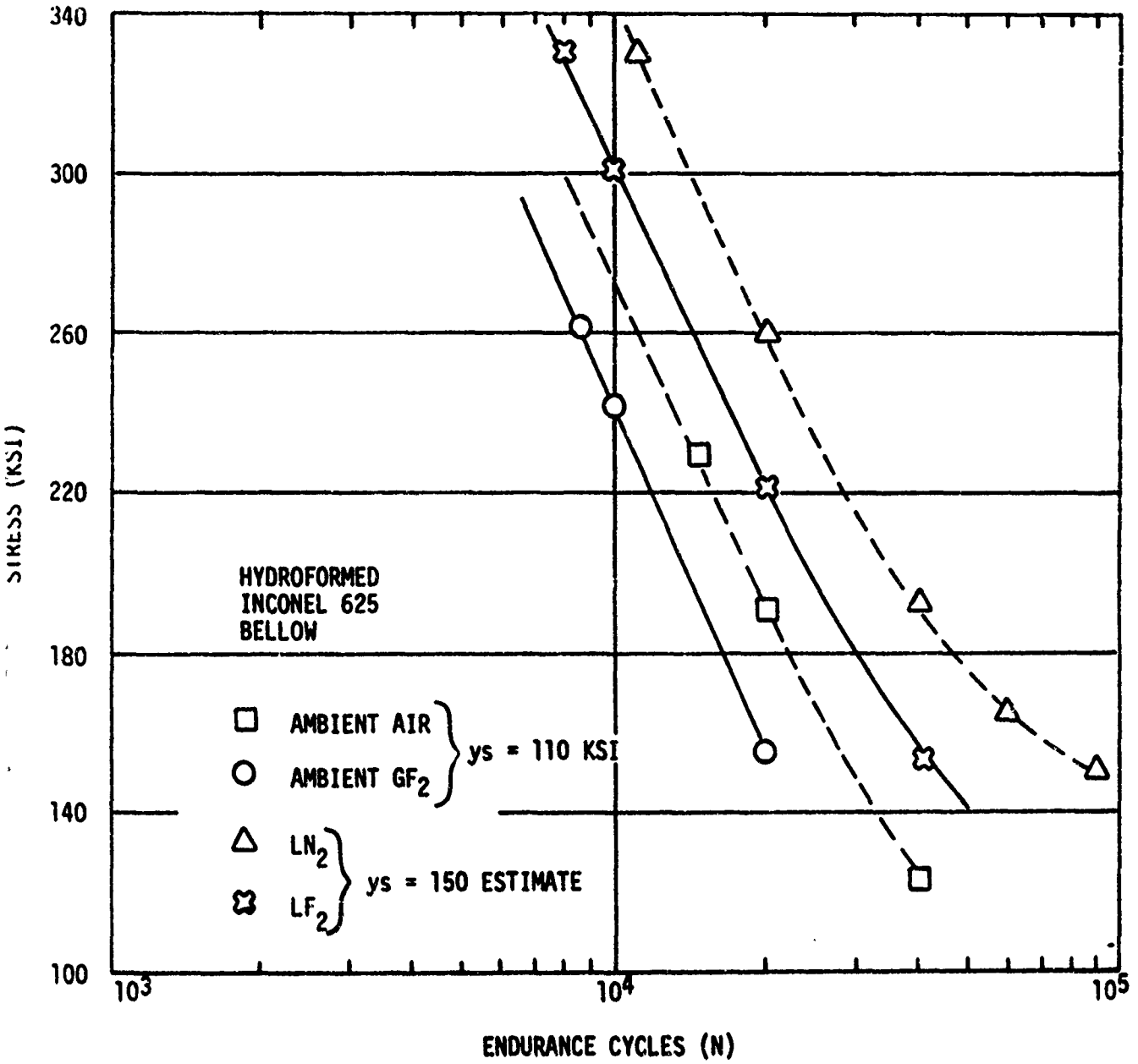

Figure 9. Fatigue Strength of Hydroformed Inconel 625 Bellows in Fluorine Environments

