GEORGIA INSTITUTE OF TECHNOLOGY — OFFICE OF CONTRACT ADMINISTRATION

PROJECT ADMINISTRATION DATA SHEET

[x] ORIGINAL [ ] REVISION NO. ____

Project No. E-27-633 GTRI/~~GIT~~ DATE 8/17/84

Project Director: Dr. F. L. Cook School/~~XXX~~ Textile Engineering

Sponsor: DOE, Oak Ridge, Tenn.

Type Agreement: Grant No. DE-FG05-84CE40702

Award Period: From 8/1/84 To 7/31/87 (Performance) 10/29/86 (Reports)

Sponsor Amount:	This Change	Total to Date
Estimated:	$ 225,000	$ 542,131
Funded:	$ 225,000	$ 225,000 (Through 7/31/85)

Cost Sharing Amount: $ 9,035 Cost Sharing No: E-27-324

Title: Applications of Chemicals to Substrates Without the Use of Liquids: Solid-on-Solid Processing

ADMINISTRATIVE DATA

OCA Contact Brian J. Lindberg x-4820

1) Sponsor Technical Contact:

Dr. R. G. Massey
Conservation and Renewal
Energy Industrial Programs, CE-12
U.S. DOE
Washington, D. C. 20585
(202) 252-2079

2) Sponsor Admin/Contractual Matters:

Ms. Joyce C. Carringer
Contract Management Branch
Procurement & Contracts Division
U. S. DOE
P. O. Box E
Oak Ridge, Tenn. 37831 (202) 576-7564

Defense Priority Rating: N/A Military Security Classification: N/A

(or) Company/Industrial Proprietary: N/A

RESTRICTIONS

See Attached N/A Supplemental Information Sheet for Additional Requirements.

Travel: Foreign travel must have prior approval — Contact OCA in each case. Domestic travel requires sponsor approval where total will exceed greater of $500 or 125% of approved proposal budget category.

Equipment: Title vests with GIT; However, none proposed.

COMMENTS:

I.D. #02.141.000.85.002

COPIES TO:

Project Director
Research Administrative Network
Research Property Management
Accounting
Procurement/EES Supply Services
Research Security Services
Reports Coordinator (OCA)
Research Communications (2)
GTRI
Library
Project File
Other I. Newton

FORM OCA 4:383

GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION

NOTICE OF PROJECT CLOSEOUT

Closeout Notice Date 03/20/91

Project No. E-27-633 Center No. R5810-0A0

Project Director COOK F L School/Lab TEXT ENGR

Sponsor US DEPT OF ENERGY/DOE OAK RIDGE - TN

Contract/Grant No. DE-FG05-84CE40702 Contract Entity GTRC

Prime Contract No.

Title APPLICATION CHEMICALS SUBSTRATES WITHOUT USE LIQUIDS:PROOF CONCEPTS POWDE

Effective Completion Date 890301 (Performance) 890601 (Reports)

Closeout Actions Required:	Y/N	Date Submitted
Final Invoice or Copy of Final Invoice	Y	
Final Report of Inventions and/or Subcontracts	Y	
Government Property Inventory & Related Certificate	Y	900315
Classified Material Certificate	N	
Release and Assignment	N	
Other	N	

CommentsSF 269, SF 270 AND SF 272 IN FINAL FORM REQUIRED TO CLOSE OUT THE FINANCIAL RECORDS.

Subproject Under Main Project No.

Continues Project No.

Distribution Required:

Project Director	Y
Administrative Network Representative	Y
GTRI Accounting/Grants and Contracts	Y
Procurement/Supply Services	Y
Research Property Managment	Y
Research Security Services	N
Reports Coordinator (OCA)	Y
GTRC	Y
Project File	Y
Other	N
	N

NOTE: ~~Final Patent Questionnaire sent to PDPI~~. Complete

E-27-633

U.S. DEPARTMENT OF ENERGY

FEDERAL ASSISTANCE PROGRAM/PROJECT STATUS REPORT

FORM EIA-459
(10/91)

FORM APPROVED
OMB No. 1905-0127

1. Program/Project Identification No.	2. Program/Project Title	3. Reporting Period
DE-FG05-04CE40702	APPLICATION OF CHEMICALS	8/1/84 through 6/30/84
4. Name and Address Dr. Fred L. Cook, School of Textile Engineering Georgia Institute of Technology Atlanta, Georgia 30332		5. Program/Project Start Date 8/1/84 6. Completion Date 7/31/86

7. Approach Changes

☒ None

8. Performance Variances, Accomplishments, or Problems

A complete literature search was conducted and evaluated. Support personnel were hired for the project, and potential members of the Industrial Working Group were defined, contacted and invited to join the project.

☐ None

9. Open Items

☒ None

10. Status Assessment and Forecast

Task I of the proposed List of Tasks has been accomplished, and Task II has begun. The project is then in-line with the proposed Schedule of Work.

☐ No Deviation from Plan is Expected

11. Description of Attachments

☒ None

12. Signature of Reviewer and Date	13. Signature of DOE Reviewing Representative and Date
3/10/85	

APPLICATION OF CHEMICALS TO SUBSTRATES WITHOUT THE USE OF LIQUIDS: SOLID-ON-SOLID PROCESSING

DOE Grant No. DE-FG05-84CE40702

Georgia Tech Project No. E-27-633

Technical Progress Report 1

August 1, 1984 - September 30, 1984

Submitted to the

DEPARTMENT OF ENERGY

by

Dr. Fred L. Cook
Principal Investigator

School of Textile Engineering
GEORGIA INSTITUTE OF TECHNOLOGY

Atlanta, Georgia 30332

December 6, 1984

APPLICATION OF CHEMICALS TO SUBSTRATES WITHOUT THE USE OF LIQUIDS: SOLID-ON-SOLID PROCESSING

Technical Progress Report No. 1

August 1, 1984 - September 30, 1984

The first two months of the project were devoted toward conducting a complete literature search in electrostatic chemical deposition processes and techniques for planer substrates, correlating the developed literature and studying the prior art for possible overlap with the current objectives.

Considerable patent and journal literature was found in the areas of xerography on paper and coatings on metal. Little prior art was discovered, however, in applying powdered chemicals via electrostatics to fabrics. Valuable information was gained from the search, though, in terms of general variables (particle size, size distribution, change characteristics, etc.) that are important in electrostatic deposition of chemical powders, and the myriad hardware configurations that have been utilized in applying powders to non-textile substrates. This background information will be utilized in determining the techniques to be utilized in the bench-scale trials with fabrics.

The support personnel on the Tech research team were finalized during the first month:

Research Associates

Ms. Elisha Edmiston, B.S. Chemist

Ms. Bronwen Kleissler, B.S. Clothing and Textiles, M.S. Textiles

Graduate Students

Ms. Karen Sudweeks, B.S. Chemist, working toward M.S. in Polymers
Mr. Gajanan Bhat, B.S. Textile Engineer, working toward M.S. in Textile Engineering.

Including the three professors on the project, the team now consists of seven (7) members.

Potential members of the DOE/Georgia Tech Industrial Working Group were contacted. After interest was expressed over the phone, an official invitation letter was sent (see Appendix 1) along with a copy of the proposal.

Plans for the 10/1/84 - 12/31/84 reporting period call for:

1. Obtaining samples of conventional, aqueous-applied resins and size formulations to determine film properties of currently-used materials.

2. Begin screening of chemical candidates.

3. Finalize the Industrial Working Group, and hold the first meeting at Georgia Tech (on November 8).

4. Contact equipment manufacturers and begin finalizing plans for system construction.

APPENDIX 1

Georgia Institute of Technology
A UNIT OF THE UNIVERSITY SYSTEM OF GEORGIA
ATLANTA, GEORGIA 30332
SCHOOL OF TEXTILE ENGINEERING
(404) 894-2490

September 26, 1984

Dr. Joe A. Mann
Corporate Director
Research and Development
Burlington Industries, Inc.
P.O. Box 21327
Greensboro, North Carolina 27420

Dear Dr. Mann:

Thank you for your interest in serving as a member of the Industrial Working Group for our Department of Energy study entitled "Application of Chemicals to Substrates Without Use of Liquids: Solid-on-Solid Processing." The enclosed proposal details the objectives and scope of the project.

In synopsis, the idea is to eliminate liquids completely in applying certain chemicals to textiles. Currently, colorants in conjunction with binders are being applied to metals and paper via electrostatics. Viewing textiles as another class of planer substrate, we feel that the "next step" forward from foam, film and spray applications is solid-on-solid processing.

The initial meeting of the Industrial Working Group is tentatively scheduled for the week of November 5. Further details will be forthcoming over the next month. We look forward to your acceptance of our invitation.

Sincerely,

Fred

Dr. Fred L. Cook
Associate Professor
Principal Investigator

FLC/ms

cc: Dr. Tincher
Dr. Carr
Mr. Massey

E-27-633

U.S. DEPARTMENT OF ENERGY

FEDERAL ASSISTANCE PROGRAM/PROJECT STATUS REPORT

FORM EIA-459 (10-83)

FORM APPROVED OMB No. 1900-0127

1. Program/Project Identification No.	2. Program/Project Title	3. Reporting Period
DE-FG05-84CE40702	APPLICATION OF CHEMICALS	10/1/84 through 12/31/85
4. Name and Address: Dr. Fred L. Cook, School of Textile Engineering, Georgia Institute of Technology, Atlanta, Georgia 30332		5. Program/Project Start Date: 8/1/84
		6. Completion Date: 7/31/86

7. Approach Changes

Three powder application modes have been selected:

1. Electrostatic spray gun
2. Fluidized bed (electrostatic, nonelectrostatic)
3. Selenium drum transfer (modified xerography)

☐ None

8. Performance Variances, Accomplishments, or Problems

Candidate resins, finishes and sizes were defined and secured, as were commercial "standard" formulations for film comparison purposes. Screening tests and film property evaluations were begun. Nordsen Corp., Electrostatic Corp. and Eastman Kodak were contacted for equipment coordination, with the former committing to systems. Design of auxiliary equipment was completed, and components ordered. The Industrial Working Group was formed, and the first meeting held on November 8, 1984.

☐ None

9. Open Items

☒ None

10. Status Assessment and Forecast

The project is progressing on schedule, with the exception of bench-scale equipment set-up. With the decision to work with the three equipment manufacturers came delays in obtaining corporate approval, as well as equipment isolation problems.

☐ No Deviation from Plan is Expected

11. Description of Attachments

☒ None

12. Signature of Recipient and Date: Fred L Cook 3/10/86

13. Signature of DOE Reviewing Representative and Date

APPLICATION OF CHEMICALS TO SUBSTRATES WITHOUT THE USE OF LIQUIDS: SOLID-ON-SOLID PROCESSING

DOE Grant No. DE-FG05-84CE40702

Georgia Tech Project No. E-27-633

Technical Progress Report 2

October 1, 1984 - December 31, 1984

Submitted to the

DEPARTMENT OF ENERGY

by

Dr. Fred L. Cook
Principal Investigator

School of Textile Engineering
GEORGIA INSTITUTE OF TECHNOLOGY

Atlanta, Georgia 30332

March 10, 1984

APPLICATION OF CHEMICALS TO SUBSTRATES WITHOUT THE USE OF LIQUIDS: SOLID-ON-SOLID PROCESSING

Technical Progress Report No. 2

October 1, 1984 - December 31, 1984

Companies were contacted, and typical resin formulations for pigment printing were obtained. Applied as emulsions, these resins were of two generic types: polyacrylates and styrene-butadiene rubbers. The films visually and aesthetically were similar to Saran® Wrap, with the following characteristics:

1. Extremely clear, with little distortion.
2. Flimsy, very flexible and "leathery."
3. Clingy, having a tendency to collapse upon itself, and the folds to tenaciously stick together.

The latter characteristic made it extremely difficult to remove the film from the glass after laying it with a Gardner knife. New techniques will have to be developed before formal testing of the films can be accomplished.

A number of resins were obtained for screening during the reporting period, including the following generic types:

1. Modified cellulose ester
2. PVC and PVC/PVA copolymers
3. Modified Polyester
4. Epoxy-Urethane Copolymers

Resin press-outs to obtain films were begun with a Rucker PHI electrically heated, hydraulic ram platen press. Due to the extremely high viscosities of the resin melts and the rapid cool-down characteristics, laying a thin film from the melt with a Gardner knife was extremely difficult. Further technique development will be necessary to achieve a uniform film of the required thickness.

In the finishing area, softeners and water/stain repellents were defined as areas of interest. Oxidized polyethylenes have made a significant impact as textile softeners. The oxidation is effected solely for emulsification purposes, as the polyhydrocarbon is sold to the industry as an emulsion. An unoxidized polyethylene, Microthene, was isolated as a powdered candidate that should have good softener properties, be applicable through SOS and give higher UV stability (and resistance to brittleness) than the oxidized analog.

The market was dicouragingly void of powdered fluoropolymer stain repellents, with the commercial materials supplied as in-situ polymerized emulsions. A segmental poly(fluoropolymer-ethylene) material was obtained in powder form from Allied Corp., and an emulsion with a procedure for isolating the polymer from 3M Corp. The former has traditionally been used to coat metal articles (with claimed electrostatic deposition), while the latter has been used as a water/stain repellent on textile upholstery fabrics.

Several size candidates for SOS slashing were identified and secured. The most promising candidates were complex acrylate copolymers currently being used as "hot melt" sizes. Traditional PVA-based sizes were largely thermosets, and thus could not be considered. However, materials were obtained to give "commercial" formulations of both PVA and starch-based sizes so that target film properties could be identified.

Several equipment manufacturers were identified and contacted. Two meetings were held with the Nordsen Co. of Amherst, Ohio. Nordsen has been a leader in applying dry powders to planer substrates (mainly metals plates and wires) using electrostatic spray guns. Powder is drawn

from a hopper and transported to the gun by compressed air. The individual particles are charged as they exit through the corona outside the gun. The part to be coated is grounded, producing a field between the gun and the part. The powder particles, attracted to the field, cling to the part until thermally cured. Advantages of the approach include:

- o excellent wrap-around and edge coverage
- o uniform coating thickness
- o controllable area of coverage

Powder recovery equipment is integral to the technology from an economic viewpoint.

Nordsen has had limited textile experience, and agreed to work with Georgia Tech as a research partner at no cost to the project. A temporary gun SOS system will be supplied to Textile Engineering during the next quarter, and a full-engineered, bench scale prototype will be supplied in April, 1985. The electrostatic gun approach has the greatest potential in application of finishes, solid-shade coloration and (possibly) yarn slashing, although the latter could present problems in around-yarn coverage with a single-side application.

The Electrostatic Equipment Corp. (EEC) of New Haven, Connecticut was also contacted. EEC has developed fluidized bed equipment for application of powdered coatings to substrates, with some research directed toward textiles (coatings for wet suits, binders for non wovens, etc.) The fluidized bed equipment can be run with charged or uncharged powder particles. The textile is passed through the fluidized powdered bed where particles attach to the fabric, followed by a thermal cure. EEC has a lab-scale unit available for testing. A trip is planned for next quarter to visit EEC and discuss equipment transfer for use on the project.

Specifically, slashing of yarns was considered the most promising candidate for SOS fluidized bed application, with finishing and solid

coloration also possible candidates. Advantages of the fluidized bed approach are both-side, uniform application. Disadvantages would be the volume of powder required to fluidize the bed and clean-up effort, both of which are minimized on long-run formulations (e.g., slashing and finishing, where the chemical mix rarely (if ever) changes composition).

Eastman Kodak Co. was contacted concerning both their resin and xerography technology. Eastman has provided multicolor xerography on paper, and with their resin/size technology, was an ideal choice to seek as a partner for the project. A xerography-type approach is considered the one with the highest probability of success in textile printing applications of SOS.

As expected, Eastman has real concerns about the proprietary nature of its xerography technology that must be addressed and satisfied before a decision can be reached. A decision will be reached during the next reporting period. Alternates such as Xerox Corp. and IBM will be approached if Eastman elects not to participate.

Designs were completed for auxiliary pieces of equipment such as take-up drives, winders, rolls and guides, and orders were placed for the components. All of the systems are expected to be completed during the next quarter.

The first meeting of the project's Industrial Advisory Board was held on November 8. The meeting was well-attended, and a number of fruitful suggestions were offered by the participants. A final listing of the members is enclosed (Appendix 1). The next meeting will be held in March, 1985.

APPENDIX I

INDUSTRIAL WORKING GROUP
DOE/GEORGIA TECH
SOLID ON SOLID PROCESSING

Fall/1984

INDUSTRIAL MEMBERS

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Market Development Specialist-Powder
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Mr. A. D. Cotney
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Mr. Robert Greene
Corporate Engineer
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Thomaston, Ga 30286
Telephone: (404) 647-7131, -6611

DOE MEMBER

Mr. Robert G. Massey
U.S. Dept. of Energy
Industrial Programs Office
Conservation and Solar Energy
CE12
1000 Independence Avenue
Washington, D.C. 20585
Telephone: (202) 252-2079

TEXTILE ENGINEERING PERSONNEL

	Telephone Nos.
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Dr. Wallace C. Carr	(404) 894-2538
Ms. Elisha Shephard	(404) 894-2490
Ms. Bronwen Kleissler	(404) 894-2490
Mr. Gajanan S. Bhatt	(404) 894-2490
Ms. Karen Sudweeks	(404) 894-2490

Common Address:

School of Textile Engineering
Georgia Institute of Technology
Atlanta, Georgia 30332

FORM EIA-459
(10/89)

FEDERAL ASSISTANCE PROGRAM/PROJECT STATUS REPORT

FORM APPROVED
OMB No. 1905-0127

1. Program/Project Identification No.	2. Program/Project Title	3. Reporting Period
DE-FG05-84CE40702	Application of Chemicals-----	1-1-85 through 3-31-85
4. Name and Address		5. Program/Project Start Date
Dr. Fred L. Cook, School of Textile Engineering Georgia Institute of Technology, Atlanta, GA 30332		8-1-84
		6. Completion Date 7-31-87

7. Approach Changes

☒ None

8. Performance Variances, Accomplishments, or Problems

The main activity during the reported period was the screening of numerous size, softener and resin binder candidates. Blends of Eastman WD (a modified polyester) and Carboset 525 (an acrylic) with low molecular weight modifiers gave the best size film performances. Eastman 252 (modified polyester) and Elvax 410 (a poly(ethylene-co-vinyl acetate)) appeared to have good binder properties. A polyethylene powder, microthene FN-500, was selected as a softener candidate.

☐ None

9. Open Items

☒ None

10. Status Assessment and Forecast

Since the concept is new, materials that "fit" the various applications are not readily apparent. Considerable materials development will be necessary before bench-scale testing is feasible. Equipment has been ordered, however, in anticipation of bench-scale testing.

☐ No Deviation from Plan is Expected

11. Description of Attachments

☒ None

12. Signature of Recipient and Date	13. Signature of DOE Reviewing Representative and Date
Fred L. Cook 10/27/87	

Georgia Institute of Technology
A UNIT OF THE UNIVERSITY SYSTEM OF GEORGIA
SCHOOL OF TEXTILE ENGINEERING
ATLANTA, GEORGIA 30332
(404) 894-2490

October 28, 1986

MEMORANDUM

TO: Ms. Marlena Clarke

FROM: Dr. Fred L. Cook FLC wwc

SUBJECT: Forms/Technical Progress Report on DOE
Grant No. DE-FG05-84CE40702

Enclosed are copies of FAP Project Status Report and Technical Report 3 for the project, covering the period January 1, to March 31, 1985, as well as DOE Form 1332.16.

You will be receiving formal submissions, of course, through GTRI within the next few weeks.

cc: Bob Massey
Albin Turbak

APPLICATION OF CHEMICALS TO SUBSTRATES WITHOUT THE USE OF LIQUIDS: SOLID-ON-SOLID PROCESSING

DOE GRANT NO. DE-FG05-84CE40702

Georgia Tech Project No. E-27-633

Technical Progress Report 3

January 1, 1985 - March 31, 1985

Submitted to the

DEPARTMENT OF ENERGY

by

Dr. Fred L. Cook
Principal Investigator

School of Textile Engineering
GEORGIA INSTITUTE OF TECHNOLOGY

Atlanta, Georgia 30332

October 24, 1986

APPLICATION OF CHEMICALS TO SUBSTRATES WITHOUT THE USE OF LIQUIDS: SOLID-ON-SOLID PROCESSING

Technical Progress Report No. 3

January 1, 1985 - March 31, 1985

A. RESIN BINDERS/FINISHES

Candidates for pigment binder formulations were hot pressed into films on a Rocker PHI platen press. Aluminum foil was substituted for the metal shim material of the press surface to minimize binder/metal adhesion. The compositions of the screened materials are located in Table 1, and the platen press conditions and film results in Table 2.

Attempts were also made to quantify the melting temperature and melt viscosity of the candidate resins. The melt viscosities were obtained by use of a Brookfield Model HBT viscometer (Spindle No. 298). The results are also included in Table 1.

Due to the high melt viscosities and high Tm's, attempted draw-downs with various Gardner Knife assemblies failed. Generally, the melts cooled too rapidly to draw the material into a film. The pool of resin accumulated in front of the knife gap and did not flow smoothly. The decision was thus made to use the platen press films for mechanical testing.

As detailed in Report 2, typical acrylic and poly(styrene-butadiene) formulations now used by the industry for pigment binding yield clear, filmsy, "leathery" films on Gardner Knife coating. The properties of the two materials, Hycar 26120 acrylic and an SBR latex, are detailed under "Base Binders" in Tables 1 and 2.

Based on the properties of the base resins and on the Table 1 and 2 data, the following resins were selected as candidates for film physical testing:

1. Eastman FA-252 - The terephthalate-based, modified polyester gave an extremely flexible film with excellent adhesion to the aluminum foil. The film was slightly "milky", but thinner,

clearer films were obtained with the help of a teflon-based release agent to facilitate removal of the film from the foil.

2. Elvax 410 - The 82% ethylene - 18% vinyl acetate copolymer gave elastic, clean films, with excellent hand. The material has good UV stability, and is "friable" enough to grind cryogenically.

Films of the Eastman FA-252, duPont Elvax 410 and B. F. Goodrich Hycar 26120 were mechanically tested on an Instron using 2" x 1" samples, a chart of speed of 2/min., a cross-head speed of 2"/min, and a 1.5 in. jaw gap. The test results are detailed in Table 3, and the averages compared in Table 4. Satisfactory films of the SBR material for testing could not be obtained due to a high static build-up resulting in self-collapse on attempted removal from the glass. Anti-static spray, release agents, etc., did not improve the quality of films obtained.

The average results in Table 4 were influenced to some degree by the differences in sample thickness, reflecting the different film-producing techniques and the adhesive character of the two solid resins. The FA-252 film, approximately twice as thick as the Hycar 26120 and Elvax 410 films, was thus considerably stronger and gave a higher percent elongation at break. The extra thickness thus contributed to the ability to withstand heavier loads at higher elongations. The three films were relatively weak, but with a high elongation-to-break.

An attempt was made to melt-blend and/or dry blend several materials to improve properties (Table 5). Analysis of the heated platen-pressed films, however, showed no advantages for the blends over the virgin FA-252 and Elvax 410 films. The Hercolyn D and Abitol were not themselves resins, but lower molecular weight property modifiers.

U.S. Industrie's Microthene LDPE was selected as the material with the highest potential as a softener (Tables 1 and 2). The FN-500 is a fine-particle, powdered version of the same material. The Microthene's mechanical properties are contained in Table 6, and the average of the results are included in Table 4.

