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# DYNAMIC AND STATIC EVALUATION OF EXPERIMENTAL INTEGRAL FUEL TANK SEALANT MATERIALS

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## NOTICES

# FOREWORD

This Final Report was prepared by Encapsulants and Sealants Technical Service and Development Laboratory, Dow Corning Corporation, Midland, Michigan, under Contract No. F33615-70-C-1422, Project No. 7340, "Nonmetallic and Composite Materials", Task 734005, "Elastomers and Compliant Materials", and covers work performed during the period 1 May 1970 to 1 April 1973. The sponsoring agency is the Elastomers and Coatings Branch, Nonmetallic Materials Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio. The Project Engineer is Mr. W. F. Anspach (MBE).

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## ABSTRACT

A dynamic test apparatus has been designed and fabricated for the purpose of testing experimental aircraft integral fuel tank sealants. The apparatus is capable of closely simulating the conditions encountered by a sealant during a typical aircraft flight. In addition, the apparatus has the flexibility of simulating a virtually unlimited number of stress, temperature, fuel, and pressure conditions, and will automatically repeat the desired test cycle until sealant failure occurs.

A fillet sealed test joint has been used in testing to date and with proper joint designs the apparatus will accommodate other types of seals found in aircraft fuel tank construction.

Dynamic testing to date has been performed on Dow Corning® 77-028, 77-085, 95-526, 77-108, 3M Polyester, a fiber reinforced polyester, and a Viton sealant formulated by the Air Force Materials Laboratory. These materials were all tested in a fillet seal configuration.

High temperature aging in JP-7 fuel vapor was performed on the above materials, and physical properties were determined on the aged specimens both at room temperature and selected high temperatures representative of possible aircraft conditions.

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## I. Introduction

The temperature extremes encountered in the fuel tanks of present and planned supersonic aircraft have pushed currently available fuel containment sealants to the limits of their capability. Now, more than ever before, it is necessary to prove the capability of a fuel tank sealant and predict its service life prior to selection for a particular aircraft.

Although there are "rules of thumb" regarding sealant physical property limits for a particular application, it is not likely that these rules can be confidently applied for all aircraft or sealants under consideration. The greatest problem in doing so is the difficulty in correlating physical properties of statically aged sealant specimens with dynamically stressed sealant in an actual fillet seal configuration.

Although efforts have been made to circumvent the problem by dynamically testing small sealed fuel tank sections, in many cases, these tests require a great deal of assembly time prior to each test, and are frequently quite cumbersome. The amount of material needed to seal one of these tanks makes it very difficult to evaluate experimental sealants as these are usually available in only limited quantities.

A dynamic test device has been designed and fabricated which optimizes on existing tests in the areas of ease and convenience of testing, and greater correlation to an end use aircraft application. The design of the equipment required the gathering of information on aircraft fuel tank conditions from reliable sources throughout the aircraft industry. The information gathered, was in some instances very specific, but required, in several cases some degree of judgement in weighing the importance of the information in relationship to the equipment design. It is felt that the finished piece of equipment is sufficiently flexible to allow for changes in the test program if and when they become necessary.

In addition to the equipment fabrication, a number of experimental sealants have been evaluated in the test apparatus until each of them failed. Failure in this case was defined as the point at which the sealant allowed an actual leakage of fuel through the test joint. The materials tested were Dow Corning® 77-028, Dow Corning® 77-085, a Viton sealant, 2 fluorocarbon/silicone hybrid sealants from Dow Corning, a 3M polyester sealant and a fiber reinforced polyester sealant. Test joints evaluated under this contract were of the fillet type and when possible were fabricated in a specially machined molding jig in order to reproduce identical specimens for every test run.

Standard physical properties were determined on separate test specimens after aging in JP-7 vapor at a specified high temperature for various periods of time. The properties were obtained at -45°F, room temperature, and at elevated temperatures, using an Instron test apparatus equipped with an environmental chamber. Low temperature testing was discontinued during the latter portion of the program, however, due to schedule pressures. The purpose of the physical properties was to provide data which might be correlated to the dynamic test results, and subsequently be used to either strengthen or modify existing "rules of thumb" used in sealant qualifications.

## II. Background Information

Prior to beginning work on this contract, a preliminary design (Figure 1) had been prepared at Dow Corning for what was felt to be an improved fuel containment sealant test apparatus. The design was quite basic in nature, but it did contain essentially all the influencing elements which might be encountered in an aircraft fuel tank, including dynamic stress, high frequency vibration, and fuel tank atmosphere. The test apparatus was designed around the idea that sealability is the key parameter in aircraft sealant performance, and further, that sealability depends upon a complex interaction of physical properties and cannot be readily determined solely from physical property data. It was, therefore, decided that the test would include a representative sealed test joint which would perform a fuel containment function just as it would in an aircraft, and that failure would be defined as the point at which leakage occurred. Other factors were taken into consideration, such as size, quantity of sealant required, and ease of operation, so that the apparatus might be a convenient evaluation tool which would be a help rather than a hindrance to subsequent sealant development programs.

Starting with the basic equipment design, a concentrated effort was made to gather as much pertinent information as possible from some of the major airframe manufacturers with regard to the conditions encountered in a number of aircraft. Personal contacts were made at the North American, Lockheed, McDonnell Douglas, and General Dynamics Aircraft Companies, and written information was obtained from the Boeing Company. The following is a summary of the most important information obtained.

### A. Joint Design

In general, it was felt that the test joint shown in Figure 2 was representative of the average fillet seal. It consists of a circular 3" diameter titanium cup sealed to a titanium plate with the test sealant. Joint deflection is applied by holding the plate stationary at its edge and rotating the cup slightly. In addition, it was suggested that there are areas such as corner joints and fasteners which seem to be critical sealing points. The corners because of the multidirectional stresses, and the fasteners primarily because of the sheer number of them to be sealed without a flaw.

### B. Joint Movement

The original apparatus design included a high frequency vibration input, which, as concluded by all persons contacted, was unnecessary. It was, however, suggested that in addition to the torsional deflection originally proposed, there should also be a joint opening deflection. The design was subsequently changed to include these modifications, as well as the fastener sealing.

The degree of joint movement depends to some degree on the type and thickness of structural members used. Aluminum military aircraft structures for example tend to have rather thick structural members (1/2" to 3/4" in some locations) and are relatively inflexible. Titanium aircraft, however, due to the type of construction used and the unique nature of the metal, tend to be more dynamic in character.

Those sources contacted regarding aluminum aircraft, felt minimal movement should be expected in the sealed joints with which they had experience, while one source working with titanium structures felt that it was reasonable to expect .008"-.010" maximum movement. In another instance, while specific joint deflections were not available, joint movement in a titanium aircraft has been translated into a sealant requirement of 18 to 20% elongation for satisfactory performance at operating temperatures.

One report (AFTR-6187) was cited as measuring the actual deflections on a B-45 aircraft by mounting a deflection meter on a structural member and monitoring the movement of the aircraft skin during flight. The report indicated frequent deflections of .005" and occasional deflections up to .030". Although this was not a current work, it was concluded by the source that it should be relatively applicable to present aircraft.

## III. Temperature Profile

Most military aircraft encounter peak skin temperatures of 375°F or less, with the peak temperature usually accounting for less than 5% of the total flight time. The remaining flight time consists of moderate speeds generating skin temperature between 200°F and 300°F, or subsonic speeds possibly accompanied by sub-zero skin temperatures.

Of more concern, at present, are aircraft cruising at extremely high speeds. Maximum skin temperatures for the SST were projected at 440°-450°F at certain points on the aircraft. Approximately 70-75% of the flight time was to have consisted of peak temperature exposure, requiring a fuel containment sealant, ideally, to last approximately 30,000 hours at that temperature.

Even more stringent are the temperature requirements of the SR-71 type of aircraft which generate peak temperatures in the 550-600°F range, with the peak temperature again accounting for a large percentage of the total flight time.

Sub-zero temperature extremes have been the subject for much discussion. The generally accepted low temperature extreme is -65°F, but at least two sources felt that -65°F was unrealistically low. Only one instance of actual temperature measurement was cited, with the lowest temperature being recorded at -45°F during flight at sub-sonic speeds. It is conceivable, however, that ground temperatures down to -65°F could be encountered by an aircraft in isolated instances, and that a sealant with poor low temperature flexibility might be caused to fail due to the high joint deflections present during taxi and takeoff.

Although the information obtained was not as specific as expected in some areas, it is felt that sufficient background was obtained to set up a reasonably realistic set of dynamic test parameters.

## IV. Test Equipment Capability

Based on specifications set forth at the inception of this contract, as well as the information summarized in the preceding section, the dynamic test device (refer to Figures 3 through 6) was designed to include the following capabilities:

1. Temperature capability between -65°F and 600°F with either extreme obtainable within 15 minutes.

2. Torsional deflection - adjustable over at least 0-.030" rotary movement measured at the perimeter of the test cup. Rate of deflection covers a range of 0-30 deflections per minute, both clockwise and counter clockwise.

3. Joint opening deflection - adjustable over at least 0-.030" opening at the perimeter of the test cup. Deflection rate is adjustable between 0-100 deflections per minute.

4. Chambers may be evacuated, pressurized with air or nitrogen, or the air or nitrogen may be bled into the chambers while evacuated.

5. Fuel may be metered into the chambers at an adjustable rate, or they may be filled at a more rapid rate through a separate fuel line. Fuel may also be rapidly evacuated from the chambers at any time.

6. Sealant failure, indicated by leakage of fuel through a test joint, is detected by a liquid sensing thermistor circuit in the vacuum outlet of the secondary chamber. When leakage is detected, the sensor automatically terminates operation of the affected chamber.

7. All test functions are controlled by a program card timer which can be programmed for the desired sequence of events and recycled indefinitely.

## V. Principles and Mechanics of Equipment Operation

Figure 3 shows a cross section of one of the three test cylinders. Each cylinder is divided into a primary and secondary chamber by a titanium diaphragm. A circular 3" titanium cup sealed onto the diaphragm with the test sealant acts as the test joint, and effectively separates the two chambers by covering four 5/8" openings in the diaphragm. Heat is applied to the cylinders via radiant heat lamps capable of producing +600°F, and the cylinders can be cooled to a possible -65°F by $LN_2$ injection through cooling coils welded to the interior of the chambers. Jet fuel and fuel vapor can be cycled in the primary chamber at temperatures prescribed by the fuel tank temperatures of the aircraft for which the sealants are being tested.

A hollow shaft through the lower chamber is attached to the test cup by means of a splined socket arrangement and is used to effect a torsional displacement of the cup. A solid shaft running through the hollow tube contacts the back side of the titanium diaphragm, and, through the use of a pneumatic cylinder, deflects the diaphragm, thereby causing an opening of the sealed joint around the perimeter of the test cup. The degree of displacement as well as the number of displacements per unit time can be adjusted to approximate the conditions in a particular aircraft.

Pressures in the two chambers are regulated in such a manner that the secondary chamber is at a slightly lower pressure than the primary chamber. In the event of a sealant failure during the course of a test, fuel or fuel vapor enters the secondary chamber and is sensed by a detection apparatus. A relay activated by the detection circuit shuts off the fuel supply, heat, and refrigeration, and failure is visually indicated by a pilot lamp.

## VI. Test Cycle Programming

Testing consists of repetition of a simulated aircraft flight cycle. In order to synchronize the various test functions such as heat, refrigeration, etc., a program card timer capable of switching up to 30 functions is used for all three units. This instrument uses easily programmable plastic cards and provides a wide latitude of possible simulated test conditions (Figure 7).

The following test cycle has been used on all specimens to date: (Note: temperatures are merely representative)

| Cumulative Time | Test Conditions |
|---|---|
| 0 Hours | Pressure control on, fuel bypass line open to fill chamber, fuel metering valve set at 2 cc/min., all other functions off. |
| 0.30 Hours | Fuel bypass closed, joint deflection on. Heat on, temperature rises to 250°F. |
| 1.00 Hours | Fuel evacuated from chamber, temperature rises to 550°F. |
| 2.50 Hours | Heat off, deflection off. |
| 2.77 Hours | Recycle to time 0. |

## VII. Sealant Materials

Sealants evaluated under this program included:

1. Viton I - A fluorocarbon sealant formulated by the Air Force Materials Laboratory.

2. Dow Corning® 77-028 - A fluorosilicone sealant.

3. Dow Corning® 77-085 - A low modulus fluorosilicone sealant.

4. Dow Corning® 95-526 - An experimental fluorocarbon/silicone hybrid sealant with polymer backbone as follows:

$$\left[\begin{array}{lcl} CF_3 & & CF_3 \\ CH_2 & & CH_2 \\ CH_2 & & CH_2 \\ Si & CH_2CH_2(CF_2)_6CH_2CH_2 & Si{-}O \\ CH_3 & & CH_3 \end{array}\right]_{12}$$

5. Dow Corning® 77-108 - An experimental low modulus fluorocarbon/silicone hybrid sealant with polymer backbone as follows:

$$\left[\begin{array}{lcl} CF_3 & & CF_3 \\ CH_2 & & CH_2 \\ CH_2 & & CH_2 \\ Si & CH_2CH_2(CF_2)_2CH_2CH_2 & Si{-}O \\ CH_3 & & CH_3 \end{array}\right]_{12-14}$$

6. 3M Polyester

7. 3M Polyester - Same as above but modified with fiber reinforcement.