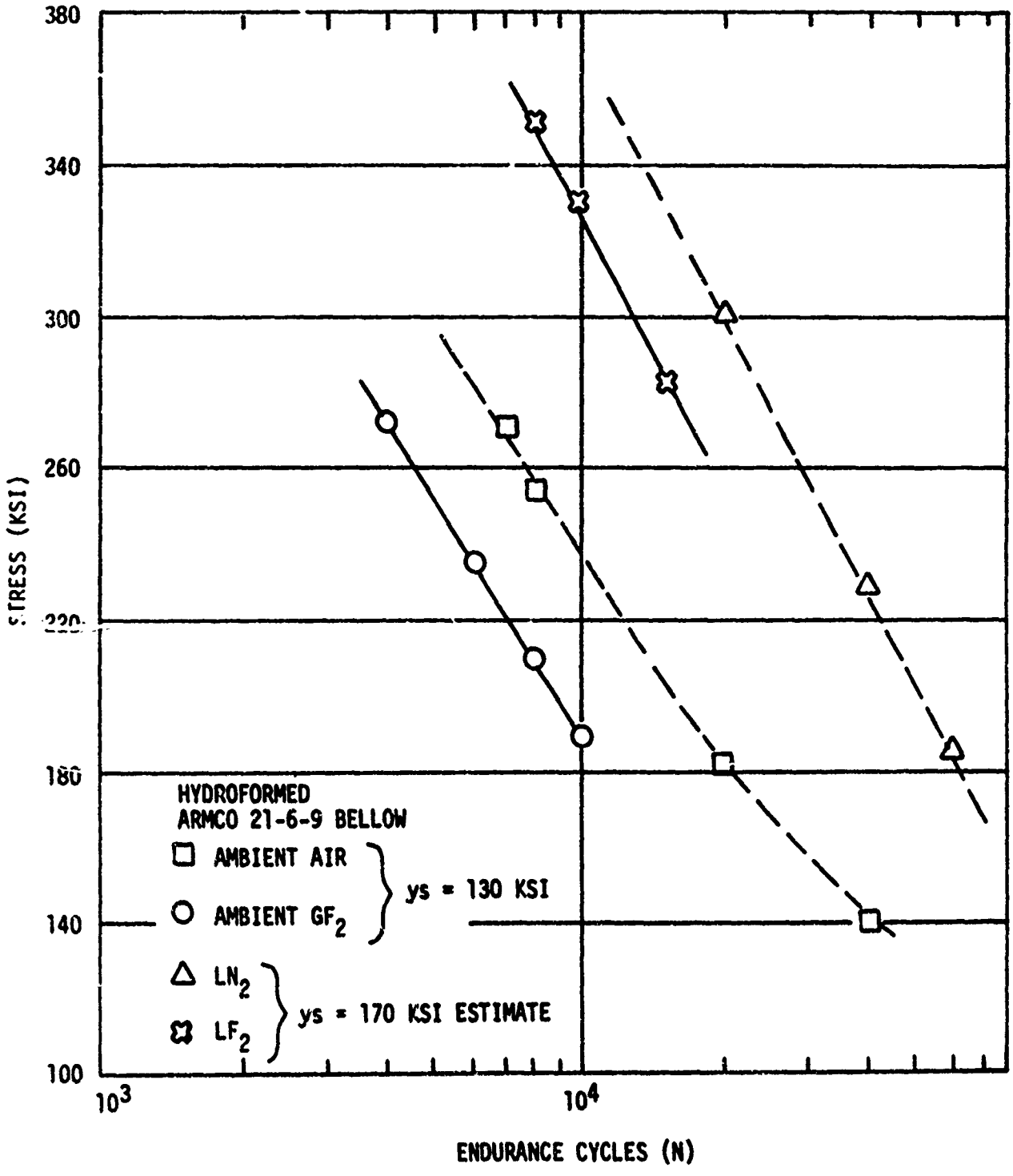

Figure 10. Fatigue Strength of Hydroformed Armco 21-6-9 Bellows in Fluorine Environments

I, C, Flexure-Type Tests (cont.)

3. Flexing Tests in Liquid Fluorine (Reference 9)

The effects of flexing thin metal strips in liquid fluorine were studied by the use of a cell designed for the purpose. The sample strips had one end fixed to the bottom of the cell and the other end fastened to a reciprocating rod. This rod passed through a brass bushing in the top of the cell, and was powered by a solenoid. The solenoid was actuated by a signal from an electric timer which operated at the rate of 1 pulse/sec.

The flexing tests were performed on copper, brass, aluminum, and Monel in liquid fluorine to provide some information on the flexibility of a protective fluoride film. If increased corrosion resulted, this would have provided evidence that a fluoride film was present and that the film was not flexible. The negative results actually obtained indicate that either the protective fluoride film is flexible or that no fluoride film is required for protection.

The results of these tests are summarized in Table XVII.

Ref. 9 Sterner, C. J., and Singleton, A. H., "The Compatibility of Various Metals and Carbon with Liquid Fluorine", WADD-TR-60-436, Air Products, Inc., Contract AF 33(616)-6515, August 1960.

TABLE XVII

RESULTS OF FLEXURE TESTS IN LIQUID FLUORINE

Test Metal	Testing Time	Initial Sample Wt.	Final Sample Wt.	Remarks
Yellow Brass	90 min	.9084 gm.	.9075 gm.	Sample was about 3-1/2" x 3/8" x .010". The only apparent change was a sparse white coating deposited uniformly along the sample. Microscopic examination at 60X revealed no further changes in the metal.
Copper	300 min	.9078	1.0840 gm.	Dimensions same as above. Due to improper attachment of the sample in the flexure cell, the sample broke at the bottom attachment sometime during the test. The breaking was not apparently accelerated by the liquid fluorine. The sample's final appearance was similar to that given above. Visual and microscopic examination failed to reveal any corrosion or reaction of the magnitude indicated by the weight change.
Copper	240 min.	.9003 gm.	.9019 gm.	Sample was about 3-1/2" x 3/8" x .010". Sample appeared bright and shiny after exposure; microscopic examination added no further information.
Monel	290 min.	.3216 gm	.3236 gm.	Sample was about 3-1/2" x 3/8" x .002". The only apparent change was the appearance of some very sparse white splotches on the convex side of the flexure sample. Microscopic examination at 60X revealed no further changes in the metal.
Alcoa Aluminum Foil	210 min	.0358 gm.	.0373 gm.	Sample was about 3-1/2" x 3/8" x .001". Sample appeared bright and shiny after exposure; microscopic examination added no further information.

I, Fluorine Dynamic Compatibility (cont.)

D. VIBRATION/CAVITATION TYPE TESTS

1. Ultrasonic Vibration/Cavitation Testing of an Aluminum Casting Alloy in Liquid Fluorine (Reference 6)

The aluminum casting alloy, A356-T6, was exposed to liquid fluorine (0.02% vol HF) at -320°F and subjected to vibration at 27.8 kilocycles/sec in an ultrasonic mechanical stepped horn apparatus in repeated cycles of 1/2 hr on and 3 hr off. Similar tests were conducted with water and liquid nitrogen for comparison. The average sample weight changes in water, LN_2 and LF_2 were 0.01, 0.02, and 0.04 mg/cm^2-min, respectively. Average corrosion pit depths in LN_2 and LF_2 were 18 and 40 microns, respectively. These tests show that liquid fluorine is somewhat more aggressive than water or LN_2 in an ultrasonically vibrated environment.

