FILE NAME: Grumman (GRU)

DATE: 1965

DOC#: GRU006

DOCUMENT DESCRIPTION: National Safety Congress Transactions with Officer Lists & Journal Article - Evaluation of Protective Clothing for use with Fluorine

1965

National Safety Congress Transactions

Volume 1

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1965 National Safety Congress

EVALUATION OF PROTECTIVE CLOTHING FOR USE WITH FLUORINE

By M. CHAIN ROBBINS, M.P.H.
Safety Engineering, Aerospace Group, The Boeing Company, Seattle, Wash.

During the past year, various government agencies have shown a growing interest in fluorine as an oxidizer in liquid propulsion systems.

Boeing has been conducting laboratory and remote-site tests with both liquid fluorine (LF_2) and fluorine-oxygen (FLOX). System design parameters define requirements for remote operations of pressurization, transfer, purging, etc. Personnel, however, must make and break connections and perform various operations attendant to testing when system failure might present exposure conditions.

Air Force and NASA concern for personnel safety has stimulated the protective clothing manufacturers to develop suits for protection against a variety of fuels and oxidizers, yet maintaining wearability and flexibility in service. (Several suits are presently on the market, but government specifications to date have not required manufacturers to demonstrate performance in dynamic exposure environments.)

The Boeing Aerospace Group Safety Engineering organization reviewed current protective clothing practices in the chemical and aerospace industries. There is no uniformity in clothing materials being used against oxidizers such as fluorine. In fact, there is very little dynamic test data available on materials presently being used or proposed for use with fluorine.

Against this background we undertook a test program whose objective has been to evaluate those materials in use or proposed for use under dynamic conditions of exposure to gaseous and liquid fluorine. From these evaluations a determination has been made of the most suitable material and a specification developed for protective-suit construction.

Technical Approach

47 possible protective-suit materials were selected for test. These included butyls, neoprenes, cellulose acetate, Teflon, FEP, Kel-F, polyurethanes, Armalon 97-001, Armalon 97-001A, and Armalon 97-001G. (Armalon is a Du Pont trade name used to identify a Teflon laminate consisting of Teflon fabric 0.008 inch thick overcoated on one side with Teflon (FEP) 0.005 inch thick film, and is identified as 97-001. Some samples had a vacuum deposition of aluminum or gold and were further identified as "A" or "G" respectively after the 97-001. The selection of Du Pont's 97-001 material was based on North American Aviation, Rocketdyne Division test report No. 2091 on Contract AF04 (611)-5963, which indicated the 97-001G material to be exceptionally resistant to gaseous F_2, chlorine-trifluoride (CTF), and pentaborane.)

The Boeing test program consisted of five phases:

1. Chemical compatibility tests on all material samples.

2. Gaseous permeation tests on materials that showed the best performance in Phase 1; in addition, test specific areas of suit construction (seams, joints, etc.).

3. Tensile strength tests on materials that survive Phase 2.

4. Gaseous fluorine impingement tests on materials that showed the best performance in Phases 1 and 2.

5. Liquid fluorine impingement tests on materials that performed best in Phases 1, 2, and 3.

Test Apparatus

The same facility was used for the gaseous chemical compatibility, permeation, and impingement tests. The facility consists of a source of fluorine gas for filling a set of six cells or jigs. Pyrex glass cells were used for the chemical compatibility studies and stainless steel jigs for the permeation studies. Fluorine gas was supplied from a cylinder enclosed in a steel barricade equipped with remote controls for operating valves. Using 0.25 inch diameter stainless steel tubing, fluorine gas was brought into the inlet mani-

ld of the test apparatus, after passing rough an absorption tube for hydrogen ioride (HG) removal and a rotameter for ow rate measurement. The HF scrubber ntained an activated form of sodium ioride. The entire test setup was assembled a laboratory fume hood. Fluorine gas was troduced into each glass cell individually id valved, allowing it to have its own mosphere independent of the others. A line om a vacuum pump, including a coil immersed in a liquid-nitrogen bath and soda-me-packed tube, was attached to the outlet anifold. The cold trap prevented any or-anic vapors from the vacuum pump from ntering the test area, while the soda-lime ibe will prevent fluorine from reaching the acuum pump. Nitrogen was dried by passing ie cylinder gases through a coil immersed ı dry ice before entering the test setup. The itrogen gas was valved for purging any or ll parts of the complete system. A pressure age was located ahead of the fluorine flow-ieter to read the pressure regulator on the uorine cylinder. A vacuum gage, calibrated gainst a mercury manometer, was located n the inlet manifold.

