NASA CONTRACTOR REPORT 166599

ADVANCED STRAIN-ISOLATION-PAD MATERIAL WITH BONDED FIBROUS MATERIAL

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PREFACE

This report was prepared by the Hughes Aircraft Company, El Segundo, California, under NASA Contract NAS2-11059. The program was administered by the NASA-Ames Research Center, Moffett Field, California, with Mr. P. M. Sawko serving as the NASA Technical Monitor.

Mr. R. W. Seibold served as the Hughes Aircraft Company Program Manager, assisted by Messrs. C. A. Saito and B. W. Buller who performed the laboratory experiments. The Rando air-laid matts were developed by Professor E.A. Vaughn and Dr. Christine Jarvis at Clemson University, and the electrostatic flocking experiments were performed by Mr. R. T. Dugan at Microfibres, Inc. Dr. J. V. Milewski at the Los Alamos Scientific Laboratory provided key technical assistance. Valuable technical consultation was provided by Mr. J. S. Tedesco and Dr. A. F. Fraser, Hughes Aircraft Company.

This report covers work performed during the period August 1981 through July 1982 and was submitted by the authors in September 1982.

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1.0 INTRODUCTION AND SUMMARY

The thermal protection system (TPS) used on the undersurface of the Space Shuttle Orbiter consists of several thousand ceramic tiles bonded to sections of low-modulus felt material which are in turn individually bonded to the aluminum skin of the Orbiter. The flexible felt material acts as a strain isolation pad (SIP) permitting relative motion between the aluminum substructure and the brittle tiles, which reduces the strains in the tiles (Reference 1). The present SIP material is a heavily needled felt made from Nomex aramid fibers, which are manufactured by the DuPont Company. This material offers the advantages of high strain capability, low bulk density, highly permeable construction to allow rapid venting, and performance capability at 316°C (600°F). However, the SIP material has two major disadvantages: (1) load history dependence, i.e., changes in elastic mechanical response when subjected to repeated deformation loads, and (2) a tendency to transmit stress concentrations into the ceramic tiles due to the presence of localized through-thickness fiber groups produced by the needling (Reference 2). The load history dependence of the needled SIP material is a consequence of the fact that the material is held together only by fiber entanglement and therefore tends to "unravel" with repeated tensile loads. The transmittal of stress concentrations into the tiles by local through-thickness fiber groups was first observed during preflight tests and was found to cause premature tensile failures within the tiles near the surfaces bonded to the SIP. This phenomenon necessitated strengthening ("redensifying") of the tile surfaces that were to be bonded to the SIP, prior to the first Shuttle launch. An optical micrograph of local vertical fiber groups in the SIP material is shown in Figure 1-1, taken from Reference 3.

Figure 1-1. Optical micrograph of present needled-felt SIP material, showing localized vertical fiber groups.

The objective of this program was to demonstrate the feasibility of an advanced concept for fabricating fibrous SIP material. The advanced SIP material was developed as a candidate replacement for the needled felt material presently used on the Orbiter. Performance goals for the advanced material included:

1. Tailorable strength, strain, and modulus in both the through-thickness and in-plane directions
2. Homogeneous isotropic construction which does not transmit localized stresses into the ceramic tiles
3. Consistent elastic mechanical response when subjected to repeated deformation loads
4. Performance at 316°C (600°F)
5. Highly permeable fibrous construction to allow rapid venting
6. Thermal properties similar to the present Nomex fiber needled-felt SIP material.
7. Compatibility with present processes for bonding to the tiles and aluminum skin with silicone elastomer adhesive.

The advanced SIP materials that were developed consisted of high-temperature aramid fibers (Nomex and Kevlar, both manufactured by the DuPont Company) deposited by controlled fluid (air or liquid) carriers, and subsequently bonded at the fiber intersections with a small amount of high-temperature polyimide resin, to form low-density bonded felts. This type of bonded fibrous construction has the potential ability to provide all of the advantages of the present SIP while eliminating the disadvantages discussed above. The bonding together of the deposited fibers can eliminate any tendency for the felt to unravel, and the homogeneous construction effectively eliminates the presence of local fiber groups such as those produced by needling.

Two fiber deposition techniques were evaluated for fabricating the felts. The first was the use of a Rando air-laying matt machine which is a common commercial device for making nonwoven textiles. The other was the deposition of very short fibers, suspended in a liquid to form a slurry, onto a filter pad. Felts deposited by both techniques were then bonded internally at the fiber cross-overs using very dilute solutions of polyimide resin. With one important exception, both of these approaches resulted in bonded felt materials having constructions appropriate for yielding the desired properties. The exception was the fact that neither fiber deposition technique appeared to produce felts having a sufficient number of fibers oriented in the through-thickness direction (or at acute angles to this direction) to provide the needed through-thickness tensile strength. Rather, most of the fibers tended to be oriented in or close to the plane of the felt. Several material variations and modifications to the Rando machine and to the liquid slurry deposition apparatus were attempted, and some increases in "out-of-plane" fiber orientations were achieved. However, none of the material, equipment, or process modifications produced felts with an adequate amount of through-thickness strength to meet requirements for the SIP material.

Late in the program, two approaches were explored that promised to provide the needed high content of out-of-plane fibers. One of these approaches was the incorporation of short fibers into expanded Rando matt (prior to impregnation and compaction to the final thickness) by use of electrostatic flocking. The other approach was to use a recently developed Rando matt compaction machine that can rearrange the fibers in the matt so that they become randomly oriented in three dimensions. The first of these machines is scheduled to become operational in late 1982.

The feasibility of using advanced air-lay and liquid-lay felt deposition techniques to fabricate homogenous SIP materials was demonstrated. However, further work is needed to achieve adequate through-thickness tensile strength in the bonded felts. The advanced fabrication approaches developed under this program can provide a range of high-temperature nonwoven bonded textile materials having unique constructions and properties that might be appropriate for future needs.

2.0 TECHNICAL DISCUSSION

2.1 SIP REQUIREMENTS AND GOALS

A list of desirable SIP properties was compiled from NASA sources and is shown in Table 2-1. Detailed requirements for the present SIP material are specified in Rockwell International specifications MB0135-051, Revision L, "Nonwoven Nomex Felt," 7 August 1981, and MA0106-319, Revision AR, "RTV Silicone Adhesive-Bonded Silica Reusable Surface Insulation (RSI) System," 29 July 1981. Specific requirements for weight, thickness and bulk density of the three current classes of needled Nomex SIP material, taken from specification MB0135-051, are shown in Table 2-2. Most of the SIP material used on the Orbiter is the Class 3 type; the experiments were therefore focused on developing bonded felts with Class 3 properties.

A primary goal was to match the strengths and moduli of the <u>fluid-laid, bonded</u> felts being developed with those of the present <u>needled, unbonded</u> SIP materials. The presence of a small amount of bonding resin in a felt will be shown to significantly increase the felt's stiffness (Section 2.2). In order to match the moduli of the bonded felts being developed with those of the present unbonded felts being simulated, an assumption was made that the bonded materials needed lower fiber contents than the present unbonded materials. As a first approximation, a goal was established to fabricate bonded felts having bulk densities approximately 20 percent lower than those of the needled, unbonded materials. This translates to the property goals summarized in Table 2-3.

TABLE 2-1. DESIRABLE SIP PROPERTIES

BASIC REQUIREMENTS

High-Strength SIP: F_{TU} = 0.48 MPa (70 psi), E = 0.48 MPa (70 psi), G $\leq$ 0.24 MPa (35 psi)

Low-Strength SIP: F_{TU} = 0.28 MPa (40 psi), E = 0.17 MPa (25 psi), G $\leq$ 0.069 MPa (10 psi)

Fully reversed fatigue lifetime (R = 1): 10,000 cycles at 60-80% F_{TU}

$F_{SU} \geq$ 0.14 MPa (20 psi)

Less than 20% reduction in strength and stiffness at 316°C (600°F)

Thermal conductivity at room temperature, $\leq$0.03 W/m·K (0.02 BTU-ft/hr-ft^2-°F)

Specific heat at room temperature, $\geq$1.0 x 10^3 J/kg·K (0.25 BTU/lb-°F)

Specific heat at 316°C (600°F), ~2.1-2.5 x 10^3 J/kg·K (~0.5 - 0.6 BTU/lb-°F)

Bulk density, ~0.08 gm/cm^3 (~5 lb/ft^3)

Thickness, 0.23 cm and 0.41 cm (0.09 inch and 0.16 inch)

Maximum strain at 0.034 MPa (5 psi) = 0.2

Must be either solid or highly permeable to allow rapid venting

Capable of being made water repellent

Compatible with present processes for bonding to the RSI and aluminum skin with RTV-560 silicone rubber adhesive

ADVANCED REQUIREMENTS

Linear stress-strain response

Minimum creep

Isotropic

Homogeneous

Not load history dependent

Low volatile condensible material, <0.1%

Low electrical conductivity

DEFINITIONS

F_{TU}, ultimate tensile strength, through-thickness direction

E, tensile modulus

G, shear modulus

F_{SU}, ultimate shear strength

TABLE 2-2. PHYSICAL PROPERTIES OF PRESENT SIP MATERIALS

Property	Class 1	Class 2	Class 3
Weight*			
$gm/m^2 \times 10^2$	2.7 ± 0.34	3.7 ± 0.34	3.4 ± 0.34
oz/yd^2	8 ± 1	11 ± 1	10 ± 1
Thickness*Δ			
cm	0.229 $^{+0.023}_{-0.036}$	0.292 $^{+0.030}_{-0.038}$	0.406 ± 0.041
in	0.090 $^{+0.009}_{-0.014}$	0.115 $^{+0.012}_{-0.015}$	0.160 ± 0.016
Bulk Density**			
gm/cm^3	0.12	0.13	0.083
lb/ft^3	7.4	8.0	5.2

*Source: Rockwell International specification MB0135-051, Rev. L.

ΔAll felt thickness measurements made under this program were performed using a Randall-Stickney gage in conformance with ASTM D1777, except a 284 gm (10 oz.) weight was used. This procedure was specified in specification MB0135-051.

**Calculated from the weight and thickness requirements.

TABLE 2-3. FIRST APPROXIMATION PROPERTY GOALS FOR BONDED FELTS

Property	Class 1	Class 2	Class 3
Bulk Density*			
gm/cm^3	0.094	0.10	0.067
lb/ft^3	5.9	6.4	4.2
Thickness**			
cm	0.229	0.292	0.406
in	0.090	0.115	0.160
Weight***			
$gm/m^2 \times 10^2$	2.2	3.0	2.7
oz/yd^2	6.4	8.8	8.1

*80 percent of Table 2-2 values.

**From Table 2-2.

***Calculated from the bulk density and thickness values.

2.2 BONDED STRUCTURAL FELTS

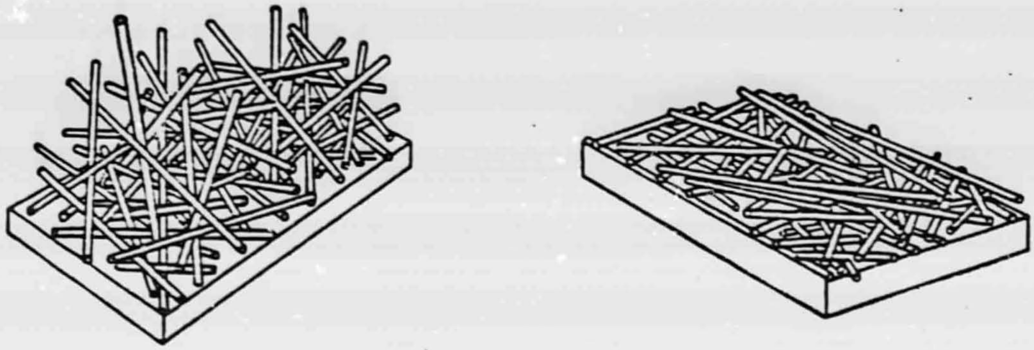
The concept of making low-density bonded structural felts is an extension of a bench top procedure recently developed by John V. Milewski at the Los Alamos Scientific Laboratory. Dr. Milewski built an apparatus in which short fibers are transported with a predominantly endwise orientation in a fluid stream directed at a flat suction filter pad, where they are deposited (Reference 4). The stacking arrangement of the deposited fibers is controlled by the nature and velocity of the transporting fluid, the diameter of the fluid stream, the fiber length and aspect ratio (L/D), the fiber population density, and the surface characteristics and amount of suction of the receiving filter. Isotropic, planar, or combined stacking arrangements can be produced (Figure 2-1). Shorter fibers tended to provide increased randomness of stacking.

An example of a polyester fiber felt made by this technique (and subsequently bonded with dilute epoxy resin) is shown in Figure 2-2, and compression properties of various versions of this felt are shown in Figure 2-3. The mechanical response of these very low density bonded felts resembles the springiness of a rubber sponge. Although the fibers are rigidly joined at their cross-overs, each fiber deforms individually when the felt is loaded, and returns to its original position relative to the overall structure when the load is removed. This characteristic was needed to minimize load-history dependence of the advanced SIP material.

A special advantage of this felting technique is that different fiber sizes and types can be mixed in the same felt, resulting in further ability to tailor the felt properties. For example, various combinations of chopped Nomex, Kevlar, and glass fibers could be felted.

2.3 FIBER SELECTIONS AND PREPARATION

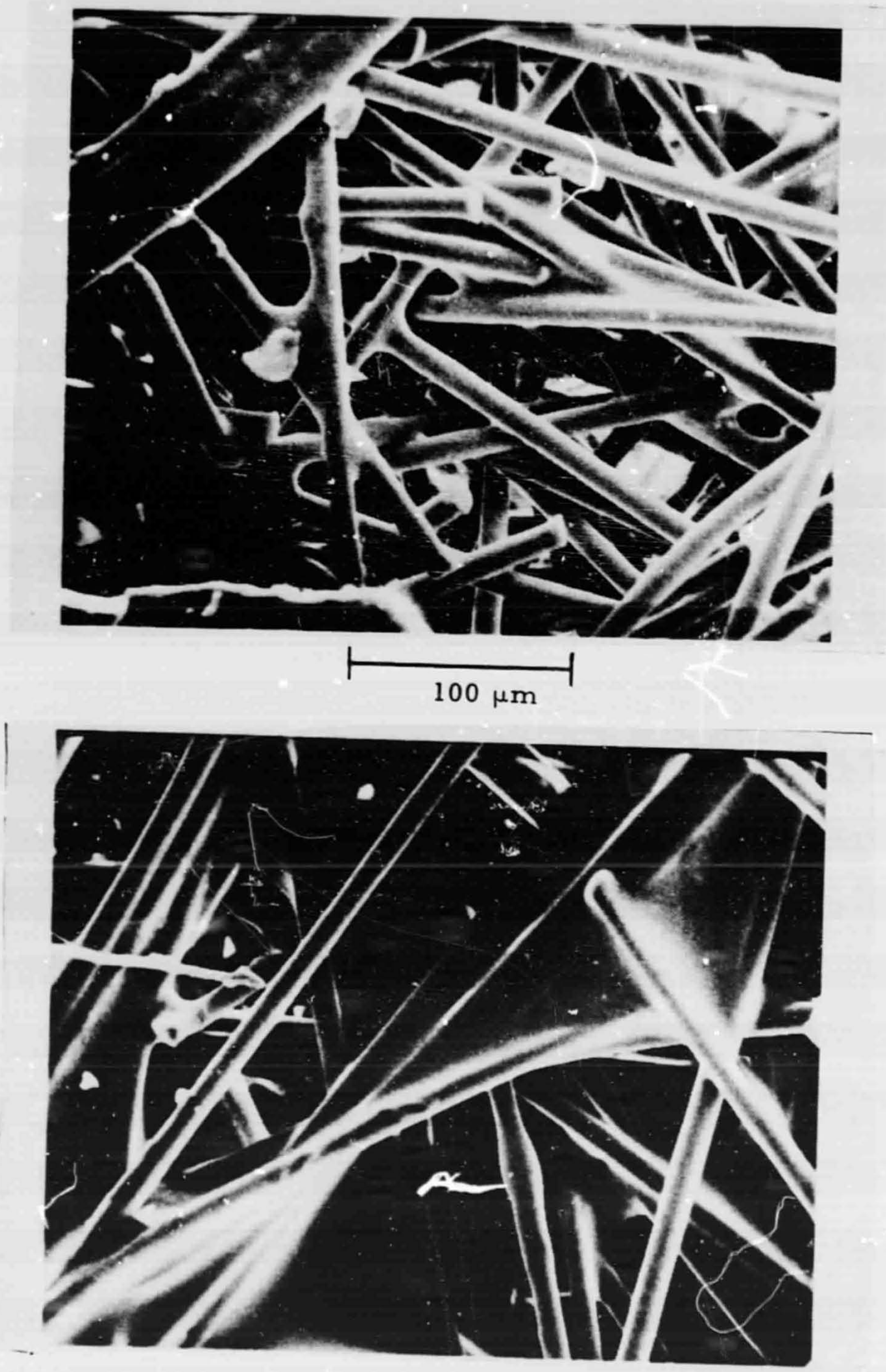
Three types of aramid fibers, Nomex, Kevlar-29, and Kevlar-49, all manufactured by the DuPont Company, were selected to fabricate the felts. Properties of yarns made from these three fibers are summarized in Table 2-4, and the specific fiber variations that were used are listed in Table 2-5. All three of these fibers provide the light weight and high elongation needed for



Near-Isotropic

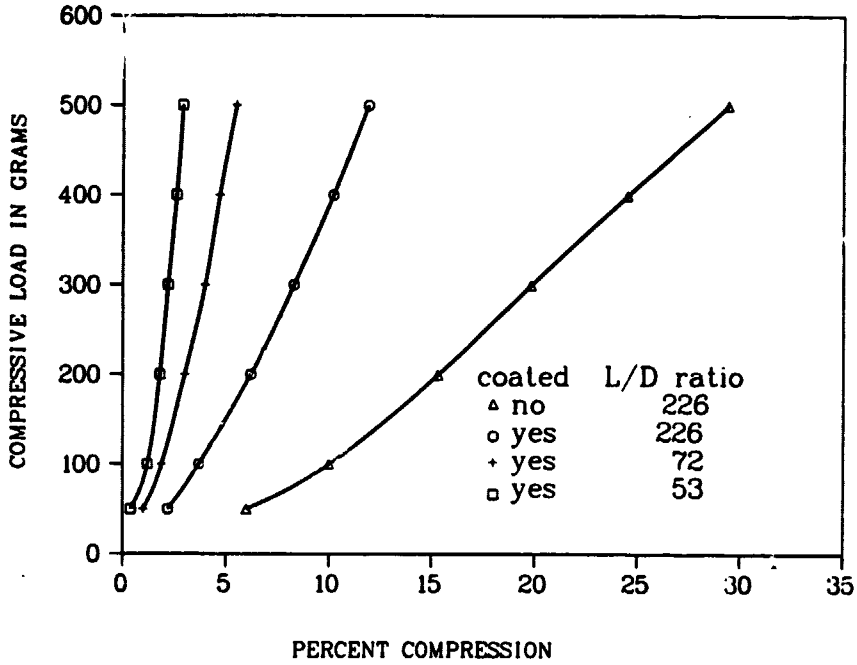
Near-Planar

Figure 2-1. Generic fiber arrangements (courtesy Fiber Materials, Inc.).