Ref. 6 "Development and Demonstration of Criteria for Liquid Fluorine Feed System and Components", Final Report, NASA CR-72063, Douglas Aircraft Co., Santa Monica, Calif., Contract NASw-1351, October 1967.

I, D, Vibration/Cavitation Type Tests (cont.)

2. Vibration Tests in Fluorine (Reference 9)

Tubes made of copper, brass, stainless steel, nickel and Monel which had been filled with liquid fluorine and impacted were immediately filled with gaseous fluorine after the impact test and subjected to a vibration test. The vibration test conditions were 30 cycles/sec at 5 mm amplitude for periods up to 5 hr. In eight such tests, the vibrations produced no observable effect on the tubes.

Ref. 9 Sterner, C. J., and Singleton, A. H., "The Compatibility of Various Metals and Carbon with Liquid Fluorine", WADD-TR-60-436, Air Products Inc., Contract AF 33(616)-6515, August 1960.

I, Fluorine Dynamic Compatibility (cont.)

E. FRICTION, ABRASION, AND FRACTURE TYPE TESTS

1. Friction Testing of Silver - 316 CRES in Gaseous Fluorine (Reference 7)

Theoretical analysis indicates that a coefficient of friction of 0.40 would be expected for dissimilar metals operating in air and a value of 0.10 to 0.18 with a fairly good lubricant. To evaluate the silver-stainless steel combination in a fluorine environment, a preliminary test run was conducted using a frictional-energy test device. To conduct this test, a frictional-energy test disk was refinished and given a silver plate. During the refinishing, a series of ventilation slots were cut into the test specimen so that the GF_2 exposure of the contact surfaces could be assured.

After installation of the test specimen in the apparatus, the test section was pressurized with ambient gaseous fluorine and a series of friction runs was conducted up to the capacity of the machine. The results of the tests are shown on Table XVIII. During the last two tests, the samples were loaded so that the mechanism could not complete enough travel for a realistic value of friction coefficient to be determined. Post-test inspection of the test sample showed no evidence of galling on the specimen after all five tests. The silver fluoride film was a yellow-green fine-grained coating which transferred from the silver to the 316 CRES in small quantities. This is an acceptable condition for dry film lubrication. The measured coefficients of friction of 0.08 to 0.20 indicate that the fluoride film provides a reasonable measure of lubrication.

TABLE XVIII

RESULTS OF FRICTION TESTS OF SILVER-316 CRES IN GASEOUS FLUORINE

Test No.	Load, psi	Coefficient of Friction
1	1,000	0.20
2	1,180	0.17
3	1,980	0.08
4	4,000	--
5	6,050	--

Ref. 7 "Development and Demonstration of Criteria for Liquid Fluorine Feed System Components", NASA CR-72543, McDonnell Douglas Astronautics Co., Huntington Beach, Calif., Contract NAS 3-11195, June 1969.

I, E, Friction, Abrasion, and Facture Type Tests (cont.)

2. Abrasion Tests of Metals in Liquid Fluorine (Reference 10)

Film disrupting studies were made in which metal surfaces were continually wire brushed while immersed in liquid fluorine. The tests were carried out in a Monel cell 1-1/4 in. ID x 4 in. deep. A wire brush 1/2 in. x 1/2 in. was made with fine stainless steel bristles about 1/4 in. long and attached to a 1/4 in. Monel rod which passed through a brass bushing in the top of the cell. The metal sample, about 2 in. x 3/4 in. was supported in the bottom of the cell at an angle of about 30° with the vertical. The brush was caused to move up and down on the sample by a solenoid outside the cell. The rate was 1 stroke/sec and the travel was about 1 in. The monel rod was hinged in the middle so that the brush was free to travel along the surface of the sample, and the force exerted by the brush on the sample was constant over the whole stroke. This normal force was due largely to gravity, and was about 0.05 lb. A helium purge was maintained on this cell during operation to prevent air from diffusing into the cell through the brass bushing.

Ten tests were made with copper and two with magnesium alloy AZ-31. In addition, three blank tests were made with copper and one with magnesium. The results of these tests are presented in Table XIX. These samples were exposed to liquid fluorine in exactly the same manner as the other samples except that the brush was not attached to the rod.

While the weight gains on these abrasion tests are scattered from 0 to 10 mg, the average for the copper and the magnesium samples is about the same as the weight changes observed on the blanks. Because the cell was not air tight, the condensation and evaporation of fluorine had to be carried out at quite a slow rate. Hence, even with the helium purge, it is quite likely that small quantities of air with its accompanying moisture were able to diffuse into the cell and cause corrosion of a slightly higher level than is commonly observed in the continuous immersion tests. The abrasion was severe enough to remove any protective fluoride film on the metals. The fact that no increase in corrosion was observed indicates that film protection is not the controlling mechanism in the corrosion resistance of these metals to liquid fluorine.

Ref. 10 Sterner, C. J. and Singleton, A. H., "The Compatibility of Various Metals with Liquid Fluorine", WADD-TR-60-819, Air Products, Inc., Contract AF 33(616)-6515, March 1961.