Several powdered candidates were isolated for other finishing applications:

1. 3M's FC - 258 - An aromatic polyester, this material imparts water-absorbent and stain-release properties to 100% polyester

fabrics. The finish has a Tm = 175°C, and gave a brittle, slightly discolored film on press-out. Application is at low levels (0.2-0.4% owf), however.

2. 3M's FC-214 - A flexible fluoropolymer, this material is supplied as an emulsion to the industry for imparting stain-reppellent/water-repellent properties to textiles. As instructed by 3M, the solid polymer was separated from the emulsion by addition of ethanol, filtered and dried to give a tan, smooth-flowing, grindable powder (Tm = 100°C).
3. 3M's FC-247 - Another suggested fluoropolymer from 3M, this polymer was not isolatable from emulsion with ethanol.
4. Halar - An Allied Chemicals product, Halar was found to be a poly(ethylene-co-chlorotrifluoroethylene) material, and thus was not suitable as a stain repellent. The ethylene component actually enhances oil penetration.

A bench-scale electrostatic powder spray gun system was obtained from Nordsen Co., and samples of polyester/cotton and 100% polyester fabrics were sprayed with FN-500 and FA-252. A dye, Disperse Blue 56, was used as a tracer. A batch oven was used for curing. The powders did not fluidize well, with the FN-500 exhibiting special difficulty in fluidizing. Several conclusions were drawn from these early trials:

1. A mounting system needed to be developed to keep the fabric taut and unwrinkled. A wooden embroidery hoop was selected as a mounting frame that would satisfy the requirements, and withstand the oven temperatures.

2. The poor flowability was attributed to the uncontrolled environment. The trials emphasized the need to carefully control temperature/humidity in the spray-room environment on a 24-hour basis. Otherwise, powder clumping was likely, inhibiting fluidity.

B. SIZE FORMULATIONS

A number of size candidates were evaluated for melting point and melt viscosity characteristics. The Tm's and physical characteristics are contained in Table 7.

Heated platen-press films were obtained for the candidates in the "as-supplied" state. The results are contained in Table 8.

Based on the performance characteristics, three major candidates were identified:
Eastman WD, Carboset 525 and Vinac ASB-576. Some candidates, eg., the Elvax series, were eliminated because of the lack of film solubility even with base. An easily desized material is necessary for slashing.

Due to extremely high melt viscosities, modifiers were sought to improve melt flow and spreadability/adhesion. The characteristics of the screened modifiers are detailed in Table 9.

The modifiers were melt-blended with the resin materials by combining in a beaker, melting, stirring thoroughly, pouring onto aluminum foil and peeling. The characteristics of the melt blends and the isolated solids are contained in Table 10.

A melting point study was conducted on the blended materials, as exhibited in Table II. Further analyses of the blends by temperature range were conducted.

Once the blends were characterized, the hot platen press was utilized to generate films (Table 12). Several of the blends produced excellent films with high adhesive character.

The lowest visual viscosity was exhibited by the 60% Eastman WD/ 40% benzoic acid blend. The solidified blends were all very brittle and hard, indicating good grindability. All the Eastman WD and PVA blends showed excellent adhesion to aluminum foil. In the press-out early trials, Samples Number 405-24-B, 405-25-B and 405-25-C of Table 12 looked especially interesting.

Table 1.

Physical/Chemical Characteristics of Screened Binder Materials

Trade Name	Chemical Type	Manufacturers	T_g (°C)	T_m (°C)	Melt Viscosity (cps)
Resin Binders					
Geon 142	PVC	B. F. Goodrich	-	200°C (Melt/Degrade)	Decomposed
Geon 141	PVC (greater inh)	B. F. Goodrich	-	198 (Melt/Degrade)	Decomposed
VROH	Poly (VC-VAC-Acrylate)	Union Carbide	65	113	
VMCH	Poly (86% VC 13% VAC - 1% Maleic Acid)	Union Carbide	74	147	Decomposed
VMCC	Poly (83% VC 16% VAC - 1% Maleic Acid)	Union Carbide	72	125	Decomposed
VAGH	Poly (90% VC - 4% VAC), W/2.3% - OH	Union Carbide	138		
VAC 1315	PVC				
Satlink 102	Acrylamidomethyl-Substituted Cellulose Ester	A.E. Staley	95	128	
FA 252	Modified, Terephthalate-based Polyester	Eastman	-	110 125	$4.8 \cdot 10^6$ (136°C) $1.4 \cdot 10^5$ (210°C)
DER 662	Epoxy	Dow Corning			
Elvax 410	Poly (82% Ethylene - 18% VAC)	duPont	-	78-83	$6.28 \cdot 10^6$ (95°C)
Elvax 310	Poly (75% Ethylene - 25% VAC)	duPont	-	72	$9.28 \cdot 10^6$ (95°C)
MU-760	Poly (82% Ethylene-18% VAC)	USI	-		$2.01 \cdot 10^6$ (150°C)
FE-532	Poly (91% Ethylene-9 % VAC)	USI	-	96	
Base Binders					
HYCAR 26120	Complex Acrylic	B. F. Goodrich	-	Degraded	N/A
SBR Latex	SBR	? (William Carter Mills)	-	Degraded	N/A

Softeners

Microthene MN 710-20	LDPE	USI	-	104	$3.14 \cdot 10^6$ (136°C)
FN 500	LDPE	USI	-	102	$5.76 \cdot 10^6$ (136°C)
Valchem Wax	Oxid PE	Valchem	-	97	-
PE-190	PE	American Hoechst	-	121	-
PE-520	PE	American Hoechst	-	105	-
CP9B	PE	Durachem		117	-
M-160	Wax (Petro.)	Sun		65	-

Table 2.

Analysis of Platen Pressed Films

Trade Name	Top Roll Temp. (°C)	Lower Roll Temp. (°C)	Time (min)	Results/ Observations
Resin Binders				
Geon 142	96	93	1.5	Translucent "donut" at center
Geon 142	127	102	2	Larger, ruffled donut
Geon 142	152	119	3	Nice, fused circle, semi-rigid
Geon 142	179	142	2	Circle, pinkish tint
Geon 142	182	149	1.5	Circle, blue/purple tint
Geon 142	166	143	1.5	Crazed circle, slight amber tint
Geon 141	132	127	1.5	Crazed circle, semi-rigid
Geon 141	171	149	1.5	Cracked circle
VROH	149	138	1.5	100% fused circle, good metal adhesion
VROH	96	93	1	Irregular shapes
VMCH	96	93		Donut in center
VMCH	96	93	1.5	Donut in center
VMCH	127	104	1.5	100% fused circle, clean somewhat flexible
VMCC	110	93	1	100% fused, circle, good adhesion, very clear, fairly flexible
VAGH	101	93	1	No fusion evident
VAGH	101	93	2	Clear, fused circle
VAGH	101	93	1.5	Clear, fused circle
VAGH	147	96	1.5	/100% fused circle
VAGH	126	109	1.5	Yellowish tinted circle
VCA 1315	135	114	1.5	Cracked circle, no real fusion
VCA 1315	152	129	1.5	Fused circle, some cracks
VCA 1315	177	149	1.5	100% fused circle

Table 2. (Cont.)

Analysis of Platen Pressed Films

Trade Name	Top Roll Temp. (°C)	Both Rolls Temp. (°C)	Lower Roll Temp. (°C)	Time (min)	Results/ Observations
Starlink 102		133	1.0		Cloudy, very brittle, good flow
FA-252	104		93	1.5	No Fusion
FA-252	138		116	1.5	Clear, stuck tenaciously to foil
DER 662	93		93	1.5	To thin, brittle to remove
Elvax 410		80	1.0		Very flexible, removable from foil, waxy feel
		75	1.0		
Elvax					Clear, very flexible, waxy, less flow
Base Binders					
Hycar 26120	-		-	-	Draw-down of 50% solids emulsion clear, very flexible (like Saran Wrap[c])
SBR Latex	-		-	-	Drawdown clear, very elastic. flexible (like Saran Wrap[c])
Softeners					
Microthene MW 710-20	93		93	1.5	No fusion
Microthene MW 710-20	121		104	1.5	Good film, clear, very flexible
FN 500	115		104	1.5	Good film, clear flexible
Valchem Way	104		93	1.5	Very thin, milky
PE-190		126	1.0		Translucent, brittle
PE502		100	1.0		Translucent, very brittle
CP9B					Whitish, opaque, very brittle
M-160		100	2.0		Too waxy, yellowish tint, padded and spread

Table 3

FILM MECHANICAL PROPERTIES

ELVAX 410	SAMPLE THICK (MIL)	LOAD AT BREAK (LBS)	TENSILE ST. AT BREAK (LBS/IN^2)	% ELONG. AT BREAK	COMMENTS
1	4.8	1.05	218.56	275.00	
2	5.4	1.16	214.95	216.67	
3	5.6	1.37	243.94	512.50	JAW BREAK
4	3.4	0.66	194.33	104.17	
5	4.6	0.97	211.57	158.33	
6	4.0	0.84	209.26	116.67	JAW BREAK
7	4.8	1.03	214.84	337.50	JAW BREAK
8	4.6	1.03	223.21	154.10	JAW BREAK
9	6.2	1.14	183.61	225.00	
10	4.2	-	-	-	
AVERAGE	4.8	0.93	212.69	233.33	

Table 3 (cont.)

FILM MECHANICAL PROPERTIES

FA-252	(MIL) SAMPLE THICK.	(LBS) LOAD AT BREAK	(LB/IN^2) TENSILE ST. AT BK.	% ELONG. AT BK.	LOAD AT YIELD (LBS)	TENSILE ST. AT YIELD (LB/IN2)	% ELONG. AT YIELD	COMMENTS
21	7.2	12.9	1791.67	1010.4	10.5	1458.33	41.67	JAW BREAK
22	8.2	11.6	1414.63	239.58	12.6	1536.59	41.67	JAW BREAK
23	9.0	13.8	1533.33	666.67	13.3	1477.78	41.67	JAW BREAK
24	11.0	16.3	1481.82	593.75	15.5	1409.09	31.25	JAW BREAK
25	7.8	15.5	1987.18	1020.83	11.60	1487.18	41.67	JAW BREAK
26	6.4	12.3	1921.88	1010.42	9.7	1515.63	31.25	JAW BREAK
27	8.6	14.6	1697.67	802.08	12.5	1453.49	31.25	JAW BREAK
28	11.0	16.5	1500.00	927.08	15.0	1363.64	31.25	JAW BREAK
29	10.0	15.8	1580.00	1145.83	14.4	1440.00	41.67	SLIPPED OUT OF JAWS
30	13.2	17.05	1291.67	156.25	19.1	1446.97	31.25	JAW BREAK
31	7.8	12.7	1628.21	625.00	12.4	1589.74	41.67	JAW BREAK
32	9.4	13.0	1382.98	645.83	13.5	1436.17	31.25	
AVERAGE	9.1	14.3	1600.92	736.98	13.3	1467.88	36.25	

Table 3 (cont.)

FILM MECHANICAL PROPERTIES

HYCAR 26120	SAMPLE THICK (MIL)	LOAD AT BREAK (LBS)	TENSILE ST. AT BREAK (LBS/IN^2)	% ELONG. AT BREAK	COMMENTS
1	4.3	1.21	281.35	416.67	
2	4.0	0.69*	172.99	133.33	FILM DEFECT
3	3.8	1.02	269.62	208.33	
4	3.3	1.27	384.88	250.00	
5	3.7	1.23	331.81	237.50	
6	3.3	1.45	439.66	345.83	
7	3.7	1.10	298.62	162.50	
AVERAGE	3.7	1.21	334.32	270.14	

*DELETED FOR AVERAGE

Table 4

Average Film Mechanical Properties

	SAMPLE THICK. (MIL)	LOAD AT BREAK (LBS)	TENSILE ST. AT BK. (LB/IN^2)	% ELONG. AT BK.	LOAD AT YIELD (LBS)	TENSILE ST AT YIELD (LB/IN^2)	% ELONG. AT YIELD
MICROTHENE [a]	12.0	17.0	1413.31	674.40	14.4	1190.80	30.93
ELVAX 410 [a]	4.8	0.93	212.69	233.33			
HYCAR 26120 [b]	3.7	1.21	334.32	270.14			
FA-252 [a]	9.1	14.3	1600.92	736.98	13.3	1467.88	36.25

a. Platen - pressed films (melt)

b. Gardner - knife films (emulsion)

TABLE 5

BLENDS OF MATERIALS

COMPOSITION NO.	BLEND MELT	TECHNIQUE DRY	Materials used (% by weight) STALINK 102	MUROTHENE MN 710-20	VMCC	HERCOLYN D	ELVAX 410	ABITOL	FA-252
1	X		90	10					
2	X		60	40					
3		X	90	10					
4		X	60	40					
5		X		10	90				
6		X		20	80				
7		X		30	70				
8		X		40	60				
9		X	90			10			
10		X	90			20			
11		X							
12		X					10		90
13		X		10					
14		X			80		20		90

Table 6

FILM MECHANICAL PROPERTIES

MICROTHENE	(MIL) SAMPLE THICK.	(LBS) LOAD AT BREAK	(LB/IN^2) TENSILE ST. AT BK.	% ELONG. AT BK.	LOAD AT YIELD (LBS)	TENSILE ST. AT YIELD (LB/IN2)	% ELONG. AT YIELD	COMMENTS
11	10.6	12.7	1198.11	303.04	11.7	1103.77	18.00	JAW BREAK
12	11.8	13.9	1177.97	773.44	12.2	1033.90	31.25	
13	11.4	17.6	1543.86	N/A*	N/A*	N/A*	N/A*	CHART ON LATE
14	12.6	17.7	1404.76	593.75	16.0	1269.84	31.25	
15	12.8	15.5	1210.94	145.83	16.0	1250.00	31.25	5"/MIN CROSS HEAD
16	13.3	24.3	1827.07	843.75	17.5	1315.79	31.25	
17	11.8	19.7	1669.49	875.00	14.9	1262.71	31.25	
18	12.0	12.6	1050.00	156.25	13.3	1108.33	31.25	JAW BREAK
19	11.8	16.8	1423.73	479.17	14.2	1203.39	31.25	
20	11.8	19.2	1627.12	822.92	13.8	1169.49	41.67	
AVERAGE	12.0	17.0	1413.31	674.40	14.4	1190.80	30.93	

*NOT INCLUDED IN AVERAGE

TABLE 7
CHARACTERISTICS OF SIZE CANDIDATES

SAMPLE	TRIAL #	TM(°C)	COMMENTS
Carboset 525	1 2	100-110° 100-107°	Softened ~60°C, noted color change from white to clear at 70°C; good flow once melted.
Carboset 526	1 2	145-155° 147-153°	Samples appeared extruded into 1/8" sheet, cooled, then cracked to any size; softened 75-77°
Carboset XL-27	1 2 3	73-85°C 80-90°C 75-85°	Similar to other Carboset melts
Eastman WD	1 2	103-113°C 97-110°C 92-100°C	Samples appeared extruded in filaments, diameter then chopped to 1/8" length. Softened ,55°C Solidified within 5 seconds from removal of heat.
T-66	1 2	196-200°C 194-200°C	Noted color change for some particles 163-170°C. Poorest melting sample of all 11 size samples, degraded easily (205°C). Many particles degraded before melting
Elvax 310	1 2	70°C 73°C	Began with uniform 'rubbery' balls. Softened 65°C Excellent flow once melted
Elvax 410	1 2	83°C 78°C	Same as Elvax 310, but better flow at melt seen here.
Vinac B-15	1 2 3	75-80°C 80-85°C 77-85°C	Small round or flat (some of each) glass-like beads. Flat ones softened 50°C , wanted to retain shape although melted. (Wrong surface tension?)
Vinac B-25	1 2	90-95°C 88-95°C	Mostly round, glass-like beads. Softened 65°C.
Vinac B-100	1 2	75-80°C 75-77°C	Clearly 2 sizes of beads Large ones are 2x diameter of small ones. Small - glass-like spheres Large - milky white, with noted color change to clear 50-60°C
Vinac ASB-516	1 2	85-90°C 89-95°C	Range of diameters Softened 62-70°C

TABLE 8
FILM CHARACTERISTICS OF "AS-OBTAINED" SIZE MATERIALS

SAMPLE #	NAME	THERMOCOUPLE Reading, °C	PLATEN Top °C	PLATEN Bottom °F	TIME	COMMENTS
405-13-A	ELVAX 310	75	-	-	1 min Pressure=10	Seems like saran wrap type. Very flexible. Good clarity.
405-14-A	ELVAX 410	80	-	-	1 min Pressure=10	Also seems like saran, only does not stick to itself like ELVAX 310 Very flexible Good clarity
405-14-B	VINAC B-100	85	-	-	1 min Pressure=15	Good clarity. Flexes, but much stiffer than ELVAX.
405-14-C	VINAC B-100	85	-	-	1.5 min Pressure=15	Thinner, slight increase in flexibility
405-14-D	VINAC B-15	90	-	-	1 min Pressure= 12.5	Less flexible than VINAC B-100 Good clarity
405-14-E	VINAC B-25	95	-	-	1 min Pressure= 12.5	More flexible than VINAC B-15 Similar to B-100, yet seems tougher
405-14-F	VINAC ASB-516	100	225	210	1 min Pressure= 12.5	Much less flexible than B-15. This sample was brittle shattered. Good clarity
405-14-G	EASTMAN WD	99	235	213	1 min Pressure=10	Similar to VINAC's in flexibility, yet seems tougher (has not shattered yet).

TABLE 8 (cont.)

SAMPLE #	NAME	THERMOCOUPLE READING, °C	PLATEN TOP °F	PLATEN BOTTOM °F	TIME	COMMENT
405-14-H	EASTMAN WD	99°C	235	213	1 min Pressure=12.5	Much more flexible than 405-14-G
405-14-I	EASTMAN WD	110°C	245	222	1 min	Similar or slightly more flexible than 405-14-H
405-14-J	EASTMAN WD	110°C	245	222	1 min Pressure=12.5	Less flexible than either 405-14-H or I,but more flex than 405-14-G
4095-14-K	CARBOSET 526	155°C	330	290	1 min Pressure=10	Similar to polyvinyl acetates (VINAC) Good clarity
405-14-L	CARBOSET 525	110°C	250	210	1 min Pressure=10	good flexibility. A close second to ELVAX 410. Good clarity
405-14-M	CARBOSET XL-27	90°C	240	210	1 min Pressure=10	Very brittle, shattered, Good clarity
405-14-N	ELVANOL T66	200°C	425	367	1 min Pressure=10 15	All adhered too much to aluminum foil to remove and give property description
		205°C			1 min Pressure=15 20	

TABLE 9

CHARACTERISTICS OF SIZE MODIFIERS

PRODUCT TRADE NAME	MANUFACTURER SOURCE	COMMENT
Adipic Acid	Aldrich	Tm 152-154°C
Bebacic Acid	Aldrich	Tm 131-134°C
Benzoic Acid	Aldrich	Tm 122°C
Resoflex R-296	Cambridge Ind.	Polyester polymer with food & drug approval for postage stamps Liquid form (100% solids) To be used as plasticizer for for VINAC ASB-516.
Vinsol	Hercules	Bottoms from tree sap. Pulverized, flaked or saponified form avail.,to use as compatible resin with Vinac ASB-516.
Santolite MHP	Monsanto	Aryl sulfonamide formaldehyde resin solid. MW 1000 Good for adhesion, plasticizer, gloss.
Santicizer 141	Monsanto	Alkyl aryl phosphate liquid good as flame retardant, plasticizer.
Santicizer 160	Monsanto	Butyl benzyl phthalate liquid much cheaper, more commonly used plasticizer with poly vinyl acetates.
Carboset 515	BF Goodrich	Liquid used as plasticizer for Carboset 525.

TABLE 10.

CHARACTERISTICS OF SIZE/MODIFIER MELT BLENDS

SAMPLE	BLEND COMPOSITION	COMMENTS
405-17-A	60% Eastman WD 40% Adipic Acid	Melt was yellowish, but smooth and clear. Viscosity of thick oil Solidified rapidly to creamy white color; seems brittle
405-17-B	70% Eastman WD 30% Adipic Acid	Seemed to have lower viscosity than 405-17-A; however, solidified slower too. Creamy white solid brittle.
405-17-C	80% Eastman WD 20% Adipic Acid	Seems slightly more flexible than 405-17-B, but would still break with fingers.
405-18-A	60% Eastman WD 40% Benzoic Acid	Similar melt characteristics to 405-17-B, but had considerabler sublimation; solidified similar to 405-17-B, but final product tougher and more pliable.
405-18-B	80% Eastman WD 20% Sebacis Acid	Similar melt viscosity as Eastman WD/adipic acid; blends solidified a little slower, but seemed to form a 'smoother' looking solid, also more pliable than flaky (seen with adipic acid & benzoic acid)
405-18-C	70% Eastman WD 30% Sebacic Acid	Very much like 405-18-B, only more pliable and less adherance to aluminum foil
405-18-D	60% Eastman WD 40% Sebacic Acid	Tough, pliable, brittle & solidified quickly - weird
405-19-A	75% Carboset 525 25% Elvax 310	Required much higher temperature to melt and even then did not form a 'clear' melt. When solidified, somewhat pebbly or bumpy.

TABLE 10 (cont.)

405-23-A	95% Vinac ASB-516 5% Sanitizer 160	Very high viscosity at melt almost like rubber cement. Hardened quickly. Pulled good fibers from it.
405-23-B	90% vinac ASB-516 10% Sanitizer 160	Slightly lower melt viscosity than 405-23-A, but still very high. Formed excellent fibers. Still very brittle.
405-24-B	95% Vinac ASB-516 5% Santizer 141	Slightly less viscous than 405-23-A, but still a lot like rubber cement. Pulled fibers readily.
405-24-B	90% Vinac ASB-516 10% Santizer 141	Tougher than 405-24-A Seems more extensible, too.
405-25-A	95% Vinac ASB-516 5% Resoflex R296	Slight yellow color (Resoflex is yellowish). Similar properties to other Vinac blends.
405-25-B	90% Vinac ASB-516 10% Resoflex	Easier to break after cooling. Also easier to remove from beaker at melt.
405-25-C	95% Carboset 525 5% Carboset 515	Very viscous at melt Very tough after cooling not brittle like Vinac's.
405-25-D	90% Vinac ASB-516 10% Vinsol NVX	Brown color due to Vinsol. Degradation observed before good melt. Elastic, brittle.

