

MTI PUBLICATION NO. 35
SUMMARY REPORT
ON ELEVATED TEMPERATURE TESTS
FOR ASBESTOS-FREE GASKET MATERIALS

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The Materials Technology Institute of the Chemical Process Industries, Inc. (MTI) is a unique, cooperative research organization representing private industry. Its goal is to conduct generic, nonproprietary studies of a practical nature on the deterioration of materials and equipment used in the process industries.

Through membership in MTI, companies can solve nonproprietary problems of major concern to the process industries, leverage research dollars by participating in the direction and results of MTI studies, capitalize on the expertise of member company representatives, and learn about MTI's accomplishments in a timely fashion.

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EXECUTIVE SUMMARY

Asbestos-reinforced sheet-gasket materials perform in a satisfactory manner at temperatures up to and beyond 750°F. Their performance has made these materials important to the chemical process and allied industries, which operate valves, pumps and piping systems at elevated temperatures. Gaskets of high integrity are vital to the safe and economical functioning of such systems.

However, asbestos has been identified as a health hazard. As a consequence, alternatives to this material are being sought and are being used in numerous applications, including gaskets. The problem is that the favorable properties of asbestos-reinforced gasket materials have not been replicated in asbestos-free compositions. Moreover, the performance of the substitute materials, especially at elevated temperatures, is not well documented. Information is lacking on their limitations and on the appropriate ways in which to use them. The result has been a significant number of failures of asbestos-free sheet-gasket materials in the field.

In order to remedy this situation, the Materials Technology Institute of the Chemical Process Industries, Inc., funded a project with JPAC, Inc., assisted by Centre de Developpement Technologique of Ecole Polytechnique, University of Montreal, to develop and evaluate test procedures and equipment with which to obtain the information needed to permit effective and safe use of asbestos-substitute gasket materials.

The contractors focused their efforts on three principal tests. One test, the creep and relaxation test, evaluates the relaxation properties and residual tensile strength of dumbbell-shaped specimens after exposure for various periods of time at elevated temperatures under a compressive load. Times up to 42 days and temperatures to 750°F were used.

Another test, denoted the hot seal test, was designed to evaluate the tightness, or resistance to leakage, of gasket materials in the form of disks at temperatures to 800°F and exposure times of several days to 2 weeks. Bolting up, heating up, and operating conditions are simulated, including joint relaxation, thermal disturbances and near blowout conditions.

The third test, the fast rupture test (FRT), was designed to measure retained stud load, thickness change and weak-direction residual tensile strength of dumbbell-shaped coupons after exposure for 30 minutes at 1200°F.

Numerous trials have been made with these tests by the contractors. The test equipment and procedures have been refined and thoroughly checked out. They are ready for general use and should provide the proper information with which to assess the probable performance of asbestos-free sheet-gasket materials for service in the chemical and allied industries.

It should be noted that these tests address only the ability of a material to function in the absence of chemical attack. Resistance to chemical attack must be dealt with on a case-by-case basis.

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The investigation was conducted in cooperation with Centre de Developpement Technologique of Ecole Polytechnique, University of Montreal (CDT) at their laboratories. Principal investigators were A. Bazergui (CDT) and J. Payne (JPAC), who were greatly assisted for what seemed countless hours by Luc Marchand (CDT) and Michel Derrere (CDT) in the design and execution of the project hardware, software and processes. The authors are also indebted to project consultants Gerry Hickey and Ed Schumacher for their expertise, guidance and encouragement, particularly in the formative stages and on the design of the hot operational tightness test (HOTT) fixture.

The contributions of R. Offutt of Key Bellvilles, Inc., and J. James of Biach Industries, and their respective companies, for their technical assistance and for the loan of equipment in connection with this work are also gratefully acknowledged.

SECTION 1 INTRODUCTION

Because asbestos has been identified as a severe health hazard, a number of industries have drastically limited their use of asbestos-containing products, including asbestos-containing sheet-gasket materials. As a result, asbestos-containing gasket materials have been rapidly disappearing from the market.

A variety of asbestos-free alternative sheet-gasket materials are now available. Often, however, the composition and properties of these new products are not well documented. In addition, appropriate procedures do not exist to evaluate these materials for the chemical process and petrochemical plant environment. As a consequence, a significant number of failures of substitute sheet-gasket materials have occurred in the field.

Accordingly, the Materials Technology Institute of the Chemical Process Industries, Inc., (MTI) initiated a project to develop equipment and procedures with which to obtain the information needed to characterize alternative sheet-gasket materials and evaluate their probable performance in chemical process and petrochemical plant service. The emphasis of the investigation was on gaskets for bolted flanged joints operating at temperatures up to 750°F.

The project was placed with JPAC, Inc., Long Valley, NJ, assisted by Centre de Developpement Technologique of Ecole Polytechnique, University of Montreal, Quebec. The work done, the results obtained and the inferences drawn from the results are reported in a document entitled "Evaluation of Test Methods for Asbestos Replacement Gasket Materials" by James R. Payne and Andre Bazergui. That document will be available as MTI Publication No. 36.

This report, which was prepared by A.M. Hall, consultant, is a condensation of that document.

SECTION 2 BACKGROUND

IMPORTANT CHARACTERISTICS OF GASKET MATERIALS

Long experience with asbestos-reinforced sheet gaskets in the process industries indicates that the following properties are important in sheet-gasket materials if they are to perform satisfactorily in the process plant environment.

Residual Tensile Strength

This is the room temperature tensile strength of the material after being exposed to an elevated temperature for a period of time. Residual tensile strength is an indicator of the degree to which gasket materials resist disintegration (crumbling and reduction to powder) as well as "blowout" (gross leakage) while in service.

Relaxation

The most common form of relaxation occurring in flanged joints using sheet gasketing results in crushing of the gasketing, causing a decrease in the load applied by the bolts.

Boiled joints sealed with gasket materials that resist relaxation are less likely to leak or fail by gasket blowout. Of course, to be truly meaningful, measurement of relaxation or leak resistance (tightness) must be carried out at the expected service temperature.

Fire Resistance

Fire resistance is the probable performance of gasket materials under the conditions prevailing in an in-plant fire. Residual strength and degree of relaxation, after the material has been exposed for a period of time at a suitable high temperature, are indicators of probable performance in a fire.

TEST MATERIALS

In order to ensure that the test procedures and equipment developed in this project would be capable of discriminating effectively among gasket materials, a number of classes of materials were used in the course of the investigation. In addition, each class was represented by several varieties of commercial gasket materials, listed in Table 1.

As shown in the table, the test materials included a variety of compressed and beater-process asbestos and nonasbestos fiber-reinforced elastomer-bound sheet materials, including sheets with aramid and with glass reinforcement. The other classes were polytetrafluorethylene (PTFE) sheet and flexible-graphite laminates.

TABLE 1
Gasket Materials Used in the Project

Description*	Identification
Asbestos-reinforced, Buna-N rubber binder, compressed sheet process	A1
Asbestos-reinforced, Buna-S rubber binder, compressed sheet process	A6
Aramid-reinforced, Buna-N rubber binder, compressed sheet process	N1
Aramid-reinforced, Buna-S rubber binder, beater process	N2
Aramid-reinforced, Buna-N rubber binder, beater process	N3
Aramid-reinforced, Buna-S rubber binder, compressed sheet process	N4
Aramid-reinforced, Buna-N rubber binder, compressed sheet process	N6
Glass-reinforced, Buna-N rubber binder, compressed sheet process	N7
Plain (virgin) PTFE** sheet	TV
Glass-filled PTFE sheet	TL
Barium sulfate-filled PTFE sheet	TG
Silica-filled PTFE sheet	TS
Flexible-graphite laminates***	
Adhesive bond with 316 SS sheet reinforcement	G2
Mechanical tang bond with 316 SS sheet reinforcement	G3
Adhesive bond, Mono cured, unreinforced	G4
Adhesive bond, Mono uncured, unreinforced	G5
Adhesive bond, with nickel sheet reinforcement	G6
Adhesive bond, with 316 SS wire reinforcement	G7

*1/16 in. thick

**Polytetrafluorethylene

***Pairs of bonded 1/32-in.-thick sheets

SECTION 3

TEST PROCEDURES AND EQUIPMENT

Numerous standard methods are available for evaluating important properties and characteristics of sheet-gasket materials.¹⁻⁴ However, they generally do not measure these properties under the conditions commonly encountered in the process industries. They do not consider the influence of exposure for extended periods of time at elevated temperatures and, hence, do not measure residual tensile strength, tightness at temperature, or fire resistance.

In developing the needed procedures and test equipment, the objective was to obtain information (1) to evaluate the probable performance of a gasket material for a period of time at a specific service temperature, (2) to estimate a suitable maximum service temperature and (3) to obtain information to assess the material's resistance to fire.

ATRS OR AGED TENSILE/RELAXATION SCREENING TEST

In this test, specimens of sheet-gasket materials were subjected to a compressive stress and, while under compression, were heated to a predetermined temperature and held (i.e., aged) at temperature for a predetermined length of time. After cooling to room temperature, the tensile and relaxation properties of the material were measured.

Specimens

Six dumbbell-shaped sheet specimens, as shown in Figure 1, were used for each test, three cut with the grain and three cut across the grain. The sheet thickness was usually 1/16 in.

In the test equipment, the specimens were generally assembled in two stacks of three specimens per stack, as in Figure 2. However, when 1/16-in.-thick material was unavailable, stacks of specimens having a combined thickness as close as feasible to 3/16 in. were used.

Equipment

The test equipment centered on a fixture designed to simulate the loading conditions prevailing in a bolted joint. The fixture, shown in Figures 2, 3, 4, 5 and 6, consisted of two heavy platens, A, which sandwiched the two parallel stacks of specimens, B.

Steel sheets measuring 1/16 in. separated the specimens to ensure that only their edges were exposed to the atmosphere. Also, the sheets aided in specimen separation after aging.

A single 1 1/4-in.-diameter stud, C, ran through the fixture and was held in place by heavy nuts. A stack of Belleville springs, D, maintained the compressive load on the specimens. Collar, E, facilitated loading. The two guide pins extending through the platens, as shown in Figure 6, aided in aligning the components of the fixture during assembly. They also served to indicate relative movement of the platens, providing thermally compensated measurement of changes in gasket thickness. With the exception of the washers, all the components of the fixture were made of AISI 316 stainless steel. A hot-work die steel, H-11, was used for the Belleville springs.

The fixture was designed for an initial specimen compression of 5000 psi, which corresponds to a uniform bolt stress of about 28,000 psi for an NPS* 6-in. ANSI Class 300 flange, or a bolt stress of about 20,000 psi for an NPS 12-in. Class 300 flange. This loading is almost four times the gasket "y" factor** of the ASME Code for 1/16-in.-thick compressed asbestos sheet gasket in a typical ANSI Class 300 joint. Higher loads would have required a heavier, more cumbersome fixture.

Other items of equipment consisted of a hydraulic stud tensioner combined with a load washer, a jig with a digital micrometer and a calibrated reference stud to measure stud elongation, electric-resistance heated ovens for heating the fixtures, and a standard low-capacity tension testing machine fitted with wedge grips for pulling the gasket specimens.

*Nominal Pipe Size

**This is the pressure over the contact area of the gasket that is required to provide a sealed joint, with no internal pressure in the assembly.

Conditioning Procedures

A program of loading and thermally cycling the fixture was conducted before actual tests of gasket materials were undertaken. The purpose was to place the fixture in a condition of thermal and mechanical stability with respect to the relationship between the applied load and the resulting displacement.

The procedure was as follows: The fixture was assembled without gasket specimens, the upper nut being made finger tight. The initial length of the stud was measured with the jig as shown in Figure 7. A load of 30,000 lbs. was then applied with the hydraulic stud tensioner (Figure 8). The stud's length was again measured and the fixture was then heated, usually to about 700°F, in an oven and held at temperature for 24 hours, after which it was allowed to cool to room temperature. Thereupon, the load and strain were measured. The fixture was reloaded to 30,000 lbs. and reheated, but the holding time at temperature was increased to 72 hours. Again, the load and strain were measured after cooling to room temperature. The cycle was repeated once more with the holding time extended to 8 days. Generally, after these three cycles, the initial and final elongations of the stud would agree within 0.0001 in.

Previously, the Belleville springs had been stabilized by loading stacks of eight washers to 30,000 lbs., heating them to temperatures of 400 to 700°F for 1 to 42 days, and repeating the cycle as much as 20 times. The largest permanent set of a stack of five washers was on the order of 0.0003 in.

Stud Calibration Procedure

The apparatus used for stud calibration consisted of a tensile testing machine capable of reaching and recording a maximum load of 30,000 lbs., equipped with two threaded grips to attach the ends of the stud. Also used were two dial indicators.

Stud elongation measurements, D_m , were obtained as follows: The threaded grips were attached to the stud ends and the stud calibration length, L_c , was recorded; this is the clear stud length between the grips. The dial indicators were then installed symmetrically. Then, with no tensile load applied, the indicators were set at zero. Five load cycles up to 30,000 lbs. were applied and the dials reset to zero. The stud was again loaded to 30,000 lbs. while recording stud elongation, D_m , at every 5000-lb. increment, the D_m being the average value shown on the two indicators.

Thread elongation measurements, D_t , were made using a shorter stud, having a length just sufficient to allow the two grips to be in contact when fastened on either end of the stud. After installation in this manner in the tensile testing machine, the load was applied and stud elongation, D_t , was recorded at 5000-lb. increments to 30,000 lbs.

Stud elongation, D_s , was determined as $D_s = (D_m - D_t) \times L/L_c$ where L was the active stud length between the end nuts in the aged tensile/relaxation screening (ATRS) fixture.

Depending on the number of pairs of gasket specimens installed in the ATRS fixture, the active stud length could vary by up to 0.180 in. However, it was considered that this difference would not affect the significance of the calibration.

To obtain the stud calibration constant, K , the curve of true stud elongation, D_s , was plotted against load, the value for K being determined by linear regression analysis. A typical value of 0.00745 in. corresponded to a tension load of 25,000 lbs., yielding a value of $K = 3,360,000$ lbs./in.

Test Procedure

Graphite dry-film lubricant was sprayed on the threads of the stud and the nuts and the platens were cleaned. Then, depending on the number of specimens to be tested (e.g., 2, 4 or 6) per fixture, 0, 1 or 2 separator sheets were placed between the platens and the upper nut was turned down and made finger tight.

The depth of the guide pins was measured and referred to as measurements SA and SB.

Using the jig with the high-precision digital micrometer and the calibrated reference stud (Figure 7), the initial length of the stud was measured and referred to as L_{01} .

Before installation, the specimens were conditioned in an oven at 212°F for 1 hour as required by ASTM Standard F104 and cooled to room temperature in a desiccator containing anhydrous calcium chloride. Then they were dipped in talcum powder to minimize adhesion problems after exposure at temperature.

A maximum of six specimens (three with the grain and three across the grain) were stacked in pairs with separator sheets in between. This assembly was placed between the platens and the top nut of the stud was turned down and made finger tight.

Again, the depth of the guide pins was measured and referred to as PA and PB.

The compressive load (generally 5000 psi) was then applied to the fixture with the hydraulic bolt

tensioner. The depth of the guide pins was again measured and noted as HA and HB. The length of the stud also was measured and referred to as L1.

The fixture was placed in a preheated oven. At the end of the aging period, it was removed and allowed to cool to room temperature.

The ends of the stud and the upper platen were cleaned around the guide pin locations, after which the depth of the guide pins and the length of the stud were measured. The measurements were referred to as MA, MB and L2, respectively.

The fixture was unloaded using the hydraulic stud tensioner, starting with a low pressure and gradually increasing the pressure until the nut was released, in this way, recompression of the specimens was avoided.

The upper nut of the fixture was then retightened to finger tightness and the depth of the guide pins was measured and referred to as RA and RB. The length of the stud also was measured as L02.

Very carefully, the specimens were removed from the fixture, records being made of appearance, adherence to the separators, flexibility, cohesion, etc. Also, the specimens were photographed.

When the condition of the specimens permitted, they were tension tested, the breaking load being recorded. The average values for the three cross-grain specimens and for the three with-the-grain specimens were calculated.

Calculations

Terms

A = initial area of specimen, 2.50 in.²

W = initial width of specimen in the gage section

n = number of pairs of specimens in the stack, e.g., 1, 2 or 3

K = stud calibration factor

S = (SA + SB)/2 = reference depth measurement of fixture without specimens

P = (PA + PB)/2 = depth measurement of fixture with uncompressed specimens

H = (HA + HB)/2 = depth measurement of fixture with compressed specimens (initial)

M = (MA + MB)/2 = depth measurement of fixture after aging and cooling to room temperature (RT)

R = (RA + RB)/2 = depth measurement of fixture after unloading (final)

L01 = initial stud length, unloaded

L1 = stud length, as loaded before aging

L2 = stud length, as loaded after aging

L02 = stud length, unloaded after aging

TLW = tensile breaking load of specimens, with the grain

TLX = tensile breaking load of specimens, across the grain

Computations

lth = (P - S)/n = average specimen thickness before aging

fth = (R - S)/n = average specimen thickness after aging

SG1 = (L1 - L01) x K/2A = initial compressive stress on specimen

SG2 = (L2 - L02) x K/2A = residual compressive stress on specimen

TSW = TLW/(W x lth) = breaking stress of specimens with the grain

TSX = TLX/(W x lth) = breaking stress of specimens across the grain

100 x (P - R)/(P - S) = percent loss of thickness

100 x (P - M)/(P - S) = percent compressibility of a specimen subjected to aging at temperature

100 x (H - M)/(H - S) = percent creep relaxation after aging

100 x (R - M)/(P - M) = percent recovery after aging and unloading

100 x (SG1 - SG2)/SG1 = percent stress relaxation

100 - [100 x (SG1 - SG2)/SG1] = percent load retained

FIRS OR FIRE SIMULATION SCREENING TEST

For exposed equipment, such as valves and flanged joints, the use of an open flame to assess fire resistance is important because it simulates the heat transfer conditions expected in a fire. Open flame was ruled out for evaluating the gaskets of concern to this project because the rate of heat transfer is less relevant to gaskets within a flanged joint. Accordingly, the decision was made to use an oven to provide the required

heat. With no open flame, the difficulty of judging, calibrating and precisely reproducing the flame was avoided. Heating in an oven to the appropriate temperature would make possible the development of pertinent information with which to evaluate the probable resistance of sheet-gasket materials to fire in plant.

The test was developed in stages or phases, the principal concern being the effect of the rate of heating to temperature and the exposure time at temperature. The objective was to arrive at a test that would be properly discriminatory among classes of gasket materials as well as among individual varieties of gasket materials within a class. Thus, the test could not be too severe nor too gentle.

Summary of the Test

Specimens of gasket material were compressed in an ATRS fixture and heated rapidly and exposed to a temperature of 1200°F for 30 minutes.

Due to the severe temperature conditions, the ATRS fixture was used without Belleville springs. Only two specimens were compressed per test and the actual specimen temperature was monitored throughout the test by a thermocouple inserted between the two loaded platens.

The test was conducted in the same way as an ATRS test except for the heating phase. In order to reach a stabilized temperature of 1200°F in a very short time (typically 15 to 20 minutes), the oven was preheated to 1500°F before loading the fixture.

Specimens

The test specimens were identical to those used in the ATRS test.

Equipment

The same equipment that was used for the ATRS test also was used for this test. A temperature recorder with a range up to 1500°F was used to monitor the heating process.

Test Procedure

The fire simulation screening test (FIRS) procedure was identical to the ATRS procedure except for the following steps.

Installation of Specimens in the Fixture

Only two across-the-grain specimens were installed in the fixture. A type-K thermocouple was inserted between the two platens and connected to the temperature recorder.

Loading the Fixture

The absence of Belleville springs made the stiffness of the fixture considerably greater than that of ATRS. Therefore, a higher hydraulic pressure in the bolt tensioner was required to get a sufficiently high residual compressive stress on the specimens. Typically, a 10,000 psi hydraulic pressure gave an initial compressive stress of 1500 psi after the upper nut of the fixture had been tightened.

Heating the Fixture

The oven was preheated to 1500°F in order to reach 1200°F in the specimens within 20 minutes. The fixture was placed in the oven and the door was quickly closed. The specimen temperature was monitored and recorded during the test. When specimen temperature reached 900°F, the oven set-point was lowered to 1200°F. The specimen temperature stabilized at 1200°F. A holding time of 30 minutes at 1200°F was used. The power to the oven was then cut off, the fixture removed and allowed to cool to room temperature.

All other steps of the FIRS procedure were the same as for the ATRS procedure.

Calculations

Calculations were identical to those used in the ATRS procedure.

HOTT OR HOT OPERATIONAL TIGHTNESS TEST

The essential feature of this test was that it provided information on the probable leak tightness or sealing capability and blowout resistance of sheet-gasket materials, while being tested under conditions that realistically reproduced pressures, bolt loads and temperatures used in the process industries.

The approach was to extend the Pressure Vessel Research Committee's (PVRC's) tightness concepts from room temperature to elevated temperatures up to 800°F.

Again, like the FIRS test, this test was developed in phases. The problem was to develop a test that would be sufficiently severe to provide a meaningful evaluation of the gasket material without requiring the use of excessively high temperatures or extremely long times at temperature. In this regard, it became evident that the parameters of the test had to be adjusted to the class of material being tested using the results of the ATRS test for guidance in this matter. As finally developed, the test took the following general form.

Specimens

The sheet-gasket material was cut into ring-shaped specimens 4 7/8 in. inside diameter (ID) by 5 7/8 in. outside diameter (OD) $\pm$ 1/32 in. The specimens' thickness was taken as the mean of three measurements made at well-spaced locations.

Hot Operational Tightness (HOTT) Fixture

The fixture was designed to accept gaskets with an outside contact diameter of 6 3/16 in. Therefore, it could accept 4-in. NPS gaskets. Maximum load was 150,000 lbs. Its overall height was 57 in. from grade and its diameter (insulated) was 21 in.

The main features of the hot operational tightness (HOTT) fixture are shown in Figure 9. It used a stacked arrangement of two gaskets {1} between load (compression) platens {4} and heating platens {3}. These were held between two blind reaction flanges {2}.

The assembly was compressed through the blind reaction flanges by a set of six ASTM A193 GrB7, 1 1/4-in.-diameter threaded bars {5} fitted with hydraulic bolt tensioners {6}. The load platens were 1-in.-thick by 7 1/2-in.-diameter.

Three 1 1/2-in.-thick heating platens {3} were used, each with an independent heat control. These platens were each embedded with six 240-watt electric-resistance heating plugs. Total design heat capacity was 4.3 kW. Copper sheets between the heating and load platens encouraged heat flow to the load platens. Insulation between the heating and blind reaction flanges minimized heat loss.

The compression and heating platens were AISI Type 316 stainless steel plate and the blind reaction flanges were ASTM A516 carbon steel plate.

The threaded bars extended well below the heated zone of the test machine to protect the hydraulic tensioners from overheating. Each bar was instrumented with a fully compensated strain-gage bridge circuit. The gages were attached near the cold end of the bars to prevent temperature-related measurement problems. The load applied to each bar was precisely known through prior calibration.

The deformation of each test gasket was monitored independently. A total of six deflection transducers {7}, linear voltage differential transformers (LVDTs), were connected to the two pairs of load platens (three LVDTs for each pair) via long rods. The transducers thus extended below and were kept away from the heated zone.

