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EVALUATION
OF
TITAN III AFT CLOSURE INSULATION

J. R. ELLISON, LT, USAF

TECHNICAL REPORT AFRPL-TR-70-89

AUGUST 1970

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EVALUATI~~ON OF~~ TITAN III AFT CLOSURE INSULATION

John R. Ellison, Lt, USAF

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FOREWORD

This report was prepared by the Motor Development Branch, Solid Rocket Division, Air Force Rocket Propulsion Laboratory (AFRPL). The subject test was conducted by Project 305903 AMG, Solid Rocket Hardware, at the request of the Space and Missile System Office (SAMSO), El Segundo, California. The test nozzle was designed and fabricated by the Aerospace Corporation under the direction of SAMSO. The AFRPL Test Engineer was Lt. R.K. Strome, and the Aerospace engineering personnel were Mr. H. Blaes and Mr. W. McDonald. The assistance of Mr. Blaes and Mr. McDonald in preparation of this report is hereby acknowledged. The test was conducted in March 1970.

This report has been reviewed and approved.

CHARLES R. COOKE
Chief, Solid Rocket Division
Air Force Rocket Propulsion Laboratory

ABSTRACT

The test firing of a rocket nozzle and insulated aft closure was conducted at the Air Force Rocket Propulsion Laboratory on a 25-inch diameter uncured propellant solid rocket motor. The nozzle incorporated insulation material at a low area ratio to simulate flow conditions of the Titan III aft closure. Other insulation materials were placed in the test motor aft closure to provide additional information. The insulation utilized was ORCO 9250, an asbestos fiber reinforced rubber. Variations in processing and cure conditions were incorporated to provide information that might explain anomolous surface recessions in some Titan III firings. Based on the test results, it appeared that the material performance did vary with process history, but an incompatible sample configuration prevented direct comparison of test results to the Titan III application. The uncured propellant test motor performed adequately, providing a 14 second test duration with a 620 psig maximum chamber pressure. The propellant was a 16 percent Aluminum, PBAN binder formulation, LPC 614A.

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SECTION I

INTRODUCTION

The first seven-segment Titan III rocket motor test conducted by the United Technology Center exhibited unacceptably high aft closure insulation erosion. Excessively high rubber insulation ablation rates were experienced, and an insulation burn through resulted. The insulation, ORCO 9250, is an asbestos filled nitrile rubber. Components of the composite are normally fabricated by layup of calendered sheets and curing with heat and pressure. Due to the thickness of the Titan closure insulation, severe thermal gradients existed in the component during the cure process. It is believed that these gradients caused the center thickness to be effectively uncured, with the subsequently poor erosion resistance. In order to assess the influence of this "under-cure", a rocket motor test was conducted that utilized rubber components fabricated with various types of curing processes.

The char motor test objective was to qualitatively evaluate the overall differences in performance of cured, partially cured, and essentially uncured specimens of ORCO 9250 in a solid rocket propellant environment. An attempt to obtain meaningful quantitative data was made, with three insulation specimens being installed in the entrance section of the rocket nozzle. The Titan exhaust chemistry was simulated with an aluminized uncured propellant, LPC 614-A.

SECTION II

HARDWARE DESCRIPTION

A. TEST MOTOR CONFIGURATION

The gas generator utilized for the test was the AFRPL 25-inch I.D. char motor. The motor was lined with a 0.5 inch wall thickness paper phenolic insulating sleeve. The sleeve was pressed into the backup insulation, V-44 silica filled rubber. The V-44 provided some structural support for the sleeve. LPC 614-A uncured solid propellant was purchased from Lockheed Propulsion Company for this test. The formulation consisted of 16 percent Aluminium, 70.5 percent ammonium perchlorate oxidizer, and 13.5 percent binder and plasticizer. Detailed ballistic properties are contained in Table I. Nominal flame temperature was 5700°F.

The materials of interest were installed in the aft closure and in the standard nozzle housing of the char motor. The configuration of nozzle materials test hardware is shown in Figure 1. The aft closure was lined with six materials specimens as shown in Figure 2. The closure contour conforms to a 2:1 elliptical surface of major diameter 24 inches. The closure materials specimens, 60 degree sectors of the ellipse, were exposed from area ratio 13.8 down to 9.7. The $\epsilon = 9.7$[*] station was at the junction of the closure specimen and graphite nozzle body. Specimens for testing were prepared 60 degree wedges of elliptical cross section with rubber ply parallel to surface.

The closure segments were molded to a finished outside contour to mate with the existing rubber insulation (V-44). The existing insulation was machined to provide the specimen bonding surface. The molded specimens were dry fit with edge trimming as necessary. Specimens were

---

$$\epsilon \quad \frac{A}{At} \quad \frac{\text{Local Area}}{\text{Throat Area}} = \text{"area ratio"}$$

bonded in place utilizing an epoxy polyamide adhesive with 10 percent chopped glass fibers. In addition each specimen was pinned by means of silica phenolic dowels, 1/8 inch diameter, at each of the four corners.

Test materials were included in the nozzle convergent section as shown in Figure 1. The nozzle test section samples were tag end sections of the closure mounted insulation. Enough material was available to allow three layers of each material to be installed within the graphite nozzle body. However, it was necessary to install material in the nozzle with the original plies normal to the axis of the nozzle. The three slabs of each test material were mounted above one another (in line axially). Figure 3 is a preassembly photograph of the nozzle test samples. Assembly in this manner prevents the performance of a specimen from influencing the behavior of a downstream material with different fabrication history.

The sectors between rubber specimen stacks were filled with blocks of an epoxy-carbon (cast carbon) material consisting of approximately 85 percent epoxy and 15 percent powdered coke.

The entire nozzle insert package was bonded together with Miller Stephenson No. 907, epoxy adhesive, primarily to aid in nozzle assembly.

The entrance section of the graphite nozzle retained the nozzle package. An exploded view of the test nozzle and specimens is shown in Figure 3.

B. MATERIAL PREPARATION

A total of six elliptical sector specimens were molded for inclusion in the aft closure.

The material was from the same batch used in the first Titan III, seven segment motor. The raw material, ORCO 9250, is an asbestos filled nitrile rubber which was utilized in the form of sheets calendered to 0.100 inch thickness. Plies were laid up in an elliptical female mold as shown in Figure 2 for all except specimens 4a. The layup method results in the final five plies terminating at the ablating surface of specimens 1, 2, 3, 4 and 6. This could possible result in plies tearing loose during test due to wall shear and the exposed leading edges. Specimen 4a was laid up in reverse order with the "short" plies positioned first. This resulted in the exposed ablating surface consisting of a continuous ply of rubber composite.

The principal variable explored during the test was the qualititive influence of the extent of composite cure upon ablative performance. The rubber cure conditions are shown in Table II. Specimen temperature during cure was measured by means of two thermocouples, one imbedded at mid-thickness and the other approximately two inches from the circumferential edge. The temperature measured near the small diameter edge, the region of greatest specimen thickness, was utilized for process control. The cure times were governed by temperature indications of this second thermocouple.

