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RESEARCH MEMORANDUM

NONMETALLIC MATERIAL COMPATIBILITY WITH LIQUID FLUORINE

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Cleveland, Ohio

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FOR AERONAUTICS
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SUMMARY

Static tests were made on the compatibility of liquid fluorine with several nonmetallic materials at -320° F and at pressures of 0 and 1500 pounds per square inch gage. The results are compared with those from previous work with gaseous fluorine at the same pressures, but at atmospheric temperature. In general, although environmental effects were not always consistent, reactivity was least with the low-temperature, low-pressure liquid fluorine. Reactivity was greatest with the warm, high-pressure gaseous fluorine.

None of the liquids and greases tested was found to be entirely suitable for use in fluorine systems. Polytrifluorochloroethylene and N-43, the formula for which is $(C_4F_9)_3N$, did not react with liquid fluorine at atmospheric pressure or 1500 pounds per square inch gage under static conditions, but they did react when injected into liquid fluorine at 1500 pounds per square inch gage; they also reacted with gaseous fluorine at 1500 pounds per square inch gage. While water did not react with liquid fluorine at 1500 pounds per square inch gage, it is known to react violently with fluorine under other conditions. The pipe-thread lubricant Q-Seal did not react with liquid fluorine, but did react with gaseous fluorine at 1500 pounds per square inch gage. Of the solids, ruby (Al_2O_3) and Teflon did not react under the test conditions.

The results show that the compatibility of fluorine with nonmetals depends on the state of the fluorine and the system design.

INTRODUCTION

In most closed systems for handling liquid fluorine there are components which should be nonmetallic. Valve seats and packings, flange gaskets, and pump seals function best if made of a pliable, elastic material. No such material is known to be thoroughly satisfactory for such applications to fluorine systems, particularly those systems required to contain liquid fluorine flowing at high velocities under high pressure.

The compatibility of several nonmetallic materials with gaseous fluorine has been examined qualitatively and is reported in reference 1. Tests were made at atmospheric pressure and temperature, and at 1500 pounds per square inch gage at atmospheric temperature. If an observable reaction (vigorous burning or explosion) occurred when gaseous fluorine contacted the sample material, that substance was eliminated from further examination. Many materials survived the low-pressure tests; only Teflon and ruby showed no reaction after several minutes of exposure to fluorine gas at the high pressure.

The present report describes similar limited work on the compatibility of some of the same materials with liquid fluorine at -320° F.

APPARATUS AND PROCEDURE

Most of the tests conducted in this investigation simply involved static immersion of the sample in liquid fluorine at 0 and 1500 pounds per square inch gage. The following procedures were used:

(1) Fluorine was condensed directly around the solid samples in a glass test tube; liquid samples were dropped into the liquid fluorine.

(2) Fluorine was condensed over the sample, solid or liquid, in a closed steel tube and then pressurized to 1500 pounds per square inch gage.

(3) Liquid samples were injected into liquid fluorine, which was at 1500 pounds per square inch gage.

In the first procedure, a sample of the material weighing about 5 grams was placed in an open, glass test tube, which was immersed in liquid nitrogen. A continuous helium purge prevented condensation of air in the test tube. When the initial preparations were completed, the purge was stopped and gaseous fluorine was admitted slowly through a copper tube extending to within 1 inch of the bottom of the test tube. The fluorine condensed to cover the test specimen. When reactions occurred, they were immediate and violent. If no reaction took place after 10 minutes, the liquid-nitrogen bath was removed and the fluorine allowed to vaporize into a hood. During the vigorous boiloff, the specimen was carefully observed for further evidence of reaction. The test was viewed through a Plexiglas shield. The sample was visually inspected after the test.

The same apparatus was used for liquid samples. Fluorine was condensed to a depth of about 2 inches in a clean test tube, and then the sample, at room temperature, was dripped into it from a separatory funnel. If no reaction took place, the sample would immediately freeze into small balls upon hitting the cold fluorine.

If the sample did not react, it was next exposed to liquid fluorine at high pressure. A closed stainless-steel cylinder 6 inches long made from 3/4-inch heavy-walled tubing was connected to a fluorine container by 100 feet of 1/4-inch copper tubing. A helium pressurizing source was connected to the copper tubing very close to the fluorine source. After the test sample was placed in the steel cylinder, the air in the whole system was removed by purging with helium and then with gaseous fluorine. The steel cylinder was placed in a liquid-nitrogen bath, and gaseous fluorine was admitted at a pressure of 50 pounds per square inch gage. When the cylinder was completely filled with condensed fluorine, helium was used to pressurize the system to 1500 pounds per square inch gage. The system was allowed to stand for approximately 10 minutes. If a reaction had not taken place in that time, the liquid-nitrogen bath was removed by remote control, and the fluorine was allowed to vaporize. The cylinder was opened, and the sample was examined visually for evidence of a reaction.