Phase 1—Chemical Compatibility Tests

The test samples were approximately 0.5 nch wide and two inches long. Prior to esting, the samples were solvent-cleaned, dis-illed water rinsed, wiped clean, and air lried.

The samples were hung on stainless steel ıooks located inside the glass cells. The ;ystem was first passivated with gaseous N_2, hen evacuated. A vacuum was drawn and gaseous fluorine (GF_2) was then introduced nto the cell until reaching a 1-atmosphere positive pressure. The samples were exposed to 100 per cent GF_2 environment at 1-atmosphere for two hours. The samples were weighed on an analytical balance before and after exposure to GF_2 to determine any weight change.

Test Results

The Armalon 97-001, 97-001A, and 97-011G, butyl-coated nlyon (parachute) cloth, Buna N coated nylon, Gentex 2 (Al_2-nylon-neoprene), and Du Pont neoprene nylon 5009 demonstrated the best performances of the supported materials. Sawyer-Tower neoprene-nylon and Gralite failed this test. Several unsupported samples demonstrated good results, such as neoprenes (2-0262) (2-0022) (#89); butyls (62.72) (8.0255) (#80-011); polyurethane (UR 29E Black); Nordell (50-001); and Surlyn (A) and Viton A, 9-0277 and 80-060.

Phase 2—Permeation Tests

The same facility in Phase 1 was used for the permeation test, however, stainless steel jigs were fabricated and used instead of the Pyrex glass cells. The jigs were fabricated into two halves, with a mating flange at the equator. The diaphragms of test materials were placed between the mating equators and torqued tight. A continuous flow of dry nitrogen was passed through the top section. A fluorine atmosphere was produced in the lower section by flowing seven liters of GF_2 into and through the chamber during a 10 minute purge. The lower section was then sealed.

Only samples that showed best performance in Phase 1 were tested. Each specimen began as a six inch square; it was then cut into the shape of a ping-pong paddle, with the "paddle" part the same diameter as the outside diameter of the permeation jig and its "handle" available for positioning the specimen in the jig.

Quantitative measuring of the permeation rate of GF_2 was accomplished by sweeping the outlet gas into a bubbler containing 10 milliliters of 0.1N NaOH through the outlet side of the N_2 jig section for five minutes. (A hose was connected to the outlet orifice of the N_2 half of the jig, and gaseous N_2 containing the permeated F_2 gas was bubbled into the 0.1N NaOH solution.) Previous tests indicated that this system will quantitatively absorb F_2 and HF from a gas stream. The pressure on the bottom on the F_2 side was built up to 1-atmosphere positive pressure; the pressure on the N_2 or top side of the jig was built up to 1-atmosphere plus two inches of H_2O positive pressure during sampling. Bubbler sampling was done periodically during the test.

Test Results

Wilgard 89 (Neoprene) Glove Material (0.030 Inch Thick)—This material demonstrated the best results of all the materials tested. Two samples were tested up to 24 hours and no detectable permeation was found. Subsequent examination showed only

that the exposed side had whitened somewhat.

Armalon 97-001—This material demonstrated the best overall Teflon results with low permeation rates.

Armalon 97-001A and G—Although some samples demonstrated low permeation rates, some had high rates. Close examination revealed pin holes through the material. It is felt that in the process of vacuum deposition, there is some tendency to cause FEP film degradation.

Armalon 97-001 Containing Sewed-Sealed Seam—High permeation rates.

Armalon 97-001G, Sewn and Sealed—High permeation rates.

Armalon 97-001A, Sewn and Sealed—High permeation rates.

Cellulose Acetate Faceshield—Two samples (approximately 0.060 inch thick) were tested. No detectable permeation was found during the first four hours, and this time could be greatly increased since it was the following day before it was retested. Equilibrium values of about 13 micrograms/inch²/hour were noted after one day. Subsequent examination revealed some attack, as evidenced by the pronounced odor of acetic acid. The material, however, remained optically clear but slightly discolored.

Butyl (Du Pont T-5595 Sheet Stock)—One sample of Du Pont T-5595 Butyl burned immediately upon F_2 exposure.

Theoretical Suit Permeation

To get some insight as to the significance of the permeation relative to full-size fuel-handler's suits, calculations were made showing the projected suit leakage rates. Times of exposure and percentages of gaseous F_2 were varied. The calculations were based on the surface area of an average size man (15.5 ft.²). The assumption is made that the suit is under two inches of water positive pressure from supplied air or its own environmental control system.

