ADVANCED SUPERSONIC TECHNOLOGY FUEL TANK SEALANTS

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SUMMARY

The Advanced Supersonic Technology (AST) Fuel Tank Sealants Program is reviewed. Status of the fuel tank simulation and YF-12A flight tests utilizing a state-of-the-art fluorosilicone sealant is described. New elastomer sealant development at Ames Research Center is detailed, and comparisons of high and low temperature characteristics are made to baseline fluorosilicone sealants.

INTRODUCTION

The continuing search for improved candidates for high temperature fuel tank sealants was begun at the NASA Ames Research Center in the Chemical Research Projects Office with the new incentives of both NASA's supersonic cruise aircraft research (SCAR) program and continuation of DOT's effort to evolve an advanced supersonic technology base.

A review of what had been accomplished synthetically suggested that the Dow Corning (DC) developmental sealant 77-028, a fluoroalkysiloxane, and derivatives of the perfluoroethers offered opportunities for further development and exploitation. Although Boeing studies had shown that DC77-028 has a tendency to degrade, it might, with the appropriate predictive analysis, be useful in meeting the high-performance sealant requirements. Research has suggested that with appropriate modifications to reduce the glass transition temperature and to improve the efficiency of crosslinking processes these basic materials have the potentiality of providing sealants with marked improvements in stability in extreme environments (ref. 1).

The overall goal of the AST Fuel Tank Sealant Program is to produce a flight-proof, fully characterized, predictable fuel tank sealant that meets high-performance supersonic aircraft requirements. Figure 1 indicates the various interrelated activities of the fuel tank sealants program. These activities comprise synthesis and characterization of new fluoroether elastomers, processing and scale-up activities, sealant-metal system development, sealant prediction studies, flight simulation, and actual flight testing of the best state-of-the-art sealants.

For advanced sealant elastomers, both new extension and new branching reactions that can be carried out with hexafluoropropylene oxide oligomers are under consideration. The reactions of these polymeric intermediates through nitrile cyclizations to give triazines, and nitrile co-reactions with acid fluorides to give polyoxadiazoles are two principal systems being studied. Early work indicated promising results for the two systems under investigation.

Both high and low temperature applications appeared feasible in spite of the initial deficiencies in physical properties. Thus, molecular weight and crosslinking control are currently the primary areas of investigation.

In principle, the desired predictive method can be developed from a suitable combination of viscoelastic measurements (leading to the construction of a characteristic failure "property surface" for the elastomer) and stress-relaxation measurements (which assess the extent and contribution of chemical degradation in these materials). Work at the Jet Propulsion Laboratory (JPL) has led to the development of short-time test methods that enable predictions to be made of the lifetime of elastomers under operating conditions, but in the absence of chemical degradation. From other studies, methods were also developed for measuring network degradation kinetics for elastomers exposed to a fuel invironment at elevated temperatures; these were principally adaptations of classical chemorheological techniques. Efforts have been made to show that these methods can be combined to yield a satisfactory prediction of long-term aging using short-term tests.

Actual time simulated tank studies were conducted at Boeing. The experimental fluorosilicone sealant was installed in a test chamber where it was subjected to the pressures and temperatures of the supersonic cruise aircraft flight cycle. Simultaneously, the tank was subjected to spanwise and chordwise loading representative of the fatigue spectrum expected under the most severe flight conditions.

The fluorosilicone DC77-028 is considered the state-of-the-art sealant candidate to date and efforts have been made to correlate its behavior in prediction studies and flight simulation. The DC77-028 is currently being tested on the YF-12A NASA aircraft, and correlation with the prediction and simulated tank studies will then provide a baseline for future testing of new elastomeric candidates in subsequent flight cycles.

FAILURE MODES

Several years ago NASA, DOT, and Boeing visited most of the NASA Centers and talked with the principal investigators who had been concerned with supersonic cruise vehicle fuel tank sealant developments. To guide future developments, the specific deficiencies of the baseline DC77-028 sealant were identified. Three failure modes were evident from these discussions: (1) reversion, (2) low tear strength, and (3) lack of reproducibility of adhesion to titanium. These failure modes are discussed below.