Source: Reference 4

Figure 2-2. Scanning electron micrographs of epoxy-bonded polyester fiber felt.



Source: Reference 4

Figure 2-3. Compression curves for 12-micron polyester fiber felt, coated and uncoated.

TABLE 2-4. COMPARATIVE YARN PROPERTIES

Property	Nomex[(1)(2)]	Kevlar-29[(3)]	Kevlar-49[(2)]
Tensile Strength*			
GPa	~0.38**	2.7	2.7
$lb/in^2 \times 10^3$	~55**	400	400
Modulus			
$GPa \times 10^1$	~1.2**	6.2	12
$lb/in^2 \times 10^6$	~1.8**	9.0	18
Elongation to Break, %	>3.0	4.0	2.5
Density			
g/cm^3	1.38	1.44	1.44
lb/in^3	0.050	0.052	0.052

(1) Properties of Nomex Aramid Filament Yarns, DuPont Technical Information Bulletin NX-17, December 1981.

(2) Characteristics and Uses of Kevlar-49 Aramid High Modulus Organic Fiber, DuPont Technical Information Bulletin K-5, September 1981.

(3) Characteristics and Uses of Kevlar-29 Aramid, DuPont Information Memo 375, September 1976.

*Unimpregnated twisted yarn test.

**Calculated at approximately 3 percent strain.

TABLE 2-5. FIBER SELECTIONS

FIBERS: AIR-LAY PROCESS (RANDO MACHINE)
• Nomex, 5.1 cm (2-inch) crimped staple, mixed with 0.64 and 1.3 cm (1/4 and 1/2 inch) chopped
• Kevlar-29, 5.1 cm (2-inch) crimped staple, mixed with 0.64 and 1.3 cm (1/4 and 1/2 inch) chopped
FIBERS: LIQUID-LAY PROCESS
• Nomex, chopped to lengths ranging from 0.079 to 1.3 cm (1/32 to 1/2 inch)
• Kevlar-49, chopped to lengths ranging from 0.16 to 1.3 cm (1/16 to 1/2 inch)
• Kevlar Pulp - serves as an entanglement aid

the SIP material. Table 2-5 shows that longer fibers were used for the air-laid felts than for the liquid-laid materials. The longer fibers were needed for proper flow through the Rando air-laying machine.

Newly developed techniques for chopping the very short fibers needed for the liquid-lay processing and as additives in the air-lay Rando felts were employed. For initial experiments, high-quality chopped Kevlar-49 fibers were prepared by Finn & Fram, Inc., Arleta, California. However, this company was not able to provide the quantities of fibers needed for this program. Because good-quality, straight, short fibers with sharp, non-tufted ends were needed to achieve non-planar deposition of the fibers in the felts, an effort was made to locate alternate sources for suitable fibers. DuPont sells chopped aramid fibers only in the following lengths:

Nomex flock (slightly crimped) — 0.64 cm (0.25 inch)
Nomex staple — 3.8 and 5.1 cm (1.5 and 2 inch) (and longer)
Kevlar-29 — 0.64, 1.3, 2.5, 3.8, 5.1, 6.4, 7.6, and 10 cm (0.25, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, and 4.0 inch)
Kevlar-49 — 0.64 and 1.3 cm (0.25 and 0.5 inch)

Lengths shorter than the above were needed for this program.

McCann Manufacturing, located in Oneco, Connecticut, routinely chops Kevlar-49 fibers into lengths as small as 0.16 cm (0.063 inch), but had never chopped Nomex fibers. Two kg (5 pounds) each of the following lengths of Kevlar-49 fibers were ordered from this company: 0.16, 0.32, 0.64, and 1.3 cm (0.063, 0.13, 0.25, and 0.50 inch). Spools of 1300 Decitex (1200 denier) 600 filament continuous filament T-430 Nomex yarn were provided to them and successfully chopped into 0.079 and 0.16 cm (0.031 and 0.063 inch) lengths.

The Mini-Fibers Company, located in Weber City, Virginia, routinely manufactures and stocks chopped Nomex fibers, but no shorter than 0.343 cm (0.135 inch). Several pounds each of the following lengths of Nomex fibers were ordered from Mini-Fibers: 0.343, 0.64 and 1.3 cm (0.135, 0.25, and 0.50 inch). Mini-Fibers also provided 0.64 cm Kevlar-29 fibers.

The Badische Corporation, Williamsburg, Virginia, is also a source for short chopped fibers.

Fibrillated Kevlar pulp was obtained from DuPont and used as an entanglement aid for the liquid-laid felts.

Scanning electron micrographs of Kevlar pulp and selected examples of the chopped Kevlar and Nomex fibers that were obtained are shown in Figures 2-4 through 2-12. As shown in these figures, the cut ends varied in appearance from smooth to very frayed, depending on the fiber type and chopping technique. Straight fibers with sharp, non-tufted ends were selected for the fabrication experiments that were performed. Fibers that were irregular and bent, or had tufted ends, appeared to "fall over flat" rather than pierce the previously deposited felt layers when slurries of the fibers were deposited.

2.4 FELT BONDING PROCEDURES

2.4.1 Resin Selections

Two families of high-temperature resins, viz., thermosetting silicones and polyimides, were considered for infiltration of the felts. Representative candidate silicone resins included General Electric SR-98 varnish (rigid), SR-323 laminating resin (rigid), and SR-224 varnish (flexible). These silicone resins are supplied in toluene solvent, which should not affect the Nomex or Kevlar fibers. They can be cured at 260°C (500°F) and will function at 316°C (600°F). However, a high-temperature polyimide was selected rather than a silicone because: (1) silicones are noted for poor bonding, and (2) polyimides with temperature capabilities higher than those of the above silicones are available. Two key criteria for selection of a polyimide binder were: (1) cure temperature low enough to not degrade the Nomex or Kevlar fibers, and (2) solubility in a solvent that would not attack the fibers. Nomex fibers tend to swell when exposed to N-methyl pyrrolidinone, a common solvent for polyimides.

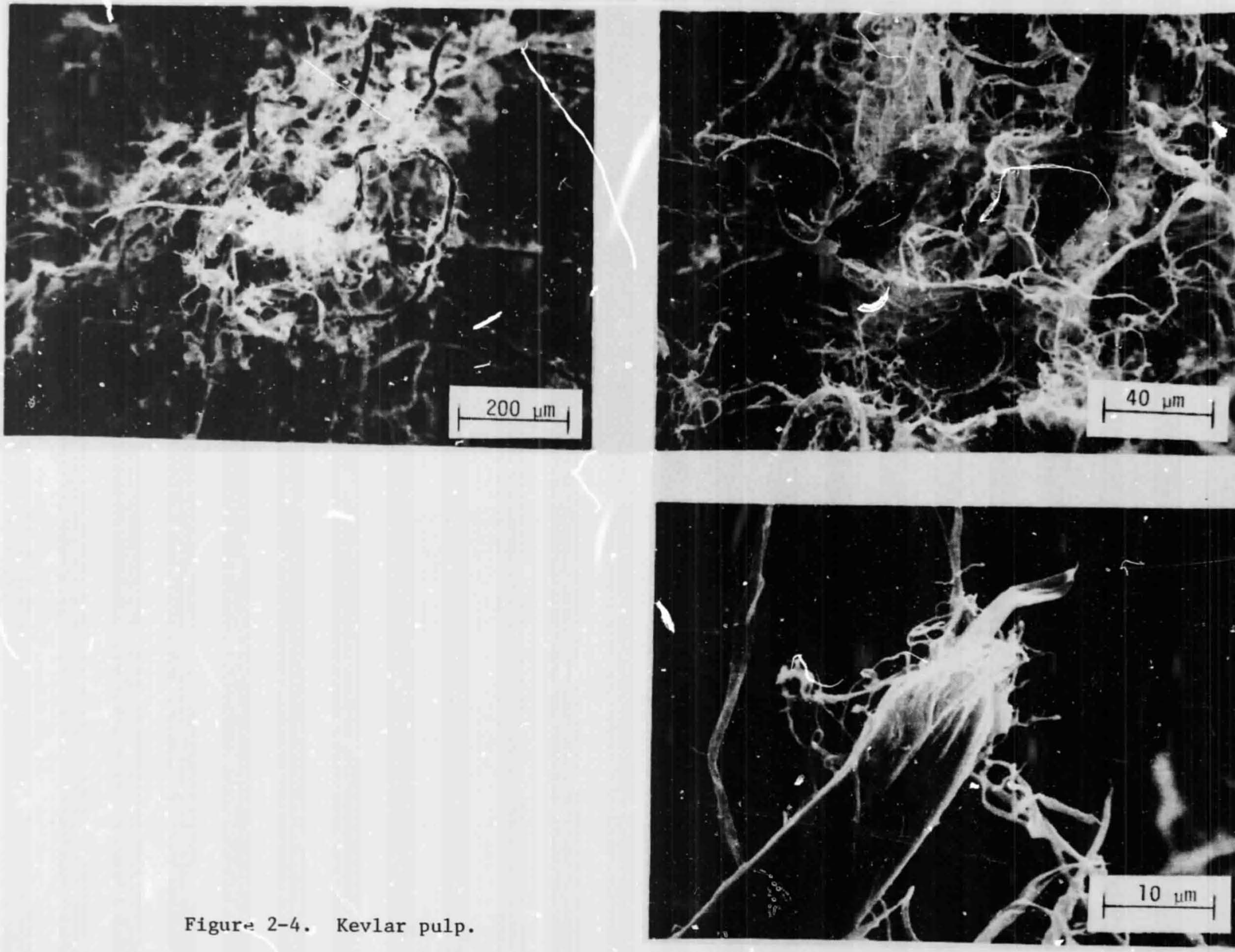
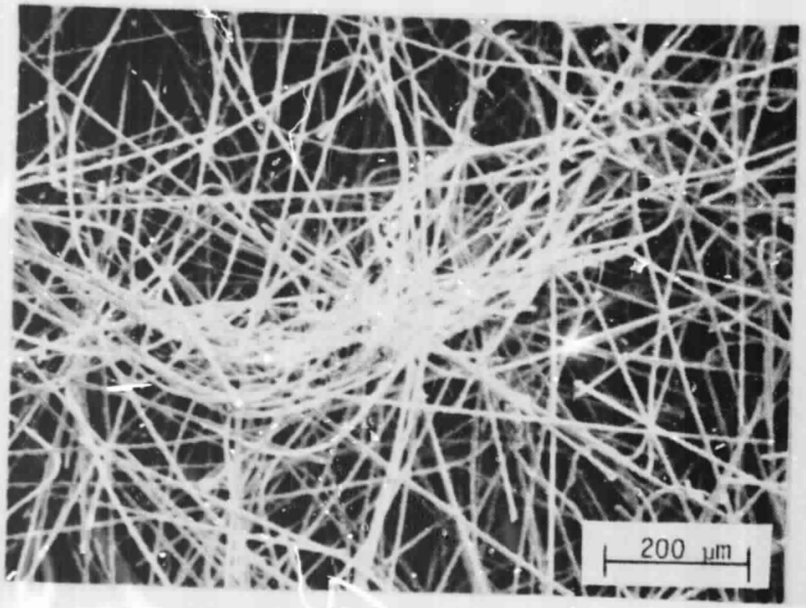
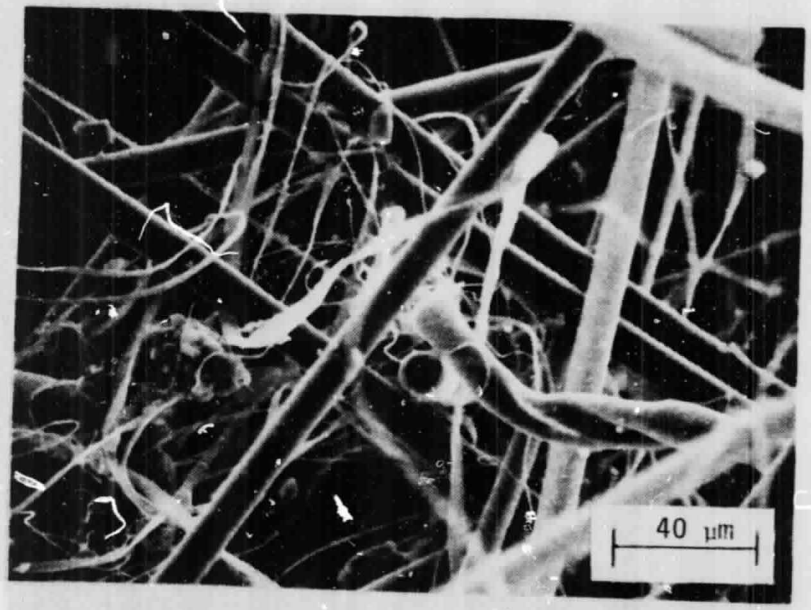


Figure 2-4. Kevlar pulp.





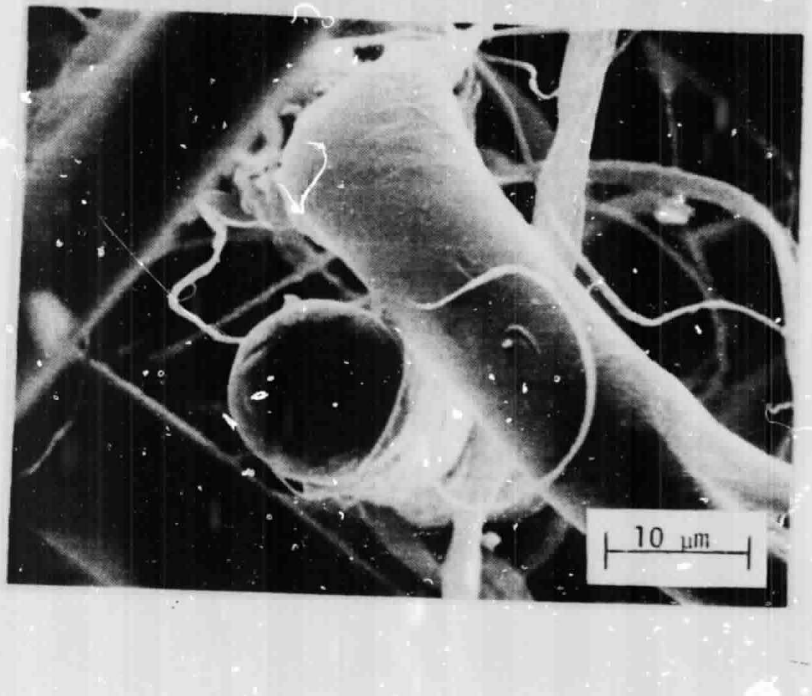


Figure 2-5. Kevlar-49 yarn chopped to 0.16 cm (1/16-in) lengths by Finn & Fram, Inc.

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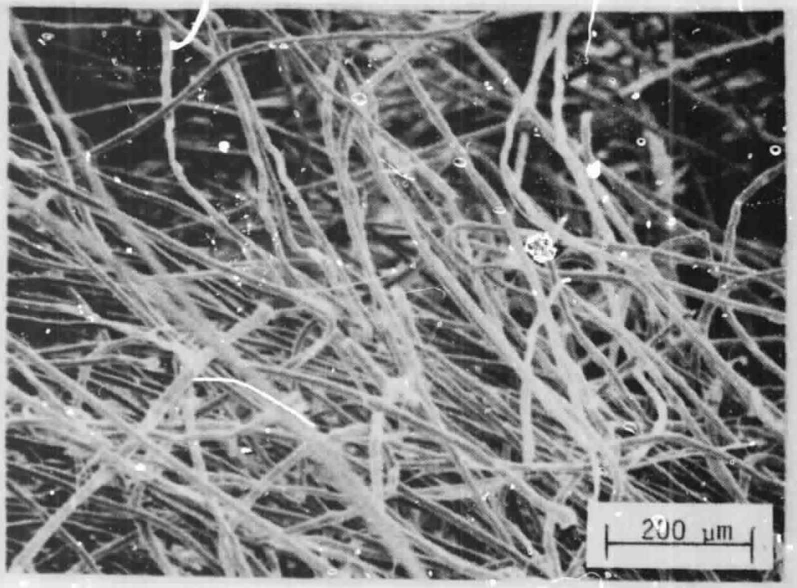
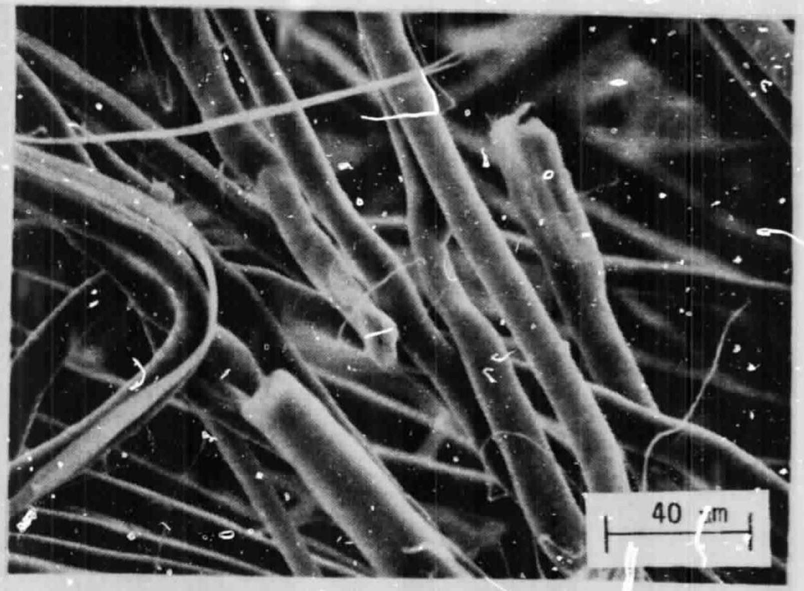




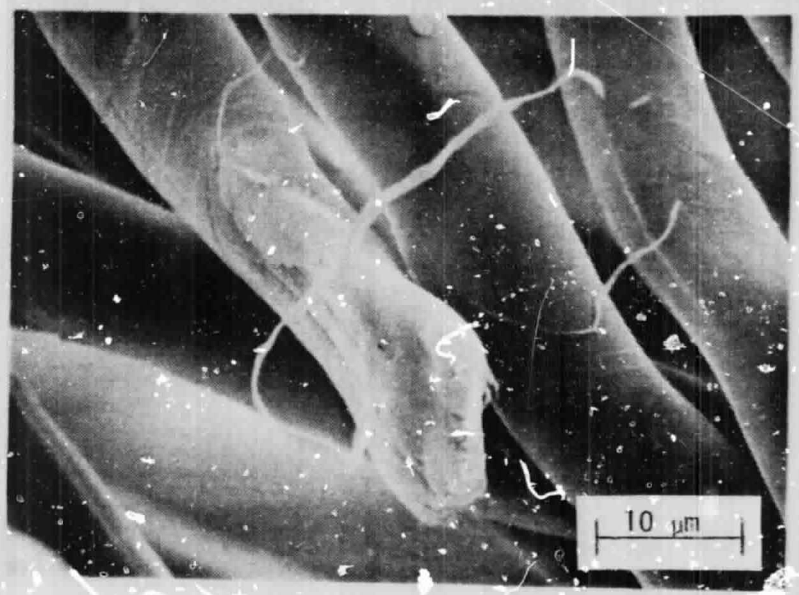
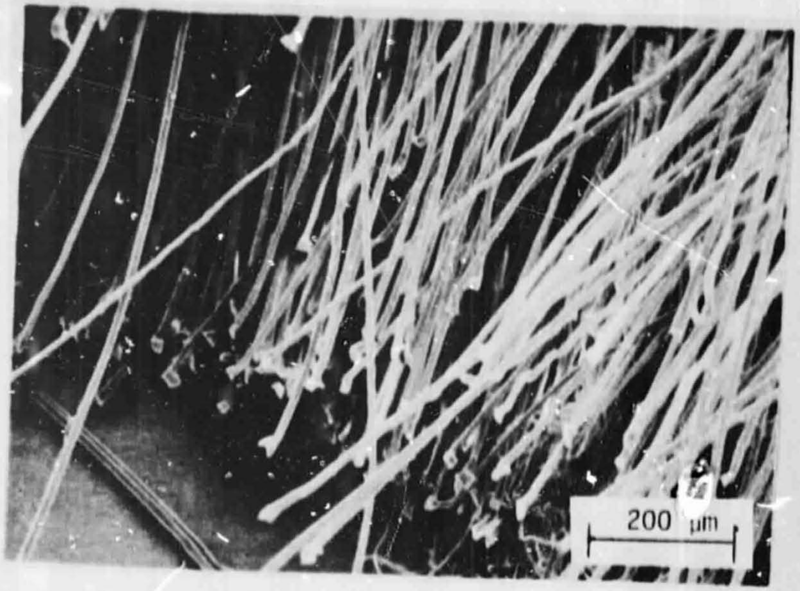


Figure 2-6. Kevlar-49 yarn chopped to 0.32 cm (1/8-in) lengths by Finn & Fram, Inc.