Evaluation of these materials was exploratory in nature and will hopefully provide a sufficiently broad base from which to direct further, more exhaustive studies.

## VIII. Static Fuel Vapor Aging

Fuel vapor aging was performed using the apparatus shown in Figure 8. Tensile and tear specimens were pre-weighed and placed into the test chamber in stainless steel mesh baskets. The chamber was sealed and vacuum was applied to maintain an internal pressure of 4 psia. JP-7 jet fuel was metered into the chamber at approximately 2 cc/min. and the temperature was raised to a point coinciding with the high temperature vapor portion of the dynamic test cycle. The jet fuel supply system used only new fuel, which was discarded after each test.

Several specimens were removed from the chamber at selected intervals of aging, and physical properties were determined after the specimens were dried for 2 hours at 200°F and reweighed for weight loss determination. Total aging time coincided approximately with the total high temperature dwell time experienced by the material in the dynamic test up to the failure point of the fillet sealed test specimen.

## IX. Physical Property Determinations

The sealant specimens used in the static aging were prepared and tested as follows.

The Viton I material was prepared by the Air Force Materials Laboratory and submitted in sheet form for testing. The polyester, fluorosilicone and fluorocarbon/silicone hybrid sealants were two-part materials as received and had to be fabricated into sheet stock at Dow Corning. These materials were catalyzed according to the individual specifications and mixed by hand with a spatula. The materials were then de-aired in a vacuum chamber for approximately one hour, and pressed into slabs .062" thick. The polyester materials were cured for 4 hours at 200°F followed by 2 hours at 400°F and the silicone materials were cured for one hour at 200°F followed by a one hour post cure at 300°F.

Die "C" tensile specimens and Die "B" (ASTM-D412) tear specimens were cut from the cured slabs. These were then tagged, weighed and placed in the fuel vapor aging chamber. As specimens were removed from the chamber after aging, they were heated in a circulating hot air oven for 2 hours at 200°F to remove any absorbed jet fuel, then reweighed to determine weight loss.

Tensile and tear strength, and elongation were then determined on an Instron test apparatus at room temperature, the temperature at which the aging was performed and, in some cases, at a selected sub-zero temperature such as -45°F.

In the early portion of the test program, adhesion evaluations were also conducted on Viton and several of the silicone materials using 180° screen peel specimens on 1" x 3" titanium panels (Ti-13V-11Cr-3Al alloy). The silicone specimens were prepared by first etching the titanium panels in a 7% hydrofluoric/21% nitric acid solution and a final cleaning in acetone before applying the appropriate adhesion primers. Following the priming procedure, the sealant was applied to the test panels to a thickness of approximately 1/8". Clean, primed, 1" wide aluminum screen was then pressed slightly into the sealant and an additional ~ 1/16" of sealant was applied over the screen and smoothed with a spatula. The specimens were cured for 8 hours at room temperature, followed by one hour at 300°F.

Later in the test program it was necessary to postpone the adhesion testing due to the quantity of tests scheduled to be run. For the most part adhesion has been secondary in importance to other factors causing failure in the dynamic testing so far, thus, lessening the immediate need for complete adhesion data. The necessary data will be picked up during the continuing work on this program.

X. Dynamic Test Procedure

Dynamic test specimens were cleaned and primed prior to sealing using the same materials and techniques as explained in the static test procedures. The cleaned, primed test cups were then placed in the molding jig illustrated in Figure 9, and sealant was injected into the jig around the outside of the cup. The titanium panels were then placed in the jig on top of the inverted cup and the top platen of the molding jig was placed on top of the plate. The entire assembly was placed in a press and vulcanized at the conditions for that particular material.

Finished specimens (Figure 2) were then bolted to the bottom halves of the dynamic test apparatus and deflection levels were set at .005" torsional movement and .005" joint opening using the deflection meter set up shown in Figures 10 and 11. The test chambers were then completely assembled and the test cycle was initiated. When leakage through the test joint was indicated electronically, the standard procedure was to first check the leak detector housing for the presence of jet fuel, and then disassemble the affected chamber and visually examine the specimen. The specimen was then removed and rechecked for leakage by pulling a vacuum on one side of the specimen and checking for air leakage through the sealant fillet. This was accomplished by placing a bell jar half filled with water on one side of the panel and inspecting for air bubbling when the vacuum was drawn.

Recently a technique was used on several specimens for determining the internal failure pattern on specimens which had leaked. It was shown that a flowable silicone fluid/lead salt paste could be drawn through the ruptured specimens under vacuum, thereby filling the leak path with a relatively radio opaque material. By exposing the specimens, backed with Polariod film, to x-ray it was then possible to obtain photographs of the internal size and shape of the leak paths (Figures 15 and 16).

## XI. Static Test Results

Physical properties on the aged sealants are shown in Tables I through VII. Adhesion was satisfactory on all static specimens tested except for the 95-526 (FCS-610). Additional effort at bonding of this system is justified. The critical areas for concern, however, are the relatively poor properties at 550°F. Hot elongation and tear propagation resistance are particularly important when viewed in relationship to the dynamic deflection model shown in Figure 12. This figure is an enlarged cross section of a test joint in the area of contact between the test cup and plate. Assuming that (c) is an imaginary sealant filament, it can be observed that as the filament moves from point (b) to point (a), it decreases in length, until at (b) the length is essentially zero. In the test specimens being used, however, it can be assumed that a very thin film of sealant could exist at that point.

Since the joint opening and torsional deflection are set at predetermined levels during the testing, it is possible to calculate the extension of any filament (c). It is recognized that the preceding model is a gross simplification of a very complex stress analysis, but it does indicate that extremely high sealant extension can occur near the joint contact point. The high extension will be somewhat tempered by the fact that during hand injection filleting of a preassembled aircraft structure, the sealant might not be forced completely into the contact area. Nevertheless, it is certain that either adhesive or cohesive failure will occur if sealant is forced within .100" of the contact point, based on the high temperature physical properties obtained thus far and the joint opening and torsional deflections of .005" each.

## XII. Dynamic Test Results

The dynamic test parameters used and the test results for each material are shown in Tables VIII through XV.

The failure of the sealants tested to date can be accounted for largely by several basic failure modes. The fluorosilicone specimens tested at high temperature (550°F), for example, failed during the first few cycles due to inadequate high temperature physical properties. This was evidenced by the small splits and/or large fractures around the fillet shown in Figures 13 and 14.

Several of the Viton I specimens under the same conditions failed after only a few cycles due to rupturing of voids which had been incorporated into the fillet during the fabrication of the specimens. As the fabrication problems were minimized, the Viton I functioned for longer periods of time in the dynamic test (19-21 cycles) and failure could be more closely attributed to splitting and a slight degree of degradation.

Thermal degradation played a more significant part as sealants were tested at lower temperatures over longer periods of time (40-100 cycles).

The degradation of the silicone and the fluorocarbon/silicone materials was generally manifested as a softening of the seal with some hardening and cracking at the outer surface, and finally, by splitting through the fillet.

The Viton I had a tendency to harden and develop a crazed surface. Ultimately, splits formed in the fillet, causing failure.

Some specimens as indicated in the tables, did not fail, but were terminated at 100 cycles in order to test other materials. These specimens all exhibited surface hardening and crazing.

## XIII. Summary and Conclusions

The ultimate goal of this contract was, of course, to provide valid and reliable assessments of the capabilities of aircraft fuel tank sealants. In order to accomplish that end, it has been necessary to design and fabricate a test apparatus completely new to the field of materials evaluations. In so doing, the already numerous considerations of the materials evaluation area have been superimposed on the design and procedures problems associated with any new test apparatus of such complexity. Thus, it has been necessary to closely scrutinize the validity of each new piece of test data with an eye toward possible improvements in the test procedures and equipment, even if those modifications would reduce the significance of data already accumulated.

Two problems have been identified during this contract period that may affect the validity of the data. The first is excessive sealant reversion during test in the cycling apparatus which contradicts results obtained in the static evaluation and also that of data obtained in actual aircraft application. It is suspected that this is due to possible air leakage in the vacuum system during testing, probably in the flange seal area between the primary and secondary chamber halves. New flange seals are being fabricated to solve this problem.

Secondly, it has just recently been discovered that a significant amount of torsional movement is being absorbed by the stainless steel torque shafts when the torsional deflections are set up at room temperature. Therefore, a torsional input sufficient to deflect the specimens plus and minus .005" at room temperature is sufficient to deflect the specimens significantly more than .005" when the natural sealant strength reduction occurs at temperatures of 450°F to 550°F during testing. This high strain condition could lead to premature failure of the test specimens. New torque shafts are being fabricated of hardened steel which is much less susceptible to flexing than is the stainless steel currently being used. This should minimize the deviation between room temperature and high temperature deflection.

Based on the preceding assumptions regarding the dynamic test apparatus, it is felt that most of the materials tested to date are capable of somewhat better performance than has been indicated by the present test data. One can, however, make some relatively sound judgements regarding the critical performance factors of the sealant materials.

High temperature physical properties are of the most critical importance if a material is to last more than a few cycles. High temperature elongation and tear propagation resistance appear to be the most important of those properties.

Once a material survives the first few cycles, thermal stability becomes an important factor, in that a significant weight loss and shrinkage or a drastic change in high temperature elongation will generally lead to splitting and, ultimately, to failure of the seal.

Adhesion, although admittedly a necessary requirement for a sealant, has not proven to be a dominating factor in the failure of any of the materials except the polyester. Adhesive failure in that case appeared to be primarily a function of the high modulus and hardness of the material.

TABLE I

Static Fuel Vapor Aging

Dow Corning® 77-028

Initial Physical Properties

| Pulled At | "B" Tear #/in | psi Tensile | % Elongation | #/in Peel | Bond Failure |
|---|---|---|---|---|---|
| -45°F | 134 | 1750 | 505 | 30 | 100% Screen |
| Room Temperature | 34 | 760 | 400 | 9 | 100% Screen |
| 550°F | 11 | 125 | 70 | 0.9 | 100% Screen |

Physical Properties

After 120 Hrs. @ 550°F in JP-7 Vapor

| | | | | | |
|---|---|---|---|---|---|
| -45°F | 168 | 675 | 360 | 27 | 100% Cohesive |
| Room Temperature | 29 | 260 | 215 | 9 | 100% Cohesive |
| 550°F | 4.9 | 8 | 5 | 1 | 100% Cohesive |

(% Weight Loss - 3.5)

Physical Properties

After 204 Hrs. @ 550°F in JP-7 Vapor

| | | | | | |
|---|---|---|---|---|---|
| -45°F | 113 | 630 | 355 | 35 | 100% Cohesive |
| Room Temperature | 35 | 295 | 235 | 9.3 | 100% Cohesive |
| 550°F | 5.6 | 6 | 5 | 1 | 100% Cohesive |

(% Weight Loss - 8.8%)

TABLE II

Static Fuel Vapor Aging

Dow Corning® 77-028

Initial Physical Properties

| Pulled At | "B" Tear #/in | psi Tensile | % Elongation |
|---|---|---|---|
| Room Temperature | 34 | 266 | 180 |
| 450°F | 8.8 | 100 | 73 |

Physical Properties

After 24 Hrs. @ 450°F in JP-7 Vapor

| Pulled At | "B" Tear #/in | psi Tensile | % Elongation |
|---|---|---|---|
| Room Temperature | 27 | 255 | 167 |
| 450°F | 5.8 | 29 | 23 |

Physical Properties

After 72 Hrs. @ 450°F in JP-7 Vapor

| Pulled At | "B" Tear #/in | psi Tensile | % Elongation |
|---|---|---|---|
| Room Temperature | 32 | 225 | 130 |
| 450°F | 9.2 | 40 | 45 |

Physical Properties

After 96 Hrs. @ 450°F in JP-7 Vapor

| Pulled At | "B" Tear #/in | psi Tensile | % Elongation |
|---|---|---|---|
| Room Temperature | 31 | 277 | 87 |
| 450°F | 11.1 | 87 | 80 |

TABLE III

Static Fuel Vapor Aging

Dow Corning® 95-526 (FCS-610)

Initial Physical Properties

| Pulled At | "B" Tear #/in | psi Tensile | % Elongation | #/in Peel | Bond Failure |
|---|---|---|---|---|---|
| -45°F | 600 | 3345 | 280 | 40 | 100% Screen |
| Room Temperature | 50 | 970 | 380 | 9.5 | 100% Screen |
| 550°F | 8.5 | 140 | 45 | 1 | 100% Screen |