The pressurizing fluid was helium. An independent high-pressure circuit was used for each test gasket. Leakage from each gasket was determined independently. This was done either directly by means of electronic mass flow meters, or indirectly from real-time calculations based on the inlet gas pressure decay.

Figures 10 and 11 show the partially and fully assembled HOTT fixture, while Figure 12 shows the fully assembled and insulated fixture.

Controls and Instrumentation

Instrumentation

Operator input and feedback were essential to understanding fixture and gasket behavior in the development stage of the gasket test program. Therefore, a mix of manual and automatic control was selected based on experience with previous room temperature gasket test programs and recent PVRC

elevated temperature work.⁵ Control schematics are diagramed in Figure 13. See Figure 9 for number references 1 through 8.

An HP*-300 computer and an HP*-3497 data acquisition control unit were the heart of the measurement control system, described below. Figure 14 is a photograph of the control panel and data acquisition unit layout.

Load Control

The air-activated hydraulic pump supplied with the hydraulic tensioners was a manually adjustable unit. To make it controllable by the computer control, solenoid valves were installed on the air supply circuit and on the hydraulic pressure relief circuit.

Figure 15 is a photograph of the air-activated hydraulic pump together with the assembled fixture.

During a control cycle, the tensioner load was increased when the air supply valve was activated and the load was decreased when the hydraulic pressure relief valve was activated. Load control details depended on whether gasket stress or gasket deflection was the control parameter, as follows:

- When gasket stress was the control parameter, the computer automatically adjusted the tensioner load to maintain gasket stress at the desired level on the basis of the following relationship:

$$\text{Gasket Stress} = \frac{\text{Tensioner Load Less Pressure Reaction}}{\text{Gasket Area}}$$

The pressure reaction was computed from the pressure and the pressurized area contained by the gasket. The gasket contact width was used to figure the gasket area.

Note that both gaskets experienced the same load, but independent gas pressures were possible (though they were normally the same). Since the pressure fed back to the stress control and might be different for each test gasket, the operator selected one of the two test gaskets as the one that was under true stress control. The other gasket followed.

- True deflection control could be achieved by using the deflection transducer signal as the control parameter and adjusting the load accordingly.

Another deflection control approach, although not as accurate, was to lock the tensioner nuts in position at some adequate load level and then release the hydraulic pressure. This method was used because it was simpler and because it mimicked gasket relaxation as it actually occurred in a bolted joint.

Temperature Control

A self-learning P-I-D** controller was used on each of the three heating platen circuits. The control thermocouples were located in the heating platens. Additional thermocouples monitored the temperature of the compression platens at several locations. Heating platen temperatures could thus be finely adjusted so that the compression platens and the test gaskets were at the required temperature.

Gas Pressure Control

A manual pressure regulator set the gas pressure at the desired level. Two circuits (see Figures 13 and 14) were connected to the pressure regulator. Each circuit was fitted with an accumulator and isolation valves so that there was no interference between the two circuits during leakage measurement (especially when one test gasket leaked more than the other).

Leakage Measurement

Leakage could be measured independently for each test gasket. Two methods of leakage measurement were used.

- An electronic mass flow meter for large leak rates (up to 8 mg/s helium) and
- The pressure decay method for the smaller leak rates (down to 0.0005 mg/s). A double block-and-bleed system provided complete isolation for this measurement.

By operating appropriate valves, one of the two methods of measurement was selected. Leak rates were computed in real time and displayed.

*Hewlett Packard

**Proportional-Integral-Derivative

Data Acquisition

A total of 31 transducers were scanned. The various measurements taken during a data acquisition scan are summarized below.

Summary of Data Acquisition Measurements

Measured Parameter	Number of Points	Type of Transducer
Load in tensioners	6	Strain gages on bars
Tensioner hydraulic pressure	1	Pressure transducer
Gasket deflection	6	LVDTs w/ integral circuit
Helium pressure	2	Pressure transducers
Heating platen temperature	3	Thermocouples
Compression platen temperature	8	Thermocouples
Temperature at one threaded bar	3	Thermocouples
Leak rate	2	Mass flow transducers

Test Procedure

Beginning of Test

One gasket specimen was installed between each of the two pairs of load platens. These platens were machined to a surface finish of 250 AARH* (± 60 AARH) in accordance with ANSI B16.5 Section 6.3.4 (1981). The surfaces were cleaned and freed of scratches and other defects.

After each gasket had been installed and the test rig assembled, the initial voltages of all transducers (no load, no gas flow, zero gas pressure, etc.) were recorded.

Each gasket was then compressed to 200 psi (± 50 psi) and the initial values of the displacement transducers were recorded. These initial displacement readings were considered to correspond to the initial gasket thickness.

The helium pressure was set at 10 to 20 psig and the purge valve was opened to remove air from the system.

Part A—Simulation of Bolt-Up Procedure at Room Temperature

This part and Part B utilized gasket stress levels and pressures that were consistent with ANSI Class 300 bolted joints.

1. Each gasket was compressed to a gasket pressure, S_g , of 1250 psi and the gasket deflections were recorded.
2. Each gasket was then compressed to $S_g = S1^{**}$ and maintained at that stress within ± 200 psi.
3. Gasket creep was recorded for 10 minutes, taking readings every 2 minutes minimum.
4. The helium pressure was then raised to 375 psi. If gross leakage (i.e., leakage exceeding 8 mg/s) was detected before full pressure was reached, Steps 5 through 8 were omitted, going directly to Step 9. This applied also to Steps 6 and 7.
5. The leak rate was allowed to stabilize and the leakage was measured as described in the section on Gasket Leakage Measurement (below).
6. The helium pressure was then raised to 750 psi and Step 5 was repeated.
7. The helium pressure was again raised to 1125 psi and Step 5 was repeated.
8. The helium pressure was then reduced to 375 psi.
9. Steps 2 through 8 were repeated for successively higher values of S_g , the gasket stress level. In raising the gasket stress from one level to the next level, gasket deflections were recorded at intermediate stress levels.
10. As soon as leakage measurements at the maximum gasket stress level were completed, the helium pressure was reduced to 750 psi. When gross leakage conditions existed at this pressure, the pressure was decreased to 375 psi. If gross leakage persisted at this pressure, the helium pressure was reduced until a level was reached at which reliable leakage readings could be obtained.

*Arithmetic Average Roughness Height

**Standardized gasket stress levels, based on nominal gasket contact area, used to perform leakage measurements in Part A and Part B: $S1 = 2500$ psi, $S2 = 5000$ psi, $S3 = 7500$ psi, $S4 = 10,000$ psi, $S5 = 12,500$ psi.

Part B—Simulation of Operating Conditions at Elevated Temperature

To prevent excessive increase in helium pressure during the heat-up process, the helium circuit incorporated safety devices such as a large volume of cold gas and an automatic pressure-regulating valve.

The heat-up procedure was performed under either constant gasket stress or under simulated gasket relaxation conditions. Simulated relaxation was used for gasket materials that were known to exhibit extensive relaxation, such as polytetrafluorethylene (PTFE). For most other materials, heat-up was performed at constant gasket stress.

Part B temperature levels were chosen to simulate long-term exposure to a specific temperature (equivalent damage) and/or to estimate the temperature above which the gasket will fail to seal adequately.

Heat-Up Under Constant Gasket Stress:

1. The gasket platens were heated to the required temperature (within $\pm 10^\circ\text{F}$). Platen temperature was monitored with two thermocouples. The test parameters (gasket stress and deflection, helium pressure and platen temperature) were recorded every 15 minutes during heat-up.
2. Time was allowed for stabilization of the temperature throughout the test rig.
3. The leak rate at the end of the heat-up period was measured.
4. Gasket relaxation was begun by locking the nuts on the fixture and disengaging the hydraulic tensioners.

Heat-Up Under Simulated Gasket Relaxation:

1. The gasket relaxation sequence was begun as in Step 4 above.
2. The gasket platens were heated to temperature (within $\pm 10^\circ\text{F}$).
3. The test parameters were recorded every 15 minutes during the first 2 hours of relaxation. The test was continued in accordance with the section on Gasket Relaxation Sequence (below).

Gasket Relaxation Sequence:

In this sequence, the gasket was allowed to relax for a predetermined period, typically over night or over a weekend, or until the gasket stress decreased to a preset minimum value. In the latter case, relaxation was stopped automatically so that the gasket stress would not fall below a minimum safe value. This precaution was necessary to prevent catastrophic gasket blowout. The steps were as follows:

1. The test parameters were recorded at regular time intervals, usually every 2 hours. When feasible, leakage was recorded in order to detect changes in gasket sealing behavior (e.g., by leaving the electronic flow meter on line).
2. At the end of the relaxation period, the gasket leakage was measured.
3. The gasket stress cycling sequence was then begun. (See immediately below.)

Gasket Stress Cycling Sequence:

During stress cycling, the helium pressure was maintained at 750 psig (or at 375 psig if leakage was excessive).

1. The gasket stress, S_g , was reduced to the next lower standardized stress level* and maintained constant. This level depended on the final gasket stress at the end of the relaxation period. (When a high initial gasket stress was used at the beginning of the cycling sequence, some intermediate stress levels were sometimes skipped, especially when the gasket sealed well.)
2. The gasket leakage was measured.
3. The above two steps were repeated for the other standardized gasket stress levels down to $S_g = S_1$.
4. At this point, an attempt was made to obtain gross leakage by gradually decreasing the gasket stress while monitoring leakage. When either the gasket exhibited gross leakage or the minimum safe gasket stress was reached (the helium pressure approaching the gasket stress level), decreasing the gasket stress was stopped and the leak rate was recorded.
The gasket stress was then increased to $S_g = S_1$ and maintained constant.

*Standardized gasket stress levels, based on nominal gasket contact area, used to perform leakage measurements in Part A and Part B: $S_1 = 2500$ psi, $S_2 = 5000$ psi, $S_3 = 7500$ psi, $S_4 = 10,000$ psi, $S_5 = 12,500$ psi.

- 6 The thermal disturbance cycle described below was then performed
- 7 Leakage at the end of the thermal disturbance cycle was measured
- 8 An attempt was again made to obtain gross leakage as in Step 4 above
- 9 Gasket stress was then increased to the next higher standardized level and maintained constant.
- 10 Leakage was measured.
- 11 Steps 8 and 10 were repeated for the other standardized gasket stress levels up to S_{max} , which could be less than the maximum gasket stress applied at the end of Part A (depending on the operating condition being simulated)

Thermal Disturbance Cycle:

- 1 Gasket stress was maintained constant at the S_1 level throughout the thermal cycle
- 2 The gasket platens were allowed to cool down to the same predetermined temperature. (The severity of the thermal disturbance depends on the extent of the cooling; it is established as a function of the operating condition being simulated. The temperature of the gasket platens should be decreased at the same rate.)
- 3 The test parameters, and, when possible, leakage, were recorded at regular time intervals during the cooling process.
- 4 When cooling was completed, one of the two platens was heated back to its initial temperature.
- 5 Test parameters and leakage were recorded at regular intervals, usually every 10 minutes.
- 6 When the thermal gradient between the two gasket platens had reached its maximum value, heating of the second platen was begun.
- 7 Recording of the parameters of the test was continued until the platen temperatures had stabilized to their initial level.

Note: The sequences or groups of procedures described under Part B constitute an operating cycle that could be repeated in order to permit estimating (a) the maximum operating temperature of the gasket material, by gradually raising the gasket temperature used in each succeeding test cycle, or (b) the operating life of the gasket material, by repeating the test cycles at the same temperature.

Termination of the Test

Following the last operating (test) cycle, the gasket stress was decreased to S_1 , stopping at each standardized level to record the test parameters and the gasket leakage.

When the measurements had been made and recorded at stress level S_1 , the helium pressure was reduced to zero while the gasket stress was held constant. The gasket stress was then reduced to zero.

The final gasket deflection was recorded for 5 minutes, after which the heating system was turned off and the test rig was allowed to cool to room temperature. The rig was disassembled and the test gaskets and test platens were photographed. The gaskets were then removed from the platens and their final thickness was measured at three locations.

Gasket Leakage Measurement

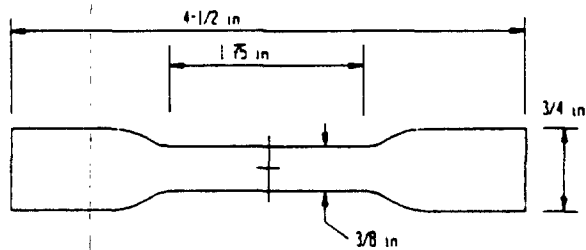
This leakage measurement procedure was applicable for helium leak rates in the range of 8 to .0001 mg/s. Large leak rates were measured using an electronic mass flow meter, while smaller leak rates were determined by the pressure decay method (Table 2). Reliable leakage measurement required a waiting period during which the leakage phenomenon stabilized.

Measurements taken by the pressure decay method were corrected to account for gasket creep and/or relaxation effects (that either reduced or increased the internal gas volume) and the temperature variation of the gas.

The respective hot and cold portion of the internal gas volume must be accurately known in order to make reliable leak measurements using the pressure decay method.

TABLE 2

Leak Rate Range	Measuring Method	Recording Interval	Total Recording Time
< 12 mg/s	Flow meter	15 s	45 s
12 to .04	Press. decay (note 3 Reference 7)	15 s	45 s (note 10)
.04 to .01	Press. decay	30 s	90 s minimum
.01 mg/s	Press. decay	150 s	900 s



Specimen Area = 2-1/2 in²

Figure 1: "Dumbbell" Sheet Gasket Coupon

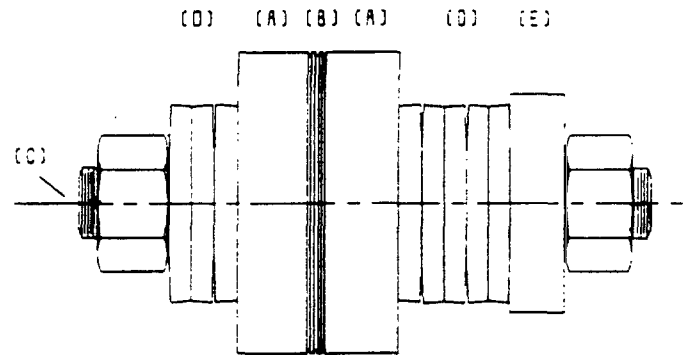


Figure 3: Aging Fixture Components: (A) Compression Platens, (B) Stacked Specimens, (C) Stud Bolt, (D) Springs, (E) Load Collar

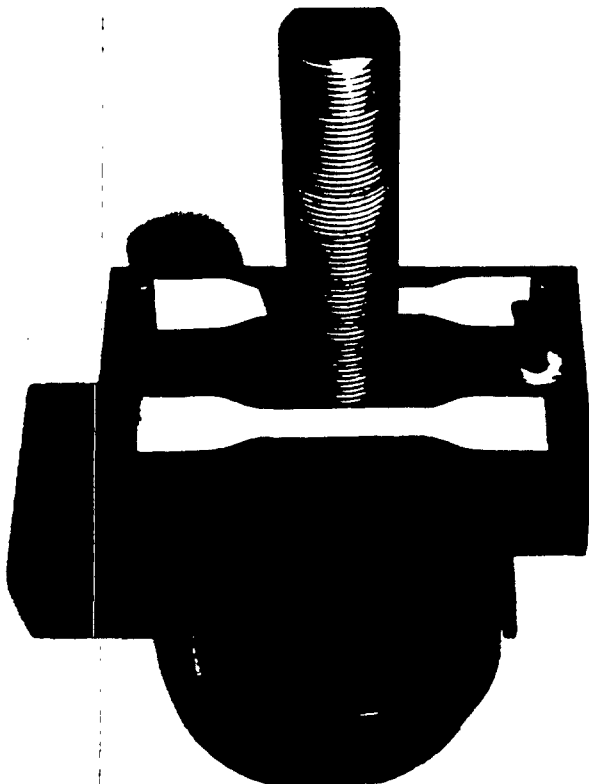


Figure 2: Side-by-Side Arrangement of Sheet-Gasket Specimens



Figure 4: Exploded Arrangement of Aging Fixture Components



Figure 5: Assembled and "Loaded" Fixture Ready for Test

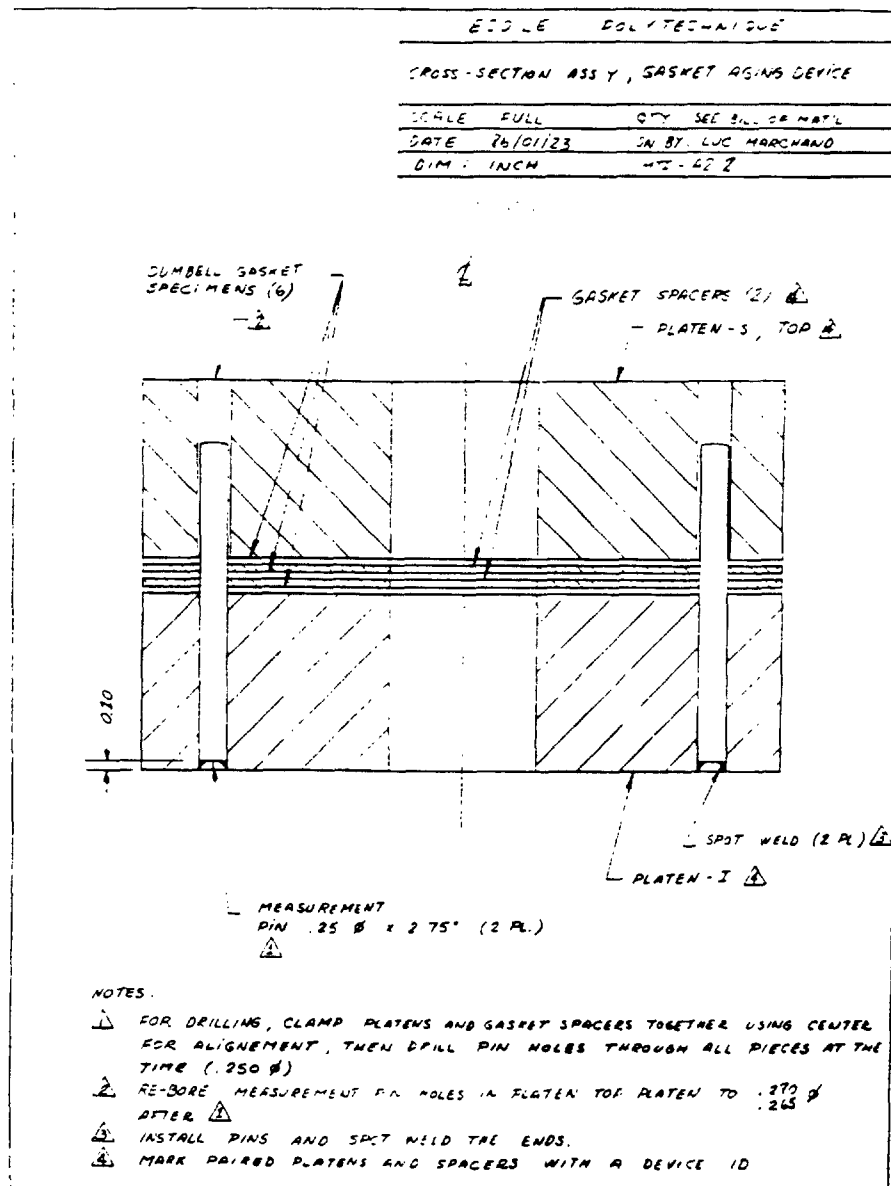


Figure 6: View of Fixture Showing Arrangement of Guide Pins

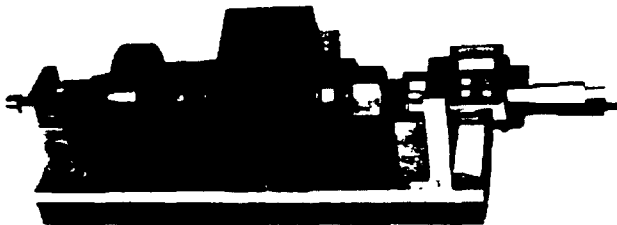


Figure 7: Stud Strain Being Measured in Special Jig

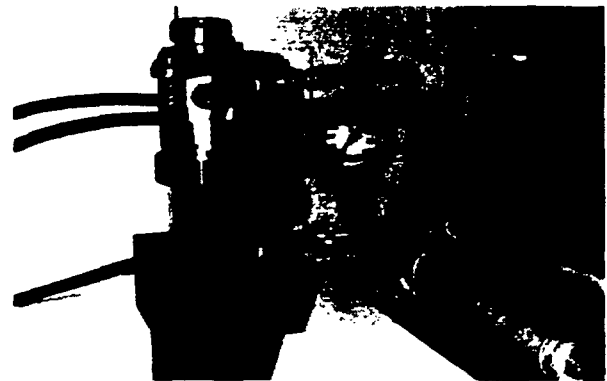


Figure 8: Stud Being Loaded with Hydraulic Stud Tensioner

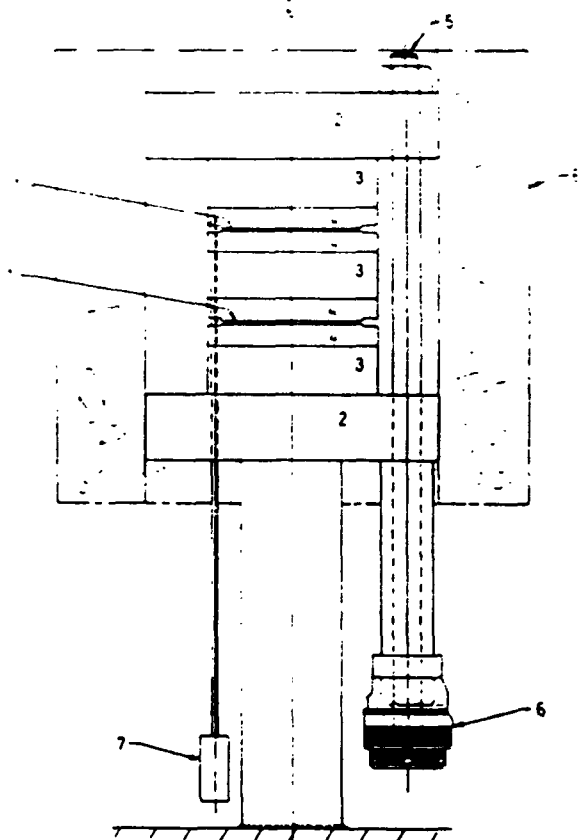


Figure 9: HOTT Fixture Diagram: Gasket (1), Reaction Flanges (2), Heating Platens (3), Load Platens (4), Threaded Bars (5), Bolt Tensioners (6), Deflection Transducers (7) (LVTDs), and (8) Insulation Casing