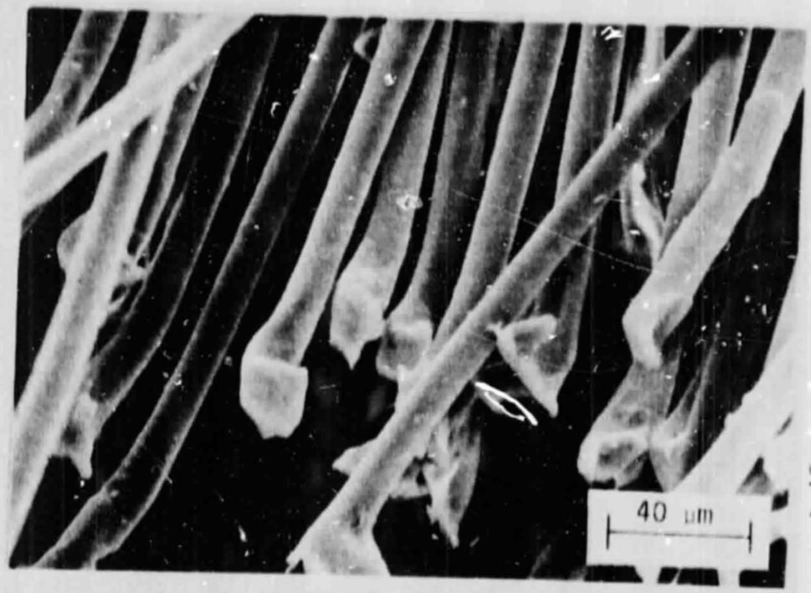
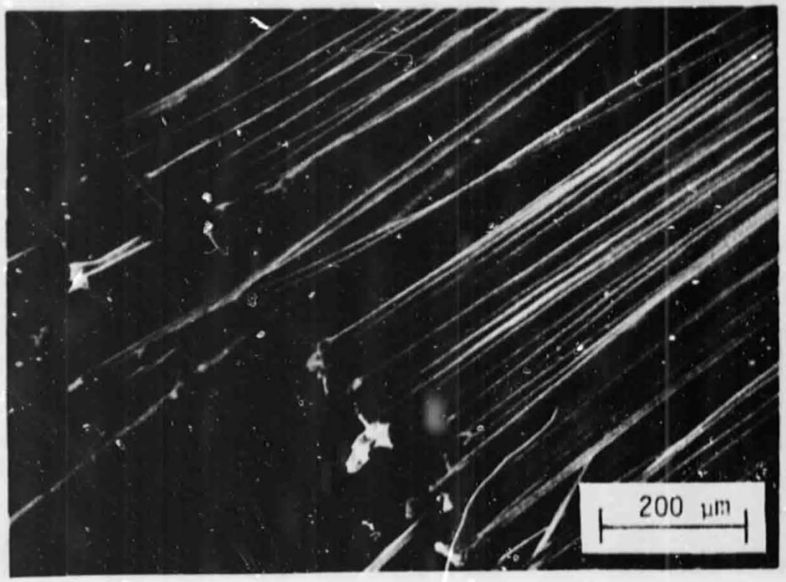
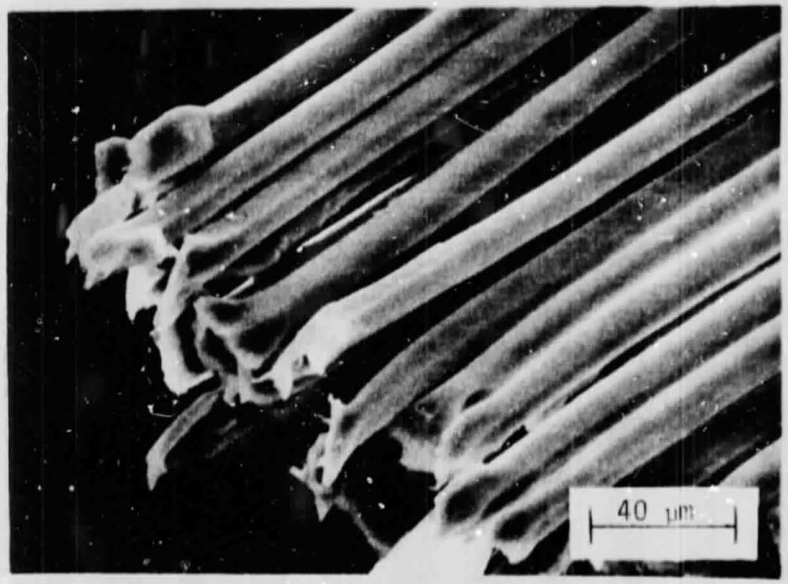


Figure 2-7. Kevlar-29 yarn chopped to 0.64 cm (1/4-in) lengths, procured from DuPont.





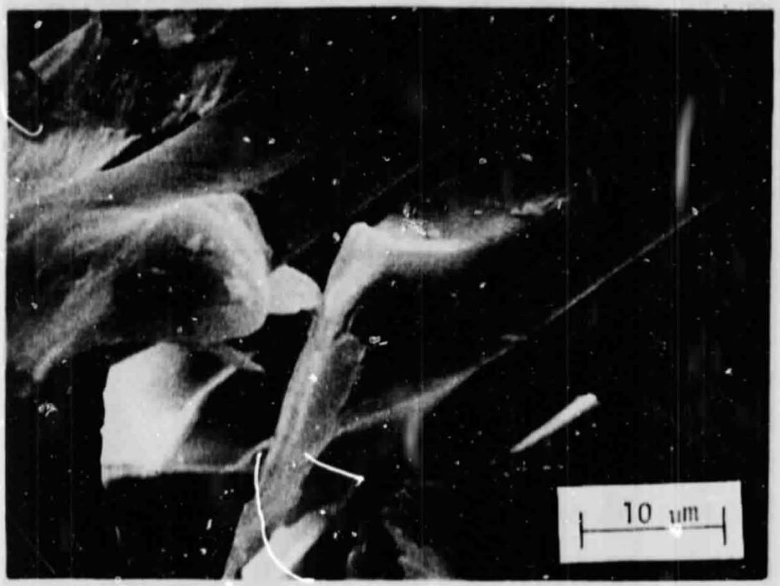


Figure 2-8. Kevlar-29 yarn chopped to 1.27 cm (1/2-in) lengths, procured from DuPont.

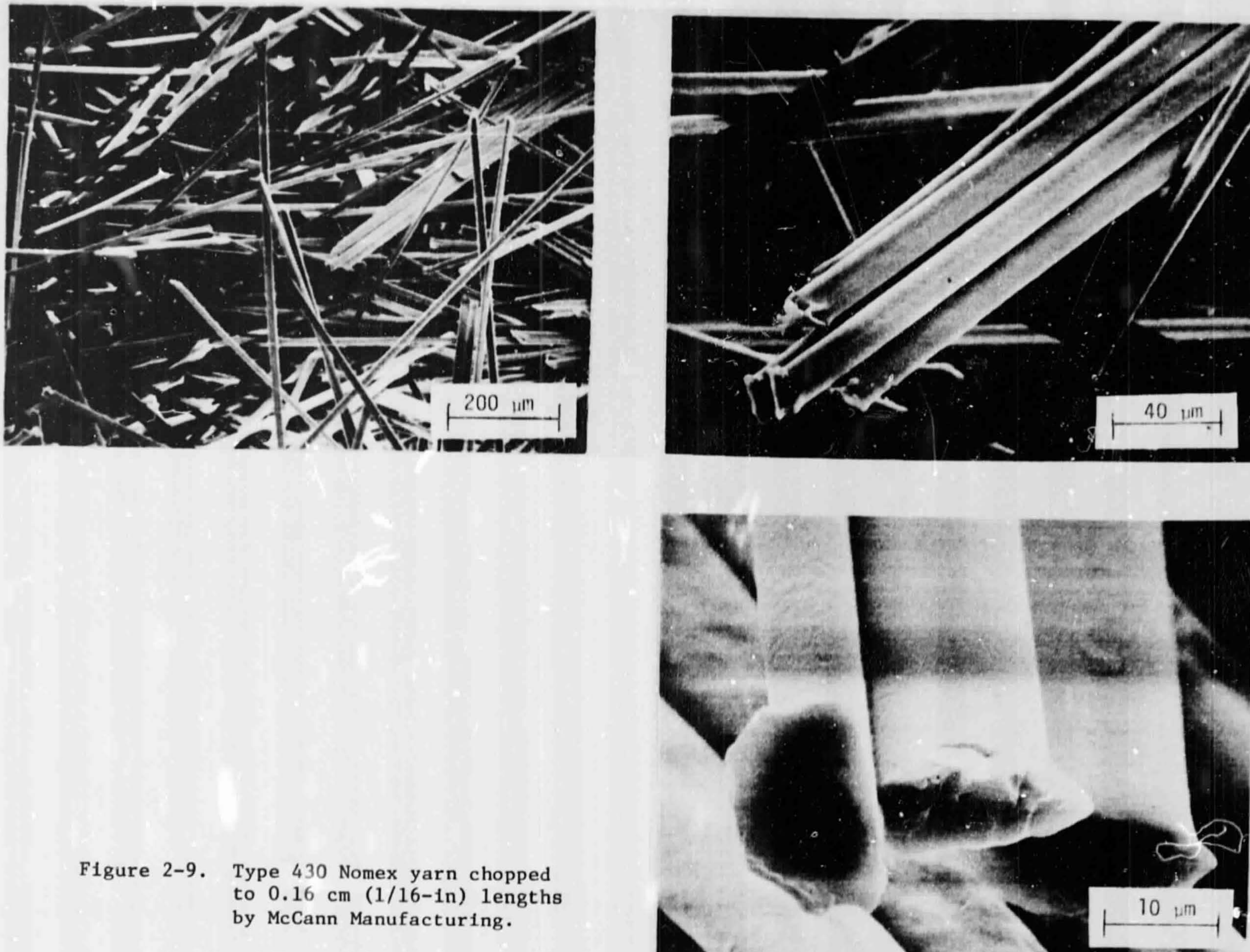


Figure 2-9. Type 430 Nomex yarn chopped to 0.16 cm (1/16-in) lengths by McCann Manufacturing.

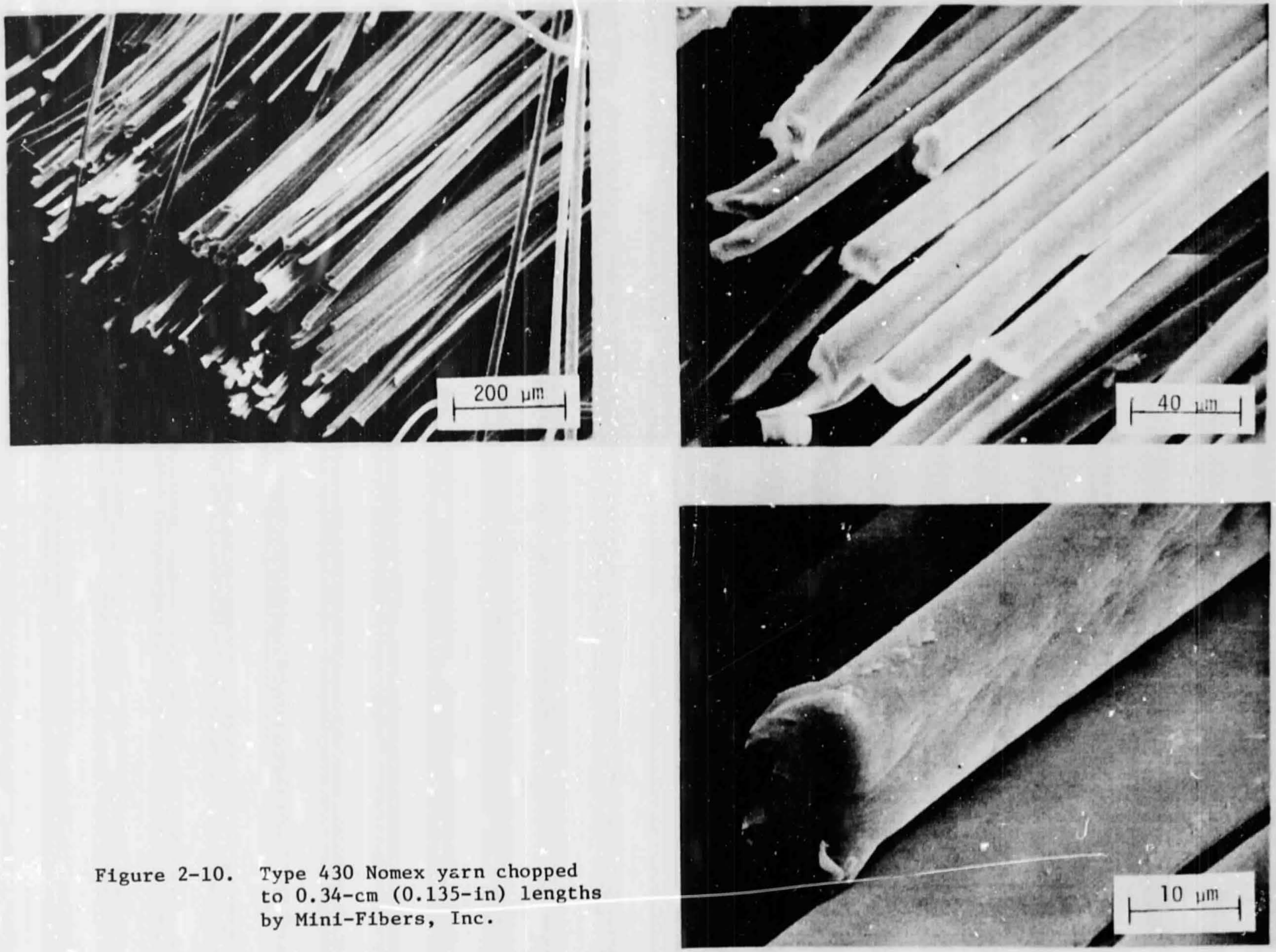


Figure 2-10. Type 430 Nomex yarn chopped to 0.34-cm (0.135-in) lengths by Mini-Fibers, Inc.

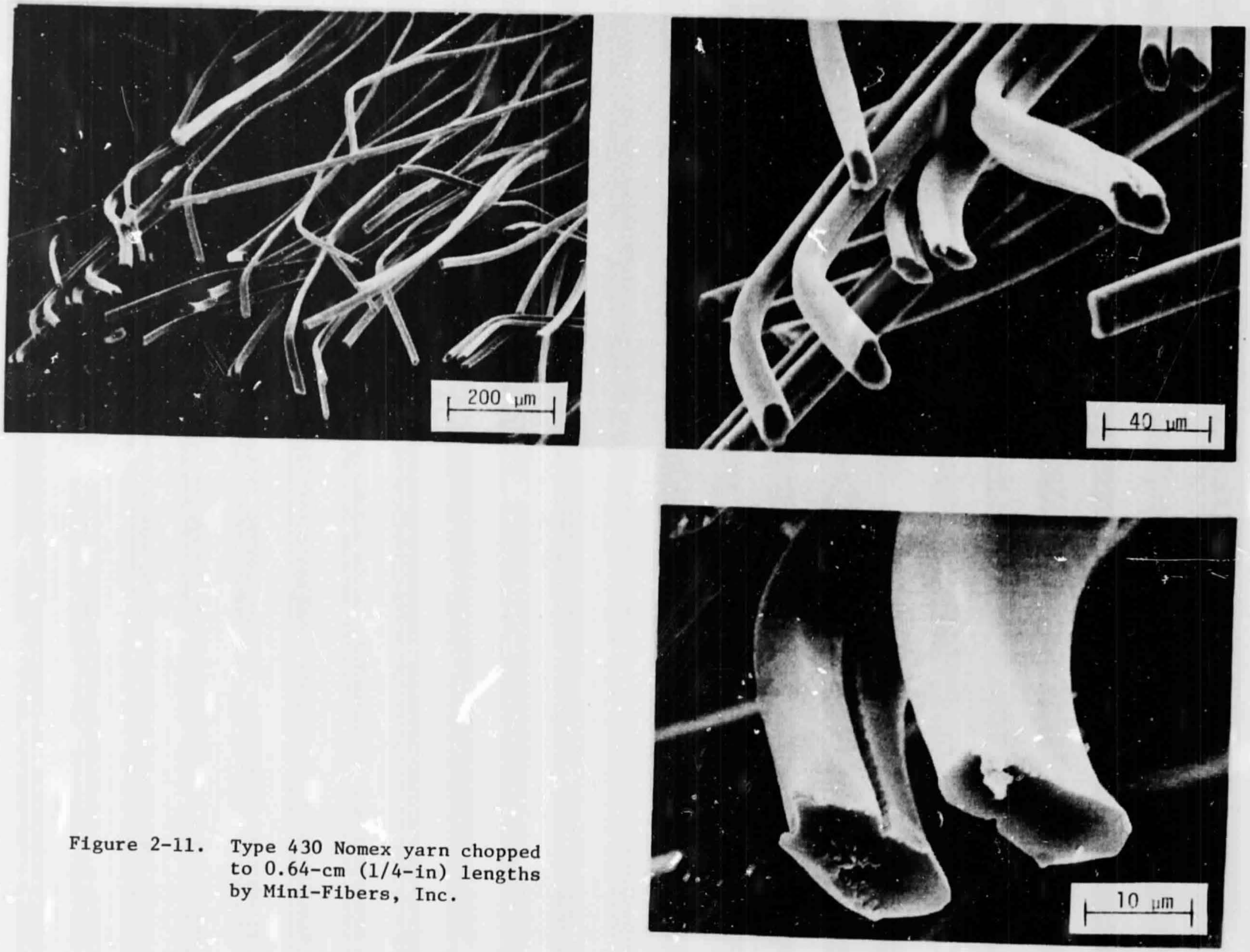


Figure 2-11. Type 430 Nomex yarn chopped to 0.64-cm (1/4-in) lengths by Mini-Fibers, Inc.