TABLE 11
MELTING POINT AND MELT CHARACTERISTIC OF SIZE/MODIFIER BLEND

SAMPLE	TRIAL #	Tm ^{o}C	COMMENT
405-17-A 60% Eastman WD 40% Adipic Acid	1 2	118-130 118-125	Became rubbery/softened at 60°C. Wax-like at 90-120°C Milky-white to clear at 125-130°C.
405-17-B 70% Eastman WD 30% Adipic Acid	1 2	100-110 95-110	Became rubber like 65°C Acted like soft was at 75-80°C. Cloudy until 135°C. Self flow at 130°C.
405-17-C 80% Eastman WD 20% Adipic Acid	1 2	90-105 85-105	Rubber-like 60-65°C Soft wax at 75°C Self flow 125-128°C
405-18-A 60% Eastman WD 40% Benzoic Acid	1 2	95-105 95-105	Did not observe rubbery stage. Soft wax at 70°C Self flow at 107-109°C
405-18-B 80% Eastman WD 20% Sebacic Acid	1 2	95-105 90-105	Softened 50°C Wax-like 60-65°C Self flow at 110-113°C
405-18-C 70% Eastman WD 30% Sebacic Acid	1 2	97-110 99-110	Rubbery at 55-63°C Wax-like 75°C Self flow at 120°C
405-18-D 60% Eastman 40% Sebacic Acid	1 2	115-120 120-125	Rubbery 85-90°C Soft wax 97-100°C Self flow 127-128°C
405-19-A 75% Carboset 525 25% Elvax 310	1 2	80-90 70-85	Rubbery at 45°C No waxy stage No self flow - too viscous

TABLE 12
FILM FORMATION AND PROPERTIES OF SIZE/MODIFIER BLENDS

SAMPLE #	NAME	THERMOCOUPLE READING °C	PLATEN TOP °C	PLATEN BOTTOM °F	TIME	COMMENTS
405-18-A	60% Eastman WD 40% Benzoic Acid	110	250	230	1 min 10 pressure	Can't get foil apart; excellent glue Low viscosity, extended to all edges of foil.
405-17-C	80% Eastman WD 20% Adipic Acid	110	250	230	1 min 10 pressure	Same glue, higher viscosity.
405-18-A	75% Carboset 525 25% Elvax 310	110	250	230	1 min 10 pressure	Same as 405-17-C
405-19-A	75% Carboset 525 25% Elvax 310	90	-	-	1 min	Feels like wax paper. Slightly brittle.
405-24-A	95% Vinac ASB-516 5% Santicizer 141	100	-	-	1 min 12.5 pressure	Smoother feel than VINAC alone. Much better flexibility, but not enough to handle weaving.
405-24-B	90% Vinac ASB-516 10% Santicizer 141	100	-	-	1 min 12.5 pressure	Excellent flexibility. Tough enough (probably) to handle weaving. Does not stick to itself. Stretches easily, but remains stretched and deformed.
405-25-A	95% Vinac ASB-516 5% Resoflex R-296	100	-	-	1 min 12.5 pressure	Smoother feel than 405-24-A, but cracked in two. Seems tough.
405-235-B	90% Vinac ASB-516 10% Resoflex R-296	100	-	-	1 min 12.5 pressur	Good flexibility. Similar to 405-24-B Tough. Low extension
405-25-C	95% Carboset-525 5% Carboset-515	110	-	-	1 min 10 pressure	Excellent flexibility Very similar to 405-24-B Better slide - smoother.
405-25-D	90% Vincac ASB-516 10% Vinsol NVX	100	-	-	1 min 12.5 pressure	Brittle beyond belief. Brown wood-stained color.

E-21-633

U.S. DEPARTMENT OF ENERGY

FEDERAL ASSISTANCE PROGRAM/PROJECT STATUS REPORT

FORM EIA-459
FORM

FORM APPROVED
OMB No. 1900-0127

1. Program/Project Identification No.	2. Program/Project Title	3. Reporting Period
DE-FG05-84CE40702	APPLICATION OF CHEMICALS ---	4-1-86 through 6-30-86
4. Name and Address Dr. Fred L. Cook, School of Textile Engineering Georgia Institute of Technology, Atlatna, GA 30332		5. Program/Project Start Date 8-1-84
		6. Completion Date 7-31-86

7. Approach Changes

The project was focused on the key areas for large-scale trials and continued research: binding of nonwovens, fluoropolymer stain repellent finishing of nonwovens, yarn slashing, xerography fabric printing and 100% solids liquid coloration. Other peripheral research areas were downgraded in priority.

☐ None

8. Performance Variances, Accomplishments, or Problems

A nonwoven bound by solid-on-solid (SOS) application of powdered binder followed by thermal flowing gave properties similar to those of the plant standard. SOS-slashed yarns performed well in abrasion tests and fray count, comparable again to plant standards. Powder spray gun trials have begun on the polypropylene nonwovens with powdered fluoropolymer.

☐ None

9. Open Items

☒ None

10. Status Assessment and Forecast

Task 9 of the proposed List of Tasks has been accomplished, and Tasks 10 and 11 are partially completed. The project remains in-line with the proposed Schedule of Work.

☐ No Deviation from Plan is Expected

11. Description of Attachments

☐ None

12. Signature of Recipient and Date	13. Signature of DOE Reviewing Representative and Date
Fred L. Cook 7/2/86	

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TECHNICAL PROGRESS REPORT 4-7

APPLICATIONS OF CHEMICALS TO SUBSTRATES WITHOUT THE USE OF LIQUIDS: SOLID-ON-SOLID PROCESSING

DOE Grant No. DE-FG05-84CE40702

Georgia Tech Project No. E-27-633

Technical Progress Report 8

April 1, 1986 - June 30, 1986

Submitted to the

DEPARTMENT OF ENERGY

by

Dr. Fred L. Cook
Principal Investigator

School of Textile Engineering
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July 3, 1986

APPLICATIONS OF CHEMICALS TO SUBSTRATES WITHOUT
THE USE OF LIQUIDS: SOLID-ON-SOLID PROCESSING

Binding of Chicopee/J & J's Nonwoven

A carefully - controlled factorial experiment has been conducted on binding of the Chicopee/J&J 100% polyester nonwoven fabric through SOS processing. Further information on the nonwoven, slated for mattress pad cover application, is contained in the Appendix A letter. The Nordson continuous powder spray booth without electrostatics was utilized for the experiment, with Eastman FA 252 as the binder resin. The nonwoven fabrics were mounted under slight tension on wooden circular embroidery hoops, and were run through the Nordson unit in multiples on the belt at 100 ypm. Curing flow was effected by a batch oven.

The factorial matrices are shown in Tables 1 and 2. The code used is:

A1←Sample #1
4.6% ←— solids pick-up
oven T (°C)→160, 30" ←— seconds at temperature
sts: 1421 ←—breaking strength (psi)
stn: 0.40 ←—strain to break (dimensionless)
mod: 3857 ←—initial modulus (psi)
Mullen: 39.2 ←— tester burst presssure (lbs)

Figures 1 and 2 graphically show the differences in the stress/strain curves in machine (MD) and cross- machine (CD) directions of the nonwoven at the defined optimum SOS conditions (solid lines)

Sample No.: 05
Solids Pickup: 9.9%
Cure Temp: 200°C
Cure Time: 30 sec.

The dotted lines on the plots correspond to the plant - bound standard, produced in a wet pad-nip-tenter oven process (poly (vinyl acetate-co-acrylic) binder). The base weight of the plant - bound standard was 1.05 oz/yd^2 due to stretching in the wet pad/tenter clip process (down from 1.3 oz./yd^2 unbound). No stretching took place on the SOS studies (minimal tension on the fabrics), and thus the density differences in SOS - bound vs. plant - bound fabrics must be considered when viewing Figures 1-2.

TABLE 1

STRESS-STRAIN RESULTS FOR MULTI-VARIABLE CONDITIONS

This set of experiments was conducted on Chicopee nonwoven unbonded fabric, C-9462, sprayed with Eastman FA-252 binder powder. There were 4 levels of binder applied, three oven curing temperatures, and two dwell times evaluated. The control fabric was C-5522, finished at Benson, NC and sent to us. The blank fabric was C-9462, with no binder applied, but placed in a 200 C oven for 60 seconds.

MACHINE DIRECTION

A1	A2	A3	A4	A5	A6
4.6%	4.6%	4.6%	4.6%	4.6%	4.6%
160, 30"	160, 60"	180, 30"	180, 60"	200, 30"	200, 60"
sts: 1421	sts: 1443	sts: 1506	sts: 1379	sts: 1432	sts: 1467
stn: 0.40	stn: 0.45	stn: 0.48	stn: 0.45	stn: 0.45	stn: 0.40
mod: 3857	mod: 3714	mod: 5263	mod: 2813	mod: 4125	mod: 4333
M.b: 39.2	M.b: 33.9	M.b: 35.9	M.b: 32.8	M.b: 33.8	M.b: 39.6

B1	B2	B3	B4	B5	B6
6.1%	6.1%	6.1%	6.1%	6.1%	6.1%
160, 30"	160, 60"	180, 30"	180, 60"	200, 30"	200, 60"
sts: 1407	sts: 1317	sts: 1349	sts: 1500	sts: 1547	sts: 1540
stn: 0.40	stn: 0.41	stn: 0.40	stn: 0.32	stn: 0.35	stn: 0.32
mod: 5000	mod: 4263	mod: 3413	mod: 7071	mod: 4800	mod: 5167
M.b: 34.0	M.b: 39.3	M.b: 41.4	M.b: 37.1	M.b: 34.1	M.b: 34.1

C1	C2	C3	C4	C5	C6
8.1%	8.1%	8.1%	8.1%	8.1%	8.1%
160, 30"	160, 60"	180, 30"	180, 60"	200, 30"	200, 60"
sts: 1600	sts: 1506	sts: 1392	sts: 1588	sts: 1523	sts: 1580
stn: 0.27	stn: 0.30	stn: 0.30	stn: 0.34	stn: 0.32	stn: 0.32
mod: 6750	mod: 4650	mod: 4250	mod: 5833	mod: 7385	mod: 7642
M.b: 38.4	M.b: 41.9	M.b: 40.1	M.b: 36.1	M.b: 31.7	M.b: 37.8

D1	D2	D3	D4	D5	D6
9.9%	9.9%	9.9%	9.9%	9.9%	9.9%
sts: 1523	sts: 1454	sts: 1488	sts: 1544	sts: 1785	sts: 1568
stn: 0.40	stn: 0.34	stn: 0.35	stn: 0.40	stn: 0.38	stn: 0.30
mod: 6000	mod: 6188	mod: 5813	mod: 7583	mod: 7385	mod: 7750
M.b: 37.7	M.b: 39.4	M.b: 40.6	M.b: 42.7	M.b: 42.4	M.b: 39.7

Table 1 (cont.)

CROSS-MACHINE DIRECTION

A1	A2	A3	A4	A5	A6
4.6%	4.6%	4.6%	4.6%	4.6%	4.6%
160, 30"	160, 60"	180, 30"	180, 60"	200, 30"	200, 60"
sts: 565	sts: 632	sts: 555	sts: 517	sts: 716	sts: 662
stn: 1.15	stn: 1.00	stn: 1.20	stn: 1.10	stn: 1.10	stn: 1.25
mod: 330	mod: 394	mod: 351	mod: 360	mod: 615	mod: 215
M.b: 39.2	M.b: 33.9	M.b: 35.9	M.b: 32.8	M.b: 33.8	M.b: 39.6

B1	B2	B3	B4	B5	B6
6.1%	6.1%	6.1%	6.1%	6.1%	6.1%
160, 30"	160, 60"	180, 30"	180, 60"	200, 30"	200, 60"
sts: 568	sts: 552	sts: 662	sts: 792	sts: 750	sts: 620
stn: 1.35	stn: 1.20	stn: 1.10	stn: 1.20	stn: 1.20	stn: 1.20
mod: 273	mod: 334	mod: 480	mod: 517	mod: 506	mod: 458
M.b: 34.0	M.b: 39.3	M.b: 41.4	M.b: 37.1	M.b: 34.1	M.b: 34.1

C1	C2	C3	C4	C5	C6
8.1%	8.1%	8.1%	8.1%	8.1%	8.1%
160, 30"	160, 60"	180, 30"	180, 60"	200, 30"	200, 60"
sts: 626	sts: 634	sts: 633	sts: 571	sts: 587	sts: 617
stn: 1.10	stn: 1.20	stn: 1.10	stn: 1.20	stn: 1.10	stn: 1.30
mod: 403	mod: 309	mod: 450	mod: 378	mod: 372	mod: 410
M.b: 38.4	M.b: 41.9	M.b: 40.1	M.b: 36.1	M.b: 31.7	M.b: 37.8

D1	D2	D3	D4	D5	D6
9.9%	9.9%	9.9%	9.9%	9.9%	9.9%
160, 30"	160, 60"	180, 30"	180, 60"	200, 30"	200, 60"
sts: 643	sts: 722	sts: 602	sts: 644	sts: 821	sts: 750
stn: 1.25	stn: 1.25	stn: 1.10	stn: 1.30	stn: 1.25	stn: 1.20
mod: 298	mod: 509	mod: 353	mod: 396	mod: 517	mod: 566
M.b: 37.7	M.b: 39.4	M.b: 40.6	M.b: 42.7	M.b: 42.4	M.b: 39.7

CONTROL
400 F, tenter frame

MACHINE DIRECTION	CROSS-MACHINE DIRECTION
stress: 1752	stress: 382
strain: 0.17	strain: 0.32
modulus: 22,450	modulus: 350
Mullen burst: 29.4	Mullen burst: 29.4

BLANK
200 C, 60 seconds

MACHINE DIRECTION	CROSS-MACHINE DIRECTION
stress: 1447	stress:
strain: 0.45	strain:
modulus: 3357	modulus:
Mullen burst: 38.5	Mullen burst: 38.5

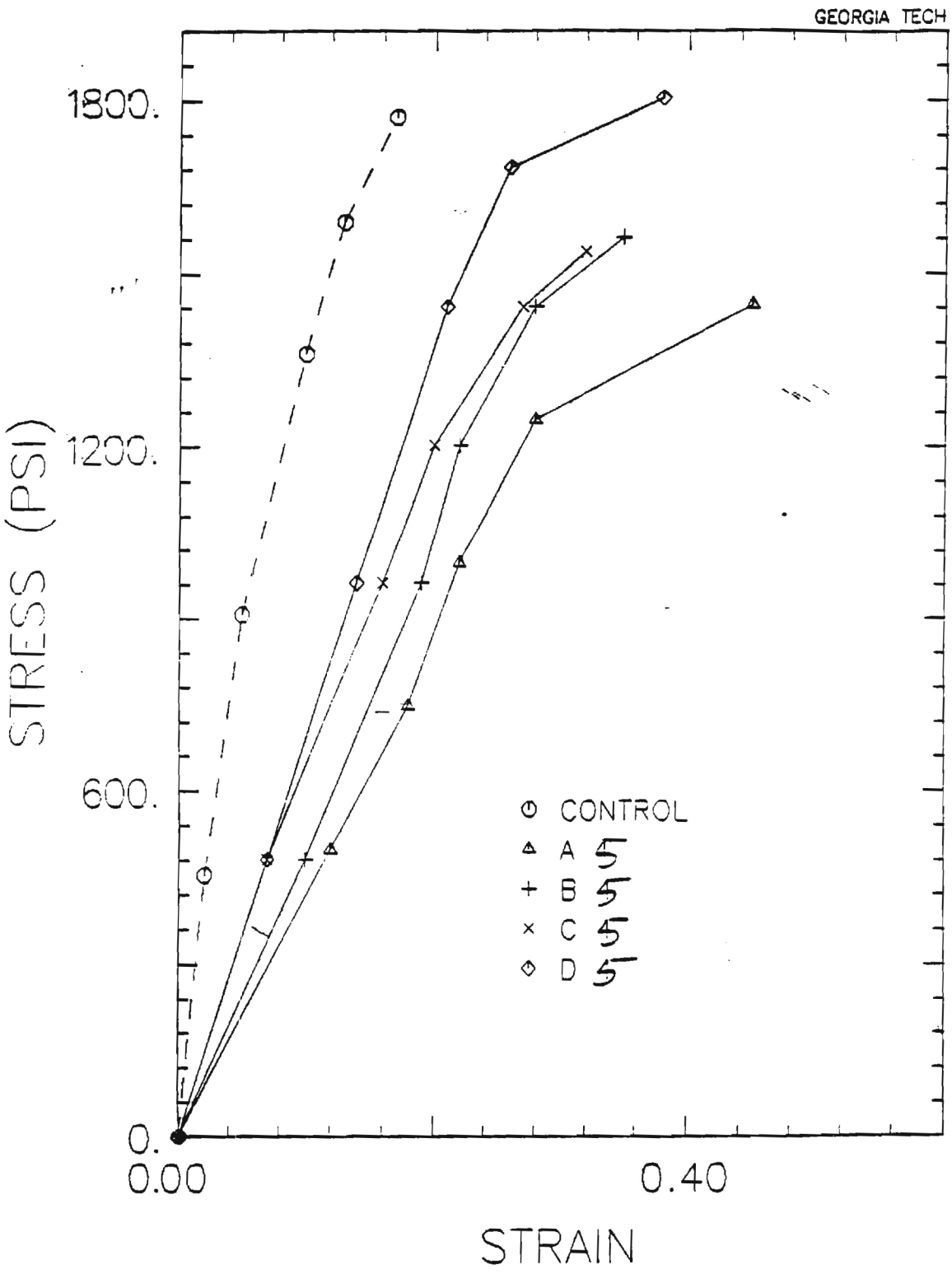
TABLE 2

HIGHER DEPOSITION STUDIES USING FA-252 ON C-9462

This is a continuation of earlier work to find optimum levels of powder deposition to match physical properties of the plant-produced nonwoven material, C-5522. Based on this earlier work, oven conditions of 200 C for 30 seconds were used to cure all samples. Five levels of powder were applied; 12.5%, 15%, 17.5%, 20%, and 30%. The results from Instron testing are reported in the chart below.

	MACHINE DIRECTION	CROSS-MACHINE DIRECTION
E 12.5%	stress: 1526.4 strain: 0.26 modulus: 7385	stress: 586.1 strain: 1.12 modulus: 383
F 15%	stress: 1732.5 strain: 0.32 modulus: 9640	stress: 763.8 strain: 1.19 modulus: 571
G 17.5%	stress: 1792.8 strain: 0.32 modulus: 9520	stress: 699.9 strain: 1.16 modulus: 595
H 20%	stress: 1648.3 strain: 0.33 modulus: 8800	stress: 819.7 strain: 1.16 modulus: 782
I 30%	stress: 1714.6 strain: 0.30 modulus: 10,625	stress: 762.5 strain: 0.99 modulus: 815

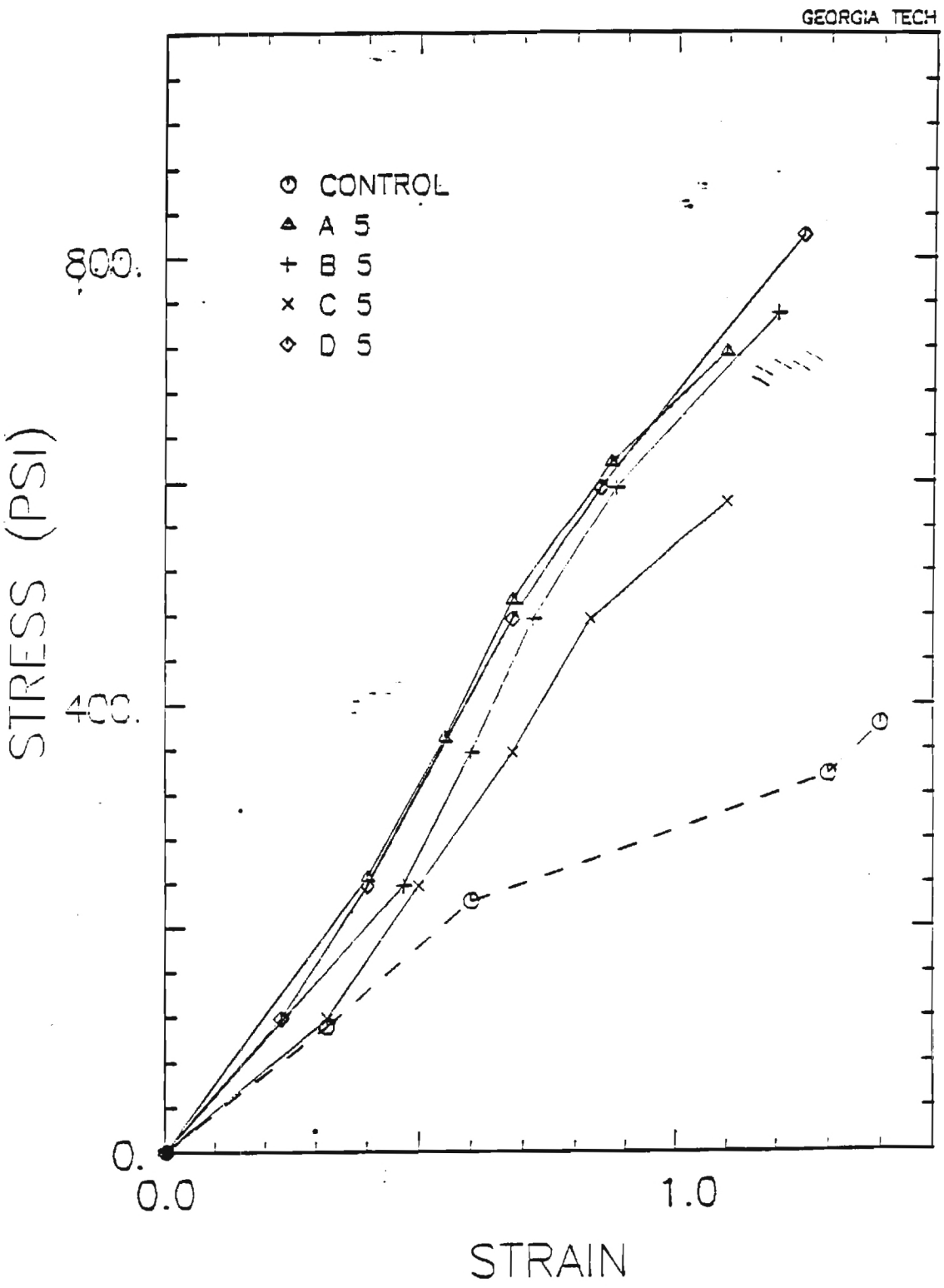
FIGURE 1



SAMPLES CURED AT 200 C/30SEC

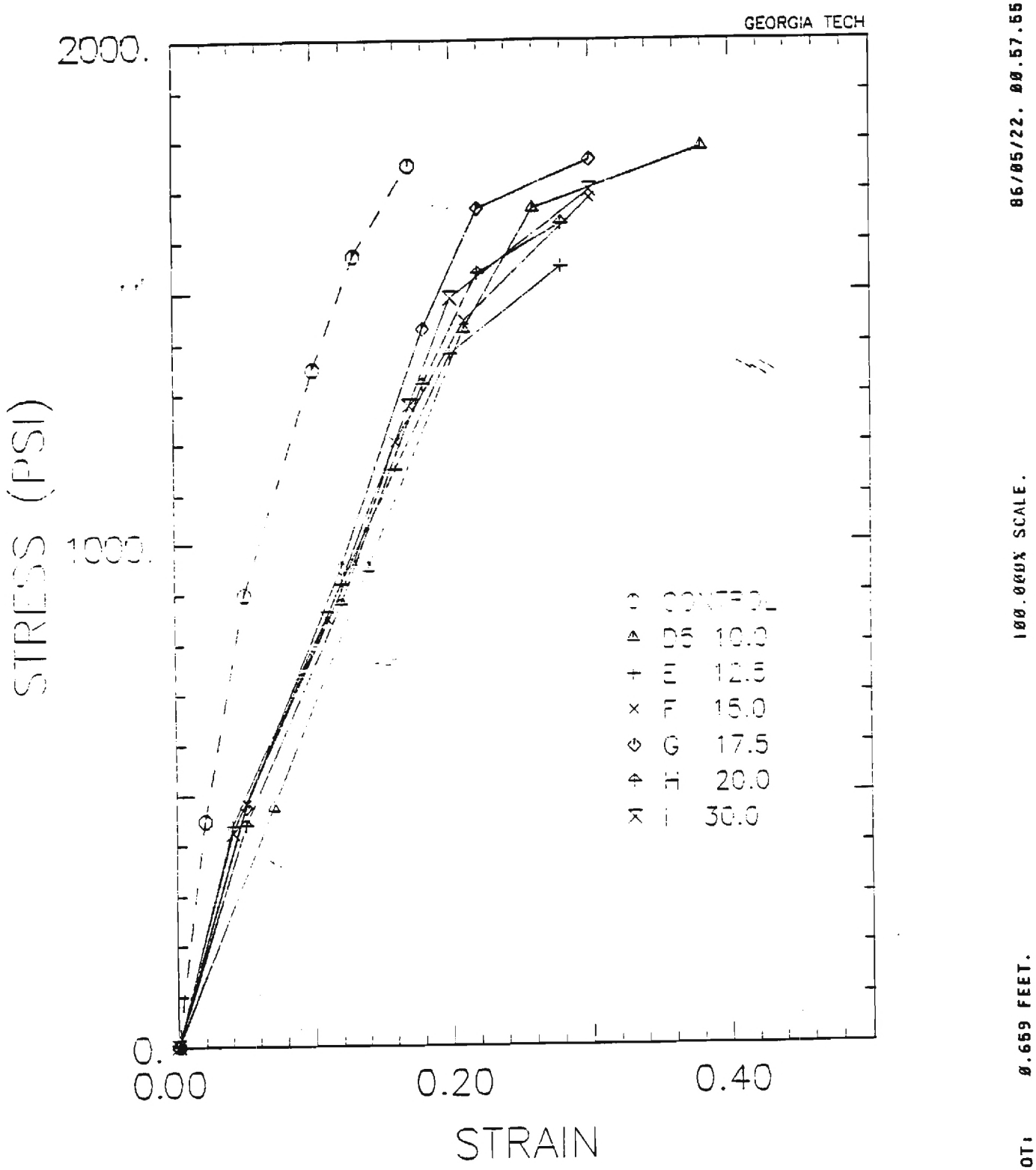
WITH DIFF DEPOSITION
(MACHINE DIRECTION)

