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HYBRIDIZED POLYMER MATRIX COMPOSITES

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FOREWORD

This final report describes the work accomplished in NASA Contract NAS3-21383, "Hybridized Polymer Matrix Composites," from August 2, 1978 to November 2, 1979.

The program was sponsored by NASA-Lewis Research Center, Cleveland, Ohio, with Dr. T. T. Serafini as the NASA Project Manager.

Performance of this contract was under the direction of the Material Technology Department of The Boeing Aerospace Company, Seattle, Washington. Mr. W. A. Symonds was the Program Manager and Mr. E. House the Technical Leader. Key personnel contributing to the program and their areas of responsibility are:

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SUMMARY

This report describes the results of a program designed to determine the extent to which graphite fibers are released from resin matrix composites that are exposed to fire and impact conditions. The conditions evaluated were laboratory simulations of those that could exist in the event of an aircraft crash and burn situation. Another objective of the program was to evaluate the effectiveness of various hybridizing concepts in preventing this release of graphite fibers. The baseline (i.e., unhybridized) laminates evaluated were prepared from commercially available graphite/epoxy, graphite/polyimide, and graphite/phenolic materials. Hybridizing concepts investigated included resin fillers, laminate coatings, resin blending, and mechanical interlocking of the graphite reinforcement. The baseline and hybridized laminates' mechanical properties, before and after isothermal and humidity aging, also were compared.

It was found that a small amount of graphite fiber was released from the graphite/epoxy laminates during the burn and impact conditions used in this program. However, the extent to which the fibers were released is not considered a severe enough problem to preclude the use of graphite-reinforced composites in civil aircraft structure. It also was found that several hybrid concepts eliminated this fiber release. Isothermal and humidity aging did not appear to alter the fiber release tendencies.

Under the burn conditions used in the program, no fibers were released from the baseline graphite/phenolic laminates. Next in order of effectivness in fiber retention was the polyimide resin, followed by the epoxy resin.

Hybridizing concepts found effective in preventing fiber release were resin fillers (boron), woven graphite reinforcement, and several coatings including aluminum flame spray, intumescent coating, talc-filled phenolic, and glass microballoon-filled polyimide.

1.0 INTRODUCTION

A potential problem has been identified (Ref. 1) with regard to the accidental release of graphite fibers into the atmosphere. The problem is that electrical equipment could be damaged (short circuit) if contacted by the electrically conductive graphite fibers. As related to composites such as the graphite reinforced epoxies which are finding increased usage in aircraft structure, the following factors add to the concern of fiber release: in case of a fire, the resin matrix of the composite would be degraded with the ensuing potential that the graphite reinforcement could be dispersed into the surroundings; then, once free of their resin matrix, the graphite fibers being of low density and small particle size could readily "float" on air currents with the distance traveled by the fibers before settling to earth being dependent on weather conditions; and further, once the fibers have settled from the atmosphere, they could be redispersed again by other air currents. The objectives of this program were: (1) to determine if a problem exists, and (2) if so, to evaluate concepts of modifying (hybridizing) the graphite reinforced composites to minimize the problem.

This document is the final report on a program performed by the Boeing Company for the National Aeronautics and Space Administration, Lewis Research Center to meet the aforestated objectives. The work was performed under Contract NAS 3-21383.

2.0 TECHNICAL DISCUSSION

The program was performed in two separate tasks. In Task I, "Concept Definition and Analysis," baseline laminates based on commercially available graphite prepreg materials were selected against which the effectiveness of various hybridizing concepts to retain graphite fibers would be evaluated in Task II. Analytical studies were then performed to predict the influence of the hybrid concepts on weight, cost, and mechanical properties of the baseline laminates. From these studies, hybridizing concepts were selected for evaluation in Task II.

In Task II, "Composite Fabrication and Evaluation," laminates were prepared and evaluated using the hybrid concepts selected in Task I. The primary evaluation procedures used were burn and impact testing of the laminates. Physical, mechanical, and NDI testing were also conducted. Evaluations were performed on as-fabricated laminates and also after thermal and humidity aging. Results obtained during this task were used in selecting the hybridizing concepts that retained graphite fibers on burning and impact exposure. The selected concepts were then incorporated into laminates delivered to NASA for evaluation.

2.1 TASK I—CONCEPT DEFINITION AND ANALYSIS

At the onset of Task I, a survey was conducted to determine what type of graphite reinforced composites were most commonly being used or projected for use in civil aircraft structure. This was a limited effort aimed at substantiating already available information that the 449.7K (350°F) curing epoxy resins are currently used more than any other resins. The graphite reinforcement to be used in the program was, by direction, to be unidirectional tape as opposed to woven fabric because: (1) tape is widely used, and (2) woven fabric provides a mechanical interlocking of the graphite reinforcement that would retain the graphite, to some extent, during a fire.

After selecting the baseline laminates, various hybridizing concepts for retaining graphite fibers during, for example, an aircraft crash and burn situation were postulated. The principal areas addressed were potential effectiveness of the hybridizing concept in retaining graphite fibers, and effect of the hybridizations on weight, cost, and performance of the composite. This effort is reported in 2.1.1.

The proposed hybrid concepts were then subjected to analytical studies to predict how the weight, cost, and structural performance of the hybrids would compare to the baseline laminates. These efforts are reported in 2.1.2.

The remainder of Task I was devoted to selecting the most promising hybrid concepts for evaluation in Task II. These efforts are reported in 2.1.3.

A flow diagram of Task I activities is presented in Figure 1.

2.1.1 CONCEPT DEFINITION STUDIES

During this portion of the program, the baseline (unhybridized) graphite reinforced composites were selected for evaluation. Then, hybridizing concepts aimed at retaining the graphite fibers during burning were postulated.

2.1.1.1 Baseline Laminates

A survey was conducted to determine which commercially available graphite prepregs are in use and are most likely to be used on future transport aircraft and space hardware. Evaluations in this program were to be performed on laminates made from these commercially available prepreg materials. Two laminate thicknesses were used: thin, 1.00 to 1.55 mm (0.040 to 0.060 in.), and thick, 6.25 mm (0.250 in.). It was required that unidirectional graphite tape be the reinforcement for the baseline laminates. The survey showed that the predominance of graphite reinforcement presently used are the high strength fibers such as T300, AS, and Celion 6000. Because of their low cost compared to the higher modulus fibers, the high strength fibers should continue to be volume usage leaders in future applications. Another class of fibers that may be used extensively in future applications are the pitch-based fibers. While the development of pitch fibers has been slower than projected, their low cost potential makes them an attractive candidate. One particular form of graphite that is expected to increase in usage and probably poses the most serious fiber release problem is chopped fiber molding compounds. The attractiveness of chopped fiber molding is its low-cost processing, especially on large production runs. Its use appears especially promising as a core in sandwich construction where it stabilizes and provides shear paths between the high modulus skins. Another attractive application is in molded fittings for cost and weight savings reasons. These applications have been

demonstrated on aircraft and missile components and it is likely that chopped fiber molding will be extensively utilized because it is amenable to high production rates processes.

With respect to reinforcement orientation, there are virtually no aircraft or space hardware structures using all 0° orientation. The most common usage is an angle-plied layup with the percentage of fibers running in any one direction being based on the load conditions of that particular application. For example, a laminate containing 25% of +45° fibers, 40% of 0°, 25% of -45°, and 10% of 90° fibers can be considered representative of frequently used angle-plied lay-ups with the percentage and direction of fibers in any one direction being adjusted to suit the application. For this program a pseudo-isotropic orientation (0, ±45, 90) was used.

The survey also indicated that present and near future airplane production will primarily utilize epoxy resin system such as Narmco 5208, Hercules 3501, Fiberite 934, and Hexcel F261 for most applications. The Boeing Company is projecting significant usage of graphite/epoxy composite structures in its new model 767 aircraft. These 449.7K (350°F) curing systems have been evaluated extensively industry wide, under both in-house and government funded programs. Within the epoxy resin family, the 354.1K (250°F) curing materials such as Narmco 5209 and Ferro CE339 were also considered in this program because of their industry usage potential and the probability that they would have different burning characteristics than the 449.7K (350°F) curing resins.

For high temperature applications above 505.2K (450°F), the most promising candidate resin systems are the polyimides. Besides the Space Shuttle weight reduction efforts, other forseeable applications that may develop are exterior surfaces of supersonic missiles and aircraft as well as in hot areas of engines. The missile applications are especially interesting because Boeing tests (Ref. 2) show the polyimides can withstand the peak temperatures above 810.8K (1000°F) for the very short periods of time required in certain missile missions.

In addition to the epoxies and polyimides which are the principal resin candidates for general and high temperature applications respectively, low cost, fast cure/processing resins such as phenolics, polyesters, and thermoplastics were also considered. These materials are ideally suited for economical, high production rate applications.

Based on the foregoing considerations, a list of candidate matrix/reinforcement combinations for use as baseline laminates was prepared (Table I).

Prior to selecting the baseline laminates and hybridizing concepts, some preliminary burn tests were conducted to obtain an indication of the seriousness of the fiber release problem. It was felt that these tests would be beneficial in establishing the severity of the fiber release problem and might provide an indication of which hybridizing concepts would best solve the problem. The laminates were 150 x 150 mm (6 x 6 in.) and of several thicknesses ranging from 0.6 to 3.0 mm (0.024 to 0.120 in.). A listing of the laminates follows:

Laminate #1--polyimide/Celion 6000, $\pm$ 45° orientation
#2--polyimide/chopped fiber
#3--epoxy/T300, 0° orientation
#4--epoxy/fabric
#5--polysulfone/AS, $\pm$ 45° orientation
#6--epoxy/chopped fiber
#7--polysulfone/fabric
#8--polyimide/Celion 6000, 0 $\pm$ 45°
#9--polyimide/Celion 6000, 0 $\pm$ 45°
#10--polyimide/Celion 6000, 0 $\pm$ 45°
#11--polyimide/Celion 6000, 0 $\pm$ 45°
#12--phenolic/fabric
#13--phenolic/fabric
#14--phenolic/fabric
#15--phenolic/fabric
#16--epoxy/chopped fiber
#17--epoxy/chopped fiber

The laminates were mounted, one at a time, in an Ohio State University Rate of Heat Release Apparatus (OSU)--see 2.2.2 and Figure 2--and simultaneously subjected to radiant heat of 5 watts/cm^2 plus direct flame impingement from 12 flamelets, 12.5-mm (0.5-in.) spacing, along the bottom of the laminate. Distance between the radiant panel heater and the specimen was 100 cm (4 in.). Duration of exposure is given in Table II. Air flow across the specimen was about 1 mph. Photographs of the burned-out laminates are presented in Figures 3 through 19 and further particulars are given in Table VI.

Because degradation of the panel due to thermal exposure was the response desired, the specimens were not impacted or subjected to high air velocity. The test results indicated that were was some release of free fibers, but not enough to be considered a serious problem.

A visual examination of the laminates during and after the flammability exposure revealed the following:

1. The structural integrity of all of the laminates was pretty well destroyed except for the thicker polyimide (Panels 2, 10, and 11) and phenolic (Panels 12 and 13) laminates.
2. Graphite fabric does not present a fiber release problem under the flammability conditions used. The laminate degrades into individual plies (Figure 6) with the graphite fibers still interlocked within each ply due to the weaving process.
3. The chopped fiber reinforcement did not present as severe a fiber release problem as expected, possibly due to mechanical interlocking of the fibers. However, the structural integrity of the epoxy matrix panels (#6, 16, and 17) was completely destroyed and free fibers would certainly be released if these panels were subjected to impact and/or high air velocities. Such is not the case with the polyimide matrix laminate (#2). Either the better char forming characteristics and/or higher temperature capability of the polyimide has effectively kept the fibers locked together.
4. Panel #5 (polysulfone/AS at $\pm$ 45° orientation) presented the worst fiber release potential of all laminates tested (Figure 7).
5. It was evident that most panels would experience very little release of individual fibers if they were subjected to an impact loading and air flow such as planned for other panels later in the program.
6. The OSU sample holder provides a certain amount of edge constraint to the laminates during exposure, which helps to retain the graphite in position once the matrix is degraded. Since various extents of damage will occur to aircraft structure during a crash and burn situation, the NASA project manager recommended that flaws be introduced into the laminates by drilling holes prior to burning. This was done to panels #8, 10, 12, 14, and 16. Comparing these panels to identical laminates (Table VI) without holes confirmed that more fiber release occurs from the flawed laminates than from the unflawed laminates. Therefore, it was decided to use flawed panels for the remainder of the program.

7. Matrix char formation appeared to aid in fiber retention in some panels but the extent could not be quantified by visual examination.
8. Visual observations of the laminates during the burn tests indicated that a few fibers, as expected, were released progressively as the matrix was destroyed. However, this release was inhibited by the char formed by the resins. Thus, it seems appropriate to speculate that the total amount of fibers released will not be proportional to laminate thickness and also that resin modifications aimed at producing better char forming epoxies are in order.
9. Panels #12 and 13, phenolic/graphite fabric, exhibited the least fiber release tendency of all the panels tested and Panel #5, polysulfone/$\pm$45° graphite tape, the greatest fiber release tendency.

These preliminary burn tests permitted a ranking to be established relating the matrix resin and reinforcement form to fiber release tendencies. (See Table III.) These tests also showed that very few fibers were released from any of the laminates burned and, at this point in the program, indicated that the fiber release problem would not be severe enough to impede usage of these materials in civil aircraft structure.

After the flammability testing, a section was removed from certain panels, wrapped in fiberglass, and isothermally aged for 12 hours at 866.3K (1100°F) in a muffle furnace. Figure 20 shows a sample prepared for aging and another sample after aging. Under these conditions, the T300 reinforcement from the thinner panels was completely oxidized while the thicker panels, such as in Figure 20, prevented complete oxidation.

Based on these considerations, the baseline laminates listed in Table IV were selected for evaluation in Task II.

2.1.1.2 Hybridizing Concepts

The primary goal of all hybridizing approaches considered was retention of the graphite reinforcement after the laminates had been burned. A secondary consideration was to maximize structural integrity after the exposure. Basic approaches considered are shown in Table V. Of these, it was felt that mechanical interlocking would be the most effective approach for fiber containment. Woven materials are readily available and can be accommodated in designs as either exterior plies or interplied with unidirectional tape. Should this concept prove to be either ineffective or

undesirable for certain applications, mechanical interlocking by weaving a hybrid fiber with the unidirectional graphite is also a possibility. It is appropriate to point out that the use of graphite fabric in lieu of unidirectional tape is increasing for two reasons: (1) cost savings due to reduced lay up time, and (2) less data scatter. The many applications that require crossplied fibers with 0^{o} and 90^{o} orientation are made-to-order for fabric. Thus, the interplying of graphite fabric with tape was projected to be a very effective method for resolving the fiber release problem with minimum weight increase and minimal property reduction for many applications.

Coatings were another minimum impact approach that was considered. It was felt that polyimide and other organic coatings that are good char formers on burning would provide fiber containment during a fire. Also considered were the intumescent coatings which expand and foam to prevent the underlying composite structure from burning. Tests at Boeing had shown that these are relatively effective. Ablative coatings and glass flakes with a low melt temperature were also considered.

Several approaches aimed at controlling the matrix constituents to effect fiber containment were also considered. The first was to determine if there were any commercially available matrix systems that would retain the graphite fibers. The second involved prepreg plies of one resin matrix interplied with prepreg of a different matrix. The third approach considered was to modify the matrix to increase its char forming characteristics and/or its ability to make the reinforcement disperse as clumps rather than individual fibers after a fire. It was recognized that the second and third approaches would impact material users because a new data base would be required.

A merit rating system was used to provide a quantitative method for guiding the initial selection of material/layup combinations for the candidate hybrid materials and for ranking them to define the most promising combination. An existing computer program was used to quickly analyze selected material candidates and ply orientation combinations for parameters such as tension and compression load efficiency (strength and weight), stiffness, and fatigue resistance (see 2.1.2).

A preliminary selection of hybridizing concepts was compiled and is presented in Table VI. Kevlar reinforcement was purposely omitted from the proposed hybrid concepts because work performed at NASA-Langley (Ref. 3) indicated that Kevlar used as

exterior plies on a graphite/Kevlar laminate were not effective in retaining the graphite fibers.

After the analytical studies were performed (2.1.2), the hybridizing concepts for evaluation in Task II were selected (see Table VII).

2.1.2 COMPOSITE ANALYTICAL STUDIES

The purpose of these studies was to predict how various hybrid concepts would affect the cost, weight, and structural performance of the baseline laminates. The basic approach used was to assume load and stiffness requirements commensurate with the structural capability of the baseline laminates. The hybridizing concepts were required to meet these same requirements, i.e., no reduction in load carrying capability was permitted. While other approaches could be used, this one appears most appropriate because it assumes that the baseline laminates were sized to carry a certain load and that load must also be carried by the hybrid laminates.

The technique used to predict the effect of various hybridizing concepts on the structural, weight, and cost properties of baseline laminates was the Boeing Composite Analysis and Optimization Computer Program "COOP" (Ref. 4). "COOP" uses classical laminated plate theory to calculate the moduli and allowable strengths of a laminate using the moduli and strengths of each layer in the laminate as input. The program then optimizes the laminate to obtain a minimum objective function subject to strength and stiffness constraints. The objective function used in the optimization is a composite functional consisting of the laminate weight, cost, moduli, and strengths. The objective function is given by:

$$OBJ = \frac{WT}{C_1} k_1 + \frac{COST}{C_2} k_2 + \frac{C_3}{E_x} k_3 + \frac{C_4}{E_y} k_4 + \frac{C_5}{G_{xy}} k_5 + \frac{C_6}{F_{xt}} k_6$$

$$+ \frac{C_7}{F_{yt}} k_7 + \frac{C_8}{F_{xc}} k_8 + \frac{C_9}{F_{yc}} k_9 + \frac{C_{10}}{F_s} k_{10}$$

where:

$$C_1 = \left(\frac{N_x}{1172.4 \text{ MPa } (170{,}000 \text{ psi})} + \frac{N_y}{1172.4 \text{ MPa } (170{,}000 \text{ psi})} + \frac{N_{xy}}{413.8 \text{ MPa } (60{,}000 \text{ psi})} \right) 0.06 \text{ (lb/in.}^2\text{)}$$

$C_2 = 50C_1$ ($/in^2)

$C_3 = C_4 = 68{,}965.5$ MPa (10×10^6 psi)

$C_5 = 20{,}689.7$ MPa (3×10^6 psi)

$C_6 = C_7 = 517.2$ MPa (75,000 psi)

$C_8 = C_9 = -517.2$ MPa (-75,000 psi)

$C_{10} = 241.4$ MPa (3500 psi)

Where C_1 is the weight of an equivalent T300 graphite/epoxy laminate subjected to the same loads, C_2 is the cost of the equivalent laminate, C_3, C_4, and C_5 are the moduli, and C_6 - C_{10} are the strengths. The weighting factors K are varied to give the desired objective function. For example, if $K_1 = 1$ and $K_2 - K_{10} = 0$, the laminate would be optimized to provide a minimum weight design. If $K_1 = 1$, $K_2 = 1$, $K_3 - K_{10} = 0$, the laminate would be optimized to provide a combination of minimum weight and minimum cost.

The optimization method used is a variation of the steepest descent-sidestep nonlinear programming method (Ref. 5). The optimization method is illustrated using the two dimensional example given in Figure 21. This example represents a 0/90 laminate being optimized subject to two constraints. Where constraint 1 is the margin of safety in the 90° layer and constraint 2 is the margin of safety in the 0° layer. For a given set of applied loads additional constraints could be present, such as minimum thickness and minimum stiffness. The purpose of the optimization is to reach the design with the smallest objective function value while remaining above the constraint curves (in the feasible region). Starting at Point 1, the code reduces thicknesses (descends) to reach the constraint curve at Point 2. A direction is then determined which will

provide the maximum increase in the constraint while maintaining a constant objective value (sidestep direction). The code moves in the sidestep direction to Point 3 and checks to see if the constraint is increasing. It continues sidestepping to Point 6, where the constraint is found to have decreased from the value at Point 5. It returns to Point 5 and descends to the constraint curve at Point 7. The sequence of steps 2-7 is then repeated until the minimum value of the objective function is found.

Eight different laminates were optimized with the "COOP" code and ranked according to their objective function values, as shown in Figure 22. The optimization factors of "K" factors given in the figure were chosen to provide laminates with low weight and cost and high moduli and strength. The loads N_x, N_y, and N_{xy} are representative of a 0/$\pm$45/90 T300/5208 laminate with a thickness of 1.016 mm (0.040 in.). These were applied to the hybrid laminates to provide strength constraints so that the hybrids would not have a lower load capability than the baseline 0/+45/90 laminate. Stiffness constraints were also applied so that the hybrids would not be less stiff than the baseline laminate.

The required thickness in each layer of each laminate are shown on the right side of the figure. It can be seen that the program removed the 0° and 90° graphite/epoxy tape layers when a 0/90 T300 fabric hybrid was used and that it removed the $\pm$45° tape layers when a $\pm$45° fabric hybrid was used. The 0/$\pm$45/90 tape laminate provide the lowest objective function value, followed by a 0/90 T300 fabric/$\pm$45 T300 tape hybrid and a 0/90 T300 tape/$\pm$45 T300 fabric hybrid.

The cost data used in these analyses were material costs. If production costs are included, those hybrids incorporating fabric reinforcement would cost less than the baseline tape laminate because fabric is easier to lay-up than tape. For the (0/$\pm$45/90) orientation under consideration, an all-fabric laminate could be used and would be the optimum laminate.

2.1.3 LAMINATE SELECTION

Based on the studies described in 2.1.1 and 2.1.2, the baseline and hybrid laminate systems listed in Table VII were selected for evaluation in Task II.

2.2 TASK II--COMPOSITE FABRICATION AND EVALUATION

The objectives of Task II were: (1) to prepare baseline and hybrid laminates selected in Task I, (2) to evaluate the effectiveness of the hybrids to retain graphite reinforcements during burn and impact testing, and (3) to determine the effect of the hybridizing concepts on laminate physical and mechanical properties. The effects of thermal and humidity conditioning on laminate physical and mechanical properties were also included in this study. The laminate fabrication efforts are documented in 2.2.1 and the evaluation of these laminates reported in 2.2.2. Based on these studies, the hybrid concepts that best retain graphite fibers during burn and impact conditions were selected as described in 2.2.3. A flow diagram of Task II efforts is presented in Figure 23.

2.2.1 LAMINATE FABRICATION

In this section, the fabrication procedures used to produce the baseline and hybrid laminates listed in Table VII will be described. These laminates will be referred to by their system numbers shown in Table VII. All laminates were autoclave cured except for systems 3, 3a, and 3b which were cured in a Pasadena Hydraulic Platen Press. Hand lay-up techniques were used. The basic orientation was pseudo-isotropic ($0/\pm45/90$) with 8 plies total being used in systems 1, 4, and 5 and 48 plies used in system 2. Particulars on each system follow:

- System #1--T300/5208 tape $(0/\pm45/90)_S$, autoclave bagging procedure and cure cycle are shown in Figures 24 and 25, respectively.
- System #1a--Same as system #1 except one ply of 120 fiberglass/epoxy with warp in the 0-degree direction cocured to each face.
- System #1b--Same as system #1 except aluminum flame sprayed film, 0.127 mm (0.005 in.) thick, cocured to one face of the laminate.
- System #1c--Same as system #1 except hand stitched with 32 end E glass in a 25.4-mm (1-in.) square pattern prior to cure.
- System #2--T300/5208 tape, $(0/\pm45/90_2/\pm45/0)_{3S}$ Bagged and autoclave cured same as system #1.
- System #2a--Same as system #2 except 42 plies of T300/5208 woven fabric (3K-70-CSW) at $(0/45)_{10}$ 0_S orientation was used.

- System #2b--T300/5208 tape $(0/\pm45/90_2/+45/0_2/\pm45/90_2/+45/0/\pm45/90_2/\pm45)_S$ with one ply of HMFW-70.5 (Fiberite) hybrid fabric, graphite (90%)/S glass (10%), cocured to each face. Prior to lay-up the hybrid fabric was impregnated with boron filled (10%) epoxy resin. The hybrid fabric is of plain weave construction with the T300 fibers all in the warp direction and the S glass being fill yarns. The orientation of the hybrid fabric was 90°.
- System 2c--Same as system #2 except 50 grams of phenolic resin, F508 (USP), was coated onto each side of the 533.4 x 381.0 mm (21 x 15 in.) laminate prior to cure. After application, the phenolic resin was staged at approximately 316.3K (150°F) for 2 hours to reduce surface tack and promote intermixing of the phenolic resin with the epoxy matrix resin of the T300/5208 prepreg.
- System 2d--T300/5208 tape $(0/\pm45/90_2/+45/0_2/\pm45/90_2/+45/0_2/90_2/0/\pm45/-45_2/+45)_S$ Boron powder was added to the outermost four plies of each face during lay-up as follows: the prepreg was wiped with an acetone dampened cloth to improve surface tack and the previously sifted boron powder was then spread evenly over the prepreg face of each ply. The weight ratio of boron to each ply of prepreg treated was 7%. Bagging and autoclave cure was the same as for system #2.
- System 2e--Same as system #2 except intumescent coating applied to one face after cure. Intumescent coating was Flamarest 1600B (AVCO Specialty Materials Division) applied per the manufacturers instructions to achieve a 0.381-mm (0.015-in.) dry film thickness.
- System 2f--T300 fabric (13K-135-8H)/5208, 18 plies at $(+45/0/-45/0)_2+45)_S$ orientation plus one ply of T300 fabric (13K-135-8H)/F805 phenolic at 0° orientation cocured to each face. Bagged and cured same as system #2.
- System 2g--T300/5208 tape and T300 fabric (3K-135-8H)/5208 interplied as follows: (Fabric 0/fabric 0/$\pm$45 tape$_2$/fabric 0/$\pm$45 tape$_2$/fabric 0/$\pm$45 tape$_2$/fabric 0)$_S$. Bagged and cured same as system #2.
- System 2h--Six plies of hybrid fabric (HMF W-705)/5208 cocured to each face of a T300/5208 tape "core": 0_3fabric/90_3fabric/$+45/(+45_2)_3$ $-45_2/+45$ $_S$. The W-705 fabric is composed of T300 graphite in the warp direction and S glass in the fill direction at a ratio of 90% graphite to 10% S glass. Bagged and cured same as system #2.
- System #3--T300 chopped fibers, 12.7 mm (0.50 in.)/EM 7125 epoxy resin by USP. Press molded at 5.5 MPa (800 psi) and 499.7K (350°F) for 1 hour.

- System 3a—Same as system #3 except graphite/epoxy mat (HY-MAT 7534 by Fiberite) cocured to form outer facings to the EM 7125 chopped fiber molding compound. Weight percentages were: 18% HY-MAT 7534 in each face and 64% EM 7125 core. Fibers in the mat material were approximately 28.6 mm (1.125 in.) long and of random orientation with a higher degree of interlocking.
- System 3b—Same as system #3 except T300 chopped fiber, 2.54 mm (1 in.)/phenolic (HY-E-1008E by Fiberite) cocured to form outer facings to the EM 7125 chopped fiber molding compound. Weight percentages were: 20% HY-E-1008E in each face and 60% EM 7125 core. Press cure cycle was 5.5 MPa (800 psi) and 477.4K (400°F) for 1 hour.
- System #4—Celion 6000/PMR-15 polyimide tape $(0/\pm45/90)_S$, autoclave bagging procedure, and cure cycle are shown in Figures 26 and 27, respectively.
- System 4a—Same as system #4 except one ply of 104 style fiberglass impregnated with NR150 B-2 polyimide resin filled with 20 weight percent glass microballons (Eccospheres "R") cocured to each face.
- System #5—T300/HY-E-1008E phenolic resin tape (Fiberite), $(0/\pm45/90)_S$. Autoclave cure: full vacuum plus 0.69 MPa (100 psi) throughout cure, heat rise rate 256.3-256.9K (2-3°F)/minute, cure at 449.7-463.6K (350-375°F) for 1 hour, cool to 338.6K (150°F) under pressure. Used two plies of 120 style fiberglass for top bleed.
- System #5a—Same as system #5 except talc filled phenolic resin coating applied to each face of the laminate prior to cure. Phenolic resin used for the coating was F508 by USP and the talc was product #8476 from Mallinckrodt Company, St. Louis, Mo. The talc was mixed into the phenolic resin at a talc to resin weight ratio of 31 to 69. Fifty grams of the talc filled resin was applied to each face of the 381 x 533.4 mm (15 x 21 in.) lay-up and staged for 2 hours at 321.9K (120°F) prior to cure.

2.2.2 LAMINATE EVALUATION

Once the baseline and hybrid laminates had been fabricated, they were evaluated for physical, mechanical, and fiber retention characteristics on burning as shown by the test matrix of Table VIII. First the cured laminates were ultrasonically "C" scanned and their specific gravity, fiber volume, and void content determined to ensure that the laminates were of high quality. The laminates were then machined into specimens and subjected to mechanical properties and flammability testing in the as-fabricated

condition and after thermal and humidity aging as shown in Table VIII. The answers to two basic questions were being sought by these evaluations: (1) to what extent would the graphite fibers be released from the baseline laminates and how effective would the hybridizing concepts be in preventing this fiber release and (2) would the hybridizing concepts adversely affect the thermal and humidity stability of the baseline laminates. Described in this section are the flammability testing (2.2.2.1) and the physical and mechanical properties evaluations (2.2.2.2) performed to answer these two questions.

2.2.2.1 Flammability Testing

As shown in Table VIII, three flammability test procedures were used during the program. They were: Limiting Oxygen Index (LOI), ASTM D-2863; Flame Spread Index (FS), ASTM E-162; and The Ohio State University Rate of Heat Release Determination (OSU). The OSU instrument (Figures 2, 28, and 29) and its operation are described in a proposed ASTM procedure (Reference 6). Burning of laminates in the OSU apparatus and then subjecting them to impact loading and high air velocities was the primary investigative procedure used to determine fiber release tendencies. Thus, discussions in this section will concentrate mainly on OSU burn results. The LOI and FS procedures were used to obtain comparative burn characteristics of baseline epoxy, polyimide, and phenolic matrix systems (#1, 4, and 5 of Table VII).

There are no established requirements for aircraft materials meeting any specific flame spread index or limiting oxygen index. Currently, aircraft materials need only meet FAR Part 25 flammability requirements. The FAA is considering imposing further flammability requirements on materials in the future to be used for new generation aircraft. However, the new requirements have not, as yet, been formulated.

The LOI procedure establishes the minimum oxygen concentration that will support candle-like combustion. This test method consists of a procedure for measuring the minimum concentration of oxygen, expressed as volume percent, in a flowing mixture of oxygen and nitrogen that will just support flaming combustion of a material initially at room temperature. This oxygen concentration is termed the limiting oxygen index (LOI). High values of LOI are desirable. As shown in Table IX, the Celion 6000/PMR-15 polyimide test results were far superior to those for the epoxy and phenolic systems.