Reversion

It is well accepted and proven that the root cause of the observed degradation of the base-line sealant DC77-028 is due to polymer reversion with the formation of cyclic tetramer from the principal chains. The practical result of this degradation process is the loss of sealant from the seal

rather than significant degradation in polymer bulk properties. For this reason, compounded sealant shrinks in the joints. This phenomenon of reversion is generally characteristic of silicones and is a limiting factor in their application for continuous service at temperatures above 204° C (400° F). Fluorosilicone hybrids, a family of new derivatives which have been proposed and synthesized for military applications, have virtually eliminated reversion but have introduced the possibility of dehydrofluorination followed by oxidative chain scission. The new perfluoroether candidates suffer from none of these deficiencies.

Mechanical Tearing of Sealant

Failure of sealant installation by the formation of cracks due to tearing of the sealant in the joint seems to be the most common failure mode. It seems to be an inherent difficulty with state-of-the-art formulated sealants, that they possess a rather large thermal coefficient of expansion and also exhibit a significant loss in tensile strength at elevated temperatures. These two properties couple to provide the tearing mechanism. A good possibility for eliminating this difficulty is the development of polydisperse blends and graft or block polymer systems.

Adhesion Failure

Difficulties with the sealant-primer-titanium interfaces manifest themselves in total lack of reproducibility in the measured peel strengths which can vary by as much as two orders of magnitude. Just how significant peel strength is as related to failure is not clear. When coupled with chemically induced stress corrosion in the joint, the lack of integrity could completely degrade the interface structure.

From the foregoing considerations, some constraints on the evolution of new polymer types can be defined. The siloxane group is a weak link and should be reduced in concentration or blocked against reversion. Cyano substitution in siloxanes degrades stability. Vicinal substitution with HF may lead to instability and chemical stress corrosion. The perfluoroether derivatives begin to meet most of these constraints, but they require innovations to reduce glass transition temperatures (Tg), by chain extension and crosslinking control.

TEST RESULTS

Flight Simulation

In 1973, NASA-Ames contracted with The Boeing Company to design, fabricate, and subject to supersonic flight environment a small tank representative of the materials and structure of a supersonic transport fuel cell. The tank was sealed using the best known sealant for supersonic use and was periodically examined and tested for leaks. Another part of the program was to conduct

studies of sealant adhesion to titanium. Lap shear specimens were subjected to elements of supersonic fuel tank environment and tested after 6 weeks of such exposure.

Table I lists the condition of the seal in the simulated fuel tank after 861 flight cycles and after 1389 cycles. Inspection after 861 flight cycles disclosed no noticeable degradation; there were no tank leaks and no perceptible cracks in the sealant. However, after 1389 flight cycles there were numerous hairline cracks in the sealant, the tank leaked badly at the corners, and minor leaks were observed in other locations. Lack of adhesion was observed at some of the cracks. The results demonstrate the importance of combining simultaneously all elements of the environment, including loads, that the material being tested would encounter in service. It is believed that stressing the sealant while at high temperature (227°C, 441°F), together with stresses due to high thermal expansion, caused it to tear.

Figure 2 is a photograph of the tank after inspection at 1389 flight cycles. The cracks are characteristic of failure under high-temperature stress. A lower modulus (higher elongation to failure) material would perhaps overcome the problem.

YF-12A Flight Tests

The objectives of the NASA-Ames program for flight testing on the YF-12A aircraft are (1) to correlate JPL prediction studies and the Boeing simulated tank test to actual flight testing, (2) to provide side-by-side comparisons of the various fluorosilicone candidate sealants, and (3) to provide a baseline for future testing of new NASA developed sealant elastomer candidates.

During the second week of January 1975, test specimens of DC77-028 sealant were mounted in a YF-12A. Some of these were from JPL and had been made with Lockheed cooperation. There were four perforated stainless steel boxes mounted on the wall of the center wing tank: two were mounted forward and two aft. In each case, one of the two was mounted near the bottom of the tank and the other near the top. Thermocouples are on the surface near each box. In each of the boxes are 22 tensile elongation specimens, 2 weight and volume loss specimens, 2 lapped titanium specimens with a fillet over the lap and a deflection of 0.01 inch at the fillet, a strip of titanium with primer only on the surface, and four strips of molded DC77-028 wrapped and clamped around a 0.32-cm (0.125 in.) rod. A sheet of titanium, coated with 0.25 cm (0.1 in.) of DC77-028, is mounted outside the boxes. One end is straight and rigid and the other is wrapped and clamped to put a variable strain in the sealant. Also, primer and sealant are applied to numerous sealant-free areas of the tank surface.

