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PROTOTYPE RIGID POLYIMIDE COMPONENTS FINAL REPORT

MARCH 11, 1975

Prepared by

D.H. Wykes
Project Engineer
Advanced Manufacturing Technology

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FOREWORD

This final report summarizes the effort under NASA JSC Contract NAS9-12302, Prototype Rigid Polyimide Components. Mr. Dale Sauers of NASA's Materials Technology Branch, Non-Metallic Materials Development Section, Structures and Mechanics Division, Houston, Texas, acted as technical monitor for the 15 tasks performed over the three-year contract period. Polyimide component fabrication and program management at Rockwell International was conducted by the Advanced Manufacturing Technology Department of the Company's Space Division, Downey, California.

The program team included S.Y. Yoshino, Manager, Advanced Manufacturing Technology Department; D.H. Wykes, Project Engineer; and A.E. Koerner, Manufacturing Research Engineer.

ABSTRACT

This report summarizes the activity conducted by Rockwell International, under the direction of NASA JSC, in utilizing spin-off Apollo base technology to fabricate a variety of commercial and aerospace related parts that are nonflammable and resistant to high-temperature degradation. Manufacturing techniques and the tooling used to fabricate each of the polyimide/glass structures is discussed under each task heading.

A brief history, tracing the development of high-temperature polyimide resins, is presented along with a discussion of the properties of DuPont's PI 2501/glass material (later redesignated PI 4701/glass). Mechanical and flammability properties of DuPont's PI 2501/glass laminates are compared with epoxy, phenolic, and silicone high-temperature resin/glass material systems. Offgassing characteristics are also presented.

This report is concluded with a discussion of the current developments in polyimide materials technology and the potential civilian and government applications of polyimide materials to reduce fire hazards and increase the survivability of men and equipment.

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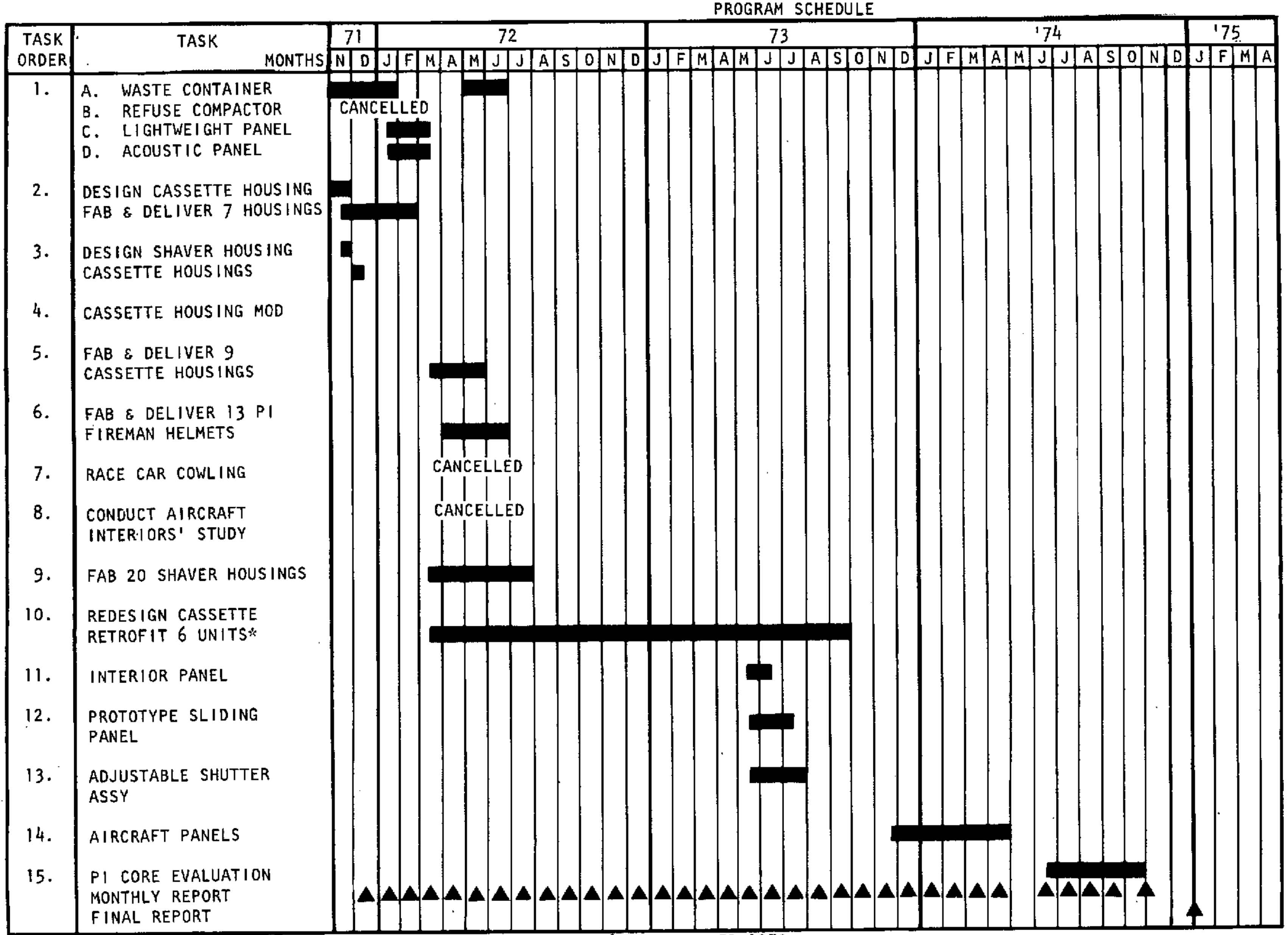


*RECORDERS REQUIRING MODIFICATION REDUCED FROM 7 TO 6 - SEPTEMBER 1973

PROGRAM SCHEDULE

Space Division
Rockwell International

INTRODUCTION

As part of its effort toward making spacecraft interiors flame resistant, NASA funded the development and/or evaluation of several families of flame resistant, nonmetallic materials. One such class of aerospace material was the rigid structural laminate fabricated from fiberlgass and a variety of high-temperature resins: polyimide, polyquinoxaline, polybenzimidazole, chem ceram, epoxies, etc. As these materials were being evaluated, parameters such as cost, flame resistance, high-temperature resistance, impact strength, tensile and flexural modulus, toxicity, durability, and availability were investigated. While the accumulated data were being reviewed, it became evident that the polyimide resin/glass fabric system had the best combination of mechanical and physical properties to meet stringent spacecraft requirements. Polyimide/glass (PI/GL) materials were subsequently qualified by NASA for unlimited use in the Apollo Spacecraft Program.

The unique properties of PI/GL were recognized by NASA as having considerable spin-off capability for commercial applications. Toward this end, NASA cooperated with several industries, among them the aircraft, fire fighting, and construction industries, to determine the potential usefulness of the aerospace materials for nonaerospace applications. The kinds of commercial items NASA was asked to supply for evaluation were diverse. A small group of these items was selected for development in this demonstration program.

Rockwell was selected to fabricate these demonstration items because of its experience in successfully working with polyimide/glass on the Apollo contract ,and the availability of high-temperature autoclaves and other equipment not normally available at companies that work only with low-temperature epoxy and polyester resin systems.

Specifically, Rockwell performed work in the following activities:

1. Recommended and provided specific resin/fabric systems which constituted a satisfactory substitute for materials presently in use.

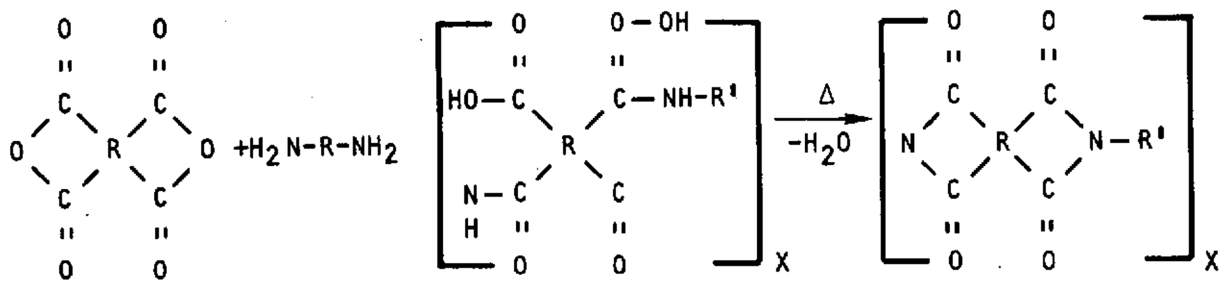
2. Developed and applied the special fabrication techniques required to incorporate the resin/fabric systems into the specific applications described in the applicable task orders (1 through 15).

3. Performed development and laboratory tests as necessary in support of the tasks of the program.

TECHNICAL DISCUSSION

BACKGROUND AND DEVELOPMENT HISTORY OF POLYIMIDES

Polyimide development and chemistry was pioneered by the E.I. DuPont Company in the early 1950's to meet the demands for more thermally stable polymers. The basic polyimide chemistry centers on the reaction between aromatic dianhydrides and dianines. The intermediate product, a polyanic-acid, is readily converted by heat or chemical methods to the final polyimide structure:



From this work evolved a series of products now commercially available. These include Kapton polyimide films, Vespel molded polyimide plastics, Pyre-ML polyimide wire enamels and varnishes, and Pyralin polyimide prepregs and precursor solutions. Most of these products found application in the aerospace industry as part of electrical or thermal insulation systems. The polyimides in the form of prepregs for the laminating industry, however, found only limited use because of the high-processing temperatures (450-650°F) and pressures 70-500 lb/in.^{2} required to produce structural laminates. These process parameters required presses and autoclaves not normally available at companies that worked only with polyesters, epoxies, or similar low-temperature resins. Additionally the polymide prepregs lack good tack, were hydroscopic, and required a long learning cycle before low-void, precipitation-free laminates could be consistently produced.

In 1967, the Space Division of Rockwell, in association with NASA, conducted an extensive program for the development of a polyimide (PI)/glass fabric laminate capable of meeting the nonflammability requirements of the Apollo spacecraft when tested in a 100-percent oxygen atmosphere at 16.5 lb/in.^{2} pressures as specified in MSC-PA-D-67-13. After numerous flammability tests, it was concluded that PI 2501/glass prepreg produced by DuPont could be made nonflammable by controlling laminate resin content and using special processing techniques. Figure 1 dramatically shows the performance of specially processed PI/glass and a silicone material it replaced.

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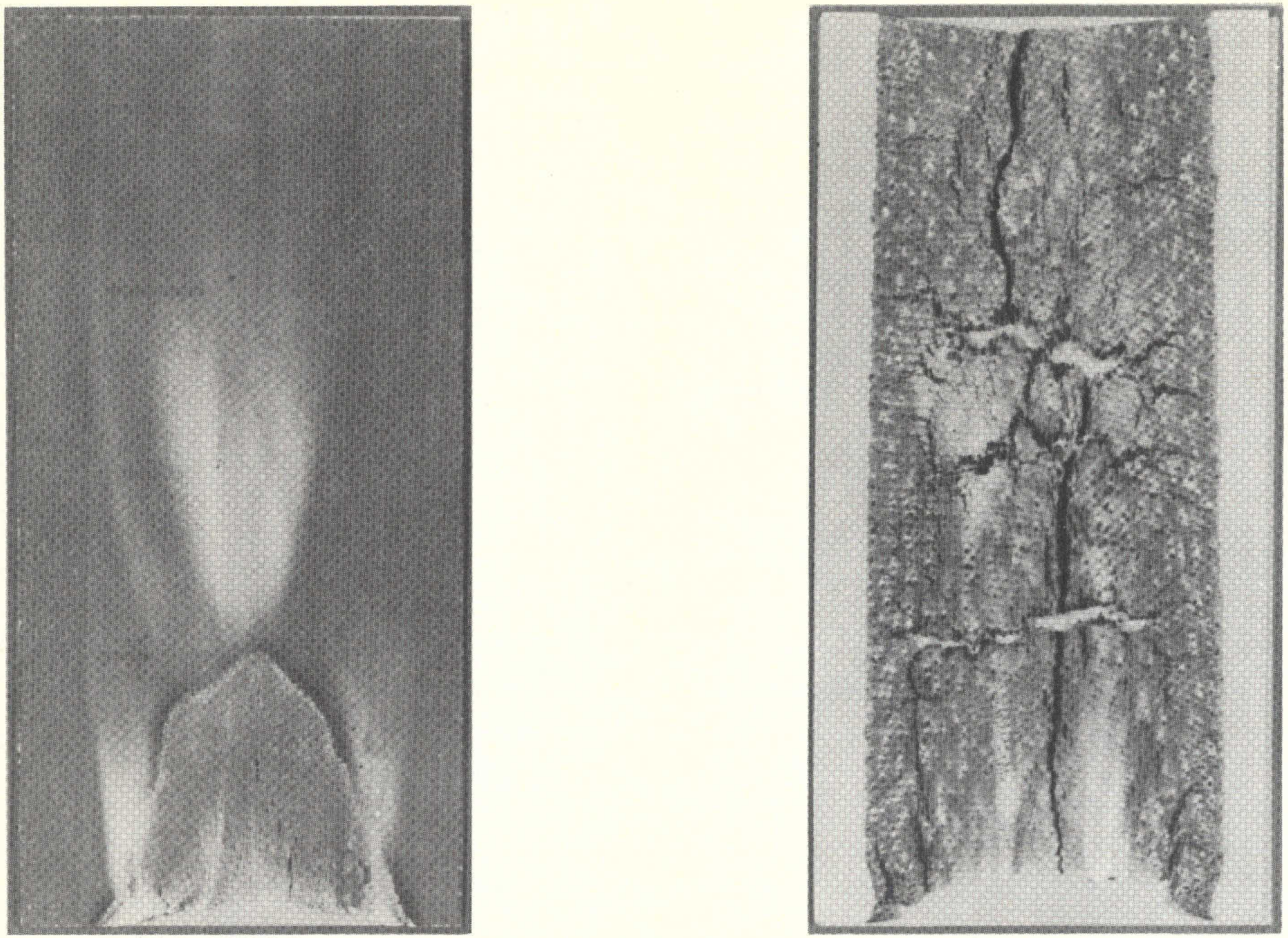
(NICHROME/RTV SILICONE IGNITOR-BOTTOM IGNITION)



P.I. 2501
POLYIMIDE LAMINATE
(2½"X 5"X0.034"/0.036")

DC 7141
SILICONE LAMINATE
(2½"X5"X0.039"/0.041")

Figure 1. Comparison of Flammability Characteristics of Four-Ply Polyimide and Silicone Glass Laminate in 100-Percent Oxygen Atmosphere at 16.5 lb/in.^{2}

PROPERTIES OF POLYIMIDE/GLASS LAMINATE

One of the most notable properties of fully cured polyimides is their resistance to burning and their low smoke generation when subjected to burning conditions. It is the only commercially available organic polymer that is self-extinguishing and will not smoke in 100-percent-oxygen atmospheres.

Table 1 summarizes the flammability properties of the polyimide laminate compared with the epoxy, phenolic, and silicone laminates. The data presented in this table show that the epoxy, phenolic, and silicone laminates will burn in 100-percent-oxygen atmospheres when subjected to the nichrome ignitor, whereas the polyimide laminate is self-extinguishing.

Numerous additional flammability tests were conducted on polyimide laminates to study the effect of different cured resin contents and ply construction (laminate thickness). From the data presented in Table 1, it can be seen that control of cured resin content and laminate thickness is critical. In Figure 2, the average combustion rate is plotted against the reciprocal of laminate thickness and number of glass fabric plies. The curve shows that laminates of two to eight plies with a cured resin content of less than 20 percent are self-extinguishing. Similarly, other curves, shown as broken lines, are drawn showing the effects of thickness and resin content on flammability. The generation of such curves permitted a more analytical approach to the design of nonflammable structural laminates. The procedure would consist of designing the component based on structural considerations, thereby establishing the necessary laminate thickness.

Offgassing characteristics of glass-reinforced plastic laminates relative to odor, carbon monoxide, and total organics are determined in accordance with NASA document MSC-PA-D-67-13 (Test No.'s 6 and 7), except for exposure duration. The Rockwell test time was 336 hours, whereas the NASA document calls for a period of 72 hours at a temperature of 155 ± 5°F. No problem was encountered, as Table 2 test data show, regardless of resin content, in meeting the toxicity requirements relative to odor, carbon monoxide, and total organics.

All work on the development of nonflammable structural glass laminates has been accomplished with DuPont 2501 polyimide resin and 181 style E-glass fabric reinforcement with A1100 finish. These applications include food containers, protective back covers for main display and control panels, many other brackets, small covers for electrical switches, etc. Table 3 summarizes various sources on material properties for polyimide laminates having a resin content approximately that desired for nonflammable characteristics. The data presented in Column 4 of this table show that the flexural properties of low-resin-content (less than 20 percent by weight), nonflammable polyimide laminates fabricated by Rockwell have strengths within the range of that reported for higher resin content laminates. Table 3 also indicates that low resin content in thin laminates is acceptable structurally.

MATERIAL: MB0130-081 - (DUPONT PI2501) FABRICATED PER MA0308-0150 BY D/071

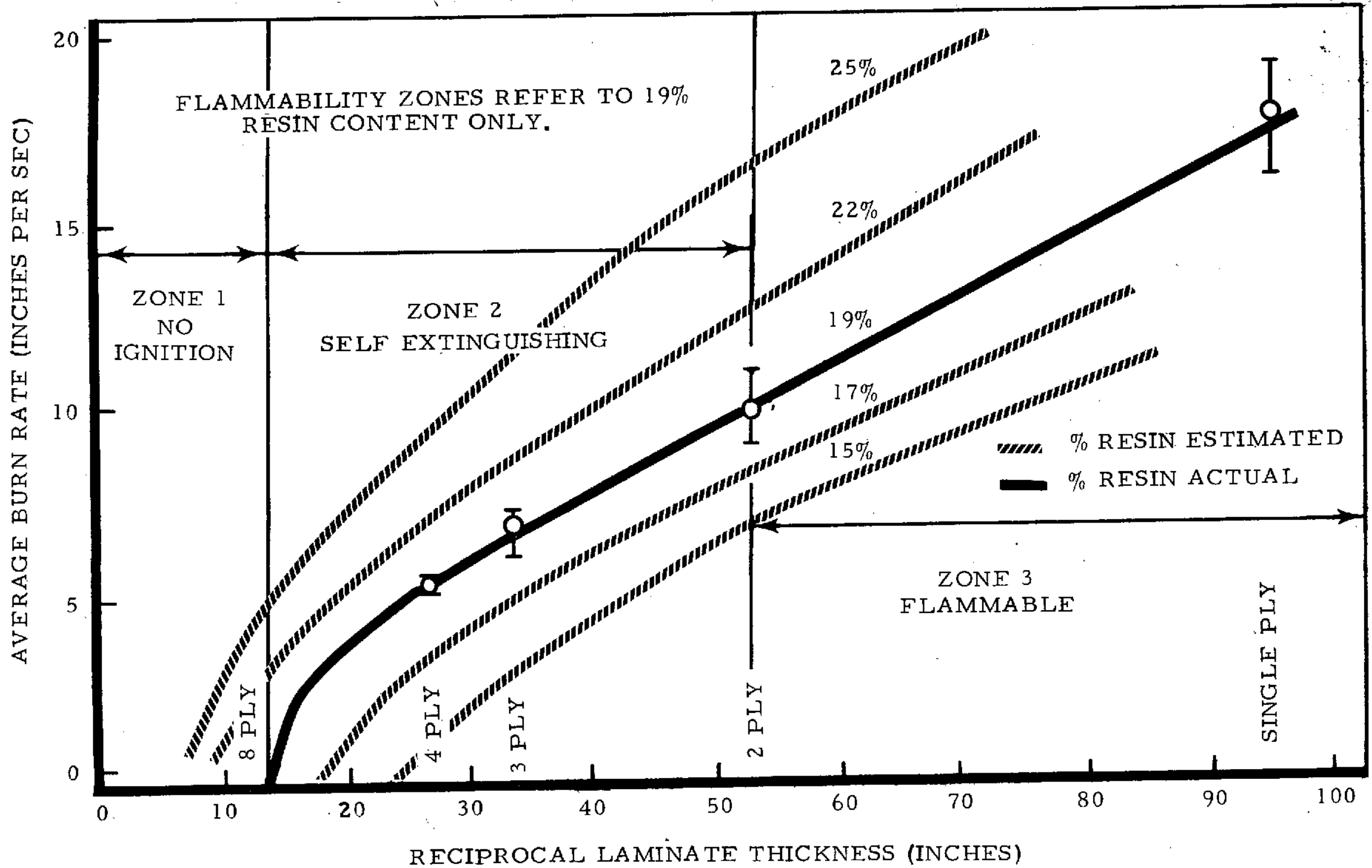


Figure 2. Flammability Versus Ply Construction of Polyimide/Glass Fabric Laminates

Table 1. Flammability Test Results - Comparison of Polyimide Versus Epoxy, Phenolic Silicone Glass Fabric Laminates

Laminate Description			Flammability Test Results (Combustion Rate - in./sec)		
Material	Thick (in.)	% Cured Resin	100% O_2 (16.5 $lb/in.^2$)	60% O_2/40% N_2 (16.5 $lb/in.^2$)	Air (14.7 $lb/in.^2$)
Polyimide (Pyralin PI 2501 prepreg)	0.045 to 0.065	15.0 to 20.0	Self-extinguishing from bottom (RTV ignitor)	No ignition from bottom (RTV ignitor)	No ignition from bottom (RTV ignitor)
Epoxy (Epoxy No. 7022 prepreg	0.070	23.0 to 28.0	0.039 - Top ignition (Nichrome/paper ignitor)	0.025 - top ignition (Nichrome/paper ignitor)	No ignition from top 0.067 - bottom (Nichrome/paper ignitor)
Phenolic (CTL 91-LD prepreg)	0.040 to 0.075	18.0 to 28.0	No ignition from top 0.085/0.120 from bottom (Nichrome/paper ignitor)	No test	No ignition from top or bottom (Nichrome/paper ignitor)
Silicone (DC-7141 prepreg)	0.040 to 0.070	26.0 to 28.0	No ignition from top 0.074/0.128 from bottom (Nichrome/paper ignitor)	No ignition from top 0.091 from bottom (Nichrome/paper ignitor)	No ignition from top or bottom (Nichrome/paper ignitor)

Table 2. Offgassing Characteristics of Polyimide Glass Fabric Laminate

	Odor	CO (ppm)	CO_2 (ppm)	H_2O (ppm)	Total Organics (ppm)
Acceptance Criteria	< 1.5	20	*	*	100
Laminate, Pyralin P.I. 2501 (8-ply construction)	1.0	1	12	700	1

*No requirements

Table 3. Typical Properties of Polyimide Glass Fabric Laminates (Suppliers and Rockwell International)

Property \ Material	Col 1 U.S. Polymeric (PI V-303/181 E Glass)	Col 2 Narmco, Inc. (PI 1830/181 E Glass)	Col 3 DuPont (PI 2501/181 E Glass)	Col 4 Rockwell International Space Division (DuPont PI 2501/181 E Glass)	Col 5 Average of Columns 1 Through 4
Tensile					
Strength (psi)	58,700	55,000	42,000	57,400	53,275
Modulus (psi x 10^6)	4.1	3.9	2.7	3.6	3.5
Compression					
Strength (psi)	64,200	49,000	52,000	43,200	52,100
Modulus (psi x 10^6)	4.0		2.7		3.3
Flexure					
Strength (psi)	84,000	76,000	57,000/70,000	77,600	74,400
Modulus (psi x 10^6)	4.0	3.7	3.1	3.8	3.65
Interlaminar shear (psi)	2,100	2,360			2,230
Specific gravity	1.77	1.65	1.64	1.77	1.70
Barcol hardness				45/55	45/55
Cured resin content (Percent)	20	22.5	23.0	17.0/18.0	Not applicable

FABRICATION TECHNIQUES FOR POLYIMIDE/GLASS PARTS

DuPont's PI 2501 polyimide resin is a condensation polymer that is dissolved in 12-15 percent n-methyl pyrrolidone (NMP) solvent. The high boiling point of NMP (395°F), combined with the condensation (H_2O) byproduct of the polyimide resin during polymerization, characterizes this system as one requiring close process control. In addition, the polyimide resin, in the B-stage condition, is hygroscopic and requires stringent control during storage and processing. Therefore, it is apparent that extreme care and accurate fabrication techniques must be exercised. For this reason, Rockwell developed a specific area for polyimide layup, which is maintained under clean room conditions with temperature/humidity controls.

The need of resin content control to achieve the nonflammability of PI glass laminates requires a unique approach to the fabrication of laminated components. Rockwell has developed a technique that assures minimum resin content variability. The process steps are not difficult, but must be strictly adhered to for production of high-quality parts under production conditions. The initial test specimen fabrication phase of this program included the use of (1) standard vacuum-bag techniques, (2) vacuum bag plus augmented autoclave pressure, and (3) platen press laminating. All were successfully used to produce PI/glass parts for Apollo. A typical layup and curing procedure followed in fabricating both parts for the Apollo vehicle and the various items fabricated in this program follows.