Surface flammability (FS) was determined by the procedure described in ASTM E-162. This method employs a radiant heat source consisting of a 304 x 457.2 mm (12 x 12 in.) panel in front of which an inclined 152.4 x 457.2 mm (6 x 18 in.) specimen of the material to be tested is placed. The orientation of the specimen is such that ignition is forced near its upper edge and the flame front progresses downwards. A factor derived from the rate of progress of the flame front (ignition properties) and another relating to the rate of heat liberation by the material under tests were combined to provide a flame spread index (I_S). Low values of I_S are desirable. As shown in Table X, the polyimide and phenolic systems are superior to the epoxy system when tested by this procedure.

For familiarization purposes, several factors pertinent to the OSU instrument and its operation as used during this program will now be described. The OSU apparatus was used to provide a controlled environment in which baseline and hybrid laminates of Table VII were burned and the release of graphite fibers therefrom visually observed. Two heat sources were used: (1) radiant heat from silicon carbide "glow bars" mounted in the radiation panel (see Figure 28) and (2) direct flame impingement from 12 natural gas pilot flamelets at 12.7 mm (0.5 in.) spacing along the bottom of the 152.4 x 152.4 mm (6 x 6 in.) laminates. These heat sources provided a constant heat flux of 12 watts/cm^2 which caused laminate ignition and violent burning within 5 to 10 seconds after its insertion into the OSU burn chamber. The laminates were exposed, one at a time, by mounting them in a sample holder (Figure 29) and then inserting them into the burn chamber (Figure 2). Referring again to Figure 28, the "radiation door" is opened and the "sample holder" moved into that position during burning. This places the laminate 4 inches from the "glow bars." A total air flow of 85 cubic feet/minute, which translates to 1 mph, is maintained through the burn chamber. The thicker laminates, 6.35 mm (0.250 in.), were exposed for 15 minutes and the thinner, 1.016 to 1.524 mm (0.040 to 0.060 in.), ones for 10 minutes. These durations permitted the temperature gradient from the front (exposed) face to the rear face of the laminate to stabilize. Determining these temperatures was accomplished by exposing a 6.35 mm (0.250 in.) thick graphite/epoxy laminate with thermocouples embedded at various depths within the laminate. After 4 minutes, laminate temperatures were 1066.3K, 845.2K, and 752.4K (1460, 1062, and 895°F) and after 11 minutes 1116.3K, 994.1K, and 958K (1550, 1330, and 1265°F) with the higher readings being those recorded by the thermocouple at a depth of 0.66 mm (0.026 in.) from the face of the laminate nearest the glow bars. Although the duration of exposure was 14 minutes, the temperatures

appeared to have stabilized after 8 to 10 minutes. These temperatures were attained using a thermal environment of 10 watts/cm^2. It is probable that slightly higher temperatures were attained during burning of the baseline and hybrid laminates since they were exposed to a higher heat flux: 12 watts/cm^2.

The OSU apparatus also monitors and records heat and smoke evolution with respect to laminate exposure time. While this information was not too applicable as related to fiber release tendencies, it did confirm that: (1) phenolic and polyimide matrix composites evolve significantly less smoke on burning than epoxies and, (2) the hybridizing concepts did not appreciably alter the smoke and heat evolution properties of the baseline laminates.

An observation port in the OSU apparatus permitted unobstructed visibility of the laminates during burning and the glow bars provided good illumination of the laminates. Thus, soot and fiber release were easy to detect. The low velocity air flow through the burn chamber removed the smoke as it was generated and also caused any fibers that were being released to gently float off to the exhaust port. Photographs of laminates for each baseline and hybrid system (Table VII) before and after OSU exposure are presented in Figures 30 through 50. As shown in these figures, the laminates had been purposely flawed with 6.35 mm (0.25 in.) diameter holes prior to exposure to facilitate fiber release. It was felt that such flaws were needed to make the laminates more representative of aircraft structure that had been damaged in a crash/burn situation. Observations recorded during the OSU exposures are presented in Table XI and summary comments follow:

1. All systems burst into flame within 10 seconds of exposure with copious amount of smoke and soot evolved for approximately one minute for the 1.016 to 1.524 mm (0.040 to 0.060 in.) thick laminates and up to 5 minutes for the 6.35 mm (0.250 in.) thick laminates. Of the systems evaluated, the epoxies evolved the most smoke with the phenolic and polyimide systems evolving about the same amount but significantly less than the epoxies.
2. There was very little fiber release from any of the laminates. Baseline systems #1 and #2 (Table VII) released the most fibers with fibers from the outermost two plies being the most fibers released during any exposure.
3. There was essentially zero fiber release from the phenolic and polyimide matrix systems.

4. The epoxy systems, which did release fibers, did so in two different ways: (a) individual fibers would float away on air currents passing through the burn chamber, and (b) clumps of fibers and resin remnants would break off of the laminate and fall to the bottom of the OSU burn chamber.
5. Individual fibers were not released from graphite fabric because of the interlocking of the fibers by the fabric weave. In some cases, chunks of burned out fabric would be dislodged from the laminate and drop to the bottom of the burn chamber.
6. Prior thermal and/or humidity aging of the laminates (Table VIII) had no observable effect on the amount or method of fiber release.
7. The baseline phenolic matrix laminates (system #4 of Table VII) does not appear to need any hybridization to prevent fiber release.
8. The hybridizing concepts listed in Table VII that were most effective in preventing fiber release are 1b, 2a, 2b, 2c, 2d, 2e, 2f, 2g, 2h, 4a, and 5a.

After the OSU exposure, the burned out panels were subjected to an impact loading of 5 foot pounds followed by 35 mph flow of air across the laminate face for 5 minutes duration. This impact/air flow test was performed in the test chamber shown in Figures 51 through 53. Portions of the laminates that were dispersed by this testing were collected on sticky adhesive coated film positioned on the floor of the impact chamber. In this manner, a permanent record (sticky film with remnants from tested panel) was made for each panel tested. A picture of remnants from each baseline and hybrid system after being subjected to the impact and air flow exposure are presented in Figures 54 through 74. Note that most of the laminates were dispersed in clumps or pieces that were too large to present a fiber float problem. After studying the laminate remnants, it appears that the best hybridizing concepts listed in Table VII were: 2a, 2b, 2d, 2e, 2f, 2g, and 4a.

2.2.2.2 Physical and Mechanical Properties

The physical and mechanical properties determinations performed on the baseline and hybrid systems (Table VII) are presented in Table VIII. Physical properties consisted of specific gravity, fiber volume and void percent. Prior to mechanical properties testing, all laminates were ultrasonically "C" scanned, using previously established Boeing procedures. The mechanical properties determinations consisted of flexural strength and modulus, and interlaminar shear strength. Tests were conducted at room

temperature and at elevated temperature before and after thermal and humidity exposure of up to 500 hours duration as shown in Table VIII. The elevated test temperatures used were selected to be within the upper service temperature capability of the various resin matrices: 405.2K (270°F) for epoxies, 477.4K (400°F) for phenolics, and 533K (500°F) for polyimides.

Test methods consisted of ASTM D790 for flexure and ASTM D2344 for short beam shear. A span to depth ratio of between 28 and 32 to one was used for flexure tests and between four and five to one for shear tests.

Test results for physical properties and mechanical properties are presented in Table XII and XIII respectively.

2.2.3 SELECTION OF BEST HYBRID CONCEPTS

In compliance with contractural requirements, a 203.2 x 203.2 mm (8 x 8 in.) laminate was prepared and delivered to NASA for each of the eight hybrid concepts evaluated that best retained graphite fibers during burn and impact exposure. Four of these concepts were for laminates with a thickness of 1.016 to 1.524 mm (0.040 to 0.060 in.) and the other four for a laminate thickness of 6.35 mm (0.250 in.). Based on the burn and impact studies described in this section, the hybrid concepts most effective in preventing release of graphite fibers were:

1. 203.2 x 203.2 mm (8 x 8 in.) x 1.016 mm (0.040 in.) to 1.524 mm (0.060 in.)
   a. Aluminum flame spray coating (System #1b of Table VII)
   b. Boron filler (7%) in all plies of baseline laminate #1 (Table VII)
   c. Graphite/fiberglass hybrid fabric/5208 at 0,45,0,45,0 orientation
   d. Phenolic matrix resin (System #5 of Table VII)
2. 203.2 x 203.x (8 x 8 in.) x 6.35 mm (0.250 in.)
   a. Intumescent coating (System 2e of Table VII)
   b. Replace 0, 90 graphite tape with grahite fabric (System 2g of Table VII)
   c. Talc filled phenolic exterior coating (coating of System 5a applied to baseline system #2 of Table VII)
   d. Graphite/fiberglass hybrid fabric for exterior ply and add boron filler to exterior ply only (System 2b of Table VII)

Before delivering these laminates to NASA, they were ultrasonically "C" scanned and determined to be of high quality. Also, a photomicrograph was made of a section removed from each laminate: see Figures 75 through 83.

3.0 CONCLUSIONS AND RECOMMENDATIONS

The conclusions reached from studies conducted during this program are presented in 3.1. The recommendations regarding severity of the fiber release problem and fu ther work to be considered in this area are presented in 3.2.

3.1 CONCLUSIONS

1. Graphite fibers were released from graphite/epoxy laminates during the burn and impact tests performed in this program. However, the extent to which the fibers were released is not considered a severe enough problem to preclude the use of these materials in civil aircraft structure.
2. The hybrid concepts selected were able to prevent release of graphite fibers under the burn and impact tests performed in this program. Also, several of the concepts have zero cost impact.
3. Thermal and humidity aging do not appear to alter the fiber release tendencies of composites.

3.2 RECOMMENDATIONS

Based on results of evaluations performed during this program, the following areas are recommended for further study:

1. The effect of particle size and percentage resin loading of boron and other candidate fillers on (a) retaining graphite fibers during burn/impact conditions, and (b) effect of these filler parameters on composite physical and mechanical properties. Besides boron, other candidate fillers include glass frit of different melt temperatures. This particular approach is projected to have minimum impact on cost and properties.
2. The effect of fiber orientation and structure edge constraints on graphite fiber retention as certain orientations may be incorporated in the design that would preclude the need of hybridization.
3. Comparison of various test techniques for measuring effectiveness of hybrids to retain graphite fibers including scale-up to full size aircraft structure such as exterior fairing assemblies.

4.0 REFERENCES

1. "A Report of Observed Effects on Electrical Systems of Airborne Carbon/Graphite Fibers" NASA Technical Memorandum 78652.

2. Boeing unpublished data

3. S. S. Tompkins and W. D. Brewer, "Preliminary Burn and Impact Tests of Hybrid Polymeric Composites," NASA Technical Memorandum 78762, July, 1978.

4. Dobyns, A. L. "COOP - Composite Analysis and Optimization Computer Program-Theory and Users Manual," Boeing Document D180-20377-1, March 1977.

5. Gellatly, R. A., Gallagher, R. H., Lubgracki, W. A., "Development of a Procedure for Automated Synthesis of Minimum Weight Structures," FDL-TRD-64-141.

6. "Proposed Test for Heat and Visible Smoke Release Rates for Materials and Products," ASTM Proposed Standard prepared by ASTM Task Group E05.02.30 on Release Rate Test Methods, 4/26/77 version.

Task I—Concept Definition and Analysis

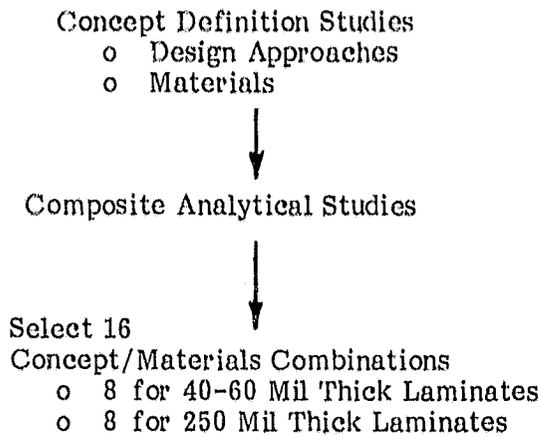

Figure 1 - Task I Flow Diagram

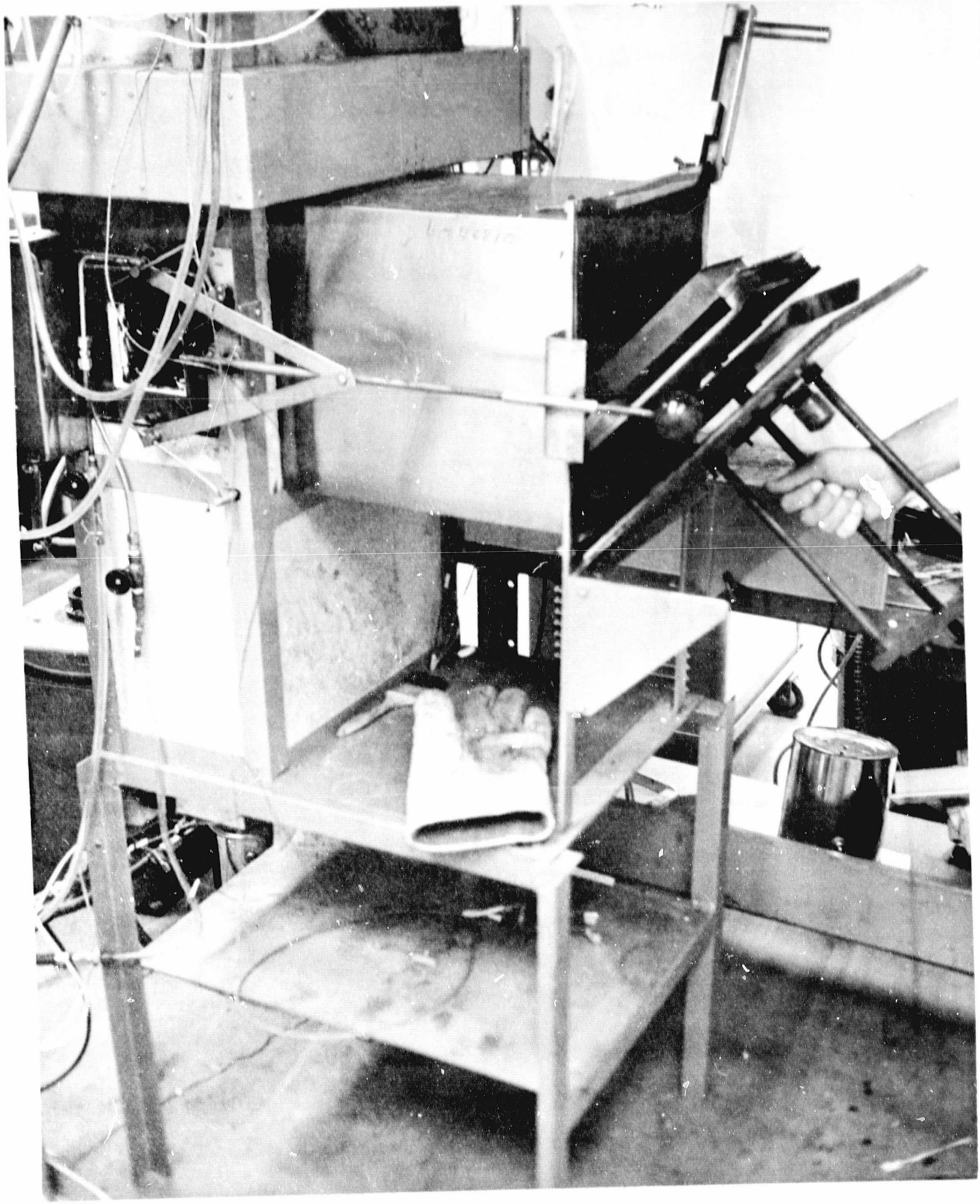

Figure 2 Laminate Being Inserted Into the OSU Apparatus

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Figure 3 Celion 6000/PMR-15, Preliminary Burn Test Laminate #1

Figure 4 Celion 6000 Chopped Fiber/PMR-15, Preliminary Burn Test Laminate #2

Figure 5 T300/934, Preliminary Burn Test Laminate #3

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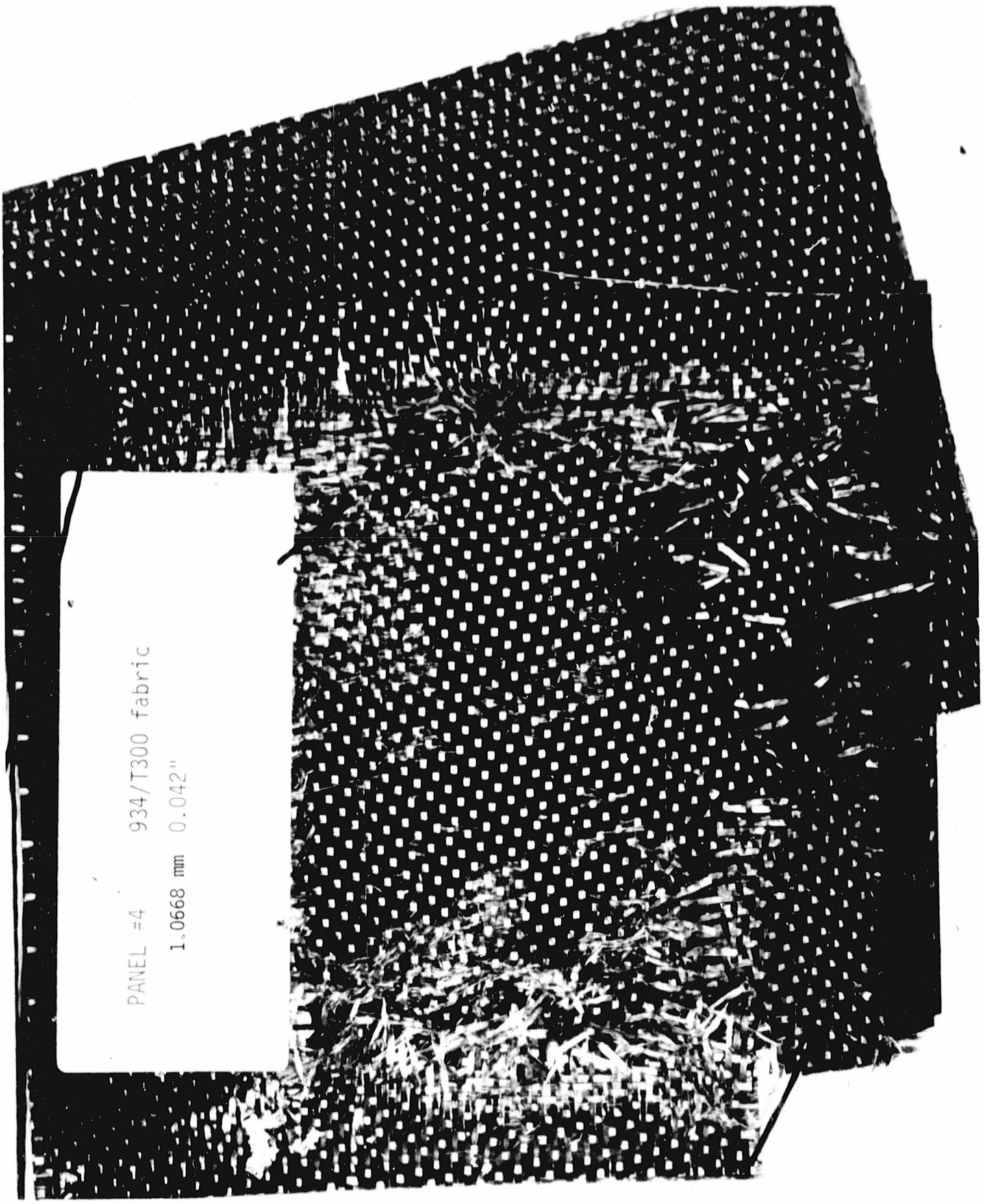

Figure 6 T300 Fabric/934, Preliminary Burn Test Laminate #4

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Figure 7 AS/P1700, Preliminary Burn Test Laminate #5

Figure 8 T300 Chopped Fiber/934, Preliminary Burn Test Laminate #6

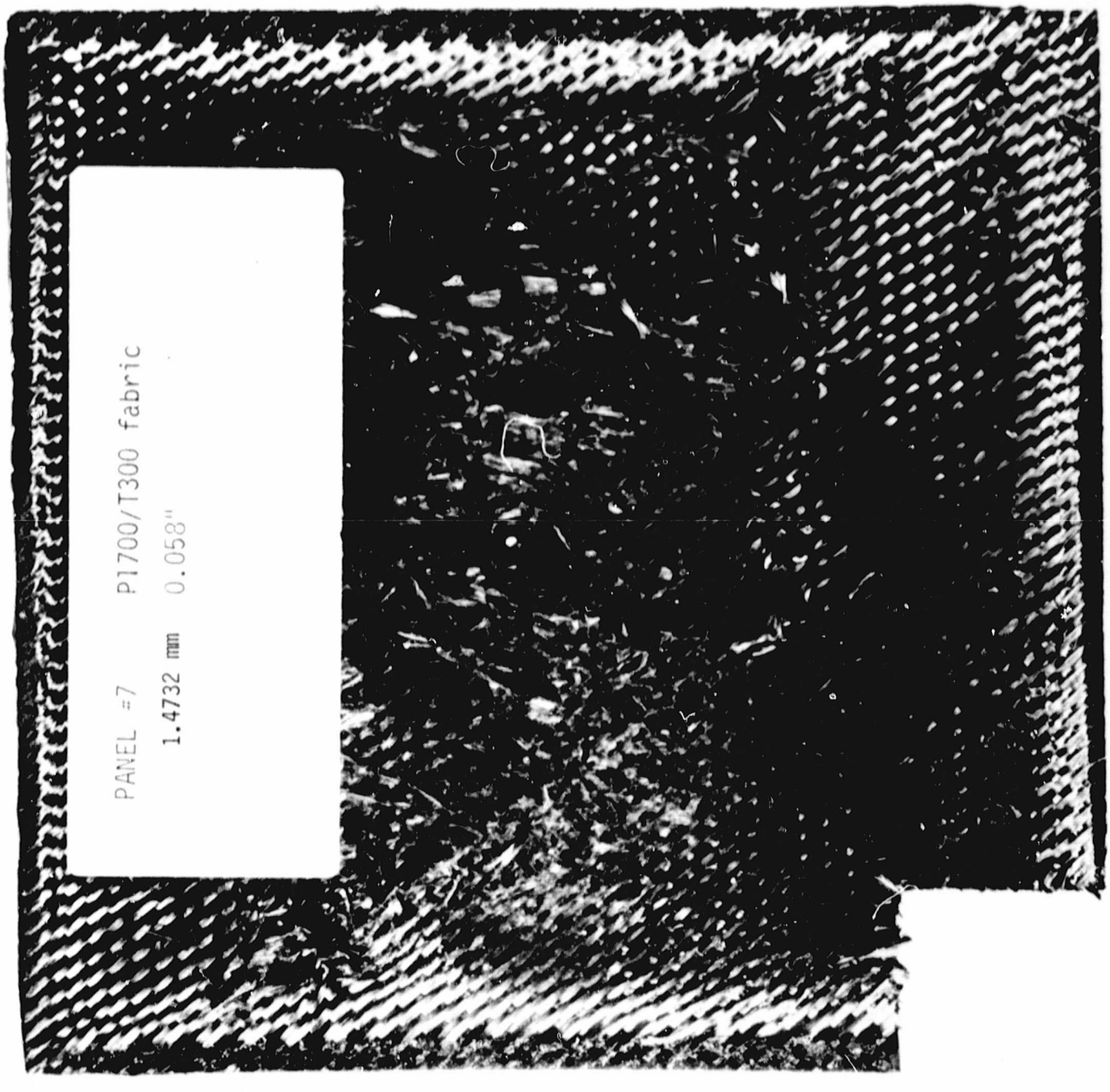

Figure 9 T300 Fabric/P1700, Preliminary Burn Test Laminate #7

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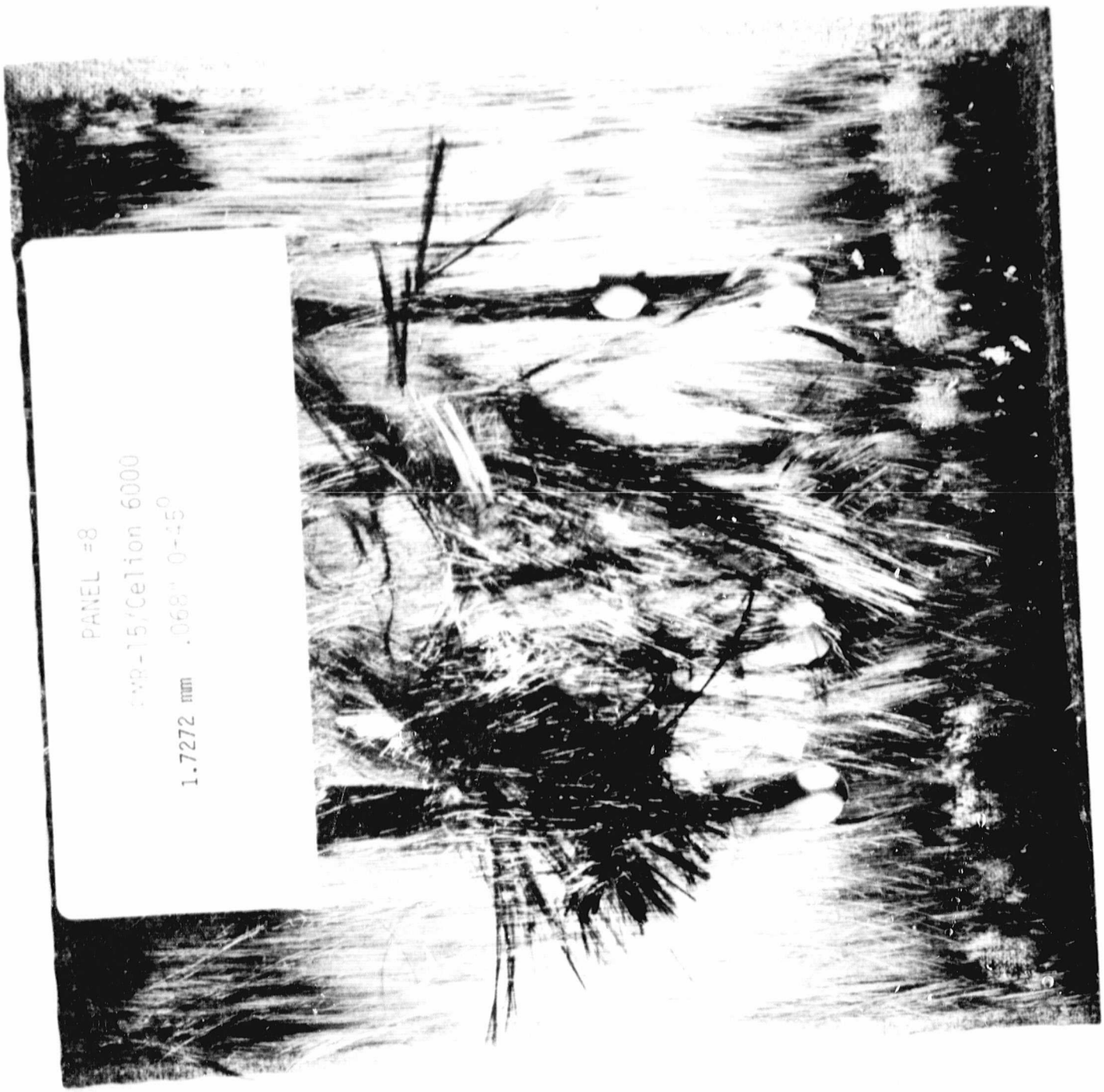

Figure 10 Celion 6000,PMR-15, Preliminary Burn Test Laminate #8

Figure 11 Celion 6000/PMR-15, Preliminary Burn Test Laminate #9

Figure 12 Celion 6000/PMR-15, Preliminary Burn Test Laminate #10

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Figure 13 Celion 6000/PMR-15, Preliminary Burn Test Laminate #11