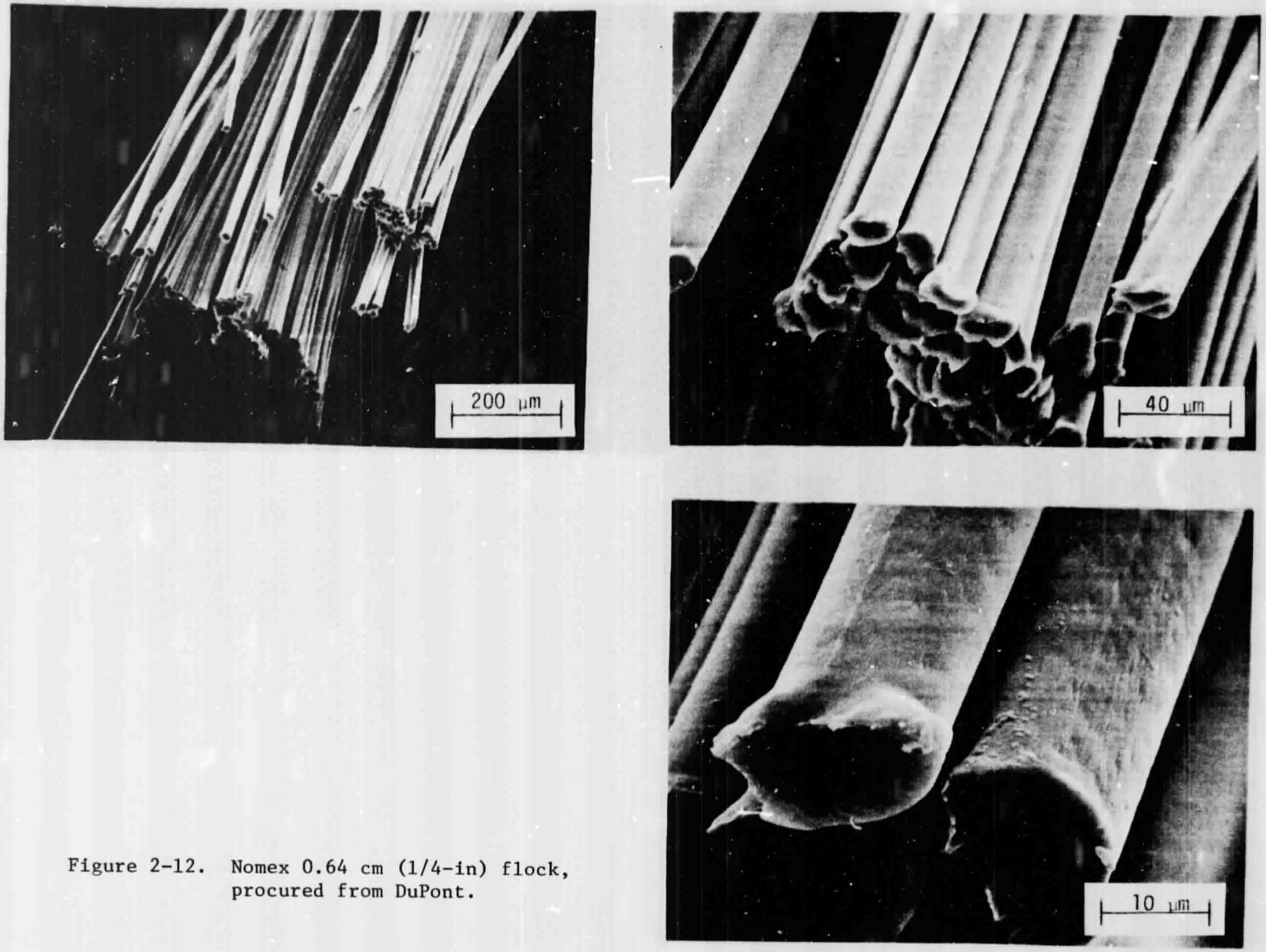


Figure 2-12. Nomex 0.64 cm (1/4-in) flock, procured from DuPont.

An acetylene-terminated polyimide, HR615P, was selected for infiltration-bonding of the deposited fibers. HR615P is a member of a family of polyimide oligomers designated HR600P, HR602P, HR603P, etc., recently developed at Hughes Aircraft Company (Reference 5). HR600P is a successor to Thermid 600 acetylene-terminated polyimide, developed earlier at Hughes. The HR600P prepolymers are isomers of Thermid 600 prepolymers and yield polymers which are believed to be identical to Thermid 600 polymers when fully cured. Their cure and post-cure temperatures are 204°C (400°F) and 371°C (700°F), respectively. The prepolymers are soluble in several common solvents and are easily processed. The cured polymers have long-term strength retention at 316°C (600°F) and short-term strength retention well above 371°C (700°F). They should easily meet standard NASA outgassing requirements.

The degree of polymerization (DP) of HR60XP prepolymers can be tailored to any value between 1 (HR600P) and 15 (HR615P). Increased DP of the prepolymer is associated with decreased cross-link density of the final polymer, and results in increased film forming capability, somewhat decreased glass transition temperature (Tg), and improved strain capability with little change in strength. Tough, free-standing films have been made from the higher DP versions. The HR615P material (DP = 15) was chosen for this program because it was believed that the high DP would maximize toughness of the bonded felts.

Other polyimide resins that were evaluated early in the program included:

- Thermid MC-600 acetylene-terminated polyimide molding powder, Gulf Oil Chemicals Company
- Thermid AL-600 amic ester polyimide precursor in ethanol (75 percent solids), Gulf Oil Chemicals Company.

However, neither of these resins resulted in adequate strength of bonding between the resin and the aramid fibers.

For some of the felts, an epoxy resin was used. This provided well-bonded felts that served as standards for room temperature comparisons with the polyimide-bonded materials. The epoxy system that was used was Epon 828/triethylene tetraamine (TETA).

2.4.2 Resin Infiltration Procedures

A resin infiltration procedure was needed that would provide: (1) good fillet bonds at the fiber cross-overs within the felt, and (2) felt properties approximating those listed in Table 2-1. Several generic approaches for infiltrating and internally bonding low-density deposited-fiber felts are summarized in Table 2-6. Preliminary experiments were conducted to evaluate three of these approaches for infiltrating samples of Rando Nomex fiber matt with a low content of polyimide resin. The three approaches were: (1) infiltration of the matt with dry polyimide powder, (2) spraying polyimide solution into the matt, and (3) immersion of the matt in dilute polyimide solution.

For approach (1), Thermid MC-600 molding powder was placed about samples of the matt, supported in an open fixture. The assemblies were vibrated and tumbled in a ball mill jar (without balls) and then cured. The resin powder penetrated the matt very poorly, resulting in concentration of the cured resin on the matt surfaces.

For approaches (2) and (3), Thermid AL-600 amic ester (75 percent solids in ethanol) was further diluted in tetrahydrofuran (THF) to various solids contents. Several spraying and dipping attempts were made using both uncompacted matt and matt precompacted to its final desired thickness between metal screens. The samples were rotated during thermal cure in an attempt to achieve uniform distribution of the resin. Results showed tendencies for the resin to concentrate near the matt surfaces. Further work was then done emphasizing more dilute solutions. Mixtures of THF and N-methyl pyrrolidinone solvents were evaluated as a means to provide enhanced solubility and better control of solvent removal rates.

The following successful technique was developed. First, a sample of the dry Nomex matt was sandwiched between plies of TX 1040 Teflon-coated fiberglass fabric and 112-weave fiberglass. The sandwich assembly was then placed in a glass desiccator modified to allow resin to be introduced through the lid (Figure 2-13). Vacuum was drawn in the desiccator, and AL-600 resin (75 percent solids in ethanol, further diluted with THF) was allowed to flow into the desiccator and impregnate the sandwich. The sandwich was then removed from

TABLE 2-6. APPROACHES FOR INFILTRATING LOW-DENSITY FELTS WITH RESIN

Infiltration Technique	Advantages	Disadvantages and Comments
Infiltration with dry resin powder.	No solvent required.	Difficult to achieve deep penetration into felt.
Spray dilute resin solution onto felt; compact to final thickness between plates or hot calenders; cure.	Standard technique for Rando matt process.	Possible resin content gradient through thickness.
Precompact felt to final thickness between screens; spray or slowly pour dilute resin solution onto felt and cure.	Good control of thickness.	Possible resin content gradient.
Precompact felt to final thickness between screens; very slowly dip into dilute resin bath.	Good control of thickness and resin pick-up.	Wetted felt might collapse under its own weight if dipped too fast.
Precompact felt to final thickness between screens; very lightly spray dilute resin and cure to rigidize felt; then dip rigidized felt as above to achieve final desired resin content.	Good control of thickness and resin pick-up.	2-step process.
Electrostatic deposition of dry resin powder.	No solvents required. Resin particles tend to migrate to fiber crossovers because of charge differences.	Experimental technique under development at Clemson University.

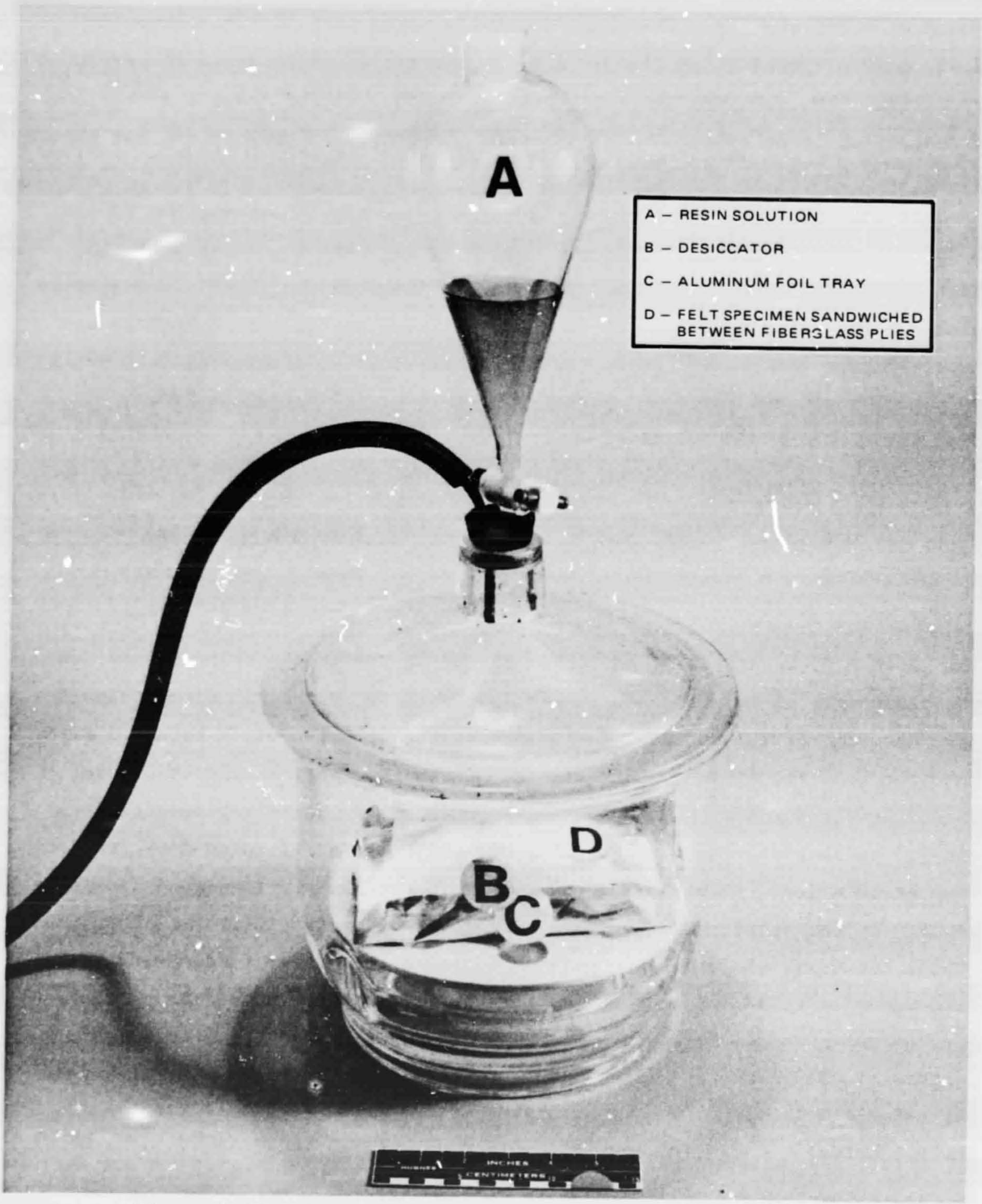


Figure 2-13. Laboratory apparatus for vacuum impregnation of felts with polyimide resin.

the desiccator, clamped between metal screens to control thickness, and oven cured to 257°C (495°F). The resulting impregnated matt was approximately 0.43 cm (0.17 inch) thick and had a resin content slightly above 25 percent by weight. The specimen was cross-sectioned and examined under a microscope and appeared to be uniformly infiltrated. However, the polymer did not appear to be well bonded to the Nomex fibers.

At this point a decision was made to use the highly processible HR615P polyimide resin for the remainder of the program, with a goal of improving both wetting of, and bonding to, the aramid fibers in the felts. A scaled-up assembly for infiltration of 15 cm x 15 cm (6 in. x 6 in.) felt samples was built (Figure 2-14), and the following infiltration procedure for both HP615P polyimide resin and 828/TETA epoxy resin was successfully developed.

First, the resin was mixed as follows:

Polyimide	or	Epoxy
HR615P - 10 gms		Epon 828 - 20 gms
THF - 200 gms		TETA - 2 gms
		Acetone - 200 gms

The 15 cm x 15 cm dry felt sample was placed in the impregnation column, and the resin-solvent solution was carefully poured in so that it slowly flowed down one side of the column. The resin-solvent solution was allowed to soak into the felt for 5 minutes and was then drawn through the felt by drawing vacuum in the chamber beneath the filter assembly. The impregnated felt sample, still supported on the filter assembly, was dried in an air-circulating oven at 80°C (176°F) for 20 minutes. (This procedure provided good infiltration by the epoxy resin; however, to provide thorough infiltration by the HR615P resin, it was usually necessary to repeat the above procedure with the felt turned over.) The impregnated felt sample was then placed in the molding fixture and cured as follows: polyimide — 2 hours at 204°C (400°F); epoxy — 2 hours at 93°C (200°F). (It would be necessary to post-cure polyimide-bonded SIP materials to 316°C (600°F) or higher before exposure to SIP thermal environments.)