SECTION III

TEST RESULTS

A. MOTOR PREPARATION AND PERFORMANCE

The AFRPL supplied nozzle and aft closure were modified by Aerospace Corporation as described in the preceding section. After completion of the modifications, the hardware was returned to AFRPL for the test. The junction of the aft closure insulation and the nozzle entrance section was coated with an annular ring of GenGard V-61 insulation to provide for a smooth contour and to insure an adequate gas seal. The V-61 was then cured at ambient conditions for approximately 24 hours. The paper phenolic insulating sleeve was coated with LPL-22 polymer 72 hours in advance of the scheduled test time. This polymer was also allowed to cure at ambient conditions. The LPL-22 was used to act as a wetting agent compatible with both the uncured propellant and the paper phenolic insulation, thus preventing flame propagation down the propellant/insulation interface. The propellant was air-cast into the test motor by pouring it directly from the shipping container into the center chamber. A delay of 24 hours was provided before the test firing to allow for trapped air to migrate to the surface and be released. Confirmation of this process was evidenced by a 1/4 inch drop in the propellant surface. Three bag igniters containing $BKNO_3$ pellets were suspended six inches above the propellant surface concurrent with the aft closure installation. The lead wires were routed through the nozzle throat. The aft closure retention bolts were torqued to 125 ft-lbs, and the motor was ready for firing.

The motor was fired, and a smoothly regressive 620-psig maximum chamber pressure trace (Figure 4) was recorded. Average chamber pressure was 600 psig over a 14 second effective burn time. A summary of significant motor preparation and performance data is found in Table III, which also contains some hardware performance information. A prediction of the maximum chamber pressure level had been made using data obtained from an earlier set of motor firings. This information is shown in the form of the K_n plot in Figure 5. The correlation of the actual and predicted values indicated the validity of this convenient prediction technique.

B. POST TEST EXAMINATION

The tested hardware was returned for post test evaluation. The tested closure is shown in Figures 6 and 7. Four panels were coated with a thin char layer. Two panels, No. 4 and No. 2, exhibited "blisters" on the exposed surface. The "blister" surfaces were one ply of calendered rubber in thickness. Delamination may have occurred during the test; however, swelling probably did not occur until chamber pressure decay. The char layer on the balance of the specimens was weak and could be removed from the substructure virgin material with ease.

The char layer was removed before post test measurements were made. The surfaces of specimens No. 2 and 4 were measured by slicing through the blister surface and measuring to the underlying ply. The thickness of the blistered ply was then measured and subtracted from the measured recession. The surface mapping measurements were made at the diameter stations shown in Figure 2. The measured recessions are shown in Table IV. A close up of the nozzle is shown in Figure 8. The surface char on the nozzle specimens was also weak. Blistering was not found as in the case of two closure specimens. This was probably due to the fact that the edges of layup plies were exposed rather than the flat surfaces. The extent to which the spacer ablators were decomposed is evident from Figure 9.

The performance of the carbon-epoxy spacers was very disappointing. It had been expected that the recession resistance of the material would be equal to or better than that of the candidate ablatives. A preliminary investigation of the poor performance revealed that the desired percentages of epoxy and carbon filler had been reversed through misinterpretation of an engineering specification. The material in the test, therefore, used only 15 percent carbon, when 85 percent carbon was required. No filler material remained, so confirmation of this hypothesis was impossible.

The post test measurements of nozzle specimens were taken at the locations shown in Figure 1. The post test measurements were taken by measuring char depth on the sides of a groove and the local char surface recession. The measured recessions are shown in Table V. The values shown are the average of the two measurements. There was some minor local variation in recession; however, measured points agreed with the averaged value to within 0.025 inches.

Cross sections of the nozzle test segments are shown in Figures 10, 11, and 12. The heat affected zone is relatively thin.

There is an obvious difference in the thermally affected depth of the various specimens. The char depth is relatively consistent between slabs of the same specimen. The undercured material exhibited the greatest depth of thermal penetration. The standard cure exhibited the least subchar thermal degradation. If this thermally reacting layer is included in the thickness of degraded material as in Table VI, the order of material performance is perturbed. Unfortunately the thermally affected sub char zone is too thin to provide adequate material for analysis of the extent of matrix decomposition. The specimen cured under condition 4 (Table II) exhibited very gummy consistency both before and after heating. A comparison of the top and bottom slabs of Figure 11 with the corresponding slabs of other specimens indicate the compressive set which occurred during test, or possibly cool down. The very limited pretest strain indicates that thermal expansion was the principal cause of deformation. This may also have led to additional radial contraction. There was no evidence of in-depth heating in terms of porosity beneath the measured heat affected zone.

The nozzle exhibited slightly variable surface loss. This was undoubtly influenced by the variation in ablation between the test specimens and the spacer ablators. Because of cure condition and loading during heating and cooling some compression deformation of specimen No. 4 occurred near the ablating surface. The post test throat diameter varied between 1.748 and 1.790 inches. Since the prefire throat diameter was approximately 1.76 inches, the maximum surface regression rate was approximately one mil/sec, and the minimum regression rate indicated deposition on the graphite surface. These measurements are consistent with previous firings of the heavyweight graphite nozzles.

C. INTERPRETATION OF RESULTS

The criteria for interpreting the test results was consideration of the magnitude of surface recession thickness added to the char layer thickness. Surface recession had been determined by measuring from the backside of the material specimens. Char layer thickness was determined by measuring the cross section at the softened, heat-affected zone. The sum of this surface regression and char layer thickness was lower in the standard cure specimen than in any of the other samples, indicating superior performance. If the measurements of the char layer thicknesses were not considered, as would be the case in total removal of the char layer by aerodynamic shear, the standard specimen was inferior in performance compared to the undercured specimens.

Delaminations between plies of the undercured material were observed in the closure section where heating had been perpendicular to the ply layer orientation. These delaminated areas would have been especially susceptible to removal by aerodynamic shear forces, and would have caused decidedly inferior performance as determined by high total surface recession.

In summary, the test results were difficult to interpret because of the uncertain relationship of char layer thickness and total surface regression in the two different ply orientations. The relative ease of removal of the char layer could not be measured, thus further confusing the interpretation. It was believed, based upon the trend of the measurements, relative qualitative appearance of the post fired specimens, and prior experience, that the standard cure specimen was the best performing sample tested.

SECTION IV

CONCLUSIONS AND RECOMMENDATIONS

Based on the results of the test firing, the following conclusions were made:

(1) The test firing failed to conclusively establish the assumed correlation of insulation undercure with poor erosion resistance.

(2) The configuration tested did not closely simulate the conditions seen in the Titan III firings resulting in confusion in interpreting post-fire measurements.

(3) The only valid conclusion produced by the char motor test relates to the strength of the insulation char layer. The undercured insulation definitely produced an easily removed (low resistance to shear) char.

(4) The low strength char would definitely result in a higher erosion rate. The undercured insulation would be subject to higher mass loss since the char would be removed by aerodynamic shear, and would not provide the underlying virgin material with the added protection from the rocket propellant combustion products.

(5) The incompatible orientation of the test specimens (parallel ply lay-up in the Titan III versus perpendicular ply lay-up in the char motor nozzle) further confuses the issue.

Based on the results of the test firing, the following recommendations were made:

(1) To obtain any additional data, further test firings of a more relevant insulation configuration should be conducted.

(2) A single nozzle Minuteman second stage motor would produce a much more reasonable simulation of the Titan III closure insulation and should be utilized as a test vehicle.