LAYUP

One ply of fluorocarbon parting fabric, Stern & Stern T-83-42 or equivalent, shall be placed on the surface and allowed to overhang the layup by 1/2 inch, minimum. Eight plies of bleeder fabric, Thalco EM 10A-1100 or equivalent, shall be applied on the parting fabric. All edges shall be sealed with a fluorocarbon tape and pigtailed to the layup to provide uniform vacuum distribution. A heat-resistant vacuum bag and bag sealant shall be used.

CURE CYCLE

Apply an initial vacuum of 26 inches of mercury, minimum, as measured from the vacuum static line and insert assembly in autoclave. Pressurize autoclave to obtain a pressure differential of 45 $\pm$ 10 lb/in.^{2} Pressure differential is defined as the difference between the vacuum static line gauge pressure and the autoclave gauge pressure. Heat the laminate to 295°F in 90-240 minutes while maintaining a pressure differential of 45 $\pm$ 10 lb/in.^{2} When part temperature (as measured by the hottest thermocouple) reaches 295°F, increase temperature and pressure differential at a gradual rate to 330°F (as measured by the coldest thermocouple) and 85-100 lb/in.^{2} in a period of 55 $\pm$ 10 minutes. Hold at 340 $\pm$ 10°F and 85-100 lb/in.^{2} pressure differential for 180 minutes, minimum. Cool to 200°F under a pressure differential of 85–100 lb/in.^{2} Cool to 150°F or less under a pressure differential of at least 10 lb/in.^{2}

POST-CURE

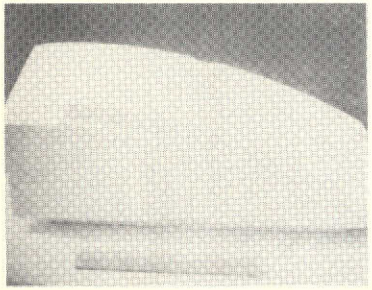
Test laminates shall be post-cured per the following cycle:

2 hours at 250 ± 10°F	2 hours at 500 ± 10°F
2 hours at 300 ± 10°F	8 hours at 600 ± 10°F
2 hours at 400 ± 10°F	Cool to 150°F or less in oven

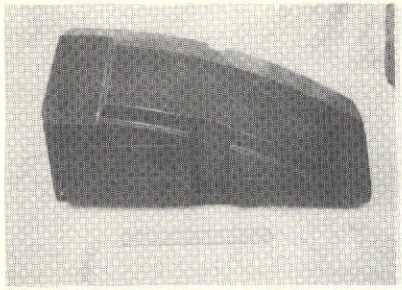
TOOLING

An interesting tooling concept used on the Apollo PI/GL program involved fabrication of the food containers. Due to undercuts, dimensional changes and schedule requirements from spacecraft to spacecraft, it was decided that the most economical tooling material was a breakaway plaster. However, the use of plaster at first was discarded because of the constant presence of moisture within the plaster. This was solved by the use of a thin spray of RTV silicone rubber over the plaster. In addition to serving as a seal, the RTV is an excellent mold release. This innovation eventually led to the first PI-laminated food container to be tested. Two weeks after receipt of an aluminum food container from Houston, Rockwell delivered an exact duplicate PI food container. A photographic sequence of the first food container production, along with other typical PI production assemblies, is shown in Figure 3. Flammability test of the first PI food container is compared against an earlier epoxy/glass test article, as shown in Figure 4. The GL/RD box was entirely burned, while the PI box shows only a slight discoloration at the ignition point.

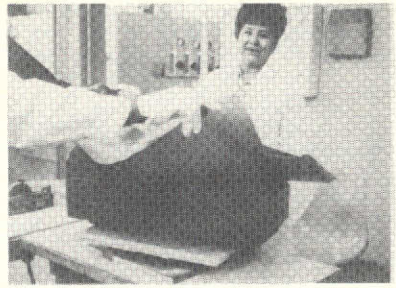
Tooling, outside of shop aids, for each of the articles fabricated is discussed under each task in subsequent section of this report.



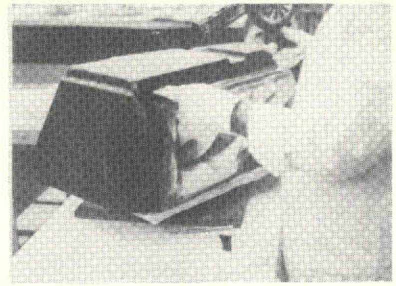
1. PLASTER MALE TOOL



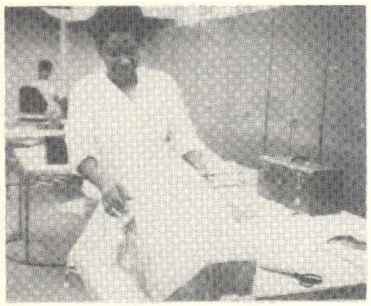
2. R.T.V. SEALANT/RELEASE



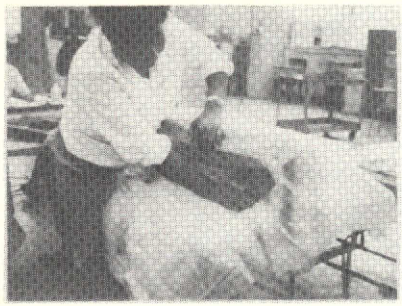
3. 1ST PLY PI LAY-UP



4. NESTLING OF OVER PLIES

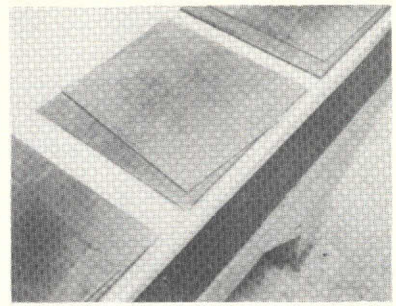


5. APPLICATION OF BLEEDER FABRIC

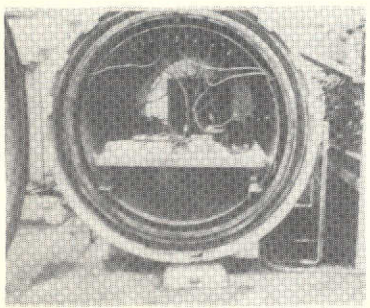


6. VACUUM BAGGING

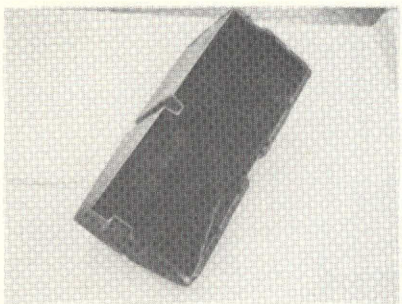
7. CHECKING FOR LEAKS



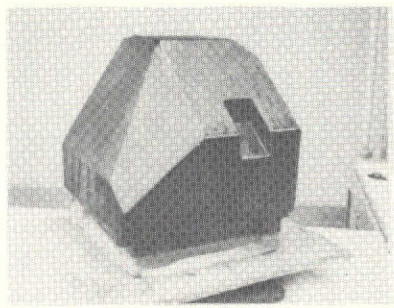
8. TAG END SPECIMEN



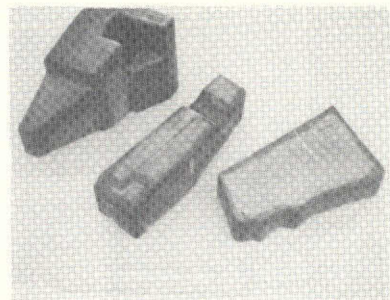
9. AUTOCLAVE CURING



10. PART FINISHED AND TRIMMED



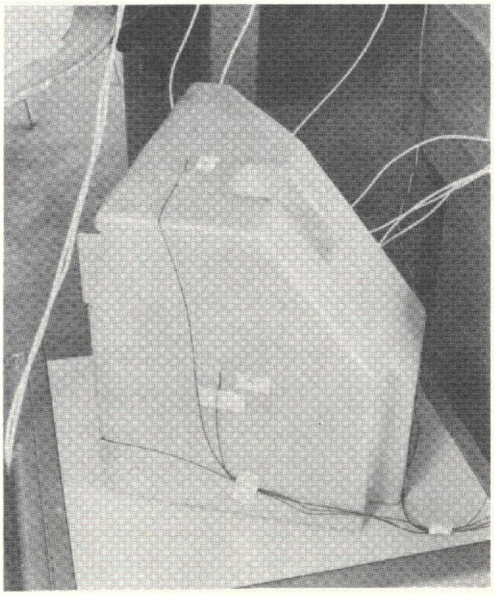
OTHER APOLLO FOOD CONTAINER



TYPICAL PI PRODUCTION ASSEMBLIES

Figure 3. Polyimide Production—Apollo Food Container

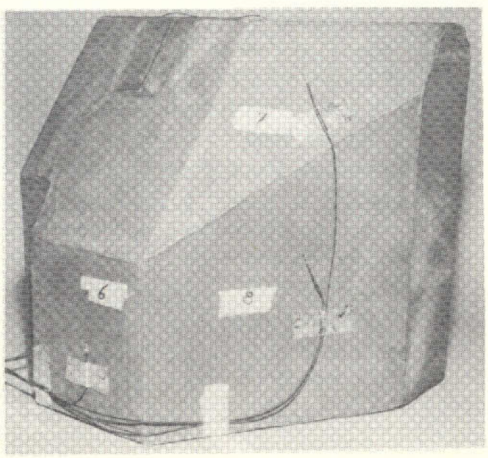
EPOXY FOOD BOX FLAMMABILITY TEST



BEFORE

AFTER

POLYIMIDE FOOD BOX FLAMMABILITY TEST



BEFORE

AFTER

Figure 4. Flammability Comparison of Epoxy and Polyimide Food Boxes

PROTOTYPE RIGID POLYIMIDE COMPONENTS

During this program, 15 tasks were worked on, at NASA JSC's direction, to demonstrate the versatility of polyimide resin materials for application in civilian and aerospace areas. Each product was fabricated from DuPont's 35-503 prepreg, which is a combination of DuPont's 4701 (commercial designation of PI 2501) polyimide resin and standard 181 weave E glass fabric with 1100 finish. Cutting and trimming of the prepreg followed standard aerospace practice. Layup, bagging, and curing procedures followed those discussed elsewhere in this report. The tooling used for each component fabricated is briefly discussed under each task heading.

TASK 1

ITEM A - WASTE CONTAINER (Figure 1-1)

Task Description

This assembly shall be 12 x 21 x 6 inches (outside dimensions) and shall have a wall thickness of approximately 0.03 inches. The exterior surface shall be smooth. The assembly shall have a removable top held in place by friction (detents are permissible). A door 5 x 5 inches shall be built into one end of the contained top and shall be supplied with a handle and some mechanism (detent, latch, or other) to hold it closed.

Discussion

This two-part waste container was fabricated from four plies of DuPont 35-503 prepreg on an aluminum tool welded up from aluminum plate. All surfaces were machined to obtain the necessary dimensions and surface smoothness. Minor oil canning of the upper and lower sections of the waste container occurred after the original post-cure. This problem was solved by using an expansion fixture and subjecting the part to a second post-cure cycle.

ITEM B - REFUSE COMPACTOR CANISTER

Work on this item was canceled at the request of NASA.

ITEM C - LIGHTWEIGHT PANEL (Figure 1-2)

Task Description

Fabricate a lightweight structural panel 12 x 12 x 1/2 inch by sandwiching rigid polyimide foam (20 pcf maximum) between two hard-surface laminates of polyimide resin and glass. This panel will be a replacement for the

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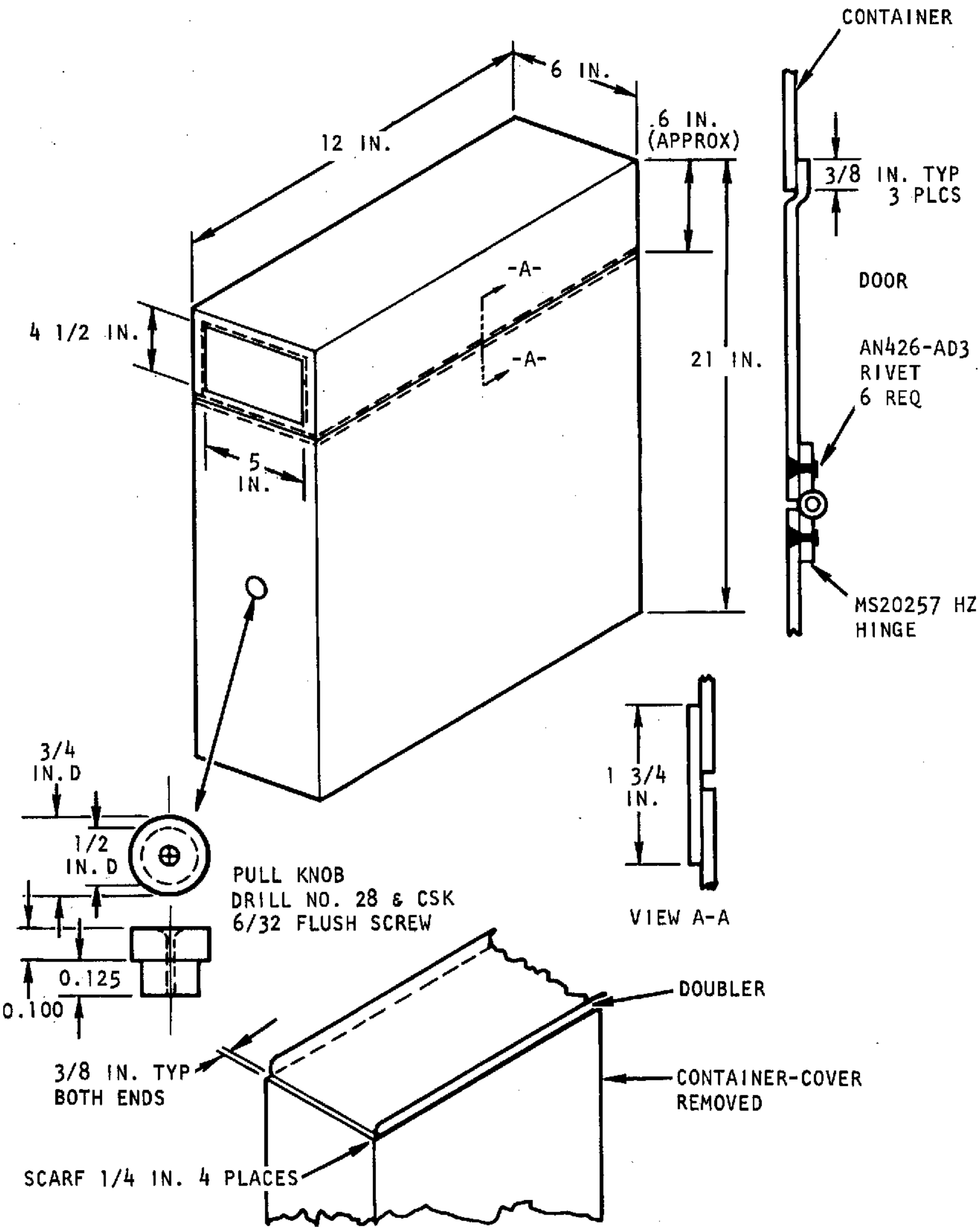

Figure 1-1. Waste Container

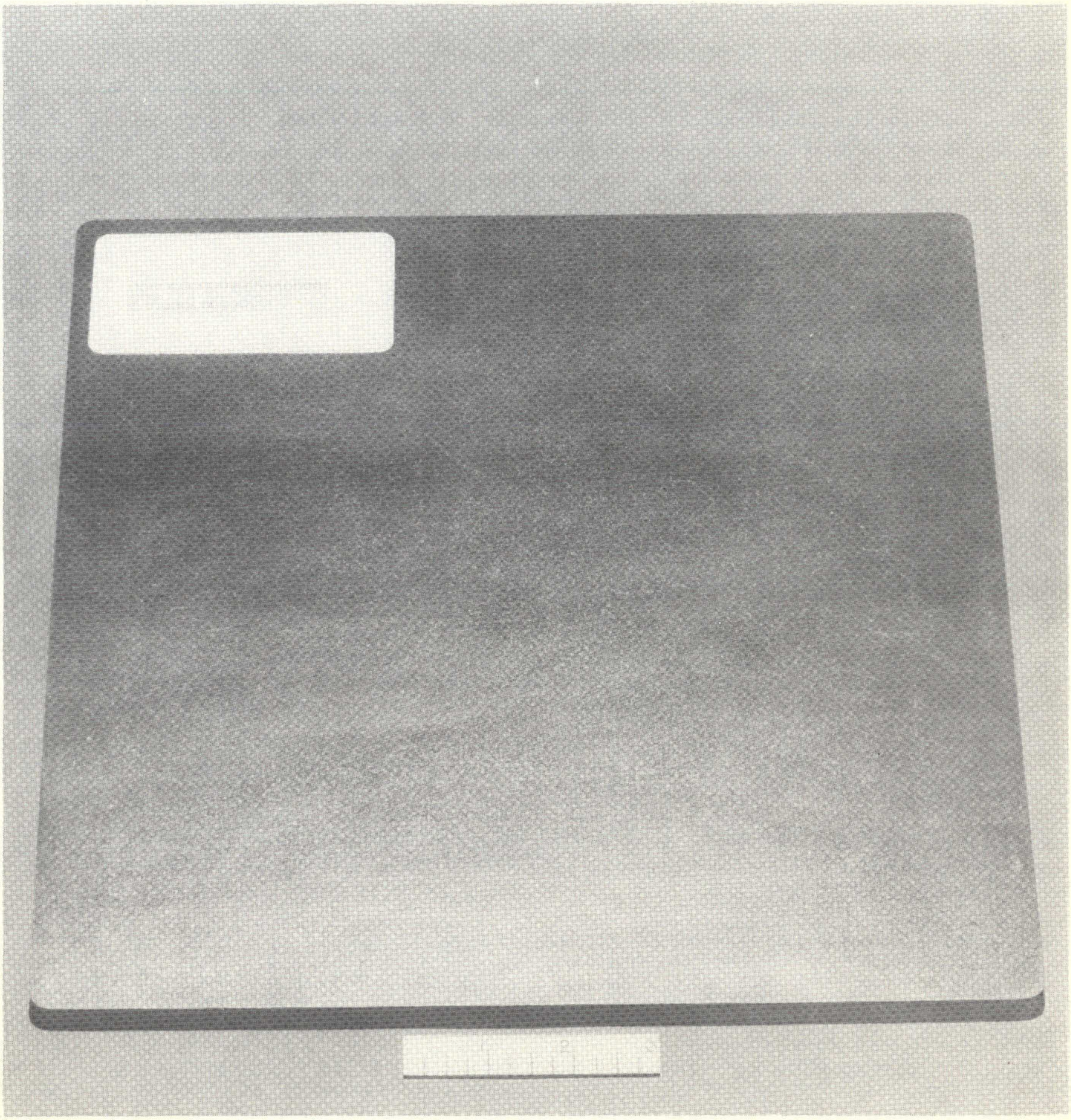

Figure 1-2. Lightweight Structural Panel

phenolic-impregnated kraft paper panel available commercially. The adhesive used must be flame-resistant as applied, nontoxic (per MSC-PAD-67-13), and humidity insensitive. The panel shall have a smooth exterior surface.

Discussion

To obtain the 20-pcf maximum foam core for this panel, a special PI syntactic foam was formulated as follows:

PI resin DuPont 4701 - 8 parts
3M glass microballoons - 8 parts
1/4-inch chopped glass roving - 1 part

Mixing, processing and manufacturing procedures for this foam are proprietary information.

The three-ply 35-503 skins for this panel were laid up and cocured to the foam under cure procedures established for the skins. No special tools, outside of normal manufacturing shop aids, were required to fabricate this sandwich panel.

ITEM D - ACOUSTIC PANEL (Figure 1-3)

Task Description

Fabricate a rigid panel 12 x 12 x 1/2 inch from polyimide and chopped glass with acoustic absorption cavities incorporated into its surface. The panel shall be as light in weight as possible concommittant with providing good structural properties. Absorption coefficient will be tested by NASA at the following vibration frequencies: 128, 256, 512, 1024, 2048, and 4096 Hertz.

Discussion

An oversize syntactic foam panel was fabricated from the formulation listed below and then machined on one surface to create the acoustical surface, shown in Figure 1-4.

PI resin DuPont 4701 - 3 parts
1/4-inch chopped glass roving - 1 part
Eccosphere EP 300 - 1 part
Cab-O-Sil - 0.1 part

Only shop aids were required for fabrication of this acoustical panel.

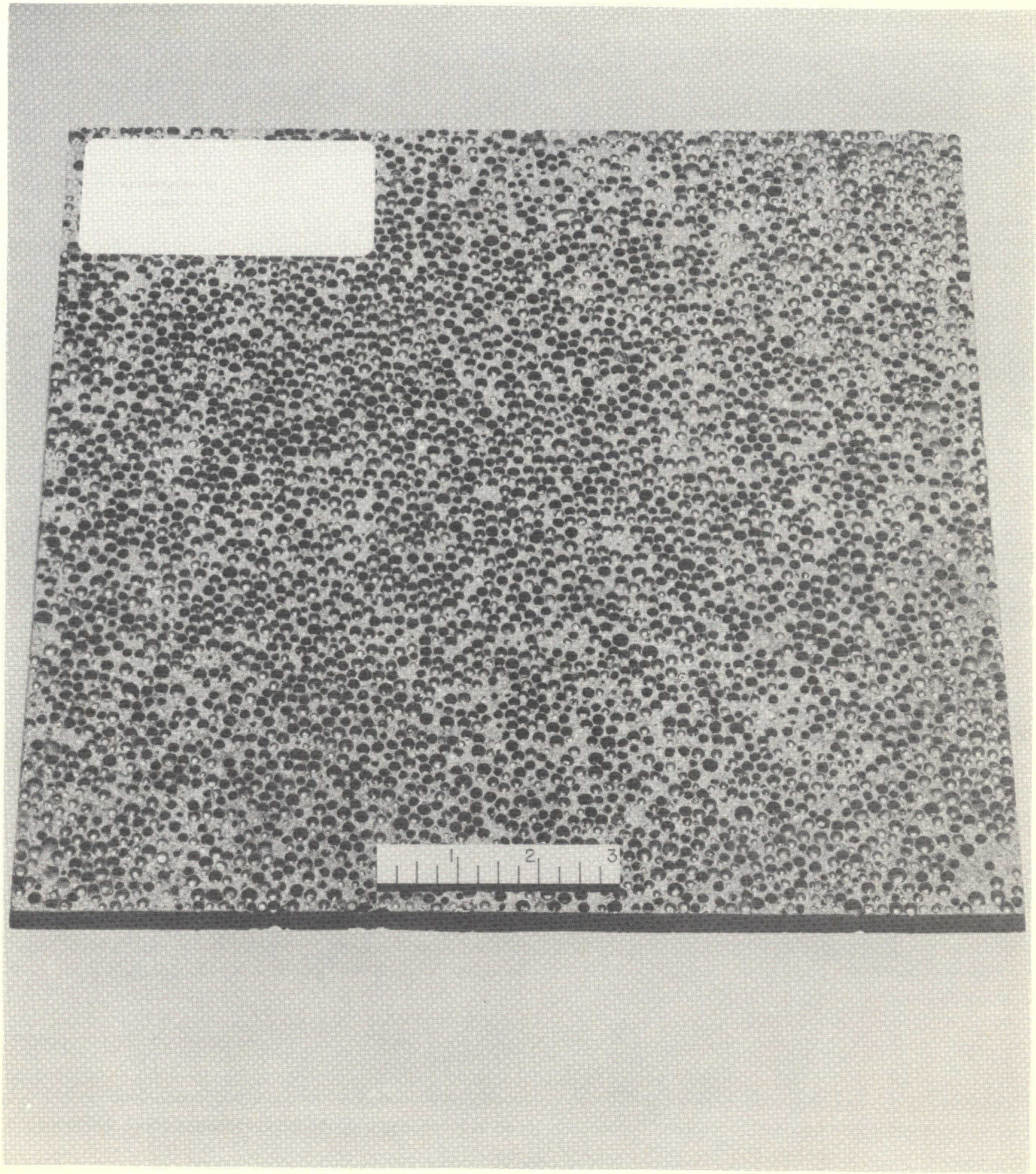

Figure 1-3. Acoustic Panel

7003-86-123A

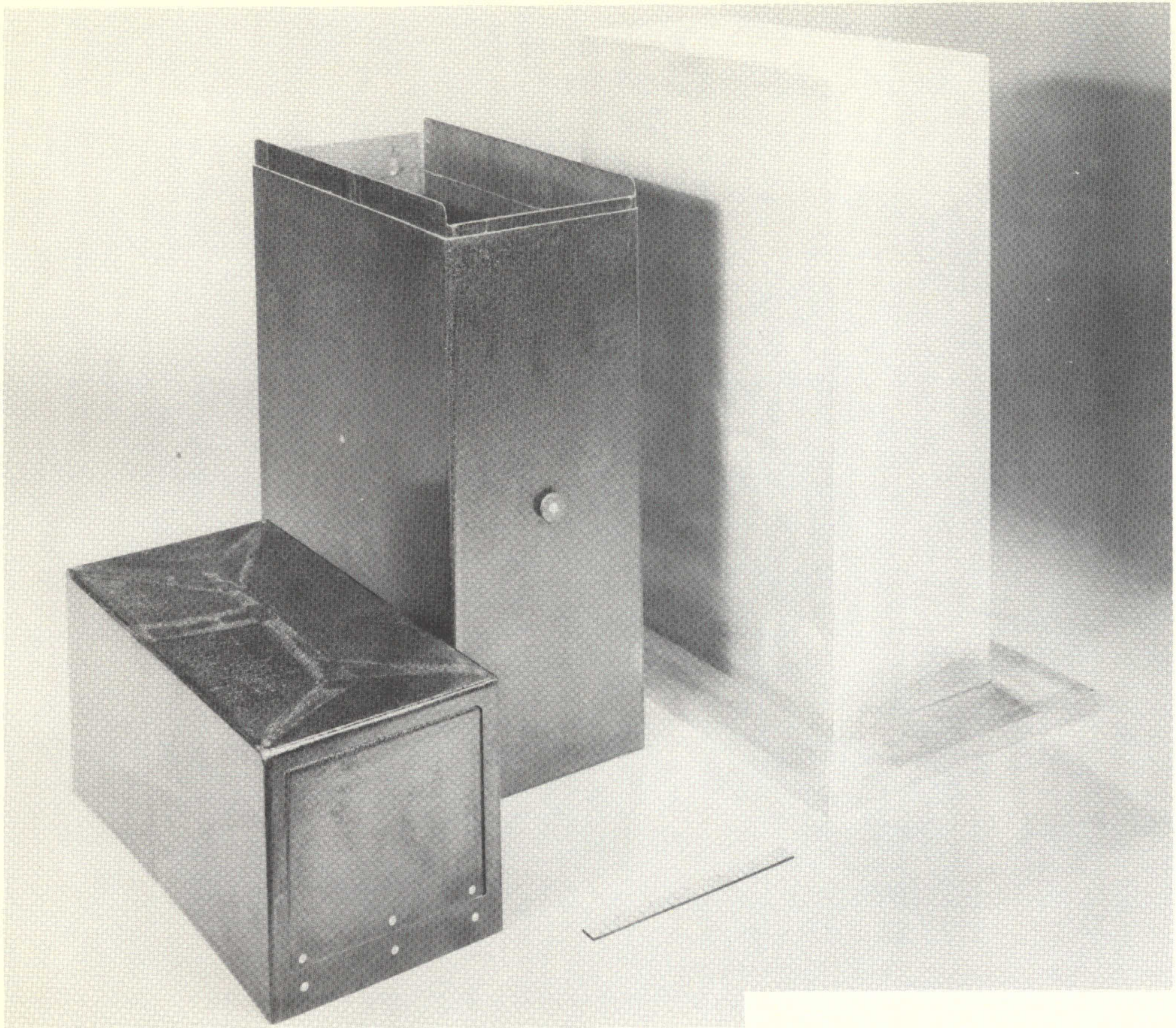

Figure 1-4. Acoustical Surface

TASK 2

TASK DESCRIPTION (Figure 2-1)

1. Design and prepare design drawings for a glass fabric/DuPont P4701 resin laminate covering to be bonded to a government-furnished tape player and microphone.