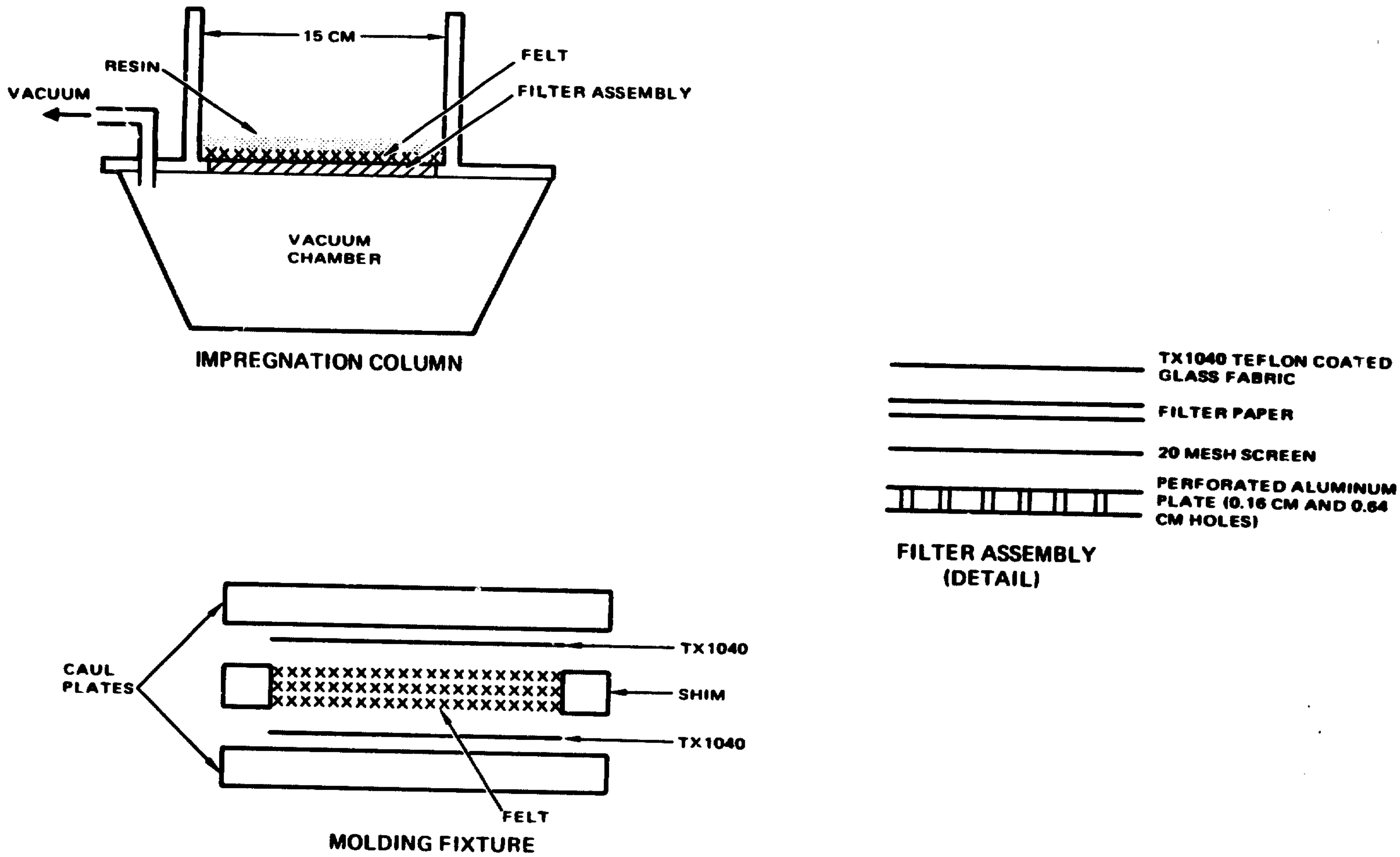


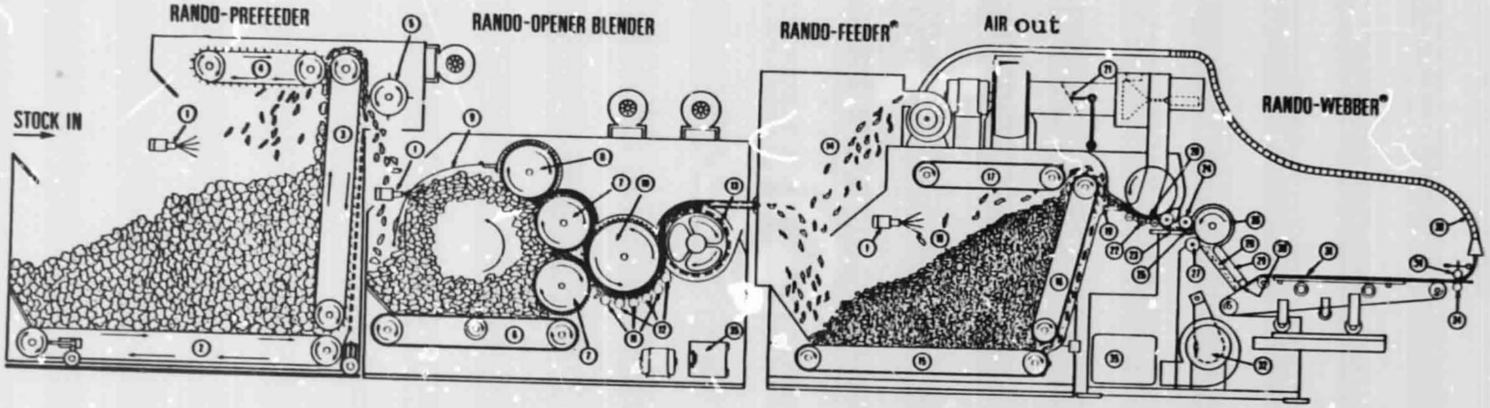
Figure 2-14. Schematic representation of scaled-up apparatus for infiltration-bonding of felts.

The above procedure provided good wetting of the aramid fibers by the epoxy resin and fair wetting by the polyimide resin. Under this initial effort, no special techniques to promote wetting and bonding of the resins to the aramid fibers at the fiber cross-overs were evaluated. Two promising approaches that should be considered in the future are plasma etching of the fiber surfaces in the felt, and coating the fiber surfaces with titanate coupling agents. Recent experiments at Hughes showed that titanate coupling agents can dramatically increase the wetting of aramid fibers by epoxy resins.

2.5 FABRICATION OF AIR-LAID FELTS

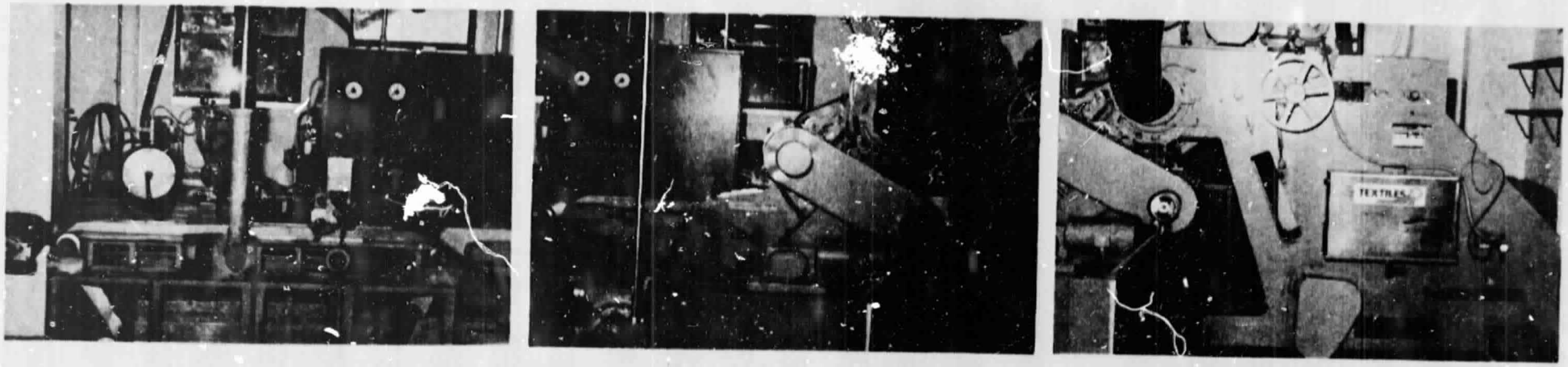
An air-laying matt machine manufactured by the Rando Machine Corporation, Macedon, New York, was used to make sheets of Nomex and Kevlar felts with various fiber orientations and contents. Production machines are available that can lay down mat' up to 122 cm (48 inches) wide, but for this program, a 30 cm (12 inch) pilot plant machine at Clemson University was employed. A schematic representation of a full-scale Rando matt laying production line is shown in Figure 2-15. Photographs of the pilot line at Clemson University are shown in Figure 2-16. As seen in this figure, the pilot line at Clemson University begins at the feeder station. A list of typical products made on Rando machines is provided in Table 2-7.

The initial experiments that were conducted at Clemson University were simply to manufacture Nomex and Kevlar-29 matt samples using conventional machine settings. The standard 3.8 cm (1-1/2 inch) crimped fibers normally used for Rando machine operation were selected for these experiments. Weight per unit area of these preliminary matt samples was measured and found to be 2.9×10^2 gm/m^2 (8.7 oz/yd^2) for the Nomex and 2.4×10^2 gm/m^2 (7.1 oz/yd^2) for the Kevlar materials. The uncompacted matts had very high loft [approximately 5 cm (1 in.)]. Samples of the Nomex material were internally bonded at Hughes Aircraft using Thermid AL-600 amic ester, following the successful infiltration and compaction procedure for this resin, discussed in Section 2.4.2. The completed impregnated specimens were approximately 0.43 cm (0.17 in.) thick and had a resin content slightly above 25 percent by weight. The impregnated felts were found to have predominantly planar fiber



1. Anti-Static Spray System
2. Floor Apron
3. Elevating Apron
4. Stripper Apron
5. Doffer Roll
6. Floor Apron
7. Worker Rolls
8. Stripper Roll
9. Hopper Cover
10. Main Cylinder
11. Small Worker Rolls
12. Small Stripper Rolls
13. Air Brush
14. Fiber Separator
15. Floor Apron
16. Elevating Apron
17. Stripper Apron
18. Hopper Level Control
19. Air Bridge
20. Feed Mat Condenser Screen
21. Air Volume Control
22. Roller Conveyor
23. Feed Plate
24. Feed Roll
25. Nose Bar
26. Lickerin
27. Saber
28. Venturi
29. Duct Cover
30. Condenser for Forming RANDO-WEB
31. Takeaway Conveyor
32. WEBBER Fan
33. FIBR-SAVR®
34. Slitter Assembly
35. Cleanout Door

Figure 2-15. Typical Rando air-laid matt production process.



Foam Bonding Area ← Webber ← Feeder

Figure 2-16. Rando matt pilot line at Clemson University.

TABLE 2-7. TYPICAL COMMERCIAL PRODUCTS MANUFACTURED ON RANDO AIR-LAYING MATT MACHINES

Automobile air, oil, and gas filters
Sintered metal filters for jet engines
Abrasive pads, floor scrub pads
Tampons
Diaper components
Bra padding
Fiberfil quilting material
Molded automobile parts
Surgical tape
Liners for ski mobile boots

orientations, with very few fibers traversing the material thickness. Clearly, a substantially greater number of fibers oriented at least partially in the thickness direction are required for SIP applications.

To verify this observation, a specimen of this material was provided to the NASA Technical Monitor, who tested the material for tensile properties in the through-thickness direction. As expected, the properties in this direction were very low due to the planar orientations of the Nomex fibers in the specimen. The test results were:

Strength:	28 kPa (4 psi)
Modulus:	41 kPa (6 psi)
Elongation:	62 percent

Several Rando machine runs were then conducted using various machine adjustments to promote non-planar fiber deposition. Nomex felts, approximately 2.6 x 10^2 g/m^2 (7.6 oz/yd^2) in weight were fabricated. The Rando air-lay process inherently produced predominantly planar orientations of the Nomex fibers, with very few fibers traversing the material thickness. Because of this, the felts were subjected to a light needling operation prior to bonding to increase fiber entanglement. They were then placed back on the Rando pilot line and bonded with rigid acrylic resin by drawing the resin, in foam form, through the felts using air suction. The resulting lightly-needled, bonded felts had good integrity. However, the use of needling, even only lightly, did not appear to be a good approach because: (1) the high proportion of planar-oriented fibers did not significantly decrease, and (2) the disadvantages of the present SIP material that are attributed to needling were likely to be introduced, to some degree.

Additional experiments were then conducted to develop techniques to mix short fibers [typically 0.64 cm (1/4 inch) in length] with the standard longer crimped fibers [typically 3.8 to 5.1 cm (1-1/2 to 2 inches) in length] normally used in Rando matts. As the basic longer fibers in Rando matts are oriented primarily in the matt plane, the added short fibers were needed, with orientations predominantly at sharp angles to the matt plane, to improve through-thickness strength. Two goals for processing the short fibers were, (1) uniform distribution throughout the matt, and (2) maximum out-of-plane orientations.

Cut polyester fibers 0.64 cm in length were fed into the pilot-line Rando machine at the Feeder-Webber junction (Figure 2-15) in attempts to mix them with standard Rando matt material as it was being deposited [polypropylene fibers, 4 cm (1-9/16 inch) length]. The polyester and polypropylene fibers were of different colors to aid assessments of fiber orientations. Some, but not a sufficient degree of, the desired out-of-plane orientations of the short fibers were achieved.

For the final set of Rando machine experiments at Clemson University, eight Kevlar matt variations were fabricated. The matts were made using crimped Kevlar-29 staple fibers chopped to 0.64 cm and 5.1 cm lengths by the Mini-Fibers Company. Some of the samples were made using the normal air flow rate in the Rando machine, which resulted in an approximately 12.7 cm (5 inch) shingle plane of the fibers in the matt. Other samples were made using modified air flow characteristics by decreasing the air flow opening width to 3.8 cm (1-1/2 inch). This modification produced matts with significantly shorter shingle planes. The machine adjustments used to make these eight matts are listed in Table 2-8.

The eight unbonded samples were shipped to Hughes Aircraft Company. A sample from roll No. 7 was compacted to the final required thickness (0.41 cm, 0.16 in.) and bonded with dilute HR615P polyimide resin. The bonded sample was sectioned; the fibers in the bonded matt were found to be oriented predominantly in the plane of the matt, with very few fibers oriented in the thickness direction. The conclusion was that compacted felts made using the Rando process tended to have predominantly planar fiber orientations regardless of the height of the shingle plane of the matt prior to compaction.

2.6 DEVELOPMENT OF LIQUID-DEPOSITED FELTS

2.6.1 Bench Top Apparatus for Felt Deposition

A bench top apparatus was constructed to make bonded felts by depositing very short fibers from liquid slurries. The apparatus was constructed with assistance from Dr. John V. Milewski, Los Alamos Scientific Laboratory, and the design was based on his previous work summarized in Section 2.2. To make

TABLE 2-8. RANDO MATTS FABRICATED USING SPECIAL MACHINE MODIFICATIONS

Roll No.	Fiber Types and Lengths*	Fiber Feed Speed**		Lickerin Speed, rpm***	Conveyor Speed****		Shingle Plane	
		cm/min	ft/min		cm/min	ft/min	cm	in
1	A	7.6	0.25	1140	73	2.4	12.7	5.0
2	A	7.6	0.25	2050	73	2.4	12.7	5.0
3	A	25	0.82	2050	73	2.4	12.7	5.0
4	B	7.6	0.25	2050	73	2.4	12.7	5.0
5	B	25	0.82	2050	73	2.4	12.7	5.0
6	A	7.6	0.25	2066	73	2.4	3.8	1.5
7	A	25	0.82	2066	73	2.4	3.8	1.5
8	B	25	0.82	1900	73	2.4	3.8	1.5

*A = 0.64 cm (1/4 inch) Kevlar-29, crimped, 2.2 Decitex (2 denier)
B = 70% A plus 30% 5.1 cm (2 inch) Kevlar-29, crimped, 2.2 Decitex (2 denier)

**Under feed roll (item 24 in Figure 2-15)

***Item 26 in Figure 2-15; covered with quick-release wire, 6.2 points/cm^2 (40 points/in^2)

****Item 31 in Figure 2-15

the felts, short chopped fiber bundles are first mixed into a liquid carrier and separated into individual fibers by vigorous agitation using a Waring blender. Vacuum is then used to draw the fiber-liquid slurry through an inverted Buchner filter funnel, and the fibers are deposited on the filter as a felt. To bond the fibers, a dilute solution of commercial hair spray is drawn through the felt, and the felt is allowed to dry. This "bonded" felt can be subsequently "coated" with epoxy or polyimide resin to produce the completed low-density structural felt. Photographs of the apparatus that was built are shown in Figures 2-17, 2-18, and 2-19.

Many types of fibers and organic coatings can be combined to form felts using this process (Reference 4). Orientation of the fibers in the felts can be controlled by varying the fiber lengths, the concentration of fibers in the slurry, and the rate at which the slurry is drawn through the filter. More rapid drawing rates promote increased non-planar orientation of the fibers, which was needed for the materials being developed.

For the initial experiments conducted to establish the process, 0.16 cm (1/16-inch) chopped Kevlar-49 fiber bundles were separated by agitation in an isopropanol carrier. The felt specimens that were made from these fibers are described in Table 2-9. The specimens were cross-sectioned, and the orientations of the short Kevlar fibers were found to be predominantly planar, with few, if any, fibers traversing the material thickness. A more powerful vacuum pump, needed for more rapid drawing of the fiber slurries through the filter screen, was apparently required to promote non-planar deposition of the fibers. Also, a larger apparatus was needed to make specimens suitable for mechanical testing.

2.6.2 Construction of Scaled-up Felt Deposition Apparatus

A scaled-up apparatus to deposit felts up to approximately 15 x 15 cm (6 x 6 inches) in size was constructed (Figure 2-20). An important feature of the apparatus was a powerful vacuum pump to allow rapid drawing of the fiber slurries through the filter screen in order to promote achievement of the needed out-of-plane orientations of the deposited short fibers.

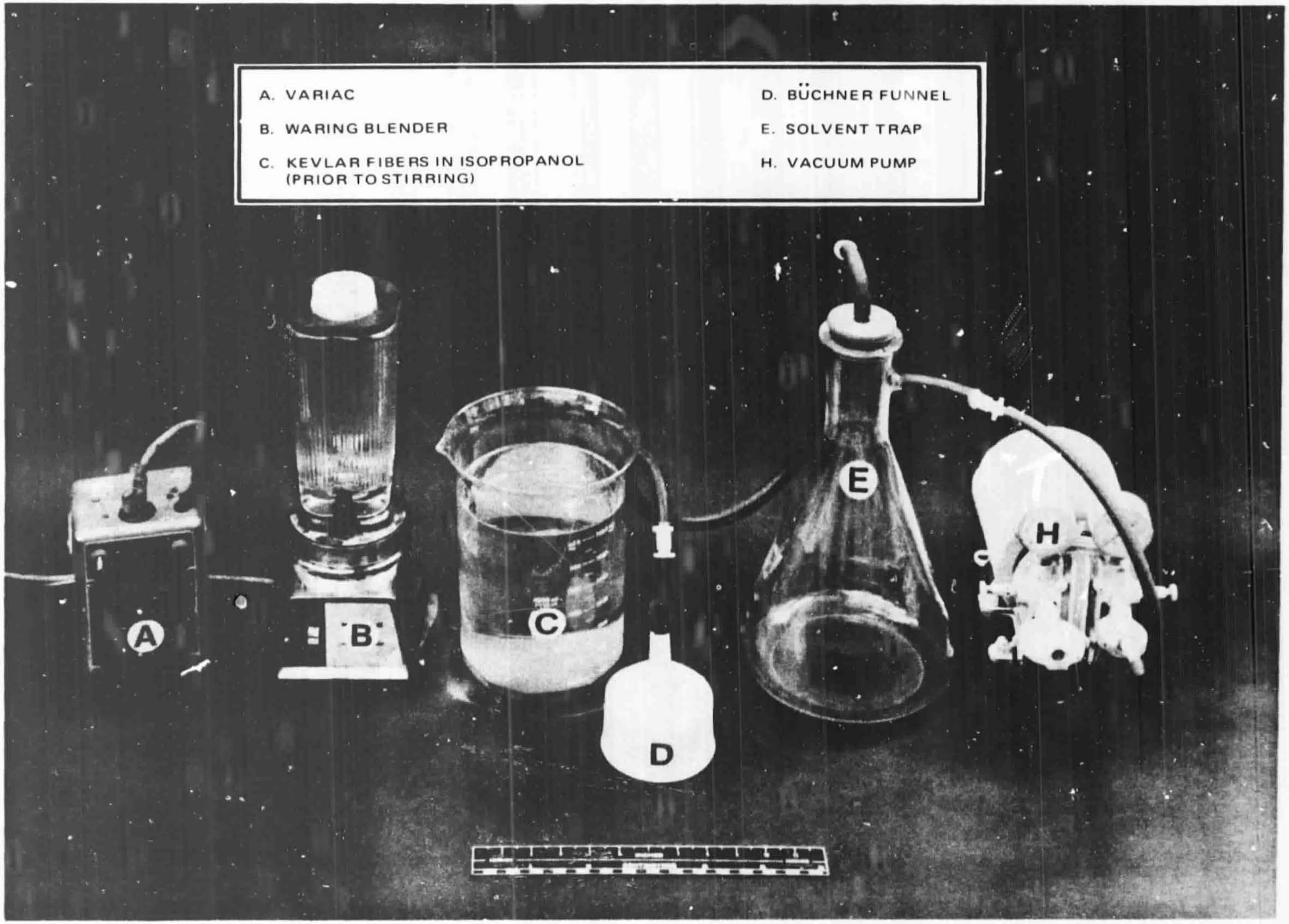


Figure 2-17. Apparatus for making short-fiber felts.