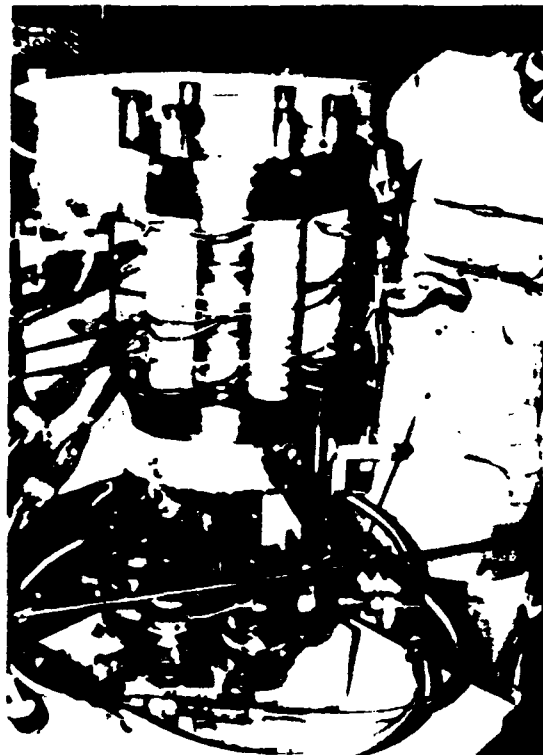


Figure 11: Assembled Fixture with Open Insulation Casing

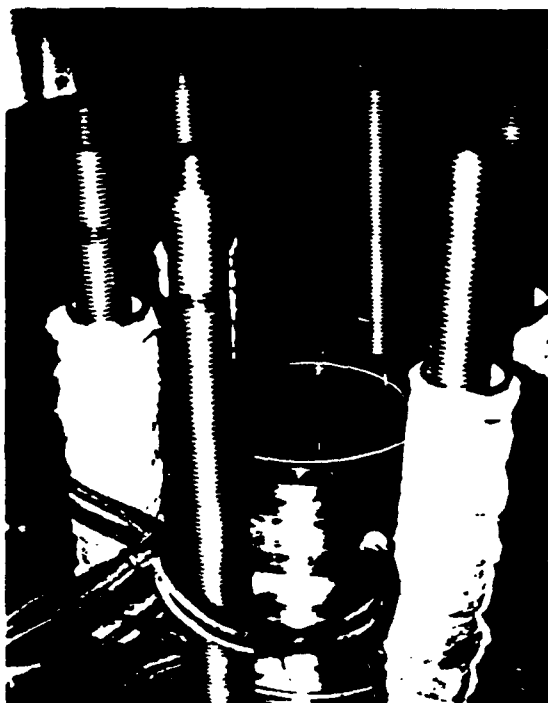


Figure 10: Partially Assembled HOTT Fixture Showing Gasket and Various Components

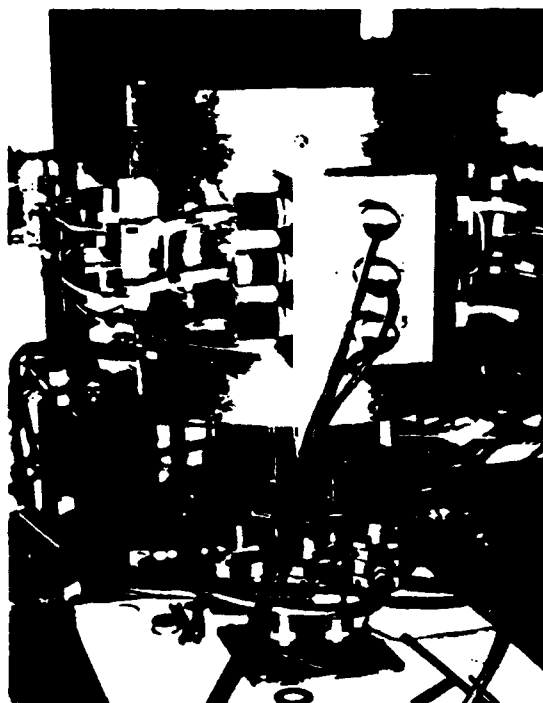


Figure 12: Fully Assembled and Insulated Fixture

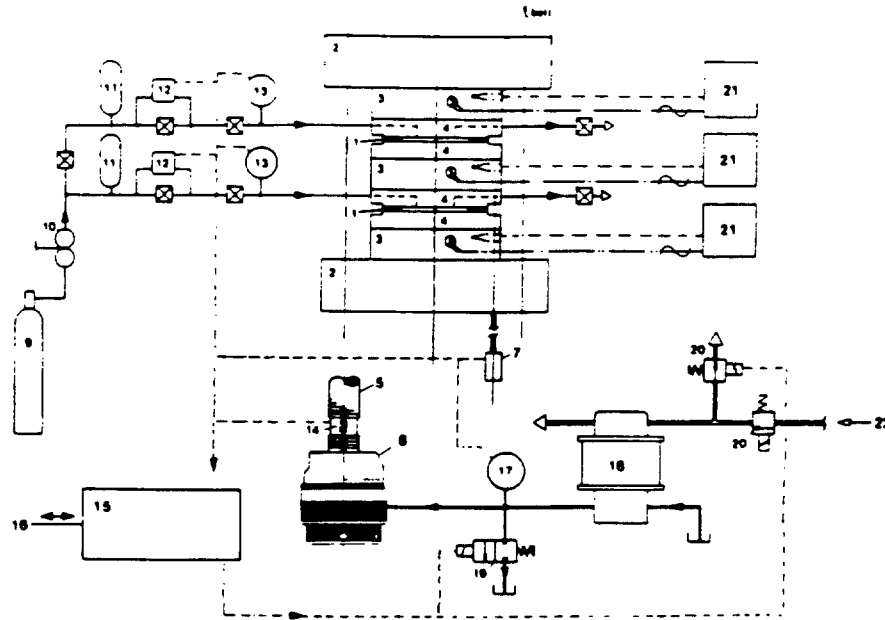


Figure 13: Control Schematics: Gas Supply {9}, Pressure Regulator {10}, Accumulators {11}, Electronic Flow Meters {12}, Pressure Transducers {13, 17}, Strain Gages {14}, Data Acquisition/Control System {15}, Microcomputer {16}, Air-Activated Pump {18}, Solenoid Valves {19, 20}, Temperature Controllers {21}, and Air Supply {22}

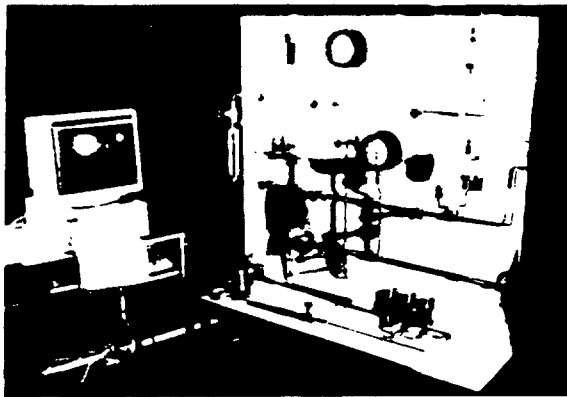


Figure 14: Photo Showing Control Panel and Data Acquisition Unit Layout

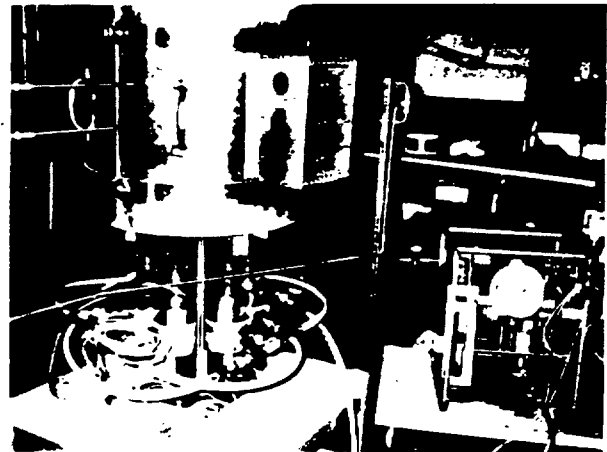


Figure 15: Air-Activated Hydraulic Pump with Assembled Fixture

SECTION 4 APPLICATION OF THE TESTS

THE AGED TENSILE RELAXATION SCREENING (ATRS) TEST

The program of tests was conducted in three phases. Phase I covered a range of temperatures and aging times in a series of tests that considered two high-quality materials. These were the elastomer-bound asbestos and aramid-reinforced sheets identified as materials A1 and N1. Six additional elastomer-bound sheet materials were studied in Phase II. Phase III covered four polytetrafluorethylene (PTFE) and six laminated graphite sheet materials. The range of exposures for each phase is summarized by the tabulation below.

Aging Times	Nominal Temperature (°F)				
	250	400	550*	700	750**
1 Day	PHASE III	III	III, IV	III	I
4 Days	PHASE III	III	III, IV	III	I
16 Days	PHASE III	III	IV	III	III
42 Days	PHASE III	I	I	I	

Note: * that the Phase III PTFE tests were at 500 F and that ** the 750 F Phase III tests provided a cross-reference for the final tightness cycle of the Phase I hot operational tightness (HOTT) test. An upper limit for many users of elastomer-bound sheet gaskets is 750 F.

Phases I and II represent over 500 individual tensile tests, while Phase III accounted for nearly 200 additional tests. The tensile and stud-bolt relaxation data trends discussed below showed consistency and useful predictability.

Phase I Materials

For materials A1 and N1, the strength first increased with moderate temperature and aging as a result of elastomeric binder hardening reactions. Higher temperatures and/or longer aging times caused deterioration. Eventually, a point was reached where only the fiber contributed tensile strength. This occurred at an exposure in the 1-to-4 day range at 700°F.

If the fiber also deteriorates with temperature, as is the case with aramid-fiber reinforcement, a brittle residue that crumbles is the eventual result of enough exposure. A comparison of the photos of Figures 16 and 17 demonstrates this. They show that while the aramid-reinforced NBR*-bound N1 material "falls apart" given exposures exceeding 4 days' aging at 700°F, the A1 material does not.

The photos also show the migration of color change and oxidation with increased exposure at temperature or time. After 16 days at 700°F, the binder of both the A1 and N1 sheets had the appearance of having turned completely to ash. A similar migration occurred from the gasket outside diameter (OD) inward on the hot operational tightness (HOTT) test.

Quantitative results for various temperatures and aging times are compared for the two Phase I materials in Figures 18 and 19. These respectively show stud-bolt relaxation and weak-direction (pulled across the fiber grain) tensile-strength trends. Room temperature base line results appear in these figures for comparison. Figure 20 shows the 550° and 700°F load-retention and tensile results plotted against time. Some further observations are as follows:

- The aramid-fiber reinforced material (N1) showed consistently lower tensile strength and stud-bolt load retention (or increased relaxation) than the asbestos-reinforced (A1) material.
- The higher fiber content of the asbestos (A1) material retarded stud-bolt relaxation. Retained load for N1 dropped 40 percentage points from about 90% at room temperature to around 50% at 750°F and 4 days' age. In contrast, material A1 dropped only 15 percentage points for the same exposure.
- The maximum across-grain tensile strength of material A1 after aging was nearly triple its initial strength (Figure 19). While this effect was much less for the N1 material, it still demonstrates a need for adequate aging in screening tests and for caution in evaluating short-term test data.

*N-Buna Rubber

Phase II ATRS Results

Phase II trends were similar to those of Phase I regarding the predictability of results with exposure. Figure 21 demonstrates again that the aramid-reinforced materials "fall apart" with exposure greater than 1 day at 700°F. Also, their across-grain tensile strength was low to nil on exposures at 550°F for 16 days or 700°F for 1 day. In contrast, Figure 22 shows that materials A6 (asbestos) and N7 (glass) fared better. The quantitative results shown in Figure 23 confirm these visual observations. Stud-bolt-load retention results for Phase I and II materials are compared for several exposures in Figure 23 (top). The aramid-reinforced materials (N1, N2, N3, N4 and N6) relaxed significantly with increasingly severe exposures. Material N2, an SBR*-bound aramid-reinforced sheet, was especially poor, while material N1 relaxed the least. On the other hand, materials N7, A1 and A6 showed little additional relaxation with increased exposure. The progressive deterioration of tensile strength with exposure is clear from Figure 23 (bottom). Only glass- and asbestos-reinforced materials (A1, A6, N7) had significant tensile strength beyond a day at 700°F (371°C).

Phase III Materials

The two different generic classes of nominal 1/16-in.-thick sheet material studied in Phase III were exposed to accordingly different test conditions. Six laminated flexible-graphite sheet products identified as G2 through G7 were tested at two exposures: 4 days at 550°F and 16 days at 750°F.

Four PTFE sheet materials identified as TL (glass), TG, TS and TV (virgin) were tested at four exposures: 1 and 4 days at 250°F and 1 and 4 days at 500°F. Aged tensile/relaxation screening (ATRS) test results for these materials are discussed separately below.

Laminated Flexible Graphite

The charts of Figure 24 compare stud-bolt relaxation and tensile-strength characteristics of laminated flexible graphite studied in Phase III. These charts compare to Figures 23 and 25, respectively, for the elastomer-bound and PTFE materials. The following observations are worth noting for laminated flexible graphite:

- The graphite product class, as expected, relaxed much less than the PTFE product class.
- Moreover, exposure-for-exposure, graphite stud-load relaxation was somewhat better than asbestos-reinforced elastomer-bound materials.

Figures 26 and 27 show the post-test condition of the six graphite materials. A tendency of the sheet, consisting of two layers of flexible graphite, to delaminate was expected. However, the tendency of these layers to stick to platen surfaces was unexpected. Sticking accounts for the rough appearance of some of the specimens. Fire simulation screening (FIRS) and HOTT graphite specimens experienced similar sticking.

A tabulation (below) comments on the degree to which adhesion and delamination occurred for each test. A notation of delamination means the physical separation of the previously bonded two 1/32-in.-thick expanded graphite layers. The degree of adhesion (sticking to the platen face) is noted on a subjective six-level scale that ranges from

No Adhesion → Very Slight → Slight → Adhesion → Strong Adhesion → Very Strong.

Tabulation of ATRS Test Comments—Phase III Graphite Material

Code	Aging	Temp°F	Comments
G2	4 Days	550	Strong Adhesion
	16 Days	750	Adhesion, Delaminated
G3	4 Days	550	Strong Adhesion
	16 Days	750	Adhesion
G4	4 Days	550	Strong Adhesion
	16 Days	750	Strong Adhesion
G5	4 Days	550	Slight Adhesion
	16 Days	750	Adhesion
G6	4 Days	550	Adhesion
	16 Days	750	Strong Adhesion, Delaminated
G7	4 Days	550	Strong Adhesion
	16 Days	750	Strong Adhesion, Delaminated

*S-Buna Rubber

It is to be noted from the tabulation that the tendency to delaminate is exposure dependent. The delamination occurred because the adhesive bond deteriorated with exposure over time. The cause of the adhesion sticking is less clear. It may have been related to the migration of volatile adhesive components although this does not explain the sticking of the mechanically bonded (ranged) G3 material.

Since sticking may be detrimental to a good seal under certain conditions of cyclic radial shear, an investigation of adhesion over a broader range of exposures seems a worthwhile future effort. Is there a lower temperature limit or a high exposure point where adhesion does not occur? Are impurities, surface finish or clamping load a factor? The effect of delamination and adhesion on tightness is discussed later with regard to tightness.

Figure 28 shows the effect of exposure on stud-load retained and tensile strength for the laminated graphite sheet materials. Some observations:

- Exposure reduces the stud-load retained. The reduction of about 15% between the two test exposures was roughly the same for all product types.
- Exposure affects post-test tensile strength little. Some increased; some did not. Except for monolithic sheets, the tensile strength is bolstered by the metallic reinforcement of the laminate.
- The monolithic sheets, G4 and G5, demonstrated that the tensile strength of unreinforced expanded graphite is between 500 and 1000 psi. This is close to the residual "weak" direction strength of elastomer-bound asbestos-reinforced sheet.

PTFE Results

The charts in Figure 25 compare the stud-bolt relaxation and tensile-strength characteristics of the PTFE materials studied in Phase III. These compare to Figures 23 and 24, respectively, for the elastomer-bound and graphite materials. The following observations are worth noting:

- The load retention quality of the PTFE group of products was very sensitive to temperature, but not to time of exposure. There was considerable variation of load retention among the four PTFE materials.
- For 500°F exposures, load retention of the PTFE products, as a class, was poorer than the aramid-reinforced elastomer-bound materials (see N1 to N6 in Figure 23).
- The post-test residual tensile strength of both the graphite and the PTFE was better than that of the aramid-reinforced products at similar exposures.

PTFE flow under compressive load was mainly temperature dependent, although time at temperature (aging) had a secondary effect of 3 to 5% more spread. In a bolted joint, spreading has the effect of increasing the gasket contact area, thereby reducing the contact gasket stress. It turns out, nevertheless, that PTFE gaskets remain tight despite spreading.

Tabulation of ATRS Test Comments—Phase III—PTFE

Material Code	Aging Time	Temp. (°F)	Comments on Deformation and Adhesion
TG barium	1 Day	75	Slight def (w = 395). No adhesion
TL glass	4 Days	75	Slight def (w = 425). No adhesion
TS silica	4 Days	75	Slight def (w = 395). Very slight adhesion
TV virgin	4 Days	75	Slight def (w = 420). No adhesion
TG barium	1 Day	250	Def (w = 440). Very slight adhesion
TG sulfate	4 Days	250	Def (w = 445). Very slight adhesion
TG sulfate	1 Day	500	Def (w = 485). Very slight adhesion
TG sulfate	4 Days	500	Def (w = 495). Slight adhesion
TL glass	1 Day	250	Def (w = 465). Very slight adhesion
TL glass	1 Day	500	Large def (w = 540). Very slight adhesion
TL glass	4 Days	250	Def (w = 495). No adhesion
TL glass	4 Days	500	Large def (w = 545). No adhesion
TS silica	4 Days	250	Slight def (w = 410). Slight adhesion
TS silica	1 Day	250	Slight def (w = 405). Very slight adhesion
TS silica	1 Day	500	Def (w = 440). Very strong adhesion
TS silica	4 Days	500	Def (w = 460). Adhesion
TV virgin	1 Day	250	Def (w = 455). No adhesion
TV virgin	4 Days	250	Def (w = 480). No adhesion
TV virgin	1 Day	500	Large def (w = 535). No adhesion
TV virgin	4 Days	500	Large def (w = 550). No adhesion

Figures 29 and 30 show the post-test condition, and spread, of the four PTFE sheet materials. Note that the elongated specimens are the result of the tensile test. Original specimen width was 0.375 in. Specimen spreading was often dramatic. The above tabulation of comments for PTFE reports post-test specimen width. Some additional observations on spreading:

- Materials TL (glass) and TV (virgin) spread the most, up to 46% at 500°F.
- Material TS (silica) spread the least, up to 23% at 500°F.
- Room temperature spread was 5 to 10% more than the initial width.

PTFE is noted for its nonstick properties. Nevertheless, all except the virgin (TV) material (see above tabulation) showed some post-exposure tendency to adhere (stick) to the ATRS plates. Mostly, there was slight or very slight adhesion, but the TS (silica) material adhered very strongly after 1 day at 500°F. Figure 31 shows the effect of the various ATRS test exposures on stud-load retained and tensile strength for the PTFE sheet materials. Note that unlike the graphite and elastomer-bound materials, the PTFE post-test tensile strength was probably much greater than the hot tensile strength. The post-test strength is based on the pull required to cause a 200% elongation. Some observations:

- Temperature governed the percent stud-load retained. Time at temperature had little effect.
- Post-test tensile strength was only slightly affected by exposure. The virgin (TV) and the glass-filled (TL) sheets were about 50% stronger than the TS (silica) and TG (barium sulfate) sheets.
- Materials TV and TL (glass) also relaxed the most, and
- Material TS (silica) relaxed much less than the others at all exposures.

General Comments

The rough predictions that are possible for the Phase I and II elastomer-bound materials imply that equivalent damage is inflicted by equivalent exposure. This behavior provides a basis for possible extrapolation of performance to longer times.

The predictive trends (for elastomer-bound materials) are clear in Figure 20, which shows load retention and tensile strength plotted against time for 550 and 700°F. For the N1 material, as an example, the 1-day strength at 700°F roughly predicts the 550°F strength at 16 days.

Only the fibers contribute to strength and creep resistance beyond an exposure of, say, 4 days at 700°F or 1 day at 750°F. For material A1 (asbestos) there was a leveling of strength to 700 to 1500 psi, while the strength of N1 (aramid) leveled to zero. This suggests a 1000-psi strength criterion as a measure of "as good as asbestos" quality.

Similarly, a second comparative measure of quality for prospective materials is suggested by the data for good quality asbestos-reinforced elastomer sheet. This is an upper limit of 75% stud-bolt-load retention as measured by the ATRS method.

A 1000-psi post-test tensile strength criterion seems reasonable for the Phase III graphite and PTFE materials as well. A stud-bolt-load-retention criterion of 75% also seems appropriate for laminated flexible-graphite materials.

However, all PTFE products would fail a stud-bolt-load-retention criterion of 75% for temperatures exceeding about 200°F. Since PTFE gasket products are exceptional sealers, as mentioned before, and are commonly and successfully used without containment at this and higher temperatures, they need special consideration.

The trends also suggest that an effective evaluation test procedure should account for time of exposure to temperature and inflict damage consistent with exposure to proposed long-term service conditions. The exposure should be sufficient to destroy the binder and fiber of reinforced elastomer-bound materials to the same extent that service would. Thus, if consistent with actual service, the effective test would ensure that only residual fiber and ash remain in the fixture after exposure. These residual materials would provide a measure of the relaxation and residual strength properties that represent exposure to service conditions.

THE FIRS TEST

As mentioned earlier, the FIRS test was developed in three phases. The third phase, designated FIRS Final, featured rapid heating of the specimens (15 to 20 minutes) to the test temperature of 1200°F and holding at temperature for 30 minutes, followed by removal from the oven and natural cooling to room temperature. The following comments on the performance of the gasket materials refer to the FIRS Final test procedure.

Graphite Sheet

As noted in the insulation pretest comments below, the flexible graphite sheets tended to stick to the platens and delaminate. This also occurred in the ATRS tests. The sticking tendency was consistently less, however, and while two of the same materials, G2 and G6, delaminated, material G7 did not.

An interesting aspect of these tests was that the graphite survived with no evidence of oxidation although it was exposed to a temperature well above the generally accepted oxidizing threshold temperature of 350°F. The brevity of the exposure is the likely reason for this.

Figures 32 and 33 show the post-test appearance of the graphite-laminated specimens. The delamination of G2 and G6 is evident. Figure 34 presents percent stud-bolt-load-retained and post-test tensile strength results. Load-retained results are less reliable than, and not directly comparable to, those of ATRS. This is because there was one instead of three layers of specimens, and because the fixture was rigid and relaxed quickly. Nevertheless, the load-retained findings are similar to those of the ATRS tests.

The tensile strength trends were also close to those of the Phase III ATRS tests. The monolithic sheets, G4 and G5, were the weakest, as expected. Nevertheless, the residual strength of these unreinforced sheets averaged about 800 psi.

Comments on FIRS Test Specimens

Code	Comments	Code	Comments
G2	Slight adhesion; delaminated	TG	Compressed powder residue
G3	Slight adhesion	TL	A little powder residue
G4	Slight adhesion	TS	Powder residue
G5	Adhesion	TV	Total sublimation
G6	Slight adhesion; delaminated		
G7	Slight adhesion		

PTFE Sheets

Figure 35 shows what remained of the four PTFE sheet materials tested. Not unexpectedly, the test destroyed the PTFE sheets, leaving only residue. There was pretest speculation that some PTFE might remain because of the shortness of the test. This did not occur. The TG sheet left the most residue and was the only sheet with a less-than-complete thickness loss (70%).