Physical Properties

After 120 Hrs. @ 550°F in JP-7 Vapor

| | | | | | |
|---|---|---|---|---|---|
| -45°F | 500 | 3675 | 235 | 48 | 100% Adhesive |
| Room Temperature | 48 | 810 | 260 | 7.5 | 100% Adhesive |
| 550°F | 9.5 | 40 | 10 | 1 | 100% Adhesive |

(% Weight Loss - Nil)

Physical Properties

After 204 Hrs. @ 550°F in JP-7 Vapor

| | | | | | |
|---|---|---|---|---|---|
| -45°F | 225 | 2755 | 203 | 38 | 100% Adhesive |
| Room Temperature | 75 | 1040 | 288 | 1 | 100% Adhesive |
| 550°F | 15 | 34 | 10 | 1 | 100% Adhesive |

(% Weight Loss - 1.8%)

TABLE IV

Static Fuel Vapor Aging

Dow Corning® 77-108 (FCS-210)

Initial Physical Properties

| Pulled At | "B" Tear #/in | psi Tensile | % Elongation |
|---|---|---|---|
| Room Temperature | 122 | 725 | 565 |
| 500°F | 42 | 120 | 120 |

Physical Properties

After 24 Hrs. @ 500°F in JP-7 Vapor

| | | | |
|---|---|---|---|
| Room Temperature | 89 | 824 | 483 |
| 500°F | 9.2 | 74 | 87 |

Weight Loss (+1.1%)

Physical Properties

After 72 Hrs. @ 500°F in JP-7 Vapor

| | | | |
|---|---|---|---|
| Room Temperature | 65 | 567 | 367 |
| 500°F | 8.2 | 160 | 127 |

Weight Loss (+5.0%)

Physical Properties

After 168 Hrs. @ 500°F in JP-7 Vapor

| | | | |
|---|---|---|---|
| Room Temperature | 64 | 454 | 213 |
| 500°F | 17 | 83 | 42 |

Weight Loss (+2.8%)

TABLE V

Static Fuel Vapor Aging

Viton I Sealant

Initial Physical Properties

| Pulled At | "B" Tear #/in | psi Tensile | % Elongation |
|---|---|---|---|
| -45°F | -- | 3485 | 255 |
| Room Temperature | 200 | 695 | 2215 |
| 550°F | 15 | 45 | 265 |

Physical Properties After Aging 72 Hrs. @ 550°F in JP-7 Fuel Vapor

| | | | |
|---|---|---|---|
| -45°F | > 800 | 2535 | 80 |
| Room Temperature | 106 | 642 | 1161 |
| 550°F | 19 | 48 | 93 |

Weight Loss - 1.4%

Physical Properties After Aging 120 Hrs. @ 550°F in JP-7 Fuel Vapor

| | | | |
|---|---|---|---|
| -45°F | > 800 | 2640 | 80 |
| Room Temperature | 145 | 615 | 1007 |
| 550°F | 12 | 40 | 54 |

Weight Loss - 3.9%

TABLE VI

Static Fuel Vapor Aging

Vicon I Sealant

Initial Physical Properties

| Pulled At | "B" Tear #/in | psi Tensile | % Elongation |
|---|---|---|---|
| Room Temperature | 146 | 825 | 955 |
| 500°F | 14.7 | 89 | 340 |

Physical Properties

After 24 Hrs. @ 500°F in JP-7 Vapor

| | | | |
|---|---|---|---|
| Room Temperature | 126 | 948 | 755 |
| 500°F | 13.6 | 98 | 190 |

Weight Loss (-1.6%)

Physical Properties

After 96 Hrs. @ 500°F in JP-7 Vapor

| | | | |
|---|---|---|---|
| Room Temperature | 110 | 731 | 605 |
| 500°F | 14.9 | 93 | 177 |

Weight Loss (-5.8%)

Physical Properties

After 168 Hrs. @ 500°F in JP-7 Vapor

| | | | |
|---|---|---|---|
| Room Temperature | 107 | 525 | 400 |
| 500°F | 11.7 | 67 | 85 |

Weight Loss (-6.5%)

TABLE VII

Static Fuel Vapor Aging

Dow Corning© 77-085

## Initial Physical Properties

| Pulled At | "B" Tear #/in | psi Tensile | % Elongation | #/in Peel | Bond Failure |
|---|---|---|---|---|---|
| -45°F | 120 | 1135 | 465 | 37 | 100% Screen |
| Room Temperature | 33 | 370 | 355 | 18 | 100% Screen |
| 550°F | 6 | 100 | 80 | 2 | 100% Screen |

## Physical Properties

## After 120 Hrs. @ 550°F in JP-7 Vapor

| Pulled At | "B" Tear #/in | psi Tensile | % Elongation | #/in Peel | Bond Failure |
|---|---|---|---|---|---|
| -45°F | 107 | 470 | 385 | 26 | 100% Adhesive |
| Room Temperature | 30 | 260 | 210 | 7 | 100% Screen |
| 550°F | 7 | 16 | 18 | 2 | 100% Adhesive |

Weight Loss - Nil

## TABLE VIII

### Dynamic Testing of Viton I

| CYCLES TO FAILURE | HIGH TEMP. | LIQUID SOAK TEMP. | LOW TEMP. | JOINT OPENING | OPENING RATE | TORSIONAL DEFLECTION | TORQUE RATE | PRESSURE | ATMOSPHERE |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 550°F | 265°F | --- | .005" | 10 cyc/min | .005" | 4 cyc/min | 5 psia | $N_2$ Bleed |
| 3 | 550°F | 245°F | --- | .005" | 10 cyc/min | .005" | 4 cyc/min | 5 psia | $N_2$ Bleed |
| 6 | 550°F | 250°F | --- | .005" | 10 cyc/min | .005" | 4 cyc/min | 5 psia | $N_2$ Bleed |
| 19 | 550°F | 250°F | --- | .005" | 10 cyc/min | .005" | 4 cyc/min | 4 psia | $N_2$ Bleed |
| 21 | 550°F | 250°F | --- | .005" | 10 cyc/min | .005" | 4 cyc/min | 4 psia | $N_2$ Bleed |

Sealant: Viton I

Joint Type: Fillet Seal Ti-13V-11Cr-3Al Alloy

Primer: None

Remarks: Titanium treated with Pasa-Jell. Failures on 1, 3 and 6 cycle specimens were primarily due to rupturing of air bubbles incorporated into the fillet during specimen fabrication and, therefore, do not represent sealant material failures. Longer term specimens showed hardening and some signs of degradation.

## TABLE IX

### Dynamic Testing of Viton I

| CYCLES TO FAILURE | HIGH TEMP. | LIQUID SOAK TEMP. | LOW TEMP. | JOINT OPENING | OPENING RATE | TORSIONAL DEFLECTION | TORQUE RATE | PRESSURE | ATMOSPHERE |
|---|---|---|---|---|---|---|---|---|---|
| 61 | 525°F | 250°F | -- | .005" | 10 $\frac{cyc}{min}$ | .005" | 4 $\frac{cyc}{min}$ | 4 psia | $N_2$ Bleed |
| *100 | 500°F | 250°F | -- | .005" | 10 $\frac{cyc}{min}$ | .005" | 4 $\frac{cyc}{min}$ | 4 psia | $N_2$ Bleed |

Sealant: Viton I

Joint Type: Fillet Seal Ti-13V-11Cr-3Al Alloy

Primer: None

Remarks: Surface hardening on both specimens. The 61 cycle specimen showed some cracking and other evidence of an improperly severe environment (perhaps air leak).

*100 cycle specimen was still functioning when terminated.

TABLE X

Dynamic Testing of Dow Corning 95-526
(FCS-610)

| CYCLES TO FAILURE | HIGH TEMP. | LIQUID SOAK TEMP. | LOW TEMP. | JOINT OPENING | OPENING RATE | TORSIONAL DEFLECTION | TORQUE RATE | PRESSURE | ATMOSPHERE |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 475°F | 225°F | -- | 0 | 10 $\frac{cyc}{min}$ | .010" | 4 $\frac{cyc}{min}$ | 5 psia | $N_2$ Bleed |
| | | | | | | | | | |
| 1 | 420°F | 280°F | -- | .010" | 10 $\frac{cyc}{min}$ | .010" | 4 $\frac{cyc}{min}$ | 5 psia | $N_2$ Bleed |
| | | | | | | | | | |
| 3 | 525°F | 230°F | -- | .003" | 10 $\frac{cyc}{min}$ | .005" | 4 $\frac{cyc}{min}$ | 5 psia | $N_2$ Bleed |
| | | | | | | | | | |
| 2 | 440°F | 235°F | -- | 0 | 10 $\frac{cyc}{min}$ | .010" | 4 $\frac{cyc}{min}$ | 5 psia | $N_2$ Bleed |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |

Sealant: Dow Corning 95-526 (FCS-610)

Joint Type: Fillet Seal Ti-13V-11Cr-3Al Alloy

Primer: Dow Corning 92-040

Remarks: 3 of the 4 specimens cured to a lower modulus than the 4th. These 3 showed small half moon splits after failure. The high modulus specimen had a more massive fracture around most of the fillet. Adhesion appeared good.

TABLE XI

Dynamic Testing of Dow Corning 77-108 (FCS-210)

| CYCLES TO FAILURE | HIGH TEMP. | LIQUID SOAK TEMP. | LOW TEMP. | JOINT OPENING | OPENING RATE | TORSIONAL DEFLECTION | TORQUE RATE | PRESSURE | ATMOSPHERE |
|---|---|---|---|---|---|---|---|---|---|
| 42 | 500°F | 250°F | -- | .005" | 10 cyc/min | .005" | 4 cyc/min | 4 psia | $N_2$ Bleed |
| 62 | 500°F | 250°F | -- | .005" | 10 cyc/min | .005" | 4 cyc/min | 4 psia | $N_2$ Bleed |

Sealant: Dow Corning 77-108 (FCS-210)

Joint Type: Fillet Seal Ti-13V-11Cr-3Al Alloy

Primer: Dow Corning 77-123

Remarks: Elephant hide cracking on surface; 42 cycle specimen was more severely degraded, perhaps because of air leakage into vacuum system.

## TABLE XII

### Dynamic Testing of Dow Corning 77-028

| CYCLES TO FAILURE | HIGH TEMP. | LIQUID SOAK TEMP. | LOW TEMP. | JOINT OPENING | OPENING RATE | TORSIONAL DEFLECTION | TORQUE RATE | PRESSURE | ATMOSPHERE |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 405°F | 210°F | -- | 0 | 10 cyc/min | .010" | 4 cyc/min | 5 psia | $N_2$ Bleed |
| 2 | 500°F | 215°F | -- | .010" | 10 cyc/min | .010" | 4 cyc/min | 5 psia | $N_2$ Bleed |
| 1 | 415°F | 250°F | -- | .004" | 10 cyc/min | .006" | 4 cyc/min | 5 psia | $N_2$ Bleed |
| 3 | 525°F | 260°F | -- | .003" | 10 cyc/min | .005" | 4 cyc/min | 5 psia | $N_2$ Bleed |
| 43 | 450°F | 250°F | -- | .005" | 10 cyc/min | .005" | 4 cyc/min | 4 psia | $N_2$ Bleed |
| *101 | 450°F | 250°F | -- | .005" | 10 cyc/min | .005" | 4 cyc/min | 4 psia | $N_2$ Bleed |

Sealant: Dow Corning 77-028

Joint Type: Fillet Seal Ti-13V-11Cr-3Al Alloy

Primer: Dow Corning 77-078

Remarks: Specimens in units 2 and 3 showed large fractures. Specimens in unit 1 showed small splits. Some adhesive failure on all specimens.

* Still functioning at 101 cycles.

## TABLE XIII

Dynamic Testing of Dow Corning 77-085

| CYCLES TO FAILURE | HIGH TEMP. | LIQUID SOAK TEMP. | LOW TEMP. | JOINT OPENING | OPENING RATE | TORSIONAL DEFLECTION | TORQUE RATE | PRESSURE | ATMOSPHERE |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 540°F | 245°F | -- | .005" | 10 $\frac{cyc}{min}$ | .005" | 4 $\frac{cyc}{min}$ | 5 psia | $N_2$ Bleed |
| 2 | 550°F | 245°F | -- | .005" | 10 $\frac{cyc}{min}$ | .005" | 4 $\frac{cyc}{min}$ | 5 psia | $N_2$ Bleed |
| 2 | 525°F | 275°F | -- | .005" | 10 $\frac{cyc}{min}$ | .005" | 4 $\frac{cyc}{min}$ | 5 psia | $N_2$ Bleed |
| *6 | 550°F | 250°F | -- | .005" | 10 $\frac{cyc}{min}$ | .005" | 4 $\frac{cyc}{min}$ | 5 psia | $N_2$ Bleed |

Sealant: Dow Corning 77-085

Joint Type: Fillet Seal Ti - 13V - 11Cr - 3AL Alloy

Primer: Dow Corning 77-123

Remarks: All specimens exhibited small splits and some adhesive failure.
*Double sealed specimen - fillet applied and cured in two layers.