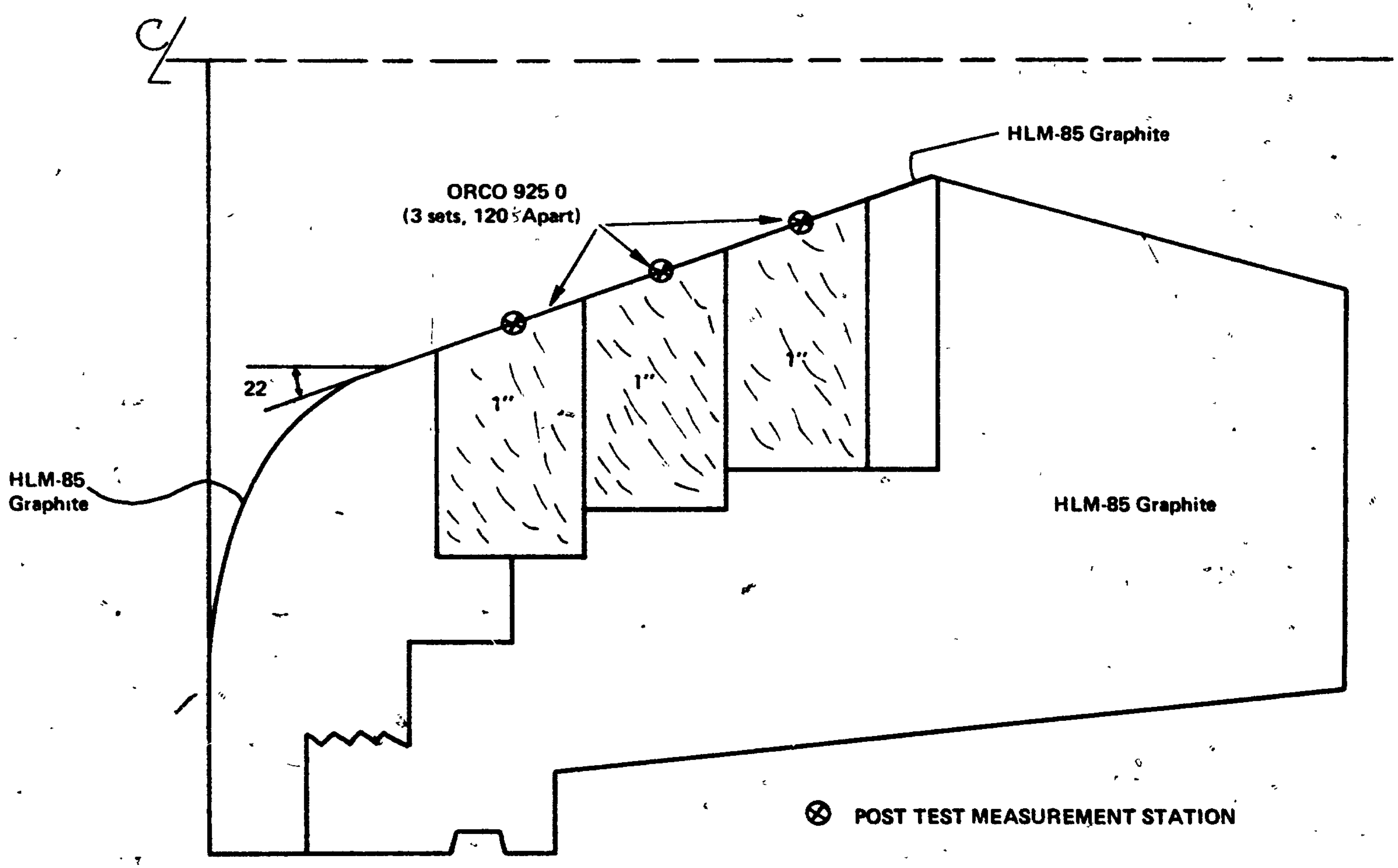

Figure 1. Cross Section of Test Nozzle

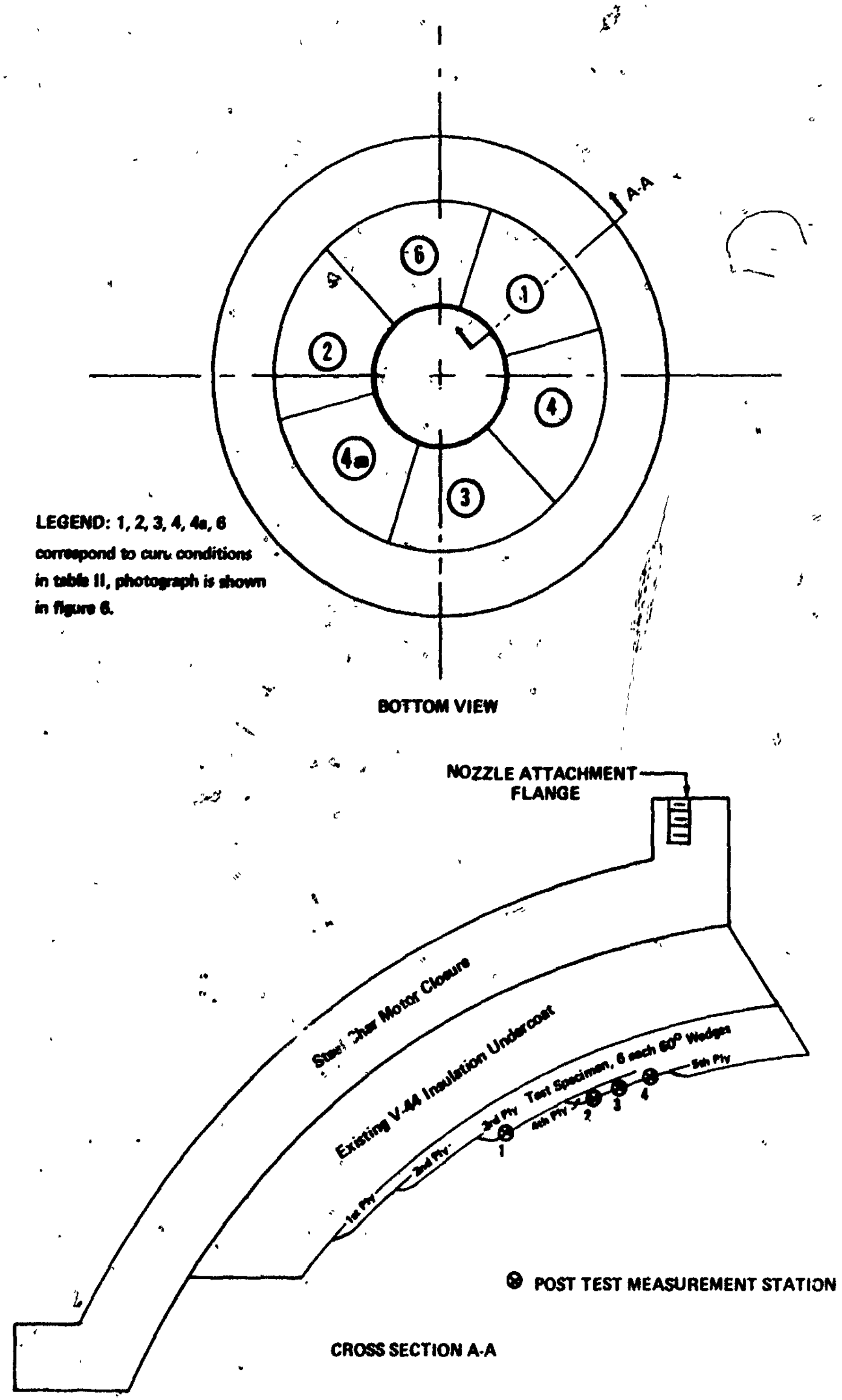

Figure 2. Char Motor Aft Closure Sample Orientation