2. Fabricate laminate covers and assembly covers on seven government-furnished Sony tape players and microphones in accordance with Rockwell drawing MD 015679 (Figure 2-1).

3. Perform functional checks on each modified tape player assembly in accordance with Predelivery Acceptance Test CSD-S-048, prior to shipment to MSC, in accordance with the terms of contract NAS9-12302.

DISCUSSION

Rockwell drawing MD 015679 (Figure 2-1) was prepared showing the basic approach taken to make the Sony tape recorder and microphone nonflammable. The recorder exterior was covered with sections of four-ply 35-503 laminate secondarily bonded in place with Epon 954 room-temperature curing adhesive. Speaker opening and tape deck view window areas were covered with a stainless steel wire cloth.

Functional tests were performed to assure proper operation of all units after all modifications had been completed. Serial and part identification numbers were applied to the initial set or recorders with Wono-ink. Later units were identified with Scotch-Cal labels, solvent-stripped of contact adhesive, and then bonded to parts with Epon 954 room-temperature curing adhesive.

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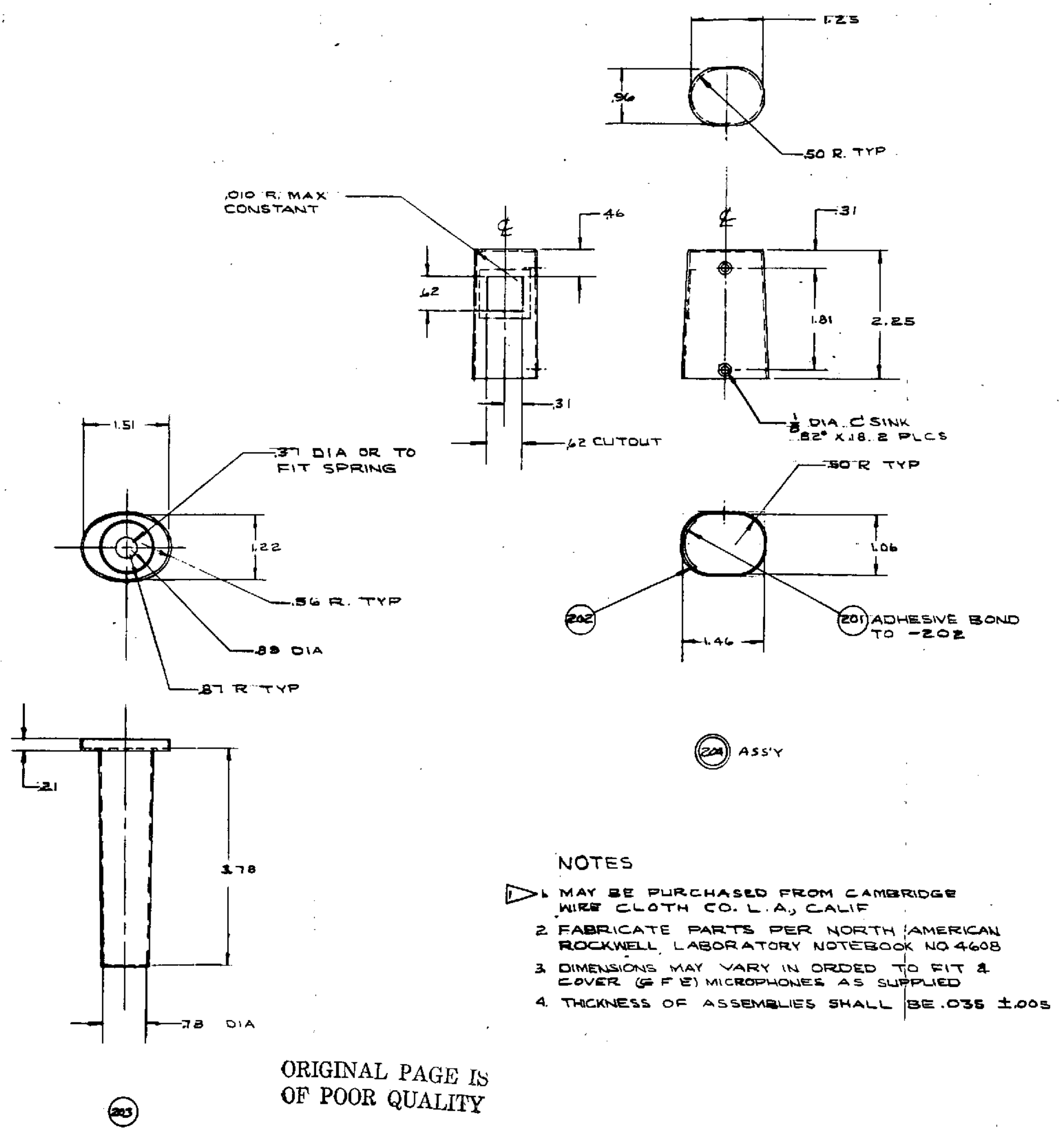

NOTES

1. MAY BE PURCHASED FROM CAMBRIDGE WIRE CLOTH CO. L.A., CALIF
2. FABRICATE PARTS PER NORTH AMERICAN ROCKWELL LABORATORY NOTEBOOK NO 4608
3. DIMENSIONS MAY VARY IN ORDER TO FIT & COVER (G F E) MICROPHONES AS SUPPLIED
4. THICKNESS OF ASSEMBLIES SHALL BE .035 ±.005

DET. NO.	NO REQD	DESCRIPTION	SIZE
204	1	ASSY CONSIST OF -201,-202 & -203	
203	1	PYRALIN 35-503 4 PLYS	.032/.036 X REQD
202	1	PYRALIN 35-503 4 PLYS	.032/.036 X REQD (OR EQUIV)
201	1	CRES TYPE (OR EQUIV) .0045 WIRE CLOTH	2 X 2

LIST OF MATERIALS

SPECIFICATION
PYRALIN 35-503 PER
N/R SPEC #MB0130-081
GLASS FABRIC POLY-
IMIDE IMPREGNATED

NORTH AMERICAN AVIATION, INC.
SPACE and INFORMATION SYSTEMS DIVISION
TOOL ENGINEERING

POLYIMIDE COVERING
TC-124 | SONY TAPECORDER
SEE SHT 1R-1
G2 | MD-015679

MD-015679

THIS PRINT REDUCED HALF SIZE

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OF POOR QUALITY

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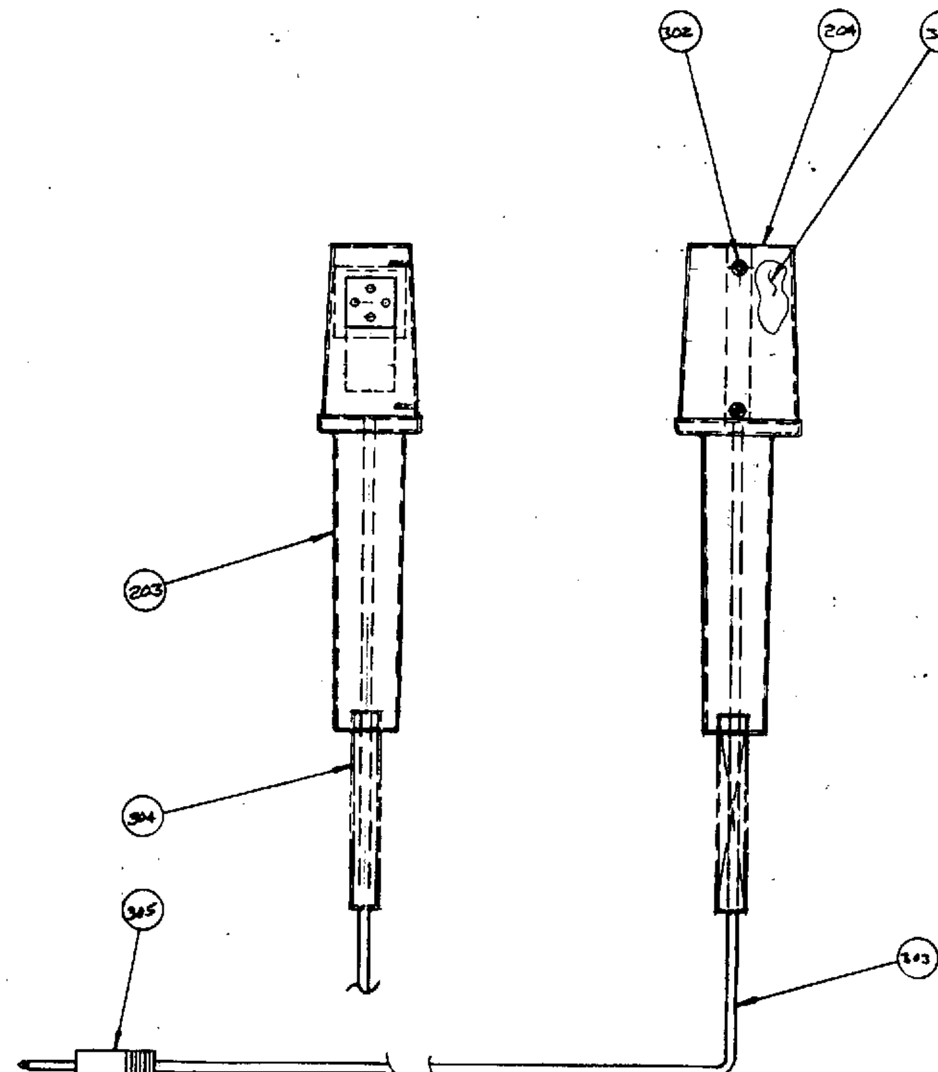
Figure 2-1. Polyimide Covering (Sheet 2 of 3)

FOLDOUT FRAME 1

FOLDOUT FRAME 2



NOTES

1. MAY BE PURCHASED FROM:
   1 ALLIED RADIO
   2 CALIF SPRING
   3 TENSOLITE
2. BOND -203 TO HANDLE OF MICROPHONE WITH RESIWELD #FE-529-5 (OR EQUIV) H.B. FULLER CO. LA MIRADA, CALIF.
3. POT WITH RTV PER MB0130-019

DET NO.	NO. REQD	DESCRIPTION	SIZE
305	1	TINI PLUG	
304	1	SPRING	#164C
303	1	WIRE	#RG188 A/U AWG 25 X 6FT LG
302	2	FLAT HD MACH SCR (CRES)	#2-56 X 1/4
301	1	~~MICROPHONE (SEE)~~	

LIST OF MATERIALS

Delete → 301

NORTH AMERICAN AVIATION, INC.

POLYIMIDE COVERING

TC-124 | SONY TAPECORDER

SEE SHT 1R-1

G3 | MD-015679

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Figure 2-1. Polyimide Covering (Sheet 3 of 3)

TASK 3

TASK DESCRIPTION (Figure 3-1)

1. Fabricate two complete shaver cases of glass fabric/DuPont 4701 polyimide resin utilizing government-furnished mold SEC 42100651.

2. Ship completed cases to NASA MSC for use on the Skylab program.

DISCUSSION

A design drawing, MD 015685 (Figure 3-2), was made of the polyimide case for the mechanical shavers. NASA-furnished stainless steel molds work well in producing close tolerance parts. Skins were made from four plies of DuPont 35-503 material.

Both units requested were tested at NASA JSC for use on the Skylab program.

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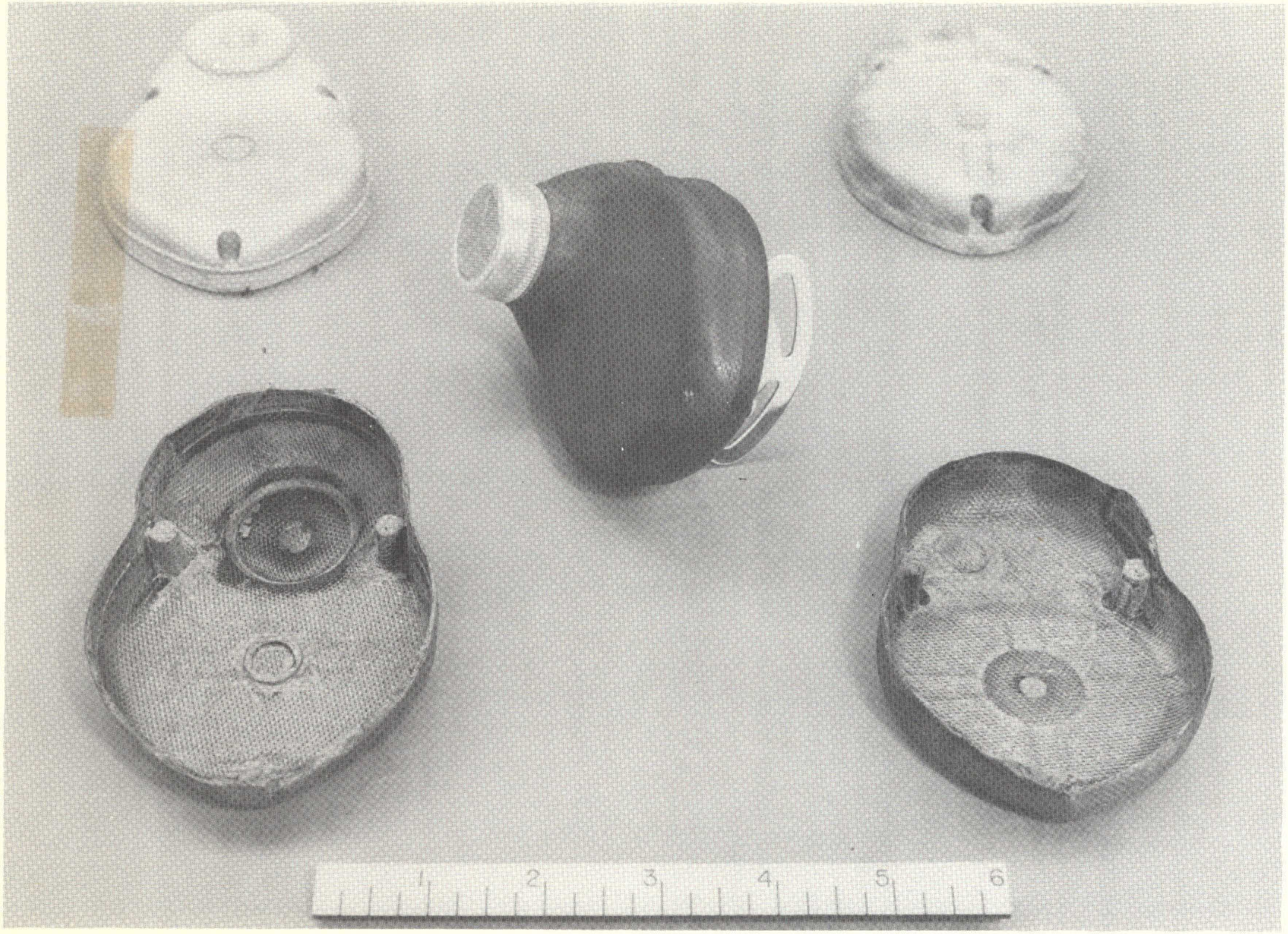

Figure 3-1. Shaver Cases

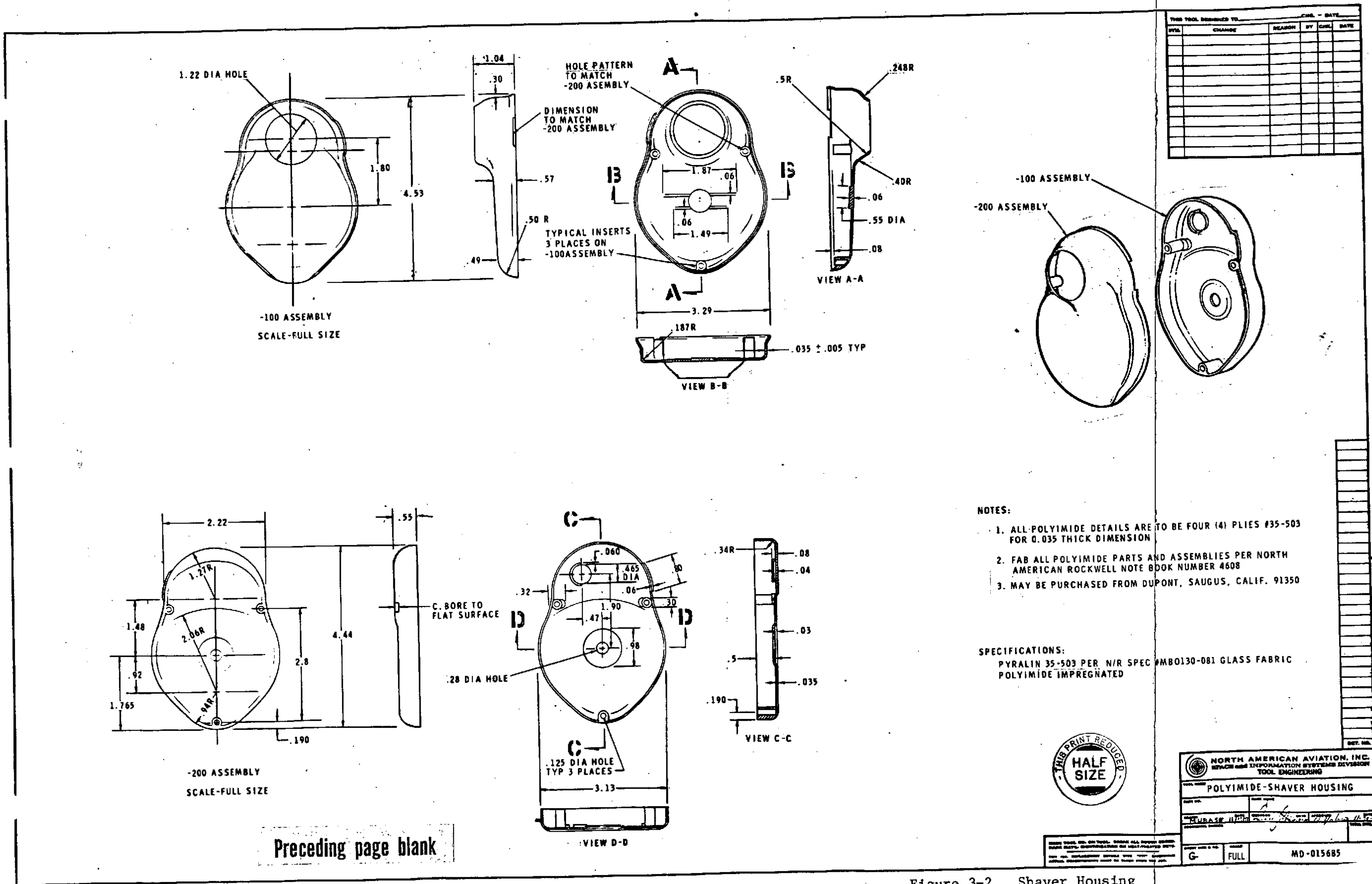

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Figure 3-2. Shaver Housing

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TASK 4

TASK DESCRIPTION (Figure 4-1)

1. Add informational decals to the seven flight class (Skylab) tape players previously defined by Task 2. Decals shall be Scotch-Cal or similar flight-qualified types and indicate "Rewind," "Stop," "Play," and "Fast Forward" positions on Attachment No. 1. Include all decals presently shown on Rockwell drawing MD 015679, Revision A. Adhesive used to bond labels to tape players must be also flight-approved.

2. Delete requirement for Rockwell to conduct functional check on modified tape players as required by item a (2) of Task 2 (all functional checks shall be performed at MSC).

DISCUSSION

This task was performed as indicated with Epon 954 used to bond labels to the tape recorders.

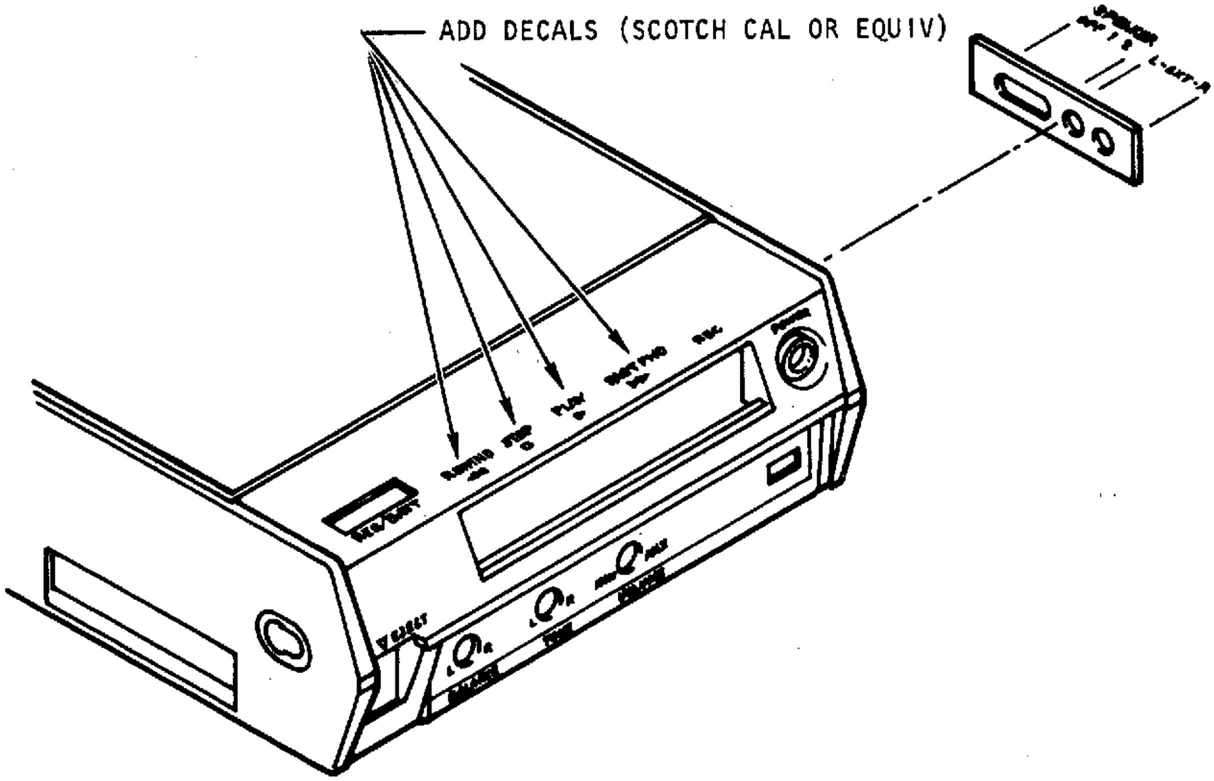

Figure 4-1. Informational Decals

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TASK 5

TASK DESCRIPTION (Figure 5-1)

Fabricate nine polyimide 35-503 covers and assemble covers on nine government-furnished Sony tape players and microphones in accordance with Rockwell drawing MD 015679 (Figure 3-1).