Comments on the Final FIRS Test

The final FIRS test procedure provided a clear indication of the behavior of sheet-gasket products under simulated fire conditions in a manner that was reasonably consistent with the exposure time and temperature of API Standard 607. An uncertainty is the temperature that gaskets in real joints experience in real fires. Gaskets in heavy flanges will experience lower temperatures compared to gaskets in light flanges. The FIRS test is convenient, portable and low in cost.

THE HOTT TEST

Phases I and II

In brief, the HOTT test involved initial gasket compression (bolt-up) at room temperature, heat-up, and then cycles of stress relaxation and reloading. For Phase I, after heat-up, there were three 96-hour load cycles at 550°F, followed by a final cycle at 750°F. The 550°F temperature of the middle cycles measured tightness consistent with a longer service time at some lower temperature. These temperatures were derived from the ATRS test data trends and exposure estimates. At least for some elastomer-bound gaskets, the tightness thus measured would represent typical leakage for a deteriorated, but acceptable, condition.

The gasket materials used in Phase I were A1 and N1. Comments on their performance in Phase I are as follows:

- Material A1 had tensile strength comparable to its counterpart ATRS samples with similar exposure.
- The N1 specimens could not be handled after test, let alone be tested for tensile strength.
- Material A1 experienced significant additional creep and increased leakage after the temperature was increased to 750°F.

- There was little relaxation of either material at low stress levels
- Leakage deterioration at 550°F after 2 weeks was less than anticipated for the same period based on ATRS results. This is apparently because the interior diameter (iD) of the gasket was exposed to a leakage flow of nonoxidizing fluid (helium)

The 2-week exposures at 550°F for Phase I revealed that insufficient deterioration (time at temperature) occurred to evaluate a material like N1. Several more weeks of exposure at 550°F would be needed to cause sufficient deterioration for failure.

Moreover, ATRS test trends for the elastomer-bound materials suggested that a long exposure would be needed for a test that is near the probable temperature limit of a material. HOTT tests for materials limited to 500°F could take months to produce sufficient damage. Such lengthy and costly hot tightness tests would not be practical or necessary.

On the other hand, the Phase I tests also demonstrated that things happened quickly for both materials at 750°F; one failed while the other did not. These results showed that shorter tests at higher temperatures would be practical. It was anticipated that performance at shorter times and higher temperatures could be correlated with behavior at longer exposures at lower temperatures.

Accordingly, in Phase II, two temperature sequences were studied. One was four 100-hour load cycles at 750°F. In the other, the temperature was increased in four steps, i.e., 650, 700, 750 and 800°F, the holding time at each temperature being 100 hours. It was thought the latter sequence might easily pinpoint a service temperature limit because it was extended to 800°F and because exposure was increased for each cycle. Since it turned out that the four-cycle 750°F sequence was the more damaging of the two, a different method was used to assess service temperature limits.

The gasket materials used in Phase II were as follows:

Material	Temperature Sequence
N3 (two tests)	650 to 800°F and 4 x 750°F
N7 (two tests)	650 to 800°F and 4 x 750°F
A6 (one test)	650 to 800°F
N6 (one test)	650 to 800°F

Materials N3 and N7 (glass fiber) were selected because they were different from each other according to the ATRS results. Materials A6 (asbestos) and N6 (aramid) were a second matched pair for comparison with A1 and N1. Because the screening tests indicated that materials N1, N2, N3, N4 and N6 were similar, all of these did not require HOTT testing. As a poorer performer in the screening tests, the selection of material N3 was balanced against N1 and N6.

Examination of the gasket materials after test showed that A6 was in relatively good condition after exposures at 650 to 800°F, as shown in Figure 36. The lower photo shows that the post-test condition of material N6 was fairly good as well. More deterioration was expected for an 800°F exposure. Although there was some flaking, the post-test condition of N6 was much better than that of N1 in Phase I. Material N3 suffered greater damage when exposed in the 4 x 750°F sequence than in the 650 to 800°F sequence, as shown in Figure 37. The same was the case for material N7, but the degree of damage was considerably less, as shown in Figure 38.

The results obtained and the observations made in Phases I and II showed that the test conditions need to be such that enough damage is produced in the test materials, particularly in elastomer-bound materials, to permit adequate evaluation of probable performance in service. Also, it is highly advisable to precede the HOTT test with either the ATRS or FIRS test in order to estimate the amount of exposure in the HOTT test necessary for the generic gasket class being considered. The details of the HOTT test procedure should be flexible enough to permit adjustments to differences among generic classes of gasket materials. Finally, load cycles at the beginning of a test run did not seem as useful as those toward the end.

Phase III

Accordingly, the four-cycle 750°F Phase II procedure was generally modified for Phase III to incorporate two load cycles near the end of the test. Moreover, the two quite different generic classes of nominal 1/16-in.-thick sheet material studied in the Phase III program were tested by different test procedures as determined from ATRS results. These materials and conditions were as follows.

Flexible-Graphite Sheets

Four laminated flexible-graphite sheet products identified as G2 (bonded Tp 316 reinf), G3 (tanged Tp 316 reinf), G4 (mono cured) and G5 (mono uncured) were tested according to a high-exposure sequence 5 days at 800°F, with two load cycles near the end.

PTFE Sheets

Three PTFE sheet materials identified as TV (virgin), TS (silica) and TL (glass) were tested according to a sequence of increasing temperatures as suggested by ATRS relaxation results: 350 to 600°F in 50°F increments. The TV (virgin) material required an additional (development) HOTT test to confirm a revised procedure specific to PTFE-type materials. The maximum 600°F temperature was selected out of a desire to measure tightness performance at this extreme.

NOTE: No one recommends 600°F PTFE gasket operation.

The Phase III HOTT sequences compared with Phase I and II as follows:

Procedure Cycles	Load Cycles	Temperature Sequence	Time (Hrs.)
Phase I	4	500, 750°F	500
Phase II: 6-800	4	650 to 800°F	100
Phase II: 4 x 750	4	750°F	100
Phase III: Graphite	2	800°F	120
Phase III: PTFE	6	350 to 600°F	150 to 200
Phase III: PTFE*	6	350 to 600°F	290

*Development test.

Final Improvements in the HOTT Test

For both types of gasket material, the procedure included aging for 100 hours prior to the load and thermal cycles, and use of two or three load-blowout-thermal cycles applied near the end of the test. For materials that relax quickly, such as PTFE types, a special relaxation sequence, which included locking the fixture prior to raising the temperature and a multiple increasing temperature sequence, which sought a service temperature limit, was used.

As a development effort, the first HOTT test on the TV (virgin) PTFE material revealed the need for a special relaxation sequence applicable to quickly relaxing materials. In the revised procedure, the fixture was first locked, and the temperature was then raised to the next highest (or initial) level. Any material deformation then immediately resulted in load relaxation, which mimicked a bolted joint.

Previously, as temperature was increased, the HOTT fixture remained under load control briefly until the temperature stabilized. The machine was then "locked," permitting relaxation. Any deformation that occurred before locking the system did not contribute to relaxation. For the elastomer-bound and graphite-gasket materials, the lost relaxation was small. This was not the case with PTFE where significant deformation occurred immediately following a temperature increase.

The test time plots of Sg and Dg in Figure 39 illustrate the lack of relaxation and the high creep deformation that occurred in the development trial test of material TV as compared to the revised procedure. Note that the deformation scale of Figure 39 is 100 x in.

As with Phase II, Part B of the procedure (heat-up and operation) began after stress level S3 had been reached and was continued for the balance of the HOTT test without interruption.

Phase III Results

Figure 40 illustrates the revised procedures by showing the test step traces of the temperature sequence for materials G3 (tanged Tp 316 reinf) and TL (glass). It should be noted that future HOTT tests of PTFE material probably would not be taken to 600°F.

An overview of the performance of the gasket materials used in Phase III is as follows:

- The graphite material class, as indicated by the ATRS tests, relaxed less than the elastomer-bound materials of Phases I and II, and much less than the PTFE class.
- The sheet-graphite materials, as a class, were much tighter than elastomer-bound materials (A1, N1,

etc. Deterioration of tightness in the HOTT test was small or nonexistent. Adhesion and delamination were consistent with ATRS results.

- PTFE materials were the tightest. Once seated, there were no important leaks up to 600°F when blowouts occurred for the TV (virgin) sheet. No PTFE sheet could be made to blow out otherwise even though the gasket stress was relaxed to almost nothing to $S_g = 0$. Deterioration of tightness with aging or relaxation did not occur.
- PTFE load retention quality, and the related spreading, were poor. These materials were very sensitive to temperature, but not to time of exposure. Relaxation was complete within a few hours of a temperature increase. There was no leakage in spite of the extreme relaxation.
- The integrity and hot tensile strength of the graphite (to 800°F) and the PTFE (to 500°F) sheets prevented blowout at low gasket stresses.

Figures 41 and 42 show the post-test condition of the graphite specimens. The tendency of the sheet, consisting of two layers of expanded graphite, to adhere and delaminate was consistent with the ATRS results for these materials. The effects of adhesion and delamination can be seen in the photos of G2, G4 and G5. More specifically:

- Specimens 1 and 2 of G2 (bonded Tp 316 reinf) separated into two and three layers, respectively. Adhesion to the load platens was strong.
- Specimens 3 and 4 of G2 (bonded Tp 316 reinf) separated into two and three layers, respectively. Adhesion to the load platens was strong.
- Specimens 5 and 6 of G2 (bonded Tp 316 reinf) separated at the midplane because adhesion to the platen faces was strong.

- Specimens of G5 (mono uncured) were almost intact. Slight adhesion.

Great similarity was seen in the stress versus tightness plots for the graphite-sheet materials. They all exhibited the favorable characteristics of:

- Good tightness at low load.
- Steep slope (tolerates unloading well).
- Little change between cycles.

Only G4 (mono cured) showed a tightness reduction between cycles. This was related to the adhesion and separation observed and to the thermal disturbance action. The performance of sheet G4 is illustrated in Figure 43.

Although quite small, and of little concern compared to the deterioration of a material like N3, the tightness reduction of G4 represented a doubled leak rate from 0.008 to 0.015 mg/s (seen as a left shift in the stress-tightness plot of Figure 43). It is not clear whether or not further tightness deterioration would occur with a larger number of cycles. A special test involving, say, 10 thermal cycles after appropriate aging would be a worthwhile future effort to explore the practical long-term significance of this.

The total deformation of all graphite sheets was within the range of 0.024 ± 0.002 in. Initial compression and creep that occurred in the first few hours accounted for 95% of this. Deformation during the balance of the test was on the order of 0.001 in. The mono sheets (G4 and G5) relaxed and crept slightly more than the others. For practical purposes, consistent with ATRS, these results indicate that relaxation is not a concern for flexible-graphite laminates (for an exposure of five days at 800°F).

For practical purposes, none of the PTFE sheets leaked. Leakage was much less than for any of the elastomer-bound or graphite sheets. Often, leakage was less than the instrument's sensitivity of 1×10^{-4} mg/s, at temperatures up to 600°F (for 6-in.-OD gaskets at 750 psi). At 600°F, blowouts occurred for the TV (virgin) sheet. The TL (glass) and TS (silica) sheets did not blow out.

On initial heat-up to 350°F, gasket stress on materials TV and TL (glass) relaxed rapidly to a low value on the order of 1000 to 1500 psi., which was close to the contained pressure (750 psi) based on the original contact area of the gasket. Since the actual contact area increased substantially because of spreading, the actual gasket stress must have been the same as, or even less than, that of the contained pressure. In ASME code terms, that means an "m" factor* of 1.0 or less. Figure 44 shows the post-HOTT condition and spread of the PTFE sheet materials TV (virgin), TL (glass) and TS (silica). Both ruptured TV specimens are shown.

Spreading at 600°F was dramatic, as expected from ATRS results, especially for the TV (virgin) sheet. The original 1/2-in. width of PTFE HOTT specimens increased 170, 140 and 80% respectively for TV, TL (glass) and TS (silica). Also consistent with ATRS observations was the relaxation of the initial stress from the point of temperature increase to 350°F. Respectively for TV, TL (glass) and TS (silica), this relaxation, expressed as a percentage of the initial (7500 psi) stress retained, was 18, 18 and 34%. These compare well with the 500°F percentages of Figure 31.

*The "m" factor relates to the additional preload capability needed in flange bolts to overcome internal pressure and maintain sealing pressure on the gasket after internal pressure is applied to a joint.

It was concluded that a HOTT test is not meaningful for the generic type of PTFE products included in this investigation. However, HOTT tests of PTFE materials should be considered for the following:

- Future types of PTFE sheet where ATRS results suggest that substantial physical deterioration (i.e. brittleness, flaking, etc.) occurs below the manufacturer's recommended temperature limit.
- An application where the owner/designer desires specific creep/relaxation, or spread, or leak-rate data for a product.

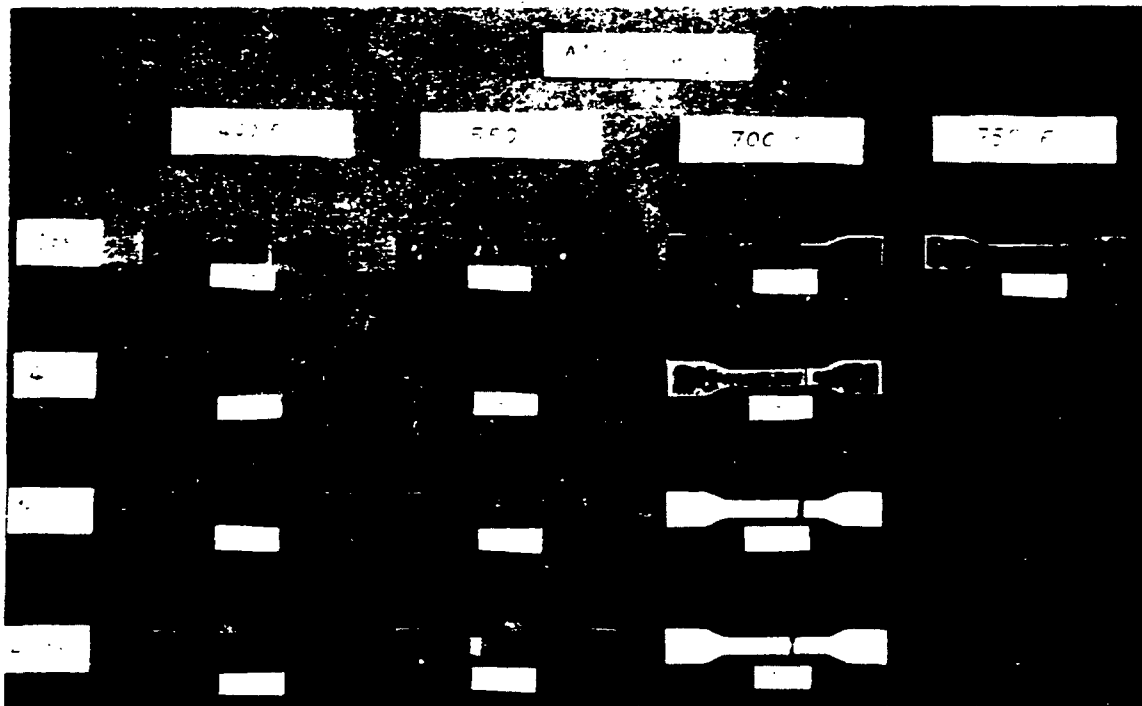


Figure 16: Typical Post-Test Specimens—Al Material—Phase I

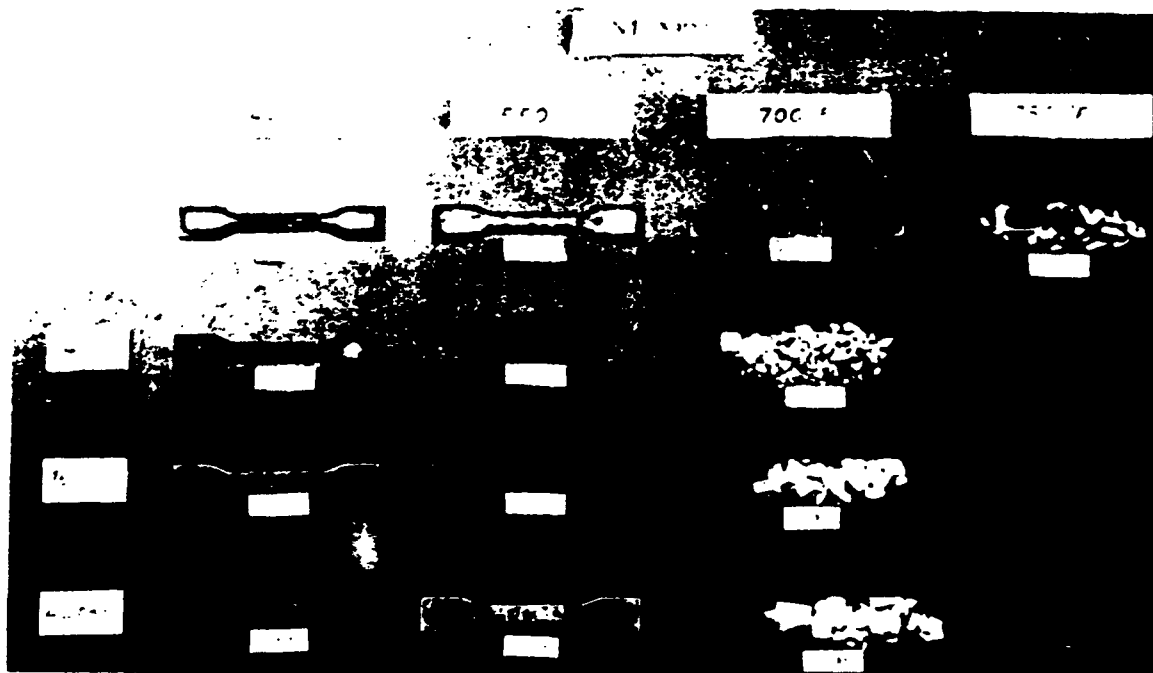
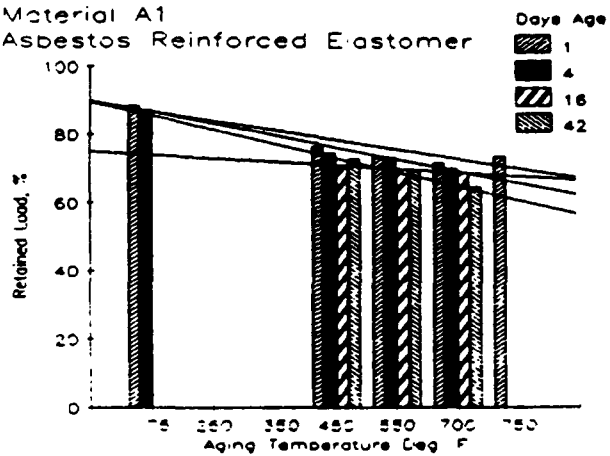


Figure 17: Typical Post-Test Specimens—N1 Material—Phase I

RETAINED STUD LOAD
Material A1
Asbestos Reinforced Elastomer



Material N1
Aramid Reinforced Elastomer

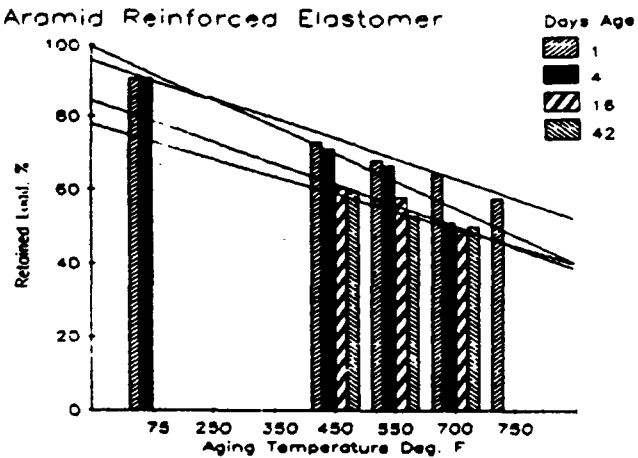


Figure 18: Percent Stud-Load Retained—Phase I Materials

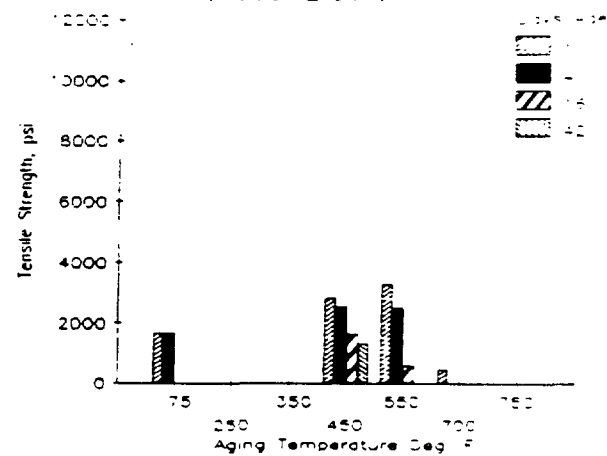
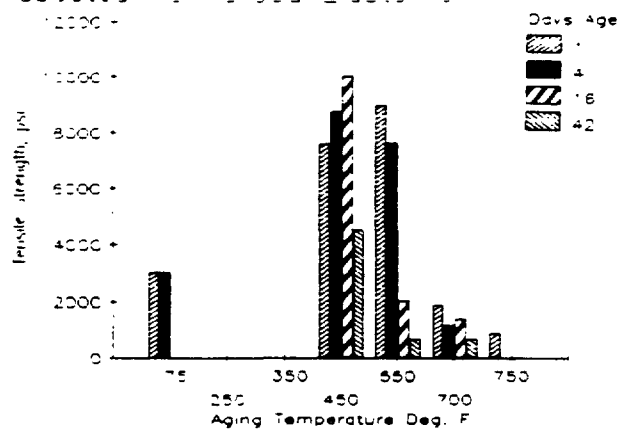
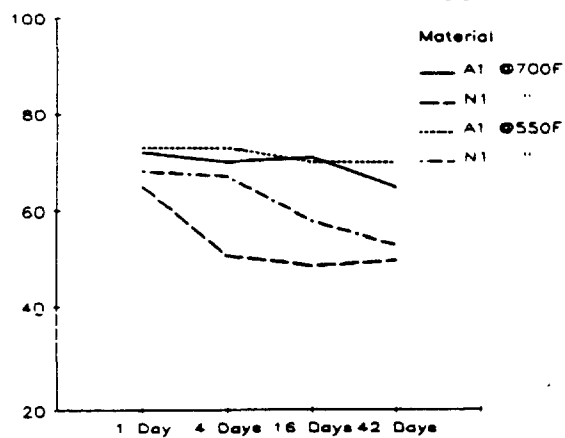