TABLE XIX

RESULTS OF ABRASION TESTS OF METALS IN LIQUID FLUORINE

Sample	Alloy	Initial Weight, gm	Weight Change, mg	Exposure Time, hr	Remarks
1	Copper	28.2264	0.1	1	All samples, except where
3	Copper	14.3360	0.9	1	otherwise designated, were
6	Copper	20.7717	5.2	1	brushed with a coarse stain-
8	Copper	22.3695	9.9	1	less steel brush continuously
9	Copper	20.7949	3.8	1	at one stroke/second while
10	Copper	21.8593	4.3	1	under liquid fluorine.
13	Copper	21.5263	-0.3	1	
14	Copper	22.2546	4.1	1	
15	Copper	22.2243	8.4	1	
16	Copper	22.1763	5.0	1	
		Avg.	4.14 (σ = 2.42)		
21	Copper	15.3295	3.9	1	*
22	Copper	20.8356	8.0	1	*
23	Copper	20.6760	1.0	1	*
		Avg.	4.3 (σ = 2.47)		
11	Mg AZ-31	5.8931	1.5	1	
17	Mg AZ-31	9.1126	2.9	1	
		Avg.	2.2		
20	Mg AZ-31	6.1560	2.0	1	*

* These samples were tested in the abrasion apparatus without brushing.

I, E, Friction, Abrasion, and Fracture Type Tests (cont.)

3. Tensile Fracture Testing of Various Metals in Static Liquid and Flowing Gaseous Fluorine (Reference 12)

Specimens of ASM 6434 steel; AM 350, 301, and 304L stainless steel; 2014-T6, 6061-T6, and 7075-T6 aluminum, and Ti-6Al-4V titanium were fractured in liquid fluorine at -320°F in the course of tensile testing. There was no evidence of ignition on any of the specimens.

Specimens of AM 350 stainless steel and Ti-6Al-4V titanium were similarly fractured in a high-velocity jet of gaseous fluorine at approximately 75°F. No indications of metal reaction were observed.

Ref. 12 Richards, H. T. and Hanson, M. P., "Influence of Fluorine Environment on the Mechanical Properties of Several Sheet Alloys", NASA TN D-1706, Lewis Research Center, Cleveland, Ohio, April 1963.

I, E, Friction, Abrasion, and Fracture Type Tests (cont.)

4. Tensile Tear Tests in Liquid Fluorine (Reference 9)

Tensile tear tests of metals immersed in liquid fluorine were conducted with titanium, brass, copper, Monel, and aluminum. The titanium samples were 1/4 in. diameter rods which were machined in the center to about 0.030 in. diameter. The rod was threaded on both ends with one end screwed into a socket at the bottom of the test cell. The other end was fastened to a Monel metal rod which in turn protruded through a bushing in the top of the cell. A helium purge was used to reduce atmospheric contamination. A hand-operated lever fastened to the Monel rod was used to tear the sample after fluorine had been liquified into the cell. The other four metals were tested in the same equipment, but the samples were in the form of thin strips 3 in. x 1/4 in. x 0.005 in. The torn edges of the samples were examined microscopically at 60X.

These tests were intended to present a freshly fractured surface to the liquid fluorine environment. Thus, there could be no possibility of a fluoride film protecting the surface at the time of fracture. It was expected that resulting ignition or corrosion products would indicate the need of a film for protection. One ignition was obtained in six tests with titanium and this would tend to support the protective film theory. However, the ignition can also be explained by a process which involves the straining heating of the specimen to its ignition temperature.

No apparent reaction occurred when thin metal strips of copper, brass, Monel or aluminum were torn in tension under liquid fluorine. Minor ignition and a slight explosion occurred with one sample out of six tests with titanium. The ignition was barely observable at one edge of the fracture. The other five samples produced no indication of a reaction.

Ref. 9 Sterner, C. J. and Singleton, A. H., "The Compatibility of Various Metals and Carbon with Liquid Fluorine", WADD-TR-60-436, Air Products Inc., Contract AF 33(616)-6515, August 1960.

I, Fluorine Dynamic Compatibility

F. EXPLOSIVE SHOCK TESTS

1. Explosive Shock Test of an Aluminum Casting Alloy in Liquid Fluorine (Reference 6)

An aluminum casting alloy, A 356-T6, Grade III-F, was exposed to an explosive shock of 120,000 psi after a 24-hr soak in LF_2 (0.02% vol HF) at -320°F using a test apparatus of the card-gap type. The test resulted in a reaction with no trace of the test coupon remaining.

Ref. 6 "Development and Demonstration of Criteria for Liquid Fluorine Feed System Components", Final Report, NASA CR-72063, Douglas Aircraft Co., Santa Monica, Calif., Contract NASw-1351, October 1967.

I, F, Explosive Shock Tests (cont.)

2. Explosive Shock Tests of Liquid Fluorine and Various Metals (Reference 10)

Tests were made to investigate the possibility of metal ignition in liquid fluorine under conditions of explosive shock. The runs were carried out in metal tubes of 2 in. x 5/8 in. ID x .065 in. wall thickness, with the tubes themselves being the samples. The tubes were closed at the ends by brass plugs silver soldered in place and fluorine was added through 1/4 in. copper tube mounted in the sample tube wall. Two tubes of each materials were tested, including Monel, nickel, brass, copper, 304 SS, 316 SS, 347 SS, and 1100 aluminum.

The experimental procedure was as follows:

(1) The 1/4 in. tube was attached to the fluorine gas manifold and a No. 6 electric blasting cap was tightly fastened to the tube wall with nichrome wire.

(2) An insulated can was positioned and liquid nitrogen was added to the can, covering the cell completely.

(3) Fluorine gas was condensed into the cell, filling it completely with liquid.

(4) Ten minutes after the tube was filled, the blasting cap was set off.