In additional tests, the liquid materials were injected into liquid fluorine at high pressure. These injection tests were intended to simulate conditions that would be present with the failure or rupture of a wall separating a hydraulic fluid (one of the test liquids) from liquid fluorine. The apparatus for this test consisted of two stainless-steel compartments separated by a thin aluminum rupture disk. One compartment was connected to a fluorine supply and submerged in a liquid-nitrogen bath. This bottom compartment was large enough to hold about $1\frac{1}{2}$ pounds of condensed fluorine. The volume of the upper compartment was about 15 cubic centimeters and could contain approximately 1 ounce of the test sample. The two compartments were simultaneously pressurized to 1500 pounds per square inch gage. The top section was then pressurized further (to about 1800 lb/sq in. gage) until the separating disk broke and the test material was injected into the liquid fluorine. In order to obtain a qualitative indication of the occurrence of a reaction, a pressure gage connected to the upper pressurizing line was observed for an increase in pressure.

Although this final step approached dynamic conditions, only a few materials were examined in this manner; hence, the present tests are essentially representative of fluorine exposure under static conditions.

The terms "reaction" and "no reaction", as they appear in this report, are defined as follows: "reaction" means that either a visible explosion or fire took place or that the sample on inspection showed signs of oxidation; "no reaction" means that, to the unaided eye, the test material appeared unchanged after exposure to liquid fluorine for approximately 10 minutes.

TEST MATERIALS

Most of the materials tested are in common use for gaskets, valve packings, lubricants, hydraulic fluids, solvents, and so forth. They included highly fluorinated polymers, which would be expected to offer the greatest resistance among organic chemical compounds to any further reaction with fluorine. Polytrifluorochloroethylene is available under several trades names, such as Kel-F and Fluorolube. Since these are considered to be equivalent, generally only Kel-F materials were tested as representative samples.

All samples were selected for high purity, cleaned with organic solvents when applicable, thoroughly dried, and handled carefully with clean rubber gloves to avoid contamination.

RESULTS AND DISCUSSION

Data from the present work with liquid fluorine and previous data obtained with gaseous fluorine (ref. 1) are presented in table I.

Reduction in temperature (use of liquid instead of gaseous fluorine) usually decreased the tendency to react, although there were some exceptions to this. Twenty-four samples were tested with liquid fluorine at atmospheric pressure. Of these, five showed a reaction although four of these did not react with gaseous fluorine at atmospheric pressure. Of the 19 remaining samples, 12 did not react with the liquid fluorine at 1500 pounds per square inch gage, although in practically every case these materials had reacted with the high-pressure gaseous fluorine. Reactions generally occurred more readily with the relatively warm gaseous fluorine than with the cold liquid fluorine.

Increase in pressure generally increased the reactivity. In the gaseous work all but two of the materials tested were found to react when the pressure was increased to 1500 pounds per square inch gage. Of the 19 samples that did not react with the liquid fluorine at atmospheric pressure, four of these did react at 1500 pounds per square inch gage, and three others each reacted in one out of two tests. Of these last three materials, two were trifluorochloroethylene polymers. This inconsistency of reaction with these three materials can possibly be attributed to the different sample sizes used. It was found in the earlier gas-phase work that, if the exposed surface area were increased, the likelihood of reaction was greater.

The reactivity in injection tests, where three liquid samples were injected into liquid fluorine at high pressure, appeared to be no more severe for these three samples than previous tests in which the samples were exposed to warm, high-pressure gaseous fluorine.

Notwithstanding inconsistencies in environmental effects, reactivity appeared to be most pronounced at higher temperature and pressure; of the two variables, temperature had the more important role.

Of the liquids tested, polytrifluorochloroethylene and N-43 ($(C_4F_9)_3N$) did not react with liquid fluorine at atmospheric pressure or 1500 pounds per square inch gage under static conditions, but did react when injected into liquid fluorine at 1500 pounds per square inch gage. They also reacted with gaseous fluorine at room temperature and 1500 pounds per square inch gage.

Water did not react with liquid fluorine, but did with gaseous fluorine at room temperature and a pressure of 1500 pounds per square inch. Reference 2 mentions unpredictable and violent reactions between fluorine and water. Pertinent unpublished investigations made at the Lewis laboratory have demonstrated that water reacts with fluorine at temperature and pressure conditions near atmospheric, if the water is first atomized into a fine spray. Such reactions, which are smooth but very rapid, apparently are directly related to the amount of exposed surface area of the water. At present N-43 and the fluorinated polymers are used as fluids in pressure transducers where the fluid is separated from the fluorine by a metal diaphragm; the N-43 is less viscous at low temperatures than are the fluorinated polymers.

The pipe-thread lubricant Q-Seal did not react with liquid fluorine but did with gaseous fluorine at room temperature and a pressure of 1500 pounds per square inch gage.

Of the solid nonmetallic materials tested, ruby and Teflon seem to be reasonably compatible with fluorine. Teflon is the best plastic for use with fluorine and should be satisfactory if it is not exposed directly to flowing liquid fluorine, when it is known to fail.