FIGURE 2



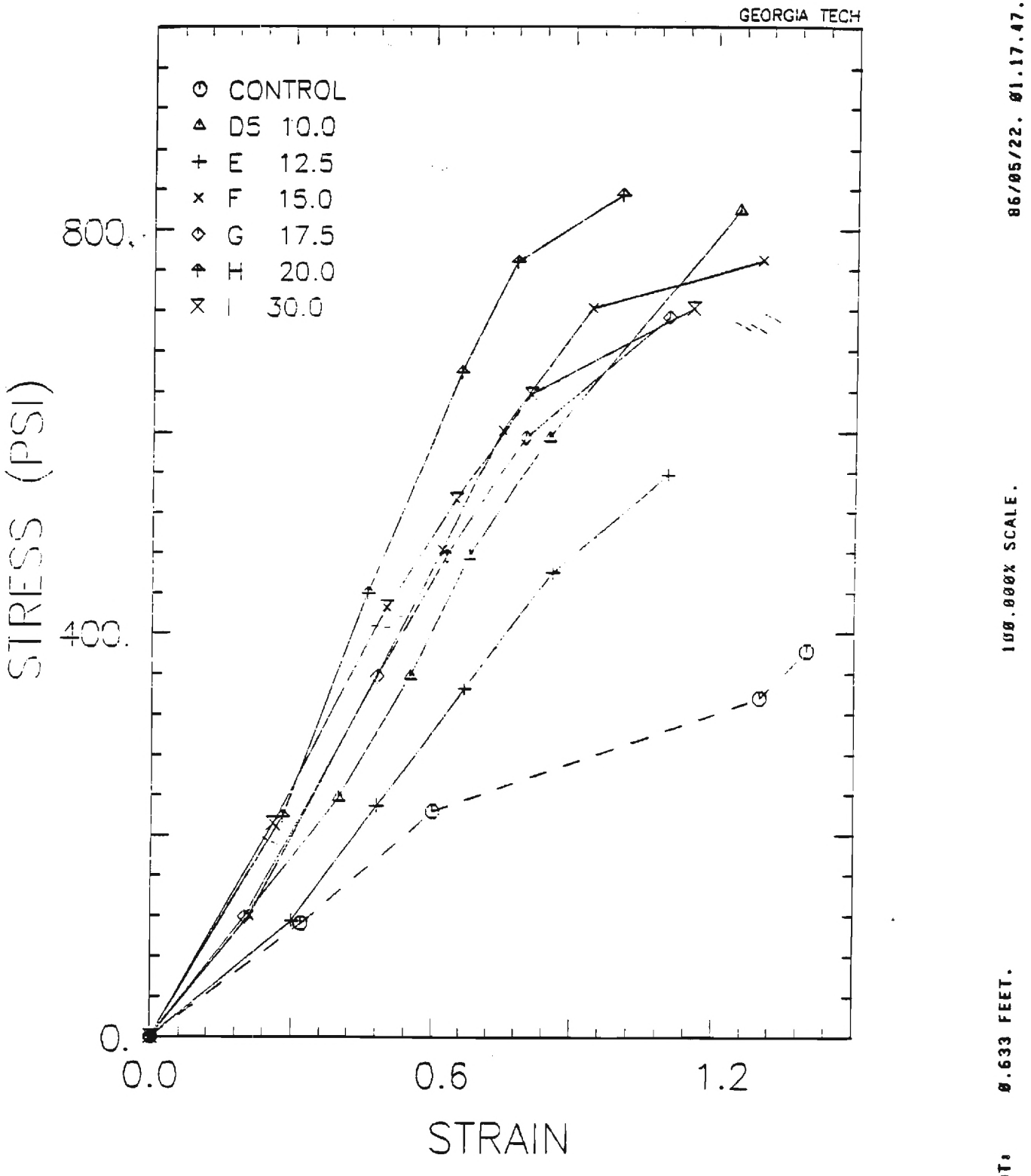
SAMPLES CURED AT 200 C/30 SEC
(CROSS-MACHINE DIRECTION)

FIGURE 3



SAMPLES WITH DIFFERENT
DEPOSITIONS (MACHINE DIR)

FIGURE 4



SAMPLES WITH DIFFERENT
DEPOSITIONS (CROSS DIR)

The data shows that the 9.9% SOS fabric (D5) in the MD approached the tensile strength of the plant standard, with a higher strain to break and a lower modulus (less stiff, Fig. 1). The CD mechanical properties were far superior to those of the plant standard, yielding a "squarer" fabric in terms of the two - dimensional properties (e.g., in tensile strength, 2.2:1 ratio vs. 4.6:1, MD: CD, D5 sample vs. plant standard, see Appendix A). The SOS produced fabric was softer, and gave a Mullen burst pressure rating 1.44 X that of the standard. Using the modified wash test prescribed by Chicopee for assessing pilling (5 rating: no pilling; 1 rating: very severe, by ASTM D-3512 pictures), the following results were obtained:

Unbound (greige) fabric:	3
Plant - Bound Control:	5
SOS, 4.3% SPU Sample:	5 (slightly fuzzier than standard)
SOS, 9.9% SPU Sample:	5 (equivalent to standard)
SOS, 30% SPU Sample:	5 (less fuzzy)

Thus the optimized conditions (sample D5) gave equivalent pilling properties on the wash test to the plant - bound standard.

Mechanical properties steadily improved from a low of 4.6% solids pickup (SPU) to 9.9%. However, as shown in Table 2 and Figures 3-4, increasing the SPU up to 30% gave practically no improvement in MD properties, and little in the CD direction.

Photographs of the plant - bound standard showed considerable fiber - bridging by binder film (Figure 5). In fact, the individual fibers appeared to be "sized" with the fiber, i.e., encased. Both factors lead to the high stiffness and recovery from deformation observed in the MD direction of the standard.

By contrast, the FA-252 gave more globular formation on melting, showing limited flowability at 200°C as a reflection of its high melt viscosity (high 10^4 cps at 200°C, Fig. 6). The dramatic improvement in mechanical properties over the unbound nonwoven, however, indicated that fiber junction binding was indeed taking place.

In summary, the SOS process with FA-252 gives an acceptable product from a mechanical property viewpoint, with pilling on washing equivalent to the plant - bound product. Eastman is cooperating with Georgia Tech in ways to decrease the melt viscosity of FA-252 without drastically affecting its film properties, e.g., by melt - blending a low MW prepolymer of the resin with the

FIG. 5 PHOTOGRAPH OF PLANT - BOUND CHICOPEE NONWOVEN

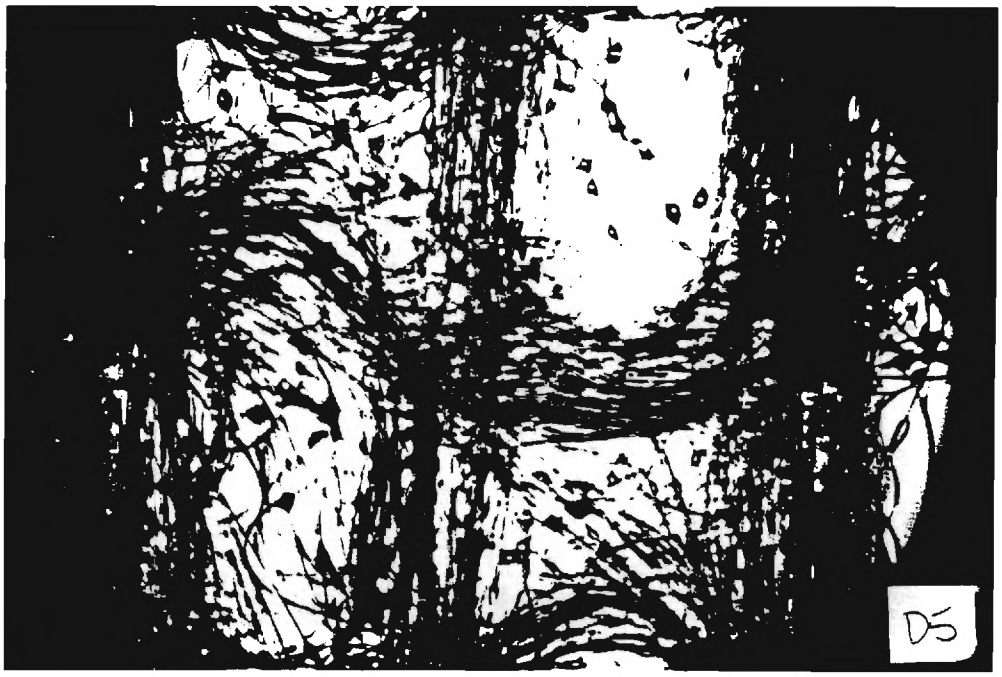


FIG 6 PHOTOGRAPH OF SOS - BOUND CHICOPEE NONWOVEN (FA-252 RESIN, D5 SAMPLE, 10% SPU).

commercial product. Two alternate resins from the H.B. Fuller Co. are also being screened in similar factorial experiments on the Nordson equipment.

Fluoropolymer Stain Repellent Finishing of Kimberly-Clark's Nonwoven

The 3M Co. has recently supplied the research team with 15 lbs. of powdered fluoropolymer blended with the proper quantity of Kimberly-Clark's proprietary antistatic agent. To optimize process utilization and to minimize clean-up time, the finishing research is being held until the nonwoven binding research is completed (July 1), at which time concentrated factorial experiments will be implemented on the polypropylene nonwoven (slated for hospital gown/drape applications).

Slashing of Polyester/Cotton Yarns

With various machinery and process modifications to gain tighter control of the Electostatic Technologies (ET) electrostatic fluidized bed system, SOS-sized polyester/cotton yarns (West Point Pepperell Co.) were produced that exhibited similar test properties to that of plant - sized yarns (PVA/starch/wax thermoset formulation). Table 3 details the conditions for a typical optimized slashing using the 60:40 Eastman WD/ adipic acid size formulation.

For Ruti Webtester abrasion resistance testing, the following instrument settings were used:

Pivot setting:	3.0
Cyclical elongation:	0.5%
Weight tensioning:	10 gm
Cycles/minute:	400
Yarn Tensioning:	7.8 N

Fifteen yarns were mounted on the device, and the number of cycles required for 10 of the yarns to break were recorded. Either four or five sets of each yarn were tested, and the cycles per break for each mounted yarn were averaged by set.

Table 4 gives the comparative averaged cycle data for the greige (unsized), plant sized (thermoset formulation) and SOS - sized (thermoplastic

Table 3. Optimized Process Conditions for SOS Polyester/Cotton Yarn Slashing

Formulation:	Melt-blended 60/40 Eastman WD/adipic acid (0-75 screen cut), with 5% Cabosil silica powder flow modifier		
Bed Vibrator:	On		
Dual Teflon Wipe Rolls, Temp:	Set/Read-out:	270°C	270°C
	Contact:	136°C	238°C
Yarn height above bed base:	3 inches		
Take-up speed:	1.6 ypm		
Charge on plate:	40 kV		
On line solids pick-up:	11.3%		
Conditioned yarn pick-up:	16.7%		
Run length:	1.75 hours		

Table 4 . Ruti Webtester

Results for Different Yarns

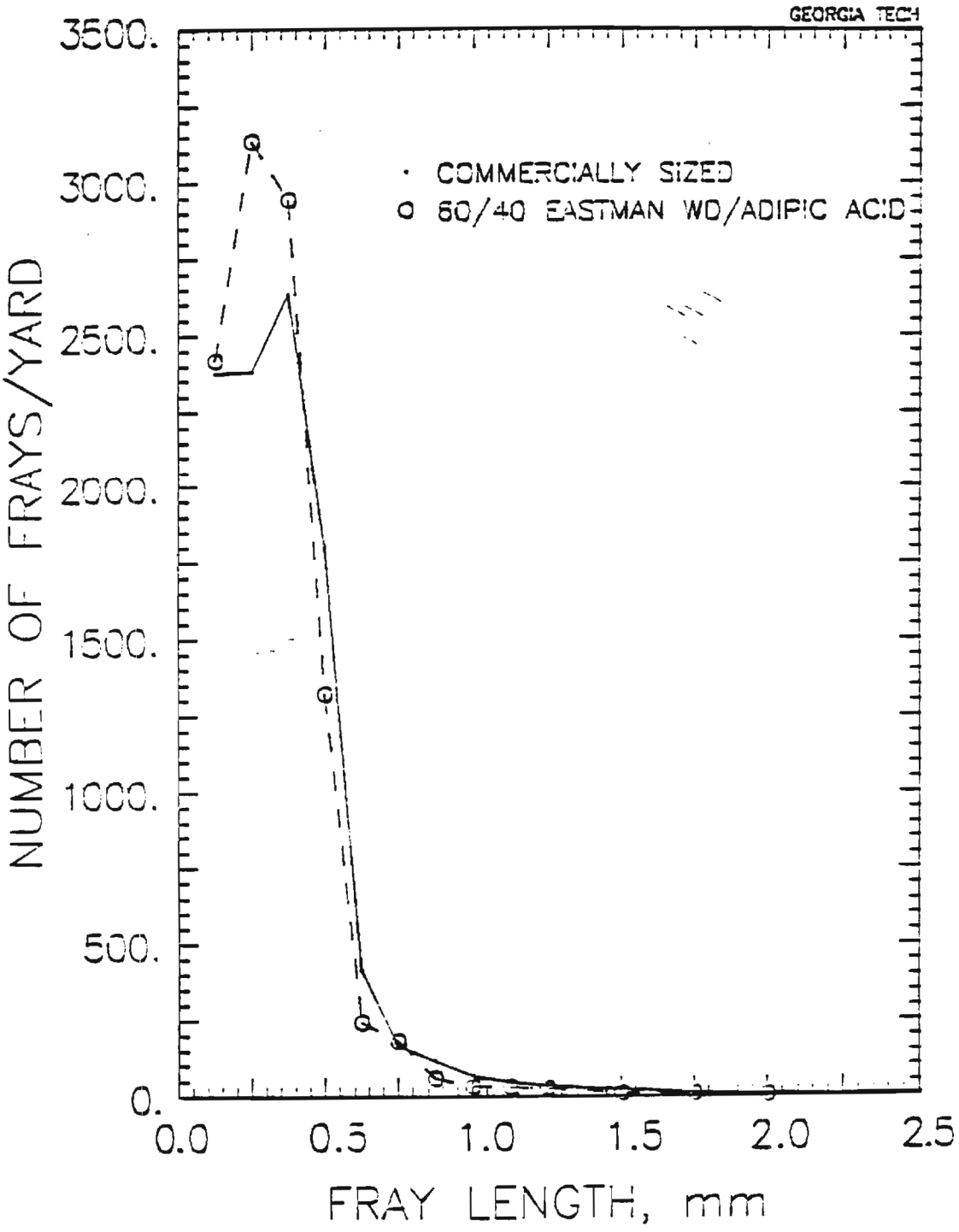
Yarn	Tenth-Yarn Break Cycle[a] (maximum/set)	Cycle Average[b]
Greige (unsized)	1,448	1,340
Plant - sized (thermoset)	18,699	16,971
SOS - sized (thermoplastic) sample 418-79-A-89D	25,760	11,915

a. Maximum recorded number of yarn cycles for any of the sets, yarn break 10.

b. Cycles recorded for each breaking yarn up to 10 of 15, 4-5 sets of tests, and the average cycles/4-5 sets obtained.

FIGURE 7

FRAY COUNTER RESULTS FOR DIFFERENT YARNS



formulation) yarns. The SOS - slashed yarn gave an average cycle-to-break/4 sets that was 70% of that for the plant - sized product, and a maximum recorded 10-yarn break 138% higher than the corresponding break for the thermoset - sized yarn.

The Toray fray counter results for the corresponding SOS - and plant - slashed yarns are plotted in Figure 7. The SOS material gave a slightly higher count of short fray lengths, which are not as important in influencing weavability as the long fray lengths. Beyond 0.5 mm fray lengths, the two yarns performed basically the same.

In summary, yarn properties from the SOS slashing process are approaching those of the plant-supplied standard. From the screening tests, the SOS yarns should provide satisfactory weavability at the end of the year 3 large trials at ET's facility in Connecticut. To complete the research phase, a larger (15 in.) fluidized bed was delivered in June, and a 10 - yarn process is being developed around it to demonstrate uniform application at multiple ends.

Xerography Printing of Polyester/Cotton Fabrics

After the last Working Group Meeting, four ground powder samples were prepared for developer/toner research:

1. Elvax 410 from duPont, poly (ethylene-co-vinyl acetate), melt blended with 5% by weight "phthalo blue" pigment (Wiley mill-cryogenic grinder-air mill-sieve sequence).
2. MU-760 analog to Elvax from U.S. Industries, but with higher film properties, pigmented and prepared as Elvax.
3. Ground/sieved blue disperse dye cake from Ciba-Geigy ($<5\mu$ average particle size).
4. Ground/sieved blue disperse dye diluted with lignin sulfonate filler/dispersing agent, same dye as (3), from Ciba-Geigy ($<5\mu$).

The materials were submitted to Mr. John Walkonis of Hunt Chemical Co. Mr. Walkonis and his staff are evaluating physical properties of the four toner systems (dielectric, tribolectric, etc.), and will supply Georgia Tech with compatible developers for the toners. Batch printing on woven 100% polyester (disperse dye toners) and 50/50 polyester/cotton (pigment/resin toners) will be resumed on the Haloid copier when the first total chemical system is supplied by Hunt.

In the interim, paper toner/developer systems, which have given clear prints on the Haloid process but which have poor textile fastness properties, are being utilized to develop a fully - continuous fabric xerography system. According to Hunt Chemical, systems will operate equally well on the same photoconductive surface (in this case, selenium surfaces). Therefore, to reduce project down - time while Hunt is researching developers for the textile toners, the paper toner/developers are being utilized for hardware system development translation from the paper to the textile developer/toner systems when they become available should be straight forward.

UV-Curable Films for Electrostatic Liquid Spray Applications

The Argus Continuous UV Processor Unit was obtained, and preliminary trials have been conducted on curing various polyurethane-based films. The unit's conveyor has a minimum speed of 3 ft/min (34.2 sec. dwell time), and a maximum speed of 30 ft/min (2.5 sec. dwell time). A plot of dwell time vs. conveyor speed is shown Fig. 8.

Several liquid oligomer systems have been cast as films and cured at 15 ft/min in the Argus unit (Table 5). Poly (ethylene glycol dimethacrylate) additive has shown promise of introducing a greater degree of flexibility into the stiff films of Table 5. Work is continuing on modifying the oligomers to give a viscosity compatible with the Nordson electrostatic liquid spray unit (< 600 cps) while yielding a flexible, UV-cured flim.

Argus UV Processor
Sample Dwell Time

FIGURE 8

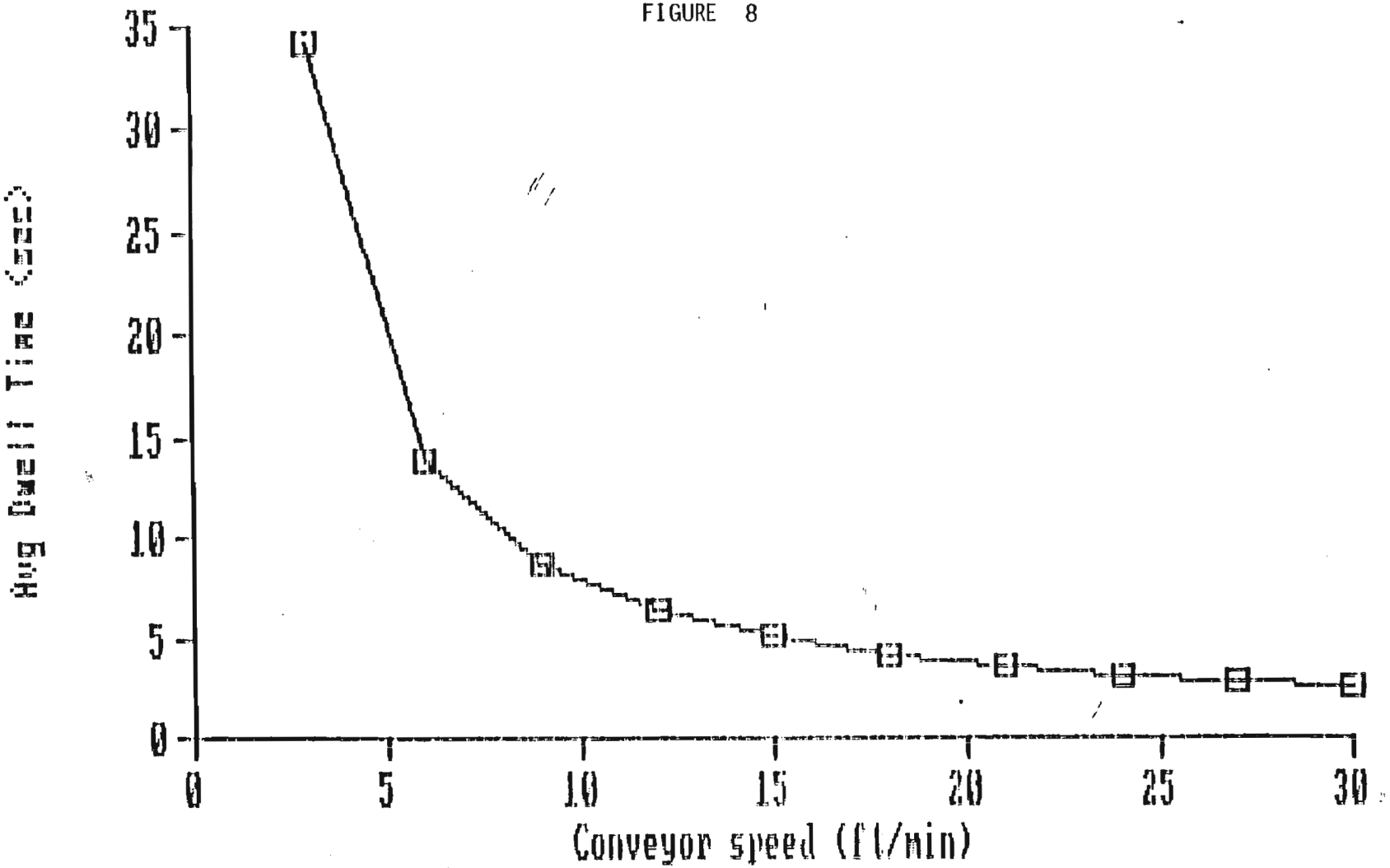


TABLE 5. Screening Test: Curing Liquid Oligomer Films

RESIN	ADDITIVES	VISCOSITY (lit.)	CURE SPEED	COMMENTS/OBSERVATIONS
Pedigree 140B RO-101 (P.D. George)	1% Irgacure-184 by weight	25,000 cps @ 25°C	15ft/min	flexible, stiff:like cellophane (designed as optical fiber coating)
Photoglaze B3693-44 (Lord Corp.)	0% (blend of oligomer, monomer, and photoinitiator)	1100-1800 cps @ 25°C	15 ft/min	flexible, stiff:like cellophane, adhered to glass plate,(may be heated to apply)
Uvithane 782 (Morton-Thiokol)	1% Ivgacure-184	Solid at Room Temp. Low Temp Melt	15ft/min	soft, very flexible, extensible
ZM-1197 (Morton-Thiokol)	0% (blend of oligomer, monomer,photointiator, etc.	11,000 cps @ 25°C	15ft/min	adhered to glass, flexible, tears easily-possible signs of reaction in storage container (blended Uvithane 782)
Ebecryl19-6230 (Radcure Spec.)	1% Irgacure 184	30,000-40,000 cps @ 25°C	15ft/min	soft, flexible, tears easily, colapses on itself
Chempot 19-4835 (Radcure Spec.)	1% Irgacure 184	4,000 cps @ 60°C	15ft/min	stiff, soft, slightly adhesive in nature
Chempot 19-4827 (Radcure Spec.)	1% Irgacure 184	3,500 cps @ 60°C	15ft/min	stiff, soft, somewhat adhesive, slightly extensible.

a. All films were 3 mil in thickness, prepared on glass plates with a Gardner Knife. All films were cured at 15ft/min exposure under 2-200 watt/in UV lamps (Argus UV Processor System).