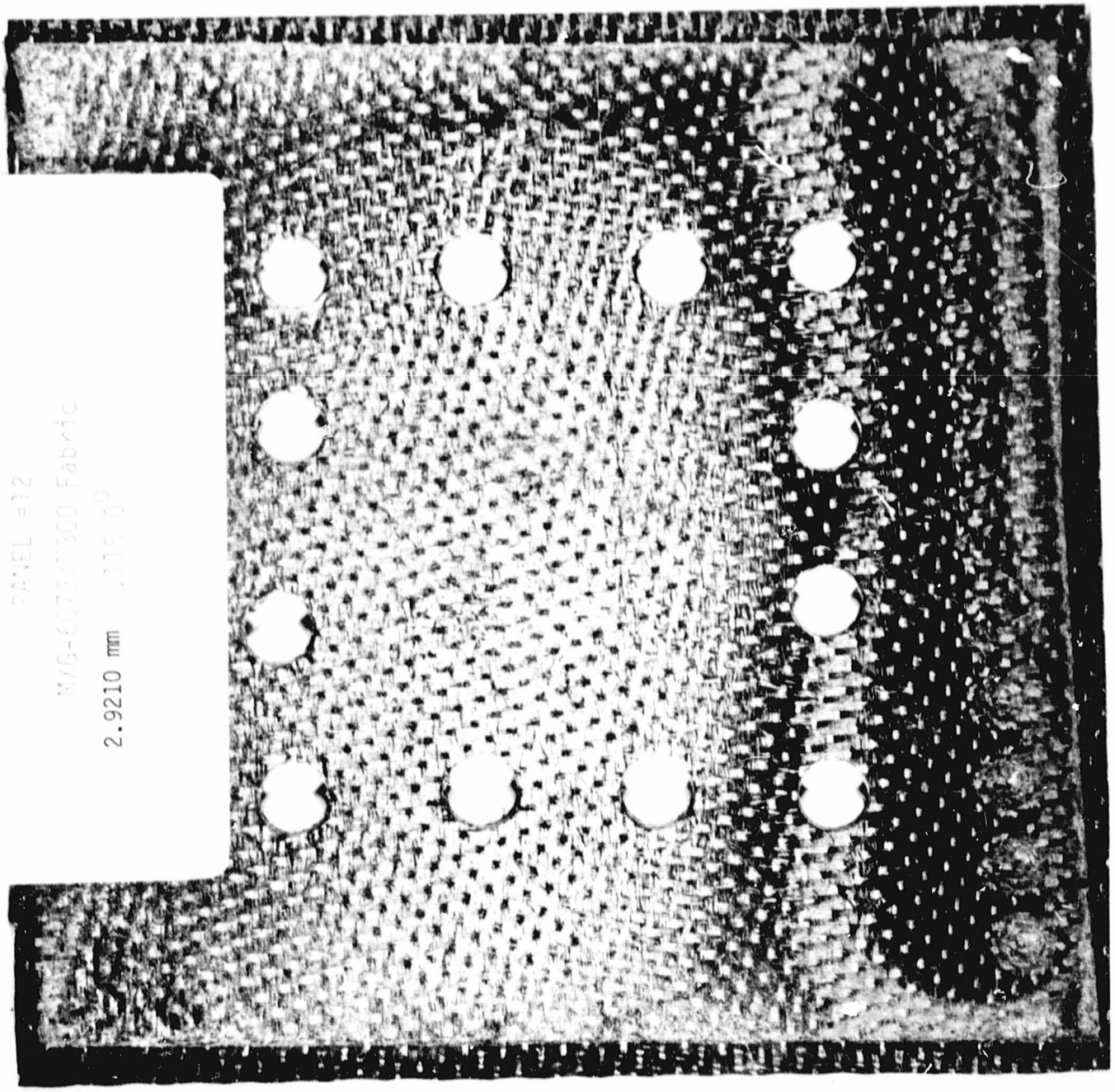

Figure 14 T300 Fabric/MXG-6073, Preliminary Burn Test Laminate #12

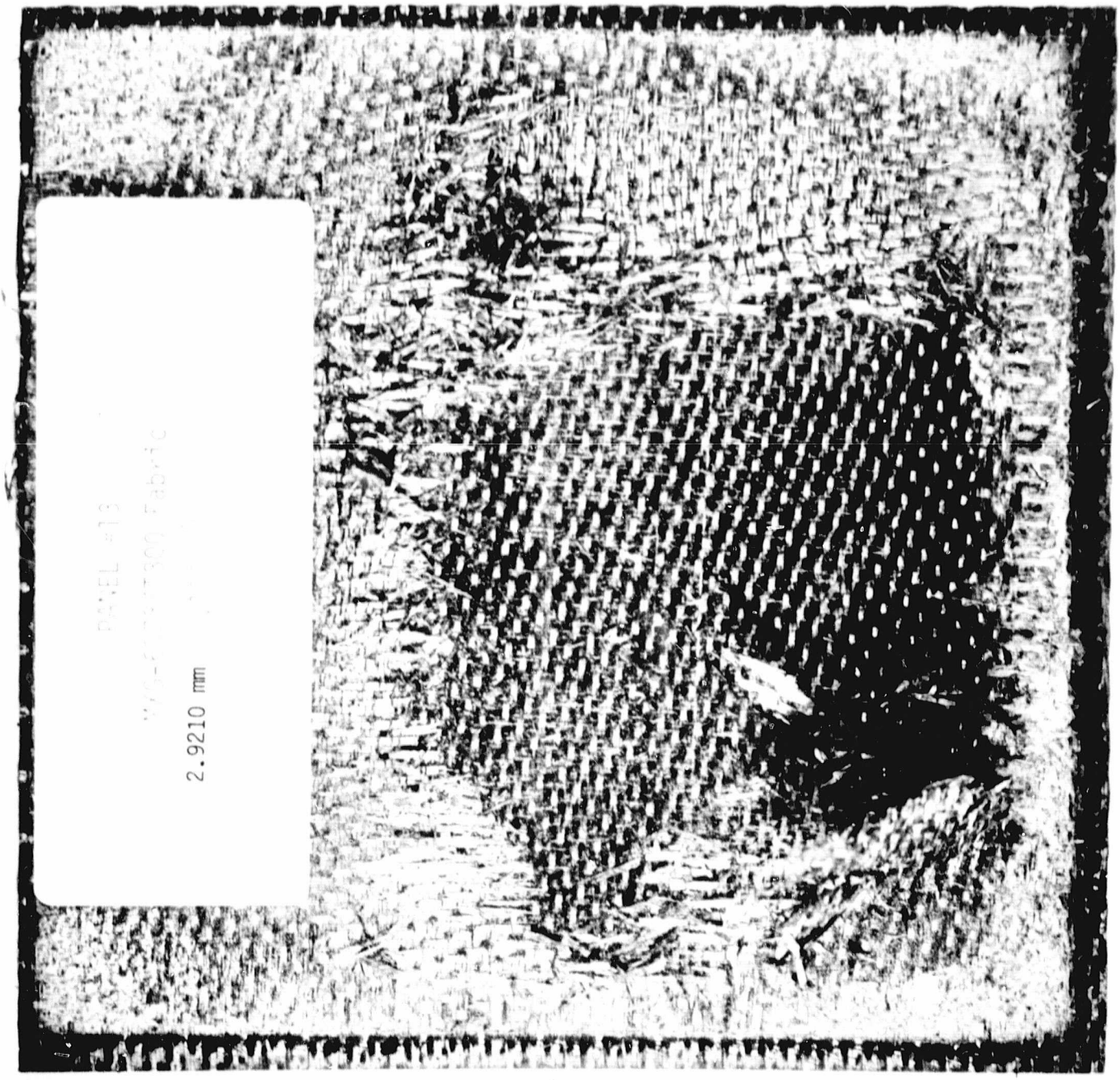

Figure 15 T300 Fabric/MXG-6073, Preliminary Burn Test Laminate #13

Figure 16 T300 Fabric/Kevlar 49/MXG-6073, Preliminary Burn Test Laminate #14

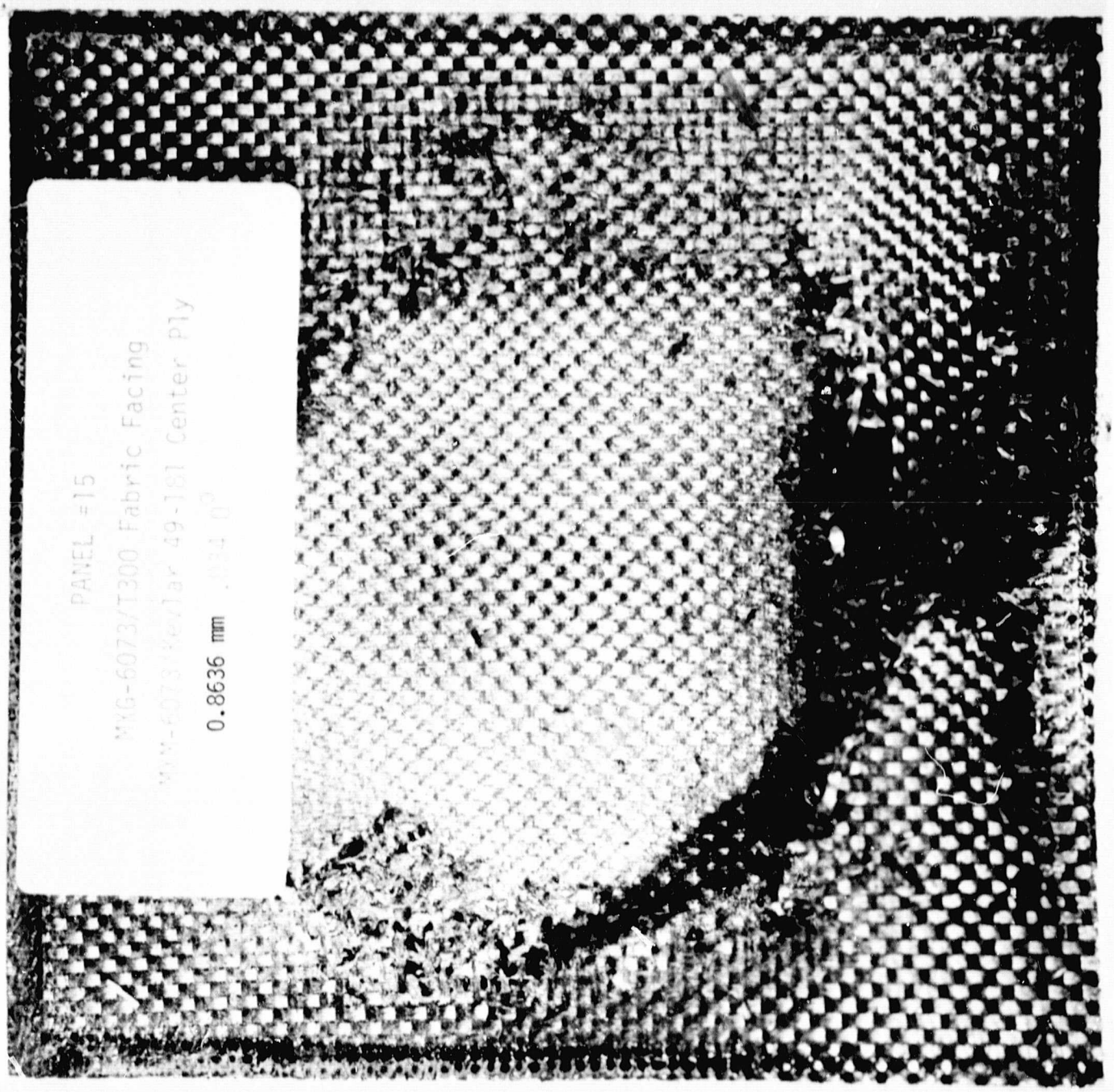

Figure 17 T300 Fabric/Kevlar 49/MXG-6073, Preliminary Burn Test Laminate #15

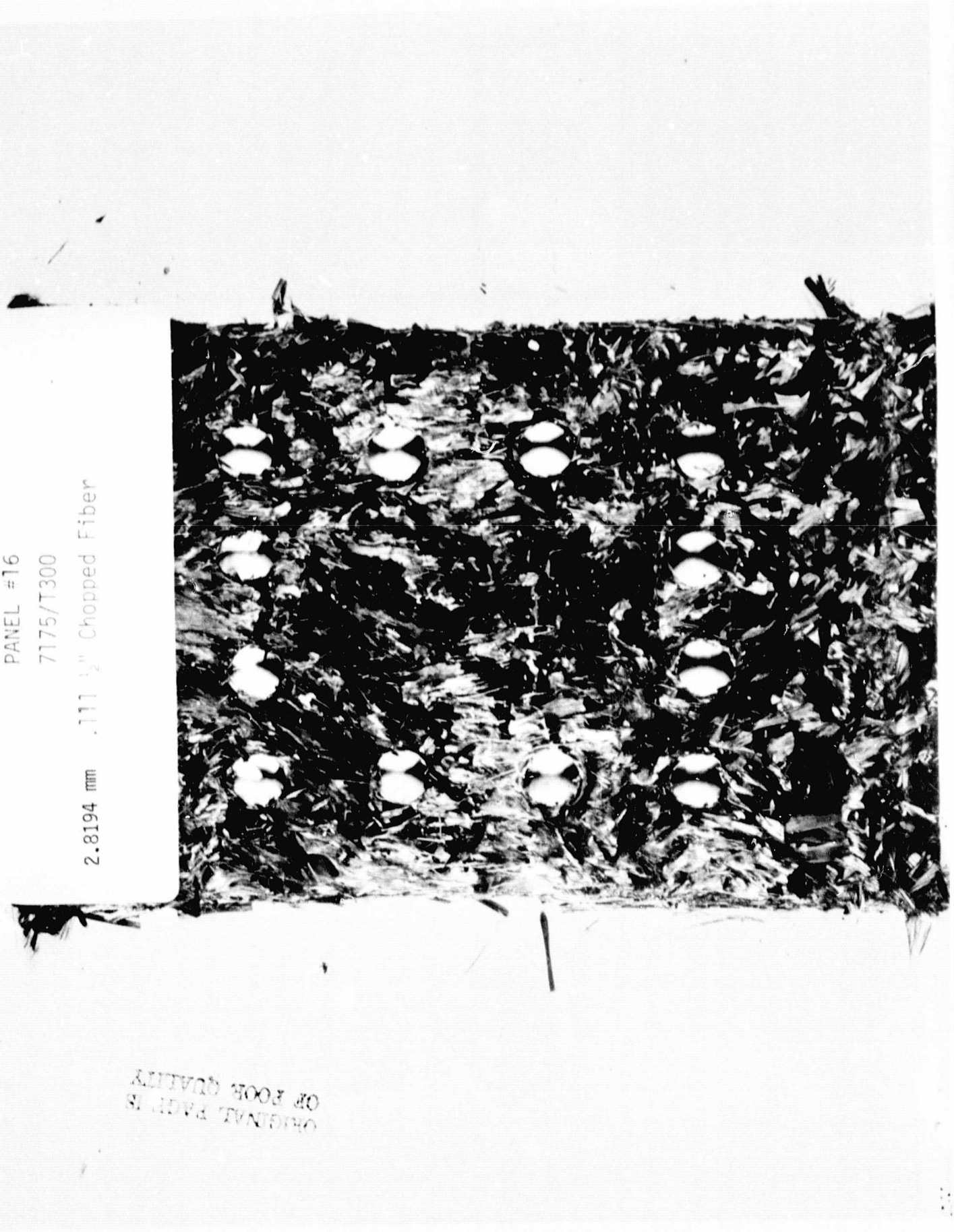

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Figure 18 T300 Chopped Fiber/7175, Preliminary Burn Test Laminate #16

Figure 19 T300 Chopped Fiber/7175, Preliminary Burn Test Laminate #17

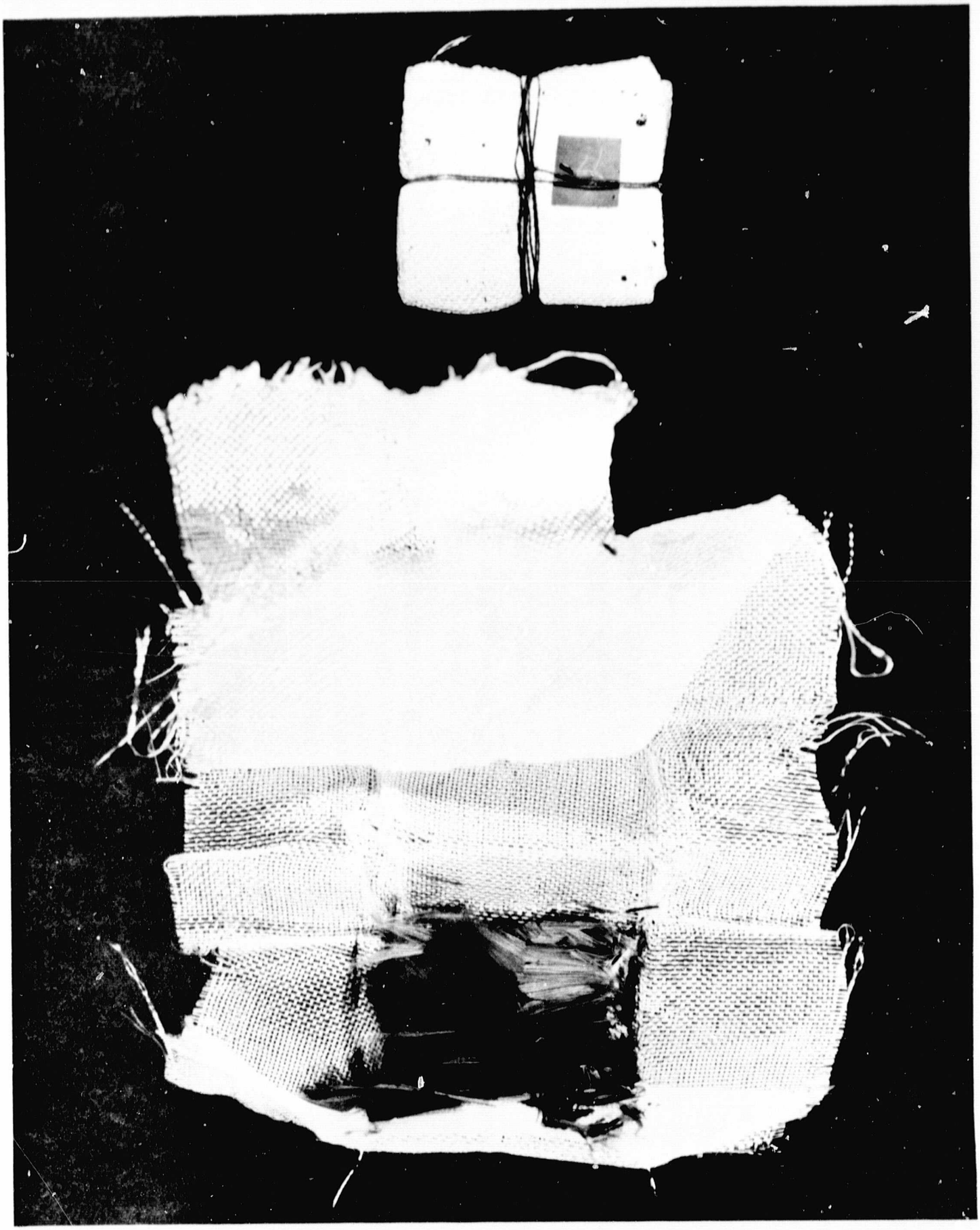


Figure 20 Isothermal Aging Specimen

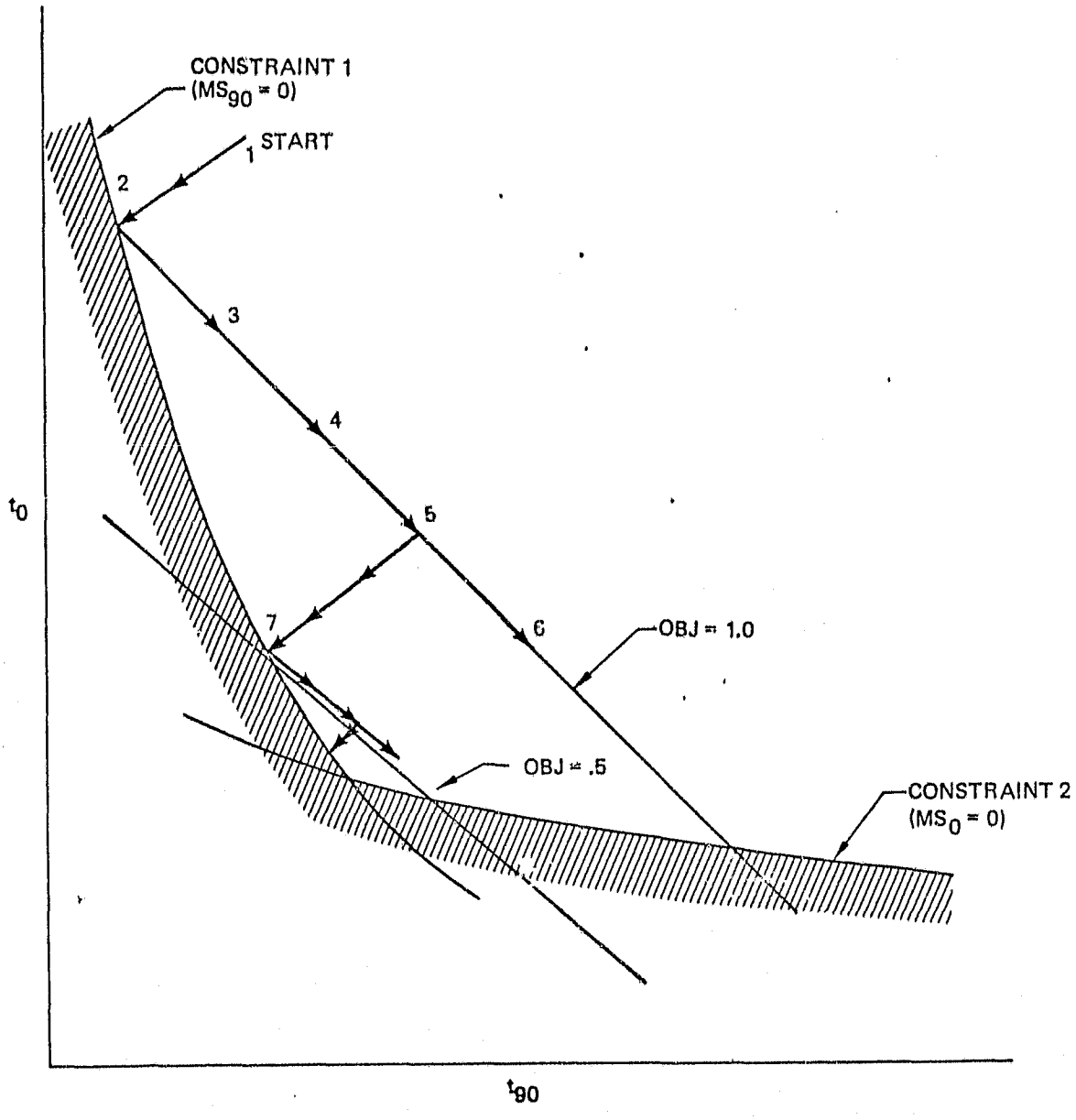


Figure 21. "COOP" Optimization Example

HYBRID STUDY

OPTIMIZATION FACTORS

WT	COST	EX	EY	GXY	FXT	FYT	FXC	FYC	FS	KC
.900	.800	.400	.400	0.000	.400	.400	0.000	0.000	.200	0.000

NX	NY	NXY
2000.	1500.	800.

RANK	0 DEG	+-45 DEG		90 DEG	HYBRID	OBJ	WT	COST	0	+45	-45	90	HYBRID
									THICKNESSES				
1	T300/934	T300/934	T300/934	T300/934		3.6893	.00220	.09800	.01000	.01000	.01000	.01000	0.00000
2	T300/934	T300/934	T300/934	T300/934	T300/FAB(0/90)	3.9244	.00253	.10120	0.00000	.01000	.01000	0.00000	.02600
3	T300/934	T300/934	T300/934	T300/934	T300/FAB(+45)	4.2220	.00253	.10120	.01000	0.00000	0.00000	.01000	.02600
4	T300/934	T300/934	T300/934	T300/934	90GR/10GL	4.3377	.00311	.12424	0.00000	.01000	.01000	.01000	.02600
5	T300/934	T300/934	T300/934	T300/934	80R6R	4.5214	.00238	.21100	0.00000	.01000	.01000	.01000	.01000
6	T300/934	T300/934	T300/934	T300/934	50GR/50GL	4.8725	.00376	.11920	.01000	.01000	.01000	.01000	.02600
7	T300/934	T300/934	T300/934	T300/934	S-GL/FAB	4.9512	.00389	.09645	.01000	.01000	.01000	.01000	.02600
8	T300/934					11.9551	.00935	.37400	.17000	0.00000	0.00000	0.00000	0.00000

Figure 22. "COOP" Hybrid Ranking Example

Task II--Composite Fabrication and Evaluation

Laminate Fabrication
- o 203.2 x 203.2 x 1.016 mm (8 x 8 x 0.040 in.) and 203.2 x 203.2 x 6.35 mm (8 x 8 x 0.250 in.)
- o Hybridized and Baseline Laminates

Laminate Evaluation
"As Fabricated" Laminates
- o Physical Properties
- o Flex. Strength and Modulus and ILS at RT and Elev. Temp.

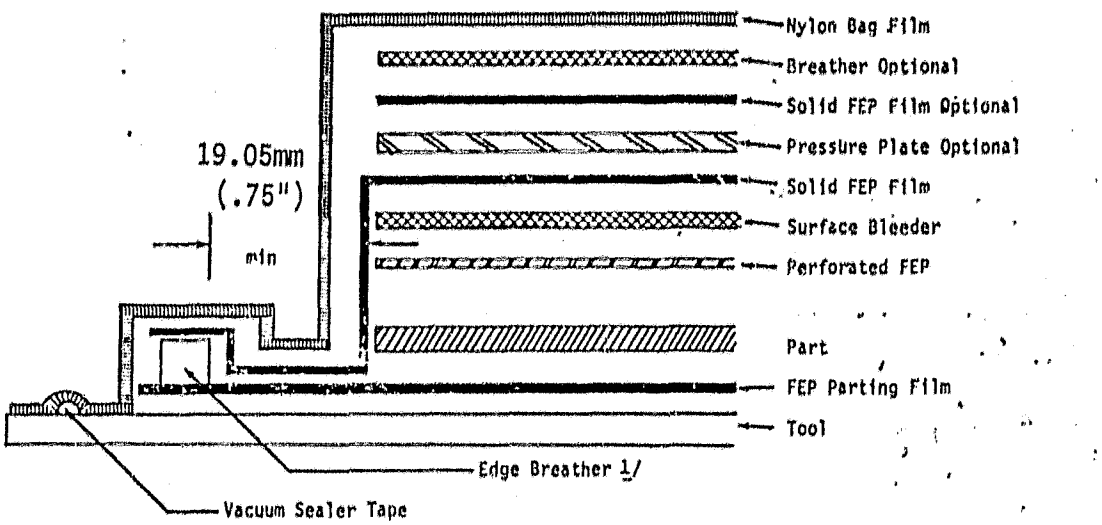
Thermo-Oxidative and Humidity Exposure
- o Flexural Strength and Modulus and ILS During and After Exposures

Flammability Testing
- o As Fabricated and After Exposure to Thermo-Oxidative and Humidity Environments
- o Flame Spread Index, LOI, OSU, Air Flow, and Mechanical Agitation

Evaluation of Structural Integrity and Residual Mechanical Properties
- o Nondestructive Tests
- o Destructive Tests

Selection and Delivery of 8 Laminates to NASA Project Manager

Figure 23 - Task II Flow Diagram



1/ Use a 1 inch minimum width edge breather with connection to vacuum source. At one corner of the layup, place a single fiberglass yarn between the edge of the layup and the edge breather to allow evacuation of air from the layup.