DISCUSSION

These additional recorders were modified with PI laminate material in the manner described in Task 2 with only minor problems encountered. The aluminum sidewall plates and edge strips of most of the recorders were scratched or gouged and had to be replaced.

7003-77-2

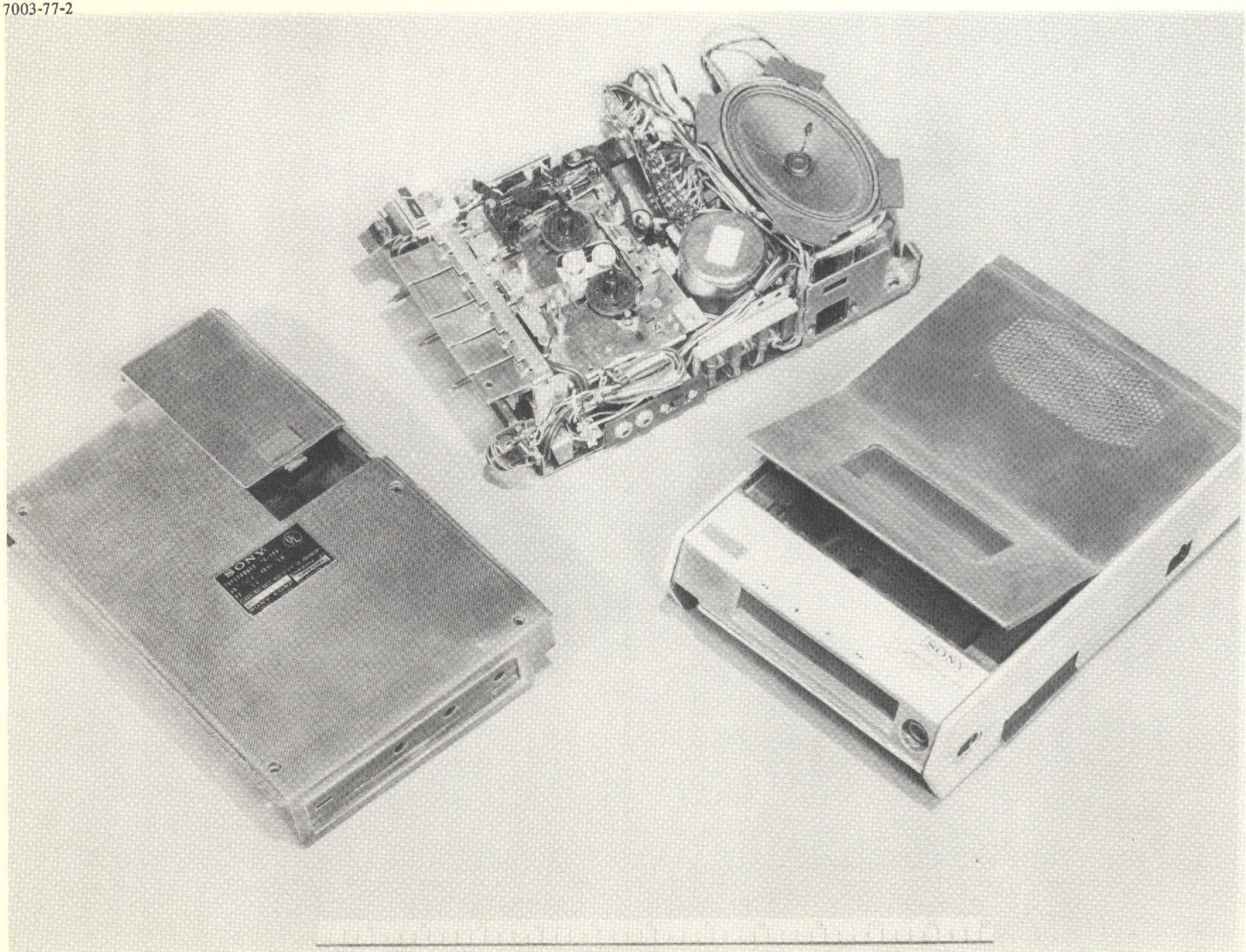

Figure 5-1. Polyimide 35-503 Covers

TASK 6

TASK DESCRIPTION (Figure 6-1)

Fabricate 13 polyimide 35-503 firemen's helmet shells and fully outfit each with liner, edge molding D ring, chin strap, and polysulfone visor in accordance with American Sports Company, Inc., Compton, California, standard design. Helmets shall be identical to prototype previously shipped to NASA JSC.

DISCUSSION

Two fireman hat tools were received on loan from American Sports Co. The first tool break-in trial run resulted in a precipitated polymer cure condition. This was attributed to the high flow (required for Apollo use to obtain low resin content) condition of the Pyralin 35-503 prepreg material. To obtain a high impact property on the helmet, a higher resin content was required. To obtain this higher resin content, the following tests were conducted:

1. Less bleeder fabric - This was attempted in the first helmet and resulted in polymer precipitation.

2. Stage cure 35-503 at a longer time in the lower temperature regime; i.e., 200°F for 16 hours under full vacuum.

3. Change to a higher staged, lower flow prepreg such as 35-512. This is extremely difficult since the 35-512 is a relatively dry "boardy" material.

4. Use combination of Nos. 1 and 3 and intermediate of 2.

The most successful approach proved to be No. 2. For aesthetics multiple coats of PI 4701 resin were applied and cured on the outside surface of each helmet.

Figure 6-1. Polyimide Fireman's Helmet

TASK 7

TASK DESCRIPTION

1. Fabricate one driver protection cowling of polyimide 35-503 for 1972 model All American Races, Inc. racing car.

2. Coordinate design details and schedule with All American Races, Inc. Intent of cowling is to protect driver from burning fuel.

3. Coordinate type and quantity of evaluation data to be obtained by All American Races, Inc. during actual usage of cowling for dissemination to NASA and other concerned agencies.

DISCUSSION

In mid-April 1972, All American Racers, Inc. informed Rockwell that the cowling delivery date of mid-April was too late for use on 1972 model cars. This task was subsequently dropped from the program.

TASK 8

TASK DESCRIPTION

1. Study commercial airliner passenger compartments (specifically the Boeing 737 aircraft) to identify flammable components and structures that could reasonably be replaced or covered by polyimide 35-503 laminate or any other polyimide system. A few possible components for replacement are floor panels (which would require a high-strength sandwich), window moldings, wall and ceiling panels, seat backs, pull-down tables, overhead luggage racks and end caps, covers for personal service units, air ducts, window shades, and side panels.

2. Prepare a data matrix containing the following minimum information:

   a. Selected component or structure name.

   b. General dimensions of the item.

   c. Weight of standard item (include specific aircraft identification).

   d. Cost of standard item (include specific aircraft identification).

   e. Estimated weight of PI (polyimide) replacement item.

   f. Estimated cost of PI replacement item based on one, 50, and full-scale production runs. Costs should be predicated on the availability of moldable polyimide, per No. 3 below if feasible.

   g. Relevant mechanical parameters of materials presently being used (tensile strength, impact resistance, etc.) and comparison of these parameters with those of proposed polyimide.

   h. Advantages of PI component over standard component (other than nonflammability) if any.

3. As part of this study, investigate the feasibility of fabricating selected aircraft items by molding instead of by the hand layup technique presently used. Report the types of forming and polyimide molding compounds investigated.

4. Propose surfacing techniques to meet aesthetic requirements (painting, etc.).

5. Submit data from Nos. 1 through 4 above to NASA MSC in the form of a comprehensive final report along with any samples of materials obtained during the study which are germane to the conclusions.

DISCUSSION

The Boeing Company, because of the proprietary information requested by Rockwell to complete this task, felt it could not put itself at a competitive disadvantage even though the study would be of great interest. This task was therefore canceled.

TASK 9

TASK DESCRIPTION (Figure 9-1)

Fabricate 20 shaver cases in accordance with Rockwell drawing MD 015685 (Figure 3-2) for use as Skylab flight hardware.

DISCUSSION

This effort resulted from the successful fabrication and test of the initial set of two shaver cases fabricated in Task 3. No changes were required either to the design or fabrication approach taken in Task 3 to fabricate these 20 additional shaver cases.

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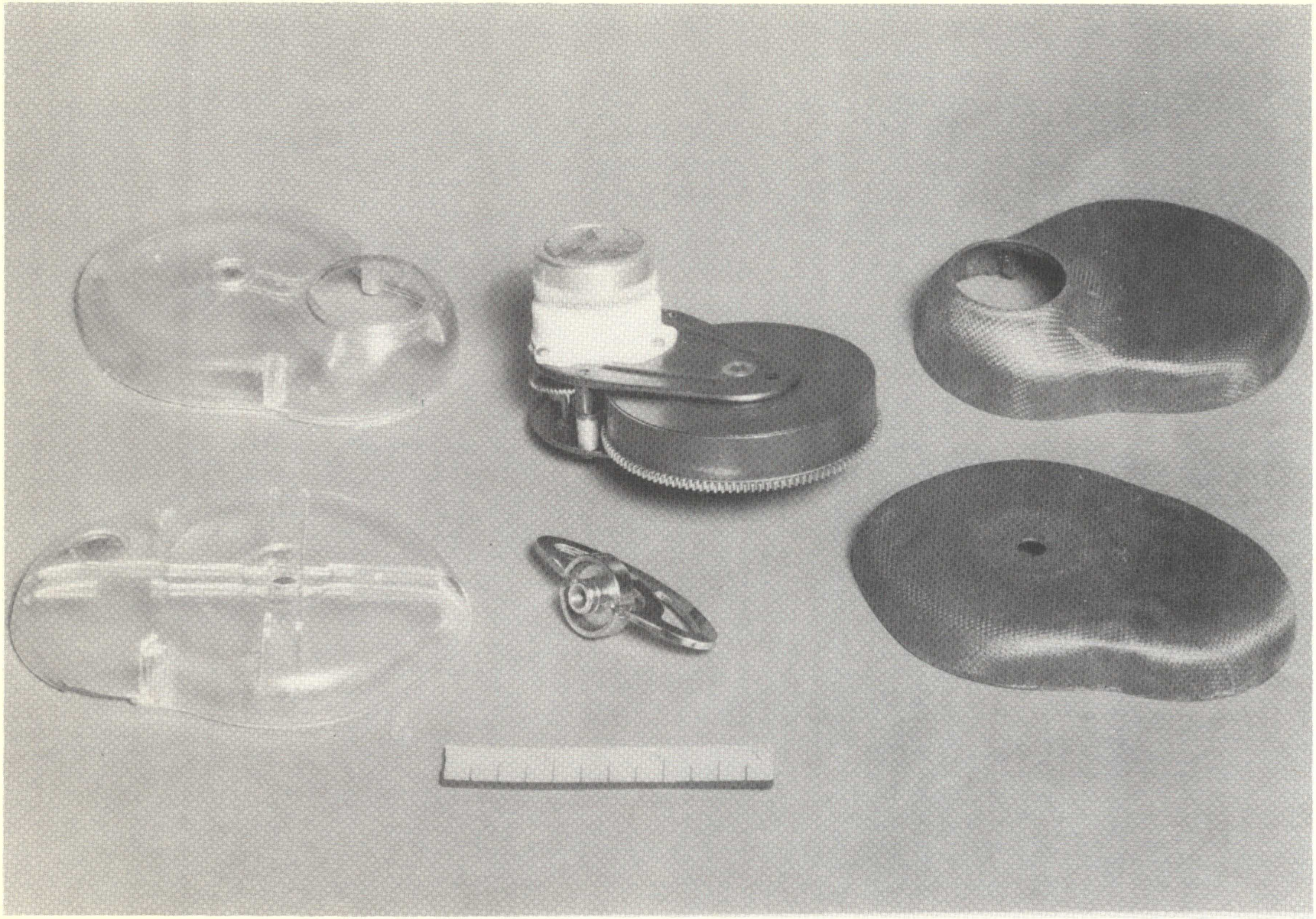


Figure 9-1. Shaver Case

TASK 10

TASK DESCRIPTION

1. Redesign the polyimide covers for the Sony tape players (P/N SEC 13100340-301) to include the following agreed upon alterations:

   a. Add a positive locking slide for the cassette door.

   b. Eliminate the sliding battery cover on the bottom of the tape player.

   c. Add an aluminum sliding battery cover and an aluminum slide track to back of tape player.

   d. Alter electrical connections of batteries to accommodate metal cover.

   e. Add decal to back of tape player indicating proper battery polarity for altered electrical connections.

   f. Apply aluminum foil (0.004-in. minimum thickness) with flight-acceptable adhesive to all seams and edges of tape players underneath (prior to applying) the polyimide cover.

   g. Identify all tape players incorporating the above design features as P/N SEC 12100240-302 to differentiate them from the earlier dash 301 units.

2. Alter Rockwell drawing MD 015679 (Figure 2-1) to reflect the above design changes as Revision B. NASA approval of Revision B of the drawing shall constitute final approval of the redesign. Provide two copies of the approved drawing MD 015679, Revision B, to EC78/ Materials Development Section.

3. Reference Task 5 of Contract NAS9-12302. The MD 015679, Revision B, configuration change shall be effected on tape player S/N 1008 through S/N 1017, inclusive.

4. Tape players S/N 1001 (prototype) through S/N 1007 shall be returned to Rockwell and altered to the MD 015679, Revision B, configuration.

5. Rockwell shall provide minor repairs as required on tape players which may be damaged at MSC during training operations.

DISCUSSION

The above effort was accomplished as directed without any problems.

TASK 11

TASK DESCRIPTION (Figure 11-1)

Fabricate one prototype interior panel of polyimide foam spacers sandwiches between PI 35-503/glass laminate skins in accordance with Figure 11-1. Dimensions are approximate, and tolerances are not critical. The panel is for a capability demonstration only. Dimensions not specified are left to the fabricator's discretion.

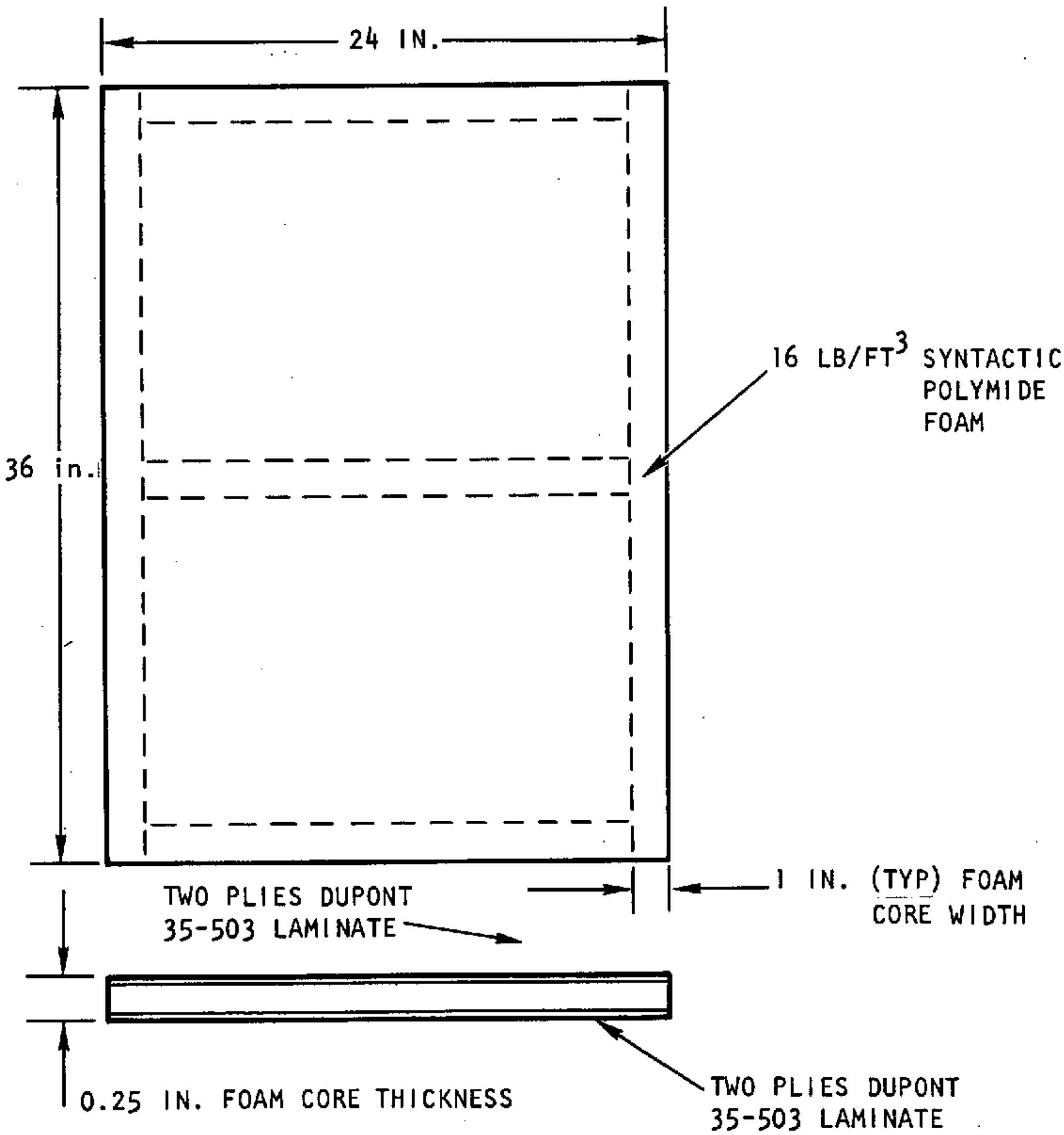
DISCUSSION

This interior sandwich panel (solid door) was fabricated from 16 lb/ft^3 syntactic foam PI core and two-ply PI 35-503 laminate skins bonded to the core with room temperature curing Hysol 954 epoxy adhesive. Dimensions of the foam core were changed to improve structural properties and fabricability of the sandwich panel. Core thickness was increased from 0.1 to 0.25 inches and width of the peripheral picture frame and all intermediate members was increased from 3/8 to 1 inch. To improve the strength further as well as reduce the oil canning of the PI skin, a third 22-inch-long intermediate foam core member, running normal to the minor panel dimension, was added to the center of the panel.

The core was made up of one-inch-wide strips of appropriate lengths cut from a solid rectangular piece of 0.25-in.-thick PI foam core. These strips were subsequently bonded at all intersections over the integral core block design selected to save material, tooling, machining, and processing costs.

Bleeding of the Hysol 954 adhesive through the porous PI skins presented an aesthetic problem, which was minimized by closer control of adhesive thickness. A doctor blade proved adequate for this purpose.

If additional panels of Task 11 configuration are fabricated, it is suggested that (1) more core members of thickness skins be used to reduce skin oil canning, (2) core strength be increased through a density increase or improved material selection or formulation to improve panel durability, and (3) peel strength be increased and bleed-through problems reduced by using an approved unsupport, high-peel, controlled-thickness adhesive such as FM-1000. This bonding technique effectively eliminated adhesive bleed-through problems encountered in Task 11 panel fabrication without effecting bond strength, which is core-property-dependent.



NOTES:

A. DIMENSION NOT CRITICAL
B. BOND CORE TO SKINS WITH APPROVED NONFLAMMABLE ADHESIVE (HYSOL 954)

Figure 11-1. Interior Panel

TASK 12

TASK DESCRIPTION (Figure 12-1)

Fabricate one prototype articulated sliding door panel utilizing Durette 400 fabric as a continuous hinge and polyimide foam faced with PI 35-503 resin/glass laminate as rigid segments in accordance with Figure 12-1. Dimensions provided are approximate, and tolerances are not critical. The door panel is for a capability demonstration only. Dimensions not specified are left to the fabricator's discretion.

DISCUSSION

An interior sliding door panel resembling a rolltop desk cover was fabricated from 16 lb/ft^3 PI syntactic foam, a two-ply PI 35-503 laminate, Durette 7 oz/yd^2 cloth, and Hysol 954 epoxy adhesive. The thickness of the PI foam, fabricated fron a proprietary Rockwell formulation, was increased from the original 0.1 inch to 0.25 inches to strengthen and rigidize the panel design. Subsequently, the main 3/8-inch-wide x 24-inch-long door segments were machined from an open-faced sandwich panel made earlier by laying up and curing the PI laminate directly on the flat machined-to-thickness foam core block. Peel properties of both ends of the panel were increased by extending the upper layer of the two-ply skin over the 24-inch ends of the PI foam core. Bond between the skin and core was effected by the excess PI resin in the PI 35-503.

The 3/8-inch x 24-inch elements were bonded to the flexible Durette fabric with Hysol 954 adhesive, carefully doctor-bladed onto the foamed surface opposite the PI laminate. While control of adhesive width and thickness was possible on the foam, the variable wetting characteristic of the Durette fabric resulted in an irregular bleed-through pattern on the opposite side of the fabric. Some resin extended into the hinge area between elements, but because the adhesive was thin, did not adversely affect fabric flexibility.

The recommendations made for improving Task 11 panel also apply here, with emphasis on controlling bleed-through and peel properties.

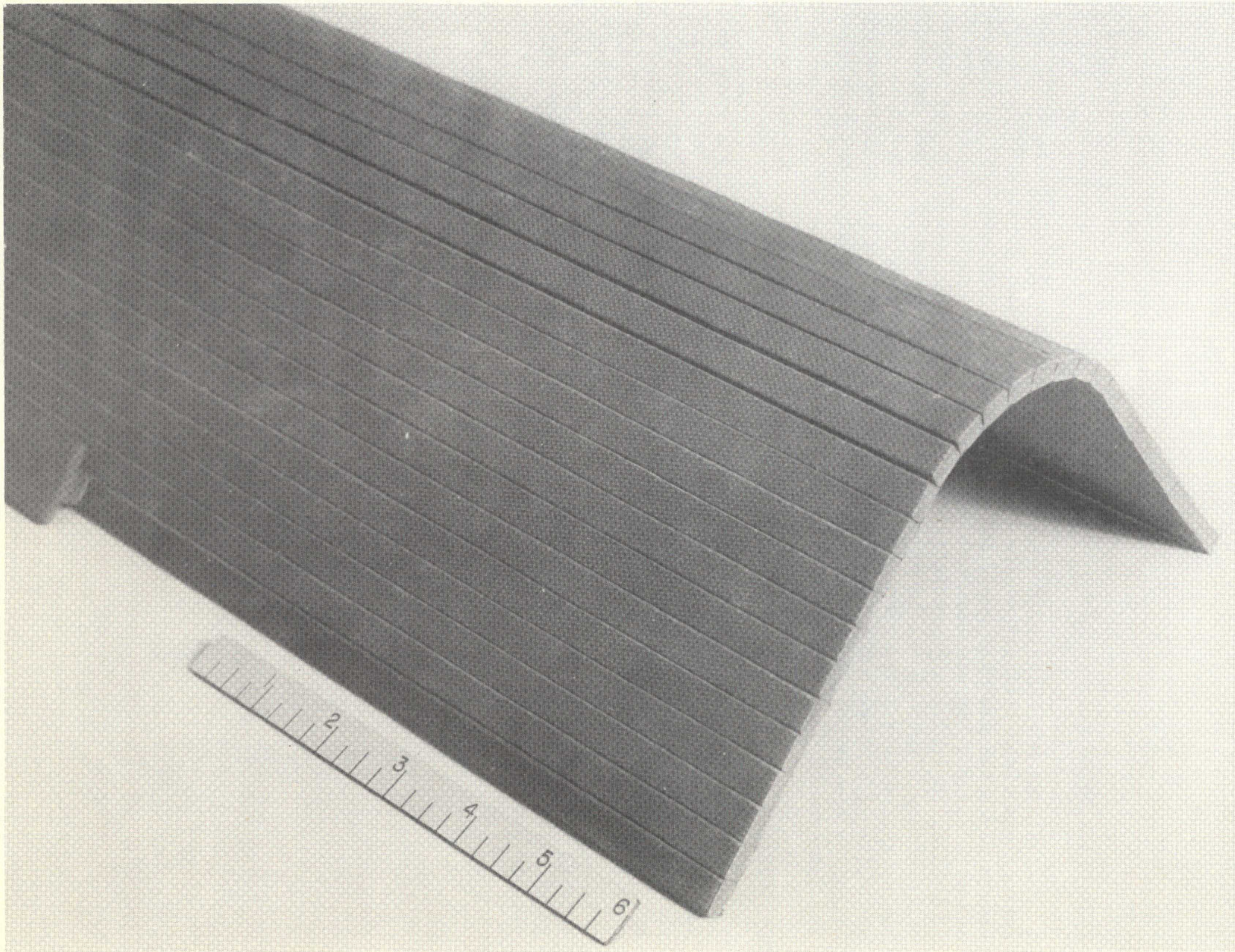


Figure 12-1. Articulated Sliding Door Panel

TASK 13

TASK DESCRIPTION (Figure 13-1)

Fabricate one adjustable shutter assembly primarily utilizing PI 35-503 resin/glass cloth laminate as the structural material. In accordance with Figure 13-2, the assembly shall have a base support adequate for standing the assembly upright for display, polyimide slats secured to the vertical members by bonding to strips of Durette tape, and an adjustment mechanism for varying the view angle through the louvers. The adjustable assembly is for capability demonstration only; dimensions provided are approximate, and tolerances are not critical. Other concepts of adjusting the lower view angle may be submitted by the vendor for consideration if desired. Dimensions not specified are left to the fabricator's discretion.