APPENDIX A

APPENDIX A

Georgia Institute of Technology
A UNIT OF THE UNIVERSITY SYSTEM OF GEORGIA
SCHOOL OF TEXTILE ENGINEERING
ATLANTA, GEORGIA 30332
(404) 894-2490

May 30, 1986

MEMORANDUM

TO: In-House DOE/SOS Group,
Dr. S. Hansen

FROM: Dr. Fred L. Cook

SUBJECT: Chicopee Feedback on Mattress Cover Nonwoven

Elisha and I, in a conference call to Gerald Hornaday of Chicopee/Benson N.C. plant on May 29, clarified several points:

1. The NW is made by laying several layers of random webs from polyester staple, and then entangling the webs by a proprietary water jet process.

2. The specs for the plant finished fabric sent to us as standard (C-5522) has a finished weight of 1.05 oz/yd^2. The unfinised fabric we are running (C-9462) has a base weight of 1.3oz/yd^2, but due to expansion during the binding process (pad-nip-tenter cure), the fabric is stretched out so that a 1.05 oz/yd^2 finished weight is attained.

3. Specs call for 6.1% (of final product weight) solids add-on. A recent chemical strip analysis, however, gave an actual 7.3 - 7.5% add-on for this fabric.

4. They are concerned about the 2:1 - 3:1 property ratio between the MD and CD directions of their product, and would like to see a "square" (1:1) property ratio.

5. They do not evaluate deformation or recovery from deformation, although they are trying to improve it (see (4)).

6. They are using a modified wash performance test (E.S. has procedure and lab contacts.).

We are sending fabrics/data for their perusal.

We are now concerned about comparison of our fabrics with their finished standards. We have no "stretching" on the current SOS powder process, and therefore increase, rather than decrease, density on finishing. Thus straight lb. comparisons on ultimate tensile strengths, etc., are not valid (apples-oranges problem). We need some suggestions on <u>logical</u> mechanical property comparisons!

FLC/cdp

E-21-633

Georgia Tech

School of Textile & Fiber Engineering
Georgia Institute of Technology
Atlanta, Georgia 30332-0295
404•894•2490
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September 28, 1990

Mr. William (Bill) Sonnett
Project Officer
Department of Energy
Office of Industrial Programs
Conservation & Solar Energy
CE12
1000 Independence Avenue
Washington, D.C. 20585

Dear Bill:

Enclosed is the first draft of the full Final Technical Report on the continuation phase of DOE Contract No. DE-FG05-84CE40702. Please review it, make any corrections/suggestions that you deem appropriate in red ink, and we will finalize the document and submit it in bound form in the required number of copies.

Much was accomplished in the 2.5 years of research, with three solid-on-solid (SOS) textile processes carried to fruition in full-scale proof-of-concept trials at West Point Pepperell and the Nordson Corp. facilities. Four other SOS processes were demonstrated at the pilot scale, with two (reactive silicone finishing and polytherm/durable press resin finishing) ready for scale up. A number of questions arose in the course of the research, not unexpected when totally new ground is being broken for the first time. Textile xerography is one such area, with much progress made in scale-up and demonstration of the capability of complex printing, but with further development research required in the materials and CAD areas before prototype machine development. We plan to submit proposals in several of these areas when the Final Report is put to bed. A total of 4-6 refereed journal papers are expected from the report, with the first on textile xerography already submitted to the **Textile Chemist and Colorist** for peer review.

I apologize for the lateness in completing the final sections of the report, but I trust you understand it was not due to wasted time. My duties as School director have overwhelmed me in the past year on a number of fronts, and one inevitable conclusion it has fostered is that I will no longer be afforded the time to function as the Principal Investigator on a major research project of this scope. Future SOS proposals to DOE will thus be headed by one of our other team members (likely Drs. Wallace (Chuck) Carr, Wayne Tincher and Lewis Dorrity, the latter whom we hired from Greenwood Mills in 1989), with me as an investigator working with them.

Hopefully, with the faculty assuming the primary responsibility for running future projects and compiling reports, we can avoid the problems the lateness of this one has caused all parties.

Bill, thank you for your understanding in this matter, and if you have any questions on the report draft that need a verbal clarification, please call.

Sincerely,

Fred

Fred L. Cook
Director & Professor

cc: Dean William M. Sangster
Duane Hutchison
DOE Project Team
Tom Gross

Georgia Tech

Office of Grants and Contracts Accounting

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March 26, 1992

Ms. Melissa Y. Johnson, Contract Specialist
Special Acquisitions Branch
U. S. Department of Energy
Procurement and Contracts Division
P. O. Box 2001
Oak Ridge, TN 37831-8757

REFERENCE: Grant # DE-FG05-84CE40702

Dear Ms. Johnson,

Enclosed in triplicate are the Final Financial Status Report (SF-269A) and the Federal Cash Transactions Report (SF-272) that is required for the closeout of Grant No. DE-FG05-84CE40702 covering the period August 1, 1984 through March 1, 1989.

If you should have questions or need additional information, please contact Geraldine Reese of this office at (404) 894-2629.

Sincerely,

David V. Welch

David V. Welch
Director

DVW/GMR/djt

Enclosures

cc: Dr. F. L. Cook - Textiles - 0295
Ms. Mary Wolfe, OCA/CSD 0420
File: E-27-633/R5810-0A0

FINANCIAL STATUS REPORT FINAL

(Long Form)

(Follow instructions on the back)

1. Federal Agency and Organizational Element to Which Report is Submitted	2. Federal Grant or Other Identifying Number Assigned By Federal Agency	OMB Approval No.	Page	of
U. S. Department of Energy	DE-FG05-84CE40702	0348-0039	1	1 pages

3. Recipient Organization (Name and complete address, including ZIP code)

Georgia Tech Research Corporation
P. O. Box 100117
Atlanta, GA 30384

4. Employer Identification Number	5. Recipient Account Number or Identifying Number	6. Final Report	7. Basis
58-0603146	E-27-633/R5810-0A0	☒ Yes ☐ No	☒ Cash ☐ Accrual

8. Funding/Grant Period (*See Instructions*) From: (Month, Day, Year)	To: (Month, Day, Year)	9. Period Covered by this Report From: (Month, Day, Year)	To: (Month, Day, Year)
August 1, 1984	March 1, 1989	August 1, 1984	March 1, 1989

10. Transactions:	I Previously Reported	II This Period	III Cumulative
a. Total outlays			1,969,768.33
b. Refunds, rebates, etc.			-0-
c. Program income used in accordance with the deduction alternative			-0-
d. Net outlays *(Line a, less the sum of lines b and c)*			1,969,768.33
Recipient's share of net outlays, consisting of: e. Third party (in-kind) contributions			754,557.00
f. Other Federal awards authorized to be used to match this award			-0-
g. Program income used in accordance with the matching or cost sharing alternative			-0-
h. All other recipient outlays not shown on lines e, f or g			9,026.83
i. Total recipient share of net outlays *(Sum of lines e, f, g and h)*			763,583.83
j. Federal share of net outlays (line d less line i)			1,206,184.50
k. Total unliquidated obligations			-0-
l. Recipient's share of unliquidated obligations			-0-
m. Federal share of unliquidated obligations			-0-
n. Total federal share (sum of lines j and m)			1,206,184.50
o. Total federal funds authorized for this funding period			1,207,000.00
p. Unobligated balance of federal funds *(Line o minus line n)*			815.50
Program Income, consisting of: q. Disbursed program income shown on lines c and/or g above			-0-
r. Disbursed program income using the addition alternative			-0-
s. Undisbursed program income			-0-
t. Total program income realized *(Sum of lines q, r and s)*			-0-

11. Indirect Expense	a. Type of Rate *(Place "X" in appropriate box)* ☐ Provisional ☐ Predetermined ☐ Final ☒ Fixed			
	b. Rate: See Attached	c. Base: MTDC	d. Total Amount: 441,478.54	e. Federal Share: 441,478.54

12. *Remarks: Attach any explanations deemed necessary or information required by Federal sponsoring agency in compliance with governing legislation.*

Questions pertaining to this report should be directed to:
Geraldine Reese (404) 894-2629

13. Certification: **I certify to the best of my knowledge and belief that this report is correct and complete and that all outlays and unliquidated obligations are for the purposes set forth in the award documents.**

Typed or Printed Name and Title	Telephone (Area code, number and extension)
David V. Welch, Director, Grants & Contracts Accounting	(404) 894-2629
Signature of Authorized Certifying Official: David V. Welch	Date Report Submitted: March 26, 1992

Attachment
Financial Status Report
Grant # DE-FG05-84CE40702
Period Covering: 08/01/84 - 03/01/89

	Direct Costs	Indirect Costs	Equipment Excluded from Indirect Costs
FY'85 - Fixed @55.3%	$129,940.58	$ 71,672.44	$ 333.98
FY'86 - Fixed @63.5%	177,193.14	107,153.26	8,447.84
FY'87 - Fixed @63.5%	187,448.40	117,762.12	1,996.25
FY'88 - Fixed @60.0%	248,626.04	131,992.04	28,639.32
FY'89 - Fixed @60.0%	21,497.80	12,898.68	

GIT CONTRIBUTIONS

	Direct Costs	Indirect Costs
FY'86 @63.5%	$ 5,521.00	$ 3,505.83

IN-KIND CONTRIBUTIONS

08/01/86 - 07/31/87	$ 332,007.00
08/01/87 - 03/01/89	$ 422,550.00

FINAL

FEDERAL CASH TRANSACTIONS REPORT

(See instructions on the back. If report is for more than one grant or assistance agreement, attach completed Standard Form 272-A.)

Approved by Office of Management and Budget, No. 80-R0182

1. Federal sponsoring agency and organizational element to which this report is submitted: U. S. Department of Energy

2. RECIPIENT ORGANIZATION

Name: GEORGIA TECH RESEARCH CORPORATION

Number and Street: P. O. BOX 100117

City, State and ZIP Code: ATLANTA, GA 30384

3. FEDERAL EMPLOYER IDENTIFICATION NO.: 58-0603146

4. Federal grant or other identification number: DE-FG05-84CE40702

5. Recipient's account number or identifying number: E-27-633/R5810-0A0

6. Letter of credit number: 89-00-30-39

7. Last payment voucher number:

Give total number for this period

8. Payment Vouchers credited to your account:

9. Treasury checks received (*whether or not deposited*):

10. PERIOD COVERED BY THIS REPORT

FROM (*month, day, year*): August 1, 1984

TO (*month, day year*): March 1, 1989

11. STATUS OF FEDERAL CASH (*See specific instructions on the back*)

a. Cash on hand beginning of reporting period	$ -0-
b. Letter of credit withdrawals	1,206,184.50
c. Treasury check payments	-0-
d. Total receipts (*Sum of lines b and c*)	1,206,184.50
e. Total cash available (*Sum of lines a and d*)	1,206,184.50
f. Gross disbursements	1,206,184.50
g. Federal share of program income	-0-
h. Net disbursements (*Line f minus line g*)	1,206,184.50
i. Adjustments of prior periods	-0-
j. Cash on hand end of period	$ 0.00

12. THE AMOUNT SHOWN ON LINE 11j, ABOVE, REPRESENTS CASH REQUIREMENTS FOR THE ENSUING _____ *Days*

13. OTHER INFORMATION

a. Interest income	$
b. Advances to subgrantees or subcontractors	$

14. REMARKS (*Attach additional sheets of plain paper, if more space is required*)

Questions pertaining to this report should be directed to:
Geraldine Reese (404) 894-2629

15. CERTIFICATION

I certify to the best of my knowledge and belief that this report is true in all respects and that all disbursements have been made for the purpose and conditions of the grant or agreement

AUTHORIZED CERTIFYING OFFICIAL

SIGNATURE: David V. Welch

DATE REPORT SUBMITTED: March 26, 1992

TYPED OR PRINTED NAME AND TITLE: David V. Welch, Director
Office of Grants & Contracts Acctg.

TELEPHONE (*Area Code, Number, Extension*): (404) 894-2629

THIS SPACE FOR AGENCY USE

Georgia Tech

Office of Grants and Contracts Accounting

Georgia Institute of Technology
Hinman Building
Atlanta, Georgia 30332-0259
404•894•4624; 2629
Fax: 404•894•5519

March 26, 1992

Ms. Melissa Y. Johnson, Contract Specialist
Special Acquisitions Branch
U. S. Department of Energy
Procurement and Contracts Division
P. O. Box 2001
Oak Ridge, TN 37831-8757

REFERENCE: Grant # DE-FG05-84CE40702

Dear Ms. Johnson,

Enclosed in triplicate is the Financial Status Report (SF-269A) for Grant No. DE-FG05-84CE40702 covering the period August 1, 1987 through March 1, 1989.

If you should have questions or need additional information, please contact Geraldine Reese or me at (404) 894-2629.

Sincerely,

David V. Welch

David V. Welch
Director

DVW/GMR/djt

Enclosures

cc: Dr. F. L. Cook - Textiles - 0295
Ms. Mary Wolfe, OCA/CSD 0420
File: E-27-633/R5810-0A0

FINANCIAL STATUS REPORT

(Long Form)

(Follow instructions on the back)

1. Federal Agency and Organizational Element to Which Report is Submitted	2. Federal Grant or Other Identifying Number Assigned By Federal Agency	OMB Approval No.	Page	of
U. S. Department of Energy	DE-FG05-84CE40702	0348-0039	1	1 pages

3. Recipient Organization (Name and complete address, including ZIP code)

Georgia Tech Research Corporation
P. O. Box 100117
Atlanta, GA 30384

4. Employer Identification Number	5. Recipient Account Number or Identifying Number	6. Final Report	7. Basis
58-0603146	E-27-633/R5810-0A0	☐ Yes ☒ No	☒ Cash ☐ Accrual

8. Funding/Grant Period *(See Instructions)* From: (Month, Day, Year)	To: (Month, Day, Year)	9. Period Covered by this Report From: (Month, Day, Year)	To: (Month, Day, Year)
August 1, 1984	March 1, 1989	August 1, 1987	March 1, 1989

10. Transactions:	I Previously Reported	II This Period	III Cumulative
a. Total outlays	1,147,331.32	822,437.01	1,969,768.33
b. Refunds, rebates, etc.	-0-	-0-	-0-
c. Program income used in accordance with the deduction alternative	-0-	-0-	-0-
d. Net outlays *(Line a, less the sum of lines b and c)*	1,147,331.32	822,437.01	1,969,768.33
Recipient's share of net outlays, consisting of:			
e. Third party (in-kind) contributions	332,007.00	422,550.00	754,557.00
f. Other Federal awards authorized to be used to match this award	-0-	-0-	-0-
g. Program income used in accordance with the matching or cost sharing alternative	-0-	-0-	-0-
h. All other recipient outlays not shown on lines e, f or g	9,026.83	-0-	9,026.83
i. Total recipient share of net outlays *(Sum of lines e, f, g and h)*	341,033.83	422,550.00	763,583.83
j. Federal share of net outlays (line d less line i)	806,297.49	399,887.01	1,206,184.50
k. Total unliquidated obligations			-0-
l. Recipient's share of unliquidated obligations			-0-
m. Federal share of unliquidated obligations			-0-
n. Total federal share (sum of lines j and m)			1,206,184.50
o. Total federal funds authorized for this funding period			1,207,000.00
p. Unobligated balance of federal funds *(Line o minus line n)*			815.50
Program income, consisting of:			
q. Disbursed program income shown on lines c and/or g above			-0-
r. Disbursed program income using the addition alternative			-0-
s. Undisbursed program income			-0-
t. Total program income realized *(Sum of lines q, r and s)*			-0-

11. Indirect Expense	a. Type of Rate *(Place "X" in appropriate box)* ☐ Provisional ☐ Predetermined ☐ Final ☒ Fixed			
	b. Rate	c. Base	d. Total Amount	e. Federal Share
	See Attached	MTDC	139,217.89	139,217.89

12. *Remarks: Attach any explanations deemed necessary or information required by Federal sponsoring agency in compliance with governing legislation.*

Questions pertaining to this report should be directed to:
Geraldine Reese (404) 894-2629

13. Certification: **I certify to the best of my knowledge and belief that this report is correct and complete and that all outlays and unliquidated obligations are for the purposes set forth in the award documents.**

Typed or Printed Name and Title	Telephone (Area code, number and extension)
David V. Welch, Director, Grants & Contracts Accounting	(404) 894-2629
Signature of Authorized Certifying Official	**Date Report Submitted**
David V. Welch	March 26, 1992

Attachment
03/26/92
Financial Status Report
Grant # DE-FG05-84CE40702
Period Covering: 08/01/87 - 03/01/89

	Direct Costs	Indirect Costs	Equipment Excluded from Indirect Costs
FY'85 - Fixed @55.3%	$129,940.58	$ 71,672.44	$ 333.98
FY'86 - Fixed @63.5%	177,193.14	107,153.26	8,447.84
FY'87 - Fixed @63.5%	187,448.40	117,762.12	1,996.25
FY'88 - Fixed @60.0%	248,626.04	131,992.04	28,639.32
FY'89 - Fixed @60.0%	21,497.80	12,898.68	

REPORT PERIOD

	Direct Costs	Indirect Costs	Equipment Excluded from Indirect Costs
08/01/87 - 06/30/88	$239,171.32	$126,319.21	$28,639.32
07/01/88 - 03/01/89	21,497.80	12,898.68	

GIT CONTRIBUTIONS

	Direct Costs	Indirect Costs
FY'86 @63.5%	$ 5,521.00	$ 3,505.83

IN-KIND CONTRIBUTIONS

08/01/86 - 07/31/87	$ 332,007.00
08/01/87 - 03/01/89	$ 422,550.00

FINAL TECHNICAL REPORT
GT Project No. E-27-633

APPLICATION OF CHEMICALS TO SUBSTRATES WITHOUT THE USE OF LIQUIDS; SOLID-ON-SOLID (SOS) PROCESSING

Prepared by
Dr. Fred L. Cook
Principal Investigator

Submitted to
DEPARTMENT OF ENERGY

Report Period
August 1, 1984 - July 31, 1986

Under
DOE Grant No. DE-FG05-84CE40702

December 13, 1986

GEORGIA INSTITUTE OF TECHNOLOGY
A UNIT OF THE UNIVERSITY SYSTEM OF GEORGIA
SCHOOL OF TEXTILE ENGINEERING
ATLANTA, GEORGIA 30332

APPLICATION OF CHEMICALS TO SUBSTRATES WITHOUT THE USE OF LIQUIDS; SOLID-ON-SOLID (SOS) PROCESSING

DOE Grant No. DE-FG05-84CE40702

Georgia Tech Project No. E-27-633

FINAL TECHNICAL REPORT

August 1, 1984 - July 31, 1986

Submitted to the

DEPARTMENT OF ENERGY

by

Dr. Fred L. Cook
Principal Investigator

School of Textile Engineering
GEORGIA INSTITUTE OF TECHNOLOGY

Atlanta, Georgia 30332

December 13, 1986

TABLE OF CONTENTS

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SUMMARY

Theoretically, the optimum method for applying chemicals to textiles in an energy-efficient manner is through deposition of powders, followed by dry thermal flow of a thermoplastic component, i.e., with no liquids involved in the process. By eliminating the necessity to elevate the temperature of, and to evaporate, liquids, energy consumption of current textile processes can be reduced by an order of magnitude.

Although SOS processing has reached the status of "best available technology" in the metals industry (electrostatic automobile painting, wire coating, aluminum can printing) and the paper industry (multicolor xerox printing), the idea has not penetrated the textile industry. Besides energy savings, the SOS technology offers several concomitant advantages, including elimination of water supply needs, elimination of steam generation needs, elimination of effluent and the potential for dramatically-increased process speeds. The latter derives from the low heat capacities of dry polymeric powders and fibers, leading to rapid heat transfer in curing ovens. The result is reduced dwell times for SOS-processed fabrics over those from comparative wet processes, since the energy-intensive slow process of water heat-up/evaporation is avoided (a limiting line speed factor). Greater productivity and reduced cost factors are projected for SOS textile processes, which will ultimately improve the industry's standing vis-a-vis foreign imports.

Bench-scale development has progressed rapidly in the first two years of the project for several SOS textile processes. Clear, well-defined color prints were achieved on prepared polyester/cotton sheeting fabric using a Haloid batch xerography copier and paper toner/carrier (termed developer) systems. However, the toner portion of the developer, consisting of a suspected poly(styrene-co-acrylate) binder and a pigment, failed to meet textile fastness requirements. Research led to identification of E.I. duPont's Elvax 410, a poly (82% ethylene-co-18% vinyl acetate), as the primary print binder candidate for fabric xerography. Carrier beads compatible with the Elvax 410 resin are currently being developed by Olin-Hunt Chemical Company.