Example 1 (Leakage Rate = 10 ug/F_2/inch²/hour)

1. Multiply the leakage rate times suit area to find the leakage per hour, expressed in ppm per average suit per hour.

2. In this example, the leakage per hour would be:

F_2 Concentration	ppm/average suit/hour
100%	169
10%	16.9
5%	8.45

3. In five minutes, the leakage would be:

F_2 Concentration	ppm/average suit
100%	14.1
10%	1.41
5%	0.71

Example 2 (Leakage Rate = 700 ug/inch²/hour

1. Multiply the leakage rate times suit area to find the leakage per hour, expressed in ppm per average suit per hour.

2. In this example, the leakage per hour would be:

F_2 Concentration	ppm/average suit/hour
100%	11,739
10%	1,173.9
5%	586.95

3. In five minutes, the leakage would be:

F_2 Concentration	ppm/average suit
100%	978.2
10%	97.8
5%	48.9

The results shown above take on added meaning in light of emergency exposure levels published recently by the National Academy of Science. See Table 1.

Table 1: Emergency Exposure Levels

Free Fluorine

Concentration Level	*Time of Exposure*
5 ppm	5 minutes
3 ppm	15 minutes
2 ppm	30 minutes
1 ppm	1 hour
0.1 ppm	8 hours

Hydrogen Fluoride

Concentration Level	*Time of Exposure*
30 ppm	5 minutes
20 ppm	15 minutes
10 ppm	30 minutes
8 ppm	1 hour

Phase 3—Tensile Strength Tests

All specimens that were permeation tested were later tensile tested per Federal Specification CCC-T-191b, Method 5102.

Specimens (one inch by six inches) were placed symmetrically into the clamps of an Instron Tester, with long dimension parallel

and the short dimension at right angles to the direction of load application. The yarn running parallel to the long dimension of the specimen was aligned with one outside edge of the front jaw of each clamp to ensure the same yarns being gripped in both clamps. The tension was evenly distributed on the yarns between clamps. Force was applied at the specimen at 12 ± 0.5 inches per minute. After specimen rupture, the breaking force was read from the dial indicator and recorded. The jaws were faced with rubber to prevent slippage.

Test Results

The test results are shown in Table II.

Table II: Tensile Strength Tests

	Tensile Strength		
Material	*As Received*	*After F_2 Contamination*	*Loss (Percent)*
97-001	75 psi	63 psi	16
97-001A	68 psi	63 psi	7
97-001G	68 psi	64 psi	6
97-001 (Seam)	120 psi	112 psi	7
97-001A (Seam)	120 psi	115 psi	5
97-001G (Seam)	120 psi	114 psi	6
Butyl (T-5595)	1960 psi	1010 psi	48
Neoprene (Wilson 89)	3760 psi	2825 psi	25
Cellulose Acetate	7166 psi	5280 psi	26

Phase 4—Gaseous Impingement Tests

Sample materials that demonstrated the best performance in the first three phases of testing were subjected to the gaseous impingement tests; in addition, three materials (8-0255 butyl, Nordel 50-001, and 62-72 butyl) that had only been given the chemical compatibility test were tested in this phase.

A test apparatus similar to the chemical compatibility test equipment was used. The system was evacuated and the sample suspended 0.1 inch from an orifice (0.16 inch diameter). Gaseous fluorine was then flowed into the evacuated chamber until the pressure reached 1-atmosphere again (about one minute of flow).

Test Results

There was no visible reaction or change in the weight or appearance of the samples.

Phase 5—Liquid-Fluorine Impingement Tests

Dynamic liquid-fluorine impingement tests were conducted at the Tulalip Remote Test Site in conjunction with Boeing Flight Technology personnel. The tests involved impinging 24 samples. Some samples were retested two or three times to verify results. Only sample materials that passed the gaseous chemical compatibility tests were liquid-impinged.

The 10 gallon liquid-fluorine transfer test apparatus was used for the impingement tests. Here liquid fluorine was made by condensing gas from cylinders in a liquid-nitrogen bath. The stand consisted of a stainless steel rack, subdivided into six individual cells. Each cell was partitioned by stainless steel baffles and copper wire screen for mounting the samples. A stainless steel manifold was located at the base of the test rack, to which six 0.25 inch diameter stainless steel lines were welded. The lines were arranged so that each sample was impinged separately. The lines were restricted at the exit end by installing an AN 818-4B nut and an AN 806-4 plug with the plug drilled to a 1/16 inch diameter orifice.

The test samples were approximately six by six inches and were solvent cleaned, water rinsed, and air dried prior to testing. One impingement was conducted per sample.