## TABLE XIV

### Dynamic Testing of Reinforced 3M Polyester

| CYCLES TO FAILURE | HIGH TEMP. | LIQUID SOAK TEMP. | LOW TEMP. | JOINT OPENING | OPENING RATE | TORSIONAL DEFLECTION | TORQUE RATE | PRESSURE | ATMOSPHERE |
|---|---|---|---|---|---|---|---|---|---|
| 1-1/2 | 550°F | 250°F | | | | | | | |
| 12 | 550°F | 250°F | | | | | | | |
| 6 | 525°F | 250°F | | | | | | | |
| 1 | 500°F | 250°F | | | | | | | |
| 4 | 500°F | 250°F | | | | | | | |

Sealant: 3M Polyester Fiber Reinforced

Joint Type: Fillet Seal Ti-13V-11Cr-3Al Alloy

Primer: None - Brush coat of sealant cut with acetone was applied and cured first.

Remarks: Possible inaccurate deflections (see text: Summary and Conclusions).

## TABLE XV

### Dynamic Testing of 3M Polyester

| CYCLES TO FAILURE | HIGH TEMP. | LIQUID SOAK TEMP. | LOW TEMP. | JOINT OPENING | OPENING RATE | TORSIONAL DEFLECTION | TORQUE RATE | PRESSURE | ATMOSPHERE |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 550°F | 250°F | -- | .005" | 10 $\frac{cyc}{min}$ | .005" | 4 $\frac{cyc}{min}$ | 4 psia | $N_2$ Bleed |
| 2-1/2 | 550°F | 250°F | -- | .005" | 10 $\frac{cyc}{min}$ | .005" | 4 $\frac{cyc}{min}$ | 4 psia | $N_2$ Bleed |
| 2 | 500°F | 250°F | -- | .005" | 10 $\frac{cyc}{min}$ | .005" | 4 $\frac{cyc}{min}$ | 4 psia | $N_2$ Bleed |
| 2-1/2 | 500°F | 250°F | -- | .005" | 10 $\frac{cyc}{min}$ | .005" | 4 $\frac{cyc}{min}$ | 4 psia | $N_2$ Bleed |

Sealant: 3M Polyester EC-2288

Joint Type: Fillet Seal Ti-13V-11Cr-3Al Alloy

Primer: None - Brush coat of sealant cut with acetone applied and cured first.

Remarks: The high modulus and hardness of this material at room temperature probably contributed to inaccurate deflections (see text: Summary and Conclusions).

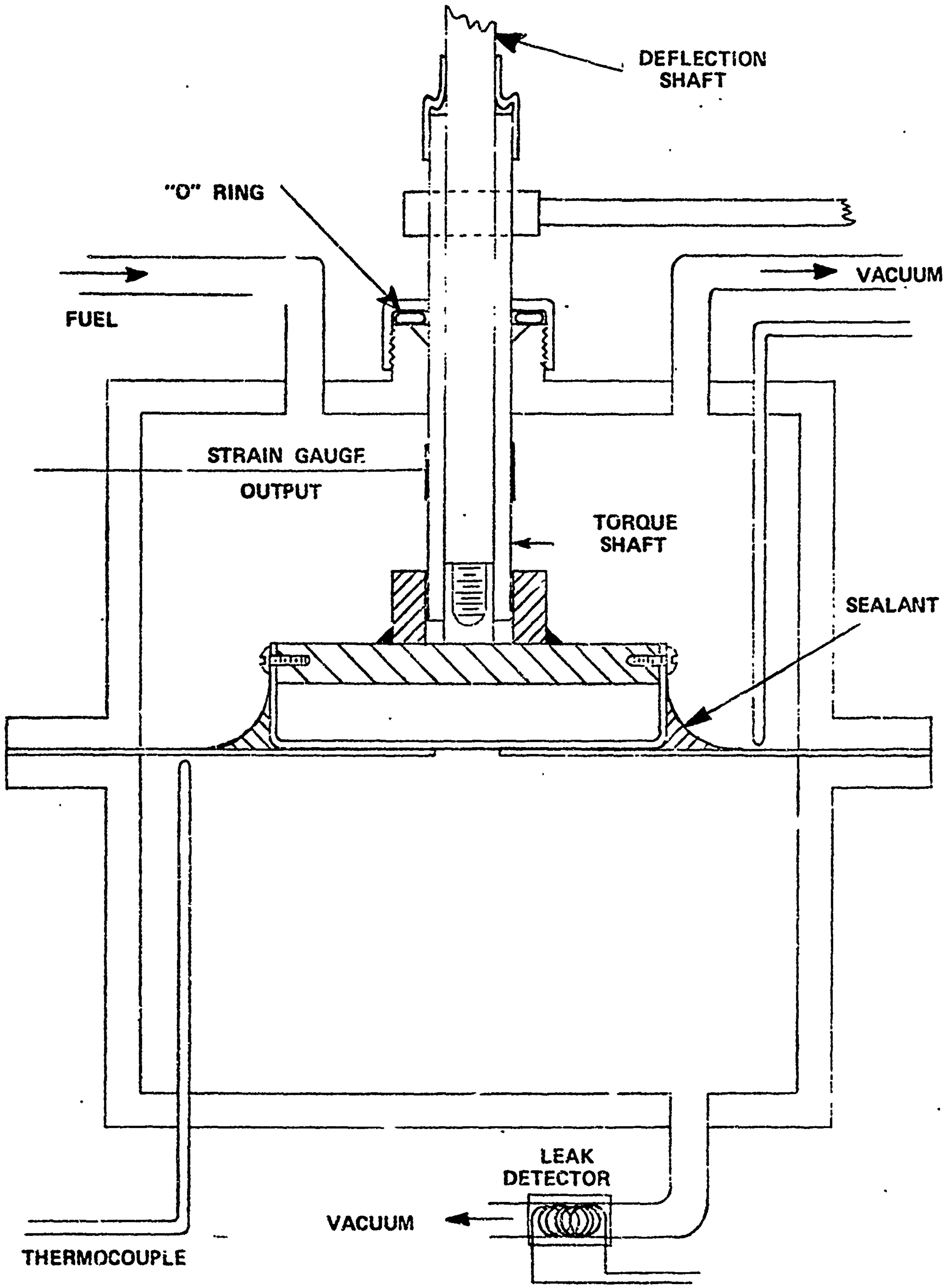
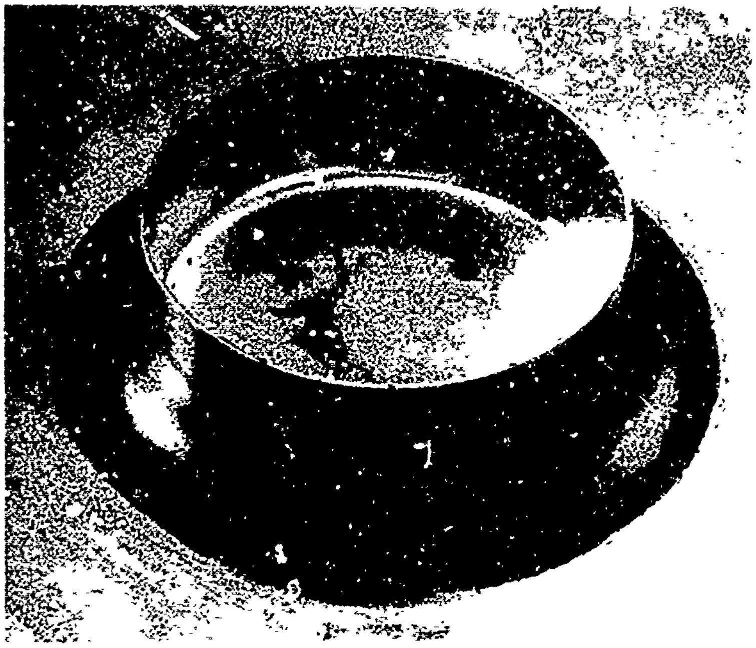
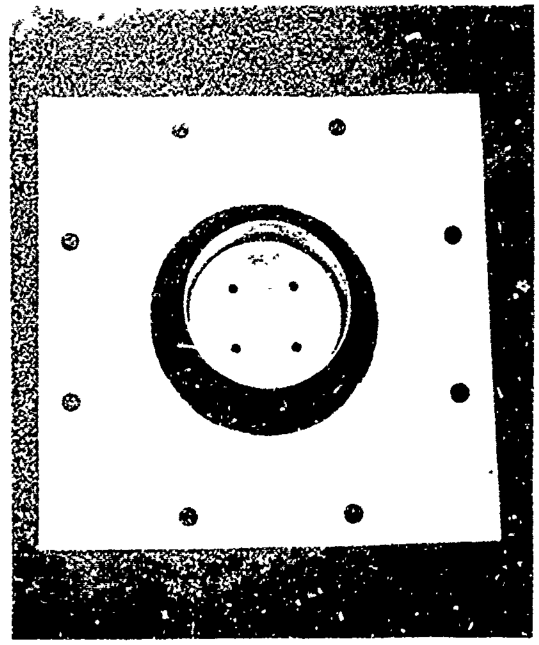