Figure 19: Residual Tensile Strength—Phase I Materials

% STUD LOAD RETAINED

Effect of Time — 550 F and 700 F



ACROSS GRAIN TENSILE STRENGTH

Effect of Time — 550 F and 700 F

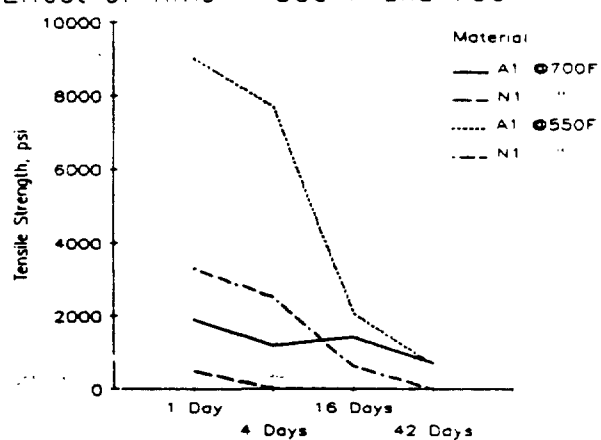


Figure 20: Time Trends—Load-Retained and Tensile Strength—Phase I

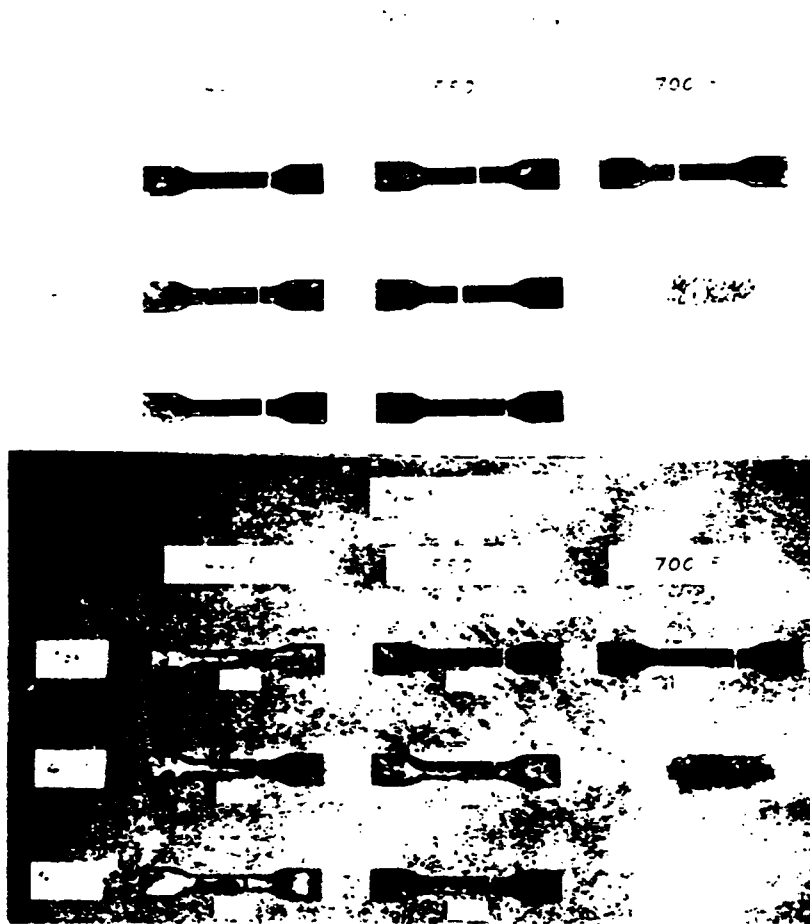
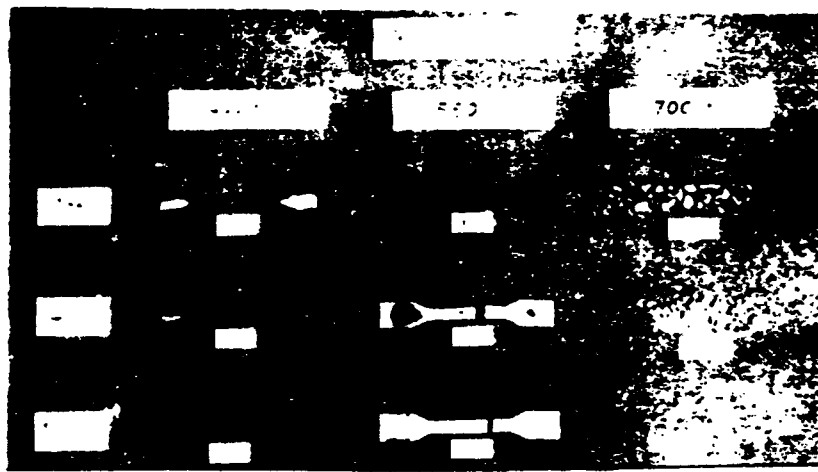


Figure 21: Typical Post-Test Specimens—Aramid-Reinforced Materials; From Top, N2, N3 and N4

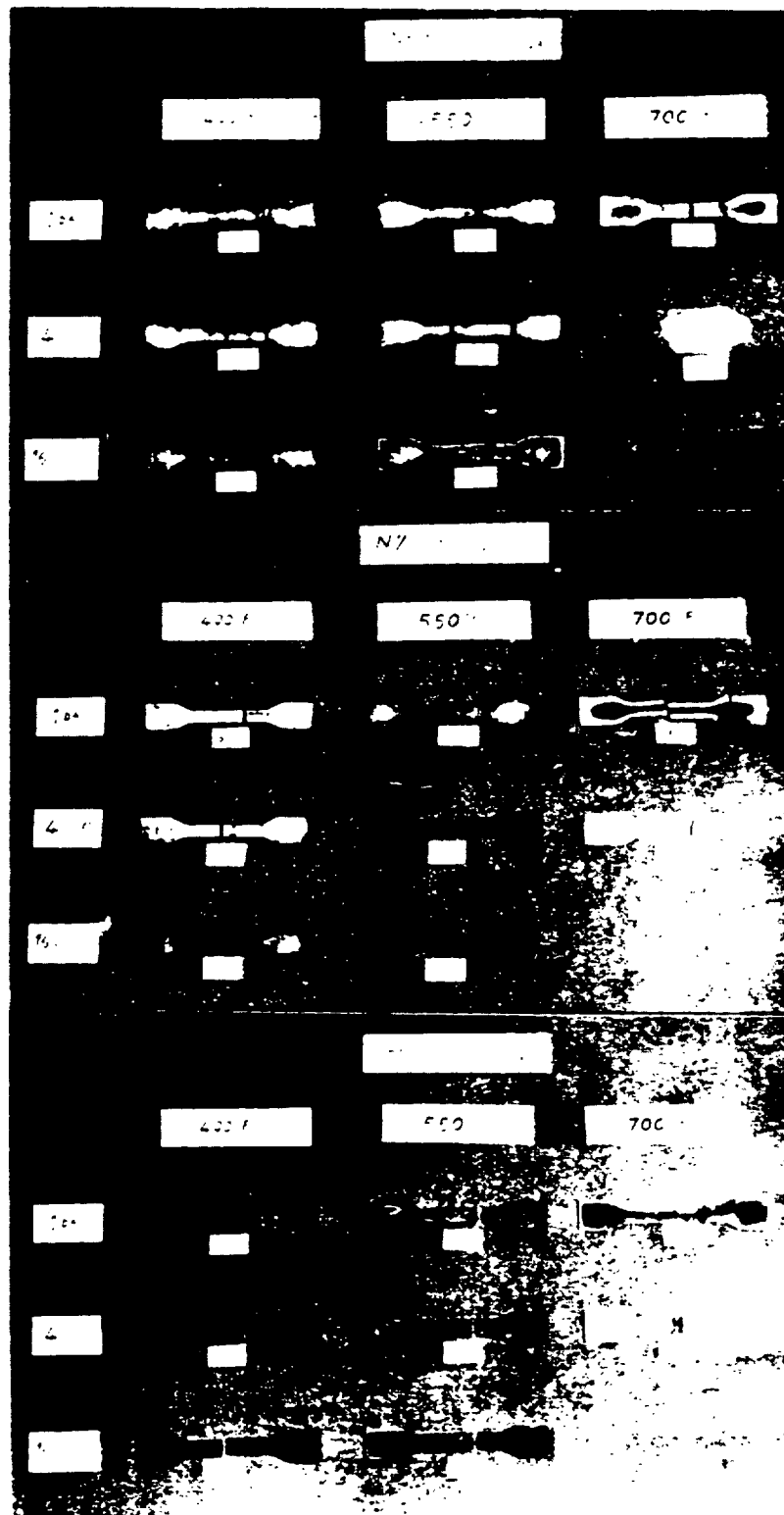
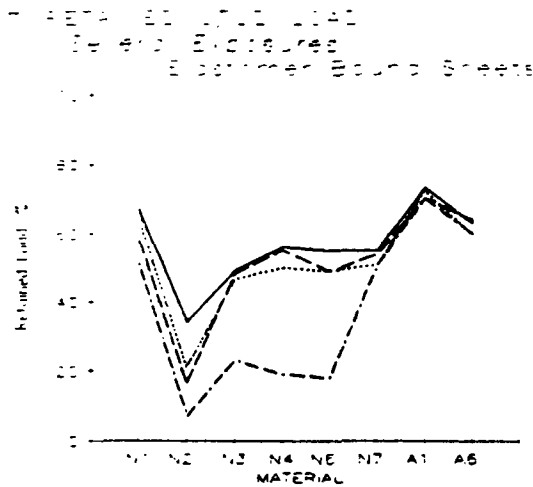


Figure 22: Typical Post-Test Specimens: From Top, Aramid (N6), Glass-Reinforced (N7) and Asbestos (A6)



TENSILE STRENGTH RESULTS
For Phase II ATRS Exposures
Elastomer-Bound Sheets

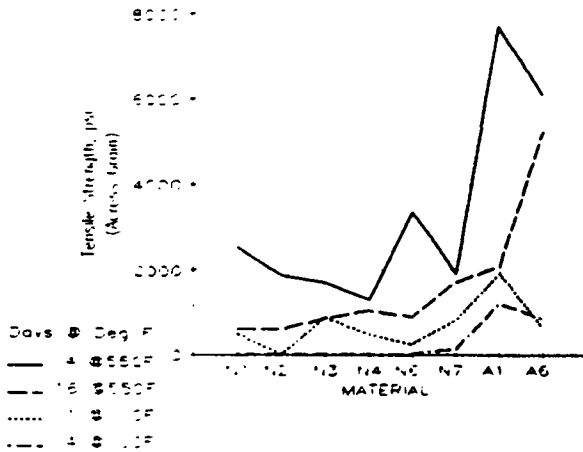
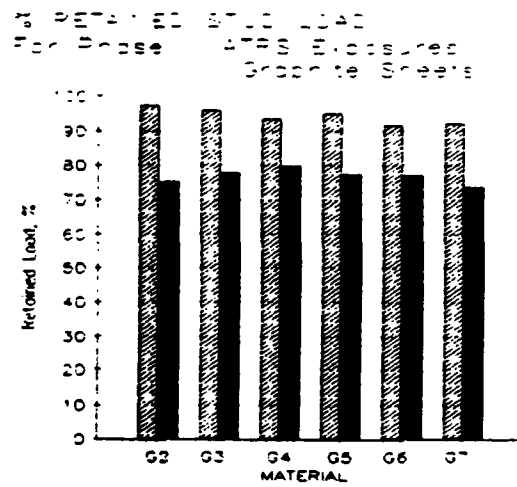


Figure 23: Phase I and II—Percent Stud-Load-Retained and Residual Tensile Strength—Elastomer-Bound Materials



TENSILE STRENGTH RESULTS
For Phase III ATRS Exposures
Graphite Sheets

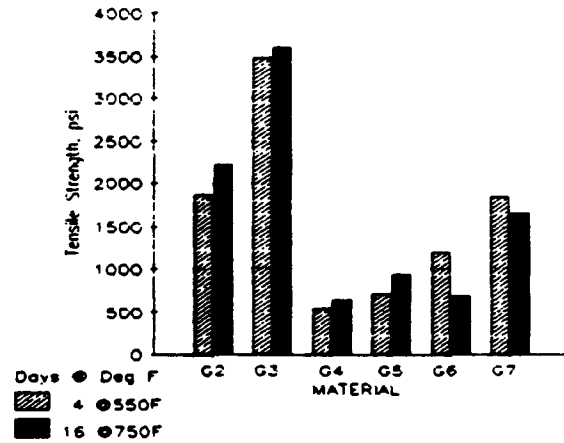
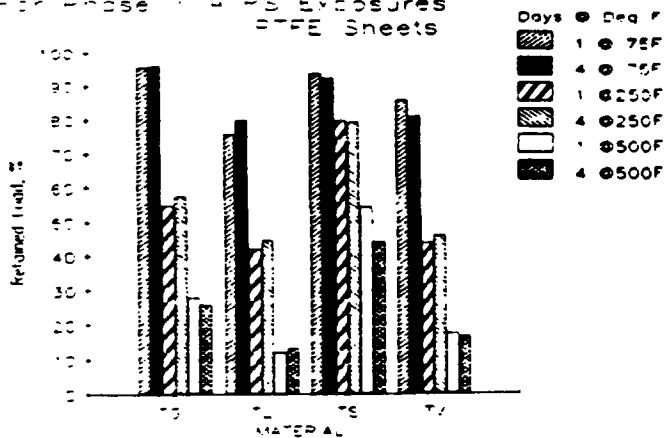


Figure 24: Phase III—Percent Stud-Load-Retained and Residual Tensile Strength—Graphite Materials

RETAINED STUD LOAD
For Phase III ATRS Exposures
PTFE Sheets



TENSILE STRENGTH RESULTS
For Phase III ATRS Exposures
PTFE Sheets

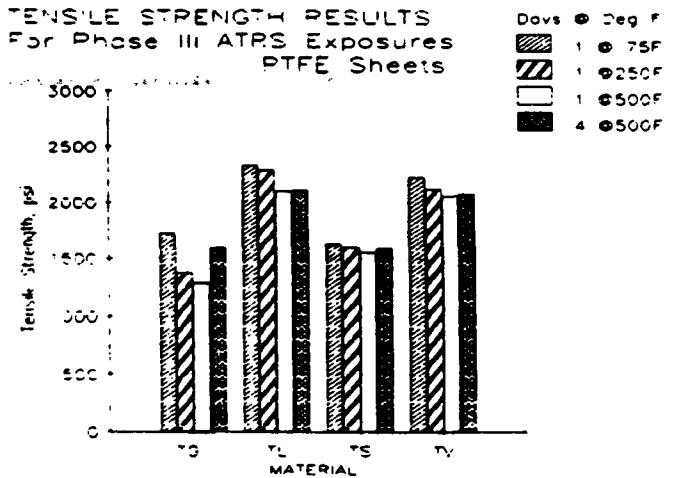


Figure 25: Phase III—Percent Stud-Load-Retained and Residual Tensile Strength—PTFE Materials

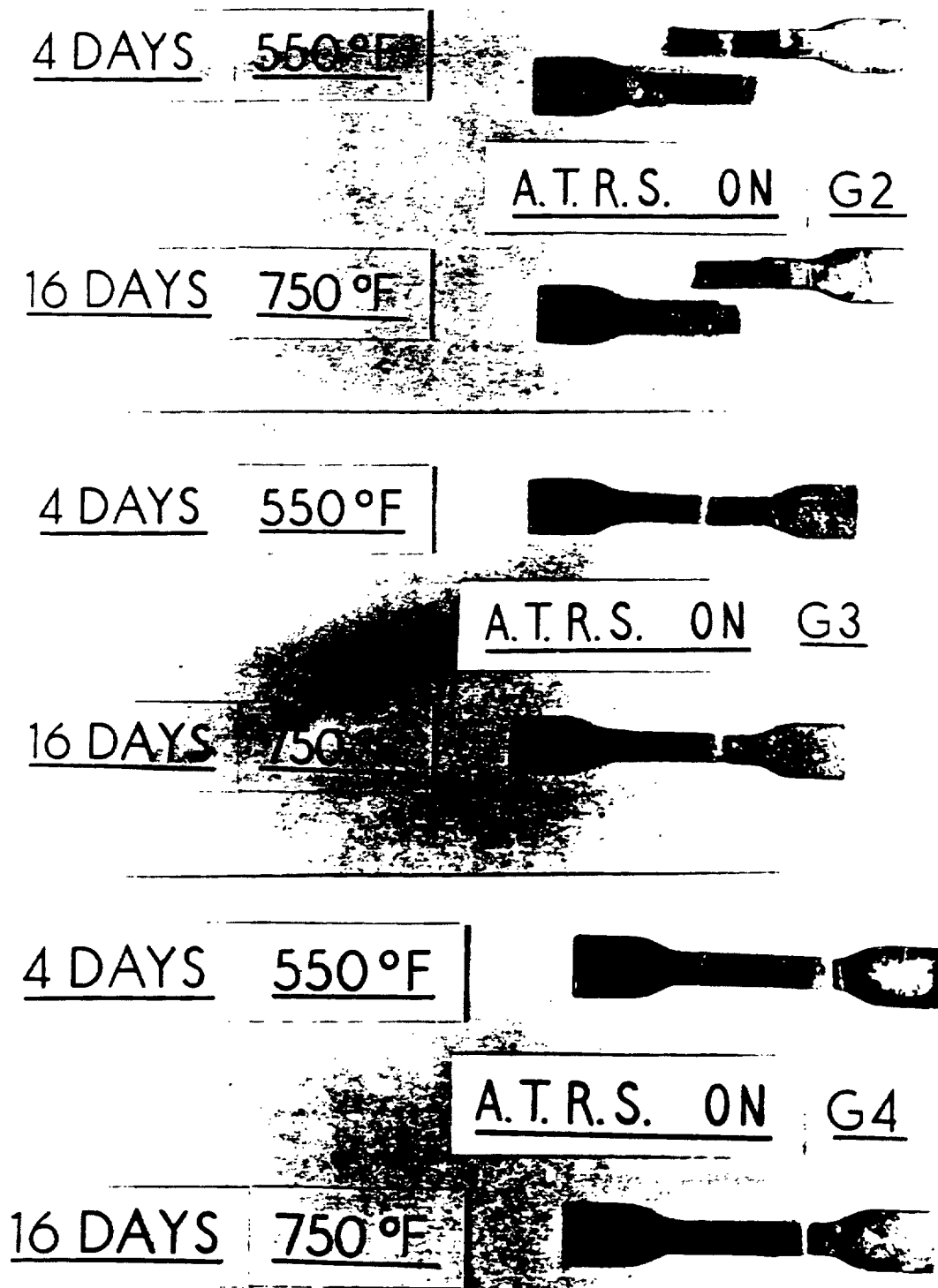


Figure 26: Post-Test Specimens—Graphite Laminate Sheets; From Top, G2 (Bonded Tp 316 Reinf), G3 (Tanged Tp 316 Reinf) and G4 (Mono Cured)

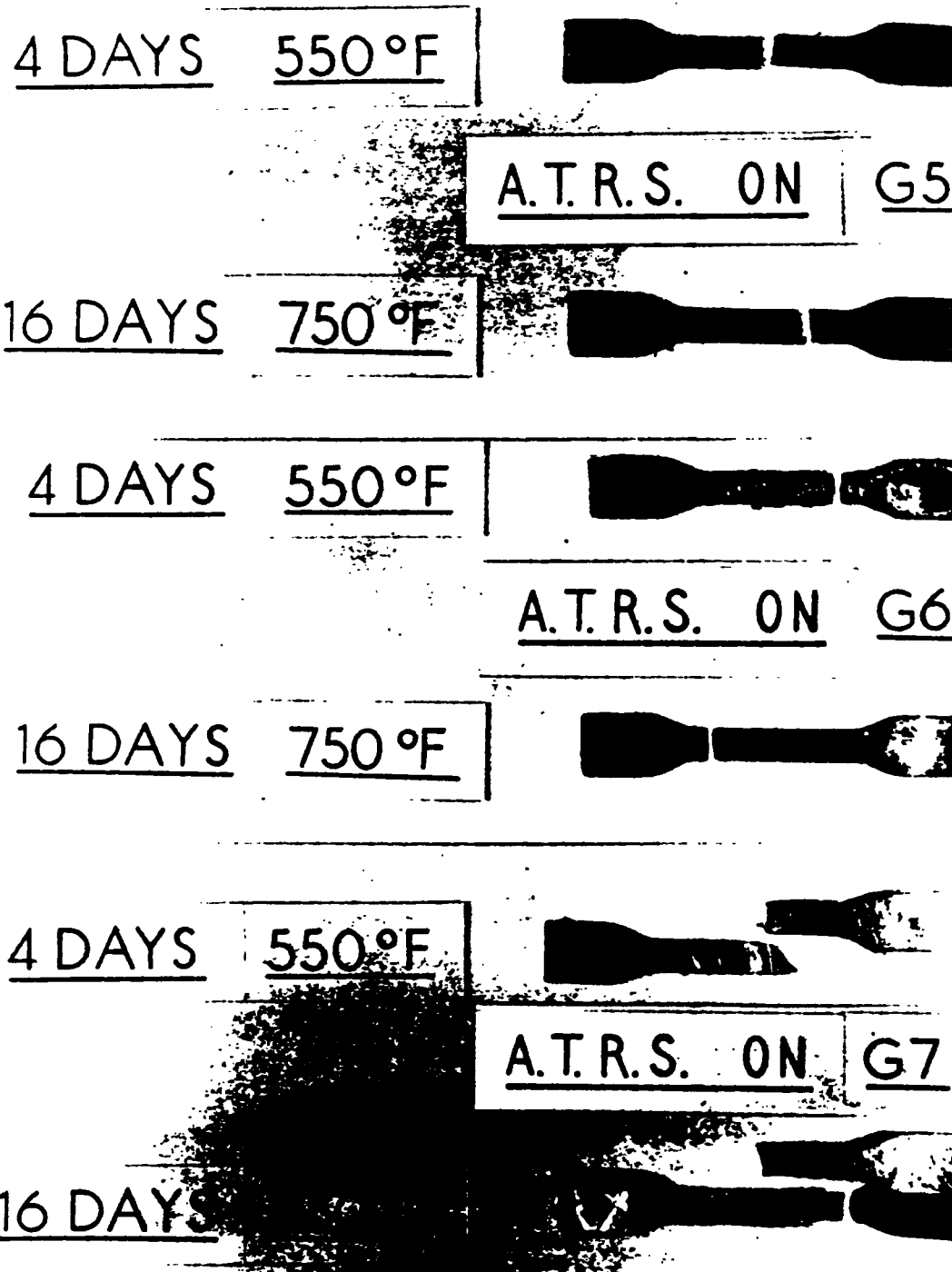
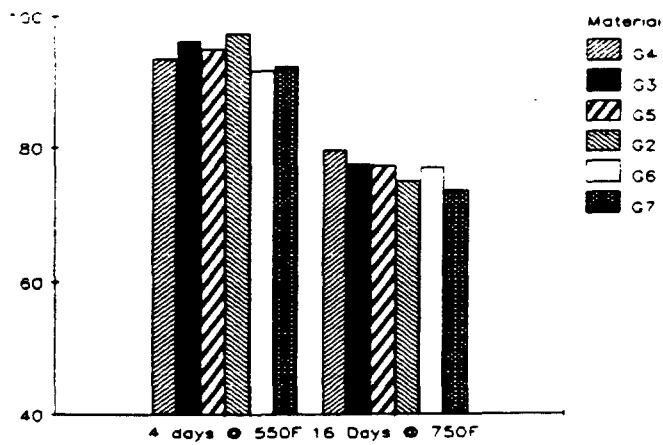


Figure 27: Post-Test Specimens—Graphite Laminate Sheets; From Top, G5 (Mono Uncured), G6 (Bonded Ni Reinf) and G7 (Wire Tp 316 Reinf)