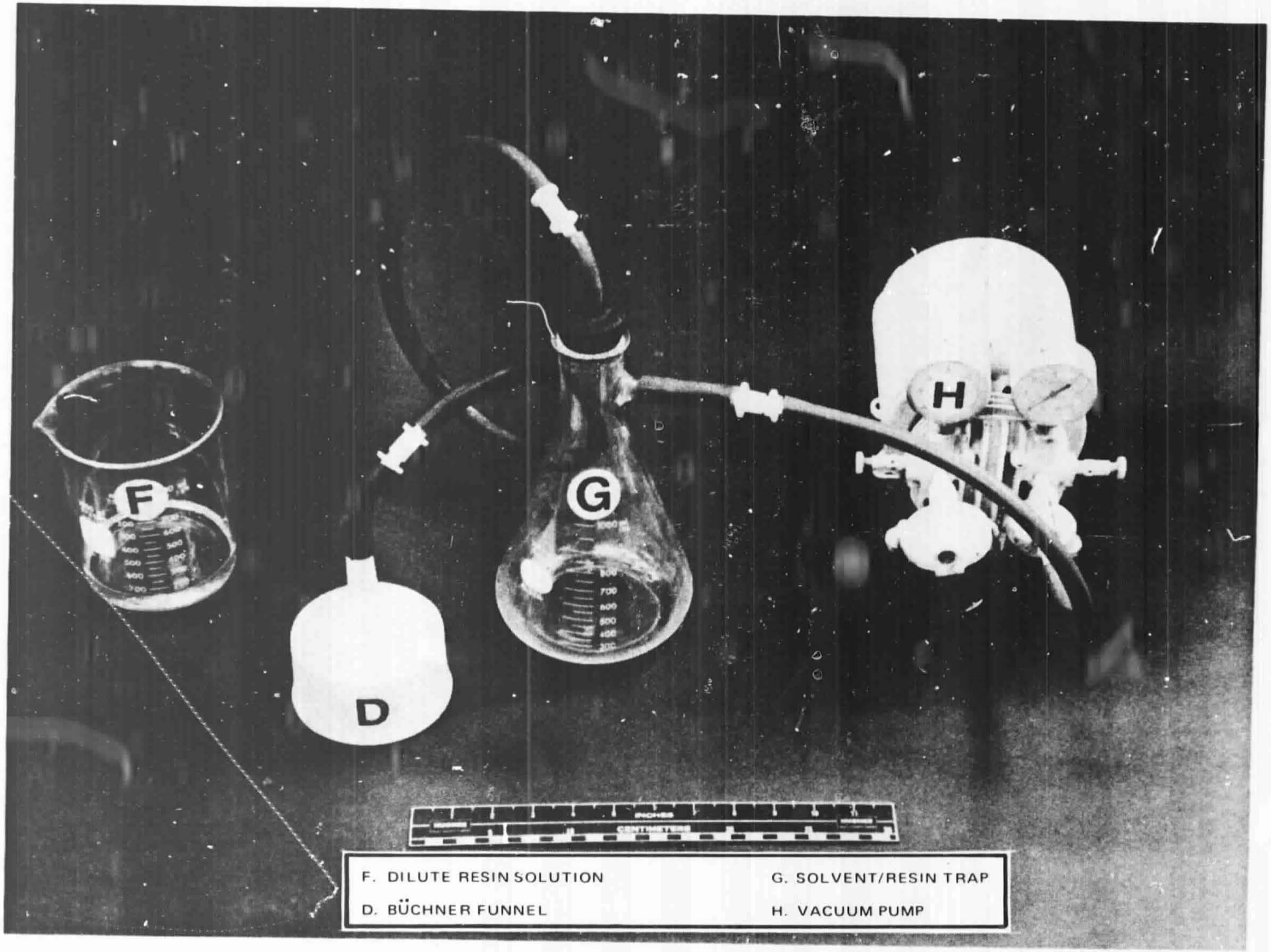


Figure 2-18. Apparatus for internally bonding felt samples with resin.

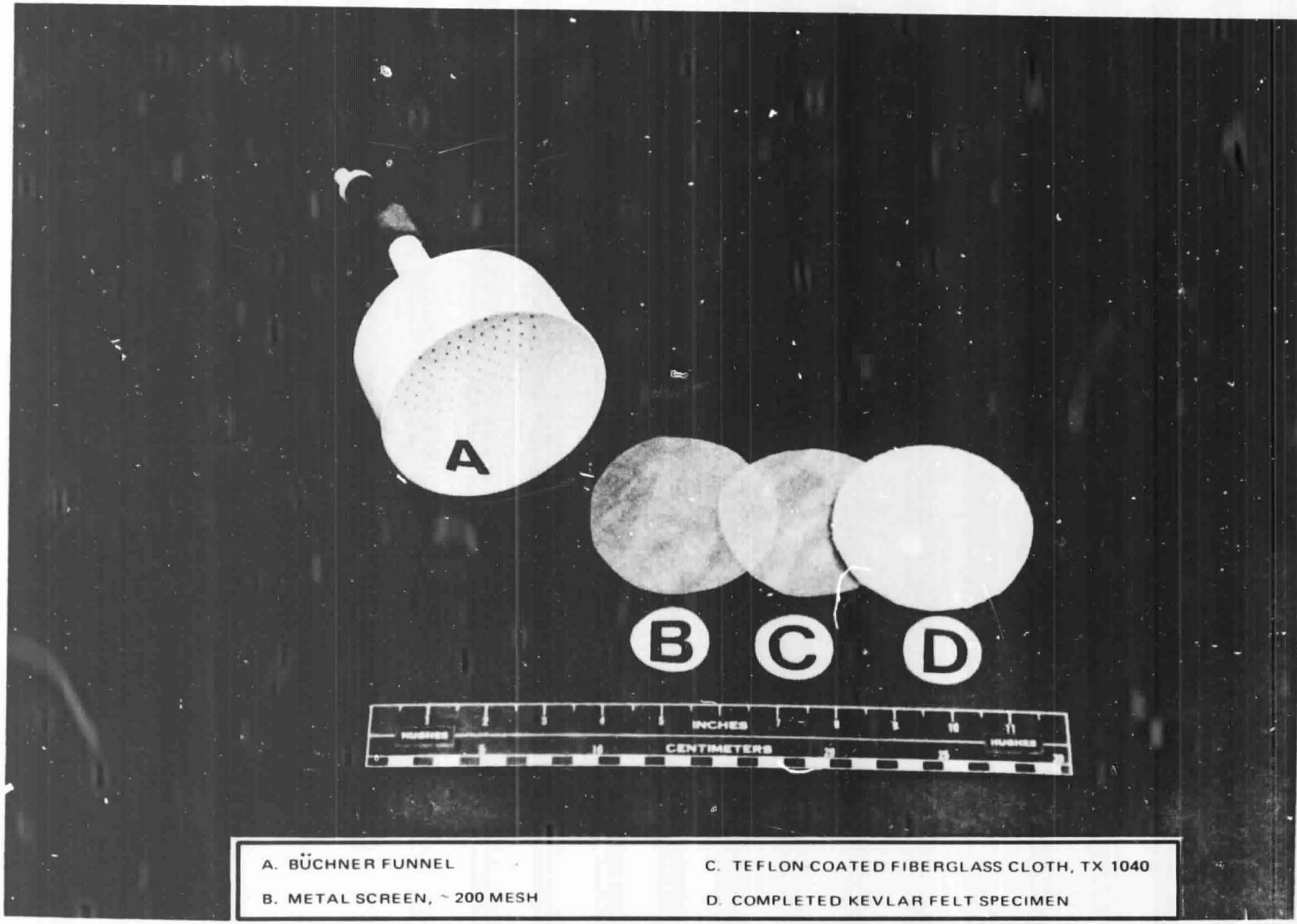


Figure 2-19. Filter assembly for deposition and bonding of short-fiber felts.

TABLE 2-9. INITIAL SHORT-FIBER FELTS MADE IN BENCH TOP APPARATUS

Specimen No.	Fiber Weight, g	Sample Size, cm		Sample Weight with Binder, g	Binder Content, %	Bulk Density, g/cm^3
		Diameter	Thickness			
1	—	4.255	0.533	0.4300	—	0.057
2	0.4065	4.648	1.016	(0.4065)	0	0.023
3	0.3000	4.648	0.457	0.3278	9.27	0.042
4	0.3000	4.648	0.533	0.3239	7.97	0.036
6	0.3445	4.648	0.470	0.5007	45.3	0.063
7	0.6000	4.699	0.749	0.6243	4.05	0.048

Fibers — Kevlar-49, 0.16 cm (1/16 inch) chopped by Finn & Fram, Inc.

Binder — Commercial hairspray

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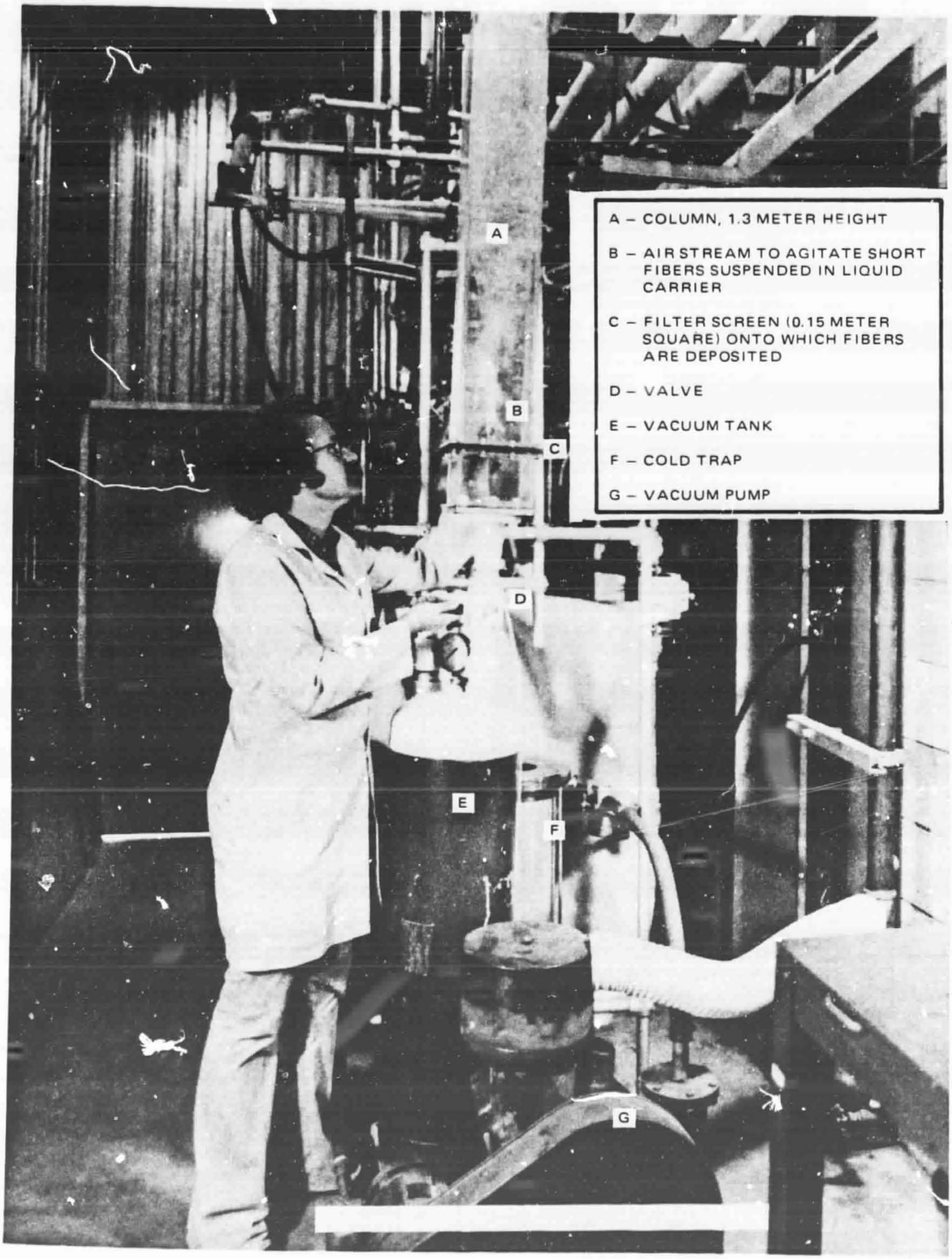

Figure 2-20. Scaled-up apparatus for making short-fiber felts.

Initial runs were made to establish operating procedures. The first step was to separate the chopped Kevlar or Nomex yarn bundles into individual short filaments. This was done by mixing the fibers in a solvent (water, isopropanol, or a blend of these) and agitating the mixture in a large Waring blender to achieve a slurry of individual chopped filaments. Before the slurry had time to settle, it was poured into the top of the deposition column, already partially filled with the same solvent. Agitation was continued in the column by bubbling air through the slurry. The vacuum tank beneath the column (Figure 2-20, item E) was then evacuated, and valve D was opened to allow the slurry to be quickly drawn through the filter screen, thus allowing the fibers to be deposited as a felt on the screen. A 1.2-meter high fiber slurry column (0.16 cm fibers) would deposit as a 15-centimeter square felt in approximately 4.5 seconds. The flow rate through the filter was approximately 27 $ml/cm^2/sec$, which was approximately eight times the flow rate achieved with the laboratory-scale apparatus described in Section 2.6.1 (3.2 $ml/cm^2/sec$).

2.6.3 Initial Felt Deposition Experiments in Scaled-up Apparatus

Thirteen felt samples were made in the scaled-up liquid deposition apparatus. The processing details and observations are summarized in Table 2-10. Some out-of-plane orientations of the fibers could be achieved by using a 50-50 mix of short and long fibers. Adding a small amount of Kevlar pulp to the liquid slurry significantly improved mechanical bonding of the felt.

2.6.4 Slow-Draw Experiments and Incorporation of Fugitive Particles

Two approaches were attempted to increase out-of-plane fiber orientations. The first approach was to slow the time to draw the fiber slurry through the filter screen from approximately 4.5 seconds to approximately 18 to 20 seconds so that this might create more laminar flow patterns which could promote end-on deposition of the fibers. The laboratory observations are summarized in Table 2-11. This procedure resulted in very uneven felt thicknesses and did not appear to increase out-of-plane orientations.

TABLE 2-10. INITIAL 15 CM^2 SHORT-FIBER FELTS MADE IN SCALED-UP APPARATUS

Specimen No.	Fiber Types and Lengths*	Draw Time, seconds	Bulk Density, Unimpregnated, gm/cm^3	Epoxy Resin Content, wt %	Fiber Orientations
1, 2	0.16-cm Kevlar-49	∿4.5	0.06	0	Layered parallel to felt surface
3	0.16-cm Kevlar-49, 90% 0.64-cm Kevlar-29, 10%	∿4.5	0.06	0	Layered parallel to felt surface
4, 5	0.16-cm Kevlar-49	∿4.5	0.06	5	Layered parallel to felt surface
6	0.16-cm Kevlar-49	∿4.5	0.06	15	Layered parallel to felt surface
7	0.16-cm Kevlar-49	∿4.5	0.05	25	Layered parallel to felt surface
8	0.16-cm Kevlar-49, 90% 0.64-cm Kevlar-29, 10%	4.2	0.06	0	Layered parallel to felt surface
9	0.64-cm Nomex	4.3	0.06	16	Layered parallel to felt surface
10	0.34-cm Nomex	4.2	0.06	16	Layered; fibers loose and easily moved; some fiber movement occurred during resin impregnation
11	0.34-cm Nomex, 50% 0.64-cm Nomex, 50%	4.4	0.06	16	Mostly layered; some out-of-plane fibers
12	0.16-cm Nomex, 50% 0.64-cm Nomex, 50%	4.4	0.06	0	Mostly layered; fibers loose and easily moved
13	0.16-cm Nomex, 45% 0.64-cm Nomex, 45% Kevlar Pulp, 10%	4.8	0.06		Mostly layered; good mechanical integrity

*Weight of fibers in slurry = 6 gms.

TABLE 2-11. RESULTS OF SLOW-DRAW EXPERIMENTS

Fiber		Suspension Fluid		Draw Time, seconds	Process Details	Result
Type	Length	Medium	Ratio			
Nomex	0.16 cm	Water	100%	18	No air agitation; no vacuum; fibers remained clumped in 4-5 strand groups; 20% floated to top.	Most of the fibers tumbled quickly to the base of the column, producing felt with uneven thickness
Nomex	0.16 cm	Water	100%	20	No vacuum; air agitation during drawing	Uneven thickness

The other approach was to incorporate fugitive small particles into the fiber slurries to enhance isotropic packing of the fibers. Requirements for the particles included: (1) specific gravity roughly equivalent to that of Nomex so that the particles would mix uniformly with the fibers in the liquid slurry, and (2) ability to be dissolved or otherwise broken down by a means that would not affect the Nomex.

Unexpanded Dylite polystyrene particles were obtained from ARCO Polymers, Inc., and mixed into the standard Nomex slurries. The laboratory observations are summarized in Table 2-12. The results showed that the beads could not be dissolved out of the felts without compromising the felt integrity.

2.6.5 Fiber Channelization Experiments

Sheets of honeycomb core with various cell sizes and configurations were procured or constructed and were installed near the base of the fiber slurry deposition column. The purpose of the honeycomb was to promote end-on flow of the chopped fibers in the slurry as they approached the filter screen, thereby increasing end-on deposition of the fibers (Figure 2-21). A total of 26 felt deposition runs were made using various fiber and pulp combinations and various types of honeycomb positioned at various heights above the filter screen. Results of these experiments are summarized in Table 2-13.

The following observations were made based on the results of these experiments.

1. Mixtures of a larger amount of shorter fibers (e.g., 0.16 cm) and a smaller amount of longer fibers (e.g., 0.64 cm) appeared to provide greater loft (thickness before resin infiltration) and fiber direction isotropy than either short fibers or long fibers alone. The shorter fibers appeared to penetrate or "shoot" into spaces between the longer fibers already deposited and thus remain in a "standing" or vertical position.
2. The bulk density goal (0.067 gm/cm^3) for Class 3 SIP material, established in Table 2-3, could be readily achieved. However, sufficient loft and out-of-plane fiber orientations to meet the 0.406 cm thickness goal were difficult to achieve. Subsequent resin infiltration bonding tended to further reduce the felt thickness.