Figure 3 is a photograph of two of the mounted boxes containing the sealant specimens. The only sealant failures were some stress rings mounted in the boxes, part of a JPL test. There were no failures in the outboard forward box, the two rings of the greatest stress had broken in the aft

outboard box, and in each of the other two boxes only the ring with next to the greatest stress had broken. Adhesion to all surfaces was good except to the old sealant.

Table II summarizes the four phases of the YF-12A flight test. In Phase 1, the test fillets now in the YF-12A will be damaged and repairs effected according to Boeing's instructions. A new "saddle" block with higher stressed sealant than the existing one will also be installed in the tank.

During Phase 2 of the YF-12A flight tests, the No. 5 tank of the YF-12A will be stripped and resealed after completion of thermodynamic (cold wall) tests. The expected completion date, after 12 to 15 more supersonic flights, is January 1977. Tank stripping will be contracted for by NASA-DFRC and performed by Lockheed. Stripping is not routinely done on the No. 5 tank and there will be some equipment costs. After initial preparation stripping takes about 24 hr. Resealing will then be carried out by NASA-DFRC technicians with assistance from Boeing. Future plans, Phases 3 and 4, call for the testing of the new NASA fluoroether sealant candidate(s) after all baseline and correlative information has been established.

Advanced Polymers

General. — The availability of hexafluoropropylene oxide and the research on the polymerization of tetrafluoroethylene oxide at the DuPont Co. (ref. 2) prompted us to consider the possibility of building long-chain perfluoroalkylene ethers that would yield a combination of properties for severe environmental applications that were not possible with any other polymer system. Our goal, then, was to obtain high molecular weight elastomers that have good low-temperature mechanical properties, high thermal stability, and high chemical resistance, while maintaining sufficient flexibility over a wide temperature range. It should be mentioned here that, until now, perfluoroether synthesis has been limited by inadequate molecular weights.

The unusual resistance of highly fluorinated materials to high temperatures, oxidation, and a variety of organic solvents has been known since Plunkett's discovery of Teflon in 1938. Efforts since then have centered on the synthesis of partially and completely fluorinated polymers designed to retain the thermooxidative and chemical stability of polytetrafluoroethylene, while adding elastomeric character, low-temperature flexibility, processibility, etc. The literature reveals many attempts to prepare fluorinated ether polymers (refs. 3-6). It is to be noted that by incorporating ether backbonnes one can design materials that will maintain flexibility below -30°C (-28°F). For example, nitroso rubber has a Tg of -50°C (-58°F), but its high-temperature stability is limited. The fluorosilicone comes nearest to meeting the requirements of a high-temperature sealant. It has excellent low- and high-temperature properties, but it tends to undergo reversion and has low tear strength.

We at NASA, together with scientists at PCR, Inc., reopened the question of how to chain extend these fluoroether systems. The objectives were to prepare long-chain difunctional polyperfluoroethers and investigate chain

extension mechanisms, as well as to convert these materials to stable crosslinked polymers for sealant applications. The nitrile, acetylene, and isocyanate groups were considered. Each of these is capable of both trimerization reactions and cycloadditions. From this base line, one could then evoke both chain extension and cross-linking with a variety of reaction schemes (ref. 7).

11.11

The general characteristics of the sealant elastomer required before formulation and mechanical property testing can begin are as follows: thermal stability from 227°C (450°F) to 316°C (600°F); low temperature flexibility to -46°C (-50°F); chemical resistance; and adhesion to metals. Our initial efforts were then directed toward increasing the molecular weights of the fluoroethers by chain extension reactions. This was to be achieved through thermally stable anchor points which could serve as "hard domains" to improve high temperature mechanical properties as well as provide the necessary crosslink sites and molecular weight control.

Two systems were selected based upon the same nitrile terminated fluoroether prepolymer backbone. The difunctional nitrile could be trimerized, that is, reacted with itself to form triazine or co-reacted with aromatic nitrile oxides to give aromatic 1, 2, 4-oxadiazoles. Both systems were investigated and results indicated good thermal stability and low temperature flexibility, but both were lacking in mechanical properties. Deficiencies in processing and molecular weights appeared to be principal reasons for achieving inadequate sealant elastomer characteristics. These materials are compared to recently developed polymers in a later section.

Model studies. — Simultaneously, we embarked upon a detailed polymer model study which would enable us to design polymers based on relating structural parameters to performance. Specifically examined were the relative effects of degradation in selected environments on the heterocyclic ring structures and ring substituents (ref. 8).