% STLD LOAD RETAINED
Effect of Exposure - GRAPHITE



TENSILE STRENGTH
Effect of Exposure - GRAPHITE

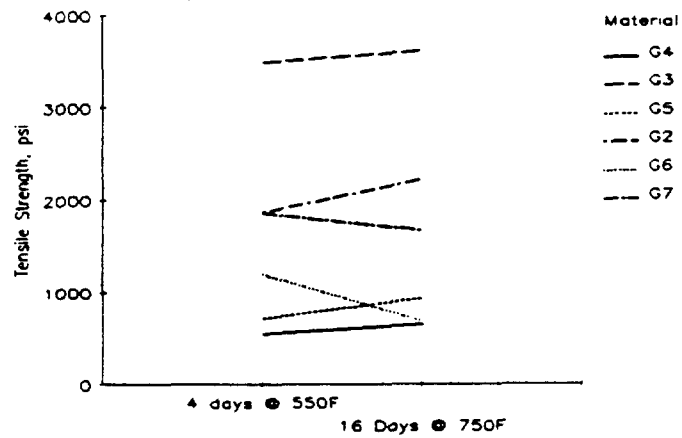


Figure 28: Exposure Versus Percent Load-Retained and Tensile Strength for Graphite Sheets

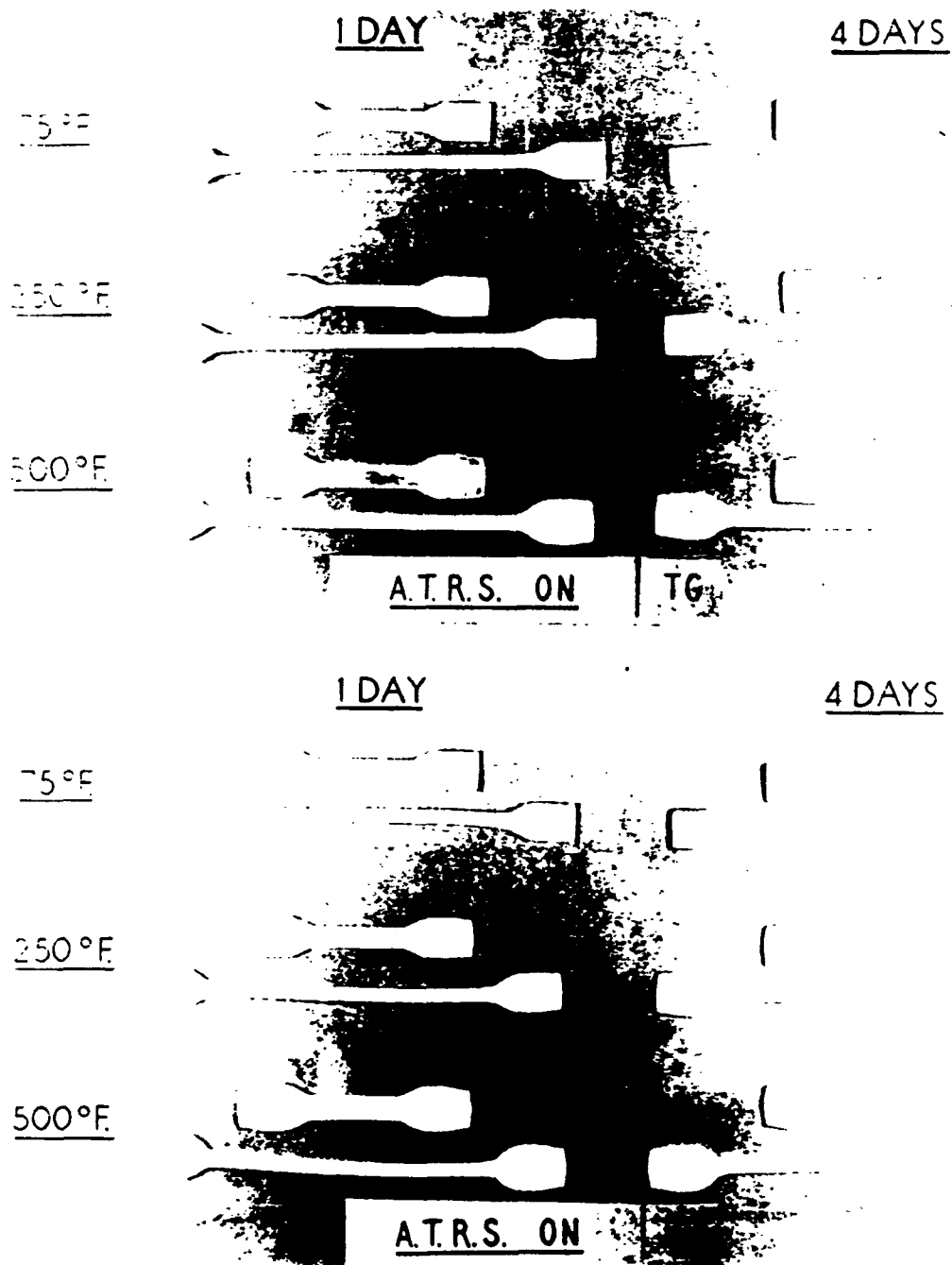


Figure 29: Post-Test Specimens—PTFE; From Top, TG and TL (Glass)

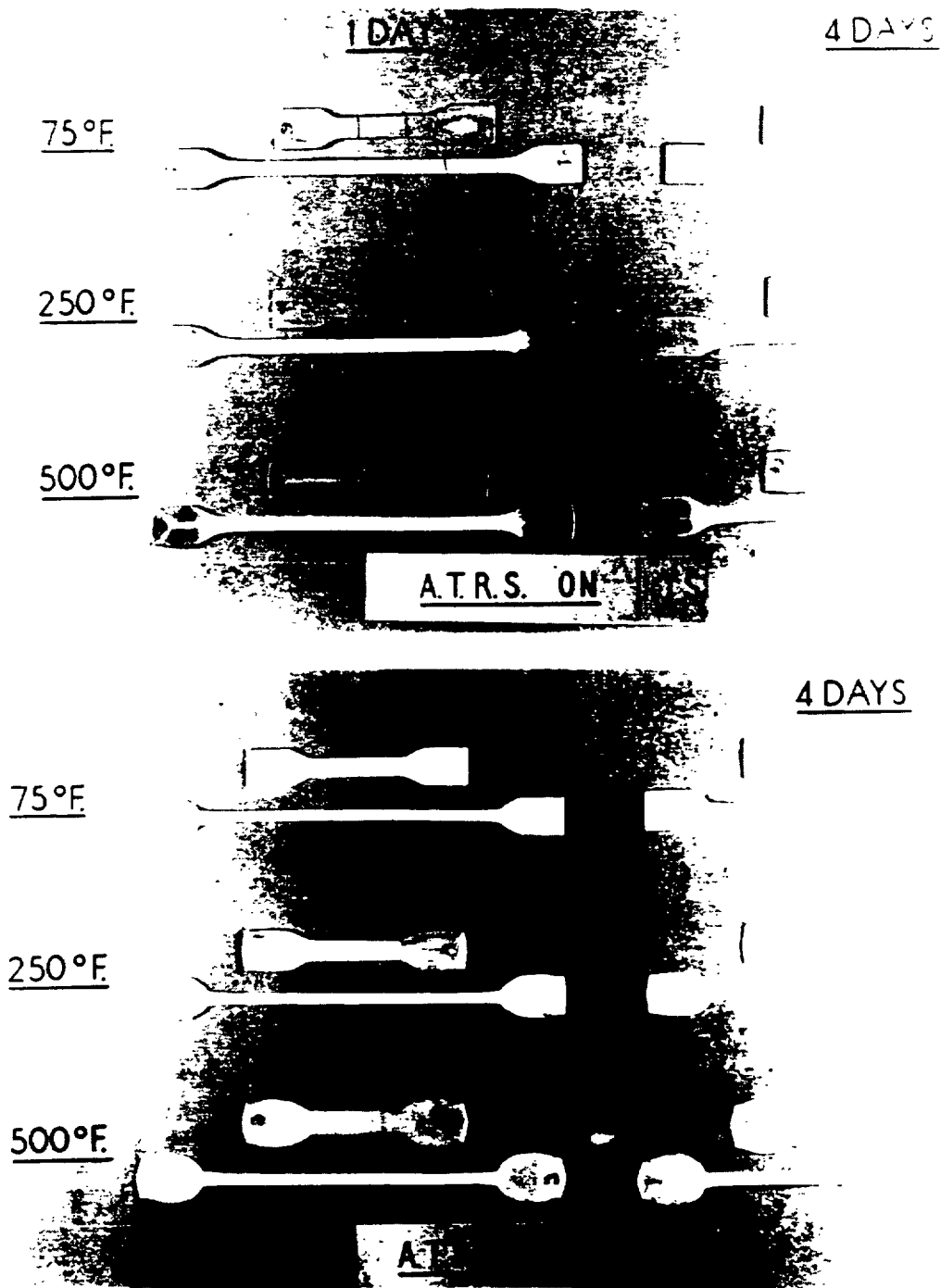


Figure 30: Post-Test Specimens—PTFE; From Top, TS (Silica) and TV (Virgin)

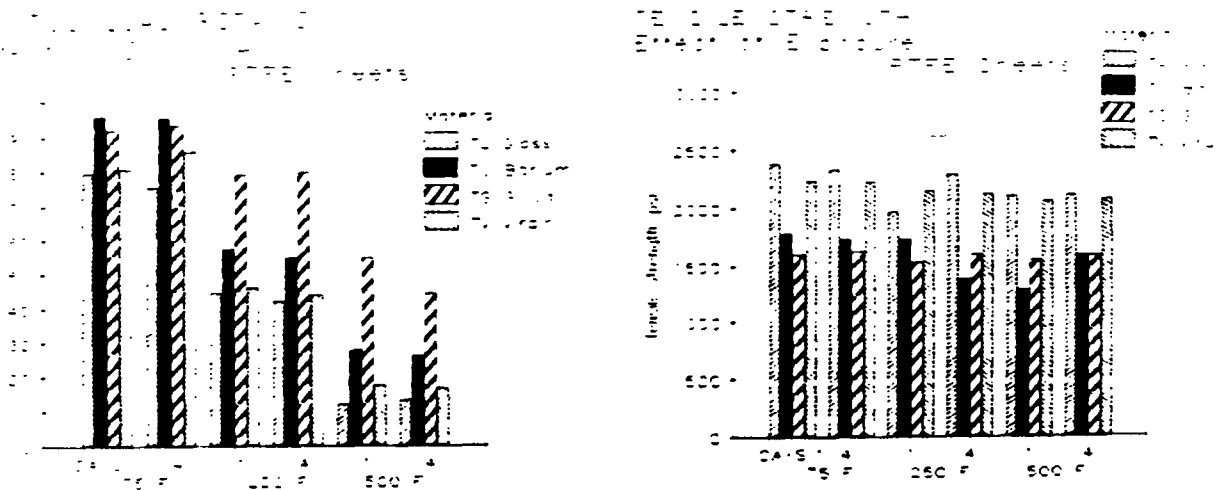


Figure 31: Exposure Versus Percent Load-Retained and Tensile Strength—PTFE Sheets

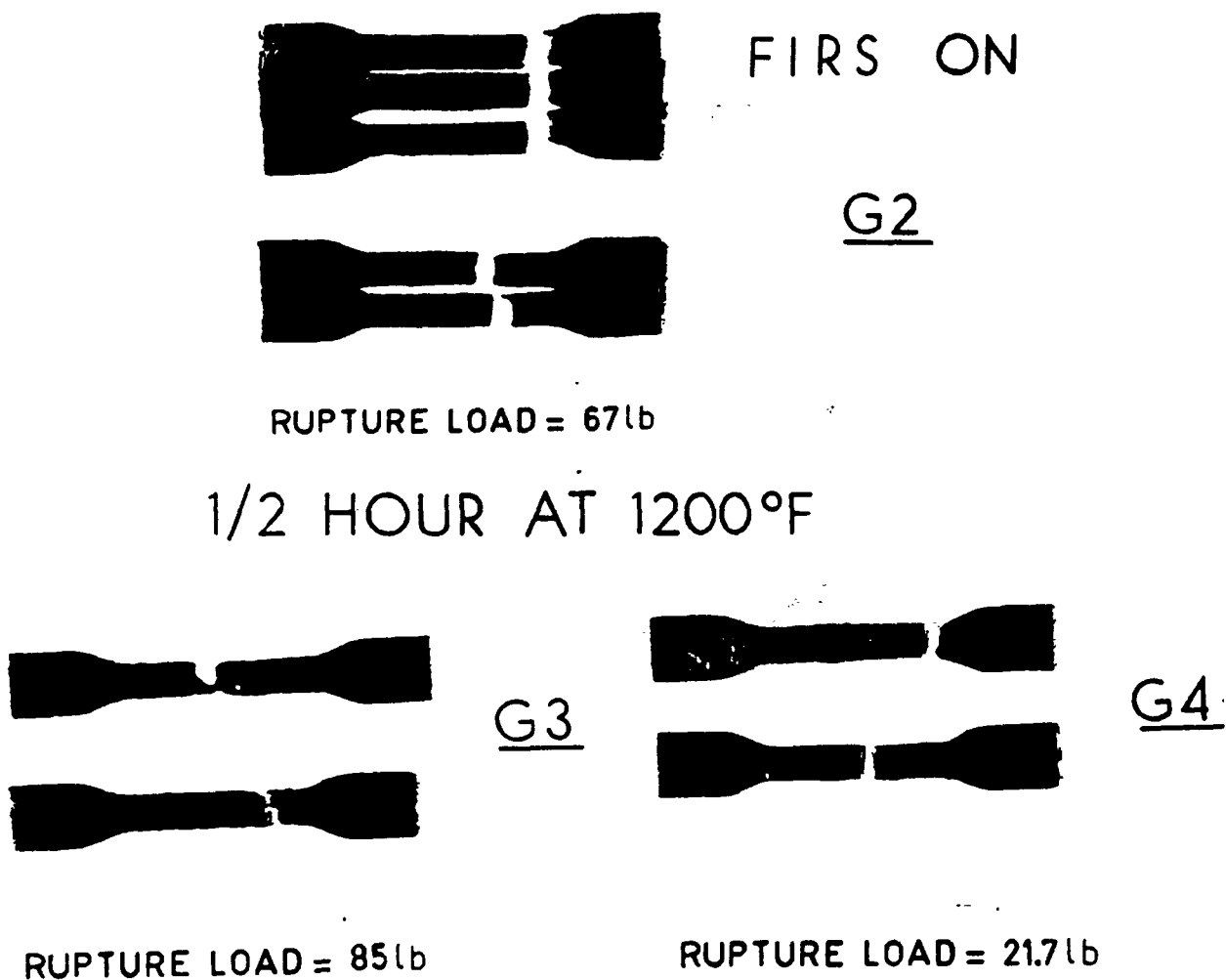


Figure 32: Post-Test Specimens—Graphite Laminate Sheets; From Top, G2 (Bonded Tp 316 Reinf), G3 (Tanged Tp 316 Reinf) and G4 (Mono Uncured)

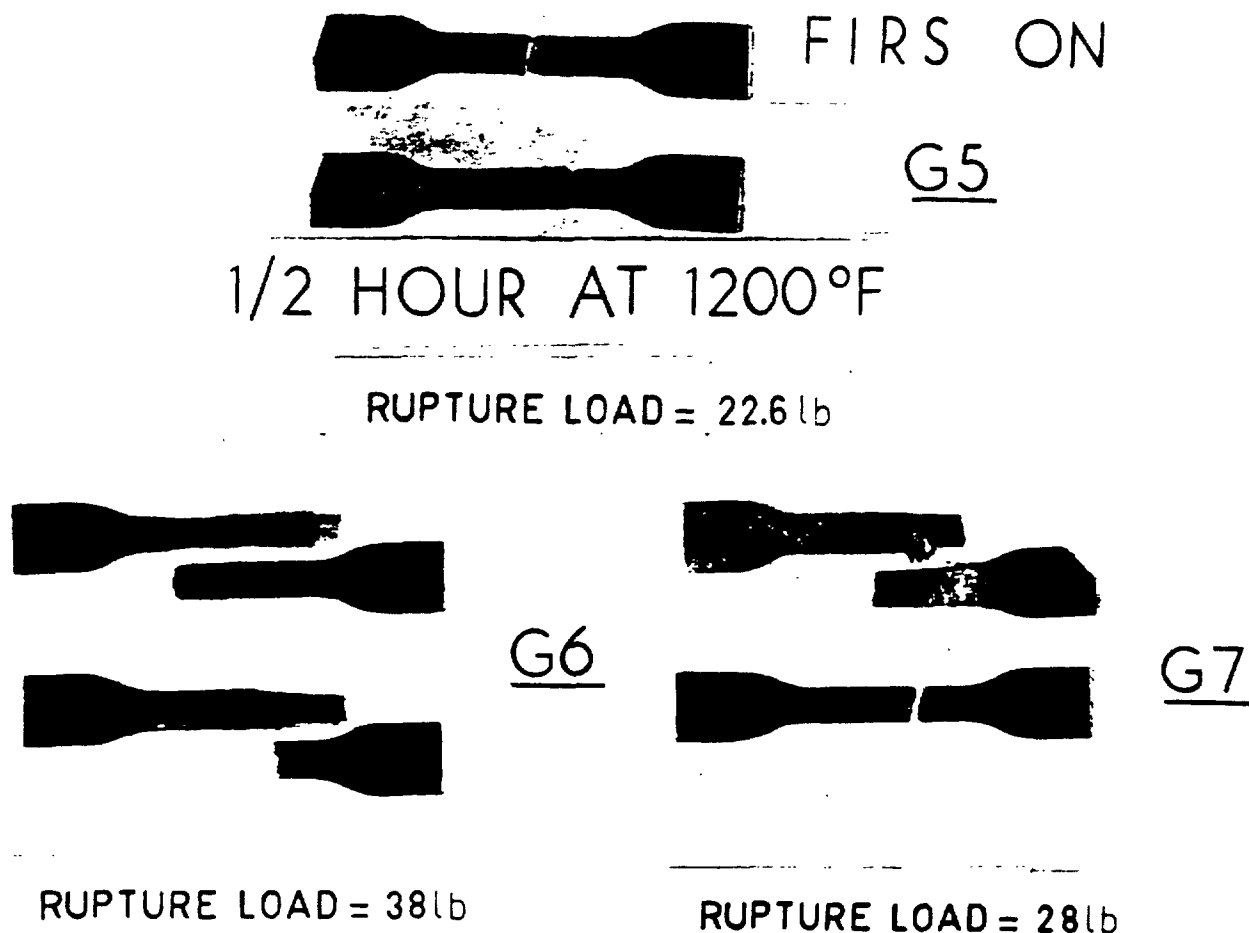


Figure 33: Post-Test Specimens—Graphite Laminate Sheets; From Top, G5 (Mono Uncured), G6 (Bonded Ni Reinf) and G7 (Wire Tp 316 Reinf)

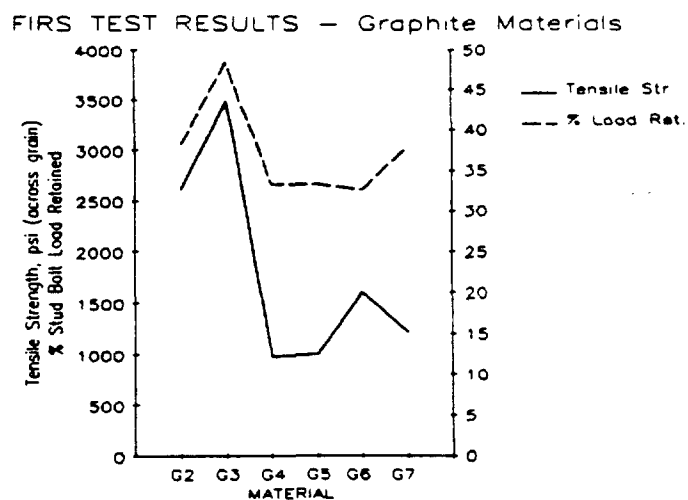


Figure 34: FIRS Test—Load-Retained and Tensile Strength—Graphite Sheets

1/2 HOUR AT 1200°F

FIRS ON

TL:  GLASS

TV:  VIRGIN

TG: 

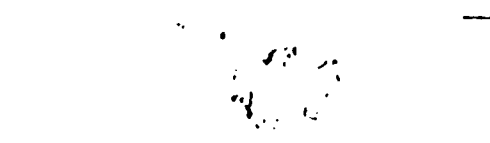
TS:  SILICIUM

Figure 35: Post-Test FIRS Specimens—PTFE

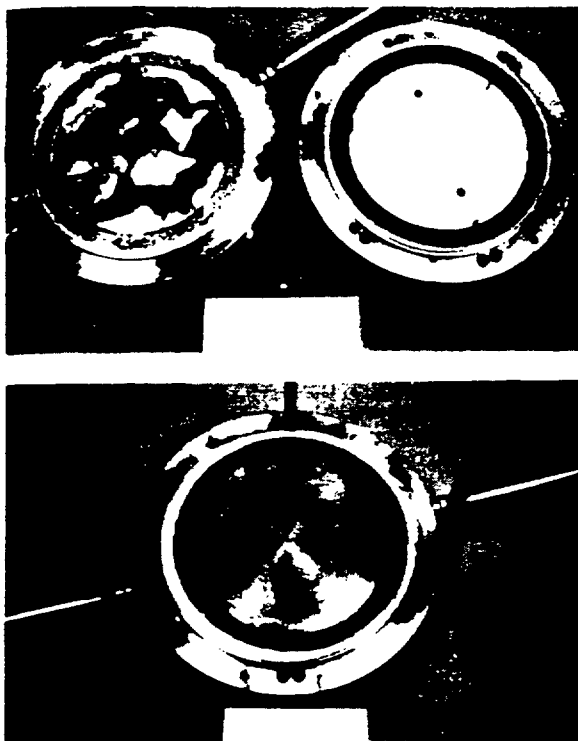


Figure 36: Post-HOTT Photo Comparison of Phase II—A6 and N6 Gaskets: Upper, Material A6; Lower, Material N6: Test Sequence, 680 to 800°F



Figure 37: Post-HOTT Photo Comparison of Phase II—N3 Gaskets: Upper, 650 to 800°F Test Sequence; Lower, 4 x 750°F Test Sequence