Figure 24. Autoclave Bagging, Epoxy Matrix Systems

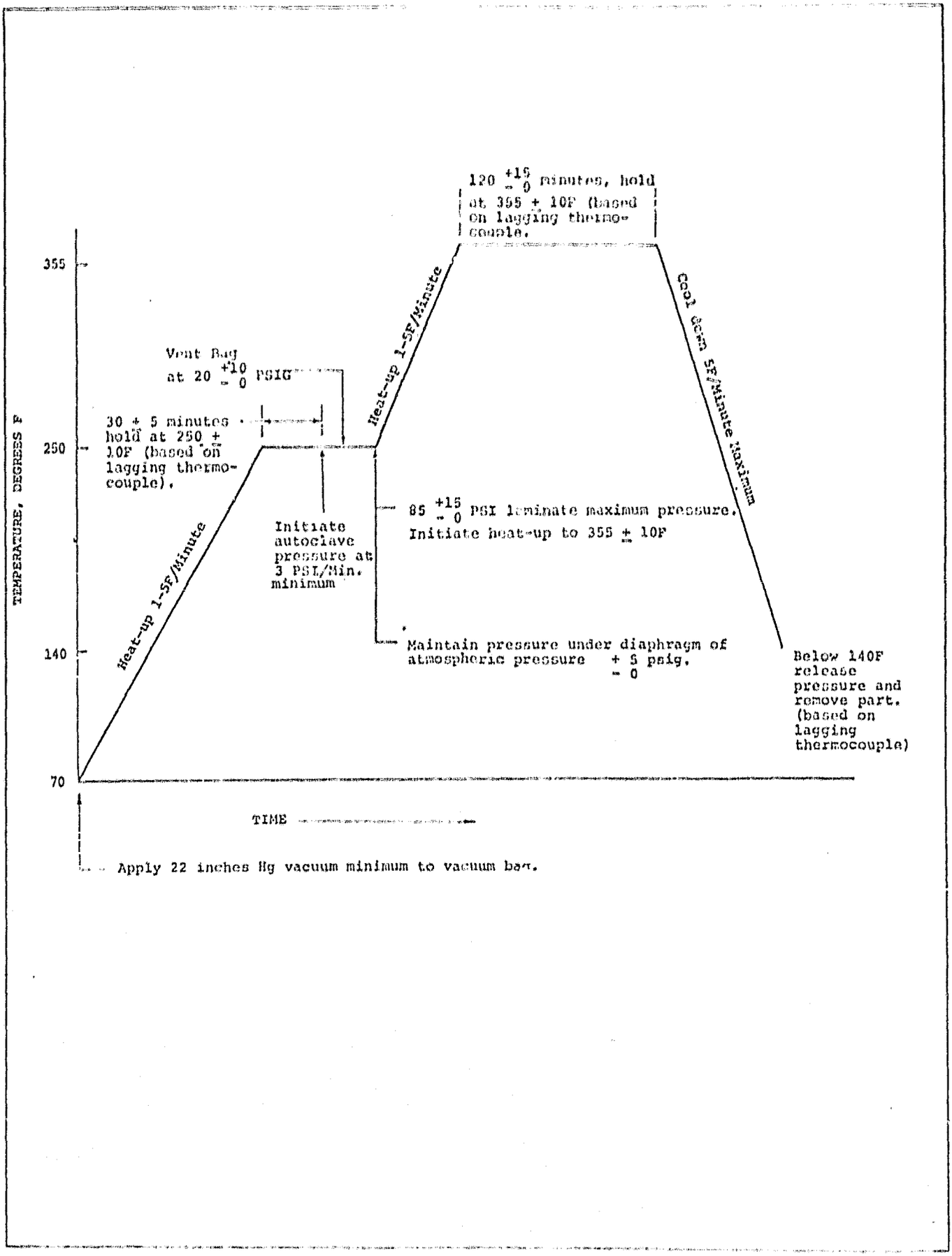


Figure 25. Autoclave Cure Cycle for Epoxy Matrix Composites

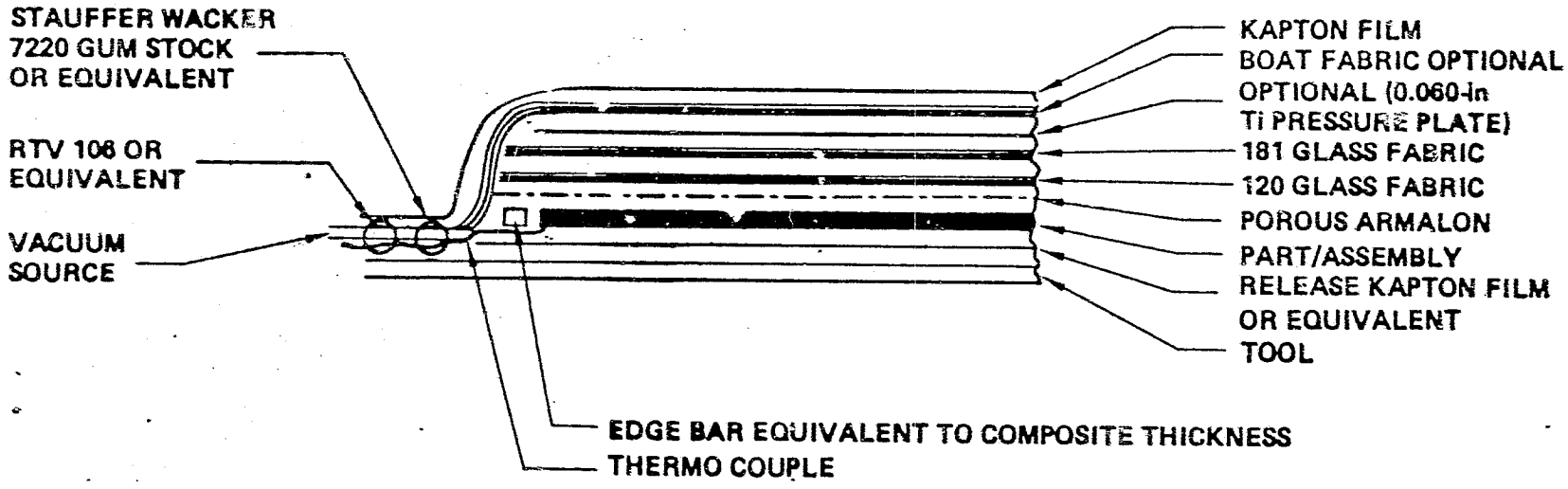


Figure 26. Autoclave Bagging, Polyimide Matrix Systems

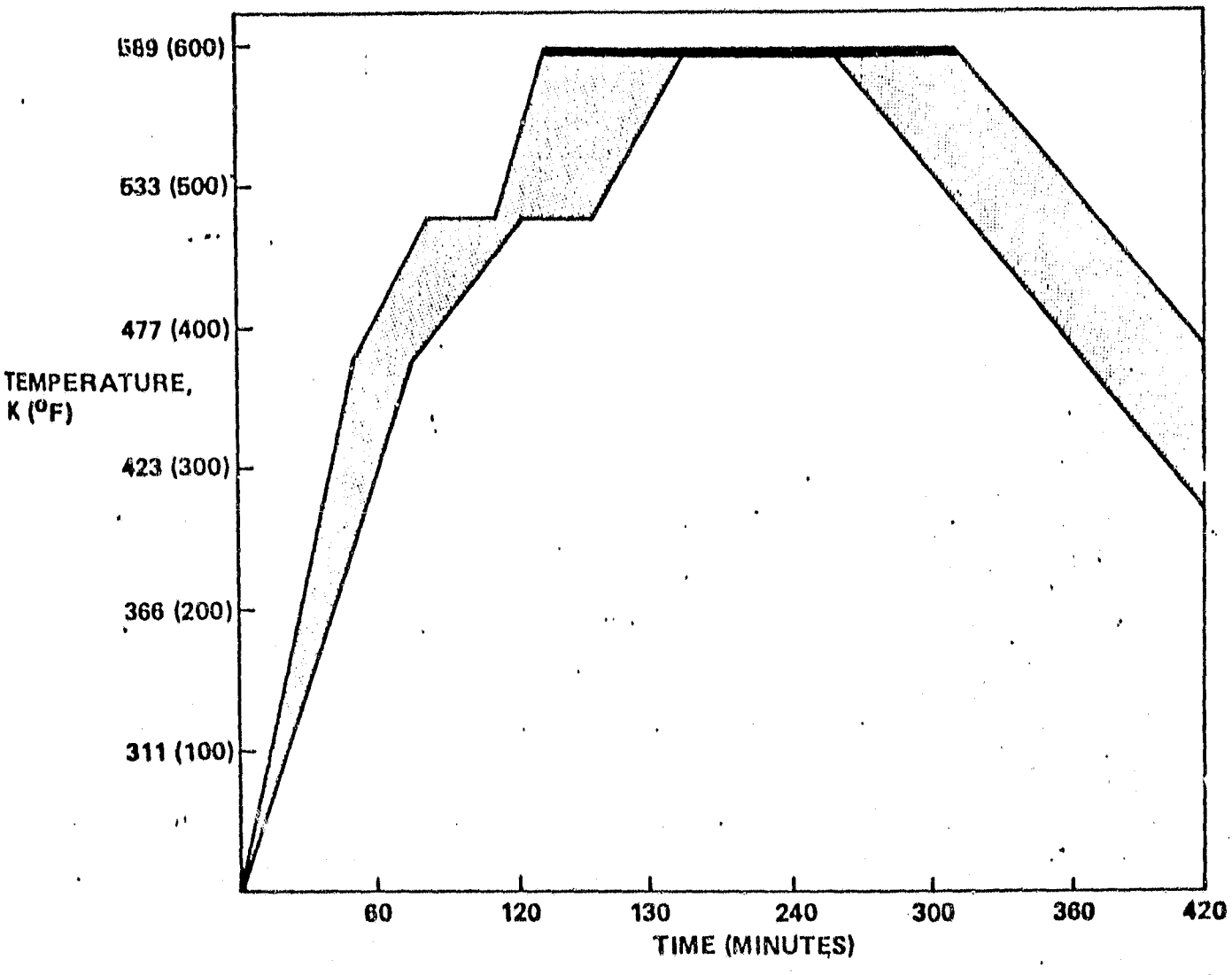


Figure 27. Autoclave Cure Cycle for Polyimide Matrix Composites

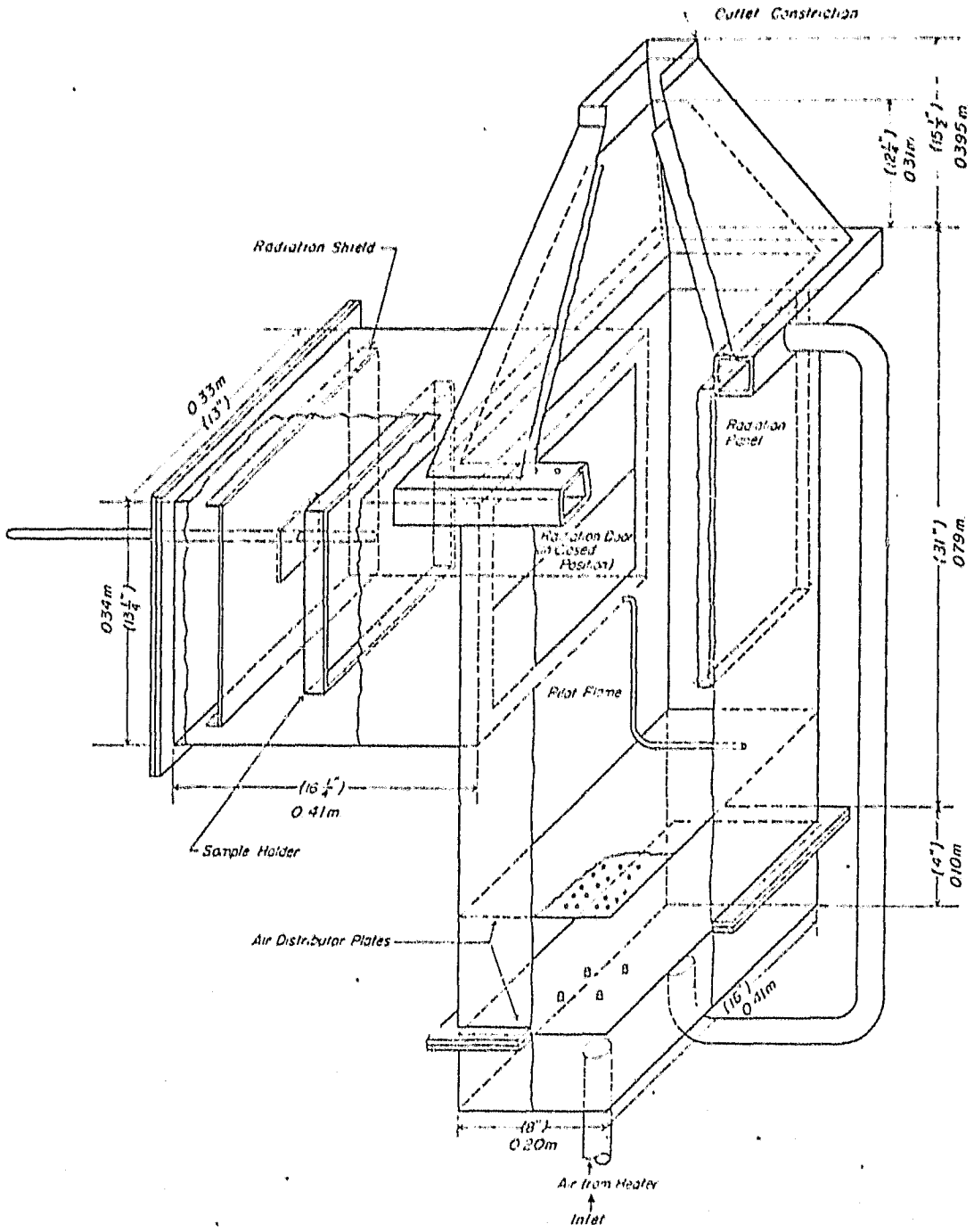


Figure 28. OSU Rate of Heat Release Apparatus, Schematic

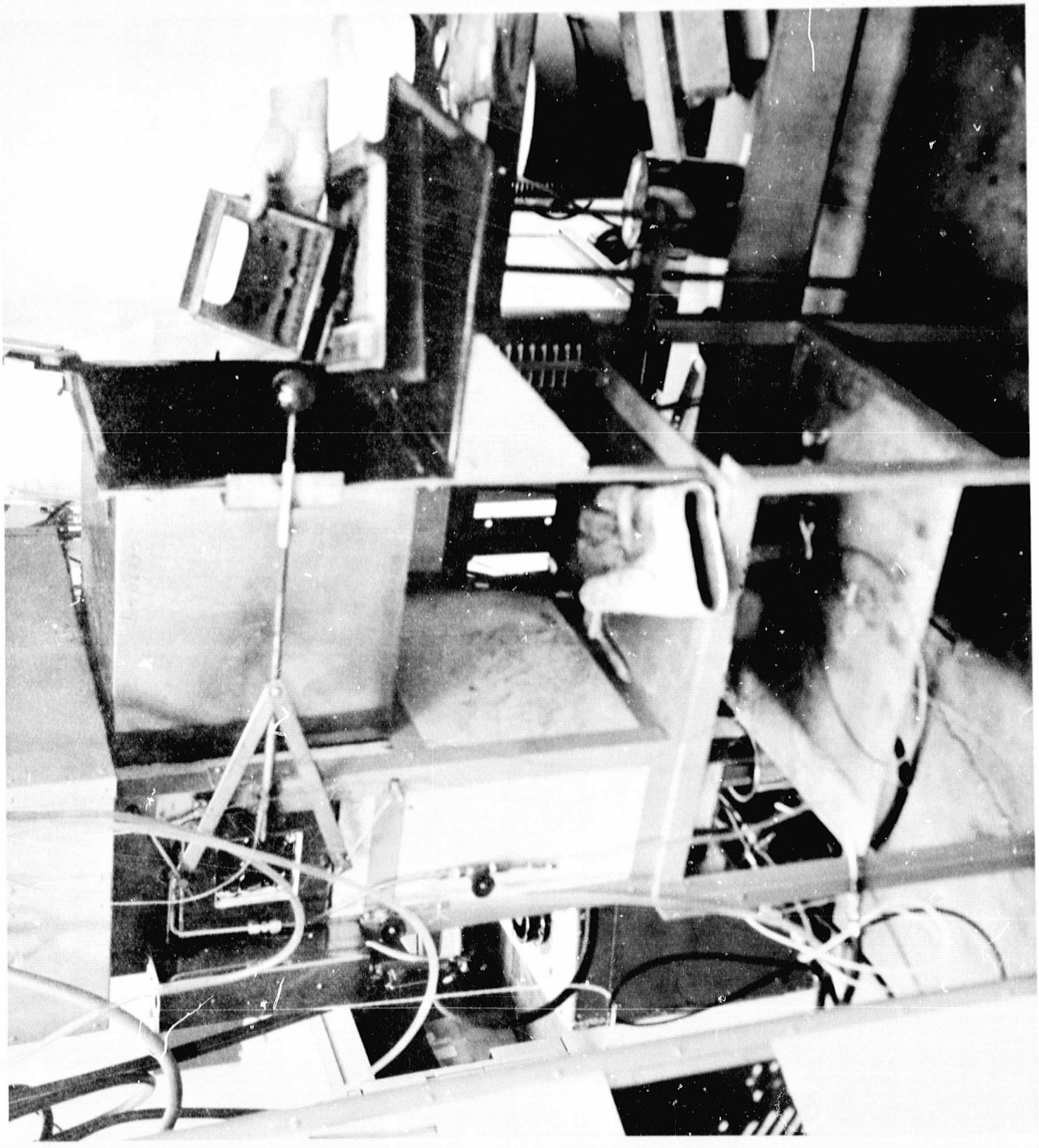


Figure 29 OSU Rate of Heat Release Apparatus and Sample Laminate

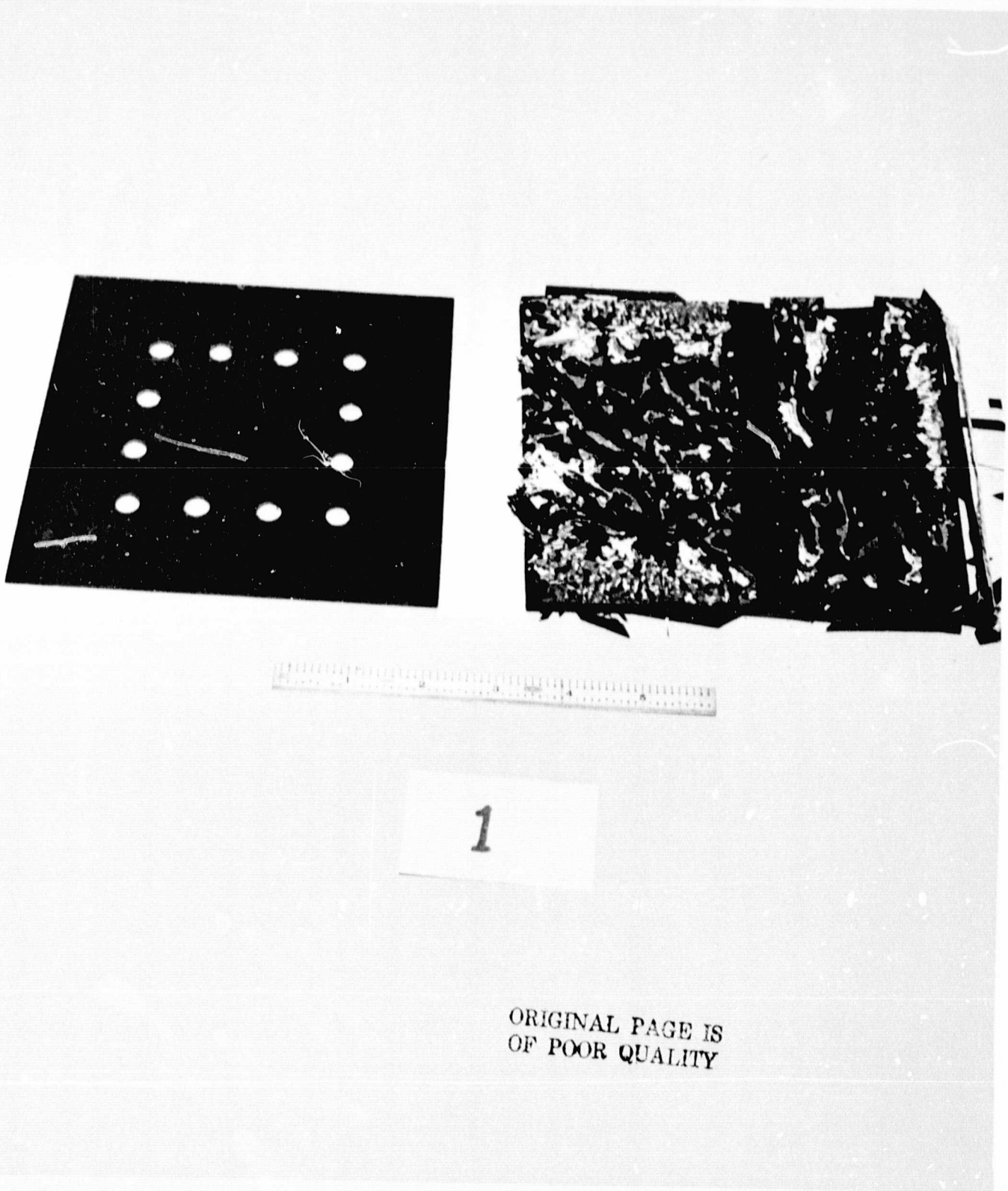


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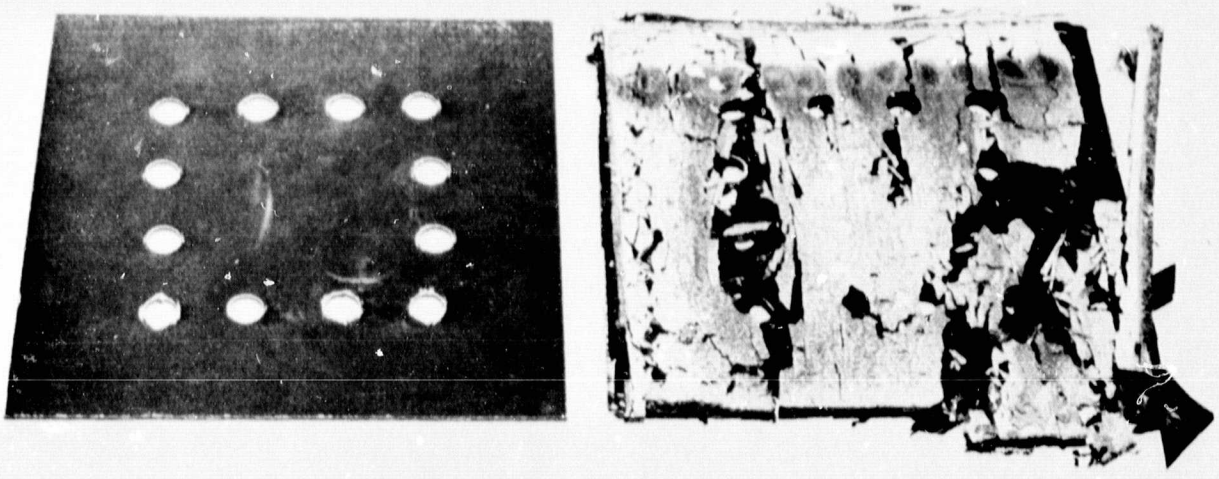
Figure 30 Baseline System #1, Before and After Burn Test



1A

Figure 31 Hybrid System #1a, Before and After Burn Test

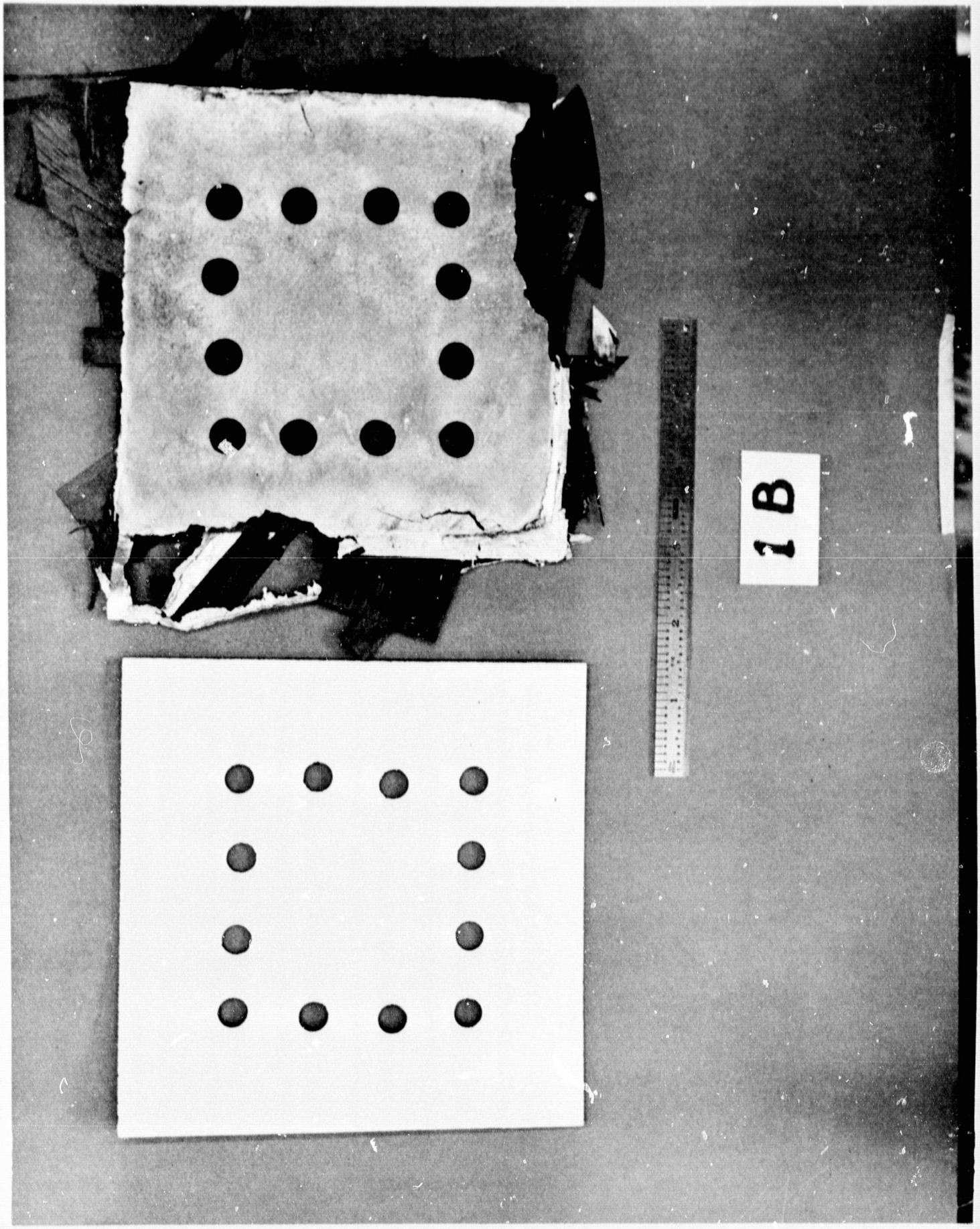


Figure 32 Hybrid System #1b, Before and After Burn Test

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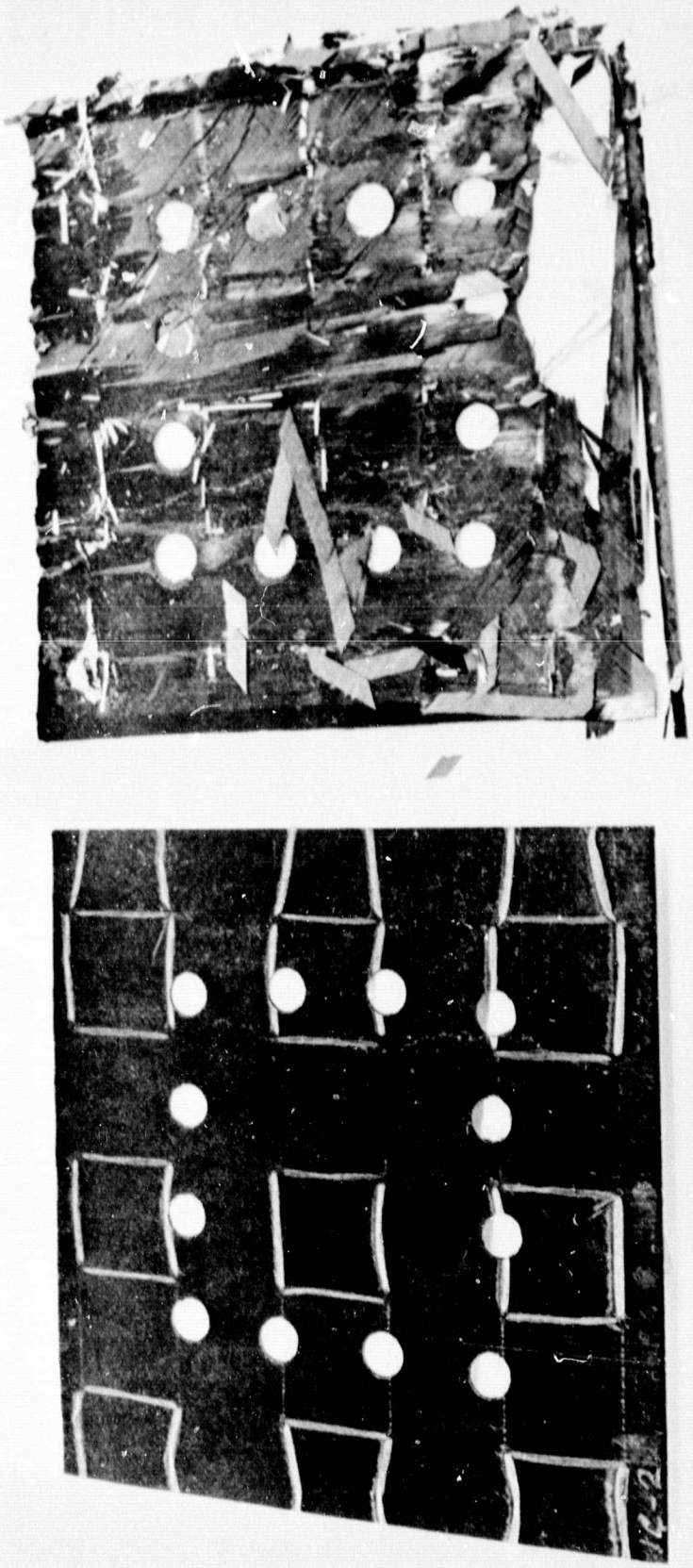


Figure 33 Hybrid System #1c, Before and After Burn Test

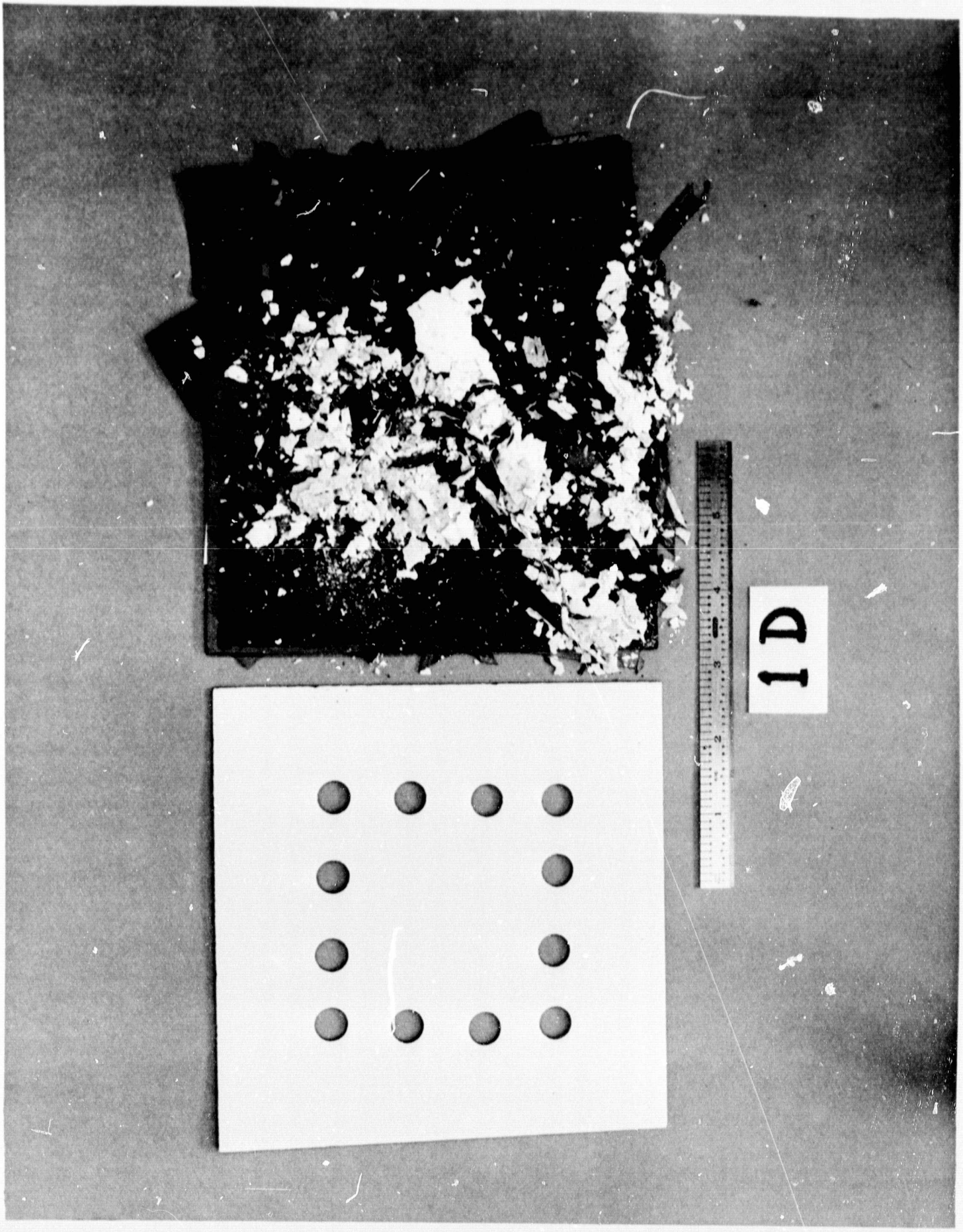
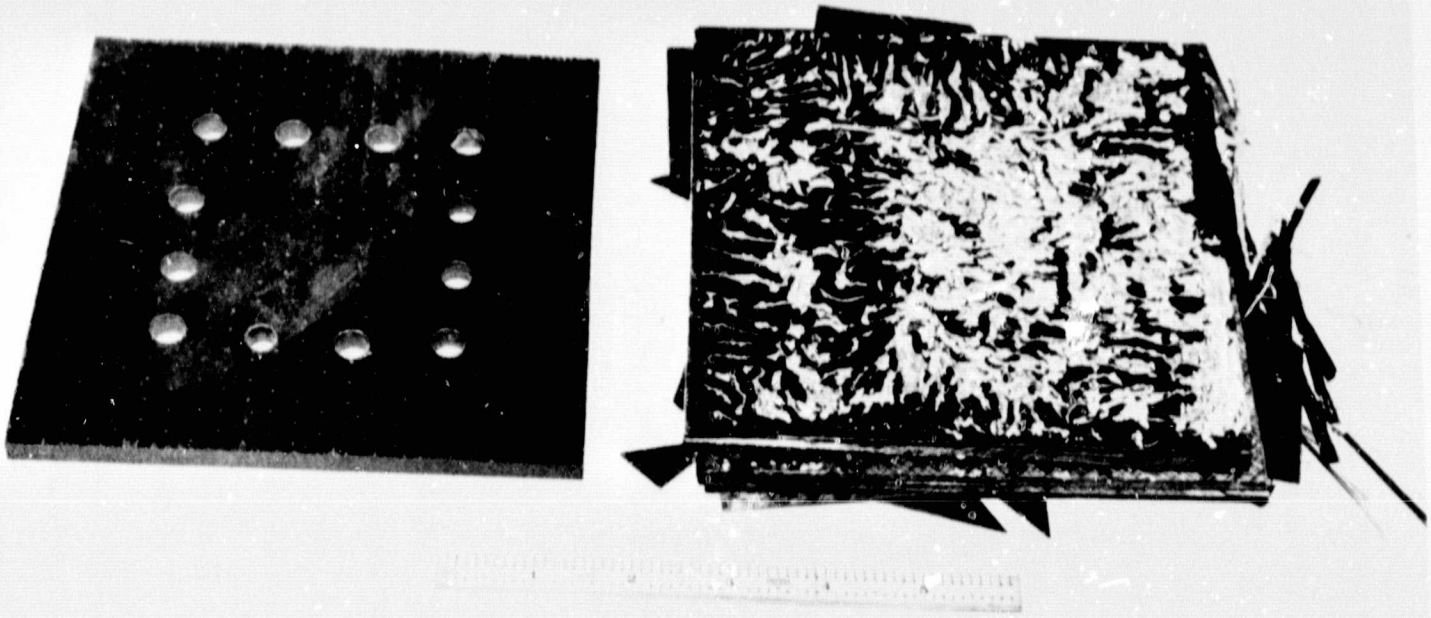
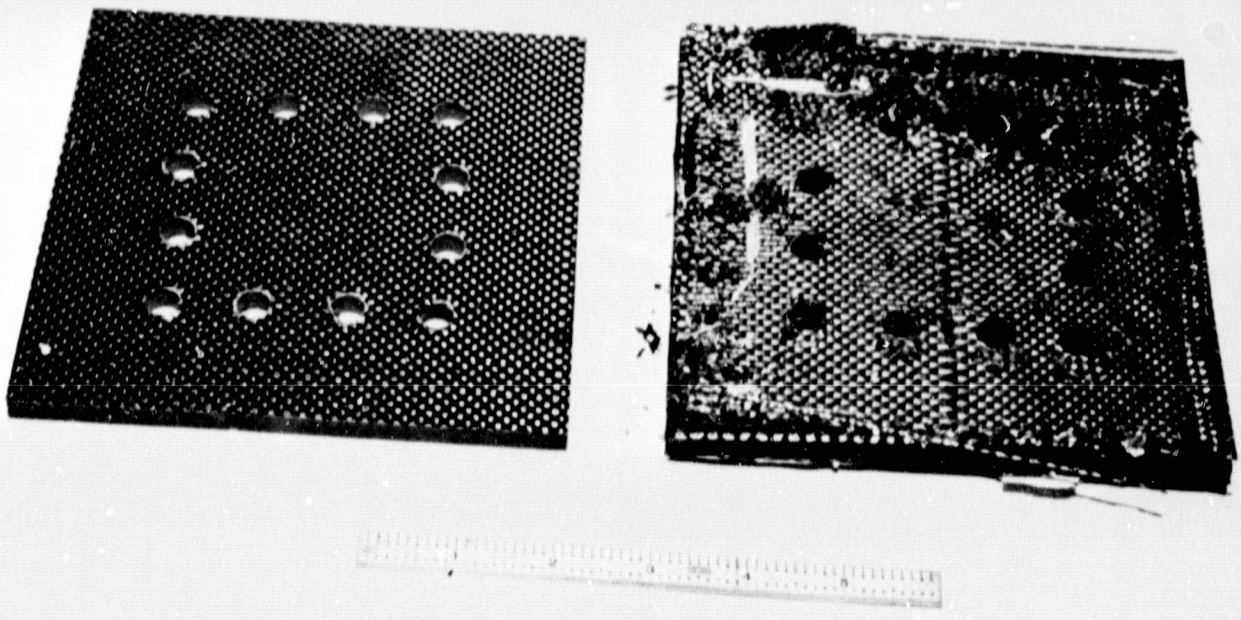


Figure 34 Hybrid System #1d, Before and After Burn Test



2.