A spun laced polyester nonwoven servicing the mattress pad cover market was successfully bound with two resins via powder spray gun deposition. The Nordson Spray Booth System, currently used to coat metals, proved adaptable to continuous powder spraying of the nonwoven. The two identified resins were Eastman Chemical's FA-252 (a modified, thermoplastic polyester) and H. B. Fuller's IF-3237 (a modified, thermoset epoxy). Both resins imparted comparable strength properties to the plant standard (bound by a wet process) in the machine direction, and ~2X superior properties in the cross-machine direction. The moduli of the SOS fabrics were lower than that of the standard in the

machine direction, yielding a much softer, less-stiff product. Pilling performance was comparable to the standard.

Fluoropolymer (FP) powder finishing of a polypropylene (PP) nonwoven with the same Nordson spray gun technology also proved feasible. Several FP's were supplied to the project in solid form by the 3M Company, including the material currently applied commercially from aqueous emulsion by the participating plant partner. The plant FP proved difficult to handle as a SOS powder, however, presenting aggregation/clumping problems in grinding and channeling/lack of fluidity problems in attempted spraying. Further research will be necessary to circumvent the hygroscopic nature of the material and the attendant problems.

Two main-component size candidates for ring-spun polyester/cotton yarns were evaluated: Carboset 525 (a complex acrylic, B.F. Goodrich) and Eastman WD (a modified polyester, Eastman Chemicals). Numerous blends of the two candidates with various melt modifiers were researched to reduce melt viscosities, improve flow and improve film properties. Optimum sized yarn properties (high abrasion resistance, low degree of "hairiness") were achieved on the electrostatic fluidized bed SOS application system (Electrostatic Technologies, Inc.) with an Eastman WD/adipic acid blend in a 60:40 weight ratio. A dual, grooved Teflon wipe-roll system was developed to spread the thermoplastic melt film and align the yarn hairs with the axis. Further research leading to proof of concept trials will be necessary to further improve film strength properties (and hence abrasion resistance), improve through-package deposition uniformity and regulate across-bed uniformity.

Preliminary research was conducted to identify 100% solid liquid resin candidates for solid shade coloration of fabric. The screened materials consisted of oligomeric/catalyst components which affected film cure without effluent, i.e., no volatile solvents were included. Blends of Lord Corporation's B3693-44 Photoglaze resin (a poly(urethane-acrylate)) with Sartomer's polyethylene glycol 200 dimethacrylate yielded UV-cureable films that had sought visual and qualitative properties. A framework was thus set for future research in this area.

INTRODUCTION AND BACKGROUND

Phase I of DOE Contract No. EY-76-S-05-5099 with Georgia Tech, "Energy Conservation in the Textile Industry", revealed that approximately 60% of the energy consumed in the industry is spent on wet processes (~37 x 10^6 BOE/year, Appendix 7). The energy is consumed in applying the desired chemicals (sizes, dyes, finishes, etc.) to the textile substrate from an aqueous bath, fixing the chemicals to the fiber by thermal energy (steam or dry heat), scouring/washing to remove loose chemicals and drying.

Most of the energy in such processes goes toward heating and evaporation of water. Water has a high latent heat of vaporization (~1000 BTU/lb. of water) and each pound of water requires a BTU to raise its temperature 1°F at 100% efficiency. At the boil, where many wet processes are conducted, the efficiency is considerably less than 100% due to the exponential nature of the vapor pressure as the liquid approaches the phase transition point. Consequently, the bulk of the energy conservation research has been directed toward reducing the volume of water required in textile wet processes or toward substituting solvents (which have a lower latent heat of vaporization than water.)

Georgia Tech has been instrumental in developing several technologies under DOE funding that reduce water volumes and energy consumption in textile wet processes. In the concept of dyebath reuse, a simple analytical scheme was developed to allow coloration of multiple batches of material from a single dyebath.

Bump-and-run technology allowed the dyebath temperature to drift during the hold cycle after "bumping" the system to the boil. In Machnozzle predrying, sonic steam flows were utilized to mechanically remove water from fabrics with little thermal loss.

More recent research at Georgia Tech, co-funded by the State of Georgia and industry, has concentrated on solvent dyeing and foam coloration. Rhone-Poulenc's STX process, in which a 90:10 volume mixture of perchlorethylene:methanol is used as the dyeing medium, has been successfully adapted to coloration of nylon carpets, polyaramid fabrics and PBI fabrics. Foam (in which air is substituted for the bulk of the water) dyeing of carpets is currently being investigated with emphasis in avoiding a final afterscour/drying step in the process. The latter constitutes a major energy consumer in current foam processes.

Although all of the developed processes to date have been extremely successful in reducing energy consumption via water volume reduction or solvent/air substitution with widespread commercial acceptance, liquids are still involved in all of the systems. Theoretically, the optimum method for applying chemicals to a surface in an energy-efficient manner is via solid-on-solid (SOS) processing. In the simplest concept of SOS, powdered chemicals containing a thermoplastic polymer component are deposited on the substrate through electrostatic attraction, followed by dry thermal flow of the polymer. In other words, the SOS concept avoids any use of extraneous liquids in the chemical application process, and reduces energy consumption by eliminating the necessity to elevate the temperature of, and to evaporate, liquids. The technology supercedes so-called low wet

pickup (WPU) systems, such as foam, film and ink applications, which still require water or solvent.

The metal industry is farthest advanced in embracing the SOS concept for coloration. Commercial processes now exist for SOS electrostatic painting of automobile body components, coating of wires and cables and printing of aluminum cans. Resins are used with the colorants to bind them together and to the metal surfaces on thermal curing.

PROJECT REVIEW

Research during the first year of the project revolved around identification of material candidates for the various areas and of powder application processes from other (mainly metal and paper) industries that presented the possibility of translation to textiles. As a result of these preliminary studies, the following conclusions were reached:

1. The two existing technologies for feasible, direct powder deposition on textiles were electrostatic fluidized bed (slashing of yarns, Electrostatic Technologies, Inc. (ETI) of Connecticut) and powder spray gun deposition (finishing/binding of fabrics, Nordson, Inc. of Ohio). Cooperative agreements were secured with the two companies and small operating systems were installed. Fig. 1 gives the basics of a typical electrostatic powder coating spray gun and Figs. 2-3 detail the installed Nordson spray gun booth system (now utilized without electrostatics). Fig. 4 schematically defines the operation of the ETI electrostatic fluidized bed, while Figs. 5-6 detail the installed hardware and the single-end slashing line at Georgia Tech.

2. Controlled room conditions were found to be necessary to avoid powder clumping/packing, especially with those of a more hydrophilic chemical nature. The application room was therefore modified with a free-standing heating/air conditioning system that would control temperature/humidity 24 hours per day, seven days per week. A dryer/filter system was also installed in the compressed air feed line to the two powder deposition systems in order to avoid both moisture and oil impurities. These actions

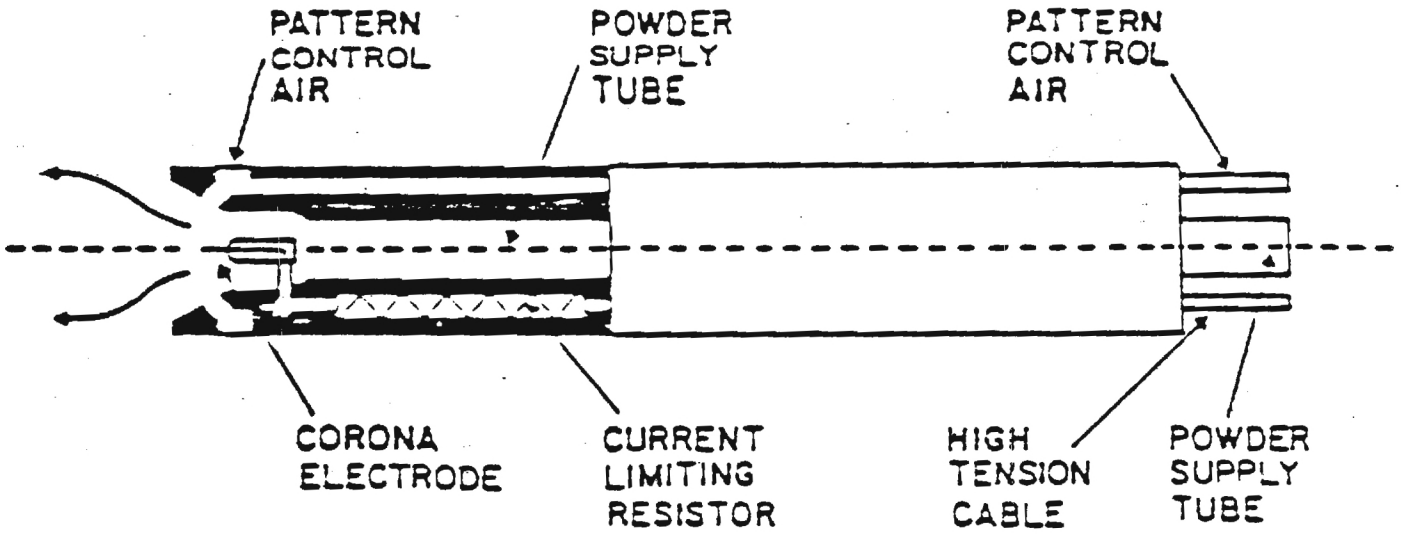


Fig. 1

Construction of a Typical Electrostatic Powder Coating Spray Gun

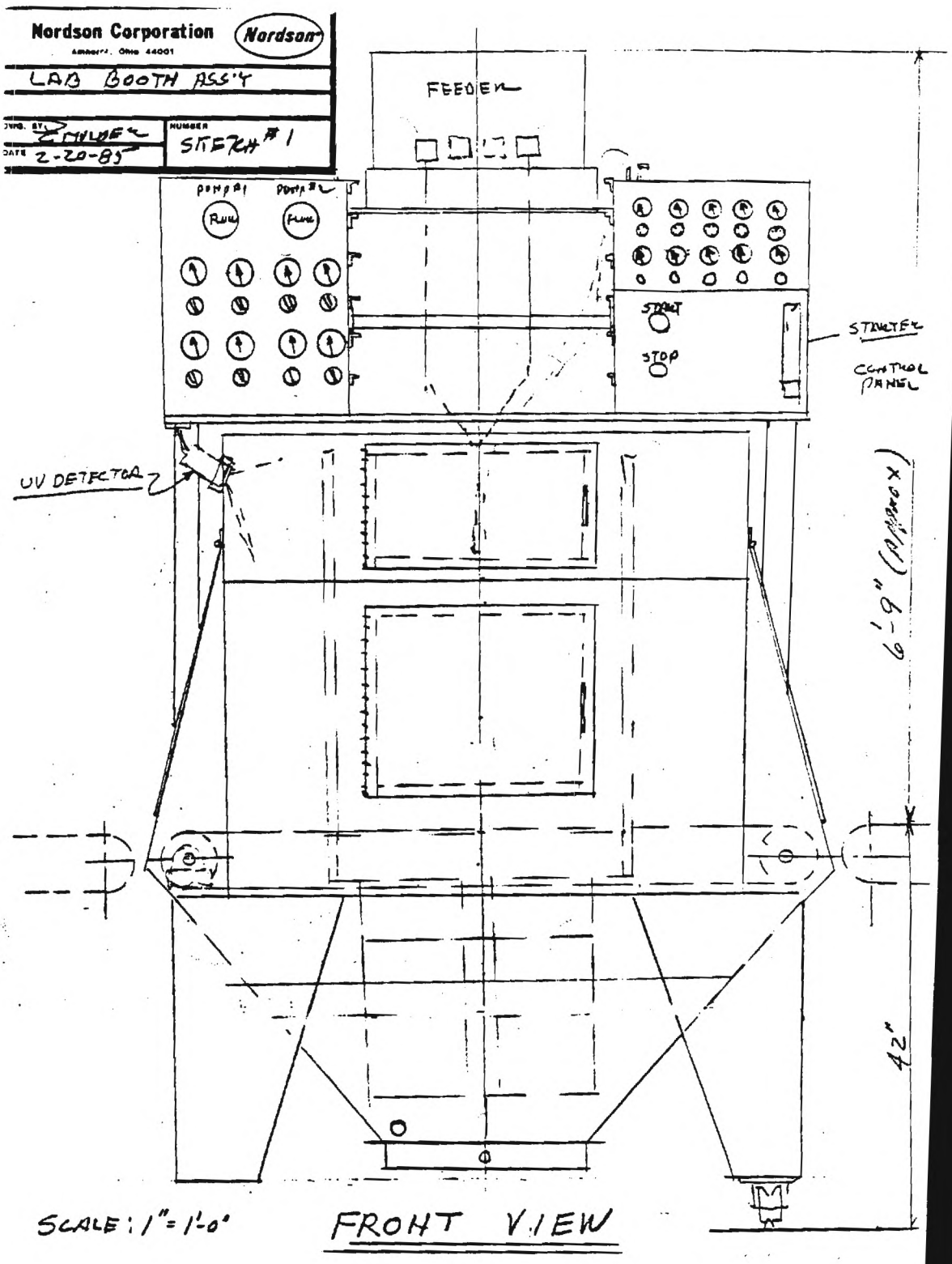
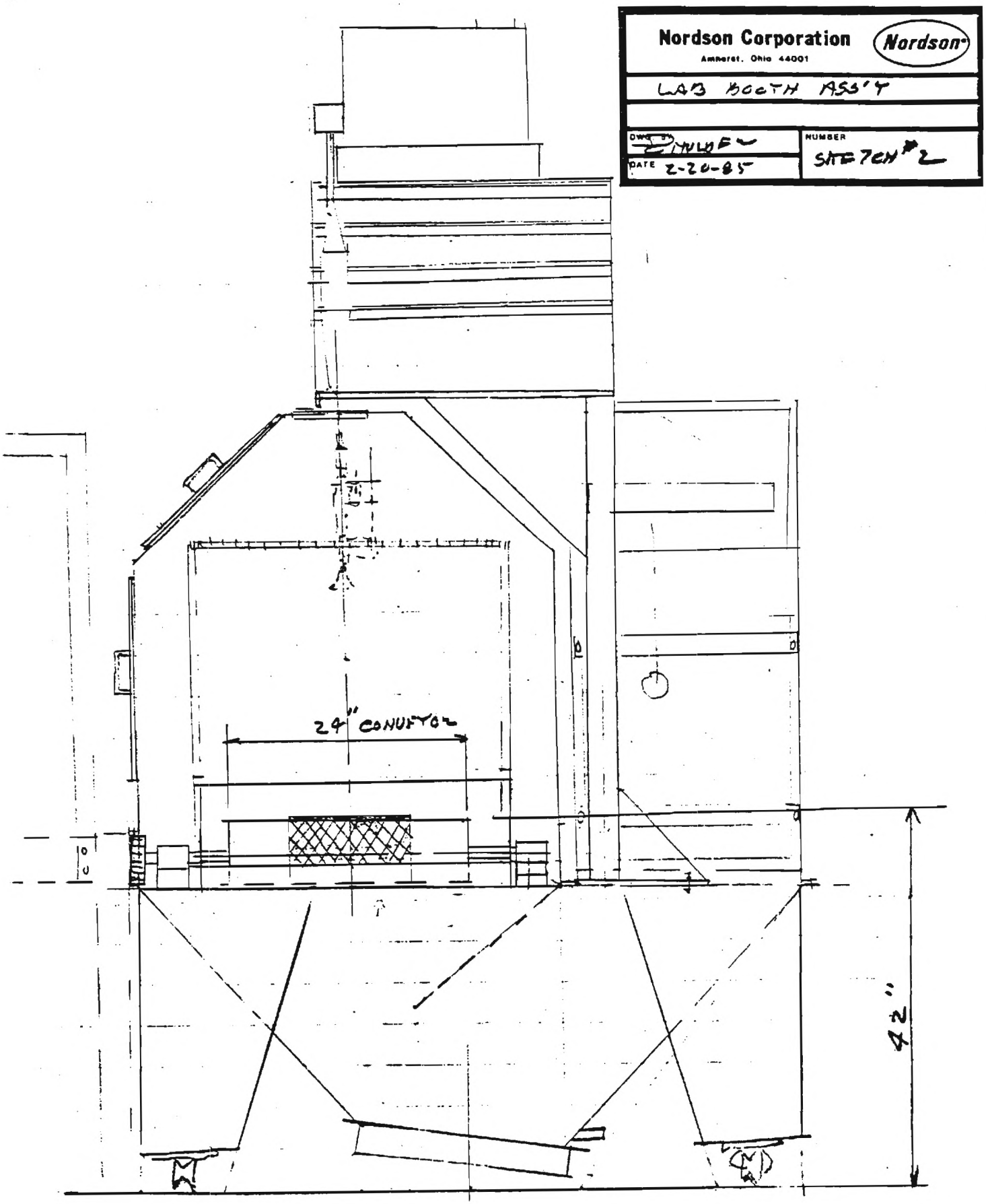


Fig. 2

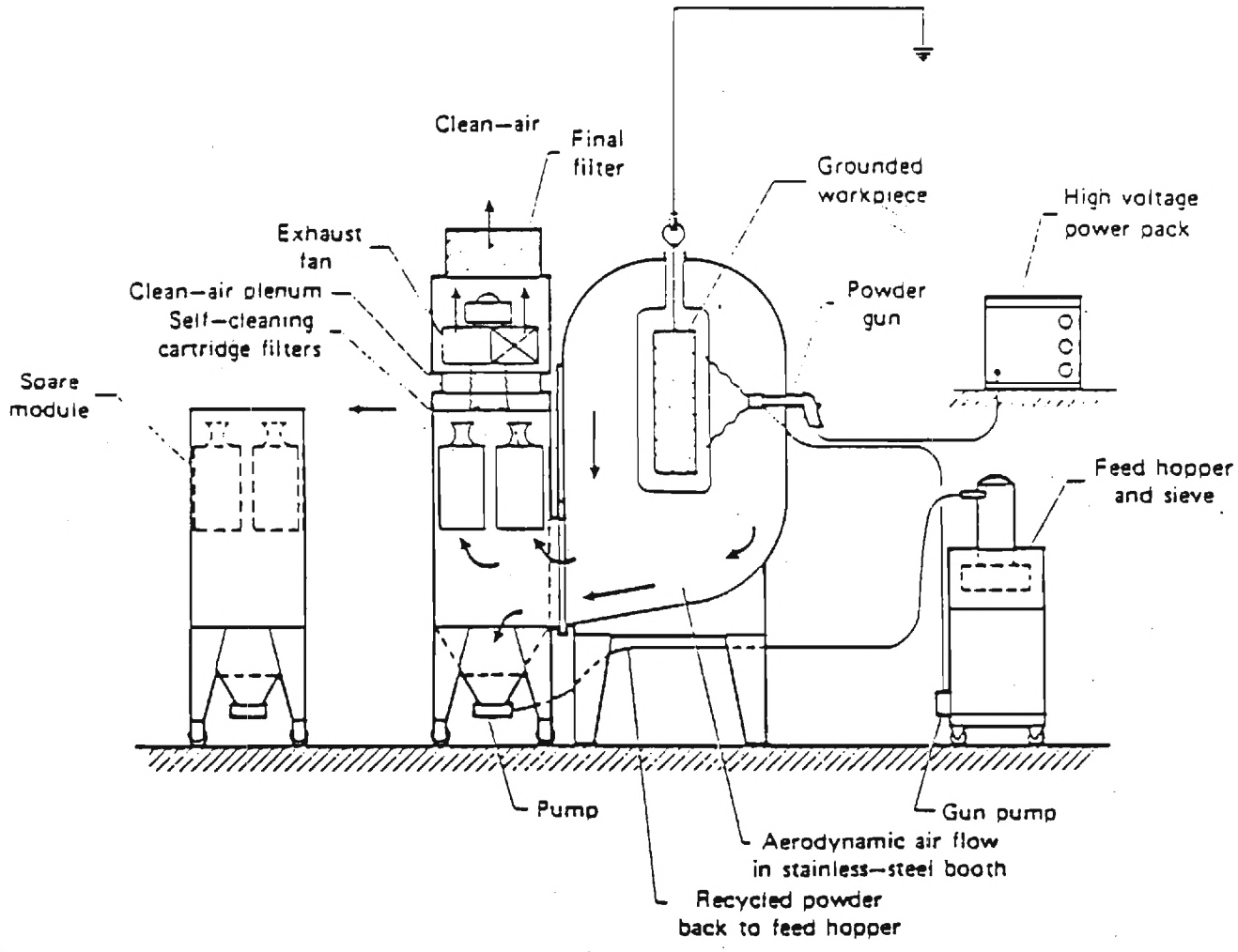
Schematic of Nordson Spray Gun Booth Assembly



SCALE: 1" = 1'-0"

END VIEW

Fig. 2 (con't.)



ELECTROSTATIC SPRAY BOOTH

Fig. 2 (Con't.)