FIGURE 1 - PRELIMINARY CHAMBER DESIGN





TEST JOINT

TEST JOINT PRIOR TO INSTALLATION

FIGURE 2

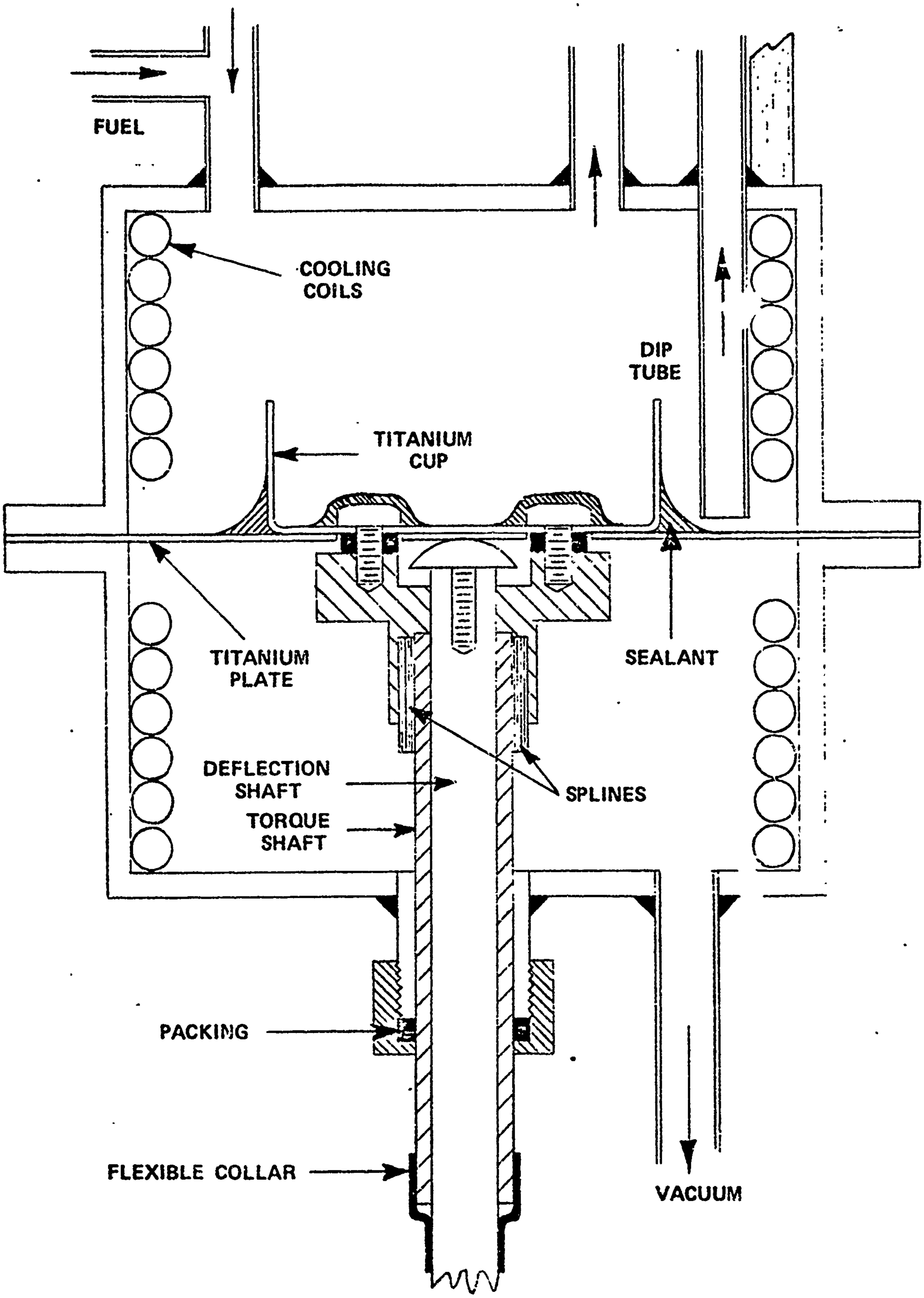
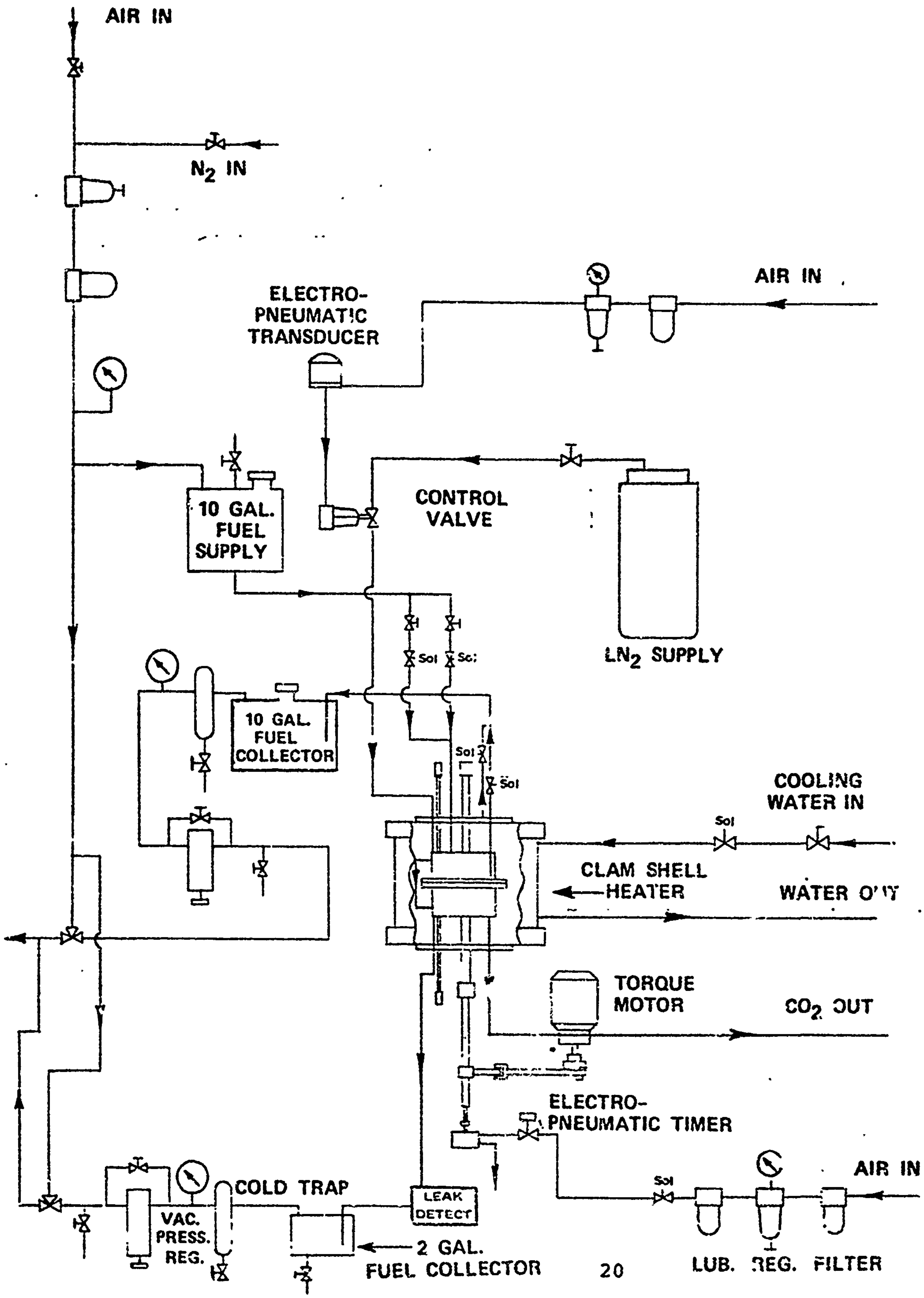
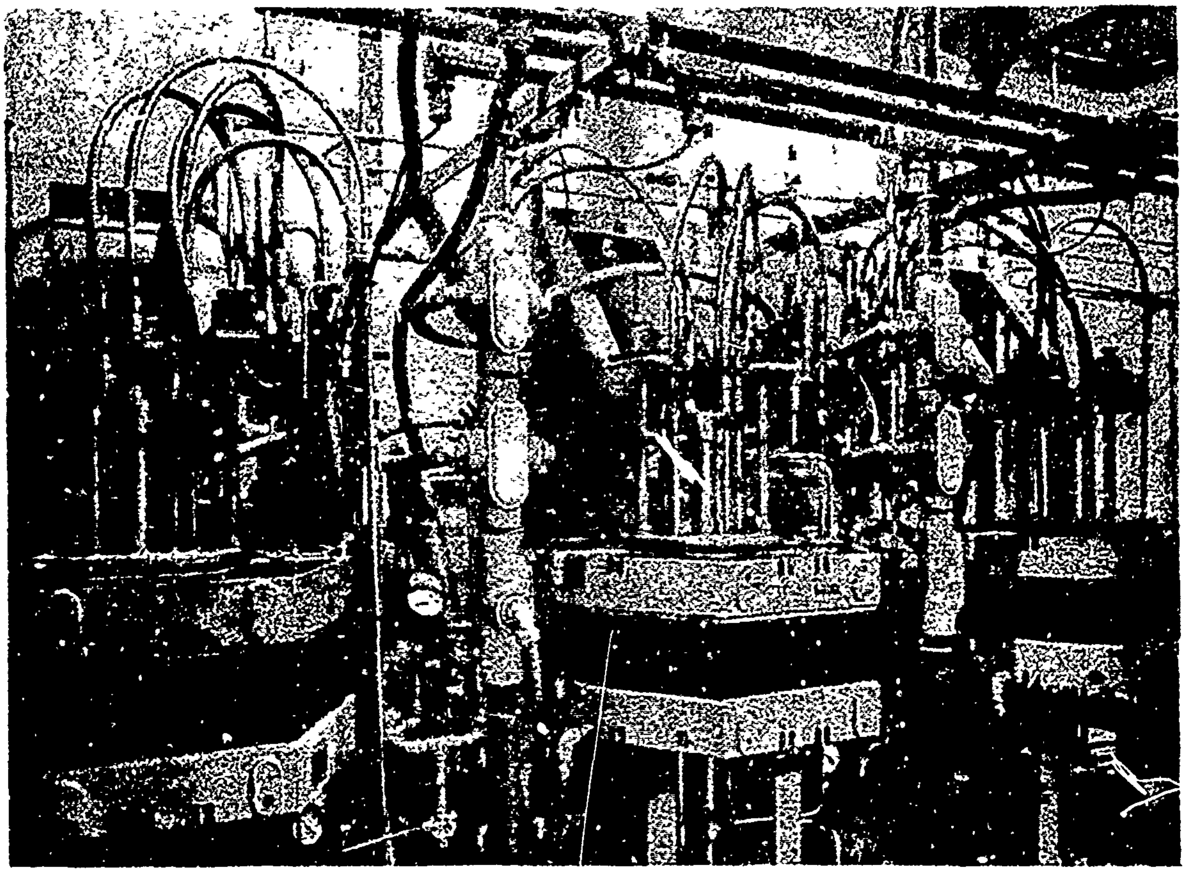



FIGURE 3 - DYNAMIC TEST CHAMBER

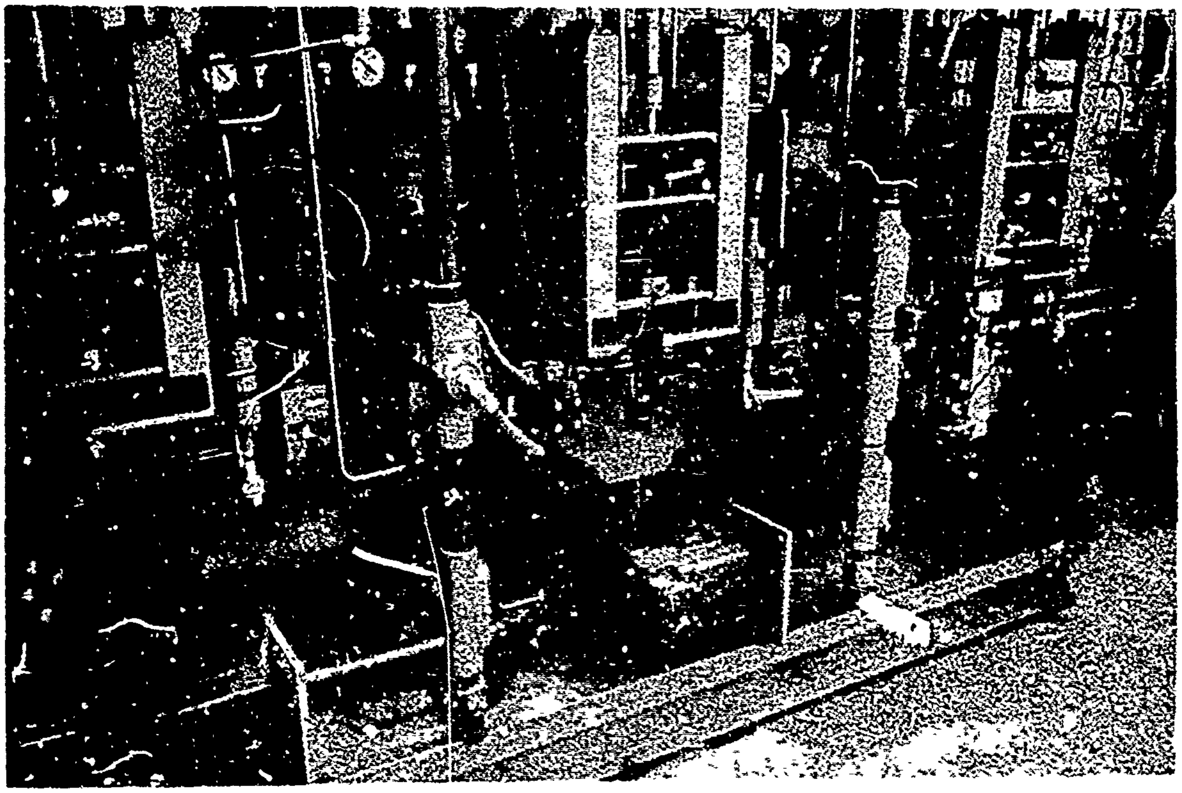



20

FIGURE 4 - PIPING/MECHANICAL SCHEMATIC



TEST CHAMBERS AND ASSOCIATED HARDWARE



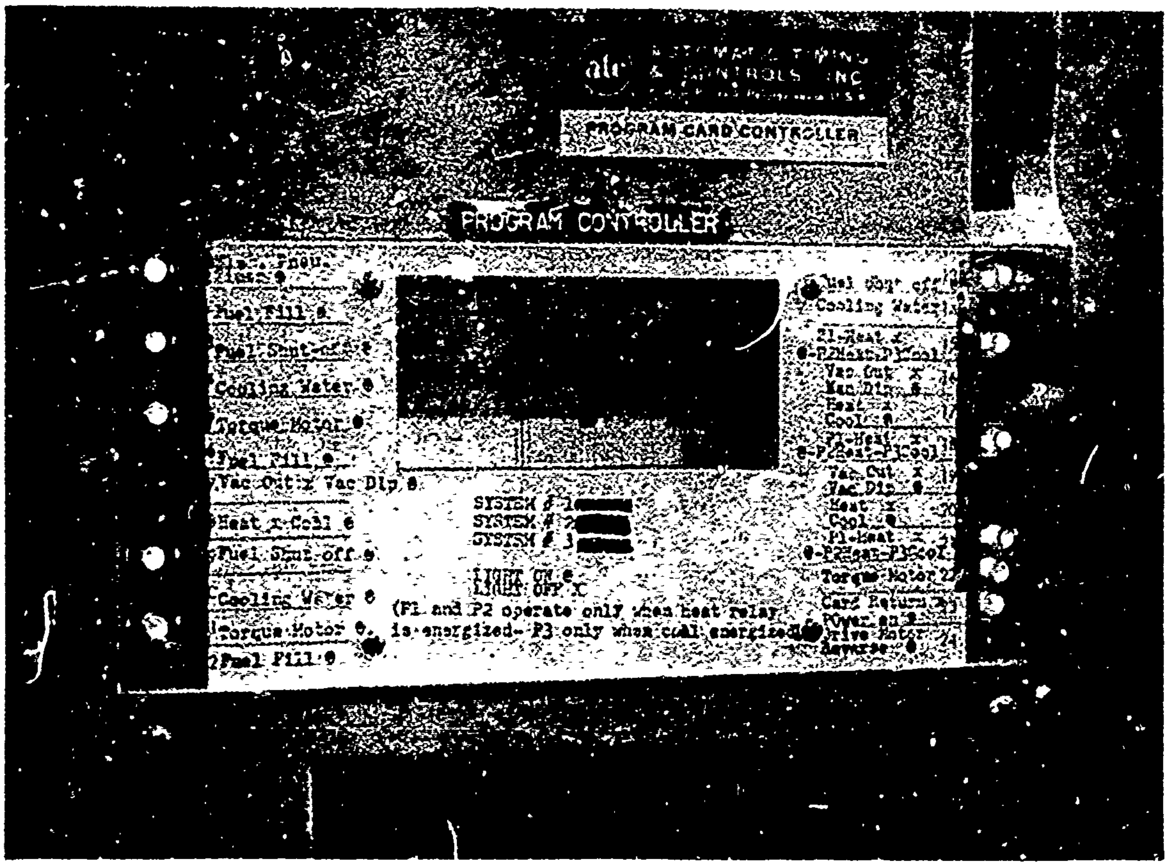
TORQUE & DEFLECTION ASSEMBLIES AND ASSOCIATED HARDWARE