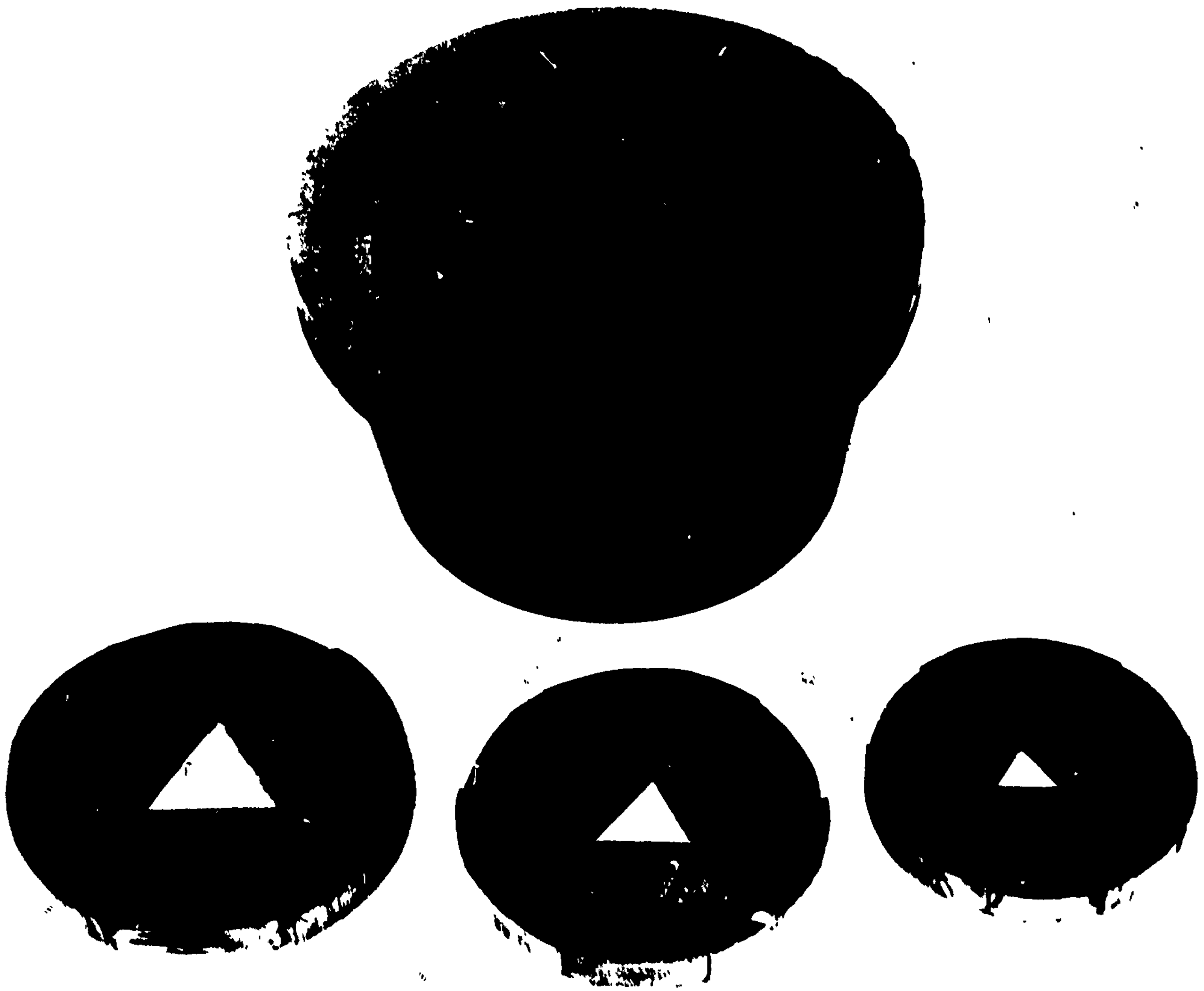

Figure 3. Exploded View of Test Nozzle and Specimens

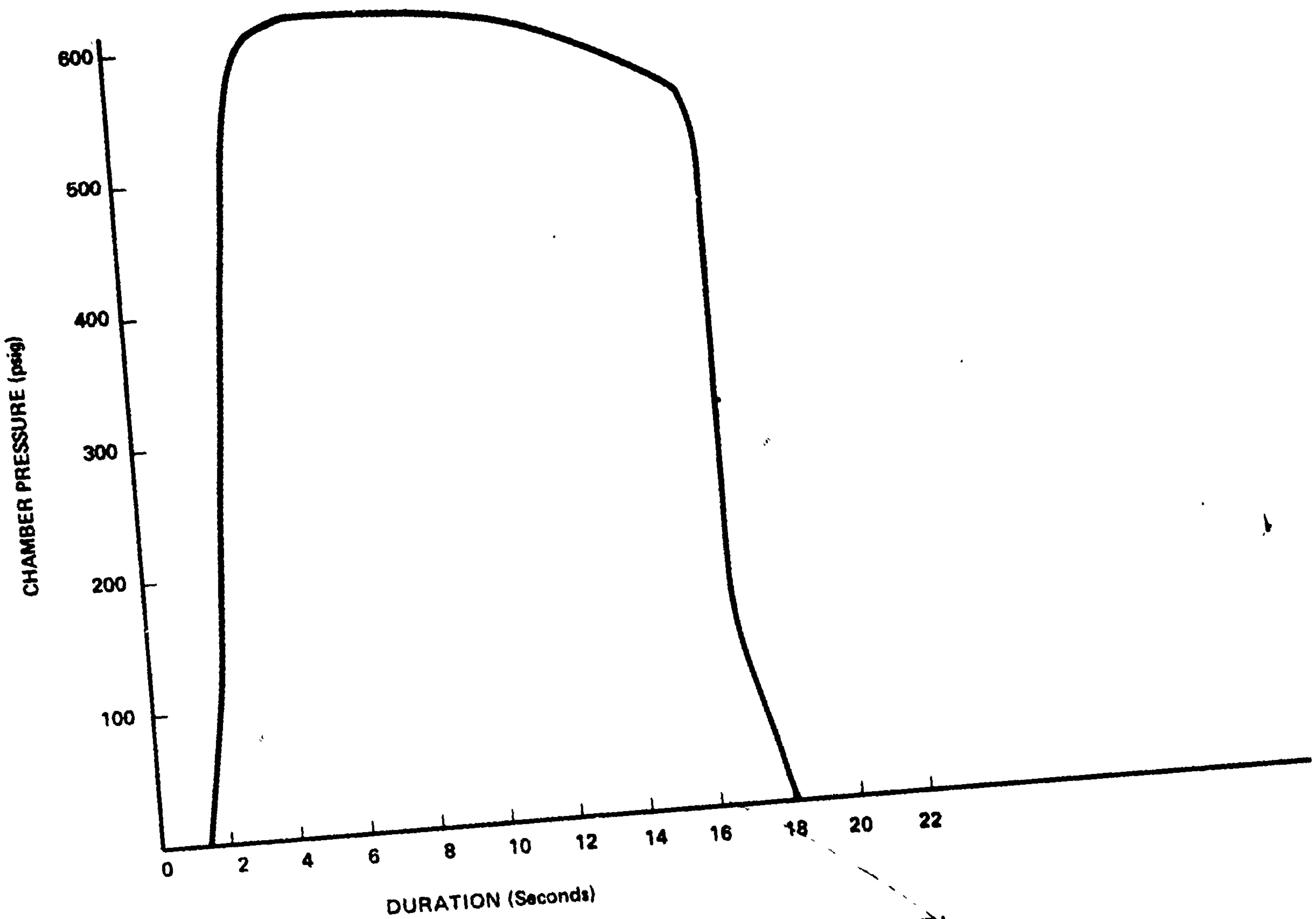

Figure 4. Chamber Pressure versus Time