DISCUSSION

The adjustable shutter assembly panel, which is essentially a space-age Venetian blind, was fabricated from PI 35-503 horizontal blinds and vertical channel frame members, Durette 400 fabric tape flexible joints, Hysol 954 adhesive, aluminum adjusting mechanism, and a limited amount of PI foam to stiffen the vertical PI channels. While it was the most complicated of the three panels fabricated, no difficulties arose, not even the adhesive bleed-through problem noted on the other interior panel structures. The only suggestion for future units of this type is to use high-peel FM-1000-type adhesive to improve long-time durability of peel prone joints.

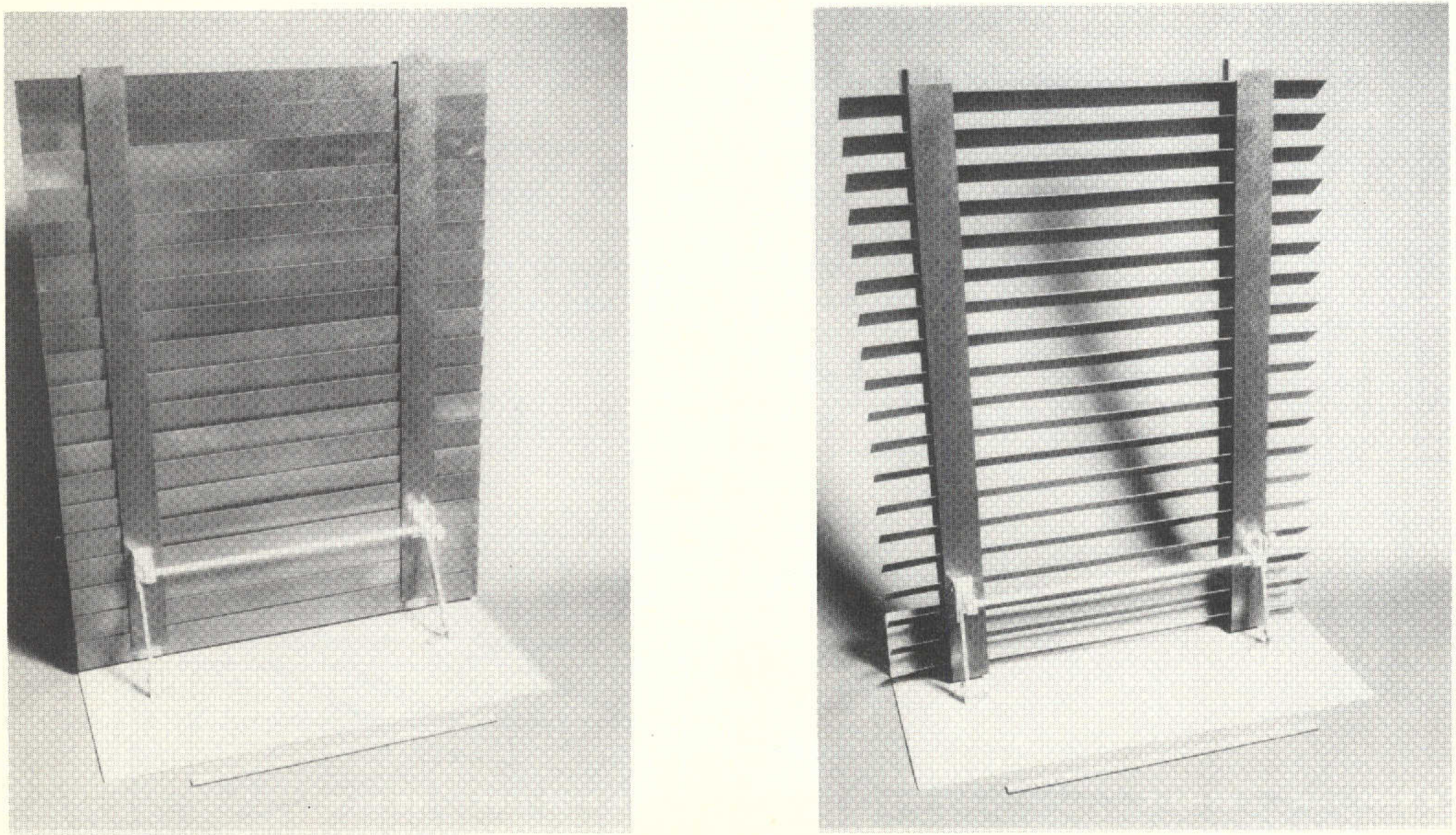


Figure 13-1. Adjustable Shutter Assembly in Open and Closed Positions

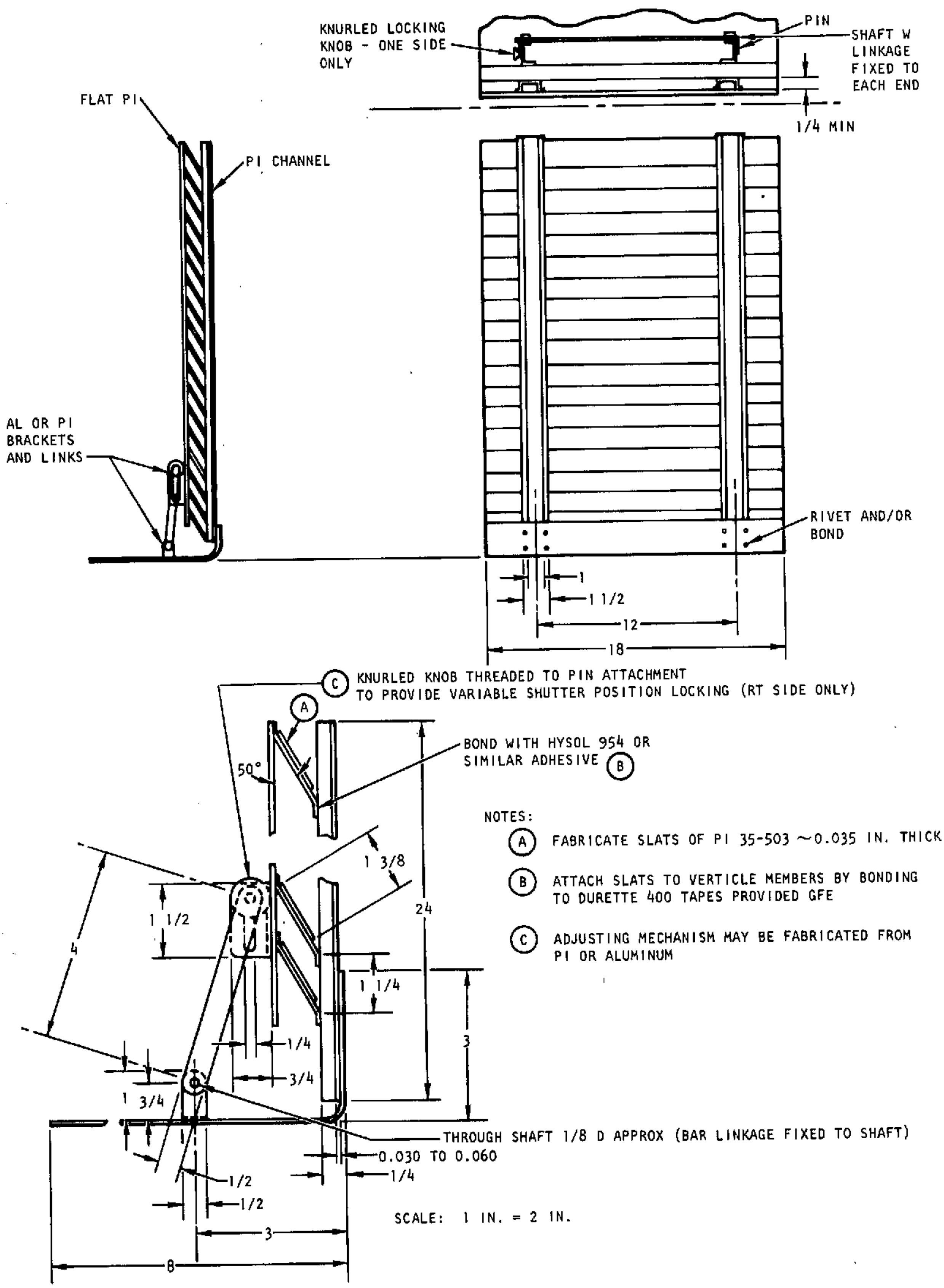


Figure 13-2. Adjustable Shutter

TASK 14

TASK DESCRIPTION (Figure 14-1)

Fabricate two prototype sound-absorption panels having a solid polyimide/glass laminate back face, an expanded polyimide/glass core filled with low-density polyimide foam, and an open mesh polyimide/glass grill cloth front face, in accordance with Figure 14-2. One panel shall incorporate a polyimide foam of approximately 0.6 pcf density and the other a foam of approximately 7.2 pcf density. The panels shall be shipped to Rockwell, Downey, for the addition of a cosmetic perforated film (to be identified at that time) on the front face.

The fabrication of the described panels is intended to:

1. Investigate the feasibility of a single-step curing, bonding, and foaming operation to fabricate the four-part sandwich.

2. Gain insight into the cost and technical problems involved in polyimide expanded core sandwich fabrication.

3. Investigate the sound-absorption characteristics of the described panel for potential use in noise reduction in vehicle interest.

Rockwell shall provide a detailed report of the fabrication operations performed on the panels, including all problems encountered and descriptions of materials used. Figure 14-2 provides general dimensions and compositions of the two panels. Tolerances are not critical. Dimensions and details not specified are left to the discretion of the vendor.

Discussion

Requirements for the acoustical insulation materials for Panels 1 and 2 were changed to 1 lb/ft^3 PI foam, for Panel 1, and 0.6 lb/ft^3 aircraft fiberglass, for Panel 2. The changes were made after efforts proved unsuccessful in foaming Monsanto's RI 7271-01 foam in closed or partially closed molds. Pressure built up to the mold, greatly inhibiting the foaming action. Various techniques such as preheating the mold and extensive venting of the mold, in combination with various cure cycles, were tried without success. A second approach, where free foamed material is shredded and sintered in a closed mold, provided good uniform foam in densities above 4 lb/ft^3. However, when the shredded foam particles were molded around the core, nonuniform densities and crushing of the low-cost PI glass core resulted. This was attributed to the low flow characteristics of the foam particles. Sintered foam specimens with densities below 4 lb/ft^3 fell apart since it was difficult to apply enough particle-to-particle bond pressure and still obtain the desired density of PI foam.

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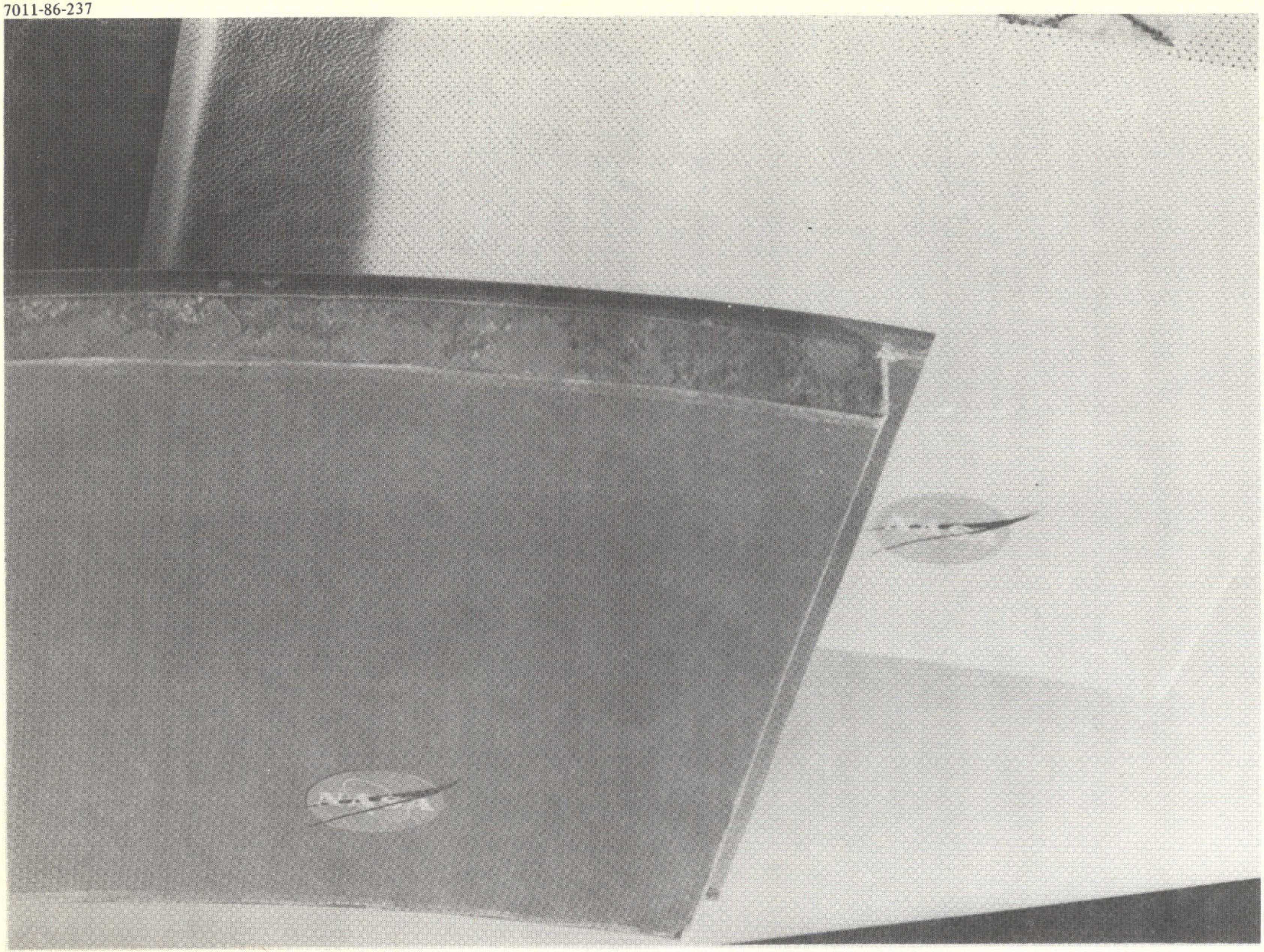
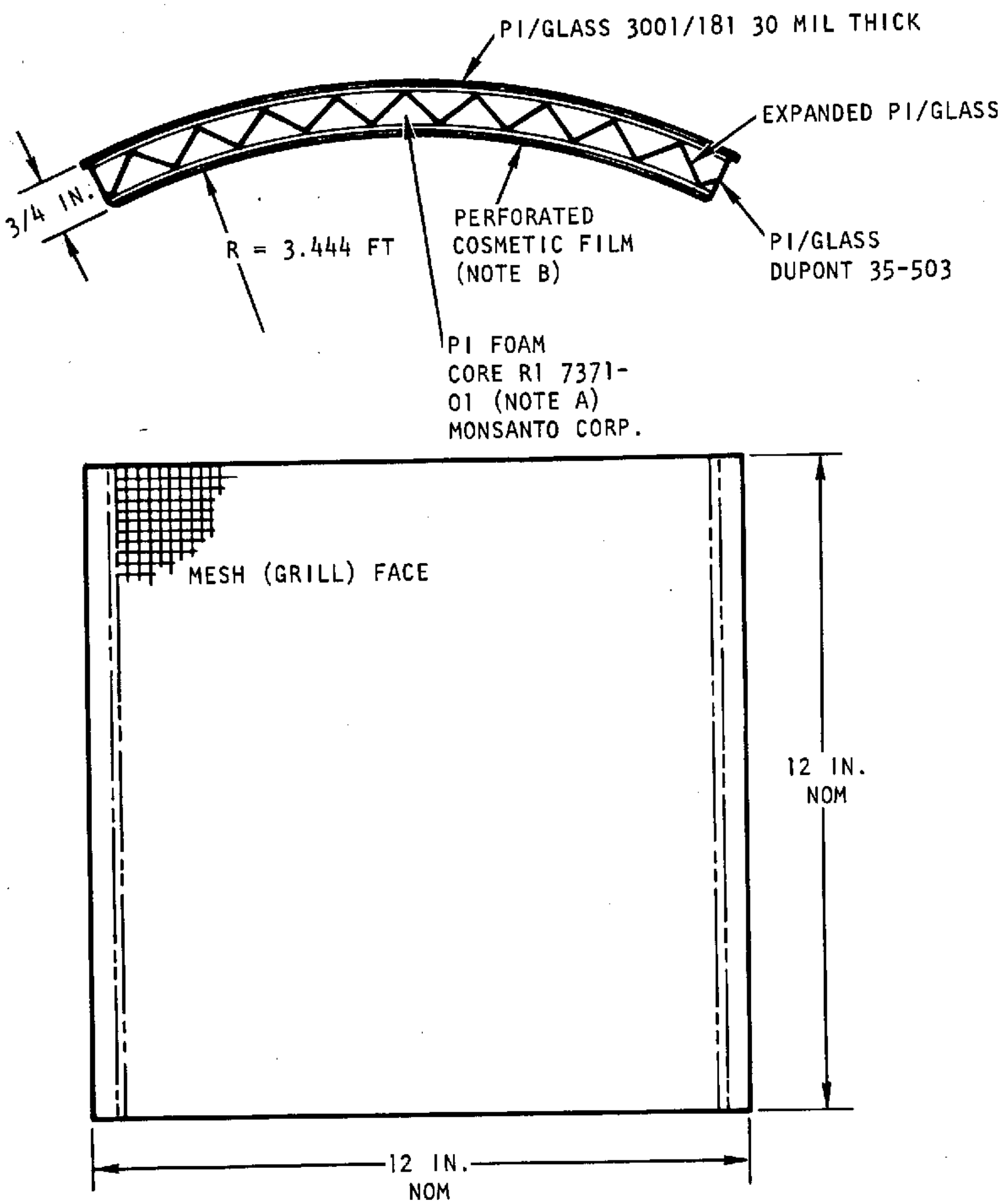


Figure 14-1. Sound Absorption Panels



NOTES:

A. PANEL "A" FOAM DENSITY 0.6 PCF; PANEL "B" FOAM DENSITY 2.2 PCF.

B. FILM TO BE IDENTIFIED AFTER INITIAL RECEIPT OF PANELS AT JSC.

C. NOT TO SCALE, DIMENSIONS NOT CRITICAL.

D. ASSEMBLE WITH POLYIMIDE ADHESIVE.

Figure 14-2. Prototype Panel Sketch

The construction of both aircraft interior acoustical polyimide sandwich panels was similar with the exception of the acoustical insulation and the adhesive materials. Three plies of DuPont 35-503 polyimide/glass and one ply of DuPont open weave 3001/16-453 acoustical material made up the back and front surface, respectively, of Panel 1 and Panel 2. Low-cost core polyimide fabricated by Rockwell was used as the sandwich material in both panels. (Refer to Task 15 for core fabrication details.) Monsanto's RI 7271-01 polyimide foam, free-foamed to an average density of 1 pcf around the low cost PI core, was used as the acoustical insulation for Panel 1, while 0.6 pcf aircraft fiberglass insulation served as the energy-absorbing medium for Panel 2.

The three-ply back face laminate for Panel 1 was bonded to the low-cost core with the Monsanto RI 7271-01 PI foam material during the free foaming of the foam around the core. No auxiliary adhesive system was used. The front one-ply acoustical PI glass laminate was bonded to the low-cost core modes with Hexcel HP 974 polyimide paste adhesive. The blue perforated Tedlar decorative surface was bonded to the one-ply polyimide glass laminate with a heat-activated thermal plastic adhesive coating, which was applied to the back surface of the Tedlar by the vendor.

Once the fiberglass insulation was in place (Figure 14-3), the three-ply back face laminate for Panel 2 was bonded to the PI core with Hysol room-temperature-curing 954 epoxy resin. Before additional insulation was placed on the opposite side of the PI low-cost core, the one-ply acoustical PI glass laminate was bonded to the core nodes with Hysol 954 epoxy adhesive. A white perforated decorative surface for Panel 2 was bonded to the PI glass laminate in the same manner as for Panel 1.

Figure 14-1 shows both acoustical panels with perforated Tedlar decorative surface bond to open weave PI/glass sandwich face.

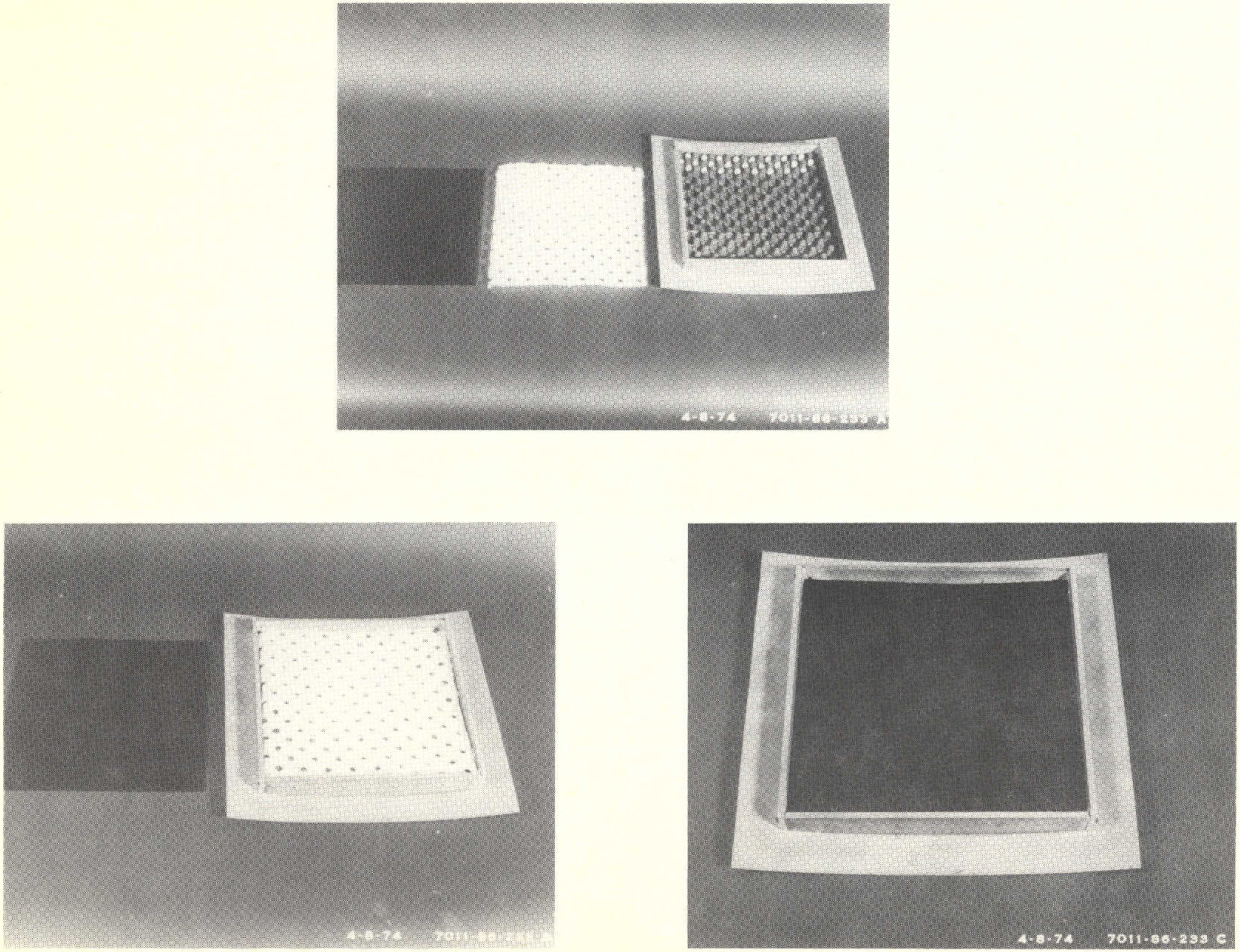


Figure 14-3. Fabrication Sequence of Sound Absorbing Panel No. 2

TASK 15

TASK DESCRIPTION

Define and evaluate the structural properties of polyimide quadricore in sandwich applications:

1. Design and fabricate two (minimum) optimized sandwich structures having polyimide/glass cores of quadricore geometry and epoxy/graphite skins.