Table III shows simplified chemical models representative of four (4) ring systems which might be synthesized from the base nitrile-fluoroether prepolymer as mentioned above. Left to right they are the s-triazine; 1, 2, 4-oxadiazole; 1, 2, 4-triazole and 1, 3, 4-oxadiazole. R, R_f and Ar indicate various substituent molecules which can be synthesized into a polymeric structure. The purpose was to degrade and analyze the materials in fuel tank environments. The investigation determined on specific model compounds the relative thermal, thermal oxidative, and hydrolytic stability of potential chain extension moieties and crosslinks useful for curing perfluoroalkyl ether elastomers. Initial results emphasized the importance of structures 1 and 2 relative to 3 and 4.

Table IV indicates that terephthalonitrile-bis-N-oxide derived oxadiazoles decomposed extensively at 325°C (617°F). Although the effect of substituents cannot be discerned, subsequent data reinforce the idea that an aromatic group adjacent to the oxadiazole leads to lessened thermal stability.

By going to a fully fluorinated 1, 2, 4-oxadiazole (compound A) as shown in table V, a significant increase in thermal stability in an inert atmosphere can be demonstrated. Moreover, at the indicated temperature of 325°C (617°F), there is adequate thermal oxidative and hydrolytic stability of the 1, 2, 4-oxadiazole for projected high-performance sealant applications. Contrast this with the 1, 3, 4-oxadiazole (compound B) where thermal-oxidative stability is lower. Subsequents runs indicate erratic behavior of the 1, 3, 4-oxadiazole in oxidative environments. Recent data reflect equal stability characteristics for the fluoroether-1, 2, 4-oxadiazole as for (compound A) which contains no ether groups.

Table VI shows the results of the alternate system, the triazine-fluoroether models. Both triazine models exhibit excellent thermal and thermal-oxidative stabilities at 325°C (617°F). However, an examination of the degradations performed under thermal-hydrolytic conditions indicates a very high sensitivity to the unbranched fluoroaliphatic group attached to the triazine nucleus (compound A). A dramatic turn around in hydrolytic stability of the triazine containing the branched fluoroether group (compound B) can be clearly seen from the data. This information points to an important structural detail in designing a stable fluorinated triazine polymer. Our polymer work utilized this result to tailor-make triazine-fluoroethers having a -CF- and not a -CF_- adjacent to the ${\rm CF}_2$

triazine nucleus. Apparently this is due to steric reasons (that is, a crowding at the site of hydrolytic attack of the triazine) rather than to electronic effects. This represents the first time that a thorough model study has been used to establish criteria for tailoring structural details in polymer synthesis design.

Synthesis of new fluoroether polymers. — Guided by the model stability studies described in the previous section, two promising sealant elastomer candidates have emerged from synthetic investigations. The candidates are a fluorinated ether triazine and a fully fluorinated ether-1, 2, 4-oxadiazole. Recent efforts have concentrated on molecular weight and it now appears that we have achieved molecular weight ranges between 15 000 and 50 000 which should be adequate for a filler type sealant. Current work involves the determination of the optimum crosslink density for attaining the requisite mechanical properties in a high-performance sealant application.

Table VII compares the relative weight loss of the Ames fluoroether polymers to the best state-of-the-art fluorosilicones. The 77-108 is the Dow-Corning hybrid fluorosilicone designed to improve the thermal stability and reversion tendency of the DC77-028 fluorosilicone currently being flight-tested on the YF-12A.

Note that weight loss does not begin at 300° C (572° F) for the two Ames fluoroether elastomer's as it does with both fluorosilicones. Weight loss comparisons are also made at 350°, 400°, 450°, and 500° C (662°, 752°, 842°, and 932° F). Even at 500° C (932° F), the fluoroether -1, 2, 4-oxadiazole at 57-percent weight loss compares favorably with the fluorosilicones while the triazine fluoroether shows a weight loss of only 20-percent.

Figure 4 shows actual traces of the thermogravimetric analyses of both fluorosilicones and the two Ames fluoroether polymers. Also included is a polyphasphazene polymer which has been proposed as a high temperature elastomer. Here again, it is clear that the Ames fluoroether -1, 2, 4-oxadiazole compares very favorably to both fluorosilicones in thermal stability while the Ames fluoroether-triazine is superior.

A summary of Ames materials compared to the fluorosilicones is shown in table VIII. In this table we are comparing the temperatures at which specific weight losses of 1, 10, 50, and 90 percent occurred. Also shown are relative comparisons of the glass transition temperatures which give an indication of the low temperature brittle or use point. Finally, a relative assessment of the elastomeric properties are compared.