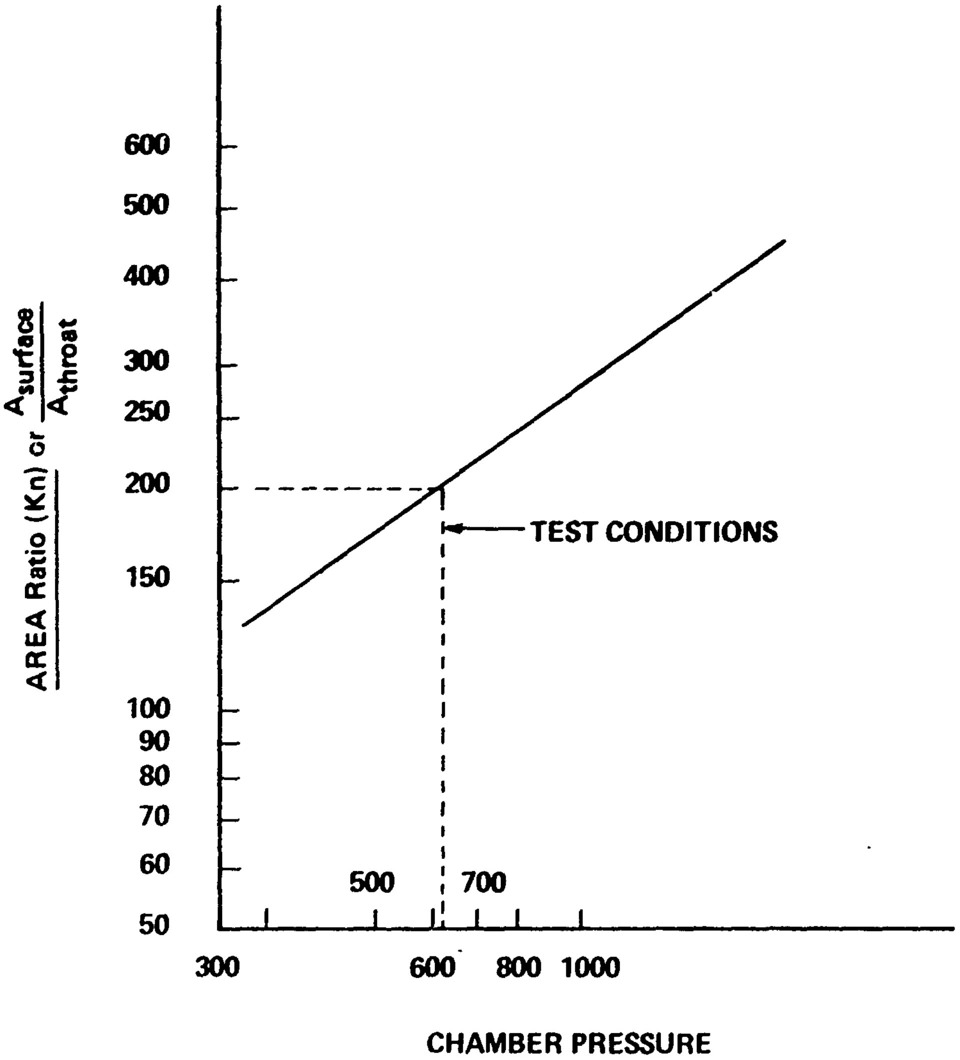

Figure 5. K_n Plot

Figure 6. Post Test View of Aft Closure

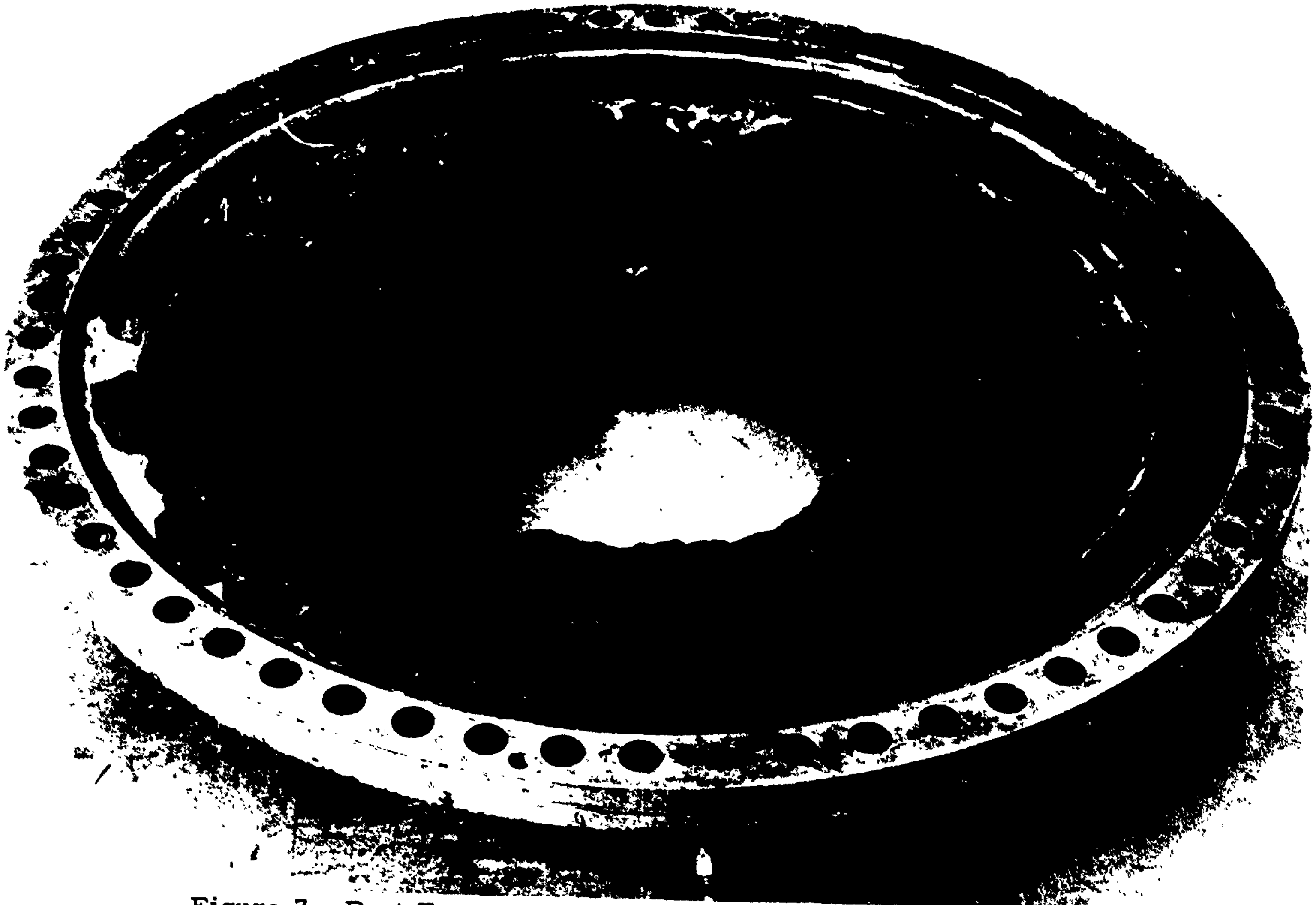

Figure 7. Post Test View of Aft Closure (Before Char Removal)