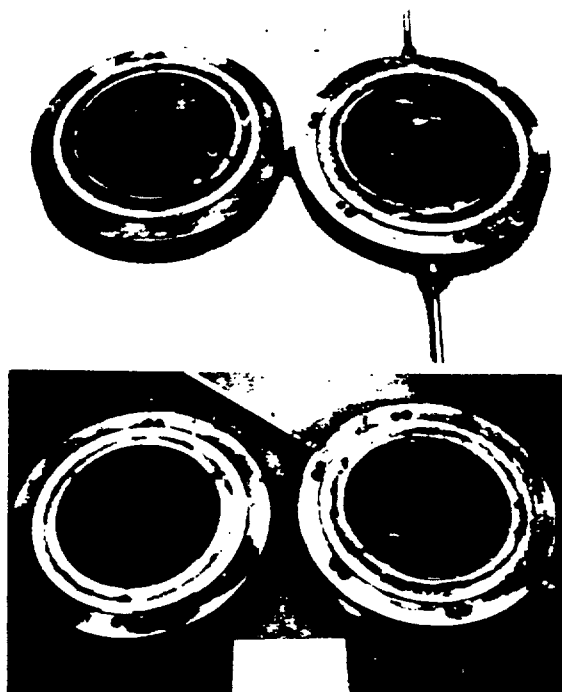
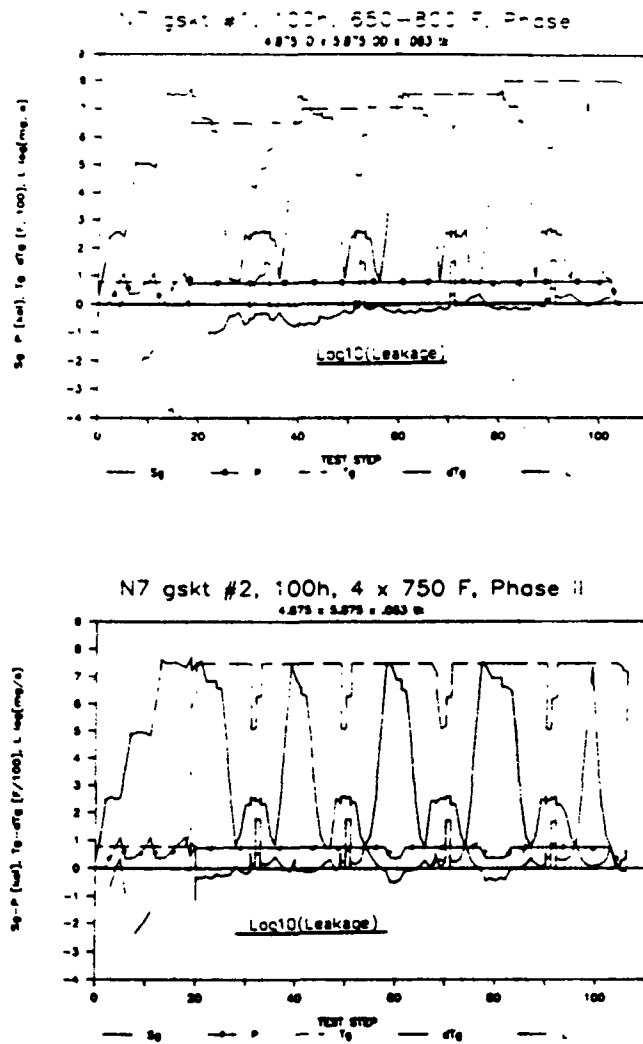


Figure 38: Post-HOTT Photo Comparison of Phase II—N7 Gaskets: Upper, 650 to 800°F Test Sequence; Lower, 4 x 750°F Test Sequence



LEGEND: S_g =Gasket Stress, T_g = Temperature, P = Pressure, L = Leakage

Figure 39: Phase III Trial Versus Final PTFE Procedures by Test Time; Upper, 12-Day Trial for TV; Lower, 7-Day Final for TV with Locked Relaxation

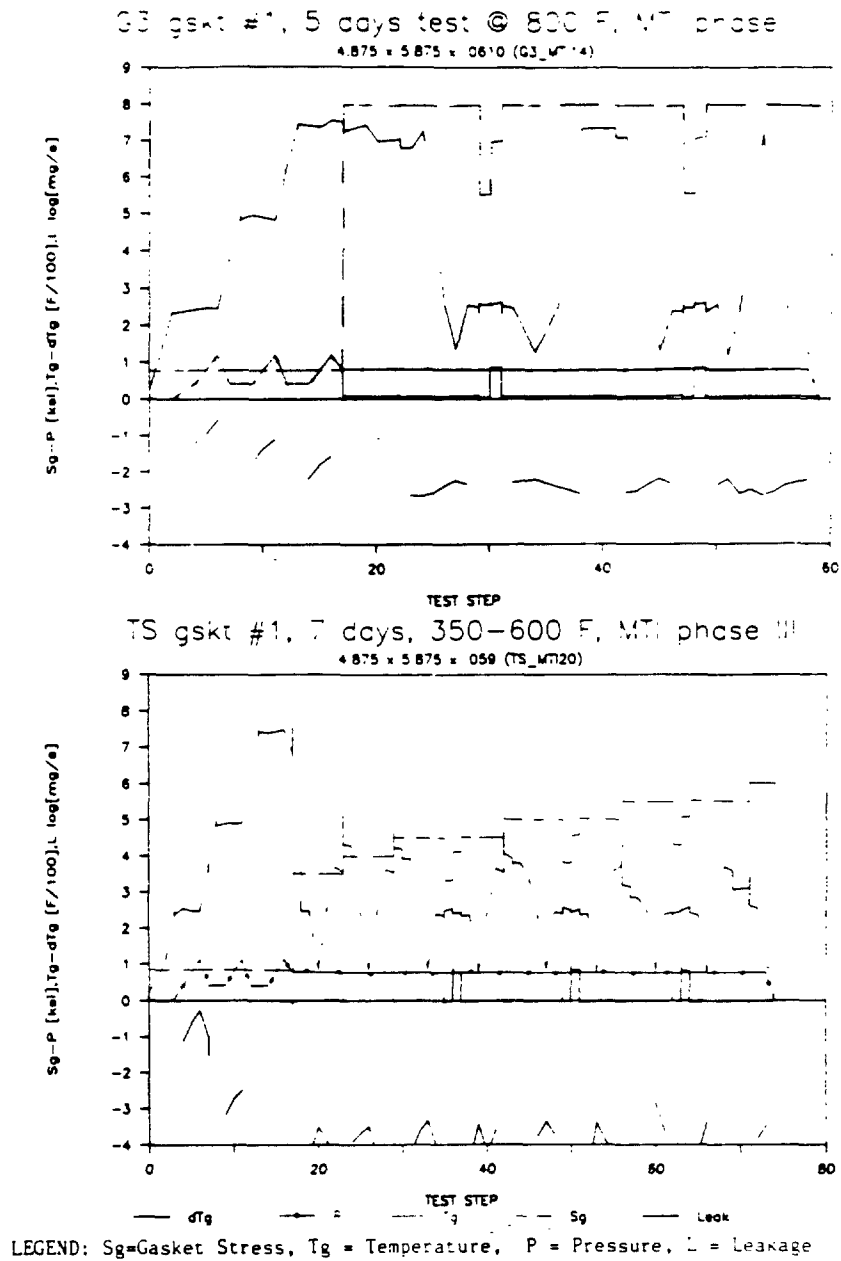


Figure 40: Phase III Procedures Illustrated by Test Step for G3 and TS

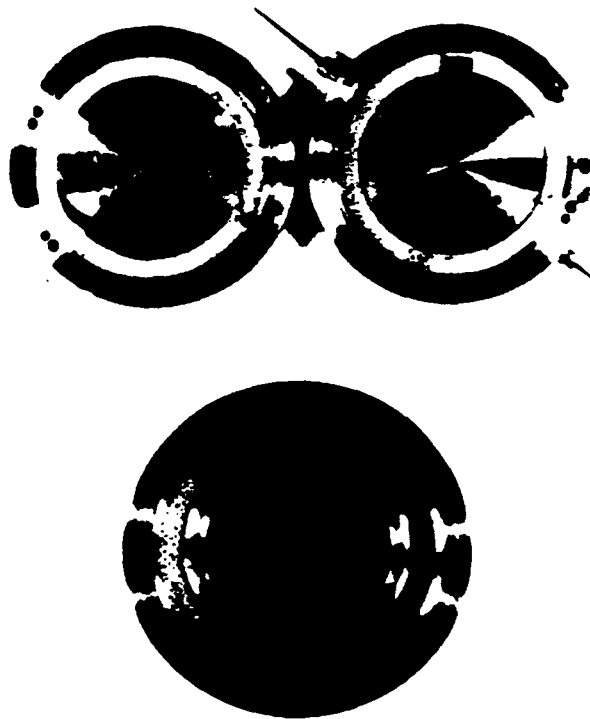


Figure 41: Post-Test Specimens—Graphite Laminate Sheets: From Top, G2 (Bonded Tp 316 Reinf) and G3 (Tanged Tp 316 Reinf)

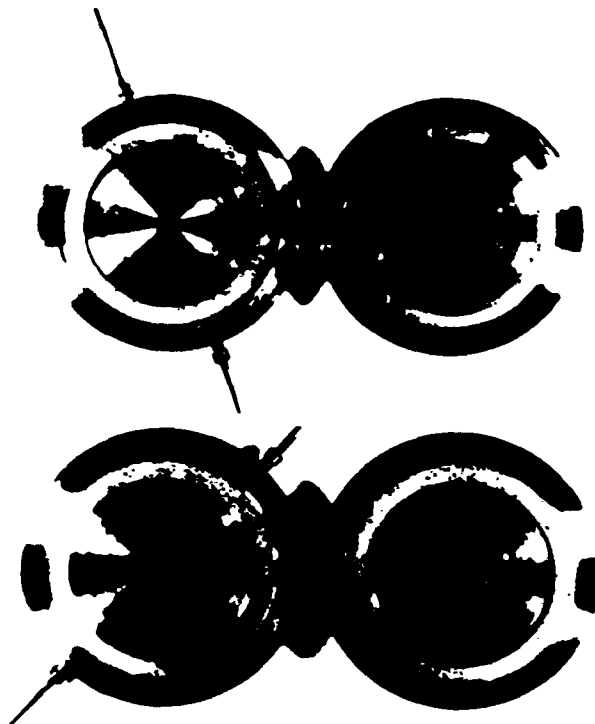


Figure 42: Post-Test Specimens—Graphite Laminate Sheets: From Top, G4 (Mono Uncured) and G5 (Mono Uncured)

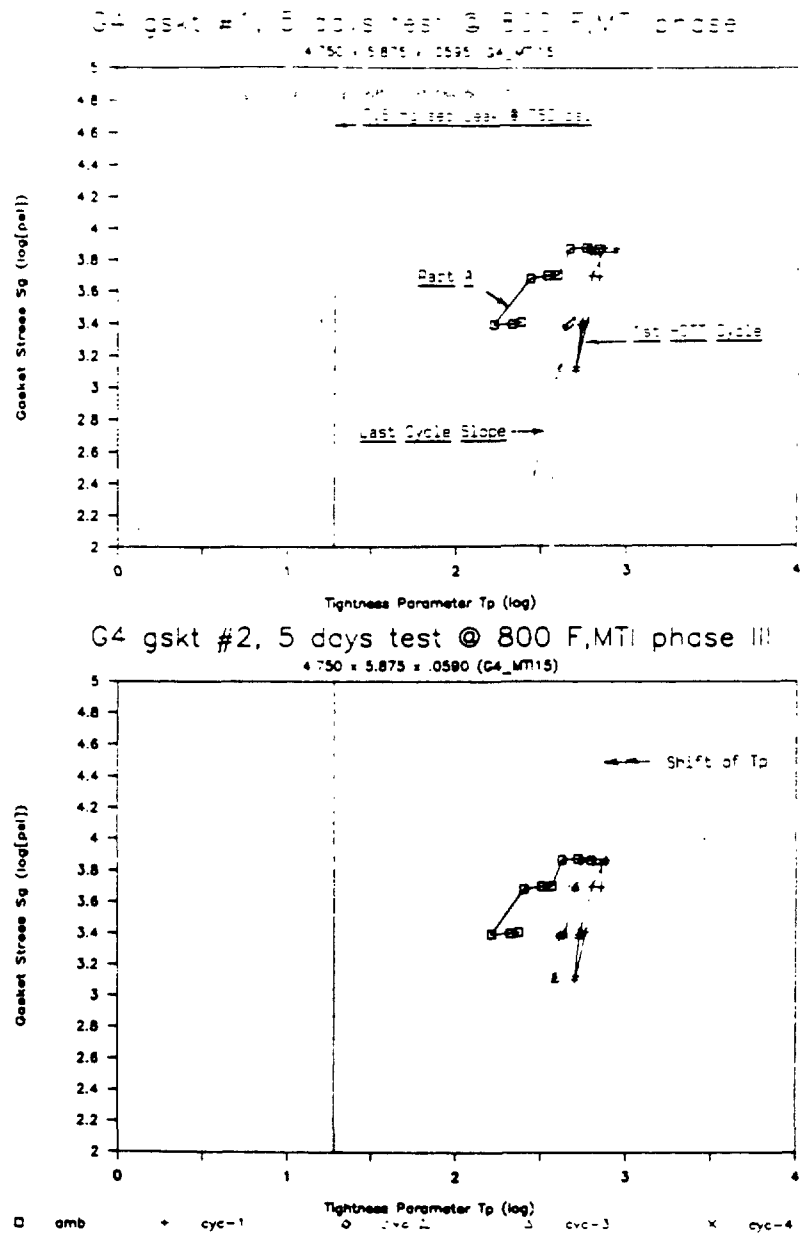


Figure 43: Stress Versus Tightness Performance for Material G4

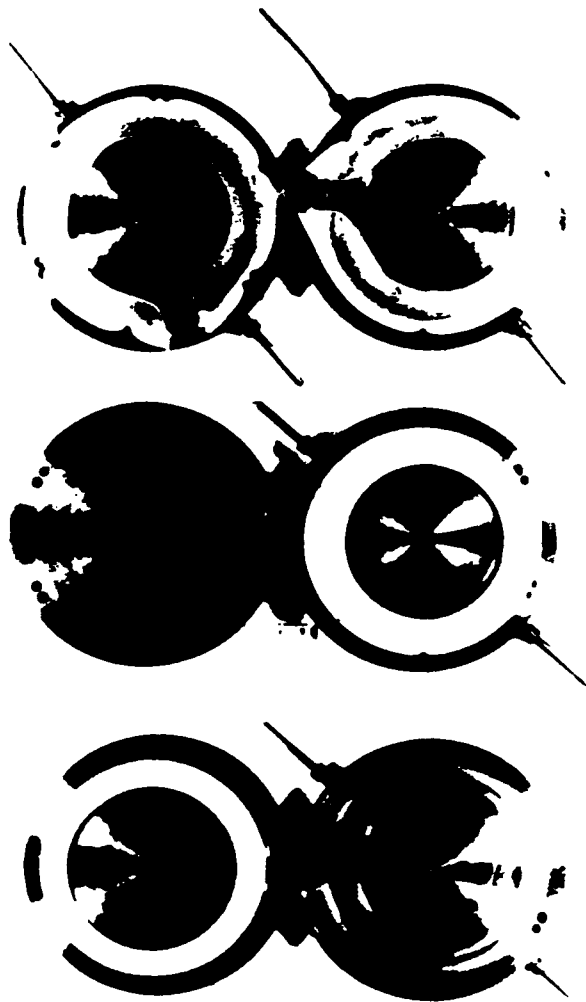


Figure 44: PTFE HOTT Specimens; From Top. TP (Virgin), TL (Glass) and TS (Silica)

SECTION 5 DEVELOPING TESTING PROGRAMS

INTRODUCTION

This chapter deals with procedures for selecting the conditions, particularly of time and temperature, for the aged tensile/relaxation screening (ATRS), fire simulation screening (FIRS) and hot operational tightness (HOTT) tests that were developed in this project. The objective was to obtain sufficient information on candidate sheet-gasket materials to permit assessment of their probable performance in service and to do so economically.

To assist in this effort, certain parameters were developed relating to stud-bolt-load retention, residual tensile strength and tightness, using the performance of asbestos fiber-reinforced sheet as a point of reference. Also, an equivalent exposure parameter, A_e , is offered for materials such as elastomer-bound types, which deteriorate with time at temperature.

EQUIVALENT AGED EXPOSURE, A_e

For materials that deteriorate on aging, it is useful to have a basis for correlating the damage done under one set of test conditions with that resulting from another set; e.g., 4 days at 550°F vis-a-vis 12 hours at 800°F. This basis leads the way to the following:

- Predictions of probable long-term performance from short-term tests.
- Setting time-temperature exposure conditions for screening tests.
- Ascertaining the need and conditions for follow-up tightness tests.
- Comparisons of performance among gasket materials.

This basis is embodied in the "equivalent aged exposure" concept, A_e , which relates change in tensile and relaxation properties to variations in exposure time and temperature. The results of ATRS tests showed that, for elastomer-bound materials, such a correlation exists and can be approximated by the following expression:

$$A_e = (T_e - T_o) (H^c) (100)/R_e$$

where T_e = Exposure temperature (°F)

T_o = Reference threshold temperature

H = Exposure time (hours)

c = Time exponent

R_e = Reference exposure corresponding to an 800°F exposure for 1000 hours*

For $T_o = 300^\circ\text{F}$ and $c = 0.20$ (see discussion below), A_e is defined for elastomeric materials as

$$A_e = (T_e - 300) (H^{0.2}) (100)/R_e \quad (1)$$

$$\text{where } R_e = (800 - 300) (1000^{0.2}) = 1990.54$$

Since this is a time-temperature exposure relationship expressed as a percentage of its value for the reference exposure of 1000 hours at 800°F (427°C), A_e is a dimensionless parameter.

The reference temperature, T_o , may be considered a threshold below which little damage occurs. It can be expected to vary with the type of binder and possibly with other ingredients, or their quantities. The task was to determine a value of T_o that would encompass the broad range of these materials. Graphical correlations indicated a range of T_o from 225 to 350 for the elastomer-bound materials studied in this project, and a value of $T_o = 300$ was selected as the most suitable. This is consistent with 300°F as a generally accepted upper limit for N-Buna (nitrile) rubber.

The time exponent, c , was determined initially by computer-assisted graphical trials. The most generally suitable value of $c = 0.2$ was also confirmed by regression analysis of a relationship between ATRS tensile results and exposure, with time and temperature as independent variables.

*An A_e of 100 represents an exposure equivalent to 800°F for 1000 hours.

$$\log \text{ Tensile Strength} = K - K' \log (T_e - 300) - C' \log (H)$$

where K , K' and C are constants determined by the regression analysis

The time exponent, c , can be reduced from the equation and the regression constants as $c = K' C'$

The exponent, c , was found to vary from 0.05 to 0.3 depending on the binder material, the reinforcement, and the range of temperatures or hours included in the regression analysis. The exponent for relaxation also differed from that for strength. Taking the results of the ATRS tests together yielded an exponent, c , of 0.17. The two most widely tested materials, A1 and N1, had exponents $c = 0.19$ and 0.23 respectively. Thus, for simplicity, $c = 0.20$ was selected.

Applicability of Ae

It is important to remember that the correlation of Ae with damage as expressed by Equation (1) was developed for the elastomer-bound materials used in this project. A somewhat different correlation or value of exponent, c , might have resulted from a different mix of materials. Also, since the correlation covers several combinations of binders and fibers, it is a compromise. This causes some of the scatter that is seen in Figures 45 and 46, and later figures, as overlapping trends for the various test temperatures.

Improved correlations of Ae with damage are possible by taking variables such as the variety of binder and fiber into account and by using expressions with a higher order of fit (such as the square of temperature). However, this defeats the purpose of having a broadly applicable correlation. For Ae to be useful for comparing materials for the class of elastomer-bound materials, it must be applicable to the entire range of materials in that class.

Thus, any improvement of Ae must reduce scatter for the broad range of materials in its class. As of this writing, no other expression for Ae has been found that reduces scatter without also limiting the range of its application. As more test data become available, work to reduce scatter and to improve broadly applicable Ae correlations would be worthwhile.

No matter what the value of the exponent, c , a large amount of scatter occurs. Fortunately, the value of c has a relatively small effect on the prediction of a service temperature limit. In a later example, the effect is seen as less than 5% for $c = 0.165$ versus $c = 0.20$.

To show how Equation (1) correlates with damage, the across-grain (weak-direction) tensile strength for materials A1 and N1 was plotted against calculated Ae in Figure 45. The trend of results for each nominal oven temperature is shown separately, as are points representing the FIRS results at 1100 and 1200°F. Since the data used for this correlation include the FIRS tests, the breadth of the temperature range of the correlation was established to 1200°F.

In Figure 45 an inflection point in the downward trend of tensile strength occurs in the 45 to 50 range of Ae for both materials. A1, the asbestos-reinforced material, had a residual strength of 1000 to 2000 psi at this point, while N1, an aramid-fiber-reinforced material, had none. It appears that deterioration of the elastomer binder was complete at the break. The better performance of A1 after the break is attributed to the strength of its intact fibers.

Reasonable trends of gasket relaxation resistance versus Ae were obtained as well. These are shown in Figure 46 where percent retained stud-bolt load was plotted against Ae for materials A1 and N1. Although an inflection in behavior occurred at an Ae around 50 for N1, the trend of A1 was a continuing slight but steady decline. The superior strength and relaxation performance of A1, the asbestos-reinforced sheet, is attributed to the greater mass of fibers plus the fact that they remained intact.

TIME-TEMPERATURE CONDITIONS FOR ATRS TESTS—CONSIDERATIONS

There are several general considerations associated with setting the time-temperature conditions for ATRS tests.

Objectives

In setting test conditions, the differences between two possible objectives should be considered:

- One is to evaluate a material for a specific exposure or temperature.
- The other is to seek the probable service temperature limit for a material which, in this project, is called the Ts.

Staged Tests

It is worthwhile to conduct tests in two stages since it is unclear how a particular material might perform beforehand. The first stage provides the results needed to approximate a service temperature limit, T_s , and the second stage provides the information needed to fine tune it. This approach has a cost benefit, elimination of unnecessary tests by early discovery of poor performers.

Multiple Candidates

Evaluating a group of candidates together permits further economies via the staged screening process wherein the poorest candidates are rejected early, thereby eliminating the expense of completely testing each candidate.

ATRS TIME-TEMPERATURE TEST CONDITIONS: ELASTOMER-BOUND MATERIALS

Using tensile strength as the criterion of structural stability, Figures 45 and 46 show that the binder was destroyed and materials A1 and N1 deteriorated to conditions dictated by fiber content alone as exposure progressed from $A_e = 30$ to $A_e = 50$. Therefore, if the objective were to determine a probable service temperature limit, T_s , test conditions for an elastomer-bound aramid-reinforced material would include exposures in the range of $A_e = 30$ to 50. Thus, for the initial test series, tests at $A_e = 30, 40$ and 50 would be conducted. The second series could provide a repeat test at $A_e = 50$ but at a different temperature.

To help visualize the relationship between temperatures and times of these test exposures, lines of constant equivalent aged exposure, A_e , for a range of times (log scale) and temperatures were plotted in Figure 47. The shaded area of Figure 47 indicates test possibilities in the important A_e range of 30 to 60. Tests shorter than 1 day would not be used, the emphasis being on the longer-term effects; the 750°F boundary reflects the limit of the scope of this project. Figure 47 is based on Equation (1).

To ensure that trends are clear and that better and poorer aramid-reinforced materials are accounted for, the conditions for a second series of tests would include a wider range of exposures, e.g., tests at $A_e = 20$ and 60 for aramid- or glass-reinforced materials.

Although these test conditions are appropriate for the aramid- and glass-reinforced materials used in this project, more severe exposures would be required to challenge future products reinforced with asbestos-like fibers. In fact, exposures far above $A_e = 60$ would be needed to assess performance that is truly asbestos-like.

A more subtle observation from the data of Figure 45 is that the tensile strength of the aramid-reinforced materials showed greater deterioration at 450°F than did the asbestos-reinforced material. While this behavior was due to scatter in the properties of the material and the imperfections of the A_e correlation, it also makes clear that an ATRS test plan needs some long exposures at low temperatures to provide a closer approach to the eventual recommended temperature.