2. Conduct the structural panel tests listed below on each of the two panel configurations at room temperature:

    a. Flat tensile
    b. Flat compression
    c. Core shear
    d. Shear modulus
    e. Edge bearing load

3. Review currently available data for specific structural parameters of thermoplastic quadricore and polyimide/glass honeycomb and conduct a structural properties and cost tradeoff analysis against the optimized polyimide/glass.

4. Prepare and deliver to NASA a formal report delineating the test data of Part 2 and the analysis of Part 3 above.

DISCUSSION

Properties of the polyimide low-cost core material were developed as requested. Test data, as well as R&D activity pursued in optimizing the PI core, are presented.

Since their conception, honeycomb sandwich structures have seen a phenomenal usage growth in both the aerospace and commercial industries. The inherent strength-to-weight ratio of a honeycomb panel in comparison to a solid sheet material has provided a tremendous advantage, primarily in the extremely weight-conscious aerospace industry. The mechanics of a sandwich structure design will not be discussed since they are quite familiar to stress, design, R&D, and process engineers.

One of the main objectives in the aerospace industry today is reduction of the initial and operational costs of structural and nonstructural hardware. One of the more costly materials used for high-temperature applications is polyimide honeycomb core. A recent review of commercial core materials and manufacturing processing has revealed a low-cost core fabrication approach that, with modification, could produce a high-temperature polyimide core that would be cost-effective in secondary and nonstructural high-temperature aerospace applications.

This material is formed into a dimpled patterm similar to quadricore to establish both height and formability for the core material. This core has the additional advantage in that its properties are isotropic, whereas honeycomb has significant directional differences in its strength. Until recently quadricore has been prepared primarily from unreinforced thermoplastic with attendant restriction upon its thermal performance. However, work at Rockwell Internation Space Division has shown that core can be prepared from glass fabric and polyimide-resin so that both high-temperature and high mechanical performance can be anticipated with it.

This effort primarily encompassed the following tasks:

1. A survey of the core industry and analyses of configurations for low-cost manufacturing applicability.

2. Determination of tooling requirements.

3. Selection of glass reinforcement materials for subsequent polyimide-resin impregnation adaptable to the selected core configuration requirements.

4. Fabrication of core samples and development of basic physical property characteristics.

Core Configuration

Industry Survey

A survey was conducted of the various core configurations used in the aerospace and commercial industries for sandwich constructions of all types. This included the hexagonal or standard honeycomb, the predominant core configuration; overexpanded (OX) core, a rectangular structure; sine wave types (both flex-core and multi-wave), which feature exceptional formable properties; and a formed core design. Figure 15-1 presents some of the various core configurations. There are, of course, other types, but they are custom, often complex, constructions and not feasible in production of a low-cost material.

The configuration selected for this program was a formed core egg-crate-type readily adaptable to the use of a stretch-type or knit polyimide resin-impregnated glass fabric for forming a single-sheet core. This would eliminate the more costly process of coating and slitting materials and bonding process involving more complex tooling in manufacture of the hexagonal or honeycomb-type core structures.

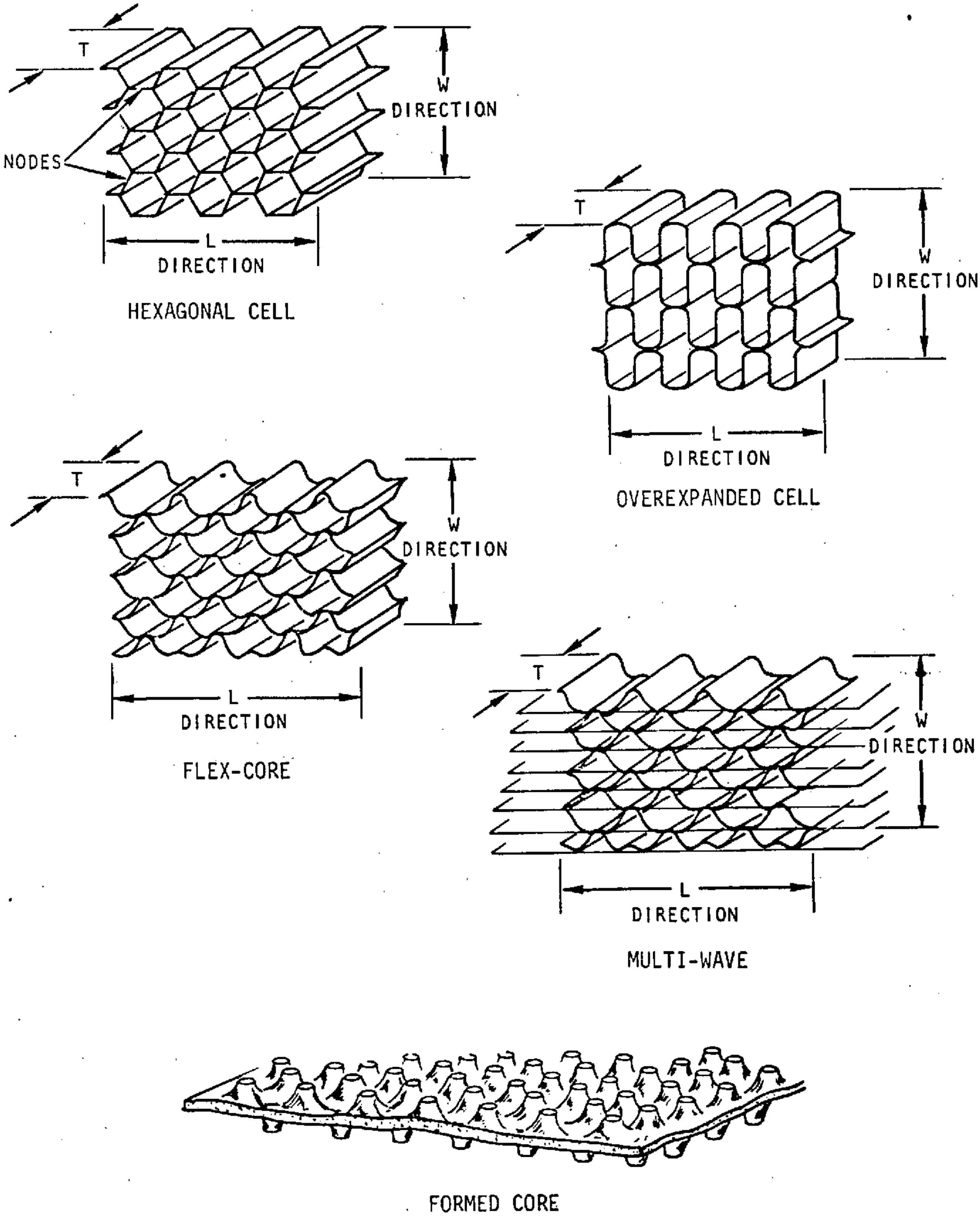


Figure 15-1. Core Configurations

Core Design Considerations

The design consists of trusses gusseted in two directions. The trusses translate most imposed flexural loads into compression and tension of the core and surfaces. Thus, core shear is minimized with this geometry.

These core structures have edge-bearing capabilities equal to or superior to honeycomb because of the core's ability to carry a major portion of the load as well as furnish relatively high values in flexure as compared to other core structures. Stacking of the core node to node, as shown in Figure 15-2, develops a multiple truss pattern that synergistically increases structural values in bending. Stacking and bonding of core, as shown in Figure 15-2, provides a structure that resists bending forces even without addition of skins to complete the sandwich structure. Note also the formability of formed core as shown in the same figure. Thus, the configuration displays both stiffness and formability.

The efficiency of the core structure is determined by a precise formula that defines node diameter, node spacing, and depth of cross section. The design is isotropic; that is, it has equal load-bearing capabilities for both length and width. It is also synclastic, having the same kind of curvature in all directions, and may be easily formed in simple or contoured curves without crushing or expensive machining.

Additional Features

Insulation. The core exhibits K factors superior to honeycomb (hexagonal) core since it provides longer paths of thermal "travel" and is readily adaptable to "dropping in" perforated sheets of foam sheet or fiberglass to greatly enhance the K factors. Figure 15-3 presents comparative thermal conductivity data on honeycomb versus formed core.

Continuous Plenum. The structure has readily available space for wiring, plumbing, or purging without modifications or cutouts, as is necessary with standard honeycomb core.

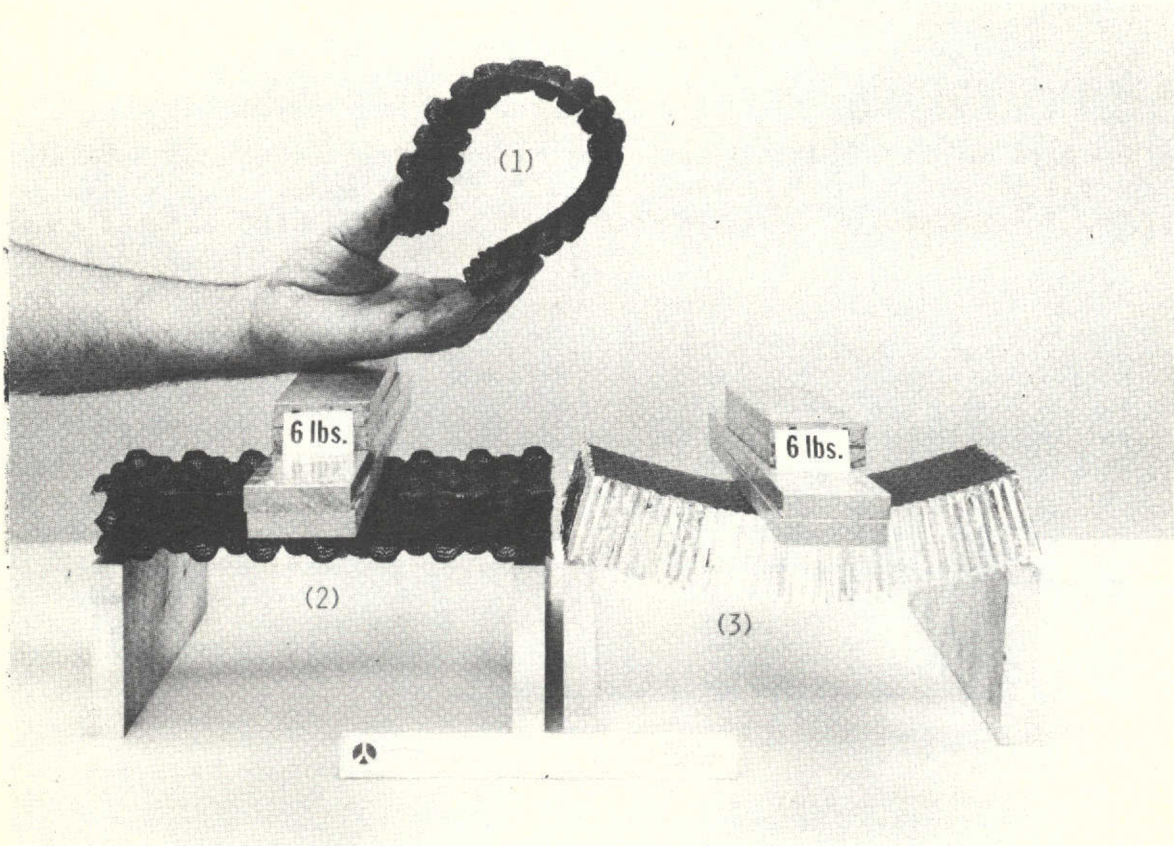
Energy Absorption. The core construction minimizes catastrophic failure resulting from localized damage as it does not readily propagate to surrounding areas.

Acoustical or Attenuation Properties. Acoustical properties of the formed core construction exhibit high sound or energy absorption coefficients, which can be significantly increased by the use of foam sheet or fiberglass. Figure 15-4 is a typical formed core attenuation graph.

Material Development

Reinforcement Selection

The reinforcement selected for this program was a high-strength, nonflammable, high-temperature-resistant, stretchable material—a good description of a knit-glass fabric. To fulfill the requirements for a simple, economical,



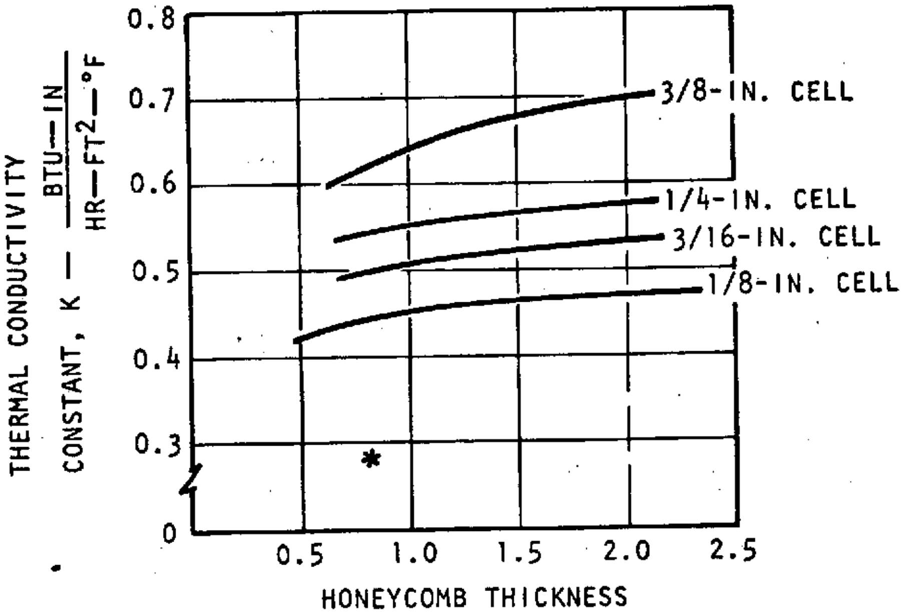
(1) FORMABILITY—3/4-INCH-THICK, 2.3-LB DENSITY FORMED CORE (HAND HELD)

(2) POLYIMIDE/GLASS STACKED FORMED CORE—2-INCH-THICK SECTION OF FORMED CORE; 3 PIECES OF (1) BONDED AT THE NODES

(3) ALUMINUM HONEYCOMB CORE—2-INCHES-THICK

NOTE: BOTH CORE STRUCTURES ARE OF EQUIVALENT DENSITIES—2.3LB/CUBIC INCH

Figure 15-2. Formability and Stiffness Characteristics of Formed Core



* 3/4-INCH-THICK FORMED CORE, 2.3-LB DENSITY

Figure 15-3. Thermal Conductivity Data

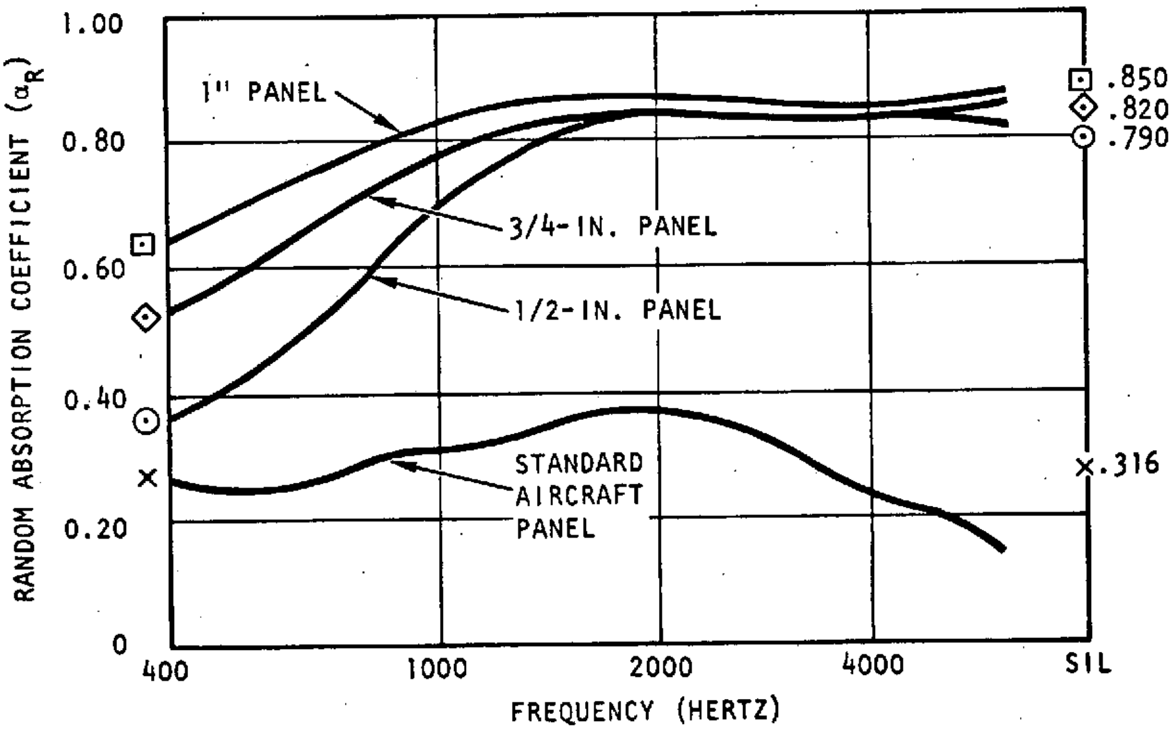


Figure 15-4. Typical Formed Core Attenuation Graph

core fabrication process, the knits must possess stretch characteristics relatively equal in all directions and display sufficient elasticity in the knit configuration to stretch over the compound curves of the core configuration.

Resin Selection

The impregnating resin selected was a polyimide-resin that, in conjunction with a glass fabric, would provide a nonflammable core material if processed by the technique developed by the Advanced Manufacturing Technology group at the Rockwell Space Division. Figure 15-5 shows the polyimide-glass prepreg used in producing the core specimens fabricated in this study. The material utilized a J.P. Stevens double-knit glass fabric (with an A-1100 finish) designated as Style 241548 and a DuPont polyimide Pyralin resin.

Material Procurement

All known suppliers of glass fabrics and specialty weaving and knitting concerns were contacted and presented the "stretch" requirements deemed necessary for adaptation to the formed-core configuration.

Availability of knit glass fabrics for adaptation to the selected core configuration proved to be scarce. Generally, the samples that were procured primarily possessed unidirectional stretch characteristics and did not avail themselves to low-cost processing techniques. They could be worked into the formed-core mold form but did not display the desired orthotropic stretch properties wherein the fabric could be applied to the female mold surface without preforming and then formed and cured with the use of matched molds. The great demand for glass fabrics (in the past year it has exceeded supplies) contributed to some lack of interest displayed by many of the firms contacted.

Owens-Corning Development Laboratories, Ashton, R.I., and Prodesco, Inc., Perkasie, Pa., however, have shown considerable interest in developing glass knits for this program. Their cooperation will be a key element in the further development of a variety of medium density, semi-structural, ultra-lightweight nonstructural cores.

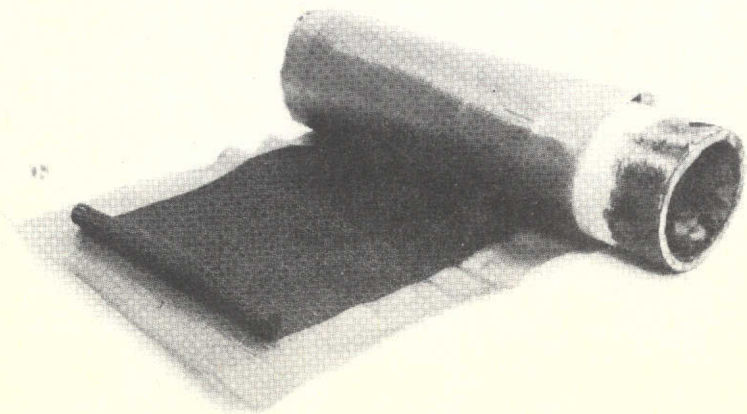


Figure 15-5. Polyimide-Resin Impregnated Double-Knit Glass Fabric

Tooling Development

Matched Tools

Development of tooling to produce the polyimide-resin knit-glass-reinforced core was initiated by obtaining Zn/Al alloy core from Quadricore, Inc., Pico Rivera, California. The Zn/Al core selected for use in producing the matched molds had node diameters of 1/2 inch, node spacings of 1 inch, and a cross-sectional height of 3/4 of an inch.

The Zn/Al core was coated with a release agent, and a high-temperature epoxy casting resin was poured onto each side to produce matched molds with an offset of approximately 10 mils, the thickness of the Zn/Al core. The molds were post-cured at 350 to 400°F for four hours to develop high-temperature properties. The molds are shown in Figure 15-6.

Though core samples were produced by using the matched mold tooling, this technique has quite a few disadvantages in making the formed core.

1. Cost of matched mold tooling is relatively expensive since new sets of matched molds with various offsets would have to be produced for different thicknesses of glass reinforcements.

2. Thermal conductivity of the epoxy mold is rather low and involves a rather lengthy cure time in a heating oven.

3. The vacuum-bagging operation to develop cure pressure is time consuming and does not lend itself to a truly low-cost process.

Vacuum Forming

Because of the listed disadvantages in using matched mold tooling, vacuum-forming tools were fabricated by casting epoxy resins onto Zn/Al and thermo-plastic quadricore to produce a forming tool. This effort was accomplished rather late in the program, however, and has not been fully developed.

A flat aluminum plate with a vacuum port in the bottom and a solid aluminum pressure frame was fabricated for use as the vacuum tool. Holes were then drilled in the lower nodes of forming tool. The core-forming operation was accomplished by laying the polyimide/knit-glass prepreg onto the mold surface (coated with a release agent), placing a silicone rubber sheet exhibiting 300 to 400 percent elongation over the prepreg, and then clamping the peripheral pressure frame over the rubber sheet. The assembly was then placed in an oven. A vacuum source was attached to the vacuum part on the tool, and full vacuum was applied (25 to 29 inches of Hg). Figures 15-7 and -8 show the vacuum-forming procedure. The prepreg material was then drawn into the mold, with the rubber acting as the diaphragm, and cured at the prescribed curing temperatures.