Figure 35 Baseline System #2, Before and After Burn Test



2A

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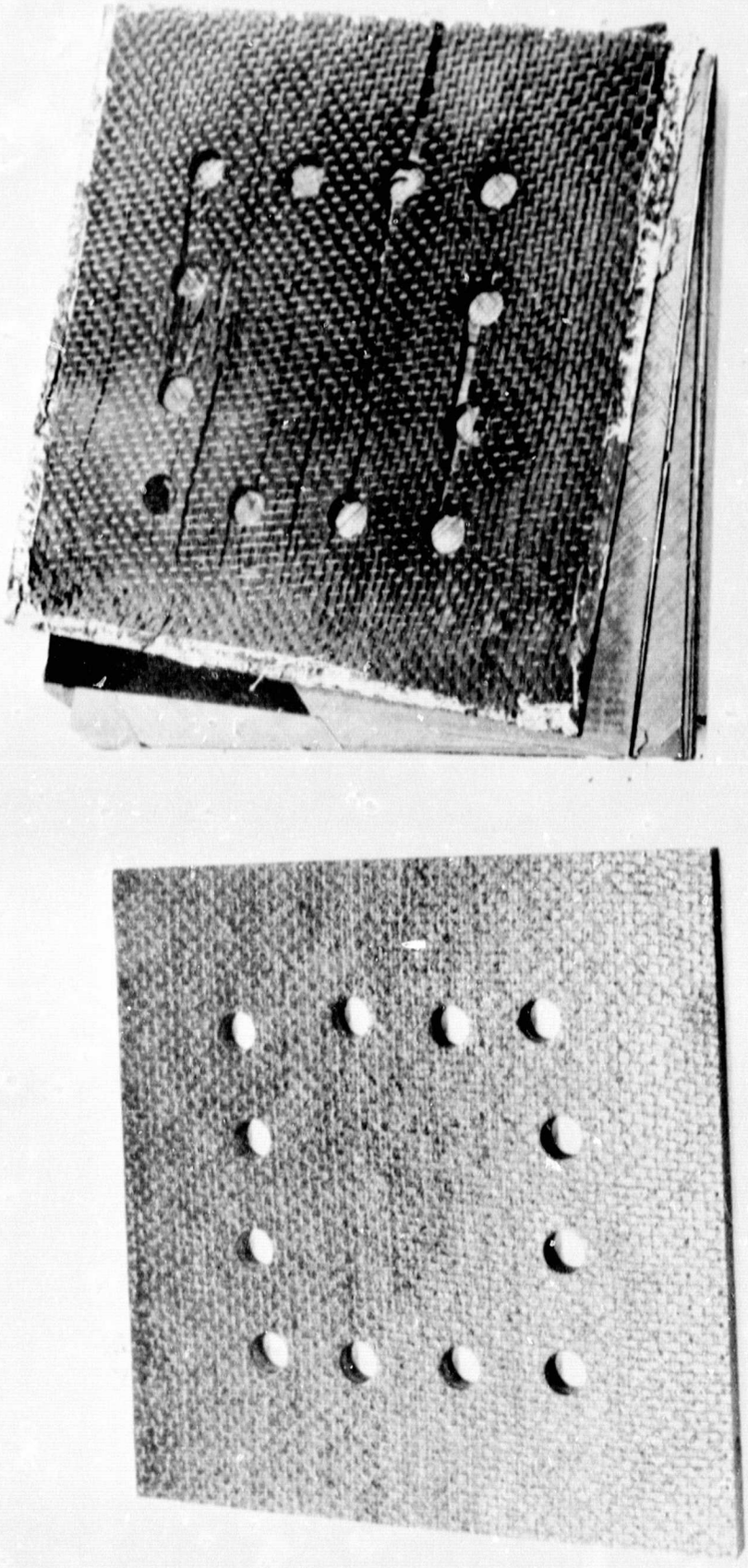
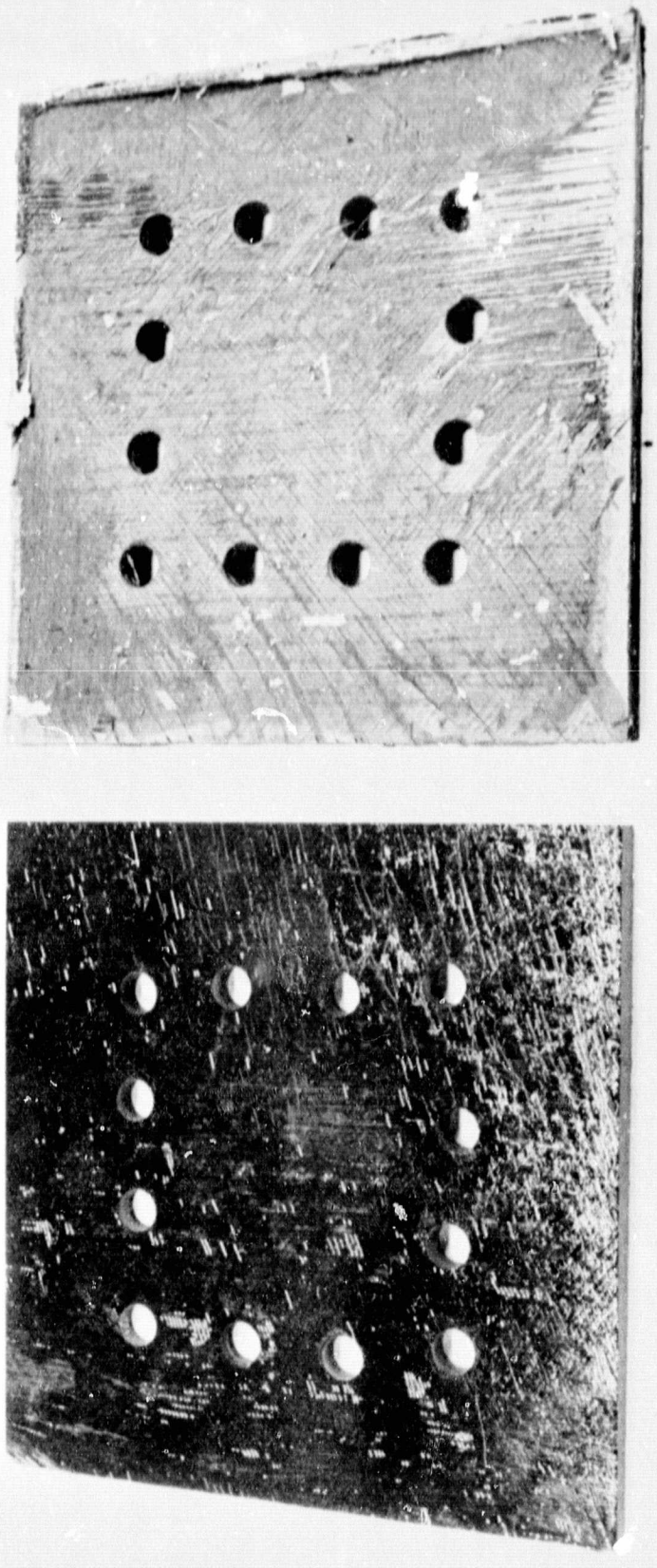
Figure 36 Hybrid System #2a, Before and After Burn Test



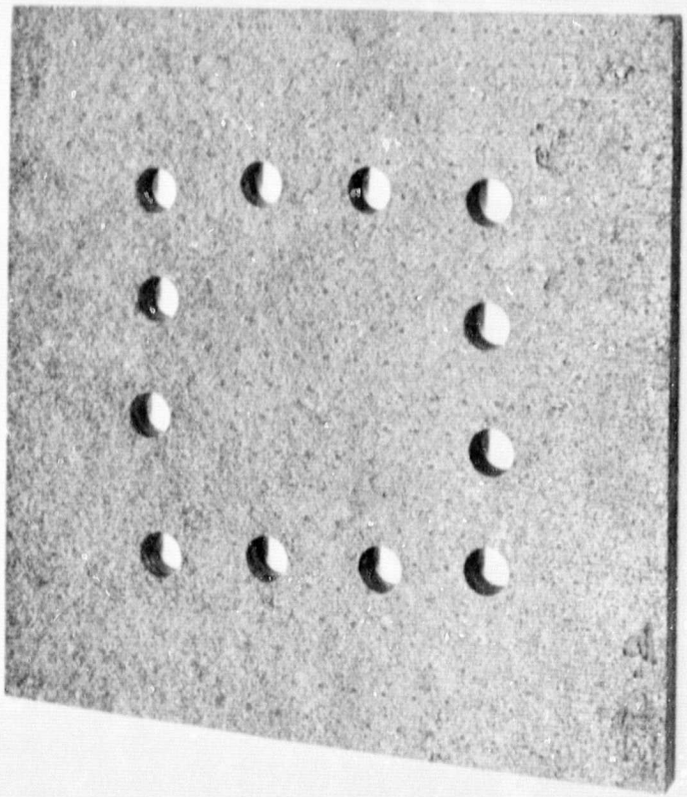
2B

Figure 37 Hybrid System #2b, Before and After Burn Test



2C

Figure 38 Hybrid System #2c, Before and After Burn Test



2D

Figure 39 Hybrid System #2d, Before and After Burn Test

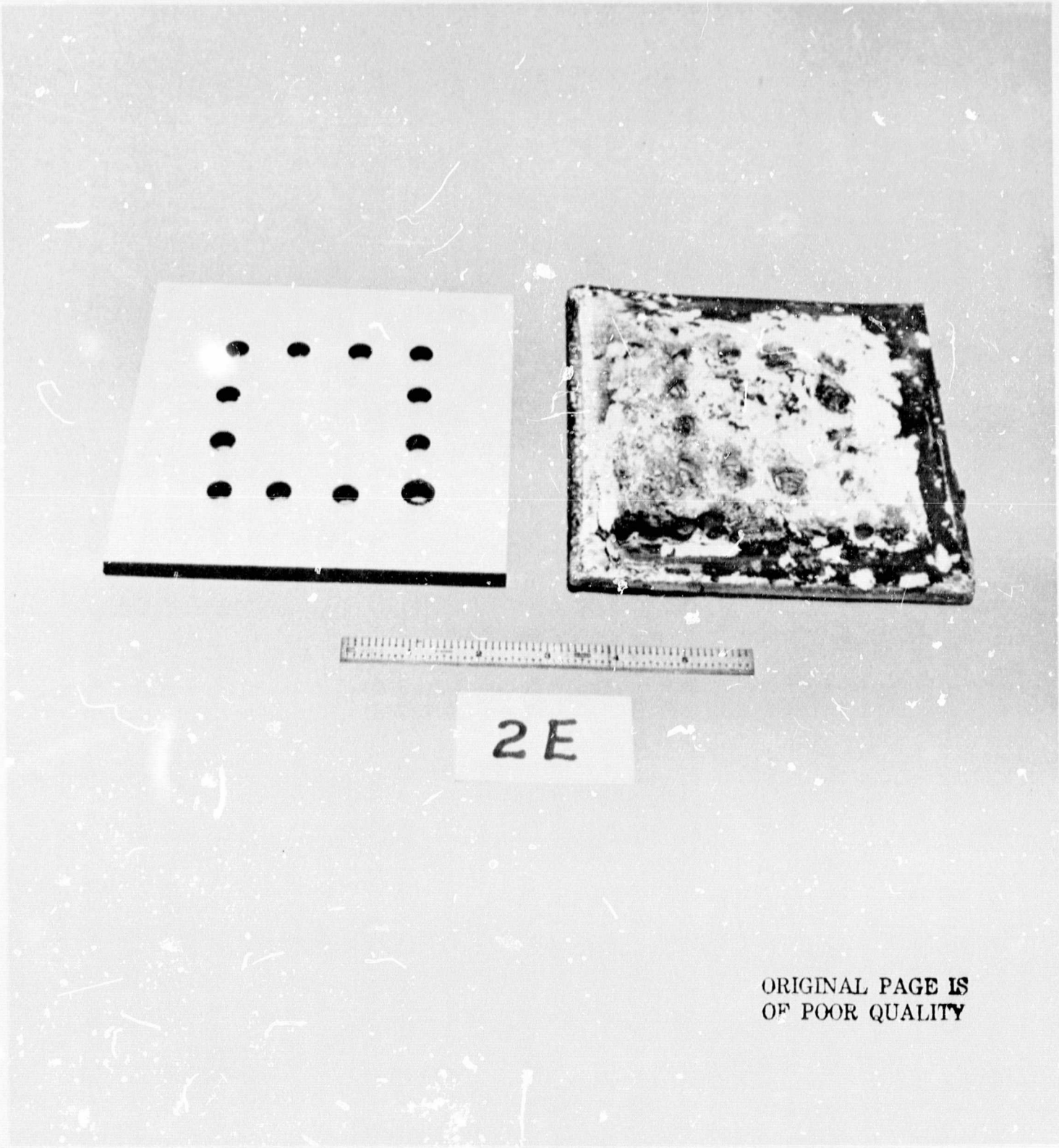


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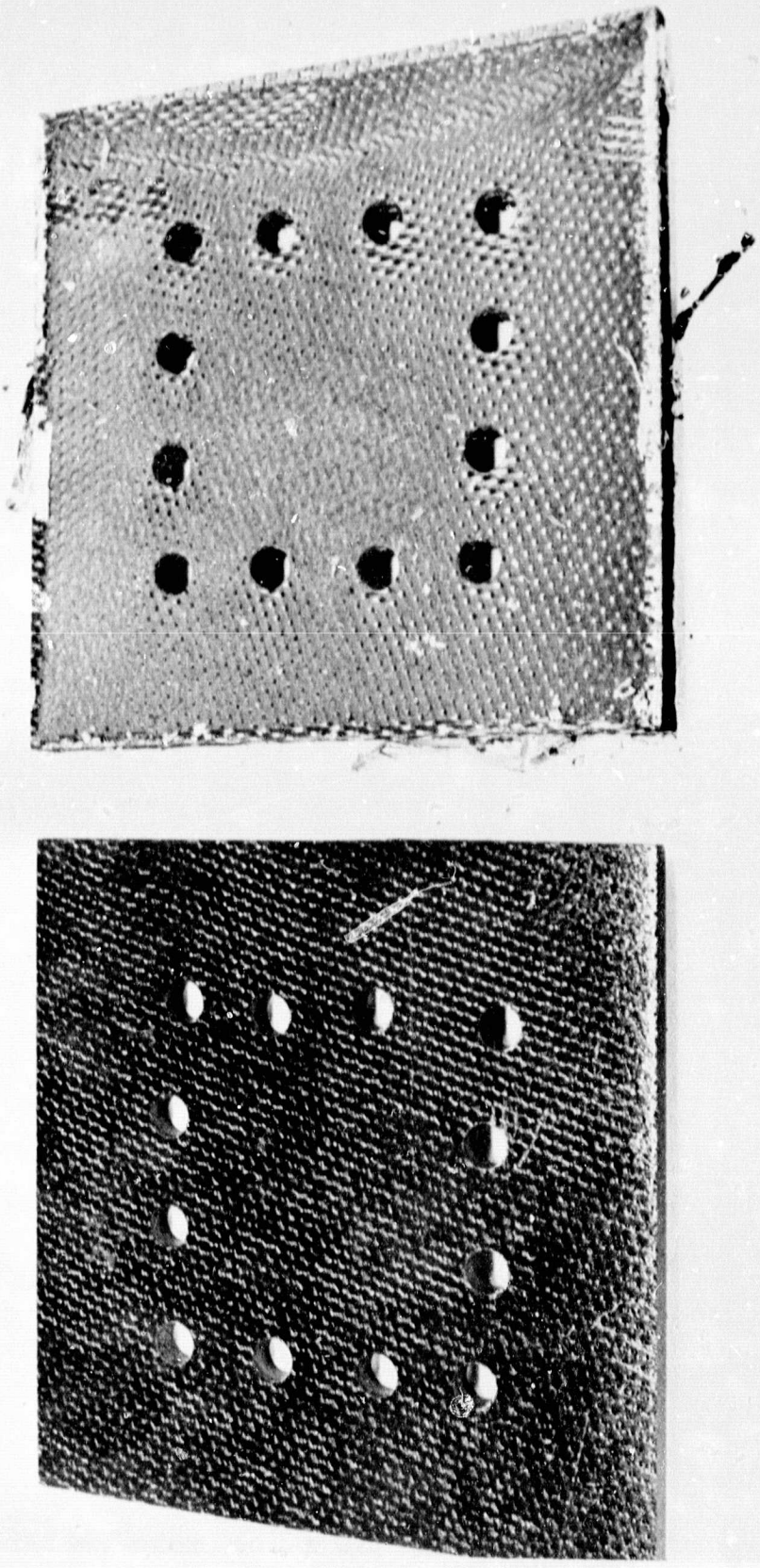
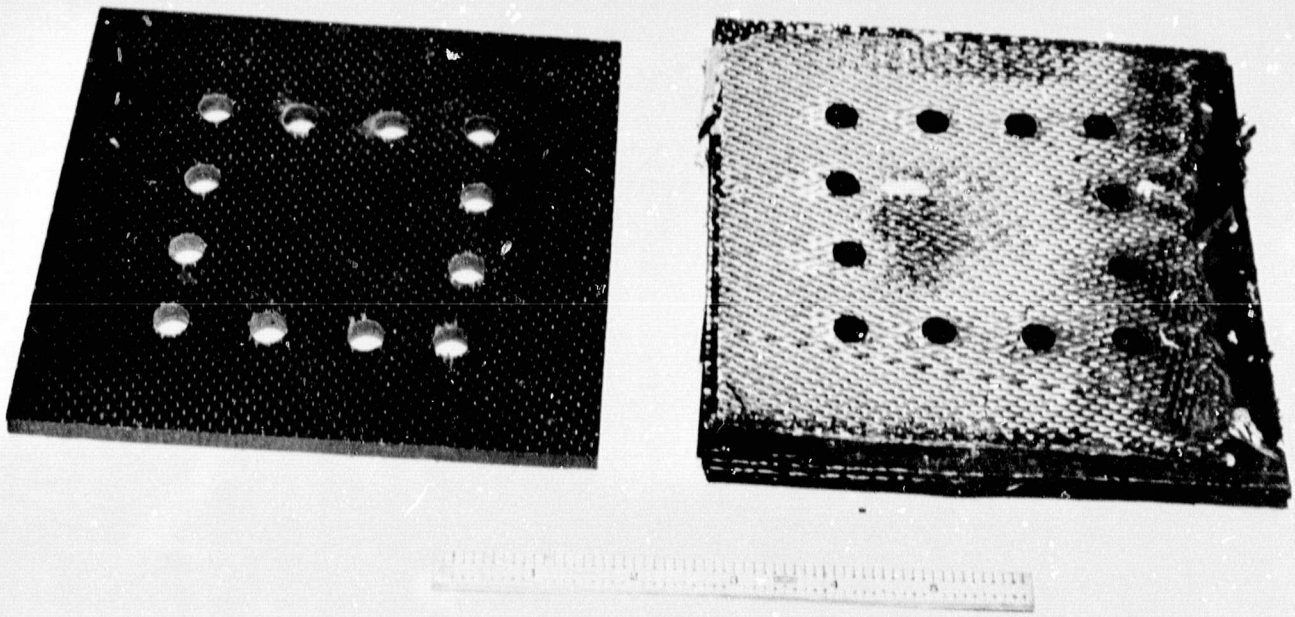
Figure 40 Hybrid System #2e, Before and After Burn Test



2F

Figure 41 Hybrid System #2f, Before and After Burn Test



2G

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Figure 42 Hybrid System #2g, Before and After Burn Test

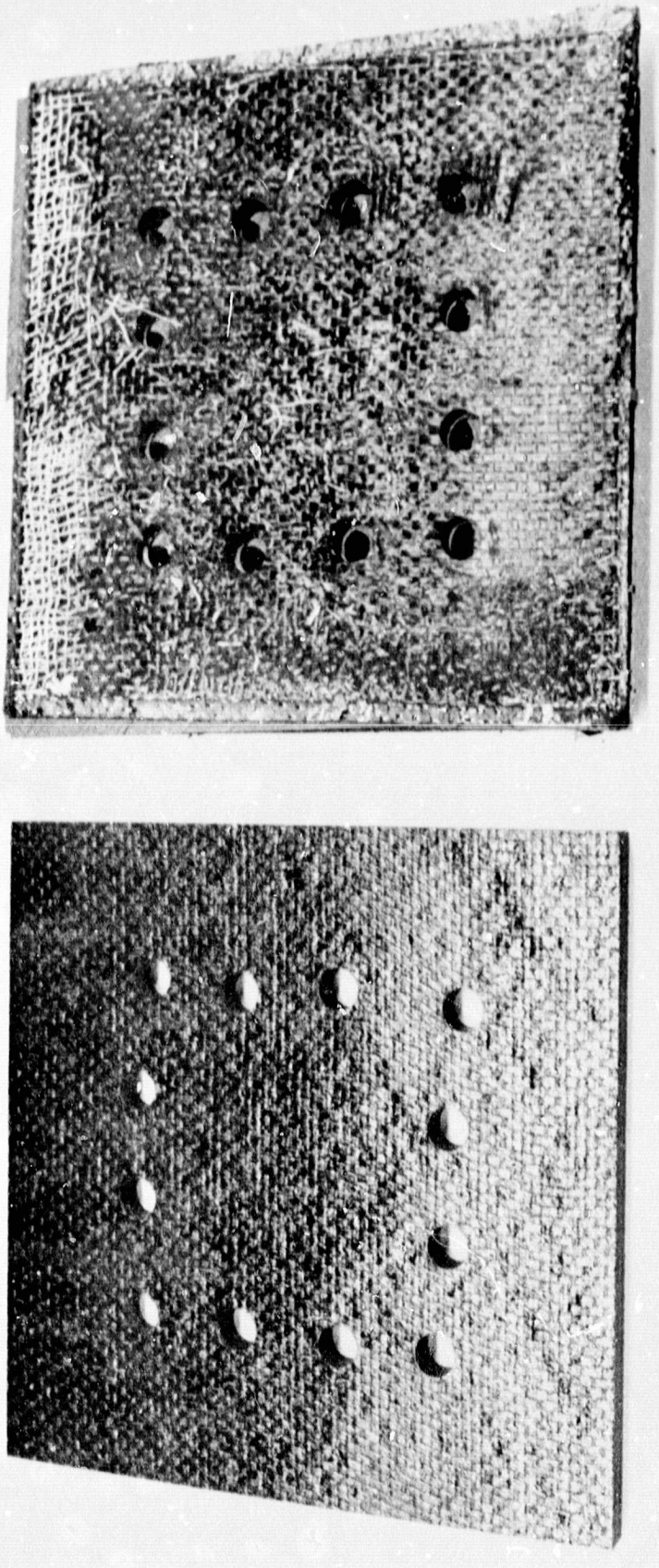
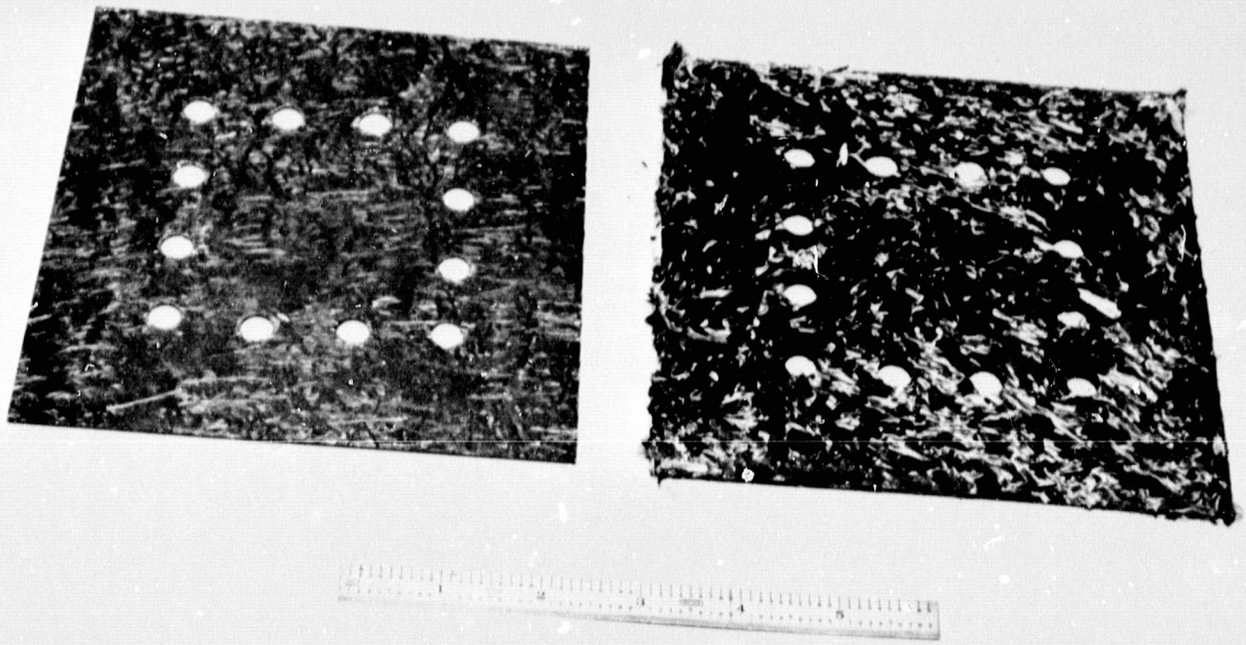
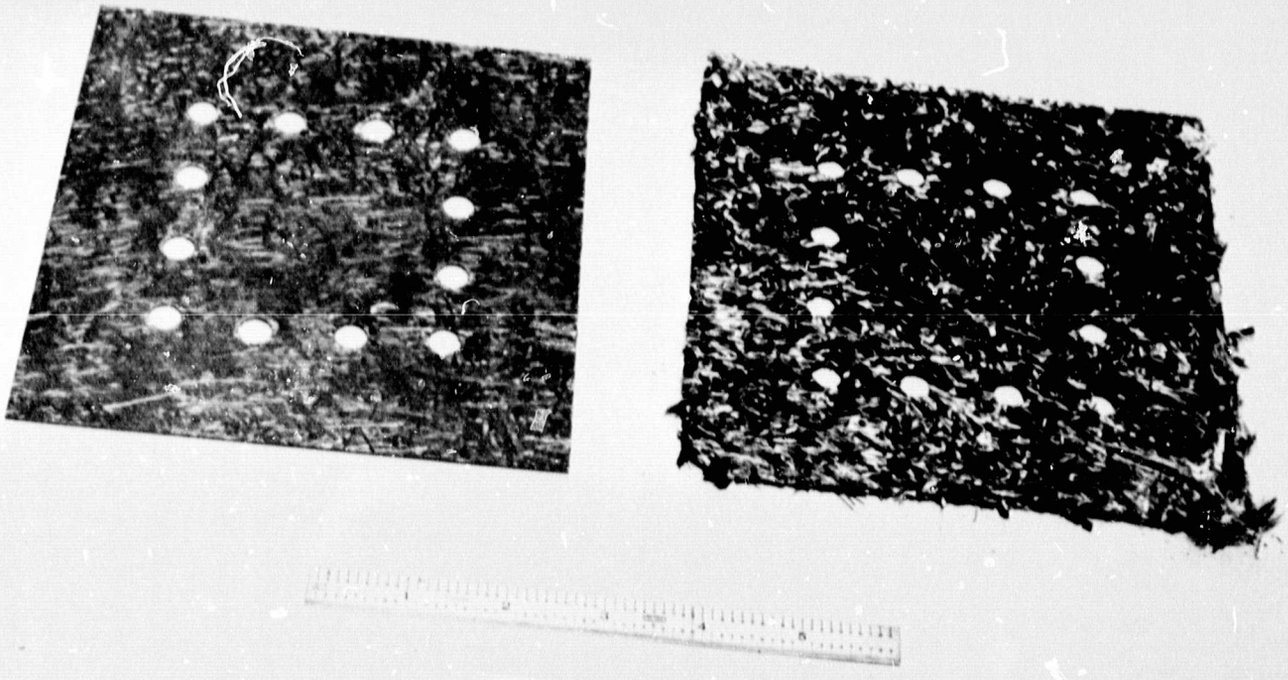