The two fluorosilicones are listed on lines 1 and 2 where it can be seen that all properties are reasonably good. Lines 3 and 4 are examples of the early Ames polymers mentioned previously. Points worth mentioning here are that the oxadiazole on line 3 is not quite up to the thermal stability or elastomeric property expectations. This was certainly substantiated in the model studies mentioned earlier. The triazine on line 4, although showing good high and low temperature properties, is deficient in the necessary mechanical properties. Lines 5 and 6 indicate that the two new Ames fluoroether polymers have very good high and low temperature properties as well as very promising elastomeric characteristics.

CURRENT AND FUTURE PLANS

The characterization and vulcanization study that will be carried out this year for polymer selection will determine the best crosslink density and will measure the properties of the polymers. This will include molecular weight and molecular weight distributions, thermal stability, aging studies, glass transition temperatures, and fuel, metal, and hydrolytic stabilities.

Simultaneously, the sealant elastomer will be formulated with the appropriate fillers and its mechanical properties determined. Sheet stocks will then be prepared for sealant-metal system development and actual flight testing in the YF-12A aircraft.

CONCLUSIONS

Preliminary results indicate that new fluoroether polymers developed at Ames Research Center compare favorably with state-of-the-art fluorosilicone sealants. The glass transition temperatures are comparable and the thermal stabilities are equal or superior to the baseline materials. It is anticipated that the fluoroethers will exhibit enhanced resistance to reversion and should provide materials for consideration as serious high-performance sealant candidates.

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TABLE I .- BOEING FLIGHT SIMULATION

77-028 FLUOROSILICONE SEALANT

NUMBER OF FLIGHT CYCLES - 46°C to 227°C (-50°F to 441°F) 3 HOURS/FLIGHT	FUEL CELL CONDITION
861	NO LEAKS, NO SEALANT CRACKS
1389	NUMEROUS BASELINE AND MOON SHAPED SEALANT CRACKS
	CORNER LEAKS
	SOME ADHESION FAILURE

TABLE II. - YF-12A AIRCRAFT FLIGHT TEST

PHASE 1:

SMALL TEST SPECIMENS—FLUOROSILICONES

- TENSILE SPECIMENS
- ADHESION SPECIMENS
- WEIGHT & VOLUME CHANGE SPECIMENS
- CONSTANT STRAIN SPECIMENS

FLIGHT TEST INITIATED MARCH 1975

30 FLIGHTS TO JANUARY 1976

INSPECTION JUNE 1976

PHASE 2:

- STRIPPING OF SEALANT IN NUMBER 5 AFT WING TANK
- RESEALING OF NUMBER 5 TANK WITH 25-30 LB. FLUOROSILICONE SEALANT (ROOM TEMPERATURE CURE)

PHASE 3:

• SMALL TEST SPECIMENS—NASA DEVELOPED FLUOROETHER SEALANT CANDIDATES

PHASE 4:

• RESEALING OF AFT WING TANK WITH SELECTED NASA FLUOROETHER

TABLE III.- STABILITY OF MODEL COMPOUNDS

PURPOSE:

TO SIMULATE ACCELERATED DEGRADATION TO PERMIT EXTRAPOLATION OF DATA TO SEALANT SERVICE CONDITIONS.

- THE-OXIDATIVE TO 316°C (600°F)
- HYDROLYTIC, 0.25 TO 3% WATER
- ANALYSIS AND MATERIAL BALANCE

TABLE IV. - MODEL STUDIES

DEGRADATION AND MASS BALANCE AFTER 48 HOURS

COMPOUND A

TEST RESULTS—COMPOUND A						
TEMPERATURE, °C ENVIRONMENT WEIGHT, % RECOVERED						
235	N ₂	98.8				
235	N₂/H₂O	98.3				
325	N ₂	-				

COMPOUND B

TEST RESULTS—COMPOUND B							
TEMPERATURE, °C ENVIRONMENT WEIGHT, % RECOVERED							
235	N ₂	100					
235	N ₂ /H ₂ O	99.1					
325	N₂						