FIGURE 5

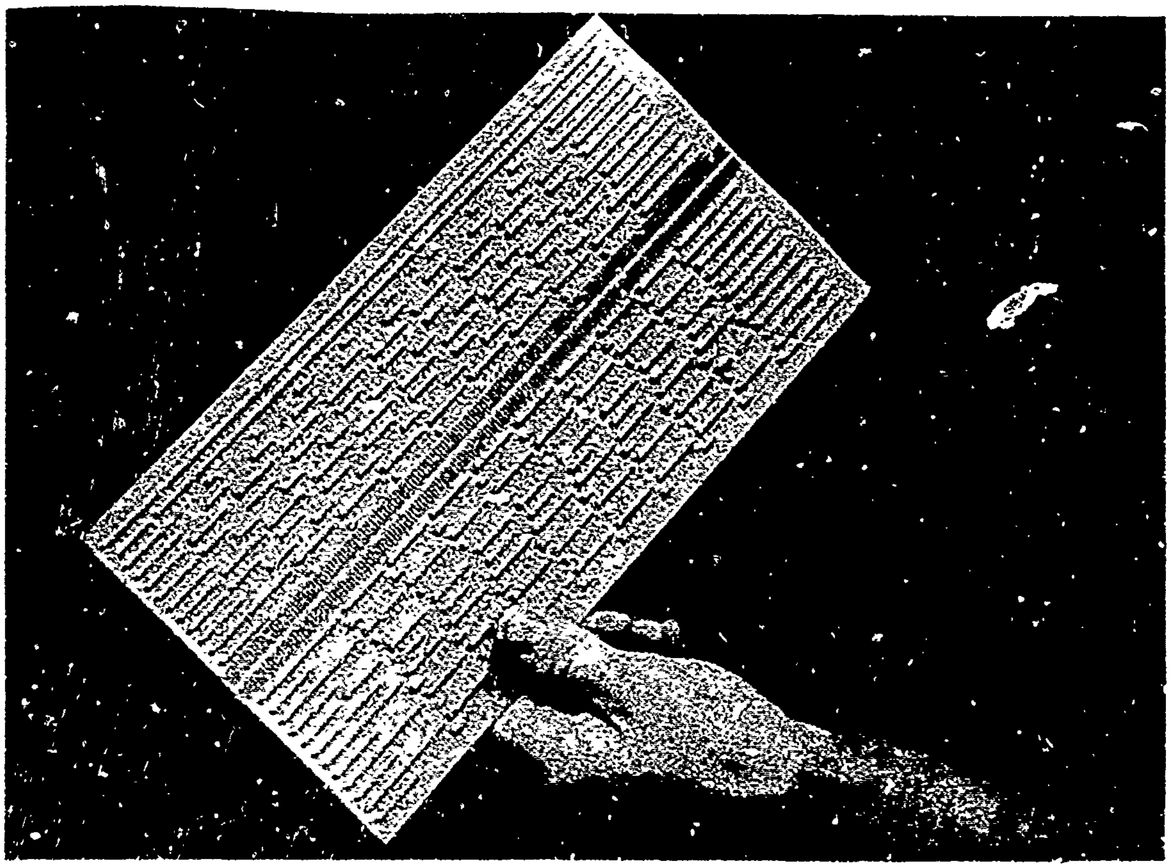
MASTER CONTROL PANEL

INDIVIDUAL UNIT CONTROL

FIGURE 6




PROGRAM TIMER



TYPICAL PROGRAMMED CARD

FIGURE 7

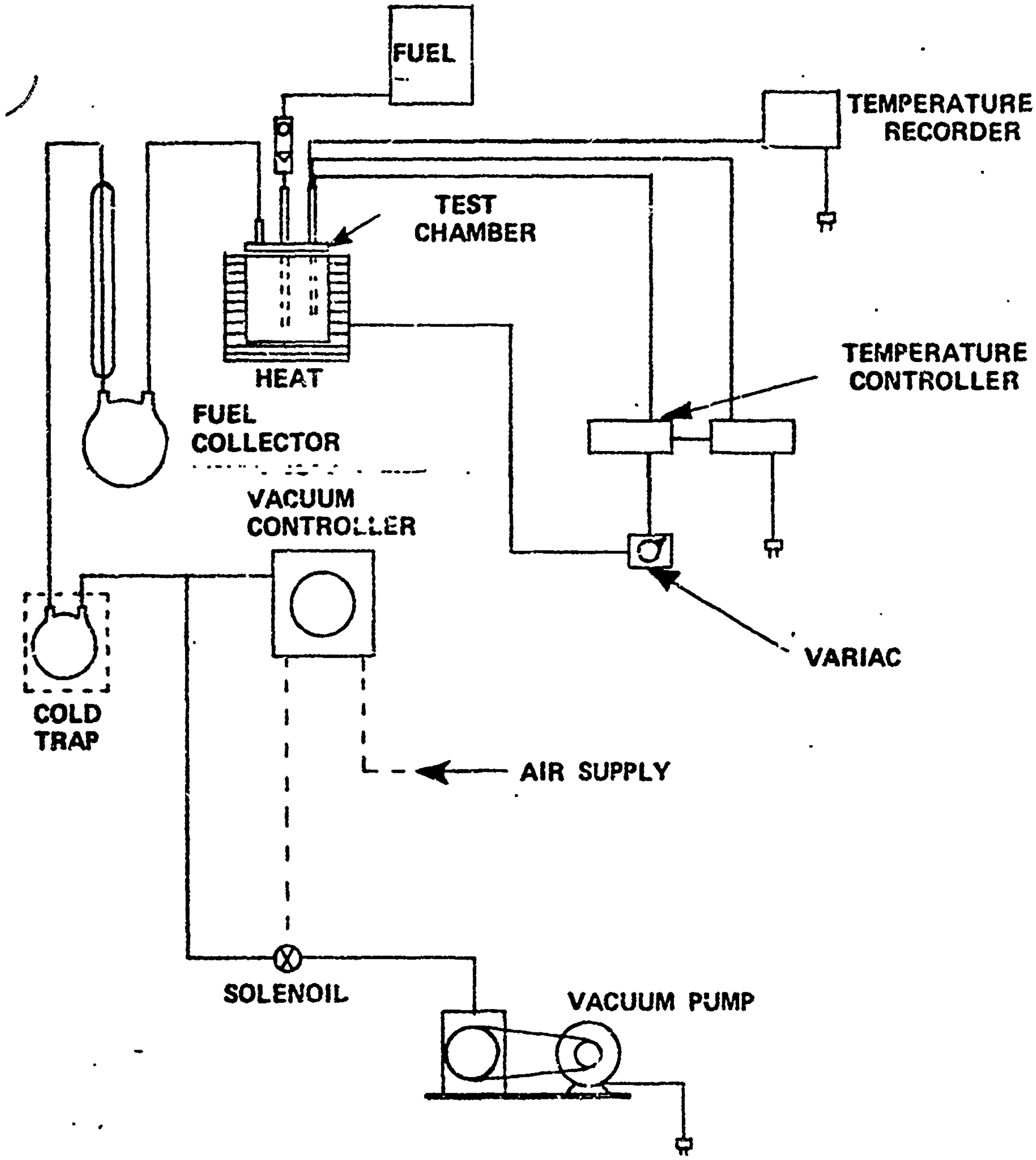



FIGURE 8 - FUEL VAPOR TEST APPARATUS

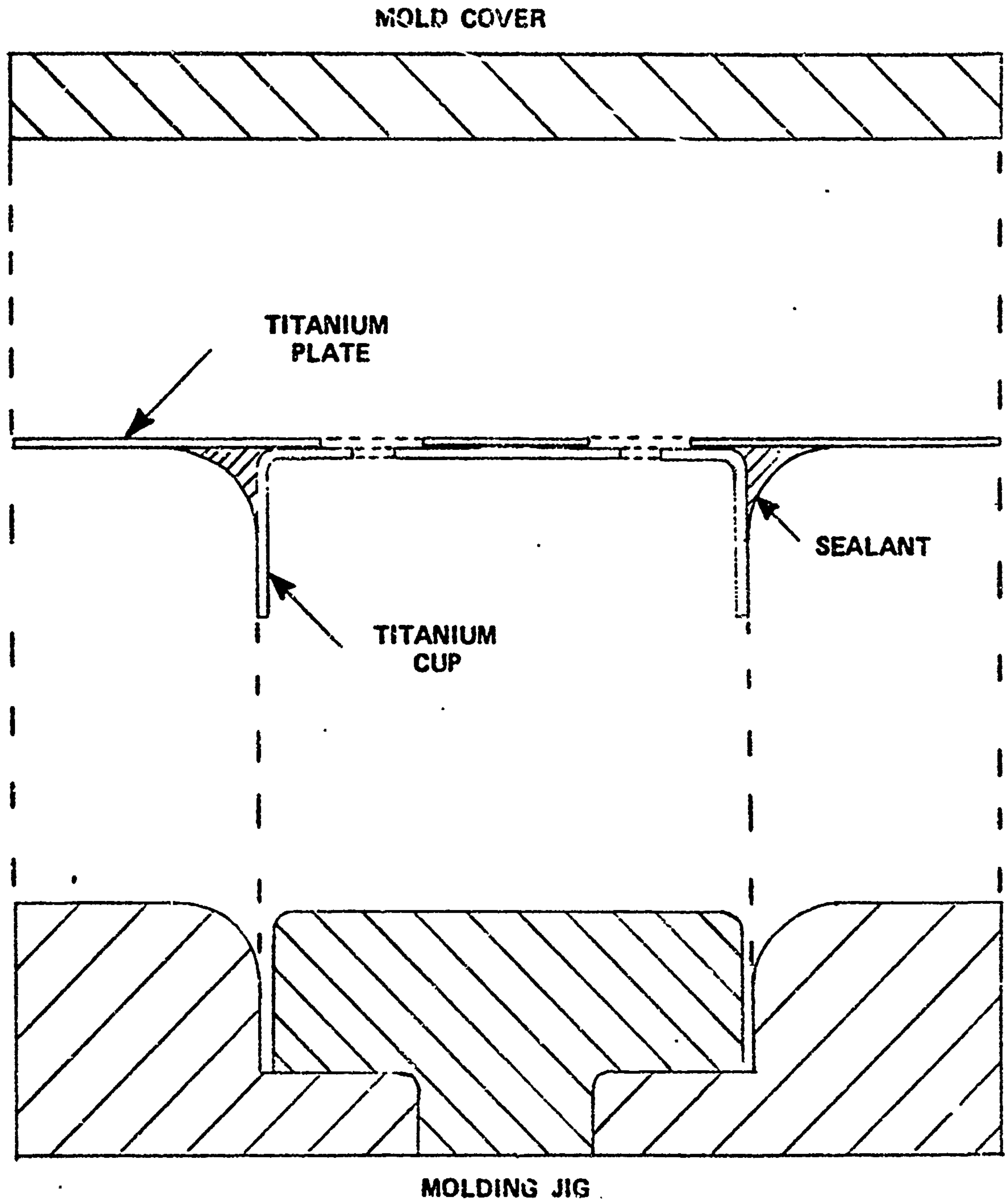