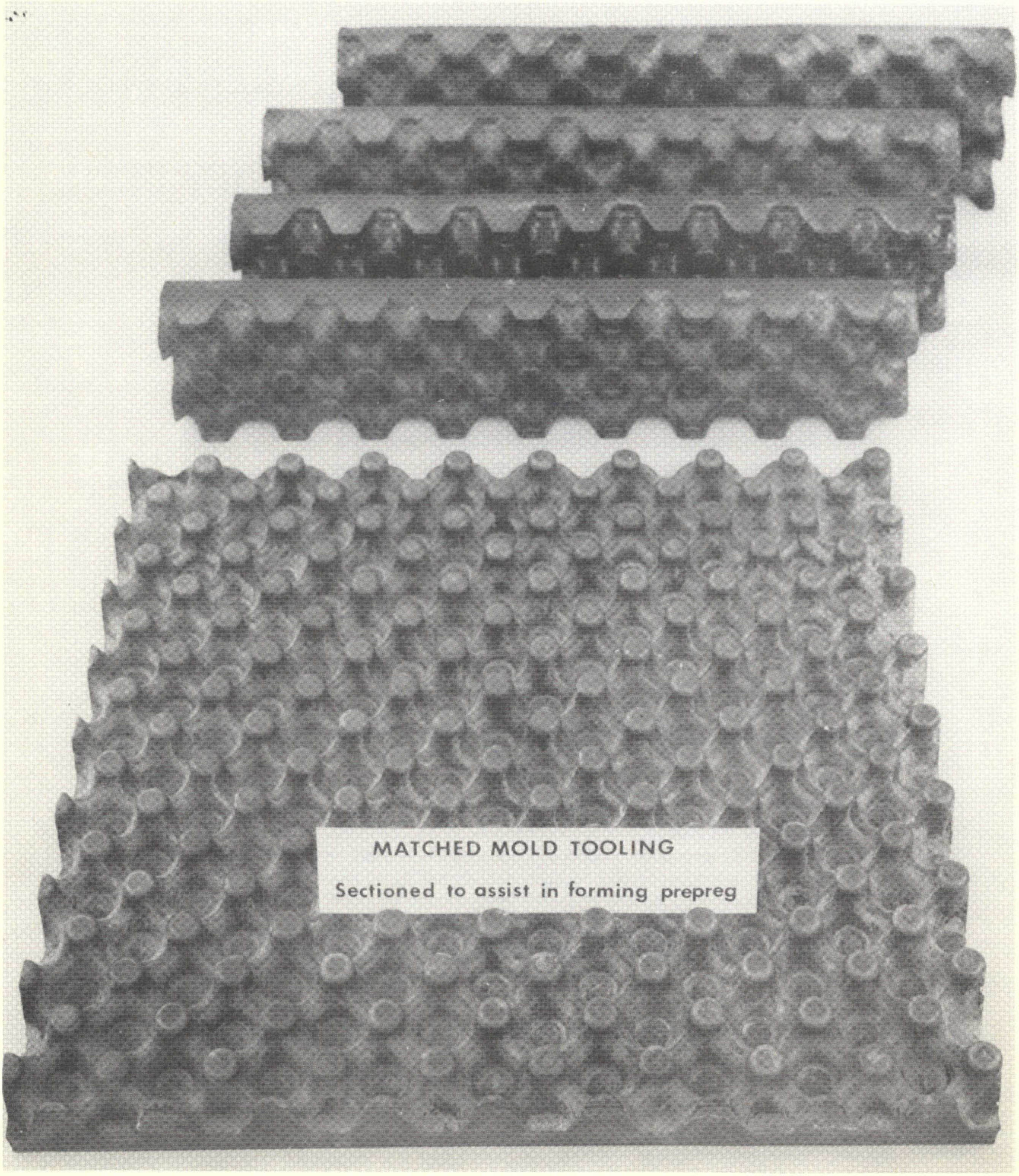


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Figure 15-6. Matched Mold Tooling Used in Production of 3/4-in.-Thick Polyimide/Glass Core

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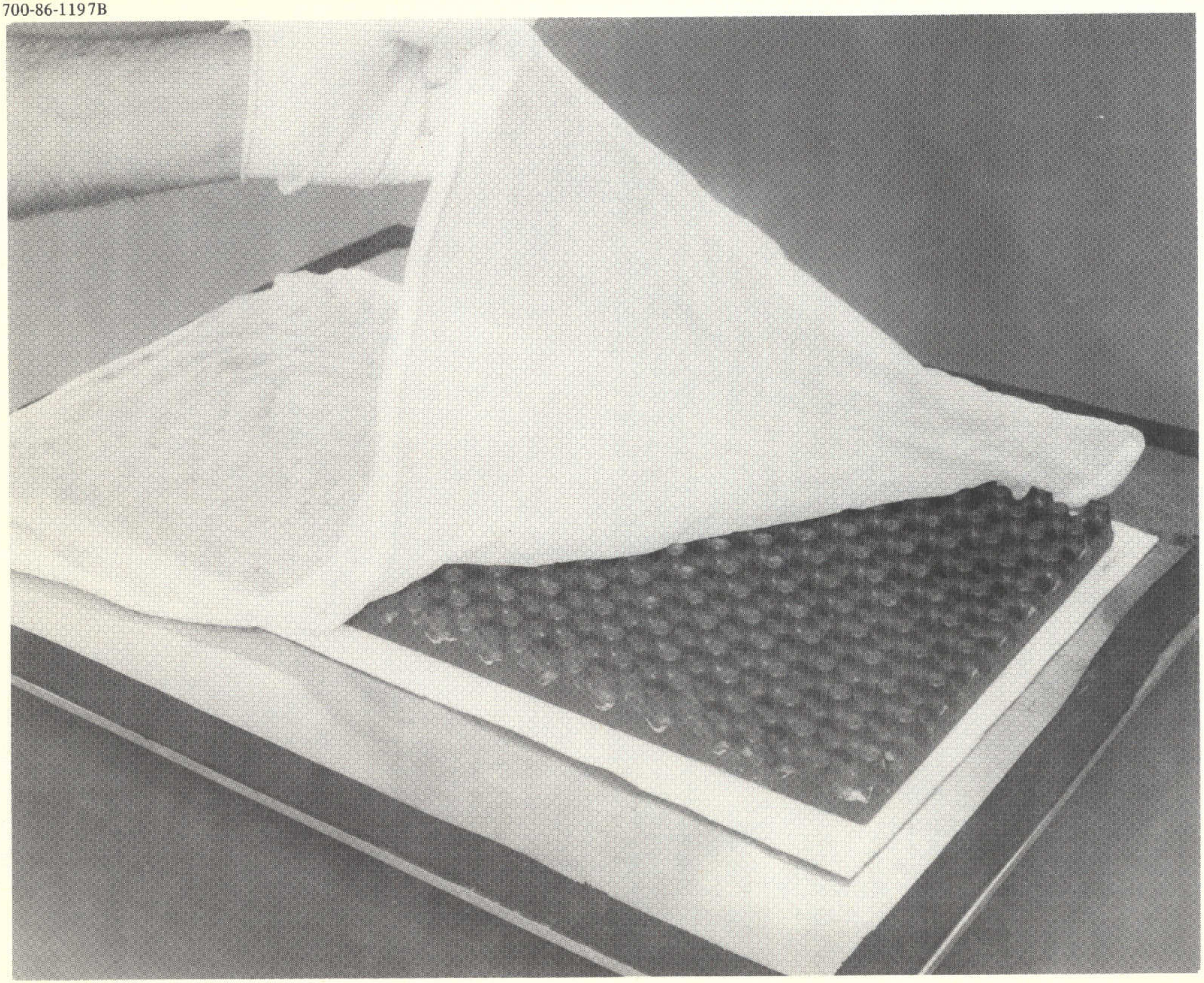

Figure 15-7. Material Draped Over Vacuum-Forming Tool

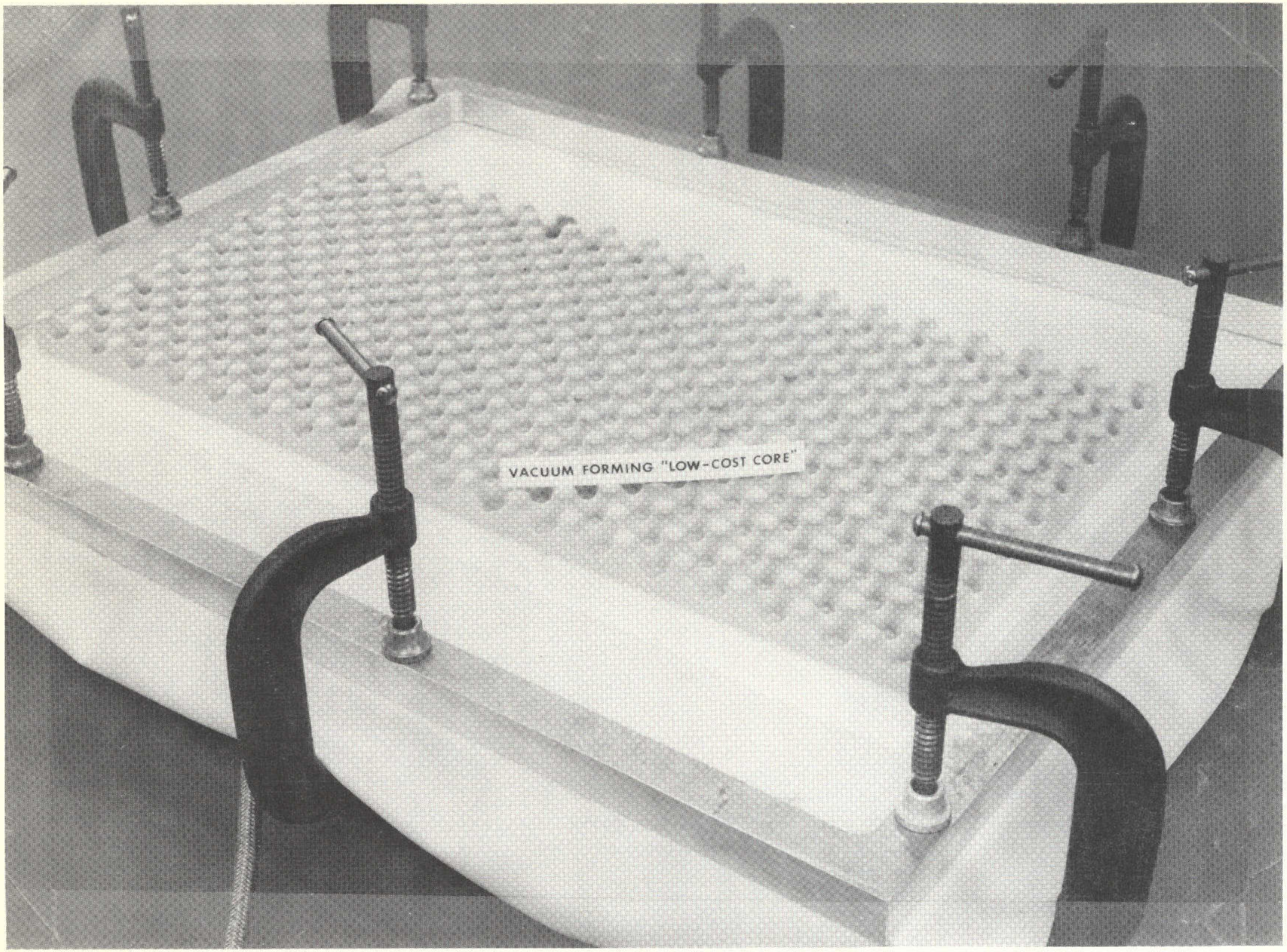

Figure 15-8. Vacuum-Forming—Application of Atmospheric Pressure Forming Rubber Diaphragm to Form Material Into Mold Cavities

Core Fabrication Procedures

A series of knit-glass fabrics was evaluated for adaptability to the contours by placing the fabric on the bottom mold and then compressing it into the contours with the matched mold. None of the materials would adequately stretch over and into the mold configuration unless material was laid over the mold and then pushed into each cell consecutively before the matched mold was applied, as shown in Figure 15-9. Even with assist tooling, which might conceivably perform this job mechanically, it is difficult to perceive using this material in a truly low-cost operation.

The tool containing the polyimide/glass prepreg was then vacuum-bagged, placed in an oven, and cured at 350°F for one hour. After the tool was cooled, the core was removed from the tool and placed in an oven. The temperature was raised from room temperature to 600°F in approximately three hours, and the core was post-cured at 600°F for one hour. This post-cure is necessary for removal of all volatile matter, complete polymerization of the polyimide, and for production of an essentially nonflammable core material even when exposed to flame in a 100-percent-oxygen atmosphere.

A core sample using a J.P. Stevens double-knit glass fabric impregnated with a DuPont Pyralin resin is shown in Figure 15-10. This material produced a relatively light core structure of approximately 2.3 lb/ft^3. In the same photograph is a sample of an ultra-lightweight core material produced from a small sample of very light, thin-gauge, glass fabric. This structure proved to be too flimsy, but does show that cores lighter than the 2.3 lb/ft^3 are feasible, and efforts to produce lighter cores for nonstructural panels are planned for the future. Figure 15-11 shows the 2.3 lb/ft^3 core bonded to polyimide/glass laminate skins with a polyimide adhesive. This combination provides a relatively lightweight, semi-structural, nonflammable sandwich panel that should find use in aerospace and possibly in the commercial industry.

Structural Properties

Specimen Fabrication

Test specimens were fabricated to provide test data on flatwise and edgewise compression strength, shear strength and modulus of elasticity, and flatwise tensile strength. In all cases, face sheets were bonded to the core material and tests performed at room temperature.

A. Materials

Quadricore - The quadricore was fabricated from Type 3001/241548 glass-fabric polyimide prepreg. The core was formed by molding the fabric in notched-metal dies having the quadricore form. Cure was accomplished at 350°F for two hours with a post-cure at 600°F for one hour. The finished material is open weave, with 0.50-in.-diameter nodes spaced on 1.00-in.-diagonal centers. Opposite sides of the panel have the nodes centered between one

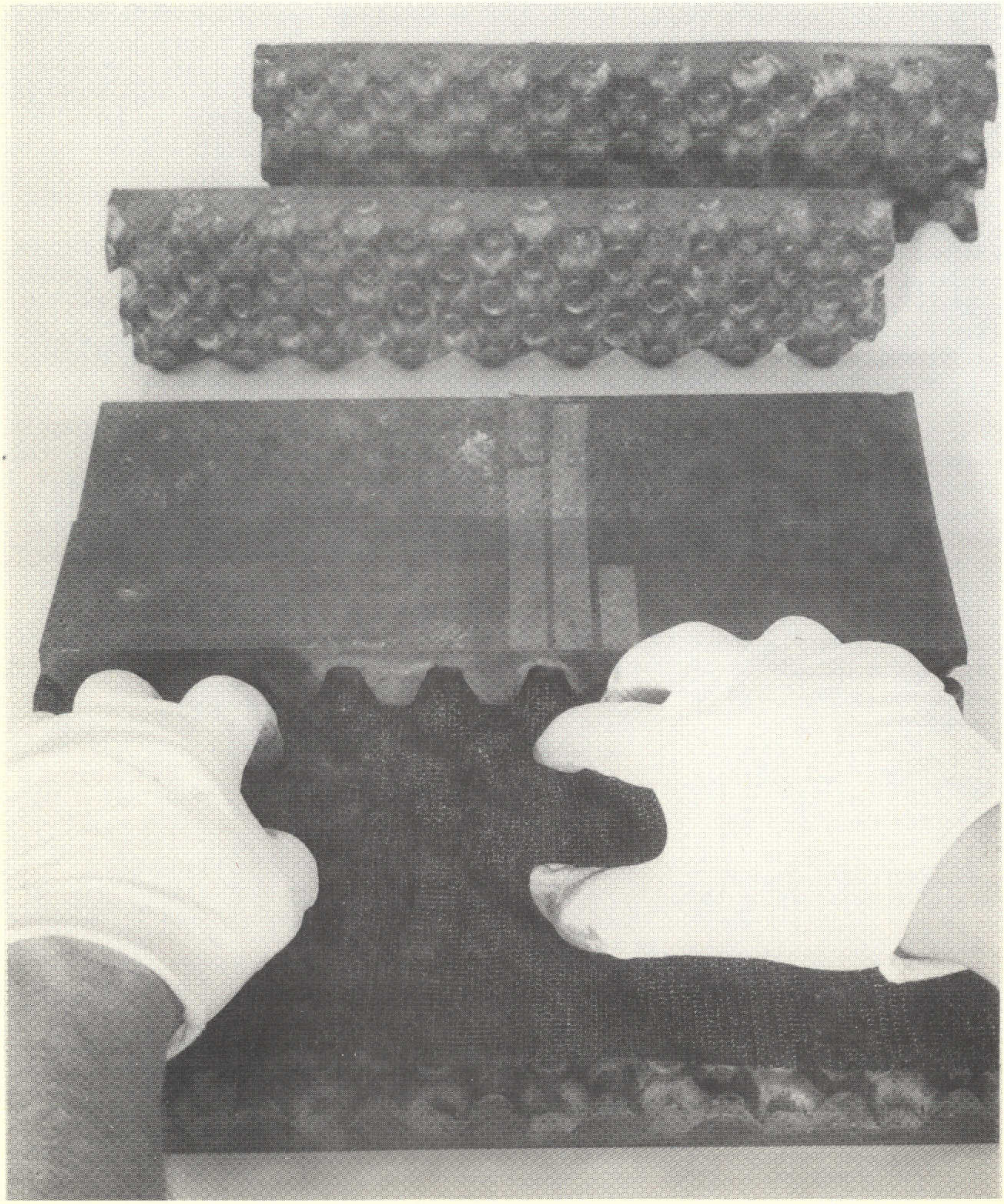

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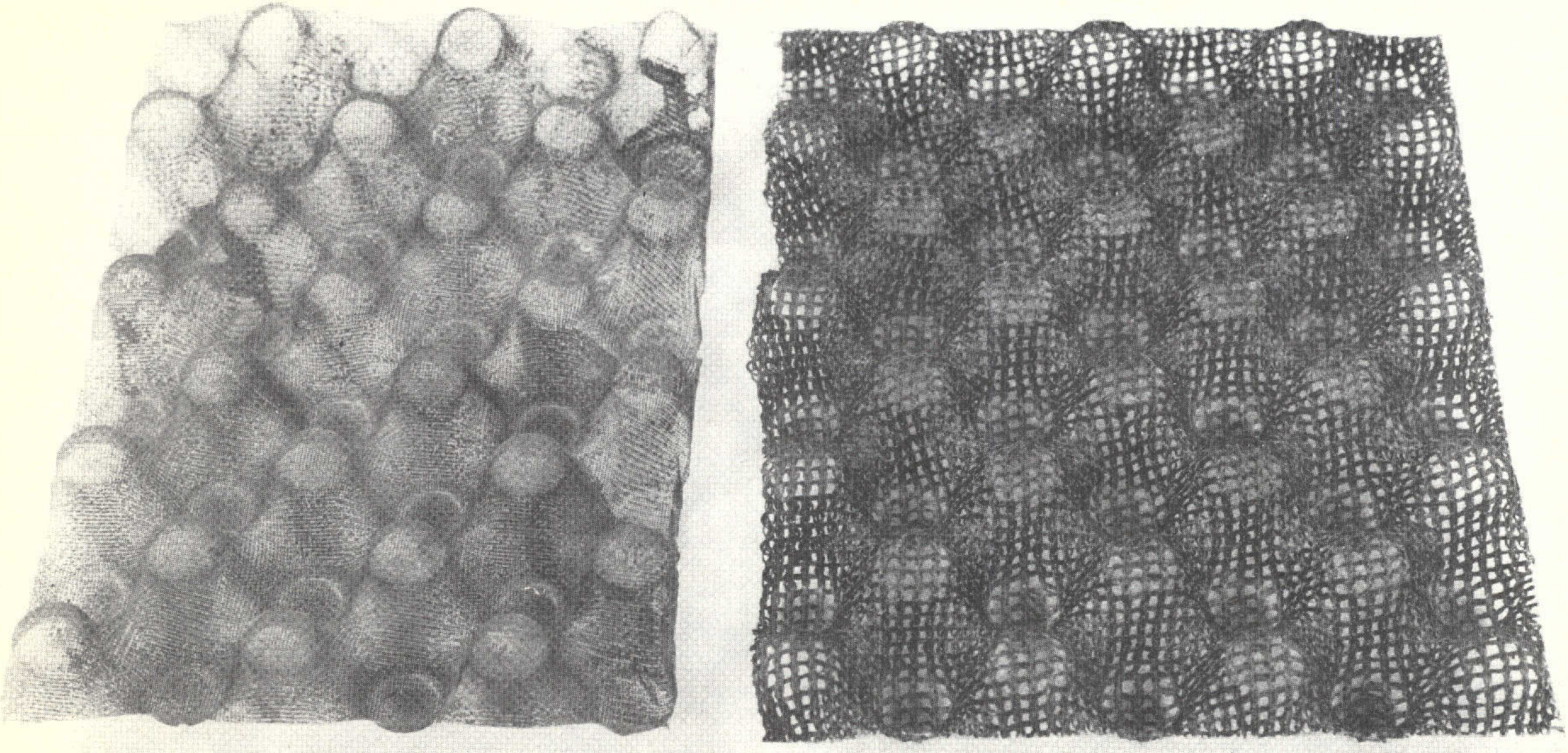
Figure 15-9. Preforming Polyimide/Double-Knit Glass Fabric (DuPont 3001/241548) Into Mold



~0.8 lbs/cu.ft. ~2.3 lbs/cu.ft.

Figure 15-10. Ultra-Lightweight Polyimide Glass Core (Left) and Semi-Structural Core—A Polyimide Resin-Glass Fabric Structure (Right)

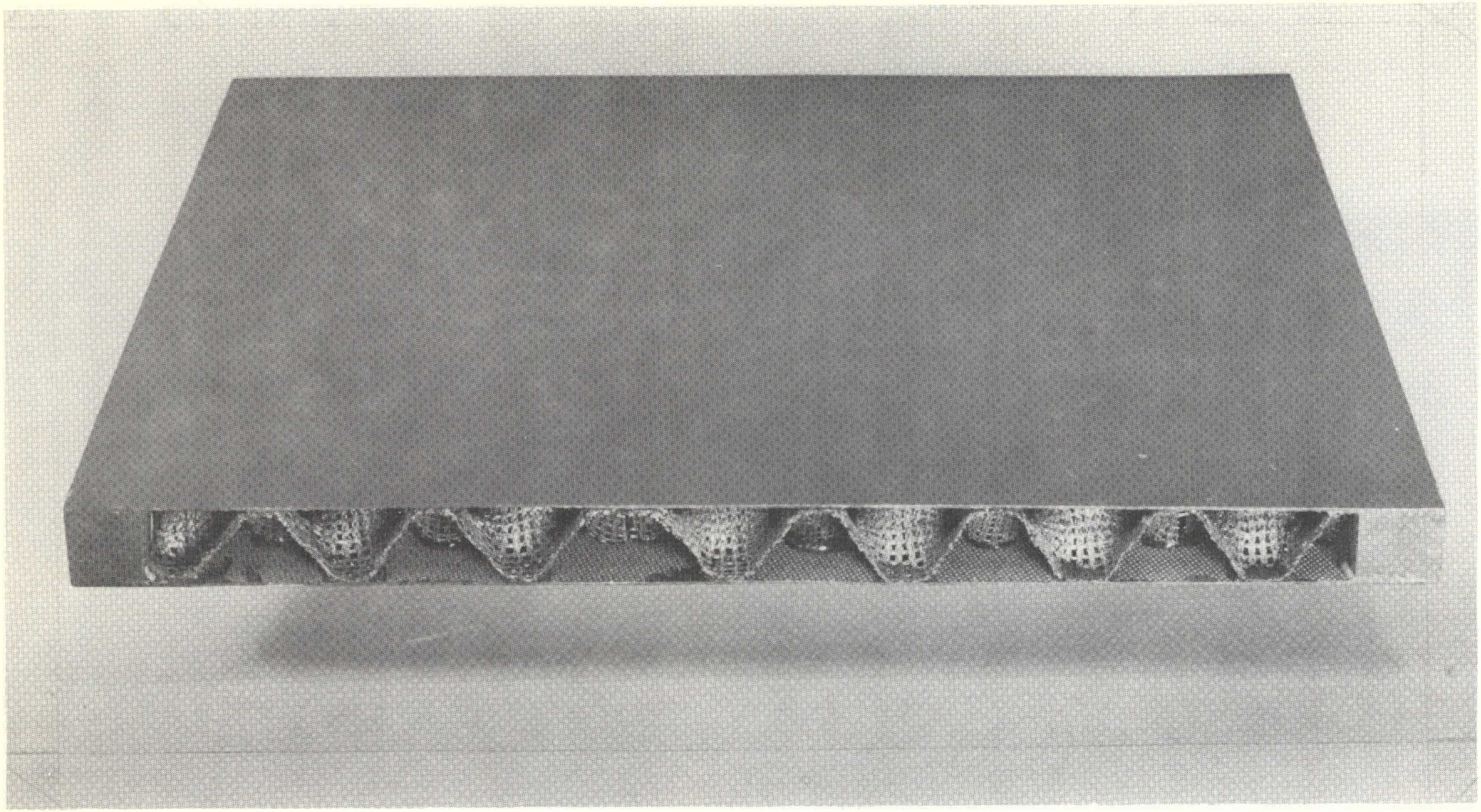


Figure 15-11. Completed Formed Core Sandwich Panel With Polyimide/Glass Skins and a Polyimide Adhesive Used to Comprise a Nonflammable Panel

another. For these tests, the panel thickness was 0.75 inches. Fabricated core density was 2.4 ± 0.2 pounds per cubic foot.

B. Facings

For shear, flatwise compression and tension, the facings were Type 6061-T6 aluminum. Thickness for the shear panels was 0.75 inches, and for the tension and compression tests 0.25 inches.

The edgewise compression specimens utilized 0.050-in.-thick glass/polyimide facings. The material of these facings was DuPont 4701 prepreg.

C. Adhesive

A paste adhesive EC 2216 (3M Company) was spread on each node. Bonding was accomplished by applying pressure of 12 lb/in.^{2} for two hours at 200° F.

D. Dimensions

Nominal specimen dimensions were as follows:

Type	Length (in.)	Width (in.)
Flatwise compression	5.00	5.00
Flatwise tensile	3.62	3.62
Core shear	11.50	4.00
Edgewise compression	3.50	7.00

Results

Table 15-1 summarizes the strength levels of glass/polyimide quadricore sandwiches measured:

Table 15-1. Mechanical Properties of Glass/PI Quadricore

Property	Specimen No. 1	2	3	4	Avg.	Aluminum
Core shear strength (lb/in.^{2})	19.8	18.3	18.5	20.7	19.3	100
Flatwise Tensile Strength (lb/in.^{2})	71.8	41.8	28.9	28.0	42.6	---
Flatwise compressive strength (lb/in.^{2})	21.2	26.4	28.2	24.0	24.9	170
Edgewise compression skin stress at failure (lb/in.^{2})	6104	4476	5058	4622	5065[1]	---

[1]Failure mode was buckling without skin fracture. Values, therefore, are conservative and a reflection of the low rigidity of the quadricore.

As shown in Table 15-1 for the type of glass/PI quadricore studied in this program, the average values for core shear, flatwise tension, flatwise compression, and edgewise compression were respectively 19.3, 42.6, 24.9, and 5065 lb/in.^{2}. These values are substantially lower than the level of strength obtainable with aluminum honeycomb of similar density.

Discussion of Results

Although the level of strength achieved with the glass/PI quadricore was discouraging, the data must be viewed in light of the state of development of this material and the types of failure observed. In all instances, buckling or yield type of failure occurred. Thus, substantially no fracturing of the glass reinforcement, which is intended to be the primary contributor to the strength of this material, was observed. In itself this observation establishes that the structural potential of glass/PI quadricore is much greater than has been achieved thus far.

For glass fiber to work efficiently, structurally in compression or shear, it must be well stabilized. One primary function of the resin in a glass fiber composite is to stabilize the fibers. The resin establishes the geometrical shape of the fiber mass and hardens it so that the fibers may work together rather than individually. If insufficient resin is present, the fibers will act as highly flexible members and will tend to display a "false" yielding behavior rather than fracturing when loaded. The minimum amount of resin required is a function of the form in which the fibers are used. Woven glass fiber cloth, unidirectional rovings, and twisted yarns each require different minimum resin content to achieve a similar level of stability.