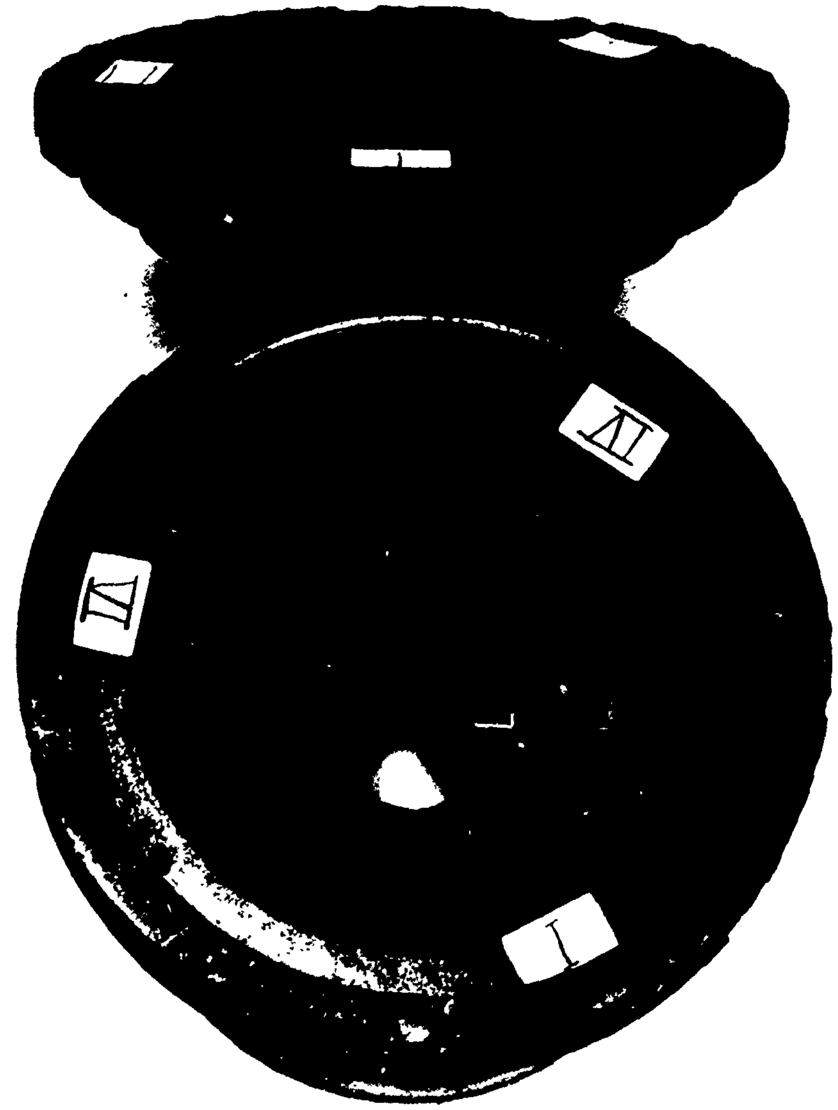

Figure 8. Close-up of Test Nozzle (Disassembled)