The ends of the tubes were usually blown out by hydrostatic pressure generated by the deformation of the tube walls, but no evidence of fluorine reaction was observed in any run.

The reactivity of titanium fillings in liquid fluorine was examined by placing filings in small Monel tubes, adding liquid fluorine, and exploding a dynamite cap outside the tube. Three tests were run. In two cases there was no apparent explosion of the titanium, although the filings were completely transformed to the white titanium tetrafluoride. In the third case, a severe detonation occurred before the cap was exploded, and the tube was torn longitudinally from the force of the blast. No trace of the filings or reaction products was found.

Ref. 10 Sterner, C. J. and Singleton, A. H., "The Compatibility of Various Metals with Liquid Fluorine", WADD-TR-60-819, Air Products, Inc. Contract AF 33(616)-6515, March 1961.

SECTION II

DYNAMIC COMPATIBILITY OF CHLORINE FLUORIDES

A. FLOW-TYPE TESTS

1. Monel Tube Burnout in High Pressure and Velocity ClF_5 Environments

In the course of determining the forced convection heat transfer characteristics of liquid ClF_5 in Monel K-500 tubes, three out of twenty-two tests were terminated by tube burnouts (Reference 13). In two of these tests, wall temperature measurements were available essentially at the time of tube burnout, while in the other case, the temperature measurement was lost shortly before burnout. The pertinent test conditions at essentially the time of burnout are summarized in Table XX and compared with that of tests at similar velocities and Reynolds Numbers but in which tube burnout did not occur at maximum temperature.

There were insufficient tests which were terminated by tube burnouts to establish a definite correlation between burnout temperature and some operating condition but the limited data do suggest that velocity or Reynolds Number is the best correlating parameter.

The limited data do show that Monel K-500 burns out in ClF_5 environments at approximately 1950°F where the superficial velocity is ~50 ft/sec and Re is ~2.2×10^5 and at approximately 1760°F where the superficial velocity is ~100 ft/sec and Re is ~6.7×10^5.

Ref. 13 Rousar, D. C., et al, "Heat Transfer Study of ClF_5", Final Report AFRPL-TR-68-53, Aerojet-General Corp., Sacramento, Calif., Contract F04611-67-C-0016, April 1968.

TABLE IX

MONEL K-500 TUBE BURNOUTS IN HIGH PRESSURE, HIGH VELOCITY LIQUID ClF_5 ENVIRONMENTS

Test No.	Heat Flux, Btu/in.^{2}-sec	Bulk ClF_5 Conditions				Tube ID, in.	Inside Wall Temp., °F	Results
		Reynolds Number	Velocity, ft/sec	Pressure, psia	Temp., °F			
104	1.49	1.9×10^5	18.9	529	122	0.209	>1580	Tube burnout
120	1.17	1.7×10^5	16.6	347	88	0.210	1564	No tube failure
116	6.53	2.2×10^5	48.8	594	7	0.137	~1948	Tube burnout
106	4.50	2.3×10^5	20.1	2015	165	0.209	1972	No tube failure
119	2.69	3.0×10^5	48.7	357	73	0.137	1594	No tube failure
108	22.8	6.7×10^5	100.4	2000	145	0.135	~1759	Tube burnout
122	13.4	5.9×10^5	64.5	1966	213	0.136	1785	No tube failure
114	20.7	6.3×10^5	98.7	1978	143	0.137	1293	No tube failure

II,A, Flow-Type Tests (cont.)

2. Ignition Temperature of Copper and 304 Stainless Steel in Flowing ClF_5 (Reference 14)

In the process of determining optimum passivation temperatures for preparing copper and 304 stainless steel for ClF_5 service, flow tests in previously passivated tubes of copper and 304 stainless steel were run to their ignition temperatures. The apparatus used consisted of an electrically heated tube furnace containing a Vycor tube. The test section was a 27-in. long section of 3/8-in. OD tubing and passed through the Vycor tube. A thermocouple was welded to the outside wall in the center of the test section. In operation the test section was heated to the desired passivation temperature (~20°C, 100°C, or 200°C) and gaseous ClF_5 at 20 psig was flowed through the section for 20 min. After passivation, the ClF_5 was purged from the test section with nitrogen. Power to the furnace was increased so that the temperature of the test section increased at 6 to 12°C/min. When the temperature reached 600°C, the flow of gaseous ClF_5 was resumed with the pressure being maintained as before at 20 psig. The ignition temperature was taken as the point at which burn-through was audibly apparent (and where a time-temperature plot dramatically changed slope).

Duplicate tests using copper tubing were completed at each passivation temperature and with 304 stainless steel using a 20°C passivation. The results are summarized in Table XXI.

TABLE XXI

IGNITION TEMPERATURES OF COPPER AND 304 STAINLESS STEEL IN FLOWING ClF_5

Test Material	Passivation Temp., °F	Ignition Temp., °F*
Copper (3/8-in. OD Tubing)	68	1445
	212	1598
	392	1463
304 SS (3/8-in. OD Tubing)	68	1218

*Average of duplicate tests

Ref. 14 Johnson, W. H. and Lawrence, R. W., "Investigation of Advanced Hybrid Propellants (U)", Final Report No. 3163, Aerojet-General Corp., Azusa, Calif., Contract NOw 65-0309-C, February 1966.

II,A, Flow-Type Tests (cont.)