FIGURE 9 - MOLDING JIG AND TEST JOINT

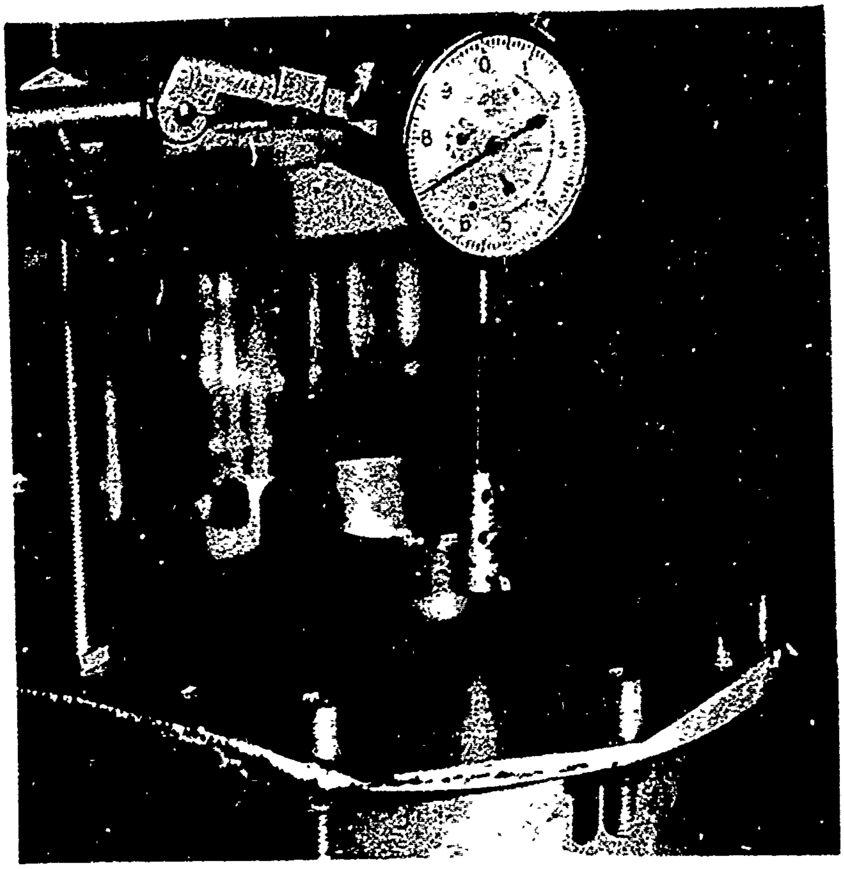


FIGURE 10 - JOINT OPENING SET-UP

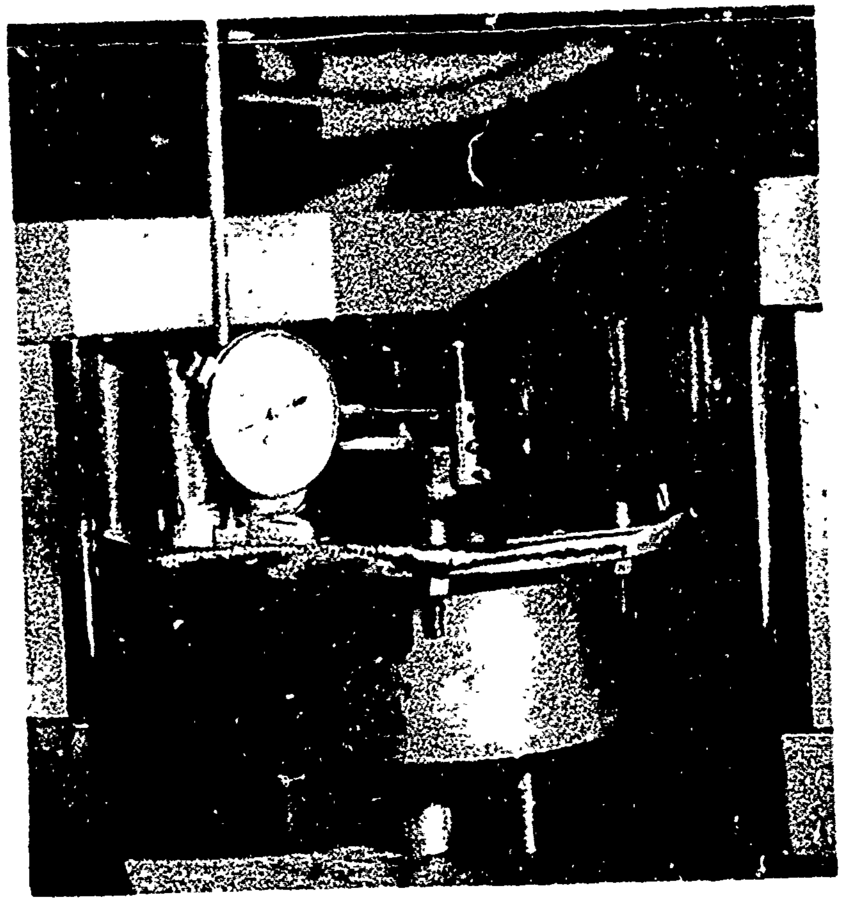


FIGURE 11 - TORSIONAL DEFLECTION SET-UP

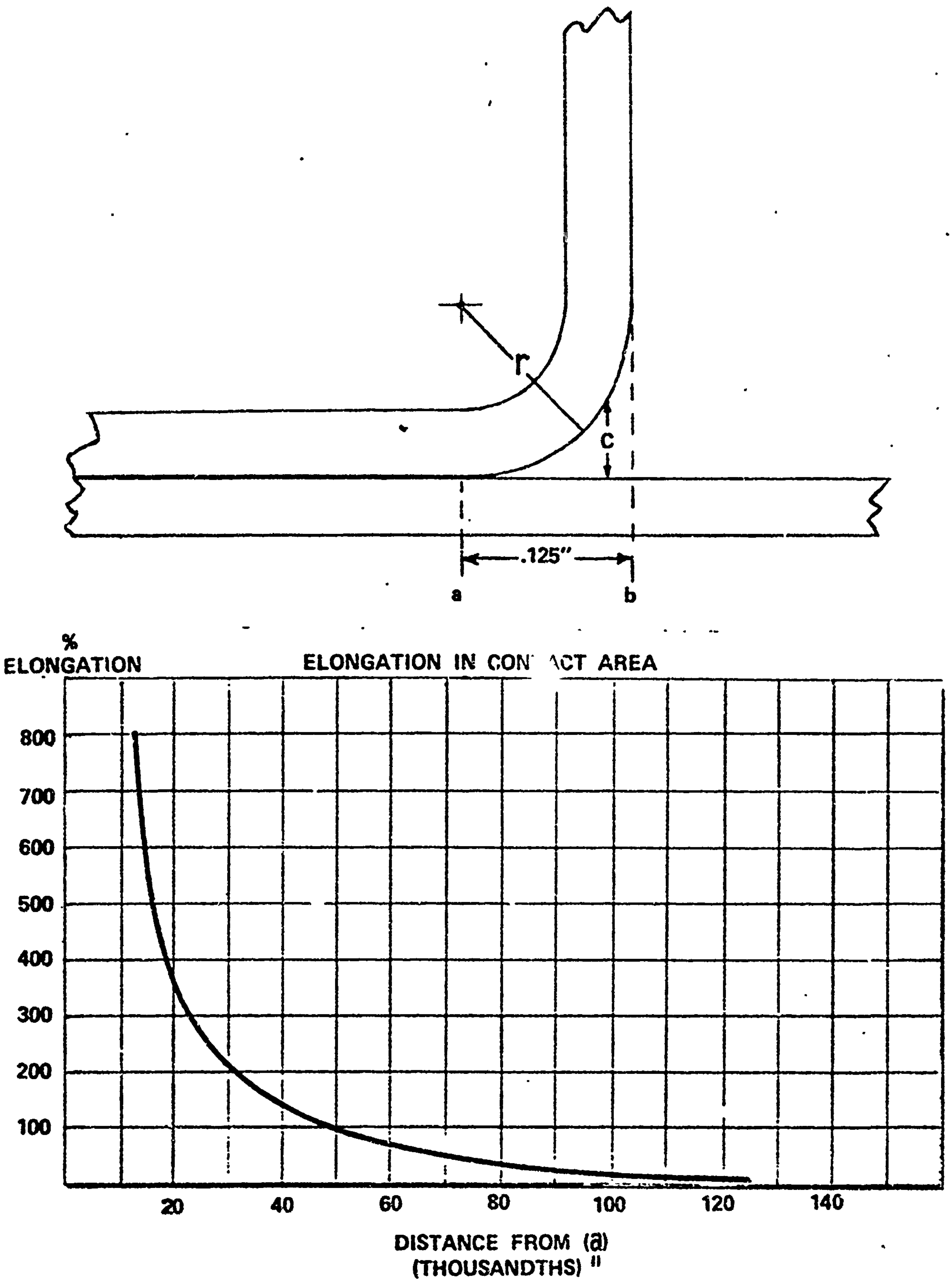
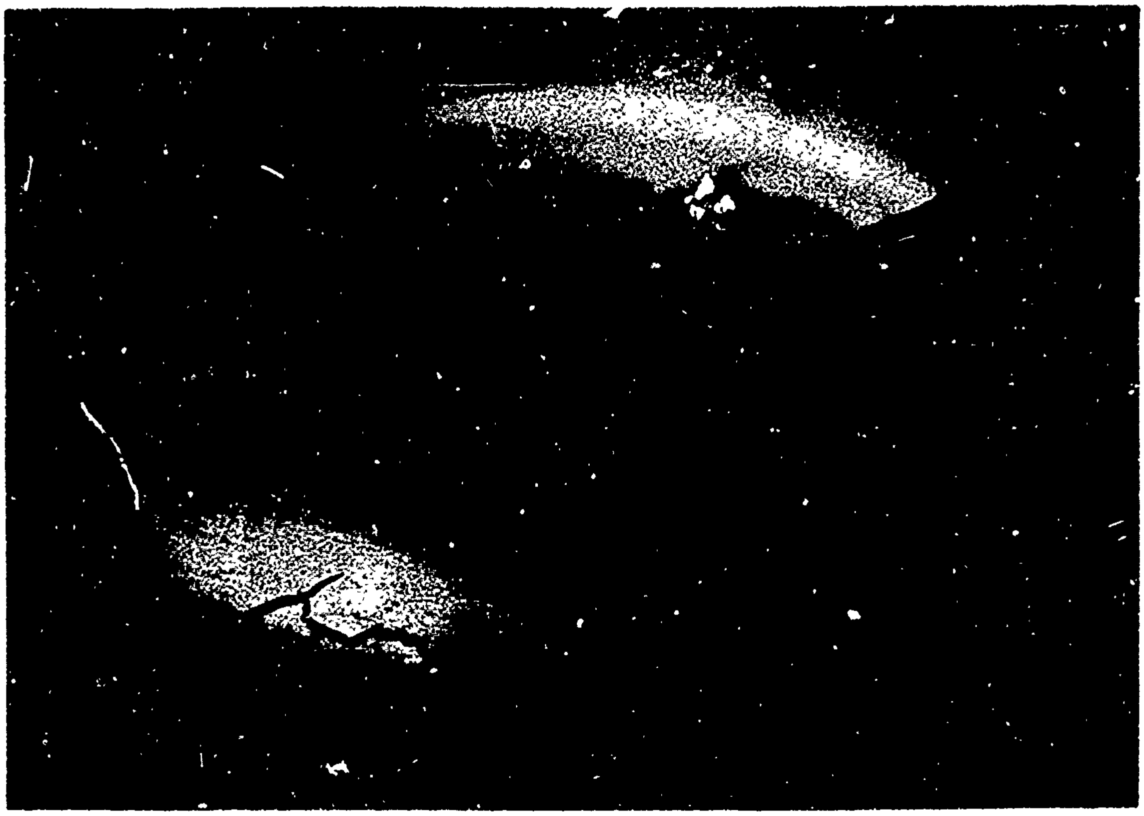