If the objective is to evaluate a material for a specific exposure or temperature, the task is simpler since it is only necessary to assess the ability of a material to withstand a specified level of exposure with acceptable results. To this end, a set of three tests at different temperatures and the same exposure, A_e , could be performed. These would be a short, a medium and a long (1000-hour) test. The short and medium tests would be conducted first to weed out poor performers prior to the long test.

LONG-TERM PERFORMANCE PREDICTIONS: ELASTOMER-BOUND MATERIALS

A_e also offers a means of estimating equivalent operating conditions and thus predictions of long-term performance, i.e., by extrapolations of A_e to times beyond the test conditions discussed above. For example, a 3-year (28,600-hr) probable service temperature limit, T_s , might be predicted from a rearrangement of Equation (1) as

$$T_s = 19.9054 A_e / (H^{0.2}) + 300 \quad (2)$$

which for $A_e = 40$ and $H = 28,600$, predicts

$$T_s = 19.9054 (40) / (28,600^{0.2}) + 300 = 405^\circ\text{F}$$

What this really suggests is that a 1-day test at 700°F, for $A_e = 40$, provides the same exposure as 28,600 hours at 405°F. Of course, one might choose a service time other than 28,600 hours. For 2 years (17,500 hours), T_s would increase to 415°F.

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It is worthwhile to conduct tests in two stages since it is unclear how a particular material might perform beforehand. The first stage provides the results needed to approximate a service temperature limit, T_s , and the second stage provides the information needed to fine tune it. This approach has a cost benefit elimination of unnecessary tests by early discovery of poor performers.

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What this really suggests is that a 1-day test at 700°F, for $A_e = 40$, provides the same exposure as 28,600 hours at 405°F. Of course, one might choose a service time other than 28,600 hours. For 2 years (17,500 hours), T_s would increase to 415°F.

The main point of this example is to show that a basis for performance prediction is available if it follows directly from the correlation of the ATRS results with Ae and carries the assumption that similar damage is inflicted for the equivalent exposure. Thus, the above example predicts that materials having adequate quality at an Ae of 40 are adequate for 28 600 hours service at a temperature below 405°F. Of course, this method represents an extrapolation of the 1000-hour ATRS data for which additional confirmation would be very welcome.

Figure 48 shows lines of constant equivalent aged exposure, Ae, for a range of times and temperatures in a manner that illustrates such service life predictions. Note that Figure 48 applies only to Ae between 20 and 80, which at present seems practical for nonasbestos elastomer-bound products.

AGED-EXPOSURE CLASSES FOR ELASTOMER-BOUND MATERIALS

In this project, the elastomer-bound materials were subdivided into Ae exposure classifications as shown in the table below. These Ae classes allow evaluation according to service temperature and corresponding years of service. Considering the complications introduced by aging, subclassification of the material by exposure, rather than by temperature, provides a convenient means of satisfying the objective of assessing a material for a specific exposure condition. The classes in turn indicate specific service temperature limits and corresponding years of service.

The tabulation below lists the exposure classes. The table is based on Equation (2) and the temperatures are rounded to the nearest 5°F. According to the table, a Class 50 elastomer-bound sheet is suitable for 2 years at 430°F and for 3 years at 400°F.

*Aged-Exposure Classification Temperatures
for Elastomer-Bound Materials*

Ae (c = 0.2):	20	30	40	50	60	70	80	90	100
Service Years 1	365	395	430	460	495	525	560	590	625
Service Years 2	350	380	405	430	455	480	510	535	560
Service Years 3	340	360	380	400	420	440	465	485	505

PTFE AND FLEXIBLE-GRAPHITE SHEET MATERIALS

One conclusion from the ATRS results was that the tensile strength and bolt-load retention capabilities of the polytetrafluorethylene (PTFE) sheet materials studied in this project were strongly influenced by temperature but very little by time of exposure. Thus, for PTFE sheets, the correlation of exposure with damage could be treated as depending only on temperature and, while it is possible to apply the idea of Ae to PTFE, the constants To and c would change (probably to To = 0°F, c = 0). There would be no benefit in complicating things with a different Ae, since temperature could be used directly for the evaluation of PTFE materials.

Similarly, application of the Ae concept to laminated flexible-graphite sheet was considered unnecessary within the 750°F scope of the project because exposure effects were small. As shown in Figure 28, the effect of exposure was nil for residual tensile strength, and stud-bolt-load retention was reduced about 15%. Further, it is unclear whether this reduction, although small, was due to the time of exposure (aging) or temperature alone. It is more likely that temperature, and not aging, was the dominant factor, because graphite tends to soften at higher temperatures.

Since aging effects may be more pronounced for flexible graphite above 750°F, a correlation of Ae with damage may eventually be needed for this material. Further tests would illuminate this aspect of flexible graphite. However, such tests would need to be done as well at temperatures beyond the scope of this project (to 1000°F or so) to properly address this question for graphite sheet. As with PTFE, there would be no benefit in complicating testing and data interpretation unnecessarily if temperature can be used directly for flexible graphite for qualification purposes.

To use Ae [for elastomers with c = 0.2 as in Equation (1)] as intended for elastomer-bound materials to establish equivalence between long and short test exposures for PTFE or flexible graphite would give misleading results. Nevertheless, strictly for purposes of comparison of PTFE or flexible-graphite sheet test results to those of other materials, it is possible to use the elastomer-bound Ae for purposes of plotting or otherwise expressing exposure, as illustrated by Figure 49.

If significant aging effects were detected in PTFE or flexible-graphite products that are different from those used in this project, then use could be made of Ae (with appropriate To and c, or other algebraic expression corresponding to that data) as an interpretive aid.

PTFE Sheet Materials

The four sheet materials identified as TL glass, TG barium sulfate, TS silica, and TV virgin are a material class that is different from the elastomer-bound materials. The PTFE sheets were tested by ATRS at the four exposures of 1 and 4 days at 250°F and 1 and 4 days at 500°F. PTFE relaxed rapidly at relatively low temperature and was unaffected by aging. The findings are discussed in Section 4.

Since time of exposure would not be a primary factor, selecting conditions for ATRS tests of PTFE sheet material would be more straightforward than for materials that deteriorate on aging. If the objective were to assess a material for a specific temperature, it is suggested that three tests be made at that temperature.

On the other hand, if the objective were to seek a probable service temperature limit, it would be appropriate to make six tests in two series, the first three at 75, 200 and 350°F. The results of these tests would be used to estimate T_s . If that temperature were found to be acceptable from the viewpoint of the intended service, it could then be used to define three additional tests in a second series that would be keyed to the T_s estimate. The 75°F test would be a comparative room temperature relaxation, spreading, adhesion reference test.

In either case, a longer-time test (e.g., 16 days) should be included for comparison with 4-day tests to check for possible aging effects. If aging effects were detected, one or two additional tests might be needed in order to more completely define this effect and to establish A_e .

Flexible-Graphite Sheet Materials

The laminated flexible-graphite sheet materials identified as G2 through G6 are a different material class from the PTFE and the elastomer-bound materials. The graphite materials were tested by ATRS at the two exposures of 4 days at 550°F and 16 days at 750°F. The test results are discussed in Section 4.

Since time of exposure would not be involved, setting conditions for ATRS tests of flexible-graphite sheet material would also be based on temperature. If the objective were to assess a material for a specific service temperature, it would be advisable to make three tests at that temperature.

A 16-day test, for example, would be included for comparison with 4-day tests to check for possible aging effects. If aging effects were detected, additional tests at 42 days would be called for in order to more completely define the effect, and to establish an A_e correlation from the data for purposes of interpretation.

If the objective were to seek a probable service temperature limit, it would be advisable to make four tests in two series, the first two for 4 days at 550°F and 16 days at 800°F. The results of these tests would be used to estimate T_s , which, if acceptable from the viewpoint of the intended service, could be used to define two additional tests in a second series that would be keyed to the T_s estimate. These would be 42-day (1000-hour) tests at T_s and $T_s + 100^\circ\text{F}$. An 800°F maximum test temperature is the limit of the current ATRS fixture's temperature capability. With this range of temperatures and time, it is possible to assess the probable performance of flexible-graphite material for temperatures to 800°F. Following study of the ATRS results, additional tests and exposures above the 800°F ATRS limit may be appropriate to seek an upper service temperature limit for flexible graphite.

The 42-day tests would be compared with 4- and 16-day tests to check for aging effects. As for PTFE, if aging effects were detected, then additional tests would be needed in order to more completely define the effect and establish an A_e correlation.

PERFORMANCE CRITERIA: GENERAL CONSIDERATIONS

Another need for a workable evaluation test plan is the definition of satisfactory gasket performance. In essence, this means freedom from catastrophic failure in the form of gross leaks in operating bolted joints. For purposes of this project, satisfactory performance is defined in terms of those properties of traditional asbestos-reinforced materials that serve to prevent gross joint failure.

The percent stud-bolt load retained is a significant aspect of performance for test plan purposes. Gross leaks occur when the bolt load is low and nearly equal to the hydrostatic end force. This means that good stud-bolt-load retention must be achieved by candidate materials.

Further, gross leaks are most dramatic when a gasket "falls apart." Therefore, a candidate material should be structurally stable after exposure so that it is not destroyed by a stream of leaking fluid under low compressive stress conditions. Accordingly, a candidate should exhibit an adequate level of residual tensile strength as assurance that it does not turn to powder, or "fall apart," after exposure.

Therefore, for screening purposes, it is proposed that gasket performance be considered in terms of the properties measured by the ATRS test, namely

- Percent stud-bolt load retained and
- Tensile strength, as measured with across-the-grain (weak-direction) coupons in the case of reinforced sheet materials.

For the ATRS tests these properties are measured in a still, hot-air environment. Beyond the rather severe influence of this oxidizing medium, the effects of chemical attack by service fluids is not considered.

THE BASIS OF PERFORMANCE: ASBESTOS-FIBER REINFORCED SHEET PERFORMANCE

Familiar, good quality, asbestos-sheet products have been studied and provide a comparative measure of performance. These do not "fall apart" after exposure. The post-test properties of good quality 1/16-in.-thick asbestos-fiber reinforced sheet (such as the A1 and A6 materials) are proposed as the basis for evaluating candidate materials. This material is still accepted for operating temperatures to 750°F and above, and one manufacturer claims over 1200°F. Since there is a large body of experience with asbestos-fiber reinforced sheet, its use as a criterion of performance is appropriate.

PERCENT STUD-BOLT LOAD RETAINED AS A PERFORMANCE CRITERION

A retained stud-bolt load of 75% is proposed as a reference measure of performance based on test results for material A1. Note that material A6 had poorer retained stud load retention.

A dimensionless measure of load retention capability, Q_r , based on percent stud-bolt load retained is given here as the square of the ratio

$$Q_r = (\text{ATRS percent stud-bolt load retained}/75)^2$$

Use of the squared function of Q_r is proposed to sufficiently acknowledge the importance and contribution of stud-bolt relaxation in gross joint failures. It means that a material relaxing twice as much is four times poorer in performance capability than traditional asbestos-reinforced sheet. While this seems severe, so is the effect of relaxation on the safety of a bolted joint.

Effect of Relaxation on Joint Safety

Consider, for example, an NPS 12 ANSI Class 300 joint with a uniform initial stud-bolt stress of 20,000 psi. This stress is "settled in" after any cold relaxation since bolt-up, and is prior to any heat-up. After heat-up and exposure at conditions of, say, 1 year at 490°F, such that $A_e = 60$, this joint, with a 1/16-in.-thick A1 material gasket would have a predicted residual stud-bolt stress that is about 70% of the cold stress, or 14,000 psi. Of course, this neglects any other environmental effects.

By comparison, the same joint with an N1 material (aramid-reinforced) gasket under the same conditions would have a predicted residual stress of about 50% of the initial stress, or 10,000 psi. At a 300-psi operating pressure on the joint, the hydrostatic end force is the equivalent of about 7800 psi bolt stress. This is the bolt stress needed to just prevent gross leakage and is calculated on the basis that the gasket stress equals the operating pressure. For the A1 material joint, the safety factor on gross leakage is reduced from the ratio $20/7.8 = 2.6$ to the ratio $14/7.8 = 1.9$ as the result of gasket relaxation. For the N1 material joint, the safety factor is reduced to about 1.3. The joint with the N1 material is, therefore, much more sensitive to upsets.

The actual situation is more complicated than this example because of such things as thermal effects, flange flexibility and bolt-load variations. However, the reason for serious leaks with some substitute products is clearer when the effect of gasket relaxation on the safety of a bolted joint is considered.

Load-Retention Capability Trends

Figure 50 shows Q_r plotted against A_e for all Phase I and II ATRS data. Q_r was relatively constant for the asbestos-reinforced materials. Material A1 showed better capability than A6. The latter reached a low of 0.65. By contrast, Q_r steadily decreased with exposure for the aramid-reinforced materials, N2, N3, N4 and N6. They reached 0.1 or less at $A_e = 50$. Aramid-reinforced N1 and glass-reinforced N7 performed with a Q_r over 0.4 at $A_e = 50$. The performance difference between the substitute and asbestos-reinforced materials is quite clear. Materials exhibiting a Q_r of 0.5 or better were judged to be "as good as" asbestos-reinforced sheet for that exposure.

TENSILE STRENGTH AS A PERFORMANCE CRITERION

The residual tensile strength of asbestos fiber-reinforced sheet is the second significant measure of performance capability for test planning purposes. Given enough exposure, A_e , the residual tensile strength decreases to a minimum level after the binder is destroyed. This minimum is fiber dependent. Specifically, fiber orientation, length, density, variety, and distribution all interact to determine the residual strength. The ATRS tests were applied to coupons cut both parallel and across the dominant direction of fiber orientation, also known as the "lay" or "grain." Weak-direction coupons were found to have about 50% the residual strength of their orthogonal counterparts.

A residual tensile strength of 1000 psi is proposed as a reference measure of tensile capability. This is based on the ATRS data for materials A1 and A6 for the cross-grain (weak) direction. As seen for the asbestos-reinforced material, A1, from Figure 45, a minimum strength around this level held to a temperature of at least 1200°F, based on the FIRS results.

A dimensionless tensile criterion, Q_x , is defined here as the ratio of cross-grain tensile strength to the tensile reference:

$$Q_x = \text{ATRS cross-grain post-test tensile strength, psi/1000 psi}$$

Q_x varied from more than 10, for light exposures, to zero, for the ATRS data on Phase I and II materials as seen in Figure 51. Within the exposure range where materials exhibit a value of Q_x above 0.5, the material was judged to be about "as good as" asbestos-reinforced sheet regarding tensile strength.

Figure 51 is shown with trend lines and in two parts. The lower part has a larger scale. This is for clarity because of the scatter. (Scatter is discussed below.) The following trends are important from Figure 51 for Q_x .

- All materials tended to reach a decisive juncture after an exposure in the range of $A_e = 38$ to 50. The binder was completely oxidized at this point.
- The asbestos-reinforced materials leveled off with Q_x at about 0.8 for A_e above 50.
- The aramid-reinforced materials reached Q_x below 1/2 for A_e above 38 on average.
- The glass-reinforced material (N7) did slightly better than the aramid materials by leveling off at $Q_x = 0.2$ above $A_e = 50$. Also, a Q_x of 0.4 was measured for material N7 from an exposure of $A_e = 55$ at 1200°F in the FIRS test.

OVERALL PERFORMANCE CAPABILITY PARAMETER FOR SCREENING TESTS

The stud-bolt-load retention and strength criteria, Q_r and Q_x , are both important and should be considered together for most applications. For example, a material with perfect load retention would not be useful if it also "falls apart" because Q_x is nil.

However, a material with high Q_r but a low Q_x should be able to handle somewhat higher temperatures than indicated by the low value of Q_x alone, and vice versa. For example, at 300°F, PTFE sheet had a low Q_r and a high Q_x relative to compressed asbestos. Its tightness performance was excellent. On the other hand, unreinforced-graphite sheet also sealed very well but had the opposite situation with high Q_r and moderate Q_x .

Therefore, a combination of Q_r and Q_x is proposed as a measure of overall performance under given exposure conditions as expressed by A_e . A dimensionless overall parameter, Q_p , is defined here as the product of Q_r and Q_x . Thus,

$$Q_p = (Q_x) \times (Q_r)$$

Figure 52 shows the quality parameter, Q_p , against A_e for the ATRS data. This effectively combines Figures 50 and 51, comparing all Phase I and II data in one figure. The trends are similar to those of Figure 51. That is, the performance capability of the asbestos-reinforced materials leveled off for A_e above 50; all others fell to a Q_p less than 0.5 at an A_e around 40. In this manner the expected performance of the nonasbestos fiber-reinforced elastomer-bound sheets of Phases I and II was clearly separated from that of traditional asbestos-reinforced sheets.

DEALING WITH SCATTER

The zigzag lines of Figures 50, 51 and 52 show the scatter for the Phase I and II elastomer-bound materials. As previously discussed, scatter arises from imperfections in the correlation of A_e , from the test method, and from sample-to-sample material variations. In situations where a number of materials are compared on the same chart, the scatter makes interpretation more difficult.

Textile Fibers Group
Celriver Plant
Hoechst Celanese Corporation
2950 Cherry Road
Rock Hill, SC 29730
803 325 3200
803 325 6079

11/24/92

Minutes of Gasket and Packing QAT meeting of 11/16/92

WGHC
293

Dist:

Hal Carson - Spartanburg
Tom Caudill - Greer
William Champion - Shelby
Russ Halberstadt - Celriver
Kathy Mitchell - Celco
R.A. Hathcock - Salisbury
Bob Harrison - Salisbury
Byron Morris - Greer
Mark Swofford - Spartanburg
Priscilla Wray - Shelby
Danny Noble - Cape Industries

CC:

D. Alexander - Charlotte
L. Ashley - Celriver
K. Dean - DRP
J. Dickinson - Vanguard
R. Graupner - Leeds
J. Heckathorn - Spartanburg
J. McKinnish - Mt. Holly
R. Dick - Greer
R. Phillips - Narrows
T. Roberts - Charlotte
D. Schumacher - Charlotte
S. Wagner - Salisbury

It was determined that the original bid sent to the RPA candidates was not a completely accurate representation of the gaskets and packing most heavily used at the plants. In order to rectify this, all team members were asked to obtain the 10 most used gaskets and 3 most used packings in their respective plants. These are to be forwarded to Hal Carson who will consolidate and send to Byron Morris for distribution to the vendors. This should be accomplished by 12/18.

The discussion then turned to the elimination of any supplier candidates based on the information that the team had gathered to date. Greenville Rubber and Gasket was taken off of the list due to location and history of non-performance. John Crane was taken off for lack of interest and low variety of products offered.

Anchor, PH Sales, CGR, and Wilmington/Charlotte Rubber and Gasket remain possibilities for an RPA.

The team is to split up and visit the above suppliers before the next meeting in January of '93.

Charlotte Rubb/ Ph Sales-
Bob Hathcock, Bob Harrison, Priscilla Wray, Tom Caudill

Anchor/ CGR-
Russ Halberstadt, William Champion, Mark Swofford, Kathy Mitchell, James Hunt

Wilmington-
Danny Noble, Bill Clark, Byron Morris

The suppliers are to be asked to make a presentation at the next QAT meeting to be held in Charlotte on January 25. A list of questions for them to answer will be forwarded to them by Byron Morris.

Russ Halberstadt

HOECHST CELWESE
SALISBURY PLANT

TO: Distribution

DATE: October 13, 1992

FROM: R. A. Hathcock

REF. NO. RAH-10-92

SUBJECT: Packing and Gasket QAT Meeting Minutes

The Packing and Gasket QAT met on October 5, 1992 at the Salisbury Plant. Those present were:

Hal Carson	Purchasing, Spartanburg
William Champion	Maintenance, Shelby
Russ Halberstadt	Purchasing, Celriver
Bob Hathcock	Purchasing, Salisbury
Kathy Mitchell	Engineering, Celco
Byron Morris	Purchasing, Greer
Dick Murph	Maintenance, Salisbury
Mark Swofford	Maintenance, Spartanburg
Ken Waypa	Engineering, Salisbury
	Pipe Standards Committee Member
Priscilla Wray	Purchasing, Shelby

The agenda was has follows:

Byron Morris opened the meeting and introduced Ken Waypa, Engineering Salisbury, Pipe Standards Committee Member, to the group.

Discussion on material type and manufacturer of gaskets which pipe standards group has recommended for piping only. A survey will be sent to suppliers who are finalist for specific gaskets for piping only to determine if suppliers can meet pipe standard recommendations. It was decided to extend the survey to P. H. Sales and Greenville Rubber and Gasket.

Friskhorn was eliminated due to only servicing one plant in the region. Others eliminated were McJunkin and East Coast Sealing.

The supplier list now includes the following to this point:

1. Anchor Packing
2. Carolina Gasket & Rubber
3. Charlotte Rubber & Gasket (Wilmington Rubber Gasket)
4. John Crane (packing only)
5. P. H. Sales
6. Greenville Rubber & Gasket

From the survey each member will rank suppliers and discuss. Suppliers will be invited to meet with QAT members and discuss capabilities. Byron will draft a letter to suppliers informing them that they have been eliminated.

Before the final committment, members of the QAT, probably consisting of one purchasing and one user representative, will visit the supplier's facilities.

The next meeting will be on November 16, 1992 at 10:00 at the Celriver Plant, Rock Hill, SC. The agenda will be:

1. Reduction of suppliers to final RPA candidates.
2. Discuss meeting and dates for supplier presentation.

R. A. Hathcock

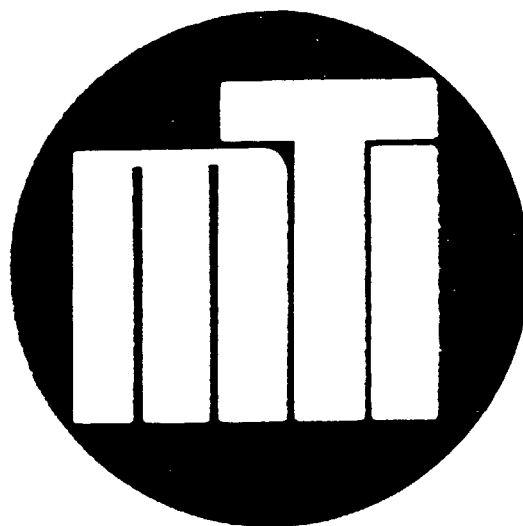
R. A. Hathcock
Buyer, Salisbury Plant

cc: D. Alexander - Charlotte
L. Ashley - Rock Hill
K. Dean - Dryefus, Charlotte
J. Dickinson - Vanguard
R. Graupner - Leeds
J. Heckathorn - Spartanburg
J. McKinnish - Mt. Holly
P. Mitchell - Greer
R. Phillips - Narrows
T. Roberts - Charlotte
D. Schumacher - Charlotte
S. Wagner - Salisbury

Distribution: Hal Carson - Spartanburg
William Champion - Shelby
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Kathy Mitchell - Celco
Bob Harrison - Salisbury
Byron Morris - Greer
Mark Swofford - Spartanburg
Priscilla Wray - Shelby
Danny Noble - Cape Industries

MTI PUBLICATION NO. 35

**SUMMARY REPORT
ON ELEVATED TEMPERATURE TESTS
FOR ASBESTOS-FREE GASKET MATERIALS**



Materials Technology Institute of the Chemical Process Industries, Inc.