A second fundamental influence upon the rigidity of a glass fiber composite is the void content of the composite. At a given level of resin content, apparent rigidity in compression and shear will decrease as void content increases. These two factors together with the physical form of the fibers and the shape of the structure member will determine the structural capability of the material. In the opinion of the author, the shape of glass/PI quadricore, although influenced by dimple dimension and area density, is such that it should function satisfactorily as a structural core in sandwich members, but only if the other requirements for a structural composite are satisfied. Thus, the deficiencies of the quadricore tested are not to be viewed as a necessary constraint upon the structural potential of this material, but rather as proof of the type of improvements necessary to develop it into a satisfactory structural material. Suggestions for approaches to the elimination of the problem are given in Conclusion and Recommendations.

Conclusion and Recommendations

Although the level of mechanical performance achieved in this study was disappointing and would not qualify glass/polyimide quadricore as a structural core material, additional development should improve structural performance greatly. There does not appear to be a theoretical basis that would prevent this achievement.

It is recommended that the primary initial effort in further development of this core material be directed toward characteristics that will improve its rigidity. Some suggestions follow:

1. A higher ratio of resin to glass. This could be achieved without substantial change in core density by reducing the coarseness of the yarn.

2. Reduce the dimple diameter, height and/or spacing.

3. Consider other than knitted fabrics which tend to have high elongation.

It is also recommended that removing (punching out) the material at the base and top of the dimples be considered. The material in these regions makes a minor contribution to the performance of quadricore as a core material, but a substantial contribution to its weight. With proper bonding, only the sidewalls of the dimples are required for strength and stiffness in the region between the skins. This removal of material would substantially reduce the weight of the core and allows this weight saving to be redistributed to the sidewalls (as additional resin, for example), which will assist in achieving greater sidewall rigidity.

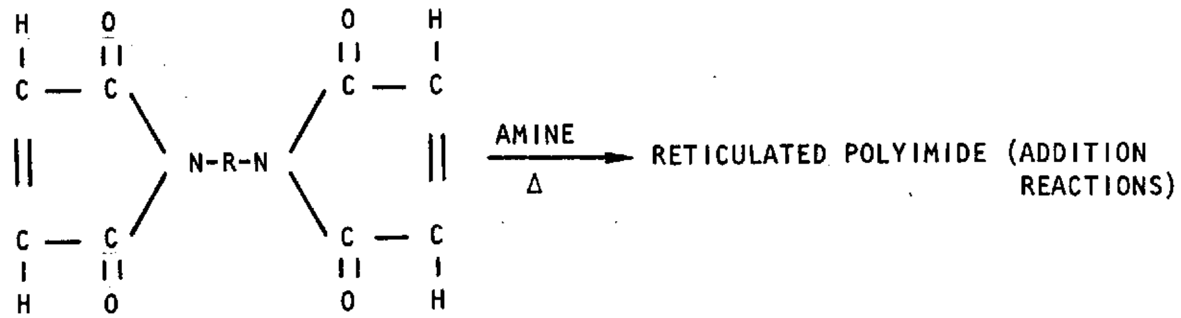
NEW DEVELOPMENT AND POTENTIAL USES

NEW DEVELOPMENT AND POTENTIAL USES

CURRENT DEVELOPMENTS IN POLYIMIDE MATERIALS

Significant changes have taken place since the development of polyimide resins in 1950's and the introduction of polyimide/glass prepregs in 1960's for aircraft/aerospace and commercial applications. The number of suppliers of polyimide resins and related products have increased, the number, type, and quality of polyimide materials have increased, and the processing characteristics of polyimides have improved significantly.

DuPont's early work was closely followed by polyimide resin developments at Monsanto and in later years by companies such as Ciba-Geigy, Rhodia Inc., Upjohn Company, TRW, and Hughes Aircraft Company. The DuPont PI 2501 and the controlled flow version of this resin, PI 4701, used in the fabrication of the various Apollo space parts and the items fabricated in Tasks 1-15 of this program are classified as condensation reaction polymers. Solvents and reaction products created by these condensation PI systems greatly complicated the fabrication of PI/glass parts. This subject has been discussed elsewhere in this report. Recently polyimides with improved processing properties have been introduced to the aircraft/aerospace industry. They differ from those described above in that they are formed from so-called bis-maleinides. These cure via an addition reaction. These polymers can be prepared in many ways, but are all exemplified by being end-capped with unsaturated linkages which, when subjected to heat, react and cross-link by addition across the double bond:



The advantages offered by bis-maleinides are ease of fabrication (low temperature - 350°F - curing), extremely low voids (no condensate needs removal) lower post-cure temperatures, and potentially lower cost. Primary disadvantages are significantly lower thermal stability than condensation polyimide (100°F lower - long term) prepregs, low drape and tack properties, and fire and smoke resistance not comparable to condensation polyimides (will burn and smoke in 100-percent-oxygen atmosphere). Nevertheless, these systems are still far superior to epoxies. Modifications of straight bis-maleinides have been investigated. A new system represented by DuPont's PI 30003 offers

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many of the advantages of bis-maleimides, but does have thermal stabilities in excess of nonmodified systems (upper use temperature 500°F). The major disadvantage is the long post-cure cycle required.

A third type of polyimide was introduced by DuPont in 1971 (NR-150). This polyimide follows essentially the condensation route, except that the final cured product is soluble in selected organic solvents (normal condensation type PI's are not, e.g., DuPont's 3001 and 3002). The advantage of such systems is that they allow for reaction with no condensate removal, only solvent. Prepregs of NR-150 are supplied as completely cured polyimides. Advantages include low voids, excellent fire and smoke resistance, and thermal stability as good as 3001 or 3002. Primary disadvantage is its severe fabrication conditions (200 $lb/in.^2$ at 600°F or higher).

Fabrication techniques generally follow standard vacuum-bag procedures. More recently, vacuum bag/autoclave techniques have been developed whereby parts with significantly lower void contents have been realized. Dielectric monitoring devices are a significant aid in preparing parts with this latter technique. Use of such instruments allows for proper selection of when to apply autoclave pressure. Yields have significantly improved when dielectric monitoring is employed. Proper layup and bleeder assemblies are also extremely important in polyimide parts manufacture. Again, through a learning curve requiring a number of years, optimum layup procedures have been developed.

This section is not intended to discuss all of the new polyimide systems that have become available, only to bring attention to the dynamic advancements evident in the development of new PI resin systems. A partial listing of those PI products which are available is presented in Table 4. It has been projected that with the combination of high-temperature strength, low flammability, ease of processing, and low moisture absorption (Figures 5 through 8) that polyimide resins will supplant epoxies as the standard resin in the aerospace industry in the next five to ten years.

Table 4. Available Polyimide Products

Company	Type	Temp Range (°F)	Prepregs	Lamin Resins	Foams	Adh	Honey-comb Core	Films	Molding Compounds
Monsanto	Condensate cond -add.	450-600	No	6	1	UD			2
DuPont	Condensate addition	450-600	Yes	4		1		2	
Ciba-Geigy	Addition	500-600	No						
Rhodia	Addition	480-550	No	2		4		2	6
Upjohn	Addition	550	No	1		1		1	1
Hexcel	Condensate addition	450-600	Yes	1	2	3	1		
American Cyanimid	Condensate	450-600	No			1			

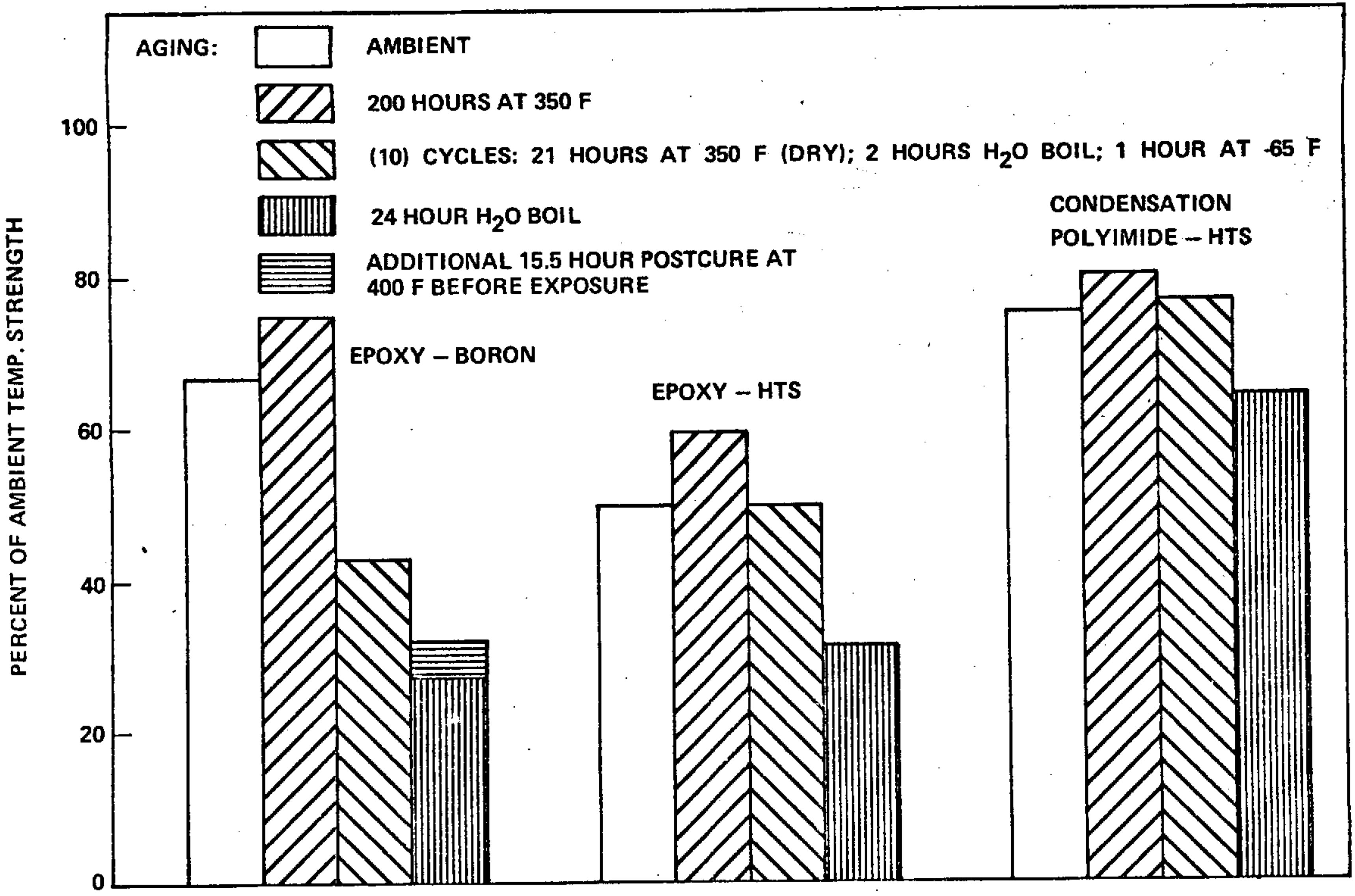

Figure 5. Effect of Various Aging Exposures Upon Flexural Strength of Unidirectional Composite Laminates Tested at 350 F After 30-Minute Preheating (Bare, Unsealed Specimens)

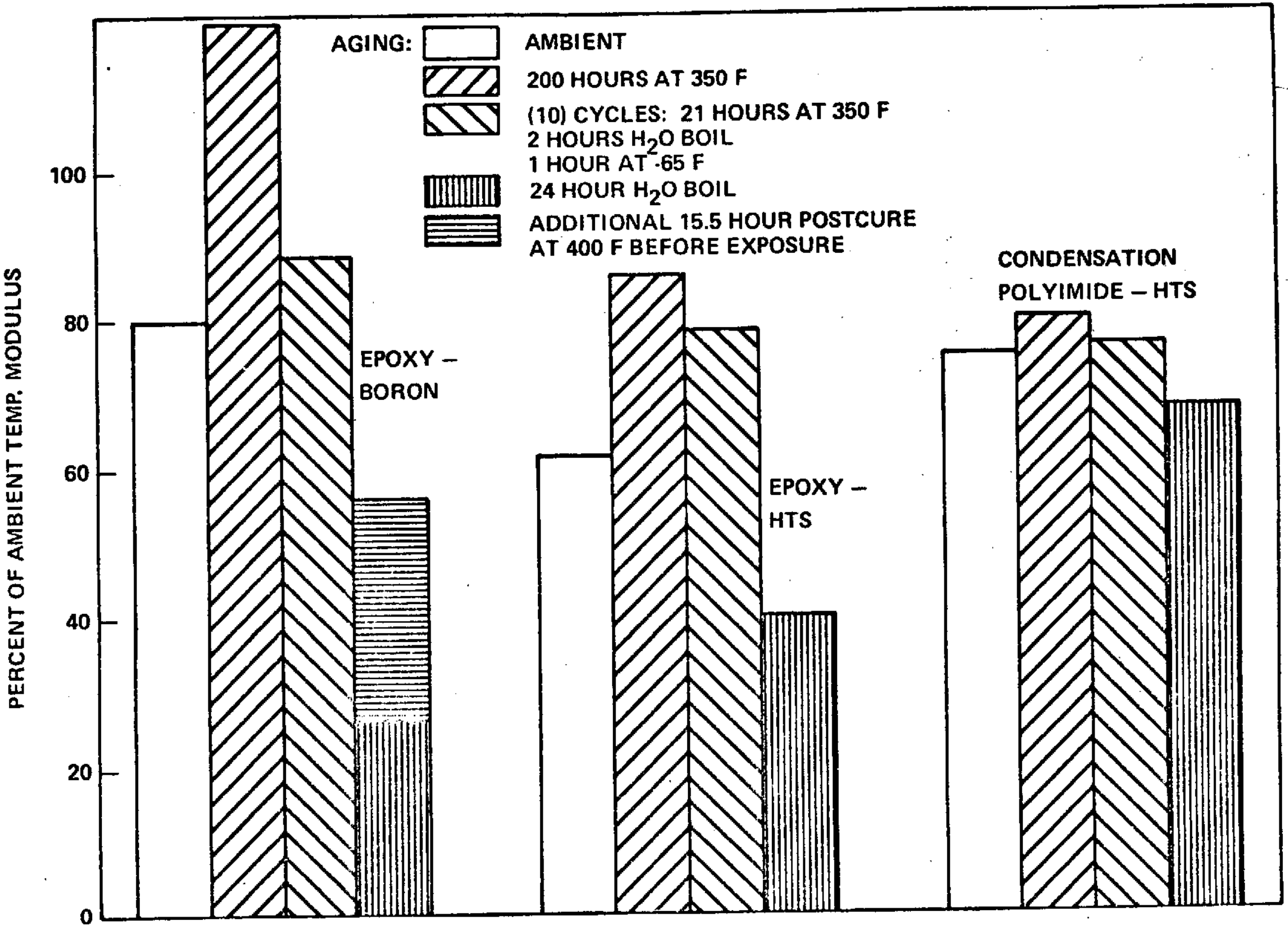

Figure 6. Effect of Various Aging Exposures Upon Flexural Modulus of Unidirectional Composite Laminates Tested at 350 F After 30-Minute Preheating (Bare, Unsealed Specimens)

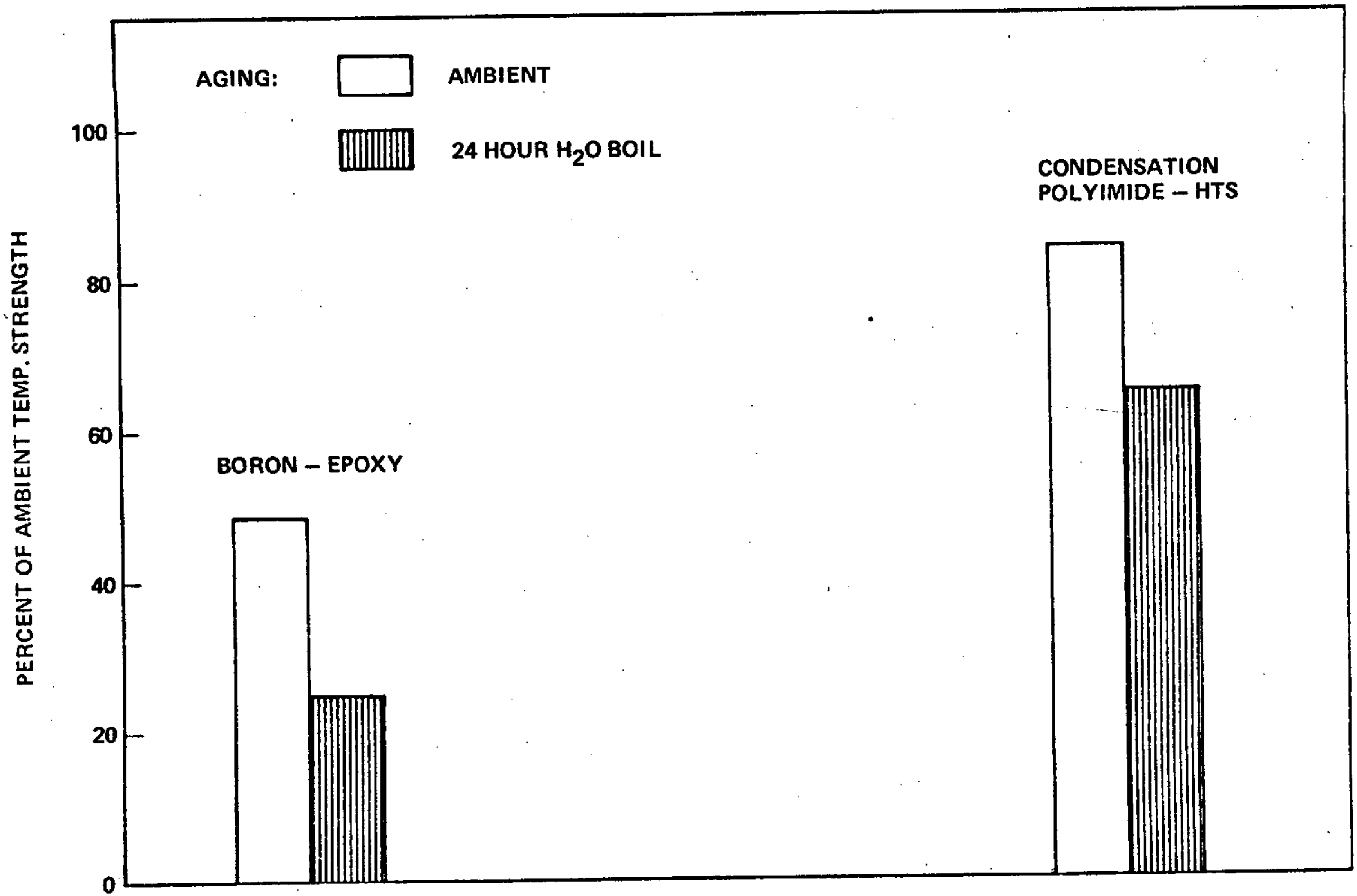

Figure 7. Effect of Temperature and Boiling Water Upon Flexural Shear Strength of Unidirectional Composite Laminates Tested at 350 F After 30-Minute Preheat (Bare, Unsealed Specimens)

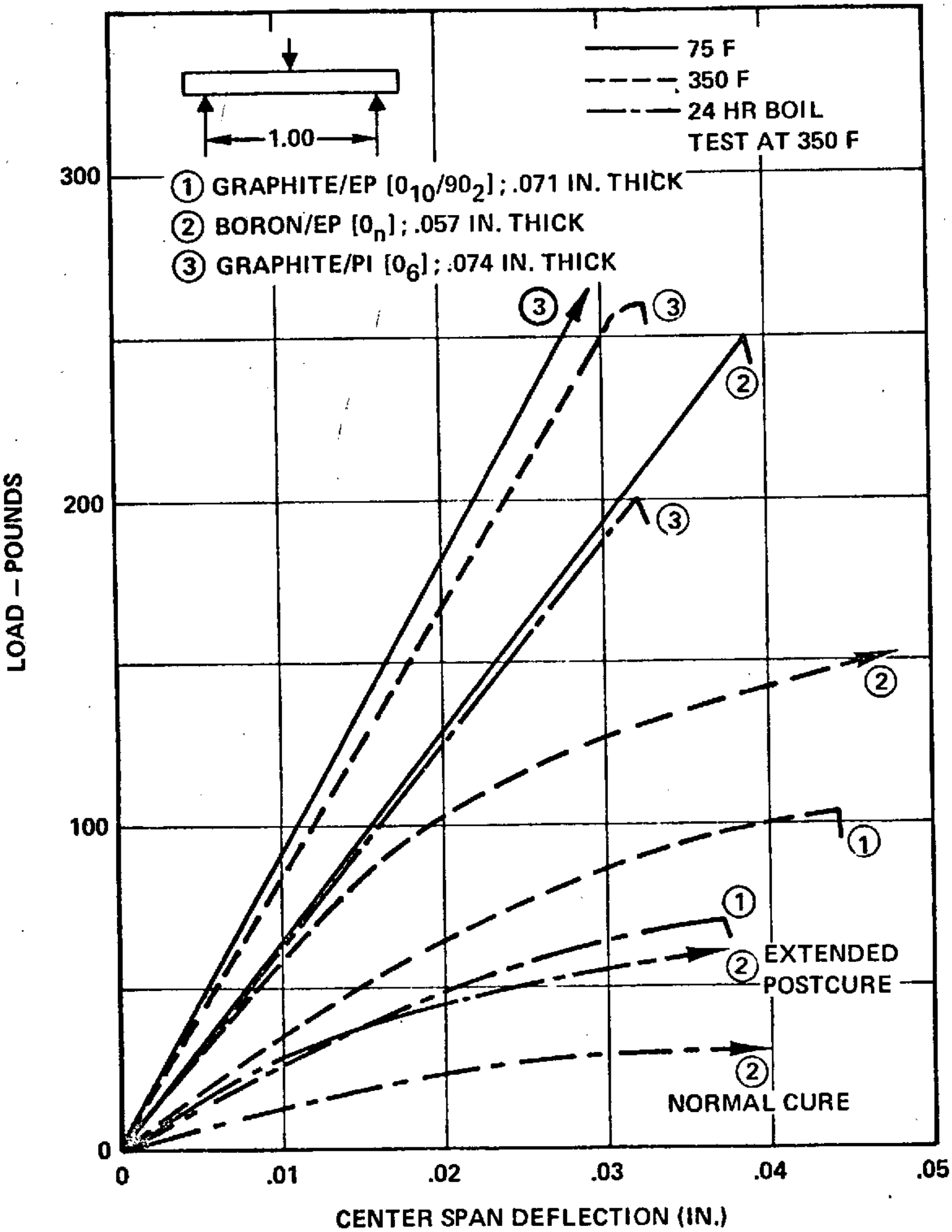

Figure 8. Typical Flexural Load-Deflection Curves for Epoxy and Polyimide Systems Reinforced with Graphite and Boron (Test After Accelerated Aging Exposures)

POTENTIAL CIVILIAN AND GOVERNMENT APPLICATIONS OF POLYIMIDES

Safety is a consideration given by every designer in the aerospace industry. Catastrophic situations cannot be tolerated in an enclosed vehicle whether it be traveling through space, under the sea, or on the surface of the earth. The Office of Safety and Health Administration has developed new and more stringent safety requirements, which must be met by careful selection of materials by designer. The courts are awarding large sums in damage suits against companies whose products have failed to operate safely in the hands of the purchaser. An Aviation Week article discusses the problems future aircraft designers and their companies can face in the courts if aircraft are not adequately designed to provide optimum survivability of passenger during air or landing mishaps. The proper application of polyimide resin base structures, while not a cureall, could contribute significantly to a lessening of safety hazards. Some of the areas that can benefit from one or all of the nonflammable, self-extinguishing, low outgassing, low-smoke-generating, high dielectric, good thermal and electrical insulating, and good elevated temperature structural properties of polyimide materials are suggested below. These PI properties can be translated into products which can:

1. Minimize the initiation of a fire.

2. Contain a fire and minimize its rate of propagation.

3. Reduce the level of toxic gases generated.

4. Generally increase the changes of passenger and crew survivability in the case of crashes.

5. Operate at higher temperatures while retaining good structural and electrical properties.

6. Reduce structural and nonstructural weight.

7. Improve transparency to radar.

8. Reduce magnetic profile.

These products can be used for:

1. Structural and nonstructural interiors for aircraft, ship, boats, submarines, homes, office and industrial buildings, test chambers, and spacecraft.

2. Electrical insulation for various products used in civilian and government areas.

3. Radomes and antenna support structure for high-power radar and communication systems use on board ships, satellites, and aircraft.

4. High-temperature-resistant printed circuit board material used in home appliances, office equipment and electronic equipment on ships, aircraft, submarines and satellites.

5. High-temperature primary and secondary structure on aircraft bodies, wings, flaps, etc.

6. Waste baskets for homes and offices.

7. Fire fighting equipment: helmets, flame and heat shields, etc.