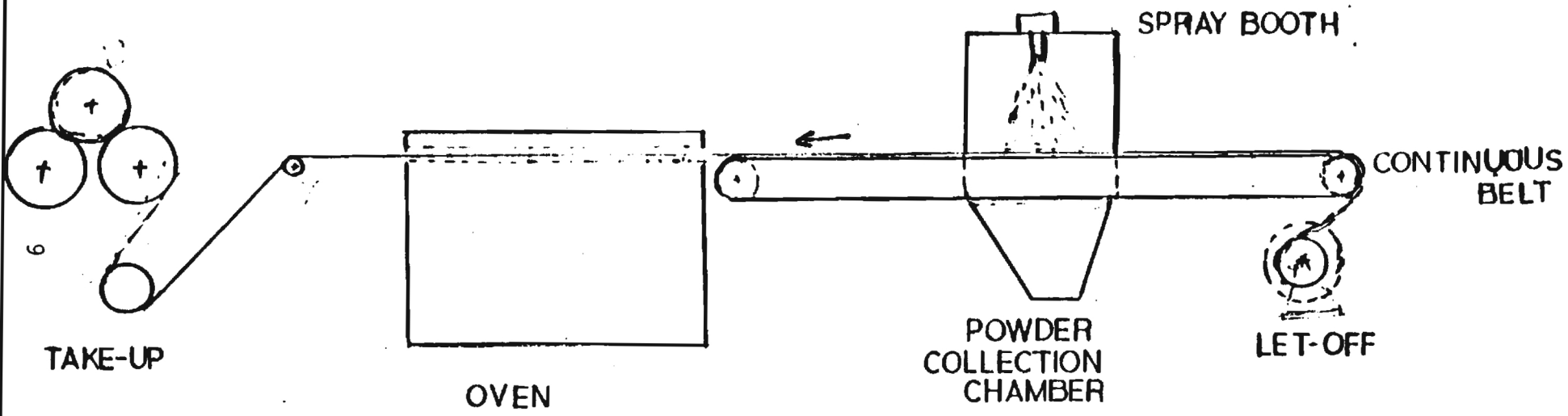


Fig. 3

Nordson Spray Gun Booth System Installed at Georgia Tech

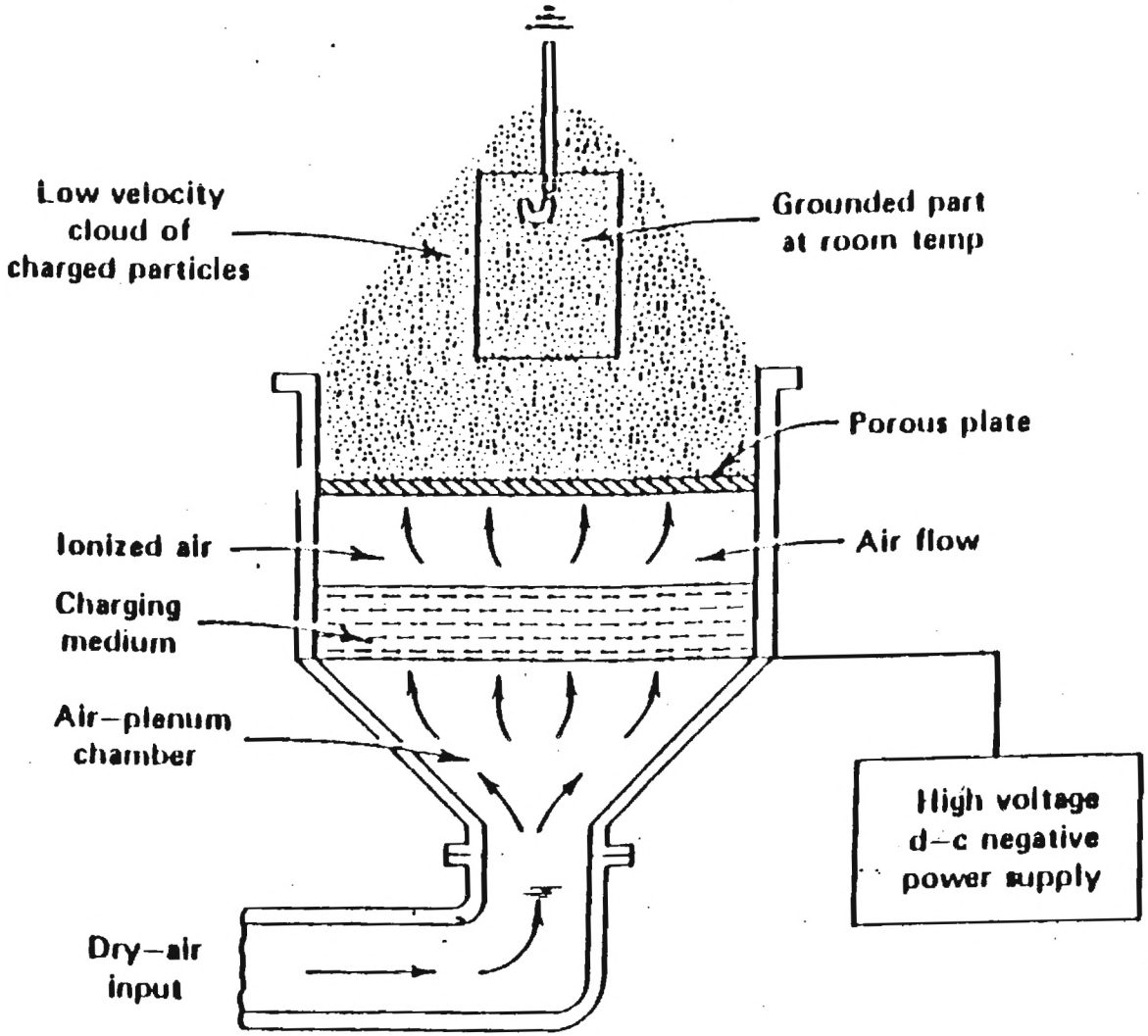
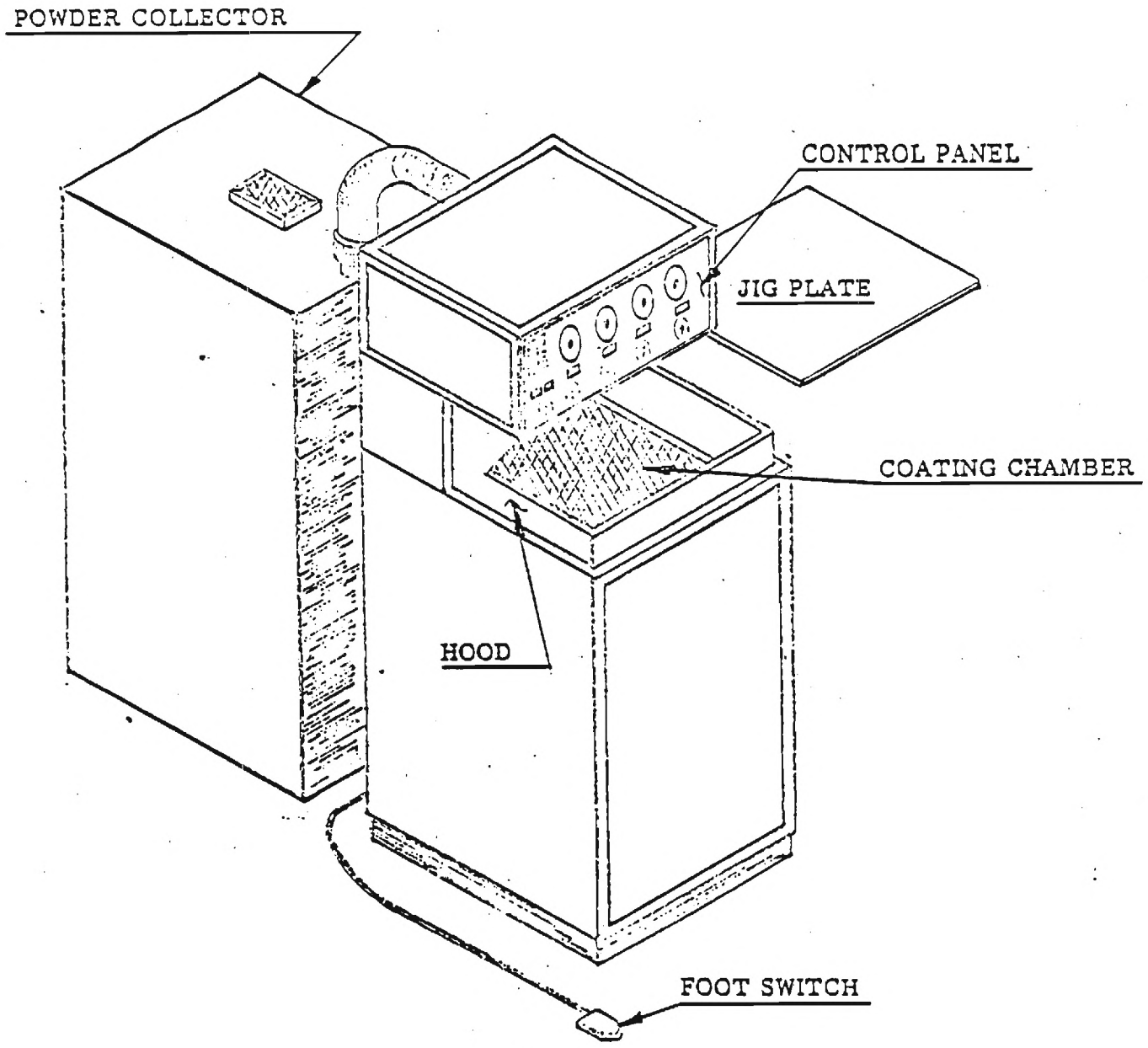


Fig. 4

Schematic of ETI Electrostatic Fluidized Bed

Fig. 5

ETI Electrostatic Fluidized Bed Lab System



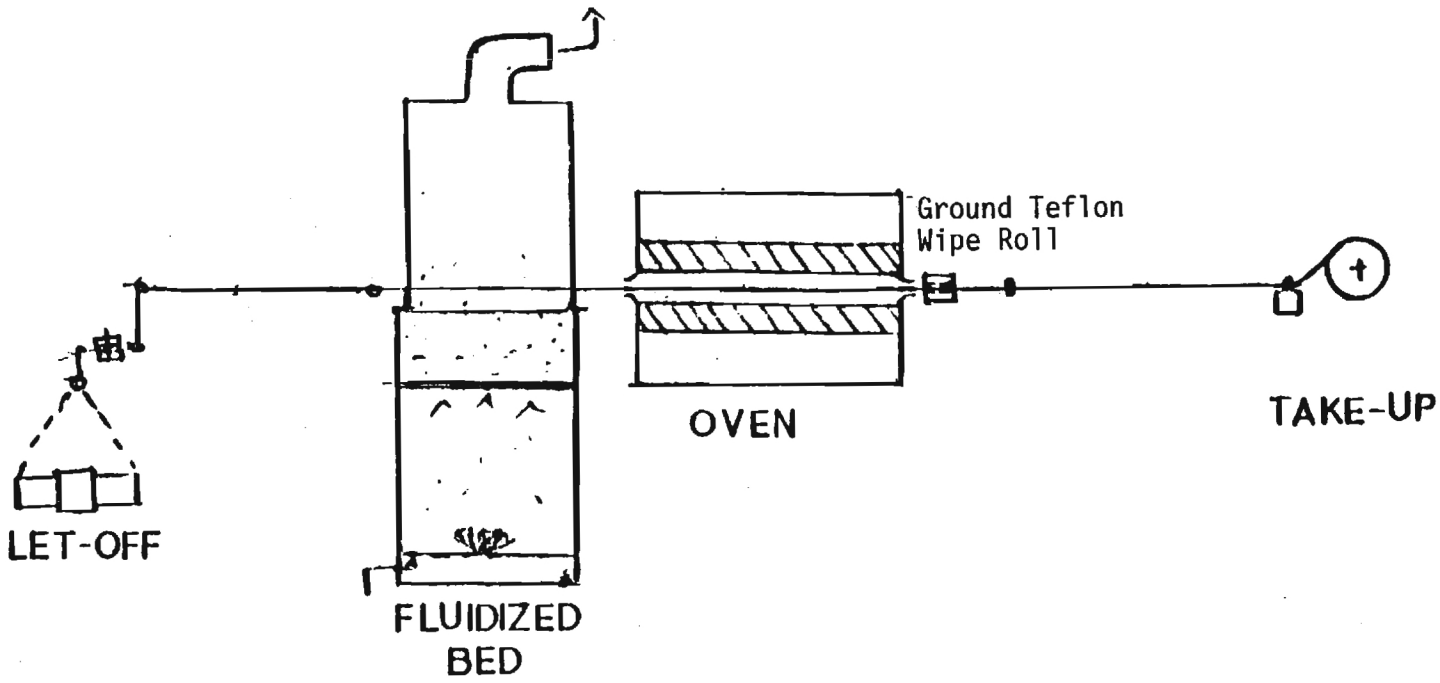


Fig. 6

Single-End Slashing Line Assembled at Georgia Tech.

alleviated the observed the powder packing problems.

3. For screening materials in modified xerography printing of polyester/cotton blend sheeting fabrics, a Haloid single-page, batch copier with selenium plate was acquired (Figs. 7-8). A non-optical, continuous xerographic process was also built by utilizing key components of a commercial paper xerography machine (Fig. 9).

4. In the xerographic printing research, excellent fabric prints were obtained utilizing typical paper toner/carrier systems. However, the binder component of the paper toners (suspected to be styrene-acrylic copolymers) did not meet traditional textile testing requirements (crock fastness, light fastness, wash fastness, etc.). Materials development was therefore required to generate powderable, thermoplastic binders that would function in a transfer xerography mode, and would exhibit acceptable textile properties once applied to the fabric.

5. An unoxidized, low-density polyethylene powder (FN-500, U.S. Industries) was shown to possess strong potential as a softener for polyester/cotton blend sheeting fabric. The Nordson spray gun system was used to apply to powder to the fabric, followed by thermal curing. The research was discontinued, however, since softeners are generally co-added to the fabric in commercial operations as part of another process, e.g., permanent press finishing (continuous) or dyeing (batch). Thus no energy penalty exists in applying softeners to fabrics in existing operations, and interest of the Industrial Working Group advising the research project (Appendix 1) was low.

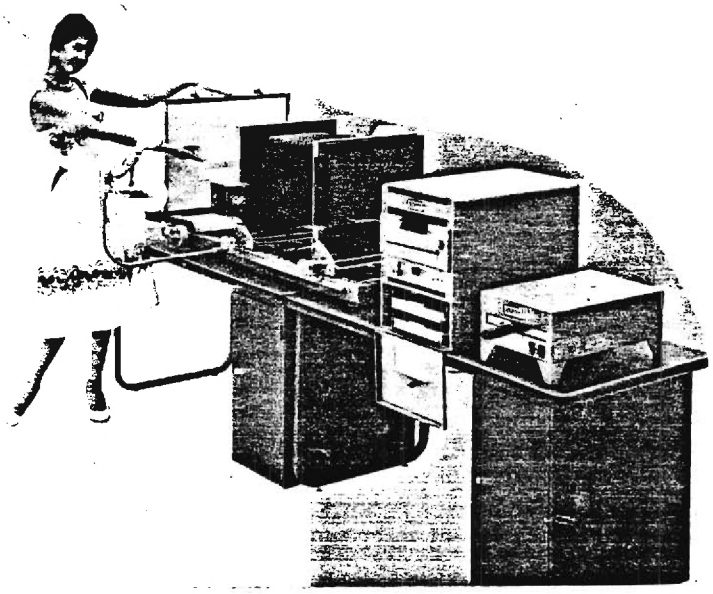
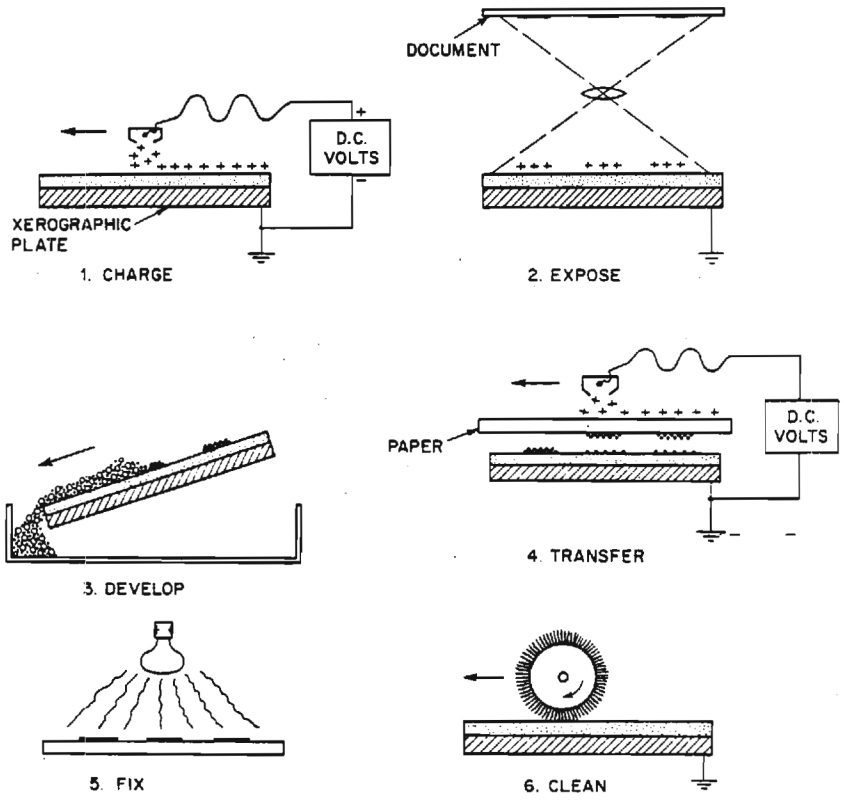


Fig. 7

Physical Arrangement of Haloid Batch Xerox Copier

Fig. 8

Basic Steps Encompassed by the Haloid Xerography Copier



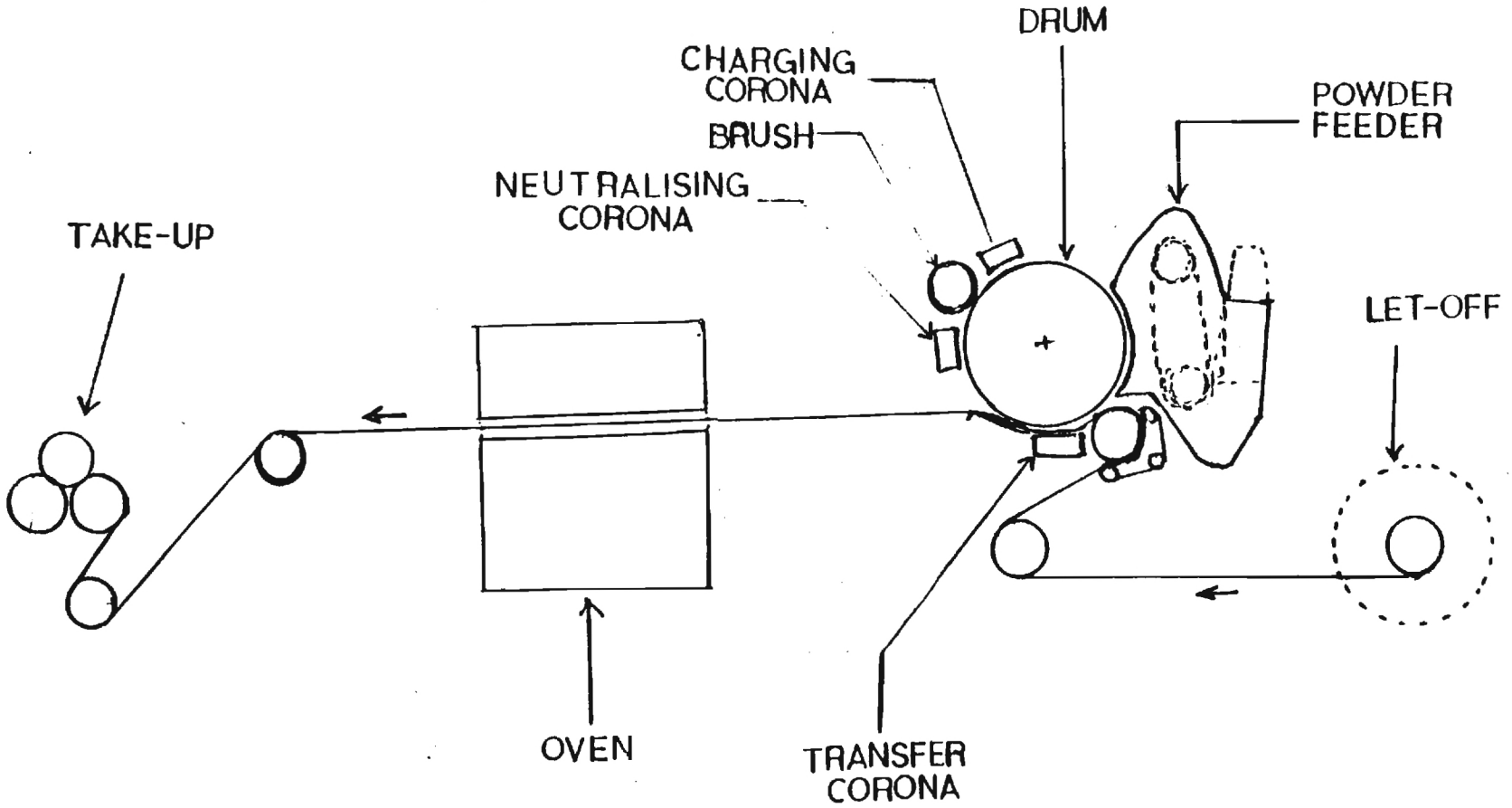


Fig. 9

Continuous, Non-Optical Xerographic Process Built at Georgia Tech

6. Solid-shade coloration of fabrics with powders applied through the Nordson spray gun technique failed for several key reasons:

a. Unlike (colorless) finishes, colorant/binder systems must form totally-uniform films on fabrics to give visual defect-free shades, i.e., to avoid slips, streaks, splotches, etc.

b. Although sufficient for finishes, the uniformity of binder/pigment powder deposition on the Nordson system was not at the extreme level required for solid-shade coloration.

c. The melt viscosity level of commercially-available resins that give proper characteristic films were too high (10^5-10^6 Centipoise, or Cps) to exhibit good flow at the fiber-limited cure temperature (<450°F). The result was discontinuous film formation.

After abandoning the powder approach to solid shade coloration, the team approached the Department of Energy on the possibility of expanding the scope of the project to include 100% solid liquid sprays, i.e., oligomeric binders that were still of sufficiently low molecular weight to be in the liquid state at room temperature, but which contain no solvents, water or other potential effluents on curing. Permission was eventually granted by DOE, and a liquid spray system capable of handling relatively high viscosities (< 600 Cps at RT) was obtained from the Nordson Company for the studies.

7. Two thermoplastic size candidates were defined for yarn slashing: Carboset 525 (an acrylic, B. F. Goodrich) and Eastman

WD (a modified polyester, Tennessee Eastman Co.). Melt blends of these two candidates with various organic acids (adipic, sebacic, etc.) also showed promise. Slashing application mode was restricted to the ETI electrostatic fluidized bed system.

8. A powderable fluoropolymer composition (coded FC-214, 3M Co.) was successfully applied via the Nordson gun system both to polypropylene nonwovens and tufted nylon carpets. Barrier properties to water, oil, solvents and isopropanol was sufficient in both products, albeit at a high fluoropolymer level. Since the energy conservation potential and industrial interest was greater for the nonwoven than the carpet application, research on the latter was discontinued in order to concentrate effort on the former.

9. Two resin candidates were defined for chemical binding of nonwovens: Eastman FA-252 (a modified polyester, Tennessee Eastman Co.), and IF-3237 (a modified epoxy, H. B. Fuller Co.). Although the melt viscosities were higher than desired (10^5-10^6 Cps), strong, flexible films were formed, giving adequate binding action in hand-sprayed trials. Further research was required to modify the melt viscosities and pursue other avenues of reaching the sought 10^2-10^3 Cps viscosity range.

With the base of knowledge built during Year 1, Year 2 narrowed to include only those areas with the highest probability of technical success leading to scaled-up, proof-of-concept trials:

- Resin Binders for Xerography Toners
- Xerography Printing of a Polyester/Cotton Woven Sheeting Fabric

- Chemical Binding of a Polyester Nonwoven
- Fluoropolymer Finishing of a Polypropylene Nonwoven
- Slashing of a Polyester/Cotton Staple Blend Yarn
- 100% Solid Liquid Spray Coloration of a Polyester/ Cotton Sheeting Fabric

Research conducted in these areas forms the basis for the remainder of this report.

EXPERIMENTAL RESULTS AND DISCUSSION

RESIN BINDERS FOR XEROGRAPHY TONERS

Although the feasibility of solid shade coloration by powder deposition had been disproven by the end of Year 1, research continued on developing a flowable resin of the proper film/textile properties to act as a toner component in the modified xerography printing of woven textile fabrics. Both new commercial materials as well as melt-blended combinations were screened.

First studies in Year 2 were conducted on duPont's Sclair 2316 resin (a linear polyethylene), along with several blends of duPont's Elvax 410 (a polyethylene-co-vinyl acetate) with other materials. Blending was implemented in an attempt to reduce the melt viscosity of the base Elvax 410 polymer, while maintaining sought print binder film properties:

- Clarity
- Low Melt Viscosity
- Good Adhesion to Substrate

- Good Physical Properties
- Good Heat and Chemical Resistance
- Good Wearability

In this series, various Allied Chemical Co. copolymers (also of the ethylene or ethylene-acrylic copolymer type, but of lower molecular weight than the Elvax 410) were used in an attempt to achieve a 10^3 Cps melt viscosity range. The results are shown in Table 1. The Sclair 2316 material was well above the sought melt viscosity in the virgin state ($1.28 \cdot 10^5$ Cps) and was eliminated from consideration. Blends of the Elvax 410 with several of the Allied materials gave promising melt viscosities in the 2000-3000 Cps range at a 1:1 weight ratio.

A standard Dot Adhesion Test was then conducted with the materials. Table 2 details the procedure, while Table 3 exhibits the adhesion test results for the Table 2 materials, as well as several additional compositions. Several of the low-viscosity blends (Sample Nos. 2, 8-11) gave good adhesion results (>83% effective) comparable to the standard, virgin Elvax 410.