Figure 43 Hybrid System #2h, Before and After Burn Test



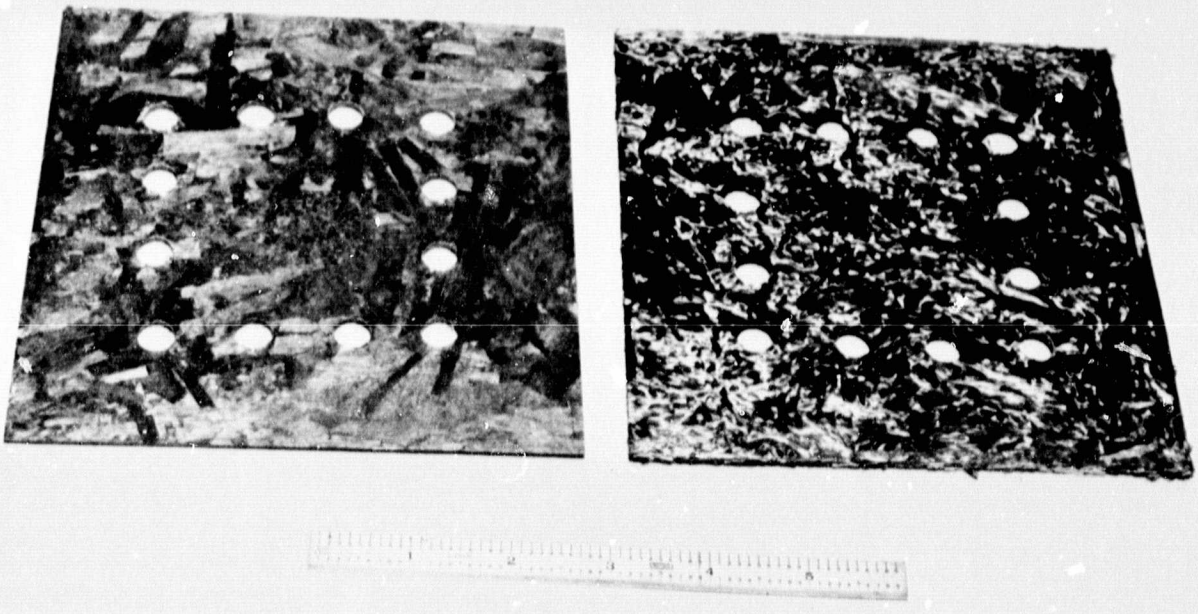
3

Figure 44 Baseline System #3, Before and After Burn Test



3A

Figure 45 Hybrid System #3a, Before and After Burn Test



3B

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Figure 46 Hybrid System #3b, Before and After Burn Test

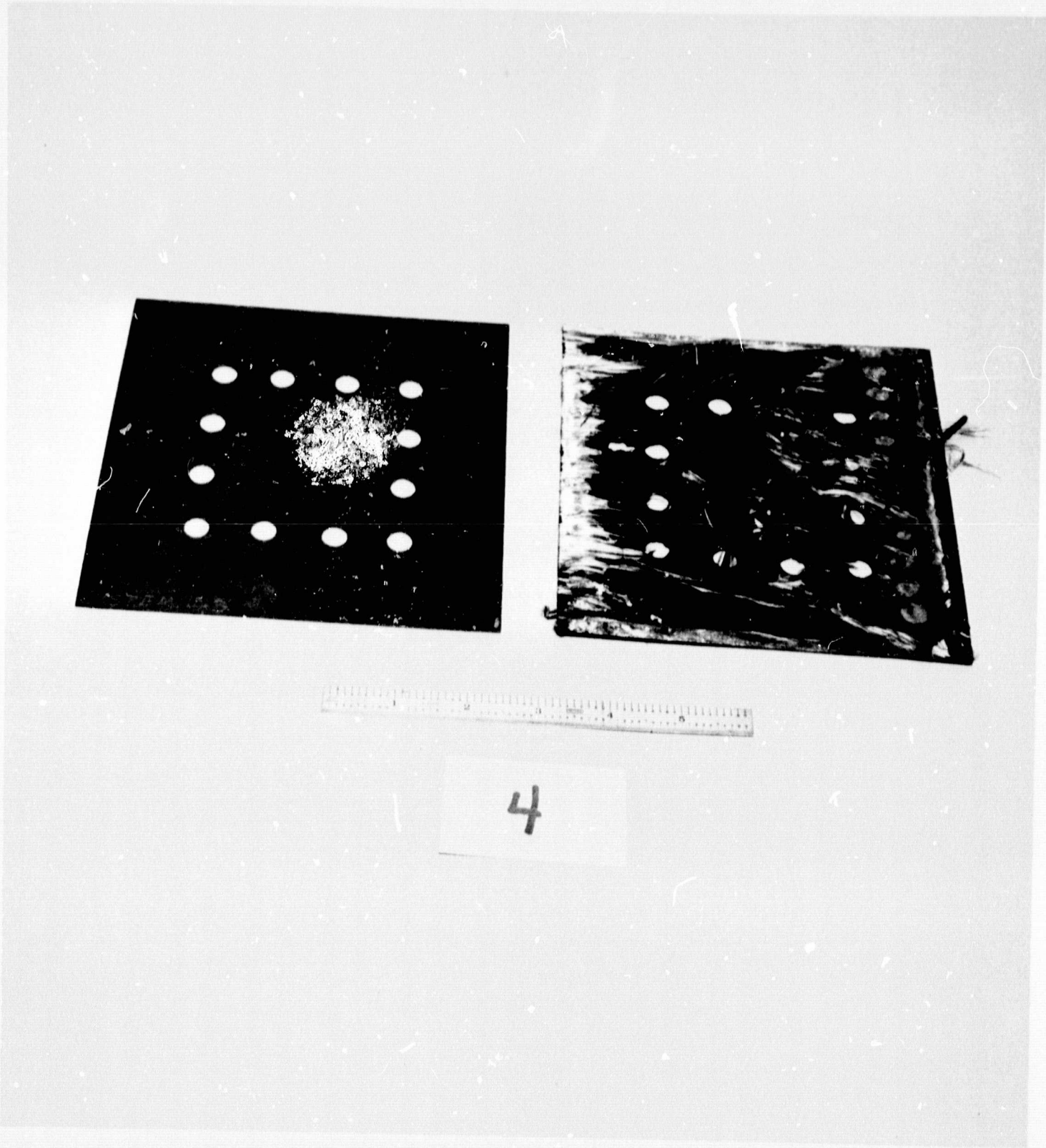

Figure 47 Baseline System #4, Before and After Burn Test

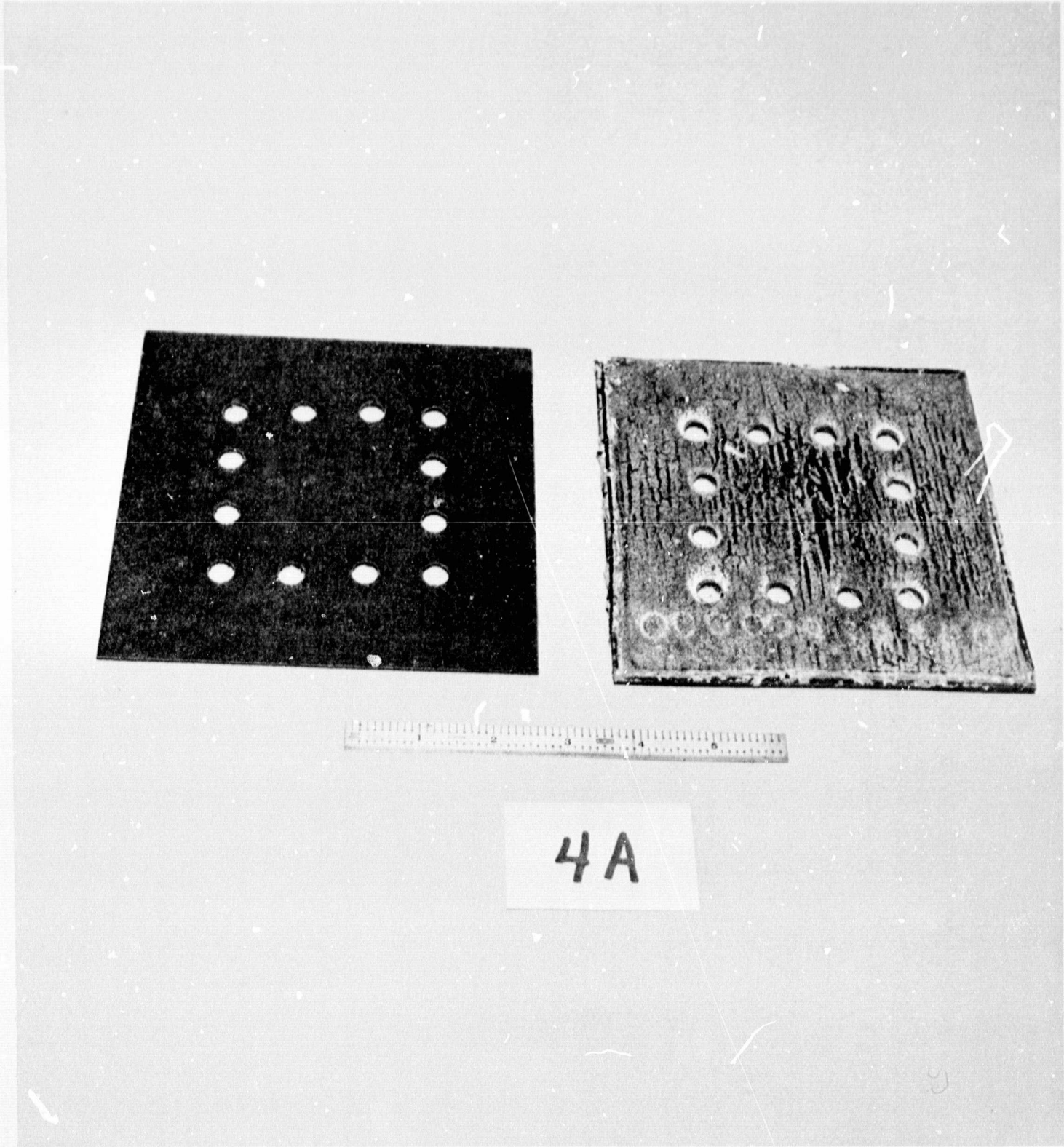

Figure 48 Hybrid System #4a, Before and After Burn Test

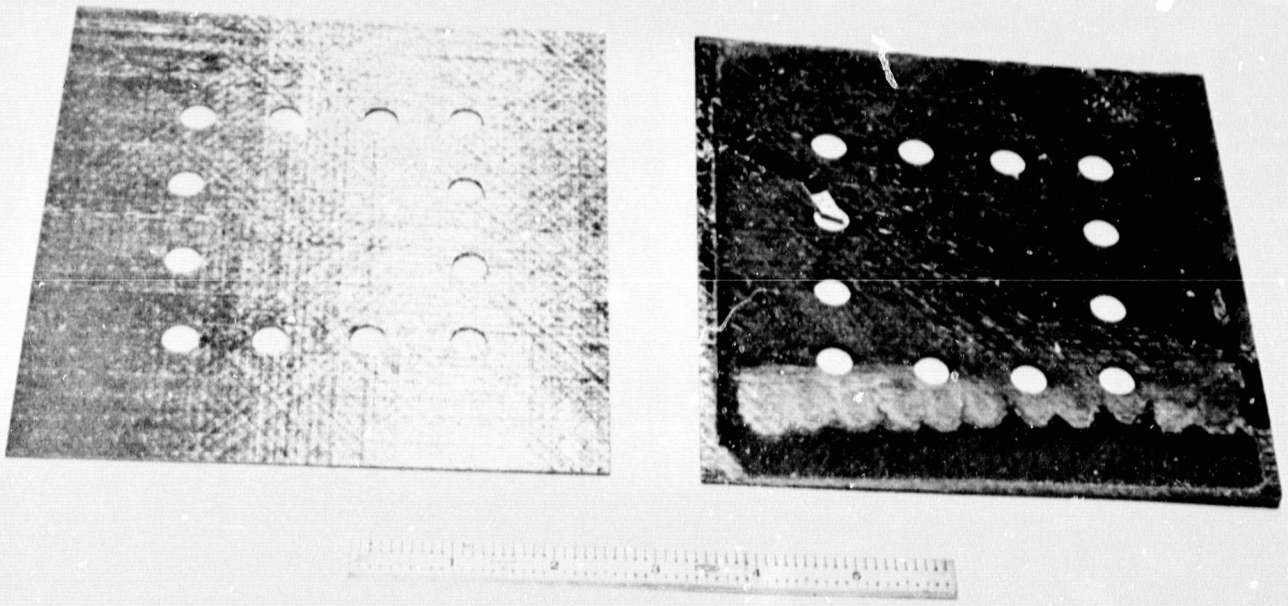

Figure 49 Baseline System #5, Before and After Burn Test

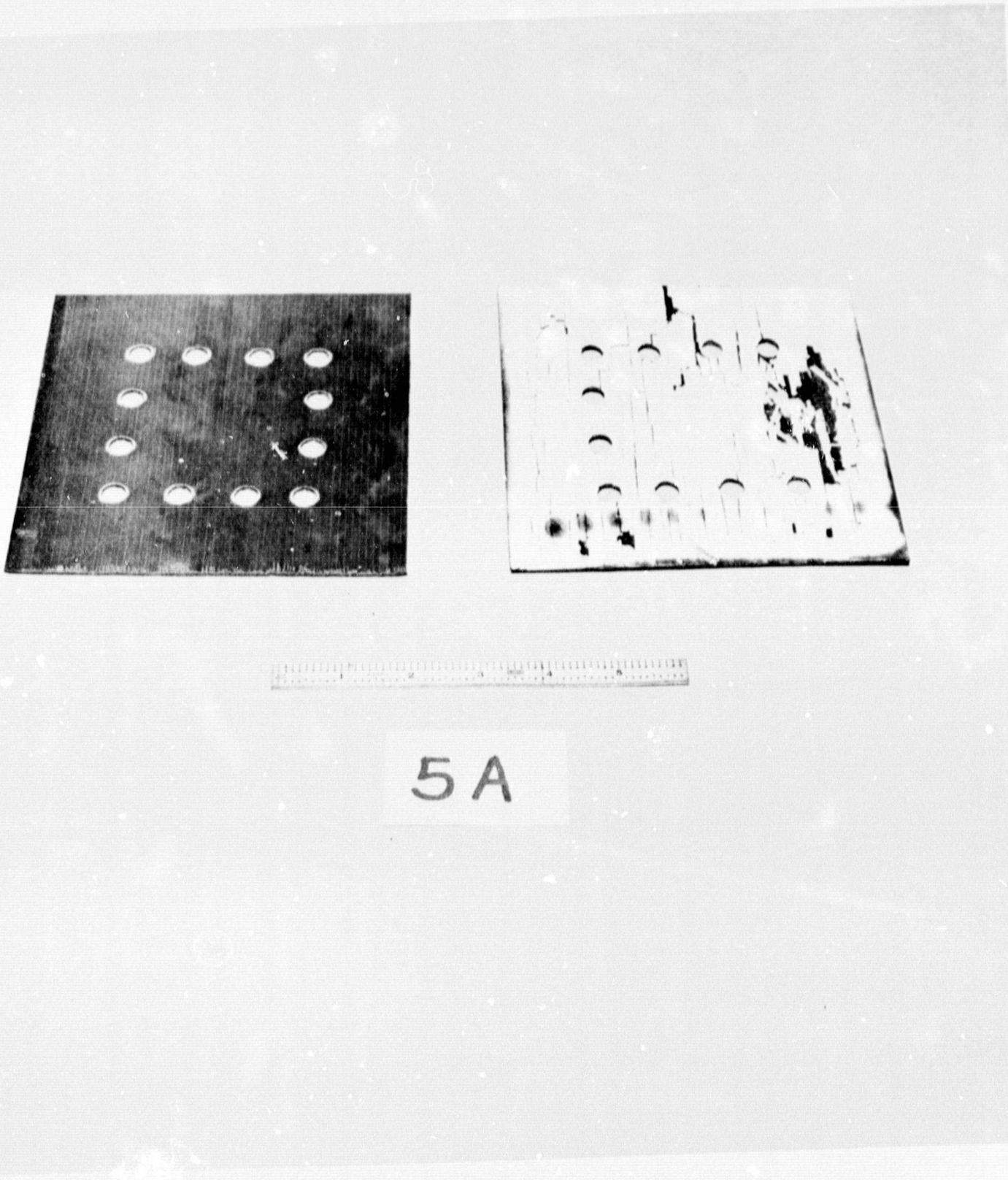

Figure 50 Hybrid System #5a, Before and After Burn Test

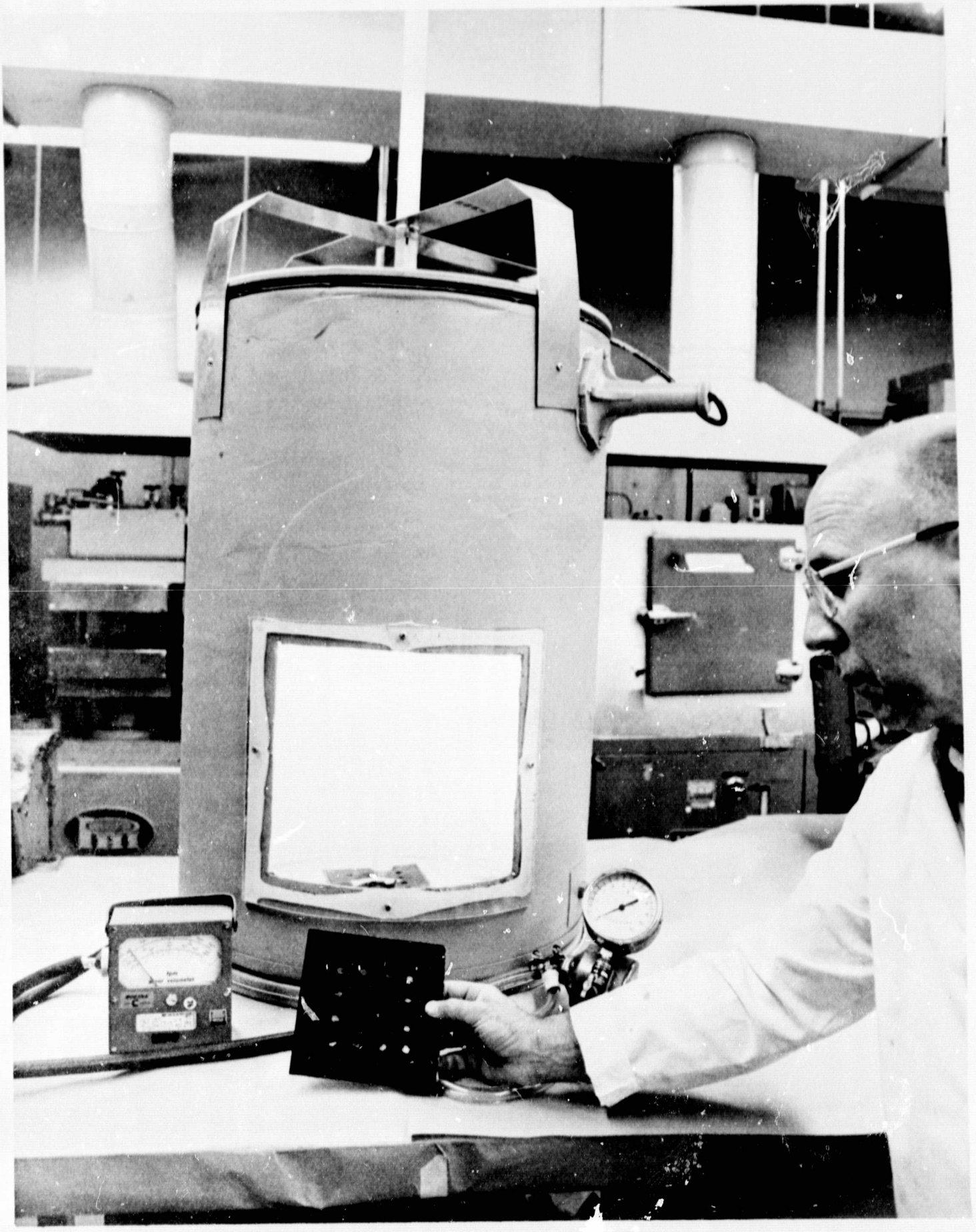

Figure 51 Impact and Air Flow Test Chamber

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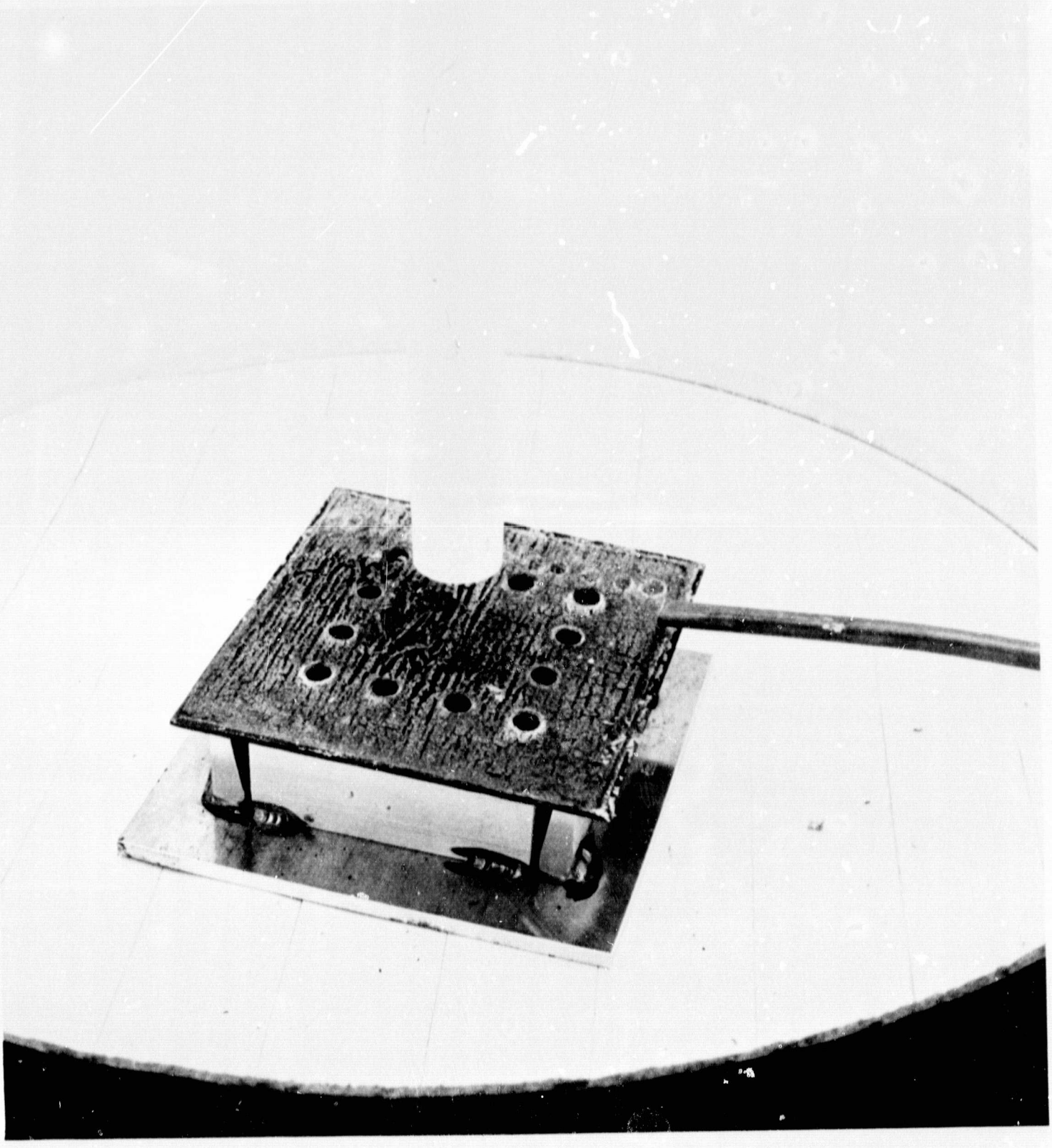

Figure 52 Specimen Positioned in Impact and Air Flow Test Chamber

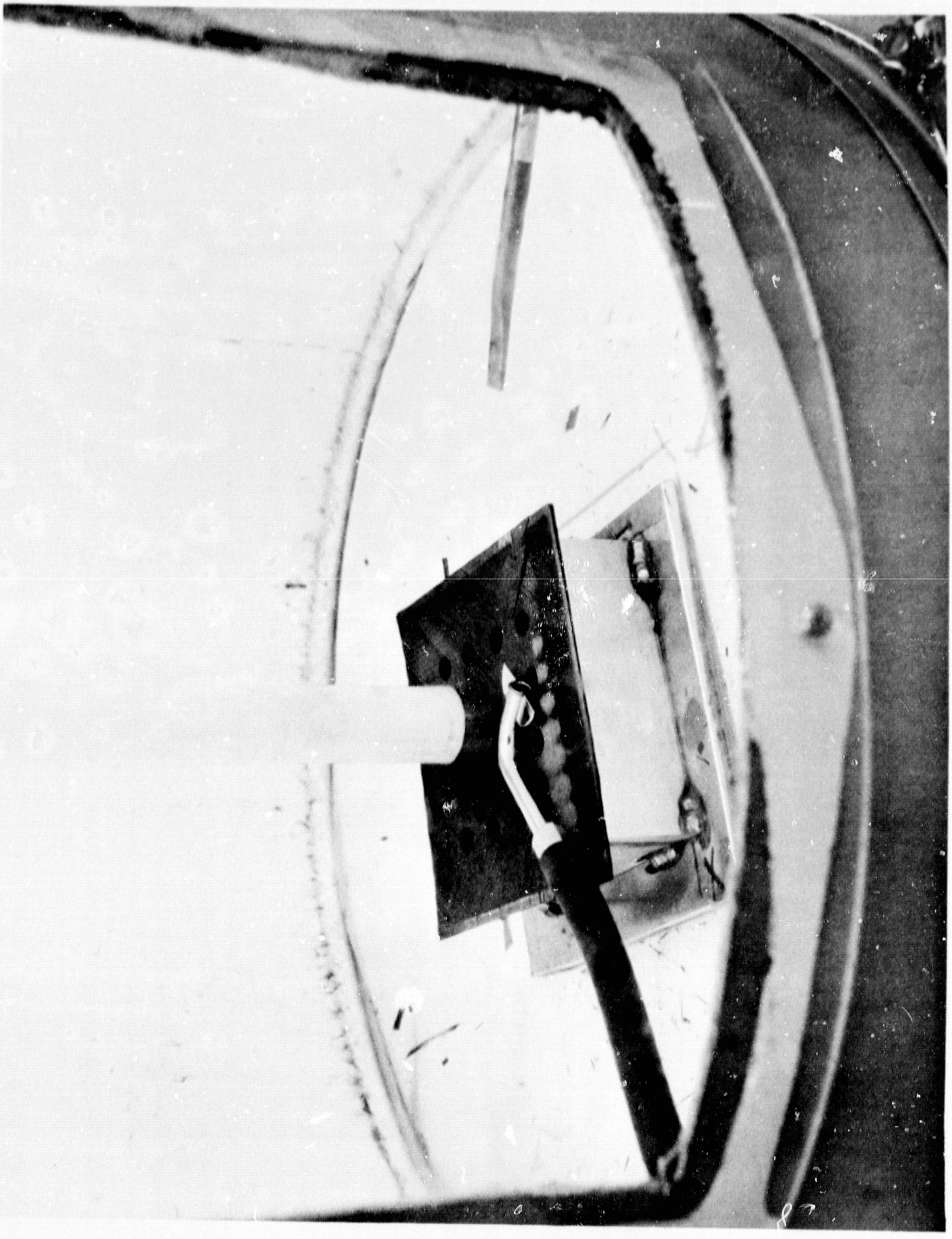

Figure 53 Specimen in Impact and Air Flow Test Chamber After Test

Figure 54 Baseline System #1 After Burn, Impact, and Air Flow Test

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Figure 55 Hybrid System #1a After Burn, Impact, and Air Flow Test

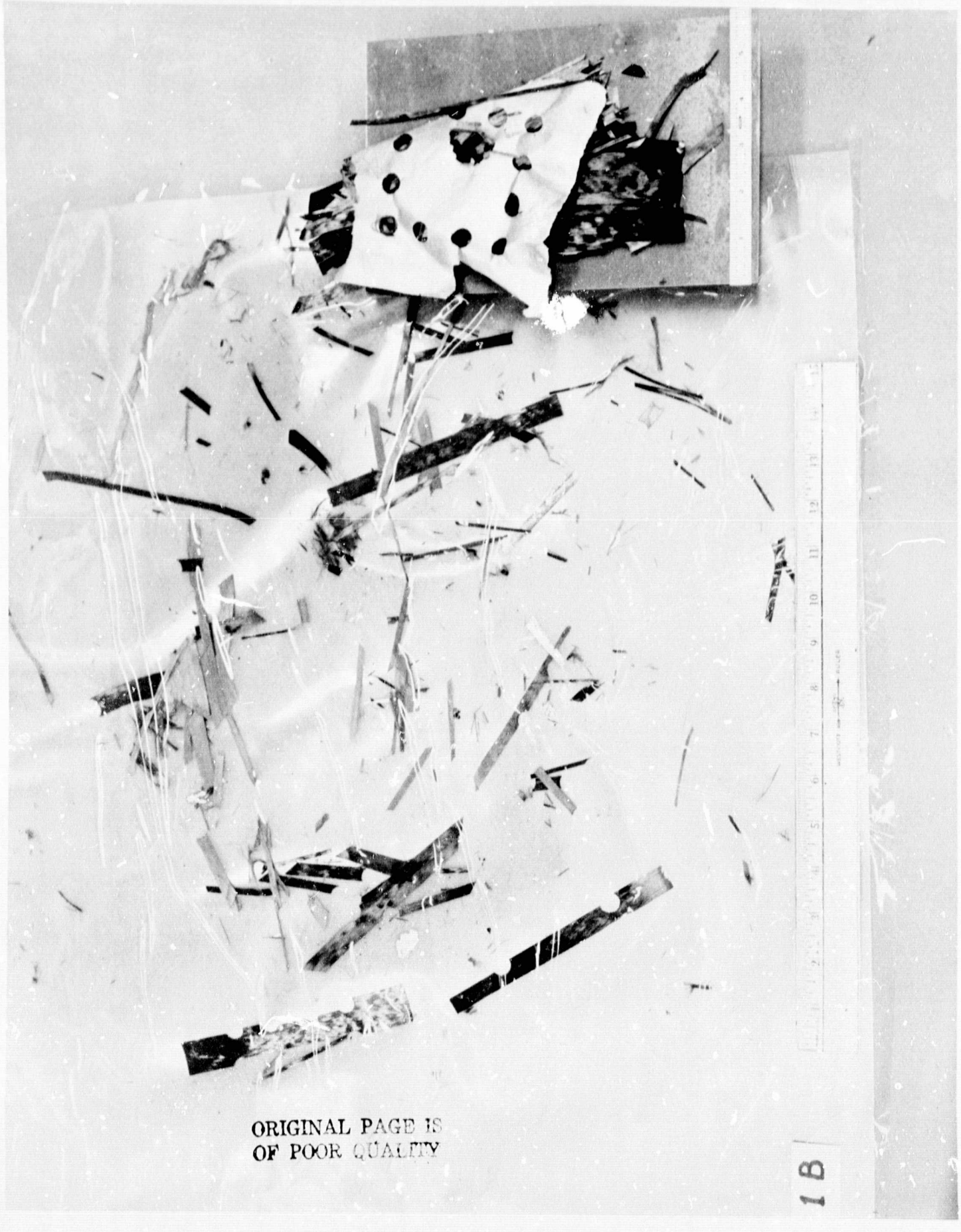

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Figure 56 Hybrid Syster #1b After Burn, Impact, and Air Flow Test