3. High Velocity Impingement and Flow Tests of ClF_3 On and In Various Metals (Reference 15)

A test was devised to determine the relative resistance of eleven metals alloys to impingements of liquid chlorine trifluoride, at very high flow velocities and high temperatures. Plate specimens, approximately 4 in. x 4 in. x 3/8 in. of the alloys were subjected to a hot, high velocity jet of liquid ClF_3 formed by ejecting the liquid through a 3/32-in. orifice. A propellant powder charge was used to generate the high pressure gas used to expel the ClF_3. Expulsion required only a few milliseconds, and jet velocities of many hundreds of ft/sec were achieved.

The test device consisted of three sections: (1) an upper section contained the powder charger and the electrical squib, (2) a center section contained the liquid ClF_3 held between two copper rupture discs, and (3) the lower section formed an "ignition chamber" which in most tests contained a small ball of steel wool which served as an igniter, and the 3/32-in. dia orifice. All parts of the tool were made of steel except the discharge head and the rupture discs, which were made of copper. All tests were run using the tool in a vertical position with the jet directed down and accurately spaced from the test metal target.

The results of 13 tests are summarized in Table XXII. A bright white flash accompanied all the aluminum tests. During the jet impingements of the iron alloys, a dull yellow flash and a red-brown puff of smoke was observed. The copper, Nickel 200 and Monel 400 tests showed no flash of light; there was a puff of white smoke and the odor of unreacted chlorine trifluoride.

When the volume of metal lost during the tests is taken as the criterion of attack, the order of decreasing resistance of the metals tested is: copper, nickel, monel, 347 SS, AM 355, 1020 carbon steel, PH15-7Mo, 410SS, 6061 aluminum, 1100 aluminum, 2024 aluminum. There was little difference between copper and nickel; both were very resistant to attack under the conditions used. Carbon steel 1020, 410 stainless steel, and PH 15-7Mo were all about equally attacked. The aluminum targets were the only ones that were cut through. Aluminum 6061 was somewhat less attacked than was 1100 and 2024.

Ref. 15 Grigger, J. C. and Miller, H. C., "The Compatibility of Welded Structural Materials with Chlorine Trifluoride, Perchloryl Fluoride and a Mixture of These", AFML-TDR-64-22, Pennsalt Chemical Corp., King of Prussia, Penn., Contract AF 33(657)-8461, Feb. 1964.

TABLE XXII

TEST RESULTS OF ClF_3 IMPINGEMENT ON METALS

Metal Specimen	Metal Loss, cm^3*
Copper (ASTM-B133)	0.0
Nickel 200	0.01
Nickel 200	0.06
Monel 400	0.62
347 Stainless Steel	1.4
AM 355 Stainless Steel	1.7
1020 Carbon Steel	1.9
PH 15-7 Mo Stainless Steel	2.0
410 Stainless Steel	2.1
6061 Aluminum	3.7
1100 Aluminum	4.1
1100 Aluminum	4.3**
2024 Aluminum	4.7

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* Metal loss normalized to average ClF_3 charge of 88g.

** No steel wool igniter used in this test.

II,A, Flow-Type Tests (cont.)

A second type of dynamic ClF_3 flow test was performed using a high pressure piping loop in which metal targets were exposed to a variety of flow conditions. The liquid oxidizer was driven through the piping loop by helium gas pressure. Two steel cylinders, each capable of holding up to 54 lb of liquid oxidizer pressurized to 1500 psig with helium gas, served as feed tank and receiver. In carrying out a flow test, compressed helium gas was used to force the liquid out of one cylinder through an air-operated safety valve, a liquid flow control valve, a special all-metal flow meter, the test chamber, and then to the second cylinder; after which the liquid was recirculated through the test chamber, and returned to the first cylinder. A continuous flow at 100 ft/sec through a 1/64-in. ID tube could be maintained for one hour. By periodically reversing the cylinders, tests for longer than one hour, or one-hour tests through larger tubes, could be made. The test specimens were held between two flanged discs connected to the rest of the apparatus by a short section of flexible tubing. This allowed several specimens to be run in series or specimens of different sizes to be run without upsetting the rest of the apparatus.

The test specimens were machined from 1-3/8-in. circular discs of the sheet metal nominally 1/8-in. thick. Two test configurations were used: (1) an orifice .020 in. diameter and .020 in. long; (2) a channel embodying two right angle turns. The second configuration was made by cutting a slot and an orifice in one disc and an offset orifice in a second disc. This arrangement of targets allowed close inspection of the metal after the exposure. Very slight erosion could be detected by changes in the markings on the metal surfaces. while larger effects could be measured by changes in the dimension of the orifice or slot.

Aluminum, nickel, and stainless steel targets were tested with ClF_3. The results of these flow tests are given in Table XXIII. No erosion of the orifice target (Type 1) was noted with ClF_3 on aluminum, nickel, or stainless steel targets. Flows of at least 100 ft/sec were maintained for one hour or longer. A Teflon orifice was seriously attacked by ClF_3 flowing at less than 100 ft/sec in less than 30 min. The only observation of attack on any of the metal specimens was on a Type 2 target made from 2014 aluminum and may be attributed to the impingement of solid particles that were inadvertently introduced into the ClF_3.