TABLE 2-12. EXPERIMENTS INCORPORATING FUGITIVE PARTICLES INTO THE FIBER SLURRIES

Fiber		Suspension Fluid		Draw Time, seconds	Description of Particles and Process Details*	Results
Type	Length and Weight	Medium	Ratio			
Nomex	0.16-cm 6 gms	Isopropanol/ water	2:1	5	Styrene, Type C, 26 gms	Majority of beads fell out of suspension; deposited at bottom of felt
Nomex	0.16-cm 6 gms	Isopropanol/ water	2:1	5	Styrene, cup size, 6 gms, air agitation prior to evacuating	Well dispersed in felt; lumping around beads
Nomex	0.16-cm 6 gms	Isopropanol/ water	2:1	5	Styrene, Type C, 6 gms, air agitation prior to evacuating	Well dispersed in felt; poor felt integrity

*Particle Type	Particle Size, cm
C	0.083 - 0.043
Cup	0.051 - 0.030

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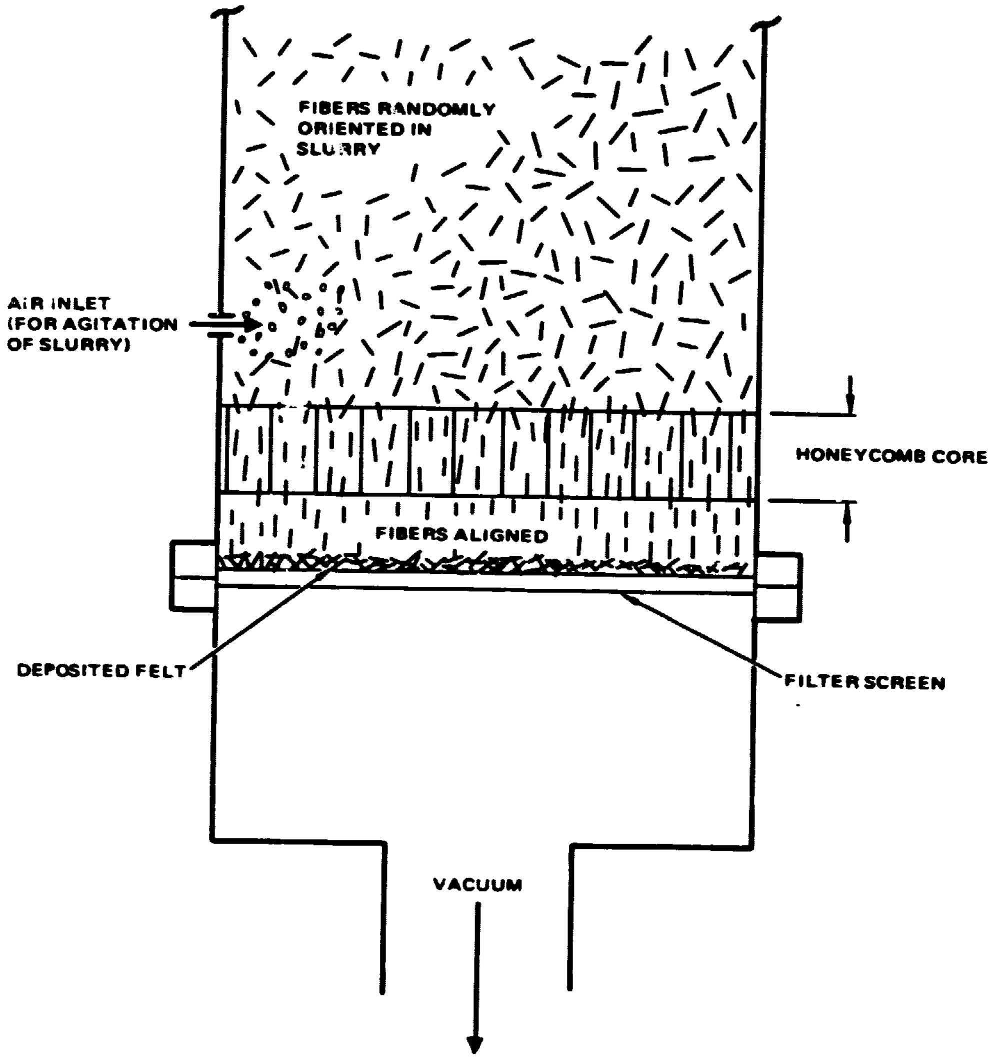

Figure 2-21. Schematic depiction of felt-making apparatus with honeycomb core installed near base of column.

TABLE 2-13. DEPOSITION OF FELTS BY CHANNELING FIBER SLURRIES THROUGH HONEYCOMB CORE — EXPERIMENTAL RESULTS

Specimen No.	Fiber Types and Lengths*	Honeycomb Description: Cell Material and Configuration	Honeycomb Description: Core Thickness, cm	Space Between Honeycomb and Filter Pad, cm	Draw Time, seconds	Results and Observations**	Resin Impregnation**
1	0.16 cm Nomex, 45% 0.64 cm Nomex, 45% Kevlar pulp, 10% Total weight, 6.0 gms	Corrugated titanium, 0.76 cm square cells	1.9	3.8	32	Approximately 60 percent of the fibers deposited on top of the honeycomb and not on the filter screen	Not attempted
2	0.16 cm Nomex, 80% Kevlar pulp, 20% Total weight, 6.0 gms	Corrugated titanium, 0.76 cm square cells	1.9	3.8	14	Fibers deposited on top of the honeycomb cell walls, decreasing the effective open cell size from 0.76 to 0.43 cm. The fibers that reached the screen deposited in uneven thicknesses in a pattern matching the honeycomb cell structure. The final felt thickness was approximately 0.28 cm.	Not attempted
3	0.16 cm Nomex, 50% Kevlar pulp, 10% Total weight, 6.5 gms	Corrugated titanium, 0.76 cm square cells	1.9	3.8	9.0	Uneven deposition on screen. Weight, 6.0 gm. Thickness, 0.27 cm Loft, poor	Not attempted
4	0.16 cm Nomex, 65% 0.64 cm Nomex, 30% Kevlar pulp, 5% Total weight, 6.5 gms	Aluminum, 0.95 cm hexagonal cells	1.3	5.1	7.5	Fiber deposition on top of the cell walls (1.5 gms) decreased the effective cell size to 0.33 cm. Weight, 5.0 gms Thickness, 0.41 - 0.46 cm Some layering	Not attempted
5	0.16 cm Nomex, 78% 0.64 cm Nomex, 15% Kevlar pulp, 7% Total weight, 7.0 gms	Aluminum, 0.95 cm hexagonal cells	1.3	5.1	5.0	Slight, feathery fiber buildup on top of the cell walls. Deposited felt had good integrity. Weight, 5.5 gms Thickness, 0.43 cm	Not attempted
6	0.16 cm Nomex, 65% 0.64 cm Nomex, 30% Kevlar pulp, 5% Total weight, 7.0 gms	Fiberglass epoxy, rectangular cells, 1.9 x 3.8 cm	4.4	1.9	6.7	Slight fiber buildup on top of the cell walls. Increased felt thickness beneath each cell wall, indicating flow of fibers toward the cell walls. Weight, 6.23 gms Thickness, 0.46 cm Good integrity	Vacuum impregnated with 5 percent solids HR615P polyimide resin. Resin deposited mostly at top of felt. Weight after cure, 8.05 gms. Fiber layering revealed when specimen dissected.

(Table 2-13, continued)

Specimen No.	Fiber Types and Lengths*	Honeycomb Description: Cell Material and Configuration	Honeycomb Description: Core Thickness, cm	Space Between Honeycomb and Filter Pad, cm	Draw Time, seconds	Results and Observations**	Resin Impregnation**
7	0.16 cm Nomex, 65% 0.64 cm Nomex, 30% Kevlar pulp, 5% Total weight, 7.0 gms	Fiberglass/epoxy, 1.9 cm square cells	4.[illegible]	1.4	6.7	Slight fiber buildup on top of the cell walls. Weight, 6.02 gms Thickness, 0.41 cm	Vacuum impregnated with 5 percent solids HR615P polyimide resin. Thickness decreased significantly, causing increased in-plane orientation of fibers. Weight, 8.09 gms Thickness before cure, 0.25 cm Thickness after cure, 0.19 cm
8	0.16 cm Nomex, 65% 0.64 cm Nomex, 30% Kevlar pulp, 5% Total weight, 6.5 gms	Fiberglass/epoxy, 1.9 cm square cells	4.4	7.6	6.7	Some layering	Not attempted
9	0.34 cm Nomex, 95% Kevlar pulp, 5% Total weight, 6.5 gms	Fiberglass/epoxy, 1.9 cm square cells	4.4	7.6	6.0	Thickness, 0.43 cm Center section layered	Not attempted
10	0.34 cm Nomex, 95% Kevlar pulp, 5% Total weight, 6.5 gms	Fiberglass/epoxy, 1.3 cm square cells	4.4	1.3	6.0	Fibers deposited in lumps	Not attempted
11	0.34 cm Nomex, 95% Kevlar pulp, 5% Total weight, 6.5 gms	Fiberglass/epoxy, 1.3 cm square cells	4.4	7.6	6.0	Weight, 4.82 gms*** Thickness, 0.25 cm Layered	Not attempted
12	0.34 cm Nomex, 95% Kevlar pulp, 5% Total weight, 6.5 gms	Fiberglass/epoxy, 1.3 cm square cells	4.4	5.1	6.0	Weight, 4.75 gms*** Thickness, 0.23 cm Layered	Not attempted
13	0.34 cm Nomex, 95% Kevlar pulp, 5% Total weight, 6.5 gms	Fiberglass/epoxy 1.3 cm square cells	4.4	2.5	6.0	Slight lumping Weight, 4.76 gms*** Thickness, 0.23 cm Layered	Not attempted
14	0.16 cm Nomex, 64% 0.34 cm Nomex, 30% Kevlar pulp, 1% Total weight, 7.5 gms	Fiberglass/epoxy, 1.3 cm square cells	4.4	5.1	6.0	Weight, 6.58 gms Thickness, 0.48 cm Poor mechanical integrity	Not attempted

(Table 2-13, continued)

Specimen No.	Fiber Types and Lengths*	Honeycomb Description: Cell Material and Configuration	Honeycomb Description: Core Thickness, cm	Space Between Honeycomb and Filter Pad, cm	Draw Time, seconds	Results and Observations**	Resin Impregnation**
15	0.16 cm Nomex, 30% 0.34 cm Nomex, 69% Kevlar pulp, 1% Total weight, 7.5 gms	Fiberglass/epoxy, 1.3 cm square cells	4.4	5.1	6.0	Weight, 6.56 gms Thickness, 0.43 cm Poor mechanical integrity	Vacuum impregnated with 2 percent solids HR615P polyimide resin; layered; final thickness, 0.30 cm
16	0.16 cm Nomex, 70% 0.34 cm Nomex, 27% Kevlar pulp, 3% Total weight, 7.5 gms	Fiberglass/epoxy, 1.3 cm square cells	4.4	5.1	6.0	Sprayed lightly with commercial hair spray to improve mechanical integrity. Weight, 6.98 gms Thickness, 0.30 cm Layered in center	Not attempted
17	0.16 cm Nomex, 50% 0.34 cm Nomex, 50% Total weight, 7.5 gms	Fiberglass/epoxy, 1.3 cm square cells	4.4	5.1	6.0	Sprayed as above. Weight, 6.79 gms Thickness, 0.32 cm Layered in center	Not attempted
18	0.16 cm Nomex, 100% Total weight, 7.5 gms	0.0018 cm aluminum, 0.95 cm hexagonal cells	5.1	2.5	6.0	Deposited in lumps	Not attempted
19	0.16 cm Nomex, 100% Total weight, 7.5 gms	0.0018 cm aluminum, 0.95 cm hexagonal cells	5.1	5.1	6.0	Sprayed as above. Thickness, 0.28 cm Poor loft, some layering	Not attempted
20	0.34 cm Nomex, 100% Total weight, 7.5 gms	0.0018 cm aluminum, 0.95 cm hexagonal cells	5.1	5.1	6.0	Sprayed as above. Thickness, 0.20 cm (Hair spray caused felt to compact.) Layered in center	Not attempted
21	0.16 cm Nomex, 50% 0.34 cm Nomex, 50% Total weight, 7.5 gms	0.0018 cm aluminum, 0.95 cm hexagonal cells	5.1	5.1	6.0	Fiber deposition on top of the honeycomb cell walls decreased the effective cell size to 0.33 cm. Thickness, 0.19 cm Poor loft and integrity	Not attempted
22	0.16 cm Nomex, 70% 0.34 cm Nomex, 30% Total weight, 7.5 gms	0.0018 cm aluminum, 0.95 cm hexagonal cells	5.1	5.1	6.0	Thickness, 0.24 cm Layered	Not attempted
23	0.16 cm Nomex, 70% 0.34 cm Nomex, 30% Total weight, 7.5 gms	0.0018 cm aluminum, 0.95 cm hexagonal cells	5.1	2.5	6.0	Thickness, 0.24 - 0.28 cm Layered	Not attempted

(Table 2-13, concluded)

Specimen No.	Fiber Types and Lengths*	Honeycomb Description: Cell Material and Configuration	Honeycomb Description: Core Thickness, cm	Space Between Honeycomb and Filter Pad, cm	Draw Time, seconds	Results and Observations**	Resin Impregnation**
24	0.16 cm Nomex, 40% 0.34 cm Nomex, 30% 0.079 cm Nomex, 30% Total weight, 7.5 gms	0.0018 cm aluminum, 0.95 cm hexagonal cells	5.1	2.5	6.0	Thickness, 0.21 - 0.25 cm Layered	Not attempted
25	0.16 cm Nomex, 40% 0.34 cm Nomex, 30% 0.079 cm Nomex, 30% Total weight, 7.5 gms	Fiberglass/epoxy, 1.3 cm square cells	4.4	2.5	6.0	Thickness, 0.20 cm Layered	Not attempted
26	0.16 cm Nomex, 70% 0.34 cm Nomex, 15% 0.079 cm Nomex, 15% Total weight, 7.5 gms	0.0018 cm aluminum, 0.95 cm hexagonal cells	5.1	3.8	6.0	Thickness, <0.25 cm Layered	Not attempted

*Fibers dispersed in a mixture of 2 parts isopropyl alcohol and 1 part water.

**Thickness goals

 >0.41 cm before impregnation
 0.41 cm after impregnation and core

All thickness measurements made using Randall Stickney gage.

***Approximately 1.5 gm buildup of 0.34 cm fibers on top of the honeycomb cell walls.

3. Adding a small amount of Kevlar pulp to the fiber slurry substantially enhanced mechanical integrity of the unimpregnated felts and prevented disruption or shifting of the Nomex fibers during subsequent resin bonding. However, use of too high a percentage (greater than 5 percent) of this highly fibrillated material caused the felt to draw together tightly during bonding, thus causing too much reduction in thickness.
4. Use of a commercial hair spray in lieu of the Kevlar pulp as an aid to enhance mechanical integrity resulted in excessive felt compaction.
5. Channeling the fibers through a honeycomb core with 1.9 cm square cells appeared to produce felts with the highest loft. Larger cells did not appear to create adequate channelization to cause the fibers to deposit vertically. Smaller cells were ineffective because the fibers tended to deposit on top of the honeycomb.
6. For each honeycomb configuration, there appeared to be an optimum size space between the bottom of the honeycomb and the top of the filter screen. If this space was too large, the fibers tended to lose any alignment achieved due to channeling before they reached the filter screen. If the space was too small, the resulting individual slurry jets coming from each honeycomb cell tended to disrupt the fibers previously deposited, resulting in uneven felt thicknesses.
7. Thicker sheets of honeycomb core appeared to produce greater vertical alignment of the fibers than did thinner sheets.

The material and processing parameters for specimen No. 7 in Table 2-13 were selected for fabrication of the felt specimens to be delivered to NASA. Scanning electron micrographs of this specimen are shown in Figure 2-22. The majority of the Nomex fibers were oriented parallel to the plane of the felt although some out-of-plane orientations were achieved.

2.7 FABRICATION OF SPECIMENS FOR DELIVERY TO NASA

Thirteen 15-cm^2 felt specimens were fabricated for delivery to NASA (Table 2-14 and Figure 2-23). Six of the specimens were made from Kevlar fibers on the Rando machine at Clemson University and were taken from roll No. 8 described in Table 2-8. Seven of the specimens were made from Nomex fibers (and a small amount of Kevlar pulp) in the liquid deposition column at Hughes Aircraft Company. The material and process parameters for these latter specimens were identical to those used for specimen No. 7 described in Table 2-13. Approximately one-half of each of the above two types of deposited

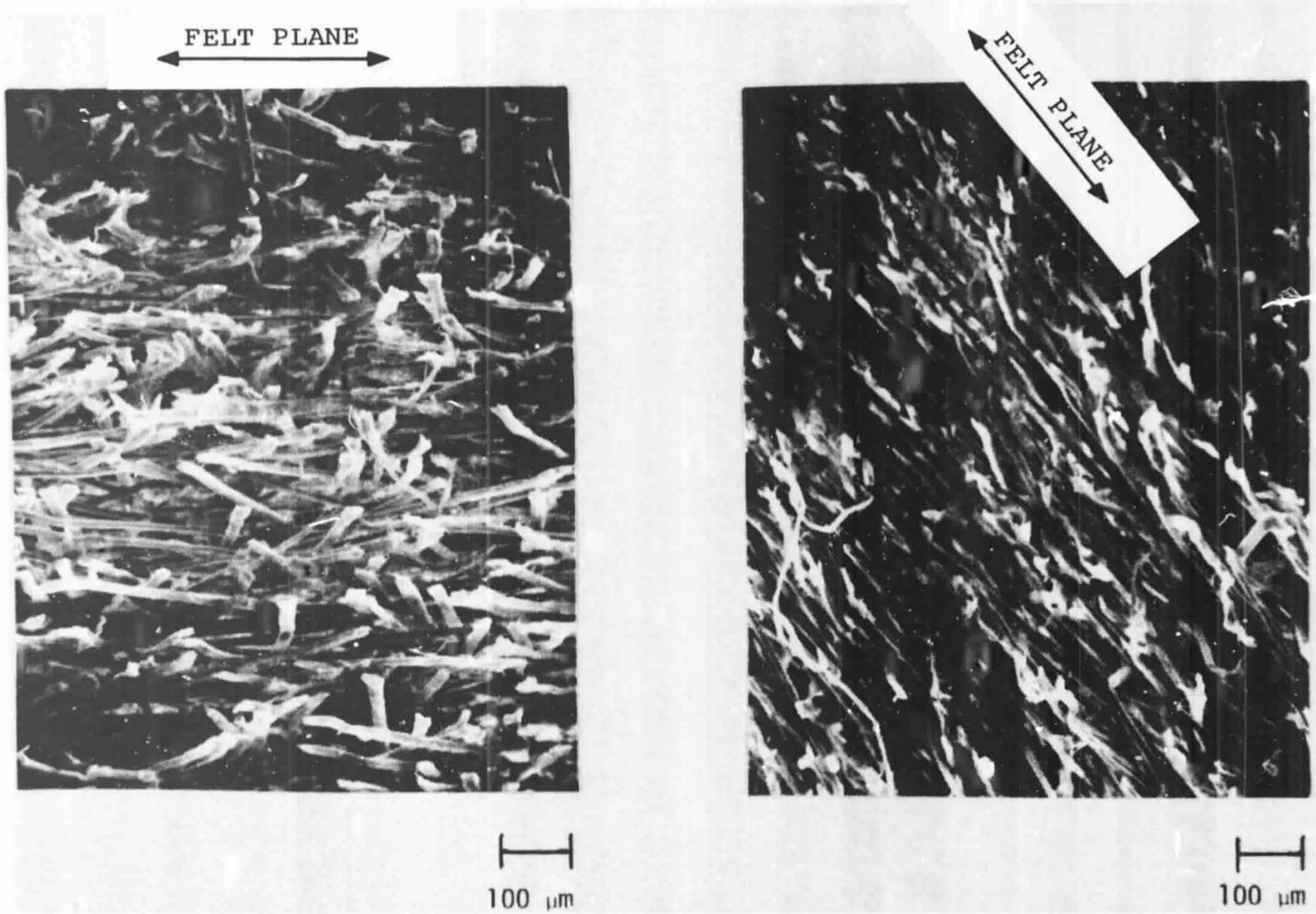

Figure 2-22. Scanning electron micrographs of specimen No. 7 showing orientations of the Nomex fibers.