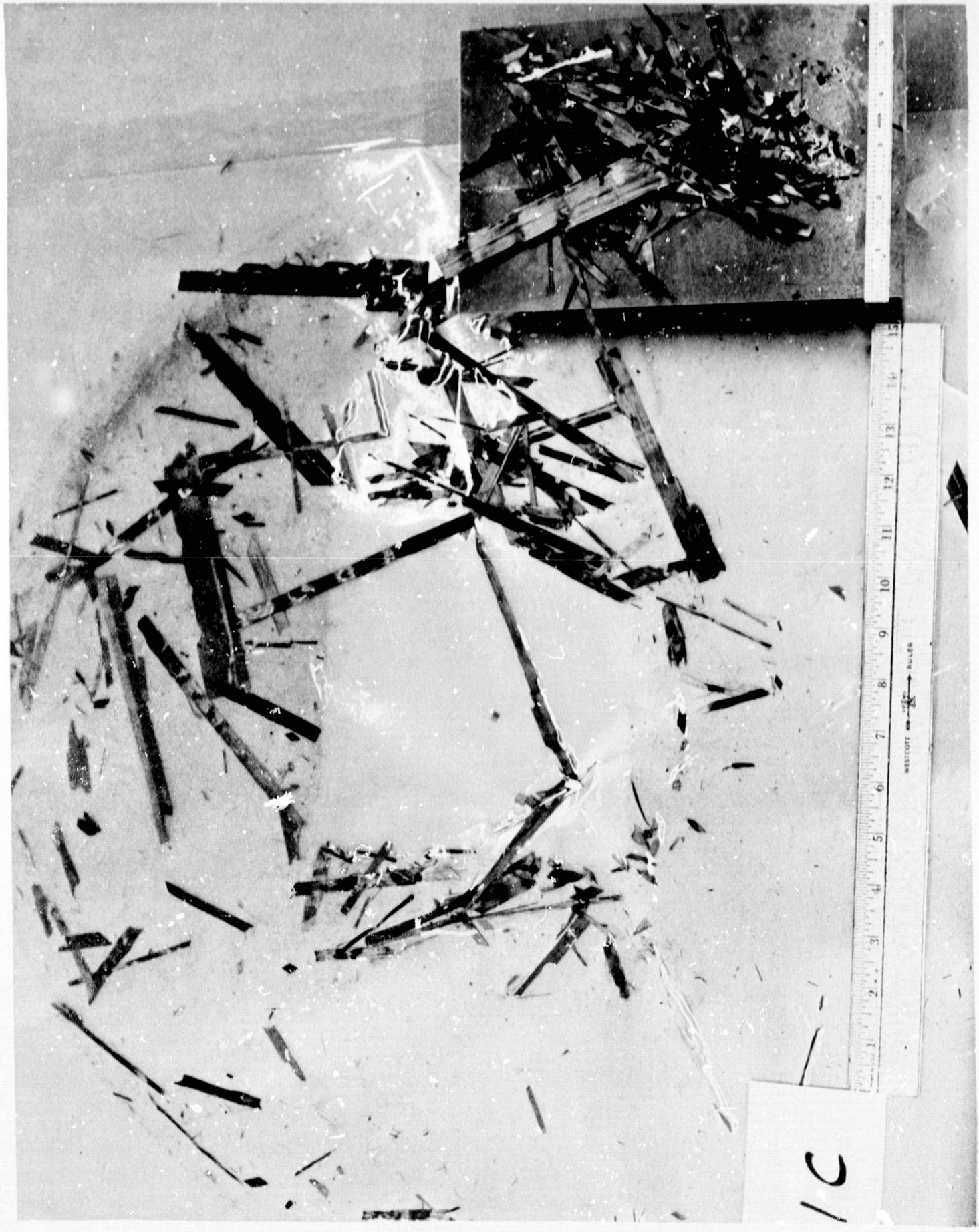

Figure 57 Hybrid System #1c After Burn, Impact, and Air Flow Test

Figure 58 Hybrid System #1d After Burn, Impact, and Air Flow Test

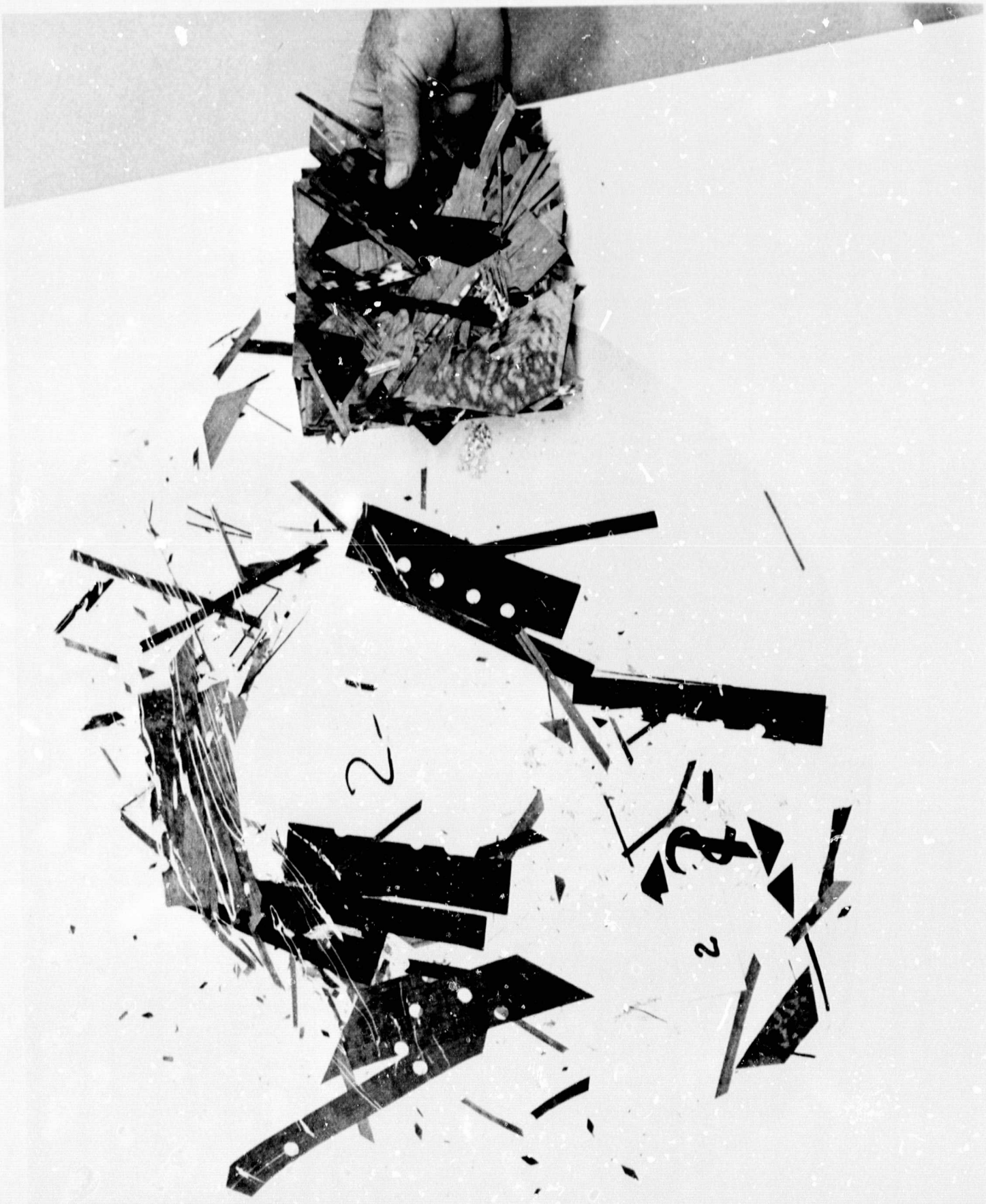

Figure 59 Baseline System #2 After Burn, Impact, and Air Flow Test

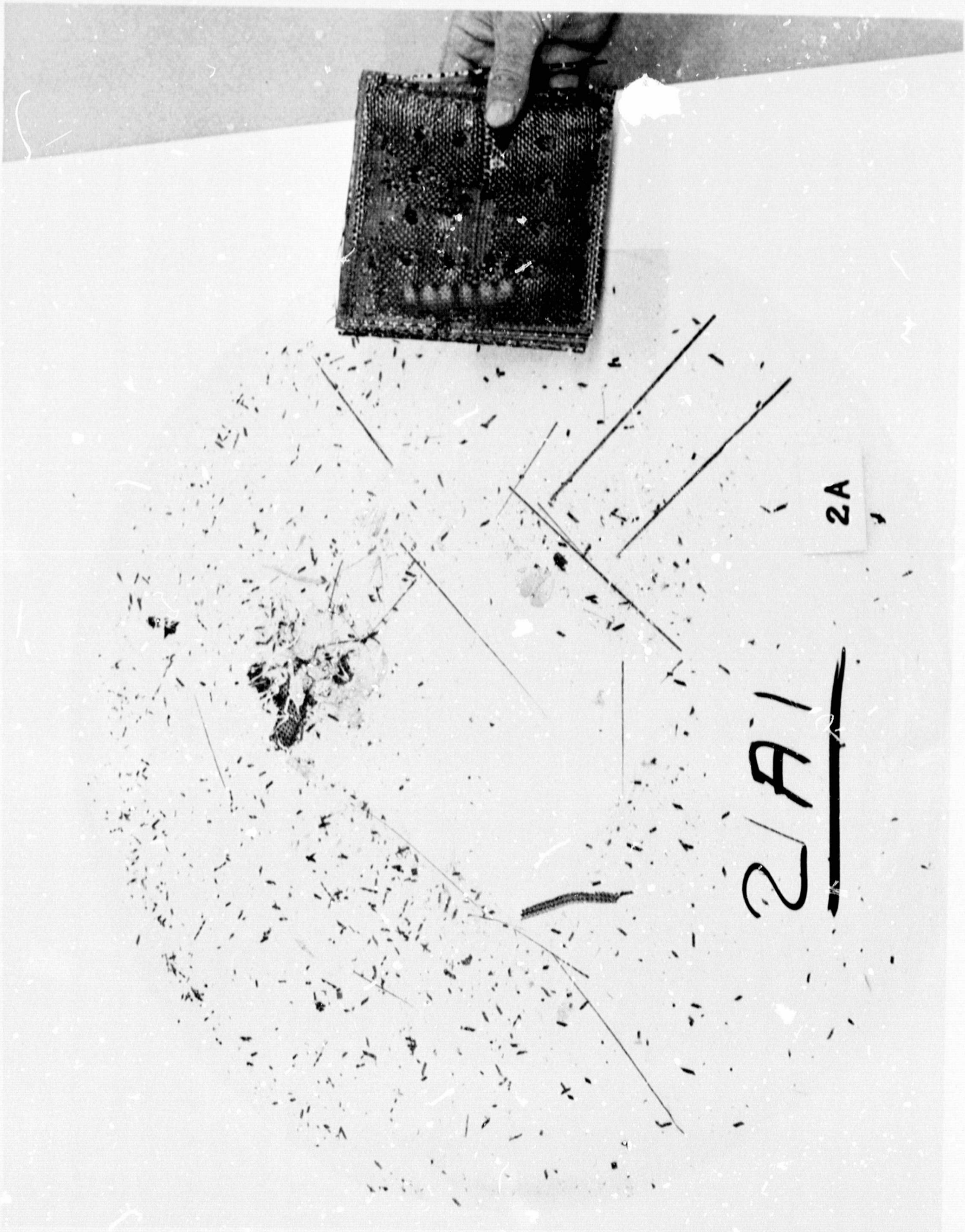

Figure 60 Hybrid System #2a After Burn, Impact, and Air Flow Test

Figure 61 Hybrid System #2b After Burn, Impact, and Air Flow Test

Figure 62 Hybrid System #2c After Burn, Impact, and Air Flow Test

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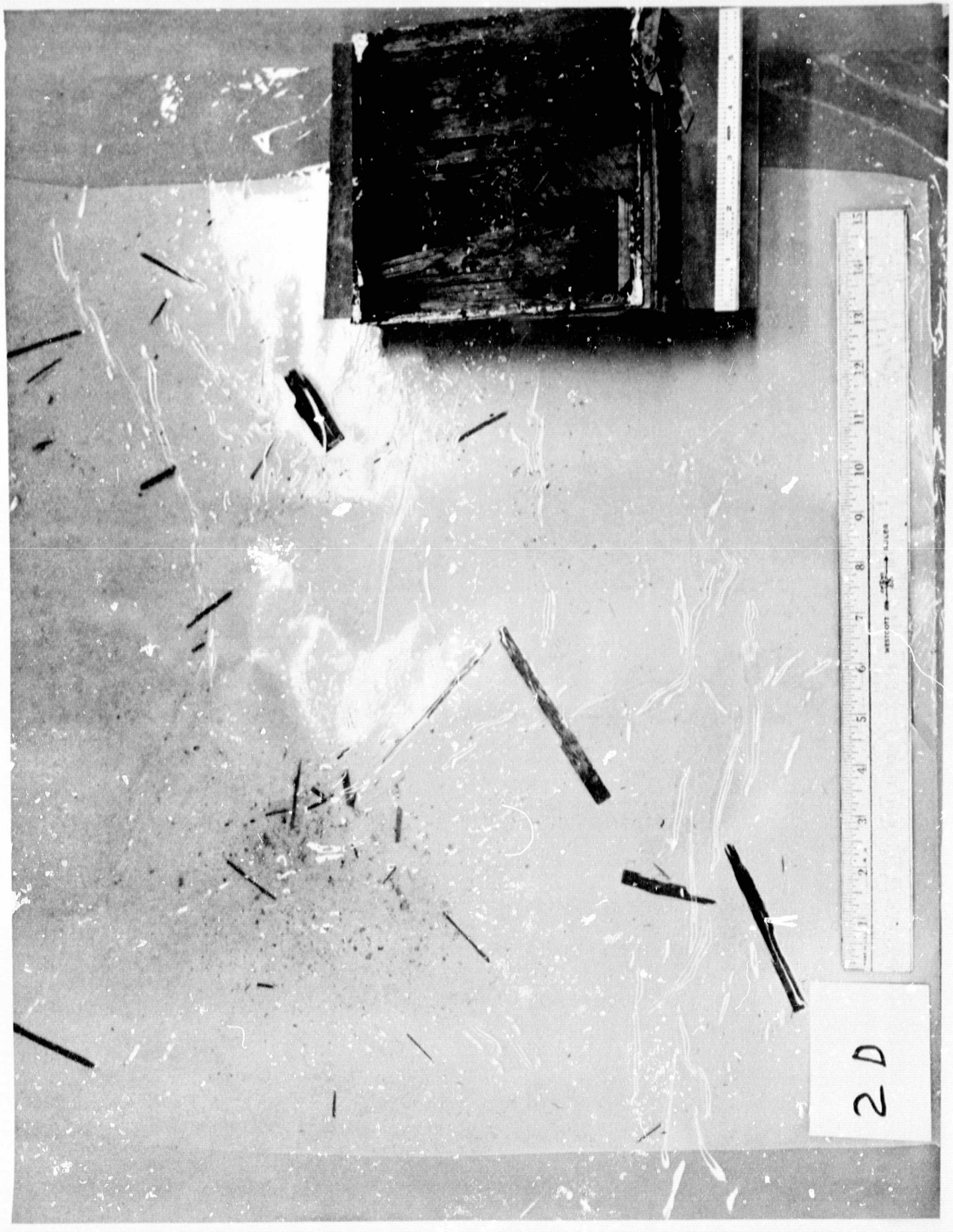

Figure 63 Hybrid System #2d After Burn, Impact, and Air Flow Test

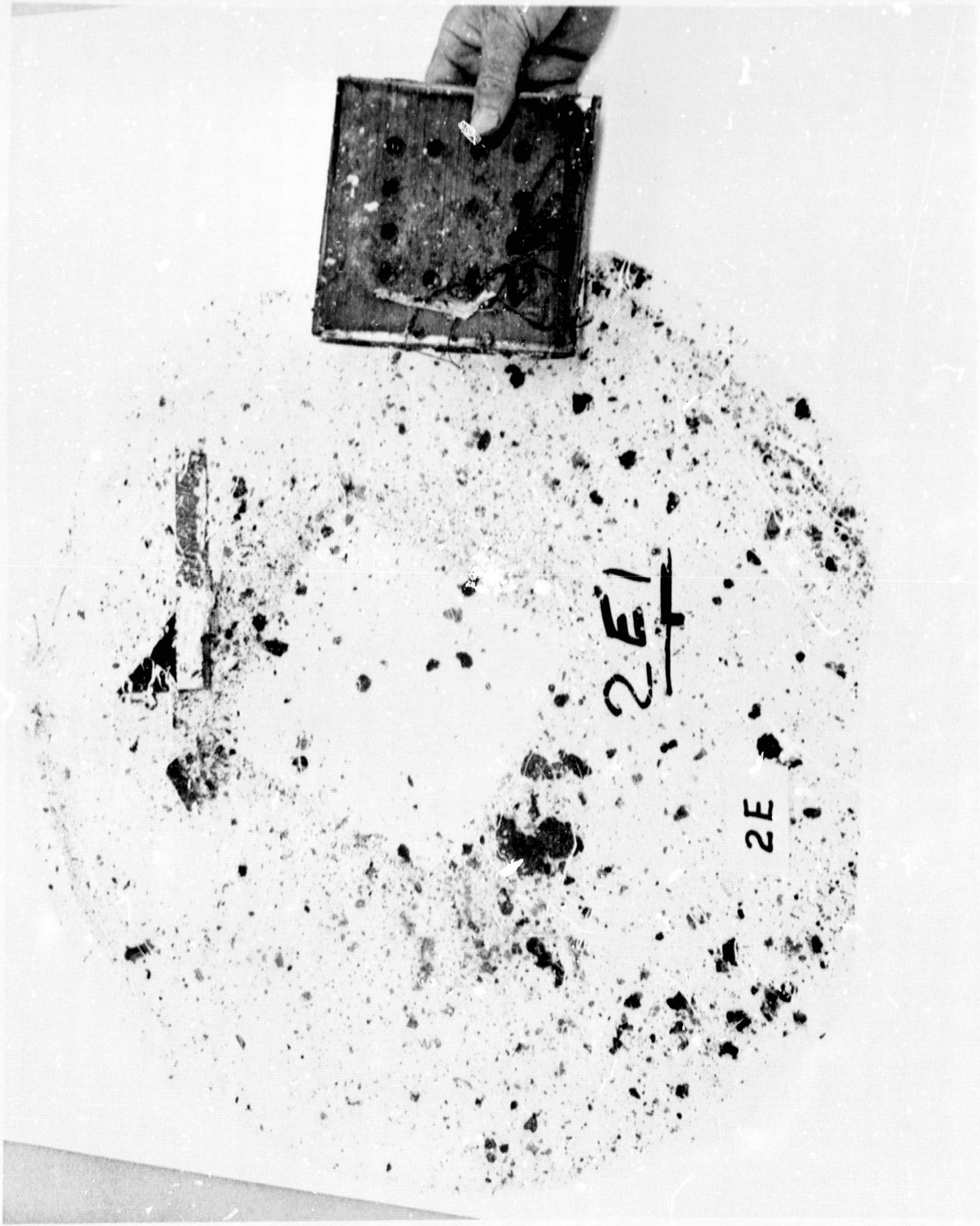

Figure 64 Hybrid System #2e After Burn Impact, and Air Flow Test

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Figure 65 Hybrid System #2f After Burn, Impact, and Air Flow Test

Figure 66 Hybrid System #2g After Burn, Impact, and Air Flow Test

Figure 67 Hybrid System #2h After Burn, Impact, and Air Flow Test

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Figure 68 Baseline System #3 After Burn, Impact, and Air Flow Test

C-2

Figure 69 Hybrid System #3a After Burn, Impact, and Air Flow Test

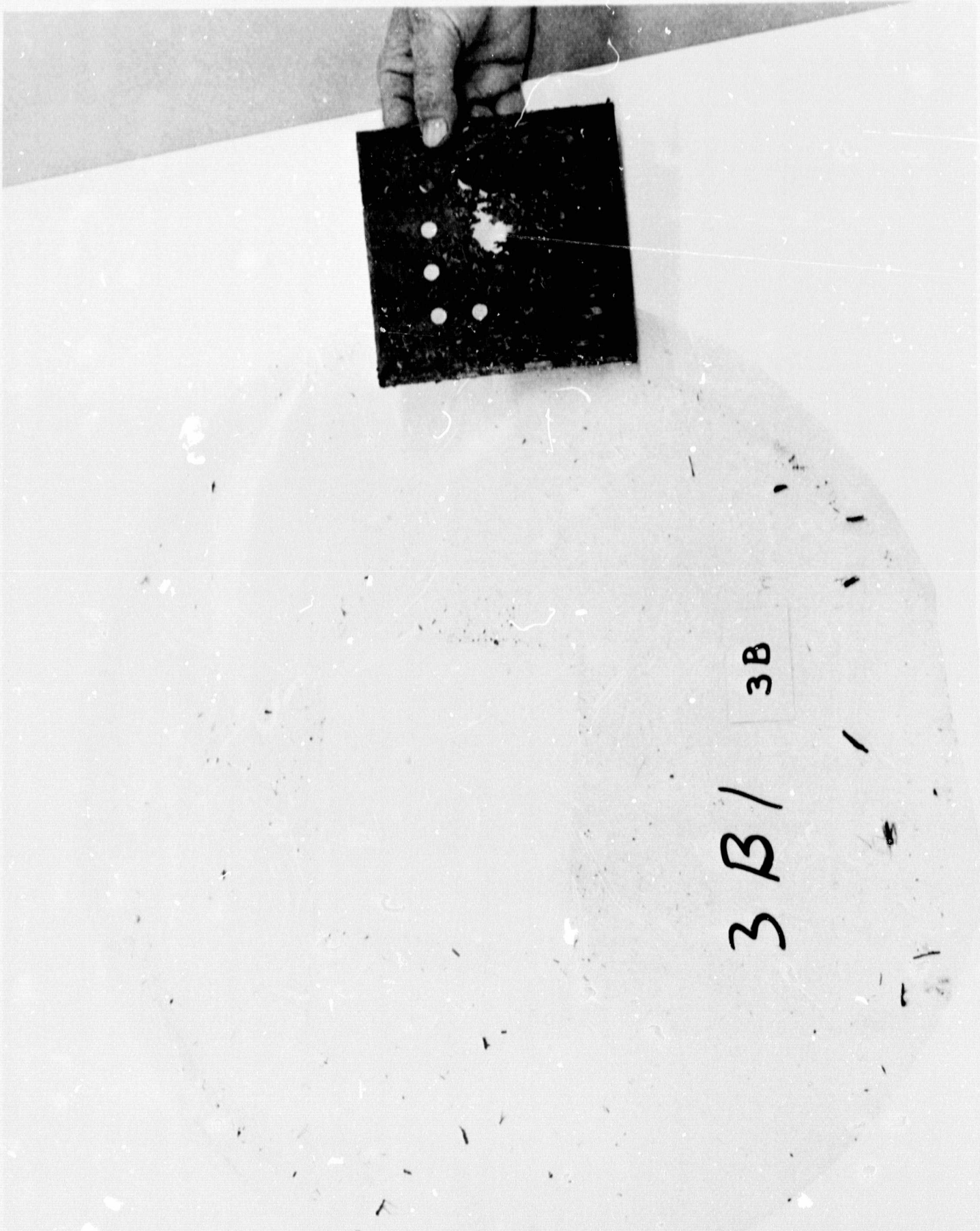

Figure 70 Hybrid System #3b After Burn, Impact, and Air Flow Test

Figure 71 Baseline System #4 After Burn, Impact, and Air Flow Test

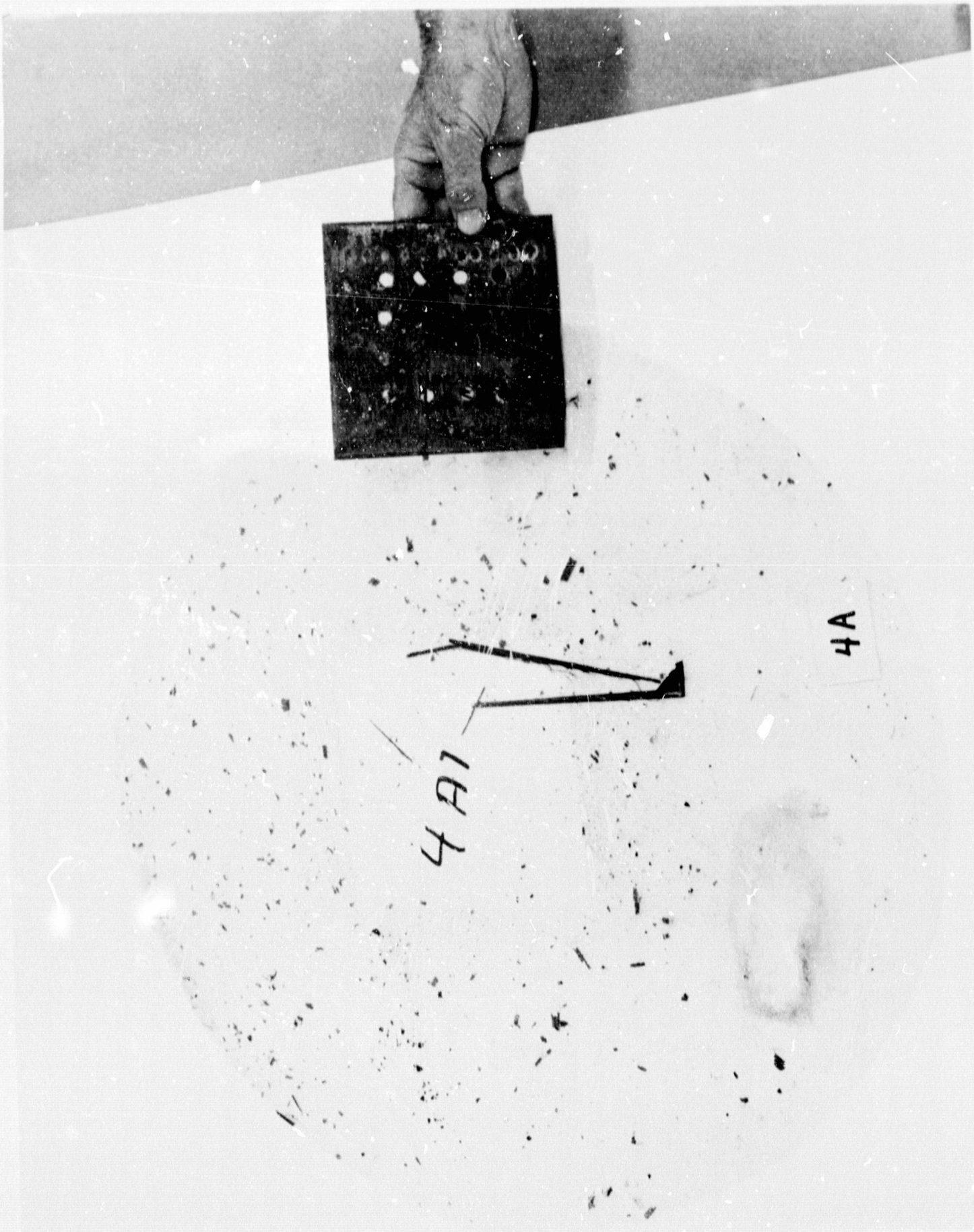

Figure 72 Hybrid System #4a After Burn, Impact, and Air Flow Test

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Figure 73 Baseline System #5 After Burn, Impact, and Air Flow Test

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Figure 74 Hybrid System #5a After Burn, Impact, and Air Flow Test

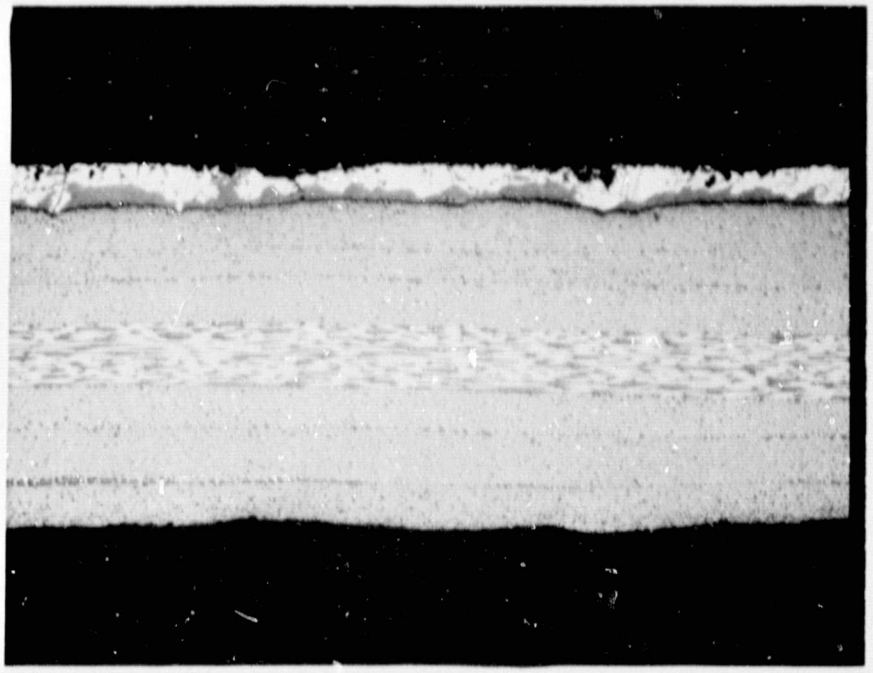

Figure 75 Selected Hybrid System #1a (50X)

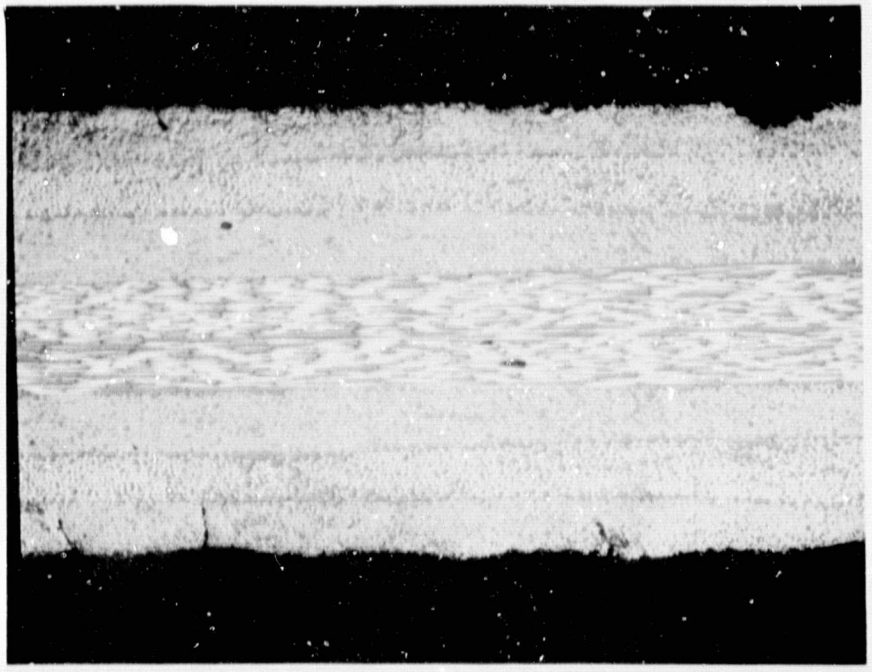

Figure 76 Selected Hybrid System #2a (50X)