TABLE XXIII

TEST RESULTS OF ClF_3 HIGH PRESSURE FLOW TESTS

Type of Flow Path	Specimen Material	Liquid ClF_3 Flow Data: Weight, lb	Volume, $in.^3$	Velocity, ft/sec	Time, min.	Results and Remarks
Orifice (Type 1)	Al 1100	35	620	?	54	Orifice distorted by pressure, no chemical attack.
	Al 2014-T6	82	1474	90	72	No orifice change.
	Al 2014-T6	119	2121	126	74	No orifice change.
	Al 6061-T6	97	1745	114	67	No orifice change, slight stain on surface.
	Nickel 200	97	1745	114	67	"
	347 S.S.	97	1745	114	67	"
	410 S.S.	81	1450	106	60	No orifice change.
	Teflon	35	629	80	35	Orifice eroded.
Double right angle channel (Type 2)	Al 2014-T6	76	1358	124	48	Orifice unchanged, small pit at impingement point.
	Al 2014-T6	66	1188	100	52	Very slight pit at impingement point.
	Nickel 220	56	1000	81	51	Channel distorted by pressure, no chemical attack.
	410 S.S.	93	1670	106	69	Stain on surface, no other sign of attack.
Inverted T with dead end (1/4-in. tube)	Al 6061-T6	54	968	6	65	Flow stopped and started every 5 sec for 5 min period, no sign of attack.

II,A, Flow-Type Tests (cont.)

4. Impingement of Gaseous Chlorine Pentafluoride on Heated Metals

The effect of gaseous ClF_5 impingement on heated samples of 2021 aluminum and 304 and 347 stainless steels has been briefly summarized in Reference 16. This is not the original source of the information. The results, as interpreted from Reference 16, are summarized below.

Material	Material Temperature, °F	Material Response
2021 Aluminum	1125	Little or no attack.
	1185	Modest attack.
304 Stainless Steel	1078	Moderate attack.
	1502	Moderate attack.
347 Stainless Steel	1125	Modest attack.
	1150	Modest attack.

The above data indicate the onset of at least some attack in the presence of impinging ClF_5 gas occurs in the range of 1080 to 1185°F in each material.

It can be seen that 304 stainless steel is only moderately attacked at approximately 1500°F according to these data and is in considerable disagreement with the reported ignition of 304 S.S. in flowing gaseous ClF_5 at 1218°F given in Reference 14 of this appendix (see Appendix A, Table XXI).

Ref. 16 "Interhalogen Handbook", Final Report, Technical Report AFRPL-TR-67-276, Rocketdyne, Canoga Park, California, Contract F04611-67-C-0006, November 1967.

II, Dynamic Compatibility of Chlorine Fluorides (cont.)

B. IMPACT AND FRICTION-TYPE TESTS

1. Impact and Rotary Friction Tests with ClF_5 and Various Materials (Reference 17)

Dynamic compatibility tests of liquid ClF_5 (-10°F) with silver-plated Duranickel 301, 304-L stainless steel, Duranickel 301, and Carmet CA-4 (tungsten carbide in cobalt) were conducted with a modified ABMA impact tester and a rotary friction tester. The energy necessary to cause initiation of surface reactions on each device was determined. The Duranickel 301 was tested in several hardnesses to determine the effect of hardness on initiation. Stainless steel 304-L was tested in the air-melt and vacuum-melt conditions to investigate the effects of impurities. The following conclusions were reached.

(1) Initiation of burning occurred only when sufficient energy was introduced to cause damage by plastic deformation.

(2) The minimum energy required was a linear function of both the yield strength and the hardness of Duanickel 301.

(3) The purer (vacuum melt) stainless steel 304-L required greater energy to initiate a reaction.

(4) The interface temperatures on the specimens were calculated from the energy supplied in the ABMA tester. In the tests where reaction occurred, the temperatures calculated were very close to those reported for thermal ignition of the metals. In tests with no reaction, the temperatures calculated were much lower.

(5) The silver-plated Duranickel 301 was found to be unsatisfactory as an ACS valve closure material, but this was traced to carbon and sulfur impurity inclusions in the plated layer of silver. No conclusions could be drawn as to the suitability of good quality silver plate.

(6) The tungsten carbide-cobalt cermet was found to be the most satisfactory material tested for use in ACS valve closures and there are indications that its ignition temperature in ClF_5 is higher than most metals.

The impact and rotary friction test data are summarized in Table XXIV.

Ref. 17 English, W. D. and Samuel, H. D., Jr., "Failure Mechanism of Attitude Control System Valve in Chlorine Pentafluoride Service", AFML-TR-71-94, McDonnell Douglas Astronautics Company, Contract F33615-70-C-1760, July 1971.

TABLE XXIV

DYNAMIC COMPATIBILITY OF LIQUID CPF AT 10°F WITH VARIOUS MATERIALS AS DEFINED BY IMPACT AND ROTARY FRICTION TESTS

Material	Vickers Hardness, Kg/mm²	Compressive Yield Strength, psi	Modified ABMA Impact Tests: Threshold Initiation: Impact Energy, ft-lb	Threshold Initiation: Interfacial Energy Flux,(1) ft-lb/sec	Threshold Initiation: Interfacial Temp,(1) °F	50% Reaction, E_{50}: Impact Energy, ft-lb	50% Reaction, E_{50}: Interfacial Energy Flux,(1) ft-lb/sec	50% Reaction, E_{50}: Interfacial Temp,(1) °F	Rotary Friction Initiation Tests: Initiation Range(3): Net Energy Input, ft-lb	Rotary Friction Initiation Tests: Initiation Range(3): Interfacial Energy Flux, ft-lb/in²-sec
Silver-Plated Duranickel 301	50(2)	-	<110	-	-	-	-	-	? to 3.5	-
304-L Stainless Steel,										
Air Melted	89	60,000 to 68,000	228	300	393	241	795	1075	2.7 to 4.7	97 to 168
Vacuum Melted	89	57,000 to 66,200	228	300	393	239			6.5 to 8.3	279 to 298
Duranickel 301,										
Annealed	147	30,000	238	319	450	252	1410	2090	17.4 to 23.1	624 to 829
Work Hardened	179	45,000	238	319	450	264	1450	2155	-	-
Thermally Aged	247	78,000	256	334	472	286	1530	2270	-	-
3/4 Hard	326	118,000	293	355	503	318(1)	1590	2365	20.4 to 28.3	733 to 1020
Full Hard	355 - 360	135,000	-	380	540	330(1)	1620	2410	23.7 to 38.8	850 to 1670
Carmet CA-4 (Tungsten carbide, 94%, in Cobalt, 6%	1800(2)	--	220	1270	2045	233	1940	3135	32.0 to ?	-

(1) Calculated values
(2) Nominal values
(3) First value is the most severe condition at which no reaction was observed and the second value is the least severe condition that reaction could be detected. Actual threshold and 50% values lie somewhere within the ranges noted.