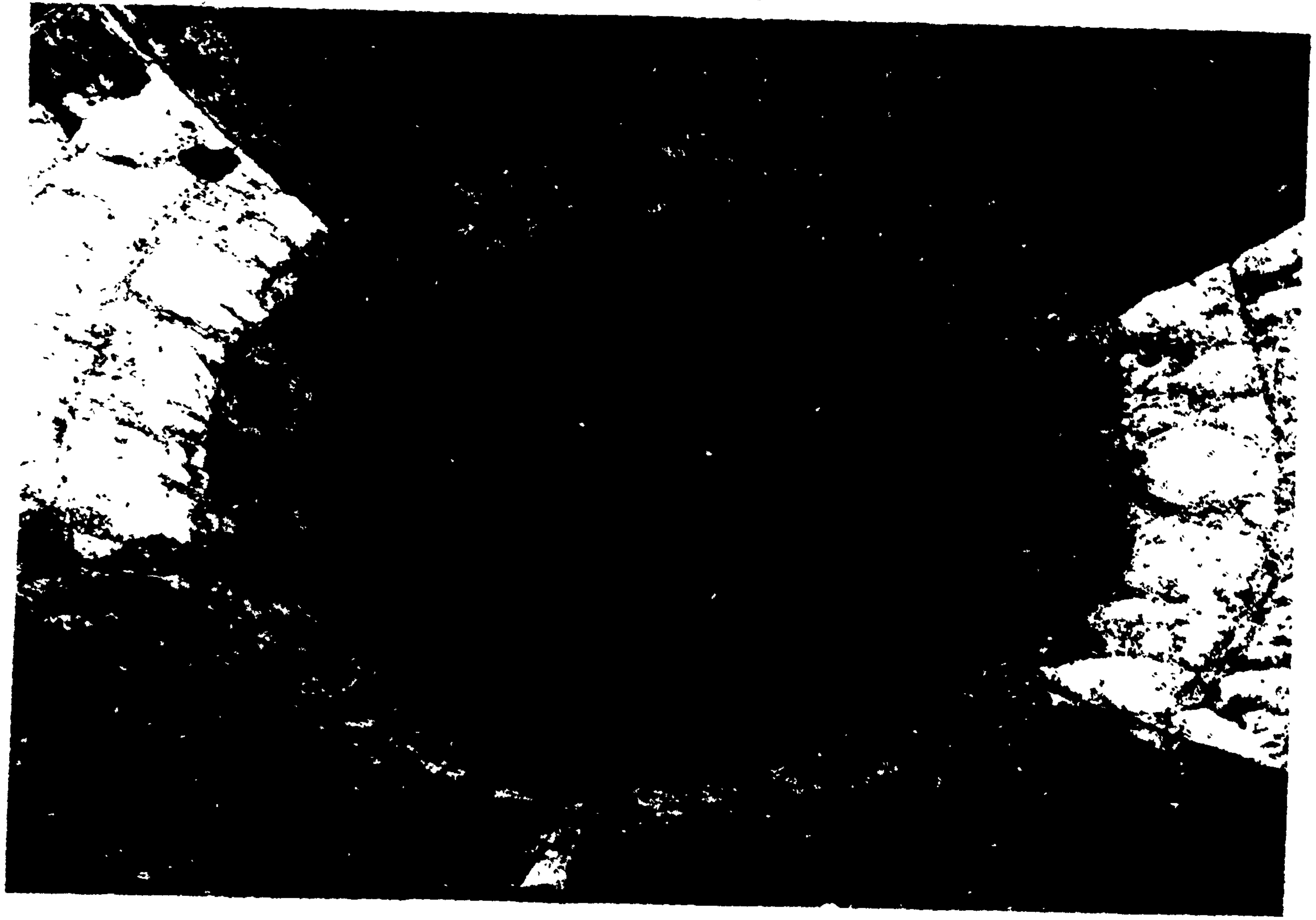

Figure 9. Close-up of Test Nozzle

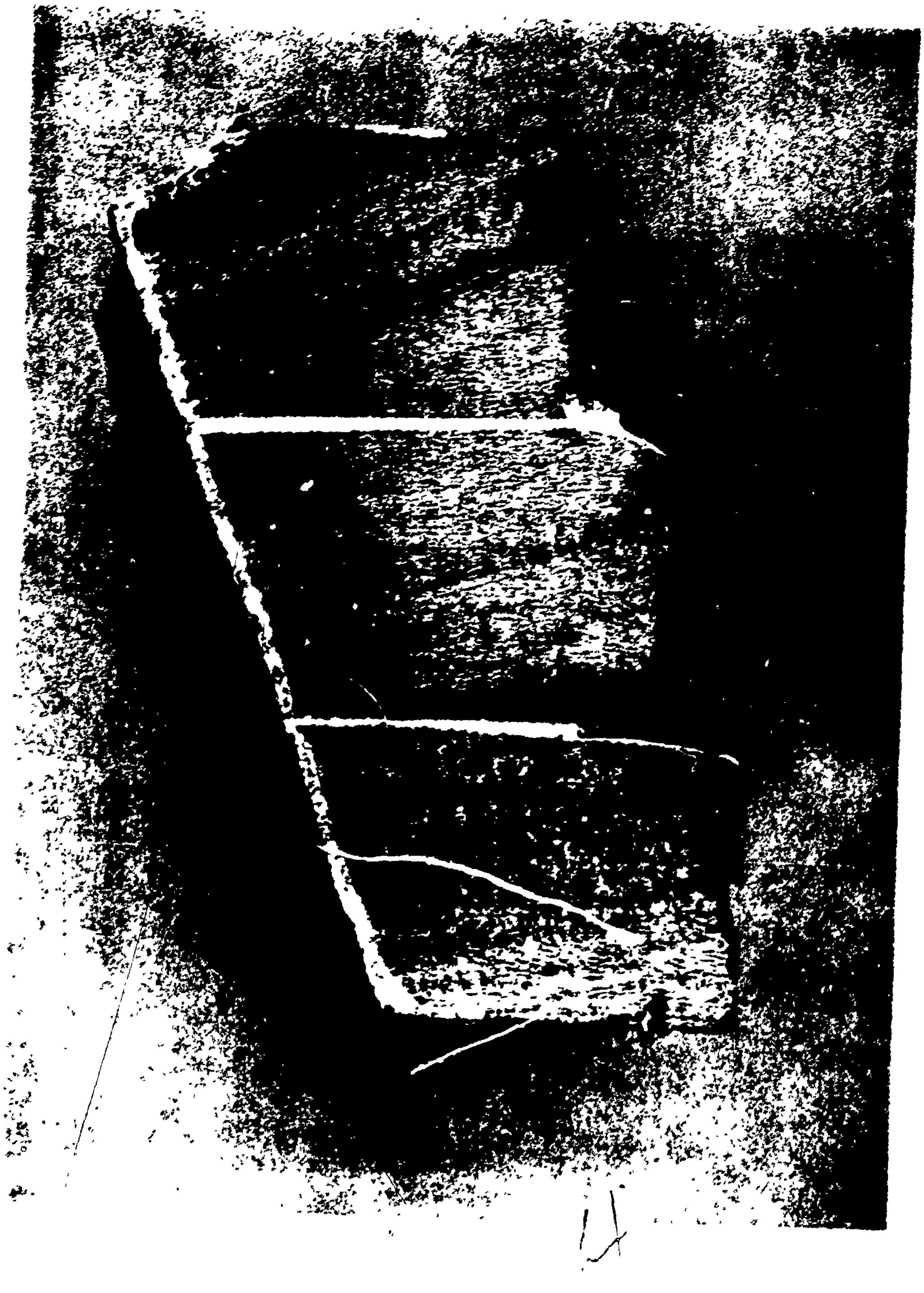

Figure 10. Cross Section of Nozzle Test Section I

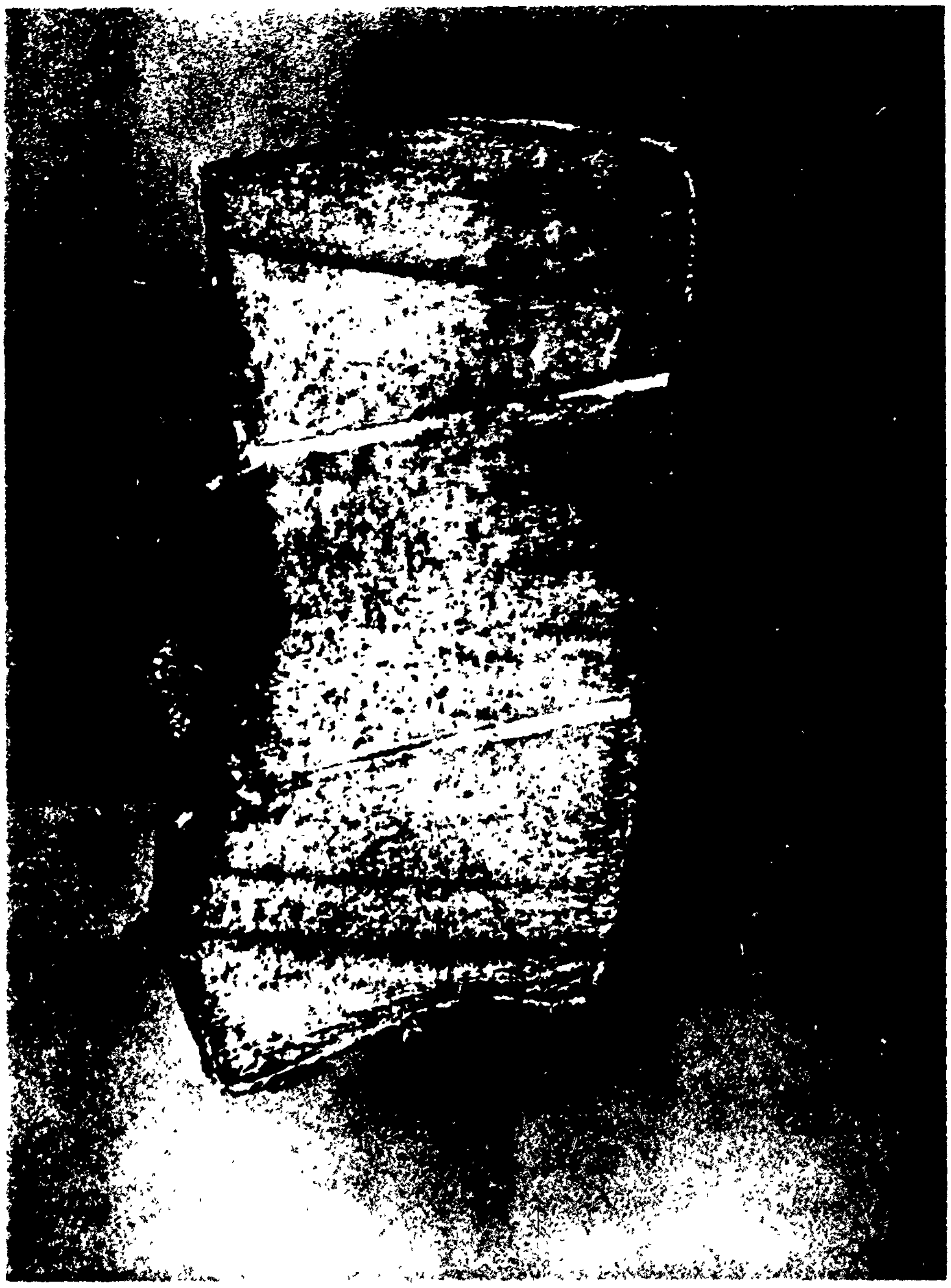

Figure 11. Cross Section of Nozzle Test Specimen IV

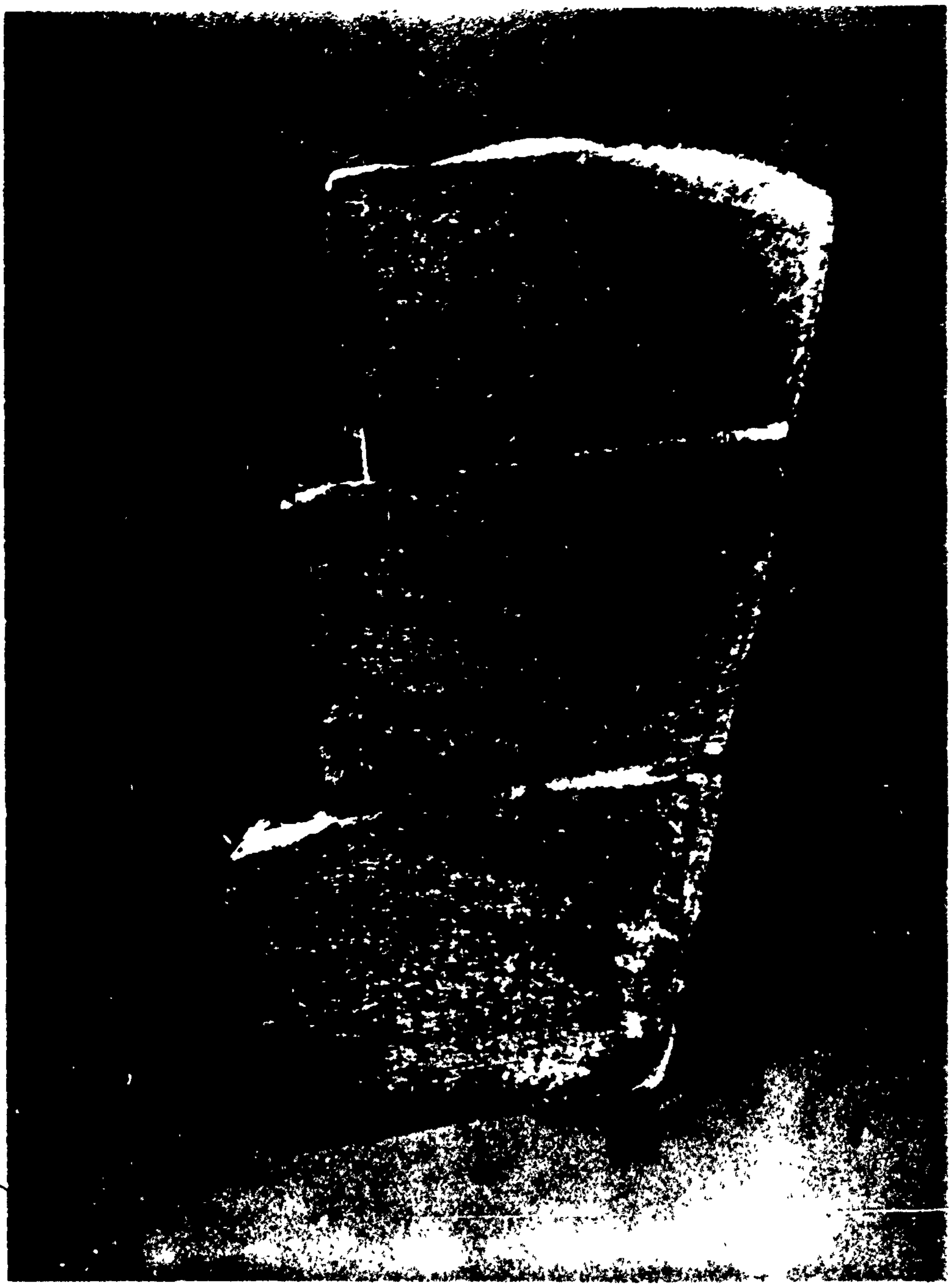

Figure 12. Cross Section of Nozzle Test Specimen VI

TABLE I. PROPELLANT BALLISTIC PROPERTIES

Combustion Products (LPC 614-A)	moles/100gm
CO	0.7846
HCl	0.5145
Cl	0.0446
H_2O	0.6563
H_2	0.9410
N_2	0.3083
Al_2O_3	0.2820

Propellant Composition	percent
Ammonium Perchlorate	70.5
Aluminum	16.0
Binder	13.5

Performance Characteristics	
Nominal r_b	0.30 in @ 800 psig
Nominal n	0.33 @ 800 psig

TABLE II. SPECIMEN CURE CONDITIONS

Spec.	Cure	Remarks
1	5 hr. at 300°F	Overcured
2	1/2 hr. at 300°F	Undercured
3	20 hr. at 250°F	Approximate standard cure
4	8 hr. at 200°F	Undercured, representative of tested motor insulation. normal layup
4a	8 hr. at 200°F	Layup reverse order
5	1 hr. at 205°F + 2 hr. at 300°F	Standard Specified T-3 insul. cure.

TABLE III. TEST DATA

Test Title: Aerospace Insulation

Prefire Throat Diameter	-	1.76 inches
Post Fire Throat Diameter	-	1.79 - 1.75 inches
Propellant Formulation	-	LPC 614-A
As Cast Propellant Depth	-	4 1/2 inches
Pre Fire Propellant Depth	-	4 1/4 inches
Burn Surface Diameter	-	25.0 inches
Propellant weight	-	approximately 125 lbs
Predicted Maximum P_c	-	640 psig
Actual Maximum P_c	-	620 psig
Average P_c	-	600 psig
Effective Duration	-	14 seconds
Ambient Temperature	-	70°F
Ambient Pressure	-	29.4 inches H_g

TABLE IV. SURFACE RECESSION OF CLOSURE SPECIMENS

	Position			
Specimen No.	1 (inches)	2 (inches)	3 (inches)	4 (inches)
1	0.045	0.025	0.064	0.029
2	0.079	0.125	0.098	0.035
3	0.042	0.081	0.068	0.078
4	0.051	0.126	0.125	0.032