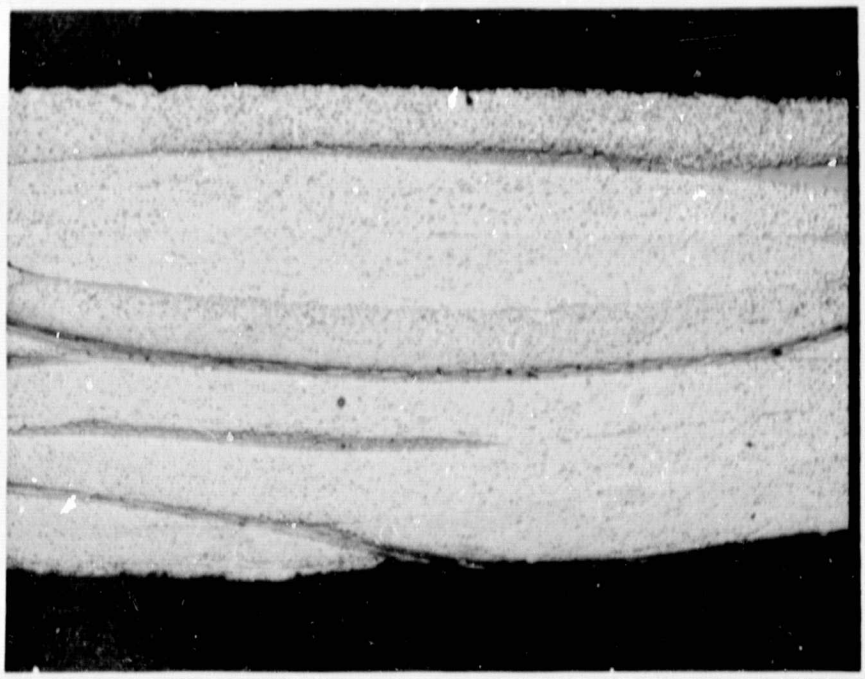

Figure 77 Selected Hybrid System #3a (50X)

Figure 78 Selected Hybrid System #4a (50X)

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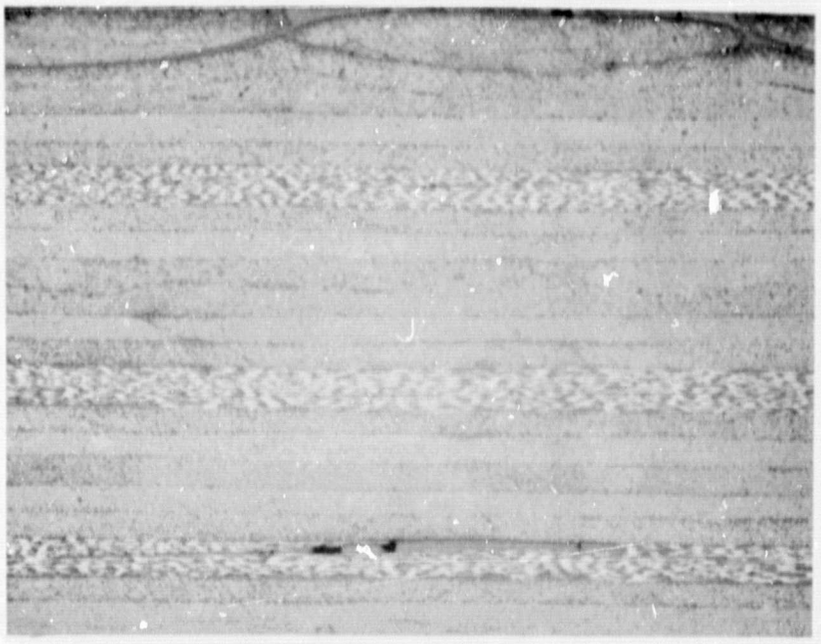

Figure 79 Selected Hybrid System #5a (25X)

Figure 80 Selected Hybrid System #6a (25X)

Figure 81 Selected Hybrid System #7a (25X)

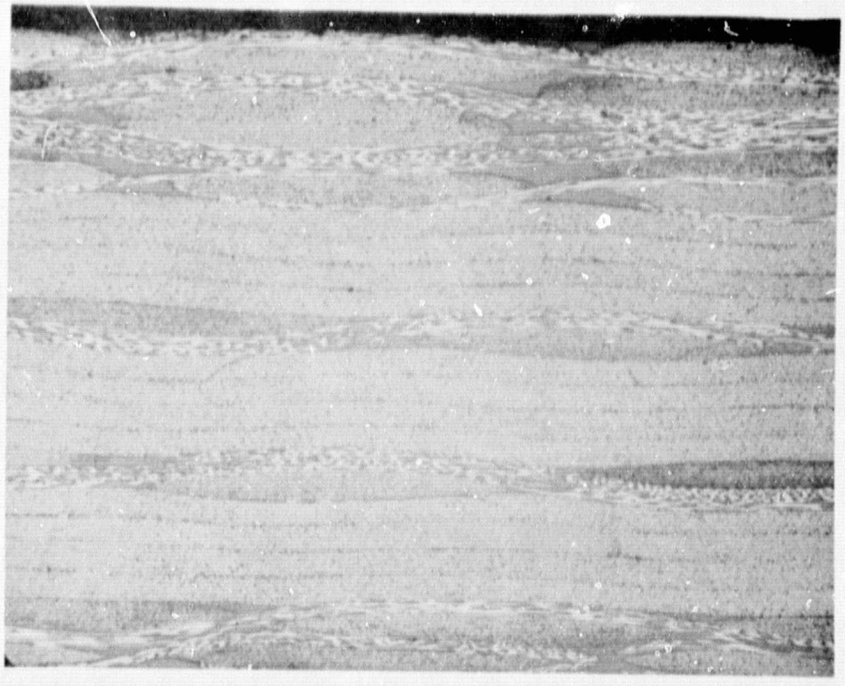

Figure 82 Selected Hybrid System #8a (25X)

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Table I - Baseline Laminate Candidates

Primary Resin/Fibers	Comments
Epoxy/Unidirectional	Most commonly used class on aircraft and space hardware.
Polysulfone/Unidirectional	Low-cost resin, structural adequacy demonstrated, projected space, aircraft, and missile application.
Polyimide/Unidirectional	High temperature service. Spacecraft exterior, supersonic missiles and aircraft and hot areas such as engines.
Polyester/Unidirectional	Low-cost resin, fast processing, aimed at high production runs.
Phenolic/Unidirectional	Low-cost resin, fast processing, moderate high temperature resistance.
Polysulfone/Chopped Fiber	Low-cost processing for high rate production.
Epoxy/Chopped Fiber	
Phenolic/Chopped Fiber	
Polyester/Chopped Fiber	
Polyimide/Chopped Fiber	Low-cost processing for high rate production.

Table II - Preliminary Burn Test Results

Panel #/ Figure #	Thickness, mm (in.)	Reinforcement	Orientation	Resin (Prepregger)	Test 3/ Duration (Minutes)	Remarks
1/3	1.60 (0.063)	Celion 6000	± 45°	PMR-15 polyimide (USP)	10	Resin apparently effects a certain amount of fiber containment as there was no disruption of fiber orientation. Matrix integrity was maintained to a limited degree.
2/4	2.95 (0.116)	Celion 6000 12.7 mm (0.5 in.) chopped fiber	Random	PMR-15 polyimide (USP)	10	Considerable panel integrity retained. Bare fibers exposed on surface, but interlocking and possibly matrix char kept them in place.
3/5	2.85 (0.112)	T300	0°	934 epoxy (Fiberite)	10	No disruption in orientation.
4/6	1.07 (0.042)	T300 (fabric)	0°	934 epoxy (Fiberite) gabric.	10	Matrix completely degraded, but no fiber release due to mechanical interlocking of
5/7	1.14 (0.045)	AS	± 45°	P1700 polysulfone (Hercules)	10	Matrix completely degraded. Fibers are easy to remove from panel.
6/8	3.05 (0.120)	T300, 12.7 mm (0.5 in.) chopped fiber)	Random	934 epoxy (Fiberite)	10	Considerable less panel integrity than Panel #2. Fiber release is less severe than anticipated because of mechanical interlocking.
7/9	1.47 (0.058)	T300 (fabric)	0°	P1700 polysulfone (Hexcel)	10	Mechanical interlocking contains reinforcement. No structural integrity retained.
8/10	1.73 (0.068)	Celion 6000	0 ± 45°	PMR-15 polyimide (USP)	8	Panel burned for 4.5 minutes. 1/
9/11	1.73 (0.068)	Celion 6000	0 ± 45°	PMR-15 polyimide (USP)	8	Panel burned for 4 minutes. 2/
10/12	3.56 (0.140)	Celion 6000	0 ± 45°	PMR-15 polyimide (USP)	12	Panel burned for 7 minutes. 1/
11/13	3.56 (0.140)	Celion 6000	0 ± 45°	PMR-15 polyimide (USP)	16	Panel burned for 6 minutes. 2/
12/14	2.92 (0.115)	T300 (fabric)	0°	MXG-6073 phenolic (Fiberite)	20	Panel burned for 4 minutes. 1/
13/15	2.92 (0.115)	T300 (fabric)	0°	MXG-6073 phenolic (Fiberite)		
14/16	0.86 (0.034)	T300 (fabric) Kevlar 49-181	0°	MXG-6073 phenolic	20	Center portion of laminate broke away during test. 1/
15/17	0.86 (0.034)	T300 (fabric) Kevlar 49-181	0°	MXG-6073 phenolic (Fiberite)	8	Small flames near pilot flame. 2/
16/18	2.82 (0.111)	T300, 12.7 mm (0.5 in.) chopped fiber	Random	7175 epoxy (USP)	20	Panel face burned for 3.5 minutes. 1/
17/19	2.82 (0.111)	T300, 12.7 mm (0.5 in.) chopped fiber	Random	7175 epoxy	20	Panel face burned for 3.5 minutes. 2/

1/ Panel flawed with 9.35 mm (0.375 in.) diameter holes prior to burning.
2/ Panel identical to next lower numbered panel, except no holes.
3/ All exposures at 2 watts/cm^2 plus direct flame impingement from 12 flamelets, 12.7 mm (0.5 in.) spacing, along the bottom of the laminate.

Table III - Fiber Release vs Matrix Material and Reinforcement Form

Matrix	Fiber Release on Burning	Reinforcement
polysulfone	most	chopped fiber
epoxy	↑	unidirectional
polyimide	↑	fabric
phenolic	least	

Table IV - Baseline Laminates

System #	Resin Supplier	Reinforcement (Orientation)	Laminate Thickness, mm (in.)	Reason for Selection
1	5208-Narmco (epoxy)	T-300; (0, ±45, 90)	1.016 to 1.524 (0.040 to 0.060)	Widespread usage; present and projected
2	5208-Narmco (epoxy)	T-300; (0, ±45, 90)	6.35 (0.250)	Widespread usage; present and projected
3	EM 7125-USP (epoxy)	T-300; 12.7-mm (0.05 in.) chopped fiber	1.016 to 1.524 (0.040 to 0.060)	Chopped fiber form of reinforcement may present the most serious fiber release problem
4	PMR-15 (polyimide)	Celion 6000; (0, ±45, 90)	1.016 to 1.524 (0.040 to 0.060)	High service temperature capability
5	HY-E-1008E-Fiberite (phenolic)	T-300; (0, ±45, 90)	1.016 to 1.524 (0.040 to 0.060)	Low-cost resin, fast processing, high usage potential

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Table V - Proposed Hybridizing Concepts

MECHANICAL INTERLOCKING

- Weaving
- Stitching

  Glass
- Cladding
- Ply Stacking
- Intralaminar Mix

COATINGS

- Heat Barrier
- Oxygen Exclusion
- Intumescent
- Char Formers
- Ablatives

MATRIX CONTROL

- Char-Forming
- Matrix Blending

  Exterior Ply

  Intralaminar
- Additives

Table VI - Preliminary Hybridizing Concepts

1.	Graphite fabric	(a) exterior plies (b) interplied
2.	Fiberglass cloth	(a) exterior plies (b) interplied
3.	Graphite/fiberglass cloth	(a) exterior plies (b) interplied
4.	Stitching	(a) prior to impregnation (b) after lay-up but prior to cure
5.	Intumescent coating	
6.	Glass-filled resin	(a) exterior coating (b) ply-by-ply coating
7.	Char-forming coating such as polyphenylene sulfide	(a) exterior coating (b) ply-by-ply coating
8.	Boron reinforcement	(a) exterior plies (b) interplied
9.	Matrix modification to increase char forming characteristics and/or promote clumping	

Table VII - Baseline and Hybrid Systems

No.	Material	Thickness, mm (in.)	Except No.	Except Description
1	T300/5208 (epoxy)	1.016 to 1.524 (0.040 to 0.060)	1a	One ply FRP (Style 120) each face-cocure
			1b	Aluminum flame spray coating
			1c	Stitching prior to cure
			1d	Glass frit filled paint
2	T300/5208 (epoxy)	6.35 (0.250)	2a	Replace all tape with graphite fabric
			2b	Boron filled resin in hybrid fabric as exterior ply
			2c	Phenolic exterior coating (co-cure)
			2d	Boron filler outer 4 plies
			2e	Intumescent coating
			2f	Graphite fabric with phenolic exterior plies
			2g	Graphite fabric interleaving (i.e., to replace 0,90 tape)
			2h	Hybrid fabric (90% graphite and 10% glass), 6 plies each face
3	T300 chopped fiber/EM 7125 (epoxy)	1.016 to 1.524 (0.040 to 0.060)	3a	Graphite mat facings cocured to EM 7125 chopped fiber "core"
			3b	Phenolic/chopped fiber facings co-cure
4	Celion 6000/ PMR-15 (polyimide)	1.016 to 1.524 (0.040 to 0.060)	4a	NR150 B-2 filled (20%) with glass microballons and coated onto 104 style glass fabric: 1 ply co-cured to each face
5	T300/HY-E-1008E (phenolic)	1.016 to 1.524 (0.040 to 0.060)	5a	Exterior coating: Talc filled phenolic (co-cured)

Table VIII - Task II Test Matrix

Condition	Tests 1/								
			Mechanicals						
			Flex & Mod		ILS		Flammability		
	Physical	NDI	RT	Elevated 4/	RT	Elevated 4/	FS	LOI	OSU
As fabricated	2/	3/	5	5	5	5	1	1	1
ITGA exposure 4/									
0F: 200 hrs			3		3				
500 hrs			3		3				
500 hrs + 24 hrs water boil			3	3	3	3	1	1	1
Humidity exposure									
0F: 200 hrs 5/			3		3				
500 hrs 5/			3		3				
500 hrs + 24 hrs water boil	6/		3	3	3	3	1	1	1

NOTES:

FS = Flame Spread
LOI = Limiting Oxygen Index
OSU = Ohio State University Release Rate Apparatus

1/ Applies to each baseline and hybrid configuration except FS and LOI performed on systems #1, 4, and 5 of Table VII only

2/ Specific gravity, fiber volume and void %

3/ Ultrasonic "C" scan

4/ 405.2K (270°F) for systems 1 thru 3b of Table VII
533K (500°F) for systems 4 and 4a of Table VII
477K (400°F) for systems 5 and 5a of Table VII

5/ 333K (140°F)/95% RH: all systems of Table VII

6/ Weight change only

Table IX

Limiting Oxygen Index (LOI) Test Results

Conditioning 1/	LOI of System # 2/ 3/ 1 (epoxy)	4 (polyimide)	5 (phenolic)
As fabricated	40.2	81.3	43.5
After isothermal aging	51.4	77.0	34.3
After humidity exposure	44.2	70.4	42.9

NOTES:

1/ See Table VIII.

2/ ASTM D-2863 Test Method

3/ See Table VII for materials.

Table X

Flame Spread (FS) Test Results

Conditioning 1/	Flame Spread Index 2/ 3/ of System #		
	1 (epoxy)	4 (polyimide)	5 (phenolic)
As fabricated	2.31	0.98	1.46
After isothermal aging	2.72	1.46	2.07
After humidity exposure	4.66	1.95	1.83

NOTES:

1/ See Table VIII.

2/ ASTM E-162 Test Method

3/ See Table VII for materials.

Table XI - OSU Flammability Test Results

System 1/ No.	Visual Observations Made During OSU Exposure
1	Fibers started to be released after 3 minutes and two plies had released after 7 minutes OSU exposure.
1a	FRP ply breaks and separates from panel and permits some fiber release but not as much as baseline system #1.
1b	No fiber release. Flames from holes in laminate only. Aluminum coating did separate from laminate but remained in place.
1c	Stitching started breaking loose after 3 minutes exposure and permitted some fiber release but not as much as baseline system #1.
1d	Coating cracked extensively and permitted some fiber release but not as much as the baseline laminate #1. Coating separated from laminate face in chunks and was completely destroyed by end of test.
2	Individual fibers started releasing near holes in the laminate after about 4 minutes OSU exposure. By 8 minutes, about 1/2 of the outer ply had released. By 10 minutes, graphite fibers were releasing from the second ply and continued until the end of exposure.
2a	After 8 minutes the first ply started to break off in small pieces and continued to do so for the remainder of test. Part of the first two plies released clumps which fell to bottom of burn chamber.
2b	No fiber release or breaks of any kind occurred in laminate face during exposure.
2c	Some fibers lifted from surface but did not float away apparently due to the holding power of the high-char forming phenolic coating. No fiber release.
2d	Same as 2b but some cracking of outer ply.
2e	Intumescent coating started swelling immediately after OSU exposure started. Coating prevented fiber release. Coating started to flake off after 11 minutes exposure. No fiber release.
2f	Same as 2b.
2g	Small chunks of outer ply started separating from the laminate about 11 minutes after OSU exposure started and fell to bottom of instrument. No fiber float.
2h	Same as 2b except some of the glass fibers in outer ply broke.

1/ See Table VII

Table XI (Continued)

System 1/ No.	Visual Observations Made During OSU Exposure
3	No apparent fiber release. The high degree of interlocking apparently prevents fiber release.
3a	Individual fibers started floating from surface after 3 minutes OSU exposure and continued throughout test. Not as bad as System 1, but worse than System 3.
3b	No fiber release.
4	Much less smoke release than epoxy systems. Some individual fibers lift from the face of the laminate, but do not float away nearly as badly as epoxy systems #1 and 2.
4a	No fiber release. Coating started to peel slightly after 10 minutes exposure.
5	No fiber release. Best looking primary system tested. Smoke release about same as for system #4 (polyimide) and much less than for epoxy systems.
5a	No fiber release. Good looking concept—should be tried on epoxy system.

Table XII

Physical Properties

System # 1/	SPG. 2/	Fiber Volume 3/ (%)	Voids 2/ (%)	Moisture Content 4/ (%)	5/ (%)	6/ (%)
1	1.557	58.4	0	0.93	1.67	2.08
1a	1.592	54.5	--	1.17	1.41	1.40
1b	1.626	61.0	0	1.1	1.95	1.86
1c	1.587	64.4	0.4	1.02	1.19	1.29
1d	1.608	50.4	0	1.53	2.37	2.81
2	1.545	57.4	0	0.67	0.95	0.54
2a	1.571	60.7	0	0.43	0.69	0.85
2b	1.565	58.3	0	0.57	0.90	0.72
2c	1.542	55.4	--	0.80	1.21	1.38
2d	1.545	54.4	0.3	0.60	1.21	1.21
2e	1.547	54.3	0	0.66	0.96	--
2f	1.512	53.0	0.7	0.70	0.96	--
2g	1.605	70.0	0	0.43	0.69	--
2h	1.542	56.0	0.8	1.86	2.31	2.19
3	1.516	51.1	0	0.83	1.49	1.86
3a	1.549	51.6	0.3	0.73	1.33	1.56
3b	1.555	52.9	--	1.39	1.45	1.57
4	1.589	66.3	1.2	0.70	1.17	1.47
4a	1.454	42.0	--	3.13	4.20	5.61
5	1.546	52.5	--	2.29	2.23	2.37
5a	1.483	43.4	--	2.93	2.56	--

1/ See Table VII.

2/ As-fabricated

3/ Not adjusted for hybridizing components.

4/ After 200 hours at 333k (140°F) and 100% RH

5/ After 500 hours at 333K (140°F) and 100% RH

6/ After 500 hours at 333K (140°F) and 100% RH plus 24 hours water boil

Table XIII - Mechanical Properties

Flexural Strength/Modulus, MPa (ksi/msi) 2/

System # 1/	As Fabricated		ITGA Exposure 4/ of:				Humidity Exposure 5/ of:			
			200 hrs.	500 hrs.	500 hrs + 24 hrs water boil		200 hrs.	500 hrs.	500 hrs + 24 hrs water boil	
	RT	3/ Elevated	RT	RT	RT	3/ Elevated	RT	RT	RT	3/ Elevated
1	751.7/ 51,724.1 (109/ 7.5)	820.7/- (119/-)	1117.2/ 86,896.6 (162/ 12.6)	1006.9/ 77,931.0 (146/ 11.3)	917.2/ 79,310.3 (133/ 11.5)	882.8/ 73,103.4 (128/ 10.6)	1089.7/ 83,449.4 (158/ 12.1)	1006.9/- (146/-)	1013.8/ 77,931.0 (147/ 11.3)	744.8/ 73,793.1 (108/ 10.7)
1a	813.8/ 46,206.9 (118/ 6.7)	642.8/ 45,517.2 (93.2/ 6.6)	793.1 46,206.9 (115/ 6.7)	912.4/ 46,206.9 (132.3/ 6.7)	896.6/ 50,344.5 (130/ 7.3)	737.9/ 40,689.7 (107/ 5.9)	858.6/ 50,344.8 (139/ 7.3)			
1b	451.7/ 18,620.7 (65.5/ 2.7)	380.7/ 17,931.0 (55.2/ 2.6)	848.3/ 50,344.8 (123/ 7.3)	979.3/ 57,241.4 (142/ 8.3)			1013.8/ 68,905.5 (147/ 10.0)	1069.0/ 67,586.2 (155/ 9.8)		
1c	993.1/ 79,310.3 (144/ 11.5)	869.0/ 75,172.4 (126/ 10.9)	1055.2/ 73,103.4 (153/ 10.6)	993.1/ 73,103.4 (144/ 10.6)	1027.6/ 74,482.8 (149/ 10.8)	875.9/ 78,620.7 (127/ 11.4)	1000.0/ 71,034.5 (145/ 10.3)			
1d	548.3/ 28,965.5 (79.5/ 4.2)	514.5/ 27,586.2 (74.6/ 4.0)	587.6/ 47,586.2 (85.2/ 6.9)	565.5/ 26,206.9 (82/ 3.8)			626.2/ 34,482.8 (90.8/ 5.0)			
2	591.0/ 50,344.8 (85.7/ 7.3)	475.9/ 44,827.6 (69.0/ 6.5)	502.8/ 40,000.0 (72.9/ 5.8)	440.7/ 37,241.4 (63.9/ 5.4)	469.0/ 37,931.0 (68.0/ 5.5)	482.8/ 41,379.3 (70.0/ 6.0)	460.0/ 40,689.7 (66.7/ 5.9)	500.0/ 39,310.3 (72.5/ 5.7)		
2a	509.7/ 51,724.1 (73.9/ 7.5)	407.6/ 44,137.9 (59.1/ 6.4)	513.6/ 46,206.9 (74.5/ 6.7)	523.4/ 46,206.9 (75.9/ 6.7)		502.4/ 48,965.5 (72.8/ 7.1)	544.1/ 48,276.0 (78.9/ 7.0)			
2b	497.2/ 35,712.4 (72.1/ 5.1)	480.7/ 40,690.0 (69.7/ 5.9)	520.0/ 37,931.0 (75.4/ 5.5)	494.5/ 37,931.0 (71.7/ 5.5)	493.8/ 39,310.3 (71.6/ 5.7)	468.3/ 41,879.3 (67.9/ 6.0)	510.8/ 25,517.2 (74.0/ 3.7)	518.6/ 38,620.7 (75.2/ 5.6)	521.4/ 37,931.0 (75.6/ 5.5)	415.9/ 34,482.8 (60.3/ 5.0)
2c	493.1/ 37,241.1 (71.5/ 5.4)	418.6/ 38,620.7 (60.7/ 5.6)	483.5/ 32,413.8 (70.1/ 4.7)	423.5/ 41,379.3 (61.4/ 6.0)	501.4/ 36,551.7 (72.7/ 5.3)	397.9/ 34,492.8 (57.7/ 5.0)	524.8/ 66,206.9 (76.1/ 9.6)	534.5/ 46,896.6 (77.5/ 6.8)	487.6/ 37,241.4 (70.7/ 5.4)	333.8/ 30,344.8 48.4/ 4.4)
2d	478.6/ 40,000.0 (69.4/ 5.8)	429.7/ 44,137.9 (62.3/ 6.4)	411.0/ 42,069.0 (59.6/ 6.1)	428.3/ 42,758.6 (62.1/ 6.2)	444.8/ 42,758.4 (64.5/ 6.2)	435.9/ 44,827.6 (63.2/ 6.5)	466.2/ 44,137.9 (67.6/ 6.4)	469.0/ 42,069.0 (68.0/ 6.1)	534.5/ 43,448.3 (77.5/ 6.3)	406.2/ 39,310.3 (58.9/ 5.7)
2e	524.1/ 45,517.2 (76.0/ 6.6)	435.2/ 37,241.4 (63.1/ 5.4)	522.1/ 36,551.7 (75.7/ 5.3)	548.7/ 35,862.1 (79.7/ 5.2)	541.4/ 37,241.4 (78.5/ 5.4)	453.8/ 38,620.7 (65.8/ 5.6)	569.7/ 38,620.7 (82.6/ 5.6)	534.5/ 35,172.4 (77.5/ 5.1)		
2f	455.9/ 40,689.7 (66.1/ 5.9)	423.4/ 46,896.6 (61.4/ 6.8)	473.1/ 39,310.3 (63.2/ 5.6)	473.1/ 39,310.3 (68.6/ 5.7)	447.6/ 36,551.7 (64.9/ 5.3)	426.9/ 44,137.9 (61.9/ 6.4)	498.6/ 40,000.0 (72.3/ 5.8)			

Table XIII (Continued)

Flexural Strength/Modulus, MPa (ksi/msi) 2/

System # 1/	As Fabricated		ITGA Exposure 4/ of:				Humidity Exposure 5/ of:			
			200 hrs.	500 hrs.	500 hrs + 24 hrs water boil		200 hrs.	500 hrs.	500 hrs + 24 hrs water boil	
	RT	3/ Elevated	RT	RT	RT	3/ Elevated	RT	RT	RT	3/ Elevated
2g	479.3/ 51,724.1 (69.5/ 7.5)	311.0/ 46,206.9 (45.1/ 6.7)	532.4/ 49,655.2 (83.3/ 7 3)	532.4/ 49,655.2 (77.2/ 7.2)	553.1/ 52,413.8 (80.2/ 7.6)	437.2/ 55,862.1 (63.4/ 8.1)	522.1/ 51,034.5 (75.7/ 7.4)	623.5/ 51,724.1 (90.4/ 7.5)		
2h	576.6/ 44,827.6 (83.6/ 6.5)	377.2/ 48,965.5 (54.7/ 7.1)	536.0/ 29,655.2 (82.4/ 6.5)	531.0/ 29,655.2 (77.0/ 4.3)	459.3/ 46,206.9 (66.6/ 6.7)	329.0/ 45,517.2 (47.7/ 6.6)	462.1/ 48,965.5 (67.0/ 7.1)	502.8/ 47,586.2 (72.9/ 6.9)	440.7/ 34,482.8 (63.9/ 5.0)	233.1/ 37,241.1 (33.8/ 5.4)
3	555.9/ 22,069.0 (80.6/ 3.2)	95.9/ 24,137.9 (13.9/ 3.5)	380.0/- (55.1/-)	262.8/ 31,034.5 (38.1/ 4.5)	202.8/ 55,862.1 (29.4/ 8.1)	103.5/ 11,724.1 (15.0/ 1.7)	270.3/ 31,724.1 (39.2/ 4.6)	240.0/ 37,931.0 (34.8/ 5.5)	189.0/ 20,689.7 (27.4/ 3.0)	97.2/ 10,344.8 (14.1/ 1.5)
3a	202.8/ 55,172.4 (29.4/ 8.0)	150.3/ 37,931.0 (21.8/ 5.5)	207.6/ 23,448.2 (30.1/ 3.4)	229.0/ 44,827.6 (33.2/ 6.5)	259.3/ 64,137.9 (37.6/ 9.3)	162.8/ 46,896.6 (23.6/ 6.8)	186.9/ 52,413.8 (27.1/ 7.6)	237.9/ 64,137.9 (34.5/ 9.3)		
3b	290.3/ 35,172.4 (42.1/ 5.1)	127.4/ 19,310.3 (18.5/ 2.8)	342.1/ 41,379.3 (49.6/ 6.0)	366.2/ 33,793.1 (53.1/ 4.9)	334.5/ 29,655.2 (48.5/ 4.3)	132.4/ 17,951.0 (19.2/ 2.6)	376.6/ 55,862.1 (54.6/ 5.2)			
4	1110.3/ 75,862.1 (161/ 11.0)	703.5/ 76,551.7 (102/ 11.1)	1165.5/ 82,758.6 (169/ 12.0)	1041.4/ 78,630.7 (151/ 11.4)	1131.0/ 78,630.7 (164/ 11.4)	834.5/ 70,344.8 (121/ 10.2)	1131.0/ 82,758.6 (164/ 12)	1048.2/ 84,137.9 (152/ 12.2)	1075.6/ 84,137.9 (156/ 12.2)	786.2/ 73,103.5 (114/ 10.6)
4a	450.3/ 21,379.3 (65.3/ 3.1)	196.6/ 13,103.5 (28.5/ 1.9)	453.1/ 28,965.5 (65.7/ 4.2)	465.5/ 26,896.6 (67.5/ 3.9)	405.5/ 22,069.0 (58.8/ 3.2)	278.6/ 20,689.7 (40.4/ 3.0)	447.6/ 28,275.9 (64.9/ 4.1)	444.1/ 27,586.2 (64.4/ 4.0)		
5	1131.0/ 77,951.0 (164/ 11.3)	411.0/ 55,862.1 (59.6/ 8.1)	951.7/ 68,965.5 (138/ 10)	744.8/ 59,310.3 (108/ 8.6)			1096.6/ 75,172.4 (159/ 10.9)	1027.6/ 75,862.1 (149/ 11.0)		
5a	862.1/ 48,965.5 (125/ 7.1)	512.4/ 32,413.8 (74.3/4.7 4.7)	951.7/ 42,758.6 (109/ 6.2)	509.0/ 38,551.7 (73.8/ 5.3)			765.5/ 48,965.6 (111/ 7.1)	696.6/ 35,172.4 (101/ 5.1)		

1/ See Table VII

2/ Average of 3 specimens except as-fabricated condition is an average of 5 specimens

3/ Elevated test temperature is 405.2K (270°F) for systems 1 through 2h, 573K (500°F) for systems 4 and 4a, and 477.4K (400° 5 and 5a.

4/ ITGA exposure temperatures are same as 3/.

5/ 333K (140°F)/95% RH; all systems

6/ All values based on actual panel thickness including hybridizing constituents.

7/ Specimen thickness of system 2 through 2h is nominal 6.35 mm (0.250 in.); all others are 1.016 to 1.524 mm (0.040 to 0.060 inch).