The samples were mounted to the copper screen holder. The jet orifices and samples were protected from moisture by placing a small bag over the purging jet orifice and a small Teflon shield over each sample to protect against atmospheric moisture. The bag and shields were removed prior to pressurizing the system.

After condensing the liquid fluorine in the 10 gallon test apparatus, it was transferred through the 0.25 inch diameter line immersed in a liquid-nitrogen bath (to maintain the density of the liquid fluorine during the transfer) into the manifold located on the test rack. The manifold was also immersed in a liquid bath and then impinged onto each sample separately. Approximately three liters of liquid fluorine were condensed from each gas cylinder. Each sample was jet-impinged with approximately 500 cubic centimeters of

LF_3 per test (1/16 inch orifice, five to eight seconds, at 50 psig).

Test Results

Table III tabulates individual test results. It indicates the specific reaction, such as immediate fire, delayed ignition time, degree of fire, no reaction, etc.

Conclusion

The results of the chemical compatibility tests indicated that, of the 47 materials tested, only four supported elastomers, 10 unsupported elastomers, and three fluorocarbons proved to be compatible.

It is not known whether the method of polymer application or compounding made the difference in compatibility. Spectroanalysis did not reveal enough differences in polymer formulation or molecular arrangement to indicate specific differences between polymers.

Several of the materials, such as butyl over nylon, neoprene over nylon, Buna-N over nylon, neoprene 89, and Gentex 2, which demonstrated good results in the chemical compatibility tests, caught fire when liquid-impinged.

The following summarizes the results of both the gaseous and liquid tests:

1. Armalon 97-001 material demonstrated the best overall performance to both gaseous and liquid tests.

2. Armalon 97-001G was demonstrated to be chemically compatible, and two samples demonstrated the lowest fluorocarbon permeability rates. A third sample, however, had high leakage rates and, when closely examined, pinholes were found. (It was hypothesized that the pinholes were caused when the gold was vapor-deposited onto the film.) Portions of the gold samples caught fire when liquid-impinged.

3. Armalon 97-001A also was demonstrated to be chemically compatible but, when permeation tested, one sample demonstrated excessive leakage rates. (Again, when closely examined, pinholes were found.) The sample caught fire when liquid-impinged.

TABLE III: RESULTS

Trade Name	Composition	Results
Gentex	Al₂-Glass-Neoprene	Caught fire immediately.
Wilgard Glove 89	Neoprene	Caught fire. Delayed fire reaction.
Wilson Rubber Co.	Butyl-coated nylon	Caught fire immediately.
Du Pont 190-27-3	Teflon fabric—Teflon FEP film	Caught fire. One end burned.
Du Pont Armalon 97-001	Same as above 97-001—gold coating	Delayed fire. Caught fire on one side.
Du Pont Armalon 97-001G	Same as 97-001G except aluminum coated	Caught fire. Delayed action. Burned quite rapidly once it started.
Du Pont Armalon 97-001A	(Retest) Same as Sample 5	Caught fire around perimeter of sample. Edge of the gold burned and flaked off.
Du Pont Armalon 97-001G	Faceshield, 0.060 inch thick Neoprene	Caught fire. Delayed reaction.
Cellulose Acetate	Neoprene over nylon	Once started, it burned up approximately 2 inches of faceshield.
Wilgard Glove	Buna-N over nylon	Caught fire. Delayed action.
Du Pont 5009	Faceshield	Caught fire immediately.
Du Pont 5085	(Retest) Same as Samples 5 and 7 with butyl rubber seam	Caught fire immediately.
Acrylic	(Retest) Faceshield	Caught fire immediately.
Du Pont Armalon 97-001G	Same as Sample 4	Seam caught fire immediately with vigorous burning.
Cellulose Acetate	Polyamide-FEP film plus gold deposition	No reaction.
Du Pont Armalon 97-001	Faceshield	No reaction.
HT-1-FEP-Gold	Faceshield	Violent fire immediately.
FEP film, 0.005 inch thick	Cotton-coated, both sides with PVC	No reaction.
FEP, 0.020 inch thick	(Retest) Same as Samples 4 and 15	No reaction.
Gra-lite	This sample had been exposed to GF_3 for 24 hours prior to liquid testing.	Caught fire immediately.
Du Pont Armalon 97-001	Faceshield Same as Sample 18	No reaction.
Du Pont Armalon 97-001	Faceshield Same as Samples 8 and 14	No reaction.
FEP film, 0.020 inch thick	(Retest) Same as Sample 19	Caught fire immediately.
Cellulose Acetate Gra-lite		