5	0.001	0.071	0.021	0.027
6	0.054	0.041	0.023	0.023

TABLE V. SURFACE RECESSION OF NOZZLE SPECIMENS RECESSION (IN INCHES)

	$\epsilon = 5.64$	$\epsilon = 3.97$	$\epsilon = 2.22$
Specimen I	0.216	0.264	0.317
Specimen IV	0.142	0.231	0.332
Specimen VI	0.154	0.260	0.359

TABLE VI. COMBINED SURFACE RECESSION AND DECOMPOSITION THICKNESS OF NOZZLE SPECIMENS

	Total Loss and Reacting Thickness (in.)		
	ϵ = 5.64	ϵ = 3.97	ϵ = 2.22
Specimen I	0.276	0.334	0.387
Specimen IV	0.247	0.336	0.412
Specimen VI	0.184	0.280	0.379

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13. ABSTRACT

The test firing of a rocket nozzle and insulated aft closure was conducted ~~at~~ the Air Force Rocket Propulsion Laboratory on a 25-inch diameter uncured propellant solid rocket motor. The nozzle incorporated insulation material at a low area ratio to simulate flow conditions of the Titan III aft closure. Other insulation materials were placed in the test motor aft closure to provide additional information. The insulation utilized was ~~ORCO 9250~~, an asbestos fiber reinforced rubber. Variations in processing and cure conditions were incorporated to provide information that might explain anomalous surface recessions in some Titan III firings. Based on the test results, it appeared that the material performance did vary with process history, but an incompatible sample configuration prevented direct comparison of test results to the Titan III application. The uncured propellant test motor performed adequately, providing a 14 second test duration with a 620 psig maximum chamber pressure. The propellant was a 16 percent Aluminum, PBAN binder formulation. ~~LPG 614A~~.

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Char Motor						
Ablative Materials						
Titan III Rocket						
Aft Closure Insulations						