TABLE 2-14. SPECIMENS FOR DELIVERY TO NASA

Specimen No.	Fiber Deposition*	Fiber Types*	Impregnant	Thickness, cm	Bulk Density, gm/cm^3
RP-1	Rando Machine	Kevlar	HR615P polyimide	0.343	0.061
RP-2	Rando Machine	Kevlar	HR615P polyimide	0.318	0.063
RP-3	Rando Machine	Kevlar	HR615P polyimide	0.318	0.062
RE-1	Rando Machine	Kevlar	828/TETA epoxy	0.292	0.087
RE-2	Rando Machine	Kevlar	828/TETA epoxy	0.340	0.065
RE-3	Rando Machine	Kevlar	828/TETA epoxy	0.356	0.076
LP-1	Liquid Deposition	Nomex	HR615P polyimide	0.244	0.127
LP-2	Liquid Deposition	Nomex	HR615P polyimide	0.249	0.139
LP-3	Liquid Deposition	Nomex	HR615P polyimide	0.254	0.167
LP-4	Liquid Deposition	Nomex	HR615P polyimide	0.257	0.148
LE-1	Liquid Deposition	Nomex	828/TETA epoxy	0.241	0.279
LE-2	Liquid Deposition	Nomex	828/TETA epoxy	0.279	0.139
LE-3	Liquid Deposition	Nomex	828/TETA epoxy	0.272	0.164

*Rando machine material and process details: Table 2-8, Roll No. 8.
Liquid deposition material and process details: Table 2-13, Specimen No. 7.

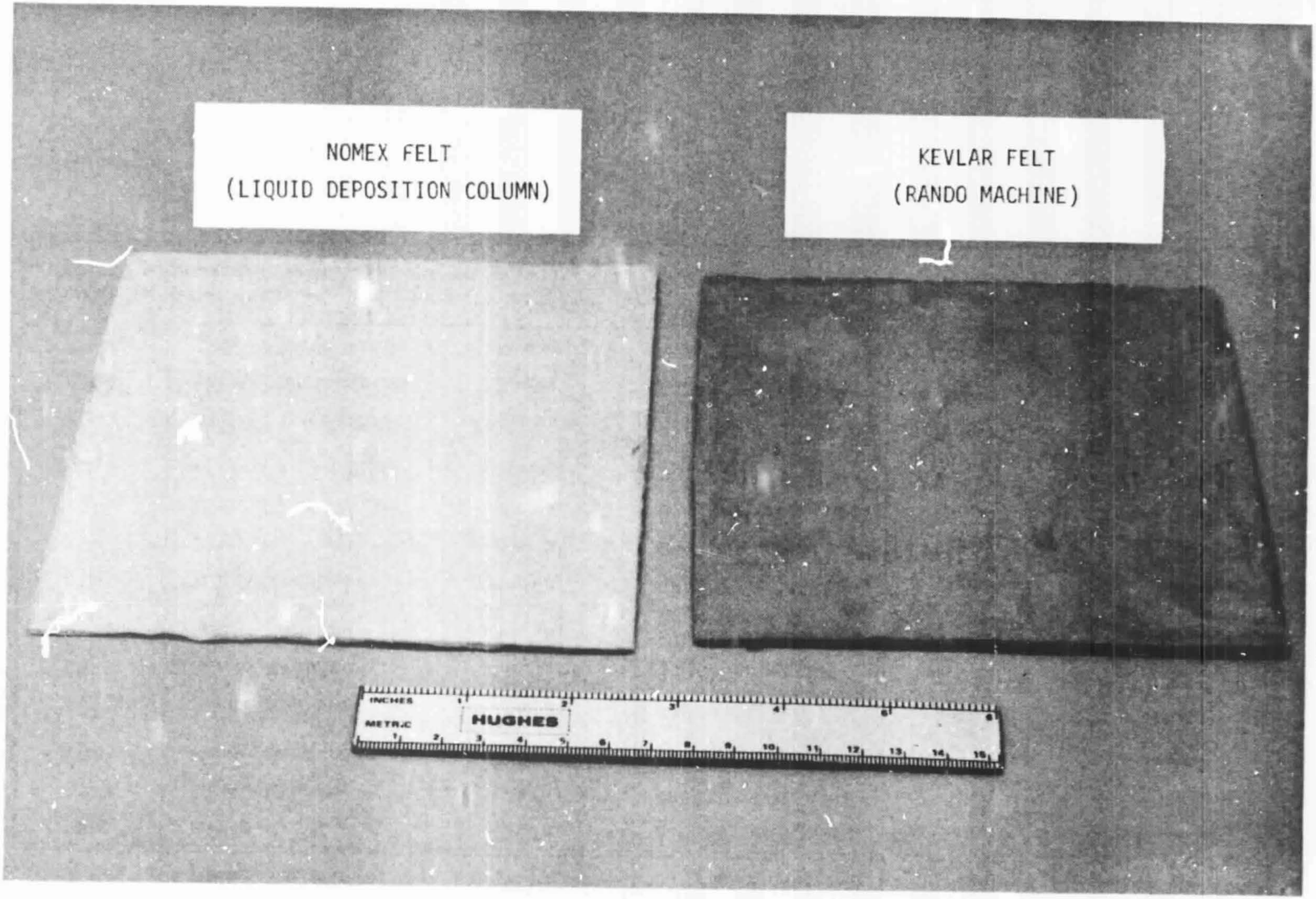

Figure 2-23. Examples of felt specimens delivered to NASA.

felt specimens were infiltration-bonded with HR615P polyimide resin, to provide the high-temperature resistance needed for SIP material. The remaining specimens were bonded with 828/TETA epoxy to provide improved fiber-to-matrix bonding relative to that achieved with the polyimide resin. (The resin infiltration procedures were described in Section 2.4.2.)

Scanning electron micrographs of one of the Rando machine specimens fabricated for delivery to NASA are shown in Figure 2-24. The predominantly layered orientations of the Kevlar fibers in this specimen can be seen. The microstructure of this specimen is representative of the Rando machine specimens that were made. (Scanning electron micrographs of a felt similar to the liquid-deposited Nomex materials delivered to NASA were shown in Figure 2-22.)

Present plans are for through-thickness tensile tests to be conducted on the delivered specimens at the NASA-Ames Research Center. Because of the predominantly layered orientations of the fibers in the Rando specimens, the test results will probably show that they do not meet the SIP strength requirements outlined in Table 2-1. The liquid-deposited Nomex specimens should have higher through-thickness strengths than the Rando specimens because of the presence of some out-of-plane fibers in the Nomex specimens. The epoxy-bonded specimens should be stronger than the polyimide-bonded specimens because of the greater fiber-to-matrix adhesion achieved in the epoxy-bonded materials (Section 2.4.2).

2.8 ADVANCED APPROACHES

Two new approaches have been proposed to increase the through-thickness strength of the felts. A new type of compactor for Rando matts has been developed by the Rando Machine Corporation. This machine compacts the matts at an acute angle and causes the fibers to become entangled in multiple directions rather than along parallel shingle planes. Such compaction could provide the isotropic constructions being sought. The first compactor of this type was scheduled to be installed on the 1-meter (40-inch) Rando machine at

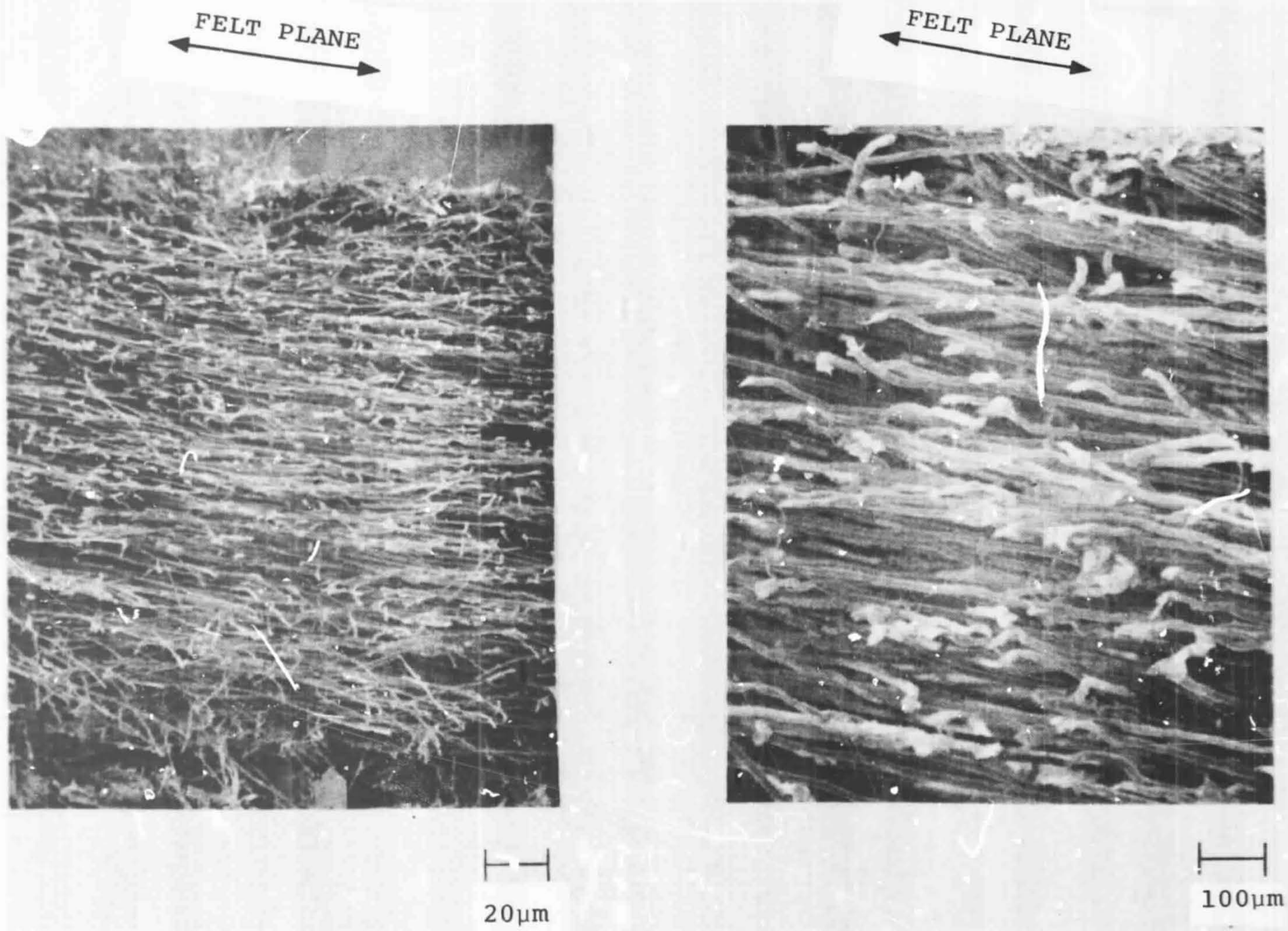

Figure 2-24. Scanning electron micrographs of Rando machine specimen No. RP-3 showing predominantly layered orientations of the Kevlar fibers.

the Fram Corporation, Providence, Rhode Island, in late 1982 and therefore was not available for use under this program. We recommend that use of this new equipment be explored to make improved SIP materials when the equipment becomes operational.

The other new approach was to incorporate short fibers into Rando matts by use of electrostatic flocking. This would result in felts having both in-plane fibers (laid down by the Rando process) and through-thickness fibers (introduced by flocking). Microfibres, Inc., Pawtucket, Rhode Island, was recommended by Clemson University as having a leading capability to do such flocking and attempted some initial experiments late in the program. Approximately 0.5 m^2 of the 2.9 x 10^2 gm/m^2 Nomex-fiber Rando matt described in Section 2.5 was shipped to Microfibres along with samples of various chopped Nomex fibers. Microfibres succeeded in flocking 0.079 and 0.16 cm (1/32 and 1/16 inch) Nomex fibers part way into the matt, but these fibers were too fine (2.2 Decitex = 2.0 denier) to penetrate very far into the matt before being bent over. Flocking attempts were then attempted using coarser Trilobal nylon (19 Decitex, 18 denier), cut to 0.48 cm (0.18 inch) lengths. These coarser fibers penetrated into approximately 1/3 of the matt thickness. Flocking was done on both sides of the matt, resulting in a specimen containing nylon fibers in the outer two thirds of its thickness but not in the center third.

The above specimens were shipped to Hughes Aircraft Company where they were compacted and infiltration-bonded with 828/TETA epoxy resin, yielding the felts described in Table 2-15. A scanning electron micrograph of specimen F-3 is shown in Figure 2-25 and shows that the nylon fibers penetrated deeply into the matt. These preliminary experiments showed that flocking of Rando matts is a promising approach for development of advanced SIP materials.

TABLE 2-15. RANDO MATT FILLED WITH ADDITIONAL FIBERS BY ELECTROSTATIC FLOCKING

Specimen No.	Fibers in Rando Matt	Chopped Fibers Added by Flocking	Impregnant	Thickness, cm	Bulk Density, cm/gm^3
F-1	3.8-cm crimped Nomex	0.079-cm Nomex	828/TETA epoxy	0.401	0.182
F-2	3.8-cm crimped Nomex	0.16-cm Nomex	828/TETA epoxy	0.401	0.169
F-3	3.8-cm crimped Nomex	0.48-cm nylon	828/TETA epoxy	0.401	0.193

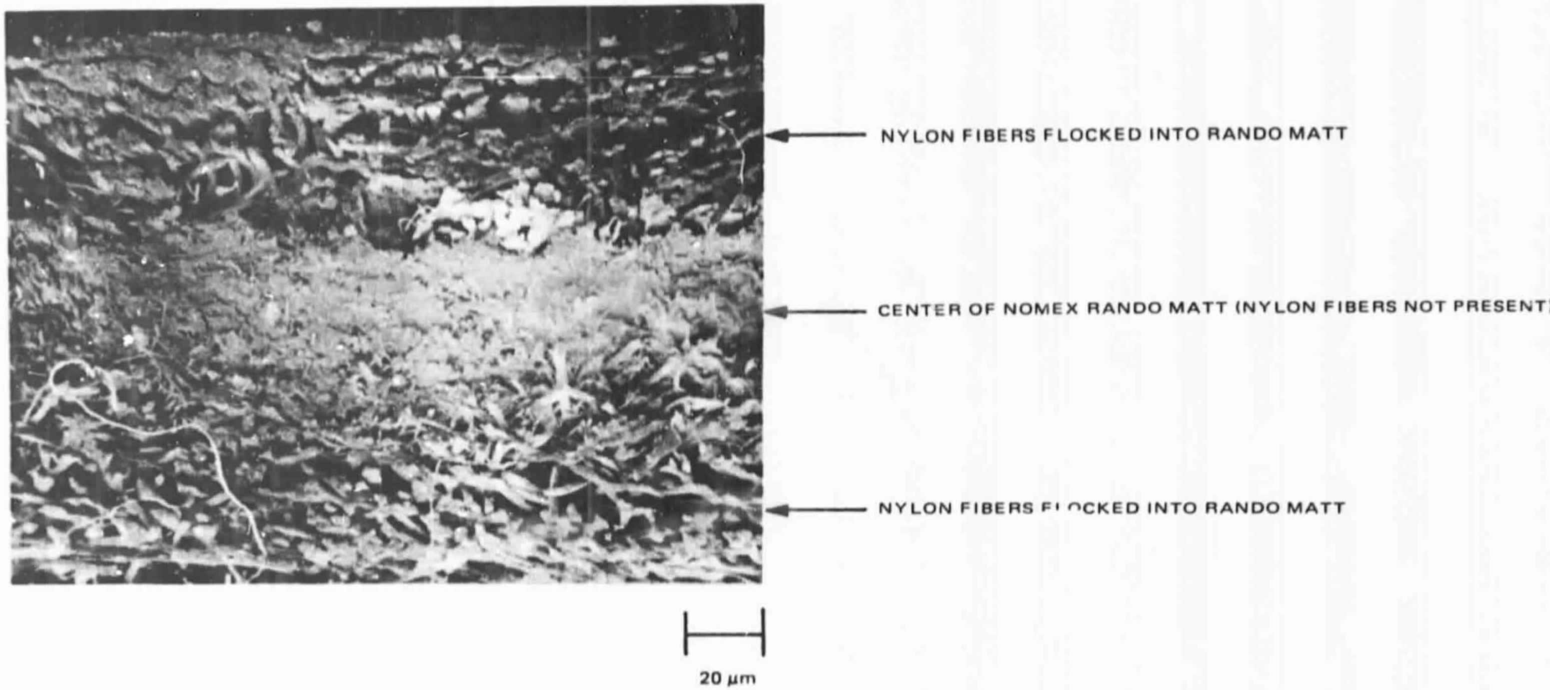

Figure 2-25. Scanning electron micrograph of Rando matt filled with additional fibers by electrostatic flocking.

3.0 CONCLUSIONS

The feasibility of utilizing air-lay and liquid-lay felt deposition techniques to fabricate strain-isolation-pad materials for the Space Shuttle Orbiter was demonstrated. With one significant exception, both of these fiber techniques, followed by resin bonding, produced felts having constructions appropriate for SIP materials. The exception was the fact that neither fiber deposition technique appeared to produce felts having a sufficient number of fibers oriented in the through-thickness direction (or at acute angles to this direction) to provide the needed through-thickness tensile strength. Instead, most of the fibers tended to be oriented in or close to the plane of the felt.

Two approaches that show promise for providing the needed high content of out-of-plane fibers are: (1) use of a newly developed angular compacting machine for Rando matts, and (2) incorporation of additional fibers into Rando matts by electrostatic flocking.

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