II, B, Impact and Friction-Type Tests (cont.)

2. Impact Tests on Aluminum and Titanium Alloys in ClF_5 (Reference 8)

Modified ABMA open-cup impact tests were conducted on Al 2014-T6 in liquid ClF_5 at a temperature between -100°C and its boiling point. The first six drop tests were conducted at 70 ft-lb and a temperature between -100°C and -50°C. Two out of five tests gave faint flashes. The sixth test was a check test run without oxidizer. No reaction occurred. The source of the observed flashes was traced to frozen ClF_5 particles floating in liquid ClF_5 contained in the Al 1100-0 cup and outside of the Al 1100-0 cup in the liquid nitrogen droplets in the moat. Identification of the location of the flashes indicated that Al 1100-0 was impact sensitive to solid ClF_5 in LN_2 and Al 2014-T6 to solid ClF_5 in liquid ClF_5. The solid ClF_5 formation was eliminated by increasing the moat base temperature from -100°C to -50°C. The seventh drop test was negative, and the eight test was negative. A faint spark was observed on the rebound outside the moat area. A test procedure for bracketing the 50% probability of initiating a reaction was attempted, beginning with the tenth drop test. One positive reaction occurred at 69 ft-lb out of 16 tests.

Modified ABMA open-cup impact tests on annealed Ti-6Al-4V (ELI) were conducted in liquid ClF_5 at the boiling point (6.7°F). There were 15 reactions out of 29 tests. The impact energy required for a 50% probability E_{50}, or in terms of drop height (H_{50}) of the alloy samples to initiate a reaction in liquid ClF_5 was calculated to be 42.5 ft-lb.

The results of these tests are summarized in Table XXV.

Ref. 8 Toy, S. M., Bell, L. E., English, W. D., and Tiner, N. A., "Tankage Materials in Liquid Propellants", AFML-TR-68-204, McDonnell Douglas Corp., Contract F33615-67-C-1718, July 1968.

TABLE XXV

IMPACT TEST RESULTS ON 2014-T6 ALUMINUM AND 6Al-4V(ELI) TITANIUM IN THE PRESENCE OF LIQUID ClF_5

Specimen Material	Striker Material	Sample Cup Material	ClF_5 Temp., °F	Energy Level, ft-lb	Number of Tests		
					Positive	Negative	Total
Al 2014-T6	17-4 PH	Al-1100	-148	70	2*	3	5
			-58	35	0	1	1
			-58	52.5	0	1	1
			-58	65.4	0	1	1
			-58	65.8	0	1	1
			-58	67	0	1	1
			-58	68	0	1	1
			-58	69	1	15	16
			-58	70	0	2	2
Ti 6Al-4V (ELI)	Ti 6Al-4V (ELI)	Ti 6Al-4V (ELI)	6.7	$\leq$42	0	9	9
			6.7	42.5	1	0	1
			6.7	43	1	0	1
			6.7	45	1	0	1
			6.7	46.5	1	0	1
			6.7	51	1	0	1
			6.7	51.5	0	1	1
			6.7	52	0	1	1
			6.7	52.5	1	2	3
			6.7	53	0	1	1
			6.7	54	1	0	1
			6.7	56	1	1	2
			6.7	$\geq$56.5	7	0	7

*These positive tests had $ClF_{5(s)}$ present.

II, B, Impact and Friction-Type Tests (cont.)

3. Impact Tests of Various Metals in ClF_5 (Reference 18)

Impact tests were run on aluminum 2014-T6, 347 stainless steel, Nickel 200, and magnesium AZ 31B in liquid ClF_5 at 86°F using a pressure type impact tester. The striker was made from 1/8-in. sheet and cut with a 77° point. It was impacted on a 1/8-in. thick base or anvil plate at energy levels from 59.4 to 71 ft-lb. For most tests the striker and anvil plate were made from the same metal. A 150-gm charge of ClF_5 used for each test. This was sufficient liquid to cover the base plate to a depth of about 3/8-in.

No signs of ignition were seen for duplicate runs of the four metals in ClF_5 where striker and anvil plate were the same metal. A Type 410 stainless steel striker was used in two of the three impacts on magnesium to avoid the cushioning obtained with the soft magnesium striker. In these cases the steel striker was driven into the soft magnesium with stress cracking on the bottom side of the anvil plate. However, there was no sign of ignition of the fresh metal surface that was exposed to the liquid ClF_5 impact. Control impacts for comparison were also made on the four metals in contact only with air. All metal surfaces exposed to ClF_5 showed a slight darkening. In all other respects, the metals that were impacted in ClF_5 and air were unaffected by the impact exposure.

Ref. 18 Grigger, J. C. and Miller, H. C., "The Compatibility of Structural Materials with Hybaline A-5 and Compound A", AFML-TR-64-391, Pennsalt Chemicals Corp., King of Prussia, Penn., Contract AF 33(657)-8461, Dec. 1964.