TABLE V. - MODEL STUDIES

DEGRADATION AND MASS BALANCE AFTER 48 HOURS

$$\begin{array}{c|c} N & & nC_7 \, F_{15} \\ & \parallel & & N \end{array}$$

COMPOUND A

TEST RESULTS—COMPOUND A						
TEMPERATURE, °C ENVIRONMENT WEIGHT, % RECOVERED						
235	N ₂ /H ₂ O	99.1				
325	99.7					
325	AIR	99.3				
325	N ₂ /H ₂ O	98.4				

$$N-N$$
 $F_{15}C_7n$
 O
 nC_7F_{75}

COMPOUND B

TEST RESULTS—COMPOUND B				
235	N₂/H₂O	100		
325	N ₂	99.5		
325	AIR	60		

TABLE VI.- MODEL STUDIES

DEGRADATION AND MASS BALANCE AFTER 48 HOURS

COMPOUND A

TEST RESULTS—COMPOUND A						
TEMPERATURE, °C ENVIRONMENT WEIGHT, % RECOVERED						
235	N ₂	100				
235	N ₂ /H ₂ O —					
325	N ₂	99.8				
325	AIR	98.3				

COMPOUND B

	TEST RESULTS-COMPO	UND B
235	N ₂ /H ₂ O	99.4
325	N ₂	98.9
325	AIR !	98.5
325	N ₂ /H ₂ O	95.5

TABLE VII. - FLUOROPOLYMERS

THERMOGRAVIMETRIC ANALYSES IN NITROGEN

WEIGHT LOSS, PERCENT

TEMPERATURE, °C	HYBRID FLUOROSILICONE 77-108	FLUOROSILICONE 77-028		TRIAZINE FLUOROETHER
300	3	2	TRACE	_
350	5	3	1.5	0.5
400	7.5	5	3.5	2
450	12	10	11	7.5
500	60	55	57	20

TABLE VIII.- FLUOROPOLYMERS

	% WEIGHT LOSS IN NITROGEN			T-+ °C	ELASTOMERIC	
POLYMER	1	10	50	90	Tg*,°C	PROPERTIES
77-028 FLUOROSILICONE	340°C	450°C	495°C	520°C	-50	GOOD
HYBRID FLUOROSILICONE	325	440	485	510	-34	GOOD
1,2,4-OXADIAZOLE-AROM FLUOROETHER	295	330	365	450	-40	FAIR
NITRILE TRIMERIZATION (FLUOROETHER TRIAZINE)	340	460	535	560	-50	POOR
IMIDOYL AMIDINE (FLUOROETHER TRIAZINE)	350	465	540	560	-61	GOOD
1,2,4-OXADIAZOLE FLUOROETHER	350	440	490	515	-45	GOOD

^{*}Glass transition temperature

INTERRELATIONSHIPS

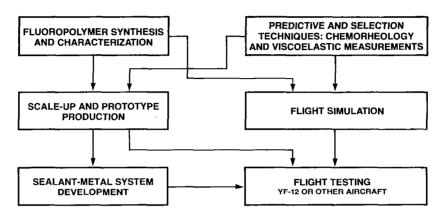


Figure 1.- AST fuel tank sealants program.

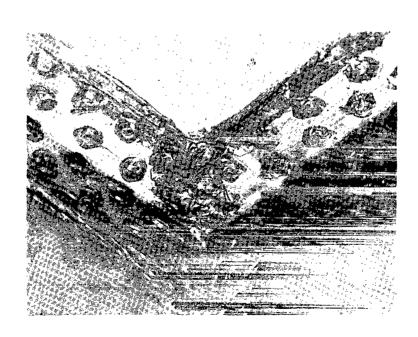


Figure 2.- Small fluorosilicone specimens mounted in fuel tank of YF-12A aircraft.

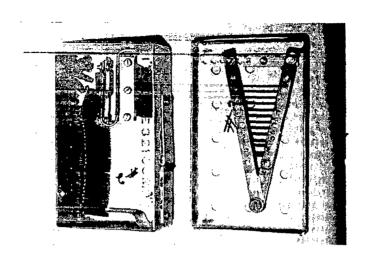


Figure 3.- Fluorosilicone sealant used in fuel tank simulator.

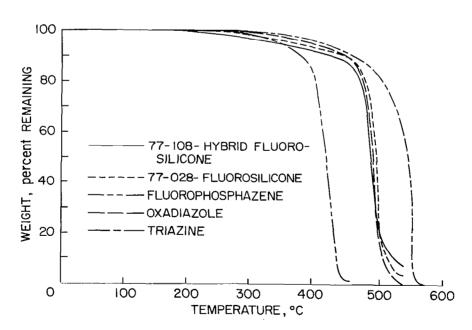


Figure 4.- Thermogravimetric analyses of fluoropolymers in nitrogen.