FIGURE 12 - ENLARGED JOINT CONTACT AREA

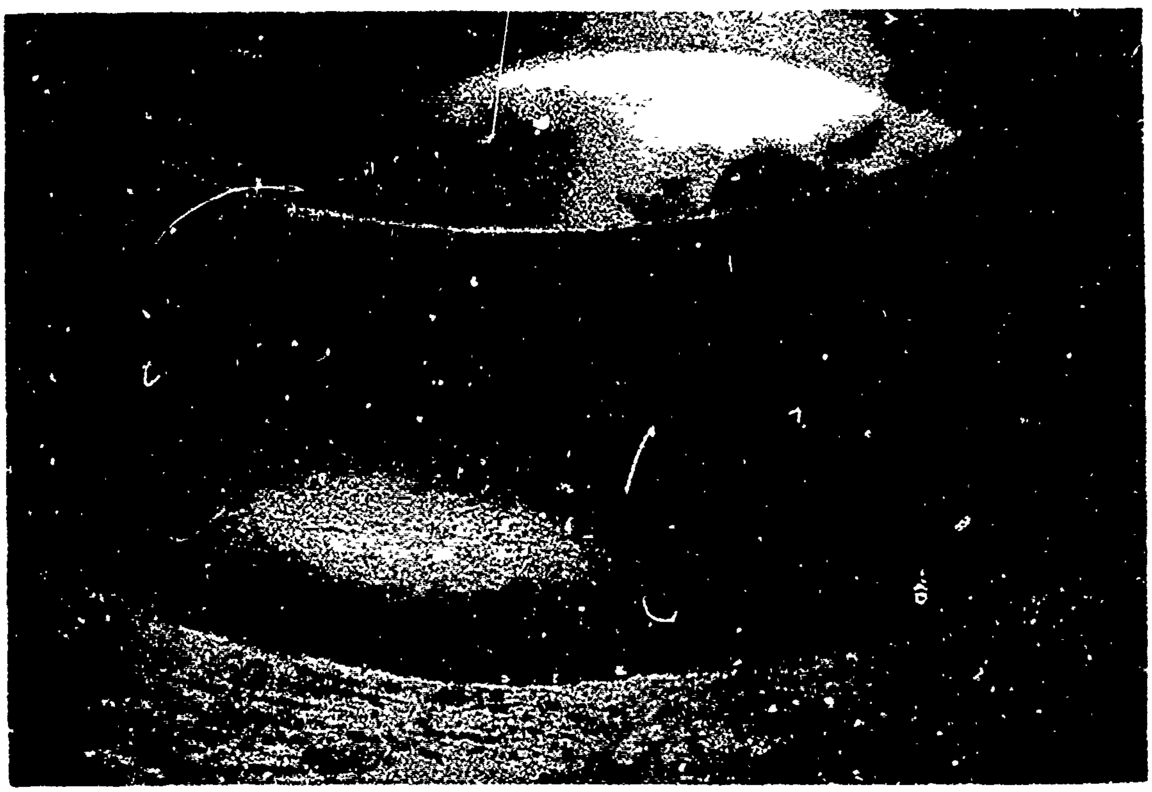


Dow Corning 77-028

Dow Corning FCS-610

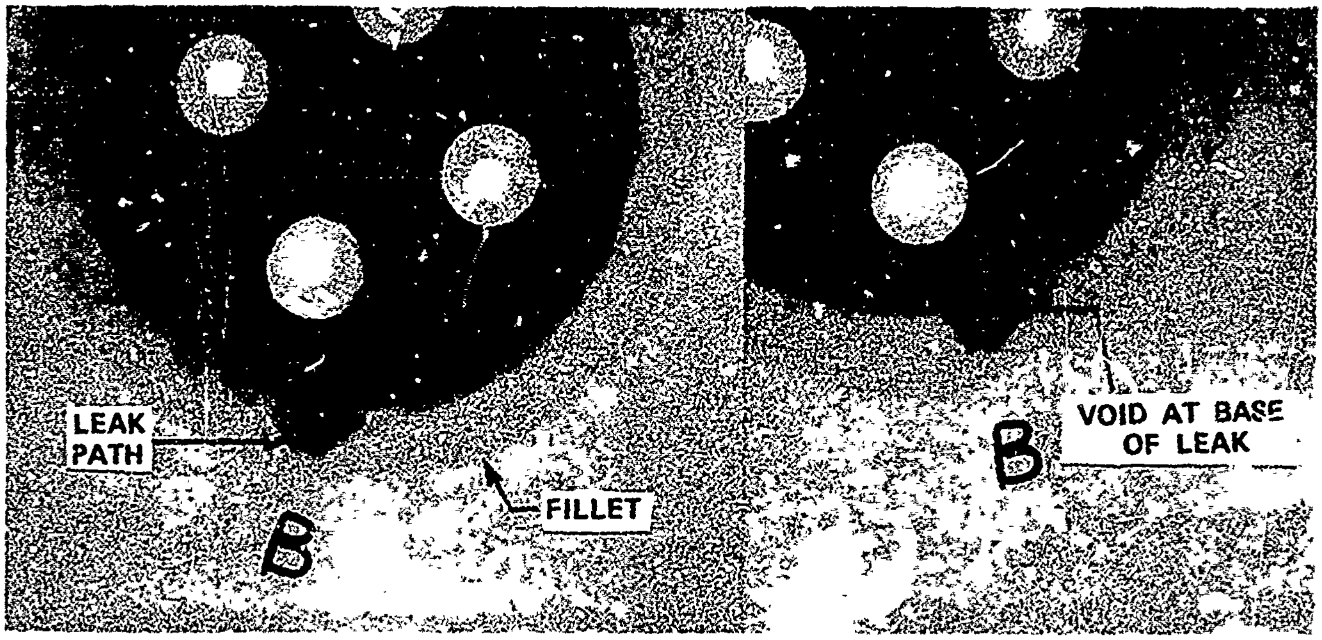
FIGURE 13 - HIGH MODULUS FAILURE

**Dow Corning 77-085**



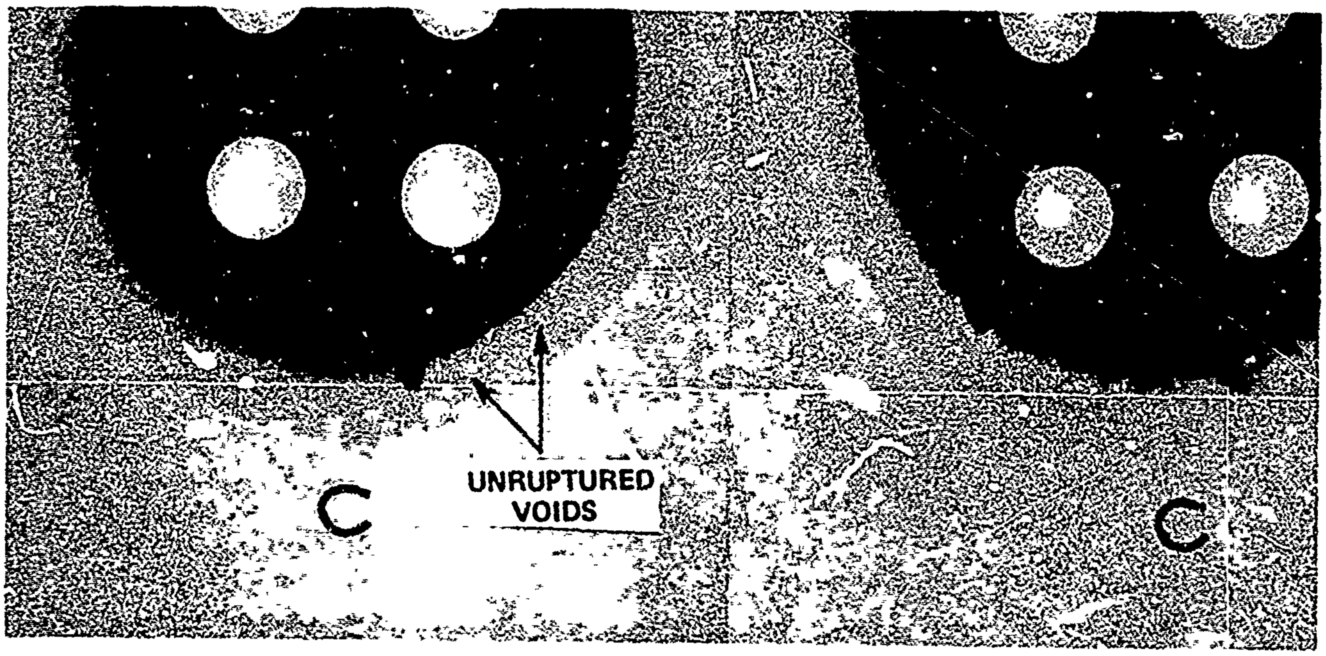
**Dow Corning FCS-610**

FIGURE 14 - LOW MODULUS THERMAL SPLITTING




OVERHEAD EXPOSURE

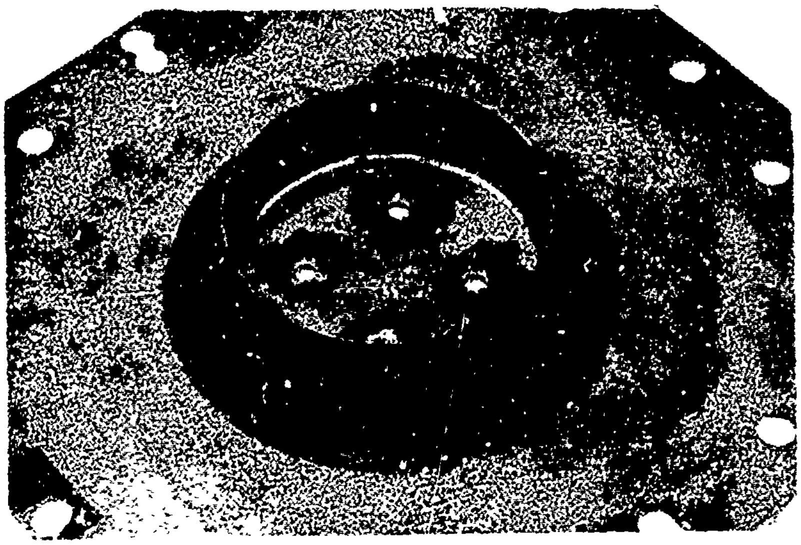
TAKEN AT 30° ANGLE



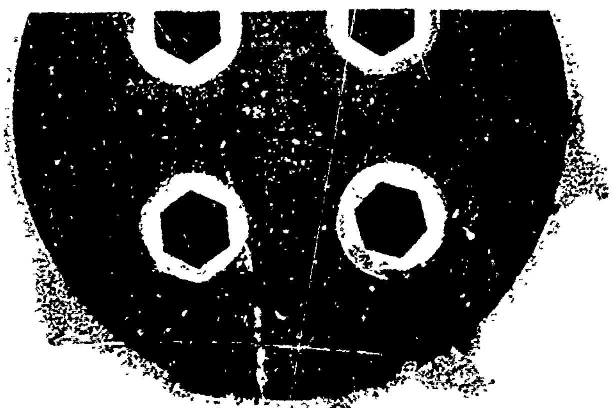

OVERHEAD EXPOSURE

TAKEN AT 30° ANGLE

FIGURE - 15 - X-RAY EXPOSURES OF RUPTURED VITON FILLETS



Overhead X-Ray of Specimen



X-Ray at 30° Angle

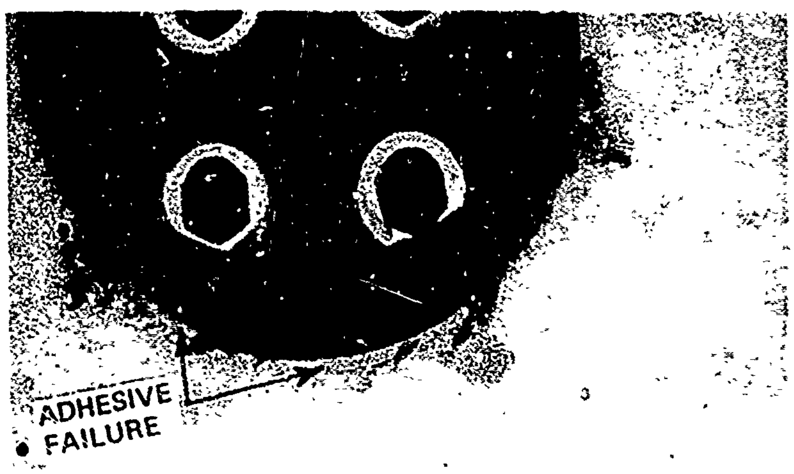



FIGURE 16 - X-RAY EXPOSURE OF SPLIT FAILURE ON DOW CORNING 95-526

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13. ABSTRACT

A dynamic test apparatus has been designed and fabricated for the purpose of testing experimental aircraft integral fuel tank sealants. The apparatus is capable of closely simulating the conditions encountered by a sealant during a typical aircraft flight. In addition, the apparatus has the flexibility of simulating a virtually unlimited number of stress, temperature, fuel, and pressure conditions, and will automatically repeat the desired test cycle until sealant failure occurs.

A fillet sealed test joint has been used in testing to date and with proper joint designs the apparatus will accommodate other types of seals found in aircraft fuel tank construction.

Dynamic testing to date has been performed on Dow Corning 77-028, 77-085, 95-526, 77-108, 3M Polyester, a fiber reinforced polyester, and a Viton sealant formulated by the Air Force Materials Laboratory. These materials were all tested in a fillet seal configuration.

High temperature aging in JP-7 fuel vapor was performed on the above materials, and physical properties were determined on the aged specimens both at room temperature and selected high temperatures representative of possible aircraft conditions.

DD FORM 1 JAN 64 1473

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14.

| KEY WORDS | LINK A | | LINK B | | LINK C | |
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