CONCLUDING REMARKS

These experiments on material compatibility and others show that the compatibility of nonmetallic materials with liquid or gaseous fluorine is highly dependent upon the environmental conditions. For this reason, materials intended for fluorine service should be checked under conditions closely simulating the intended application.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, July 18, 1957

REFERENCES

1. Price, Harold G., Jr., and Douglass, Howard W.: Material Compatibility with Gaseous Fluorine. NACA RM E56K21, 1957.

2. Anon.: Fluorine - Properties and Methods of Handling. General Chem. Div., Allied Chemical and Dye Corp.

TABLE I. - NONMETALS EXPOSED TO GASEOUS AND LIQUID FLUORINE

Sample[a]	Sample exposed to liquid F_2 at atmospheric pressure	Sample exposed to gaseous F_2 at atmospheric pressure[b]	Sample exposed to liquid F_2 at 1500 lb/sq in. gage pressure	Sample exposed to gaseous F_2 at 1500 lb/sq in. gage pressure[b]	Sample injected into liquid F_2 at 1500 lb/sq in. gage pressure
Liquids:					
Kel-F LO No. 10 (M. W. Kellogg Co.)	No reaction	No reaction	No reaction	Reaction[c]	Reaction
Fluorolube HO (Hooker Electrochemical Co.)				Reaction[c]	(d)
N-45, $(C_4F_9)_3N$ (Minnesota Mining & Mfg. Co.)				Reaction[e]	Reaction
Tap water	↓	↓	↓	Reaction	No reaction
Cenco Hyvac Oil (Central Scientific Co.)	Not tested	Burned	Not tested	Not tested	Not tested
Glyptal (General Electric Co.)	Not tested				
Dow Corning 200 fluid (20 centistokes)	Exploded				
Water glass	Not tested				
Safety Solvent 17o (Fine Organics, Inc.)					
Leak-Tec (American Gas & Chemicals Inc.)					
Leak-Tec with glycerine					
Dry JP-4 fuel		↓			
Carbon tetrachloride	↓	Exploded	↓	↓	↓
Greases:					
Kel-F Med. Wax (M. W. Kellogg Co.)	No reaction	No reaction	Reaction[f]	Reaction	Not tested
Kel-F No. 1 Grease (M. W. Kellogg Co.)	(d)		(d)	(d)	
Fluorolube LG (Hooker Electrochemical Co.)	(d)		(d)	(d)	
Fluorolube MG (Hooker Electrochemical Co.)	(d)		(d)	(d)	
Permatex No. 2 (Permatex Co.)	No reaction		Reaction	Reaction	
Q-Seal (Quigley Co.)	No reaction		No reaction		
Blue Goop (Crawford Fitting Co.)	Exploded		Not tested		
Molylube (Bel-Ray Co., Inc.)	Exploded				
Plast-O-Seal (The Colonial Plastics Mfg. Co.)	Burned	↓		↓	
Permatex No. 1 (Permatex Co.)	Not tested	Burned		Not tested	
Permatex No. 2 (Permatex Co.)					
X-Pando seal coating (X-Pando Corp.)					
Tyte Unyte (J. C. Whitlam Mfg. Co.)					
White lead	↓	↓	↓	↓	↓
Solids:					
Ruby (Al_2O_3)	No reaction	No reaction	No reaction	No reaction	Not tested
Teflon (E. I. du Pont de Nemours & Co.)			No reaction	No reaction	
Kel-F Solid (M. W. Kellogg Co.)			Reaction[f]	Reaction	
Kel-F Elastomer 5500 (M. W. Kellogg Co.)			No reaction	Reaction	
Graphitar (United States Graphite Co.)	↓	↓	No reaction	Reaction	
Powdered Graphitar (United States Graphite Co.)	Not tested	Burned	Not tested	Not tested	
Neoprene-covered Fiberglas	Exploded	No reaction		Reaction	
N-45 plus neoprene-covered Fiberglas	Not tested		↓		
Plexiglas (Rohm & Haas Co.)	No reaction		Reaction		
Tygon tubing (U. S. Stoneware Co.)			Reaction		
Vinylite (Carbide and Carbon Chemicals Co.)			No reaction		
Pennsalt PCC (Pennsylvania Salt Mfg. Co.)			No reaction		
Pennsalt PCI (Pennsylvania Salt Mfg. Co.)			Reaction[f]		
Flux on silver-soldered copper-to-brass joint	↓		Not tested		
Slag on stainless-steel weld joint	No reaction		No reaction		
Flux on silver-soldered stainless-steel joint	No reaction	↓	Reaction	↓	
Dow Corning Elastomer	Not tested	Burned	Not tested	Not tested	
Molykote Type Z Powder (Bel-Ray Co., Inc.)					
Litharge and glycerine					
Palmetto (Greene, Tweed & Co.)	↓	↓	↓	↓	↓

[a]Samples are listed by trade name in most cases; the compositions were not available.

[b]Taken from ref. 1.

[c]Reacted when exposed surface area was increased.

[d]Not tested; Kel-F and Fluorolube materials are all polymers of trifluorochloroethylene; see preceding entry for representative results.

[e]Reacted in two out of four tests.

[f]Reacted in one out of two tests.