Property sheets were compiled in order to more clearly compare the properties of the various resin candidates and their blends (Table 4). Some of the materials were eliminated in their virgin states due to poor adhesion (e.g., Allied's AC-629), and in blends due to color formation (e.g., 1:1 Elvax 410/AC-629 composition).

Films were melt formed to 5:4:1 and 6:2:2 blends of Elvax 410/AC-400/AC-580 on a heated platen press, and the stress/strain curves were obtained. Table 5 gives the properties of the films compared to Elvax 410 and a standard pigment printing resin

Table 1.

Melt Viscosities[a]. of Various Resin Material Candidates

Sample (#)	Composition	Viscosity (Cps)
1	Sclair 2316[b].	1.28 x 10[5]
2	5:4:1 410[c]. :400[d]. :580[e].	3150
3	6:2:2 410:400:580	3500
4	7:2:1 410:400:580	6010
5	7:1:2 410:400:580	5150
6	8:1:2 410:400:580	9000
7	9:1:2 410:400:580	10000
8	1:1 410:400	2600
9	1:1 410:629[f].	2870
10	1:1 410:580	1910

a. Viscosity measured with a Model LVT Brookfield Viscometer, spindle 4, at 200° C.
b. A linear polyethylene, duPont.
c. A poly(ethylene-co-vinyl acetate), duPont.
d. Also a poly(ethylene-co-vinyl acetate), Allied.
e. A poly(ethylene-co-acrylic acid), Allied.
f. An oxidized polyethylene, Allied.

Table 2.

Standard Dot Adhesion Test for Screening Materials

1. Apply ~ 100 dots of melt to Mylar film.
2. After solidification, cover with tape strips.
3. Record number of dots removed by tape.
4. Flex film containing post-tape dots, and record number.
5. Calculate percent of dots remaining adhered after 3, 4.

Table 3. Dot Adhesion Test Results for Resin Candidates

Sample (#)	Composition	# Drops Original	# Removed by Tape	% Removed by Tape	# Removed by Flex	% Removed by Flex	Total Adhesion	Melt Viscosity (Cps)
1	Elvax 410	100	3	3	8	8	89	6.28 x 106
2	50% Elvax 410 50% AC-580	99	7	7.1	5	5.4	87.9	1910
3	50% Elvax 410 50% AC-629	98	28	28.6	61	87.1	9.2	2870
4	50% Elvax 410 50% AC-400	100	1	99	46	46	54	2600
5	AC-629[a]	100	72	72	16	57	12	200
6	AC-580	100	38	38	20	32	42	650
7	AC-400	100	4	96	61	63	35	610
8	5:4:1 Elvax:400:580	100	0	0	3	3	97	3150
9	6:2:2 410:400:580	100	0	0	12	12	88	3500
10	7:2:1: 410:400:580	100	0	0	16	16	84	5150
11	7:1:2 410:400:580	100	0	0	17	17	83	6010

[a]. An oxidized polyethylene, Allied.

Table 4. Direct Comparison of Properties of Candidate Resins

Product	Chemical Composition	Tm (°C)	Melt Viscosity (Cps)	Observations on Press-Out	% Total Adhesion	Comments
FA-252 Eastman	Aromatic Polyester	110 (mfg) 125 (exp)	1.4×10^5 @ 210°C (mfg)	w/mold release very flexible & milky	91	Powder
Elvax 410 (duPont)	82% Ethylene 18% Vinyl acetate	78-83 (exp)	6.28×10^6 @ 95°C (exp)	clear, very flexible; great hand	89	Pellets
MU-760 (USI)	82% Ethylene 18% Vinyl acetate	----	2.01×10^6 @ 150°C (exp)	------	60	Powdered form
FE-532 (USI)	91% Ethylene 9% VA	96 (exp)	N/A	clear, flexible looked like Elvax	no adhesion	------
AC-400 (Allied)	86% Ethylene 14% Vinyl acetate	95 (mfg) 86 (exp)	550 @ 140°C (mfg)	cloudy, somewhat flexible, but can't peel sections, just chunks	35	Possible Viscosity Modifier
AC-580 (Allied)	Ethylene/ Acrylic Acid	102 (mfg) 96 (exp)	650 @ 140°C (mfg)	translucent somewhat flexible great flow	42	Acid no. = 75 Possible Viscosity Modifier
AC-629 (Allied)	Oxidized Polyethylene	104 (mfg) 98 (exp)	200 @ 140°C (mfg)	translucent somewhat brittle can't remove film	12	Possible Viscosity Modifier
Sclair 2316 (duPont)	High Flow Linear Polyethlene	125 (exp)	1.28×10^5 @ 200°C (exp)	transparent, not flexible & rubbery enough	no adhesion	Not viable; not rubbery or elasitc enough
50/50 Elvax 410/ AC 629	EVA ox. PE	----	2870 @ 200°C (exp)	translucent, good flow, too brittle, not enough film strength	9.2	yellowing seen when checking melt viscosity

Table 4. Con't.

50/50 Elvax 410/ AC-580	EVA EAA	96 (exp)	1910 @ 200°C (exp)	milky, film strength better, but not rubbery enough	87.9	Lowest viscosity w/highest adhesion, just not rubbery enough
50/50 Elvax 410/ AC-400	EVA EVA	-----	2600 @ 200°C (exp)	milky, good film, rubbery strength	54	----
5:4:1 Elvax 410/ AC-400/ AC-580	EVA EVA EAA	92 (exp)	3150 @ 200°C (exp)	milky, flexible good film properties,	97	----
6:2:2 Elvax 410/ AC-400/ AC-580	EVA EVA EAA	96 (exp)	3500 @ 200°C (exp)	milky, flexible good film properties, more elastic than 5:4:1	84	----
7:2:1 Elvax 410 AC-400 AC-580	EVA EVA EAA	-----	6% @ 200°C (exp)	milky, hard to peel from foil even w/ TFE, film strength seems better	84	----
7:1:2 Elvax 410 AC-400 AC-580	EVA EVA EAA	----	5150 @ 200°C (exp)	no significant differences from 7:2:1 seen	83	----

Table 5. Stress/Strain Properties of Blended Resin Candidates

	Tensile Strength at Break (lb/in^2)	Strain at Break (% Elongation)
5:4:1[a.]	394.5	26.05
6:2:2[a.]	478	35.40
Elvax 410	212.7	233.33
Hycar 26120	334.3	270.14
7:2:1[a.]	249.8	43.80
7:1:2[a.]	351.9	56.30
8:1:2[a.]	442.1	88.8
9:1:2[a.]	475.9	80.3

a. Mass ratio of Elvax 410: AC-400: AC-580

currently used by the textile industry, Hycar 26120 (a complex acrylic, B.F. Goodrich). The latter film was laid from an emulsion utilizing a Gardner knife, and was considered a "standard" in terms of desired film properties (Fig. 10).

As seen in Fig. 10, the desired print binder film should have an extremely low initial modulus, an extremely high elongation-to-break with a definite yield point and a moderate tensile strength. By blending the Elvax 410 with the lower molecular weight materials, the films became more brittle, with a higher tenacity and drastically-reduced elongation-to-break. The 5:4:1 and 6:2:2 blends were thus eliminated as resin candidates due to their brittle behavior.

A higher percentage of Elvax 410 in the blends was postulated to regain the lost mechanical properties, while striking an intermediate melt viscosity. Blends of 7:1:2, 7:2:1, 8:1:2 and 9:1:2 (Elvax 410:AC-400:AC-580 mass ratio) were then produced, films were pressed and stress/strain curves obtained. The tabulated results are contained in Tables 1 and 5, with complete physical data for the 7:1:2 and 7:2:1 blends included in Table 4.

In Figs. 11-15, the various materials are compared in the plotted stress/strain curves. Fig. 11 shows the relationship between the "standard" Hycar resin and the candidate powdered resin, Elvax 410. Although the latter material showed a much higher initial modulus than the former, the elongation-to-break and breaking tenacities were in the same ranges, with the former property (most important) showing only a 36.8% difference (Table 5).

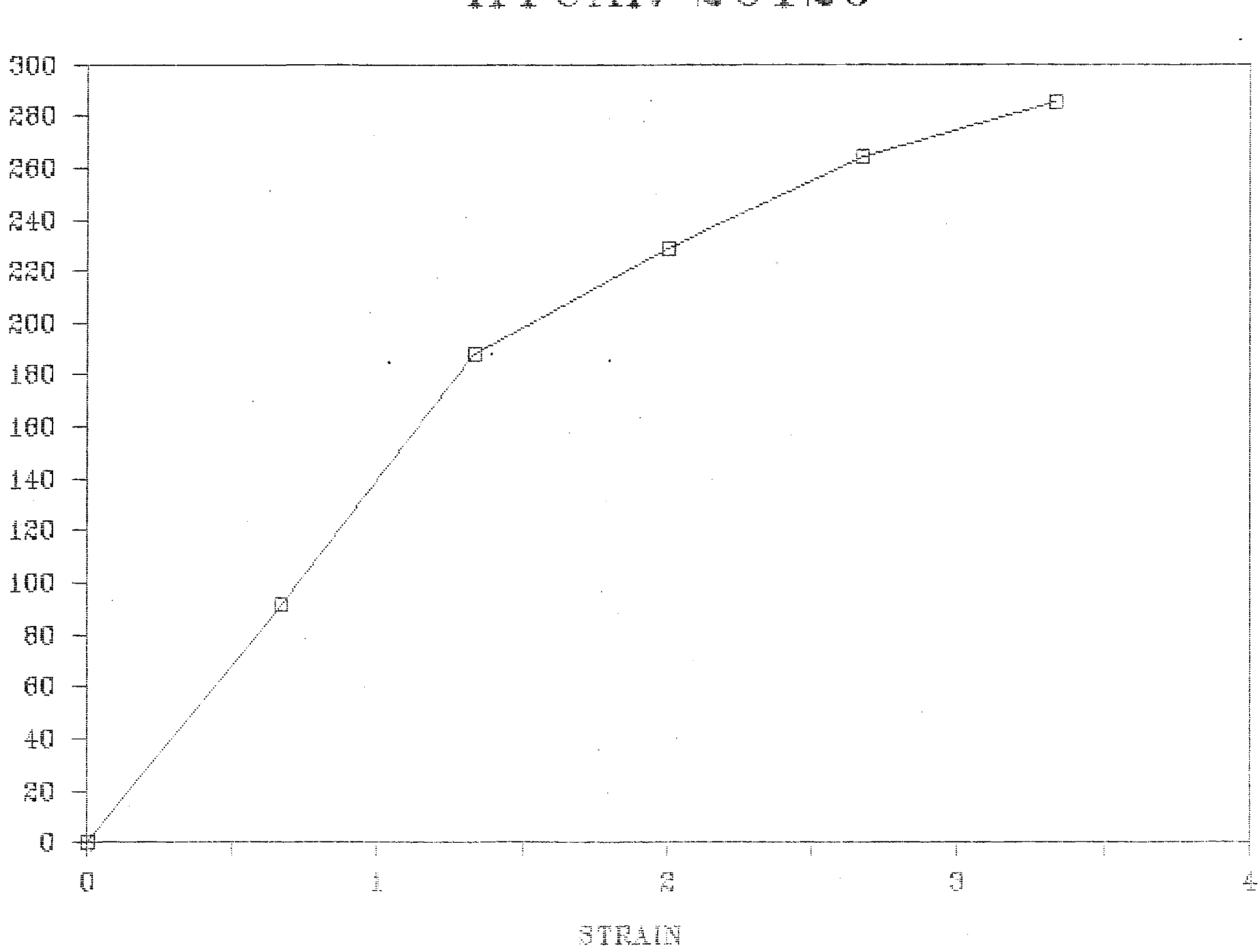


Fig. 10 Stress/Strain Behavior of "Standard" Hycar 26120 Resin Film

Fig. 11

Film Stress/Strain Curve Comparisons of Hycar 26120 and Elvax 410 Resins

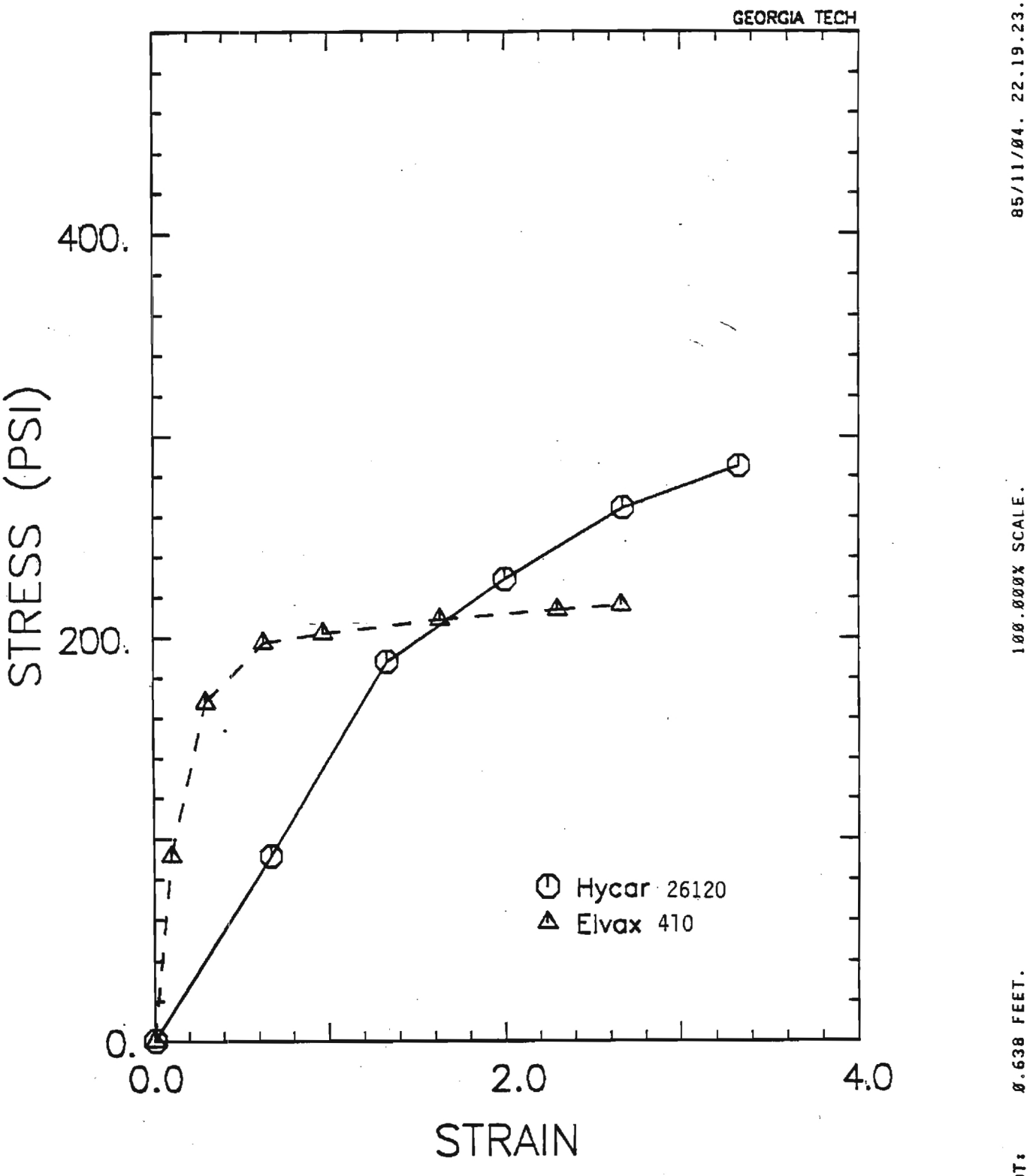


Fig. 12

Film Stress/Strain Curve Comparisons of 7:1:2 Blend and Hycar 26120 Resins

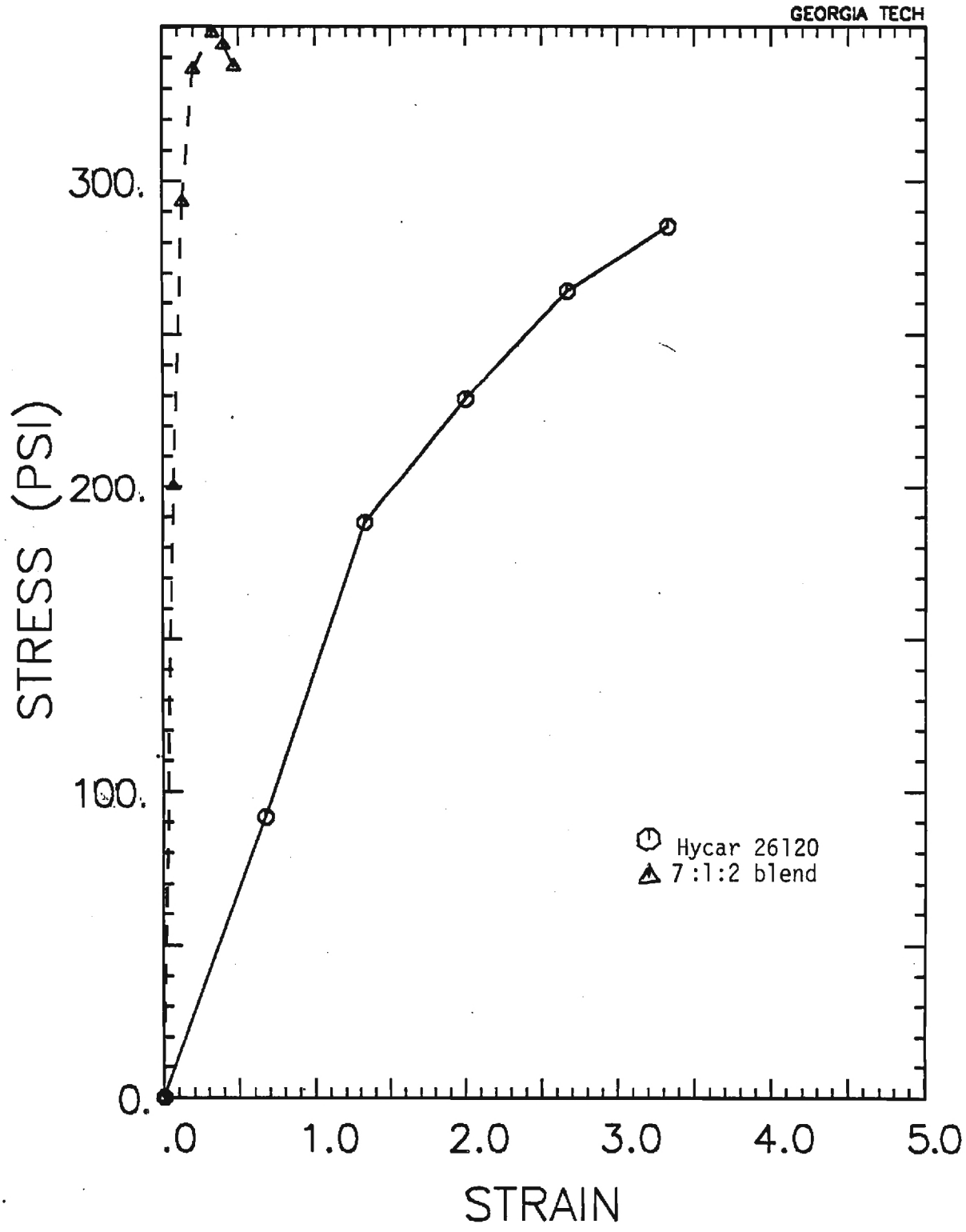


Fig. 13

Film Stress/Strain Curve Comparisons of 8:1:2 Blend and Hycar 26120 Resins

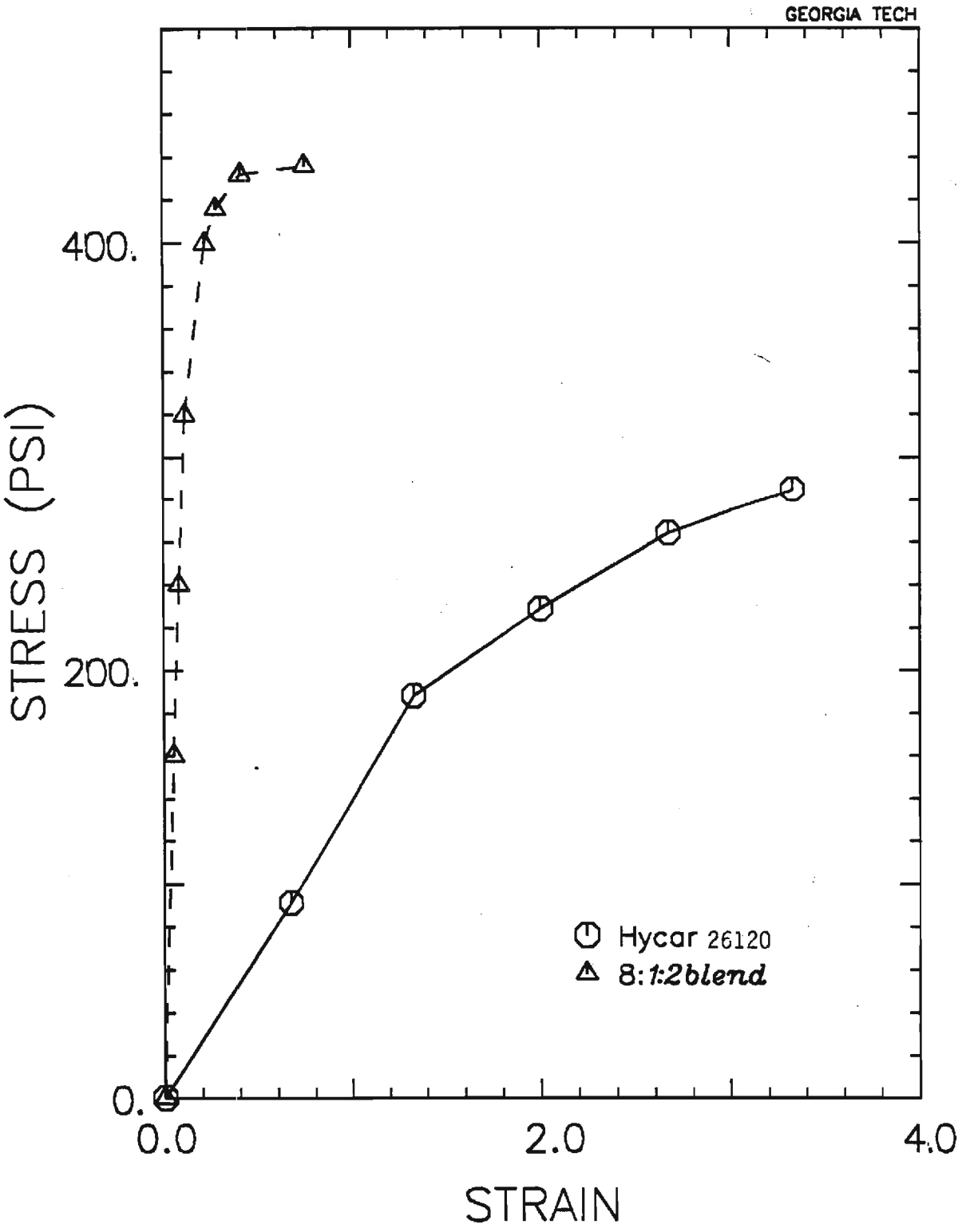


Fig. 14

Film Stress/Strain Curve Comparisons of 9:1:2 Blend and Hycar 26120 Resins

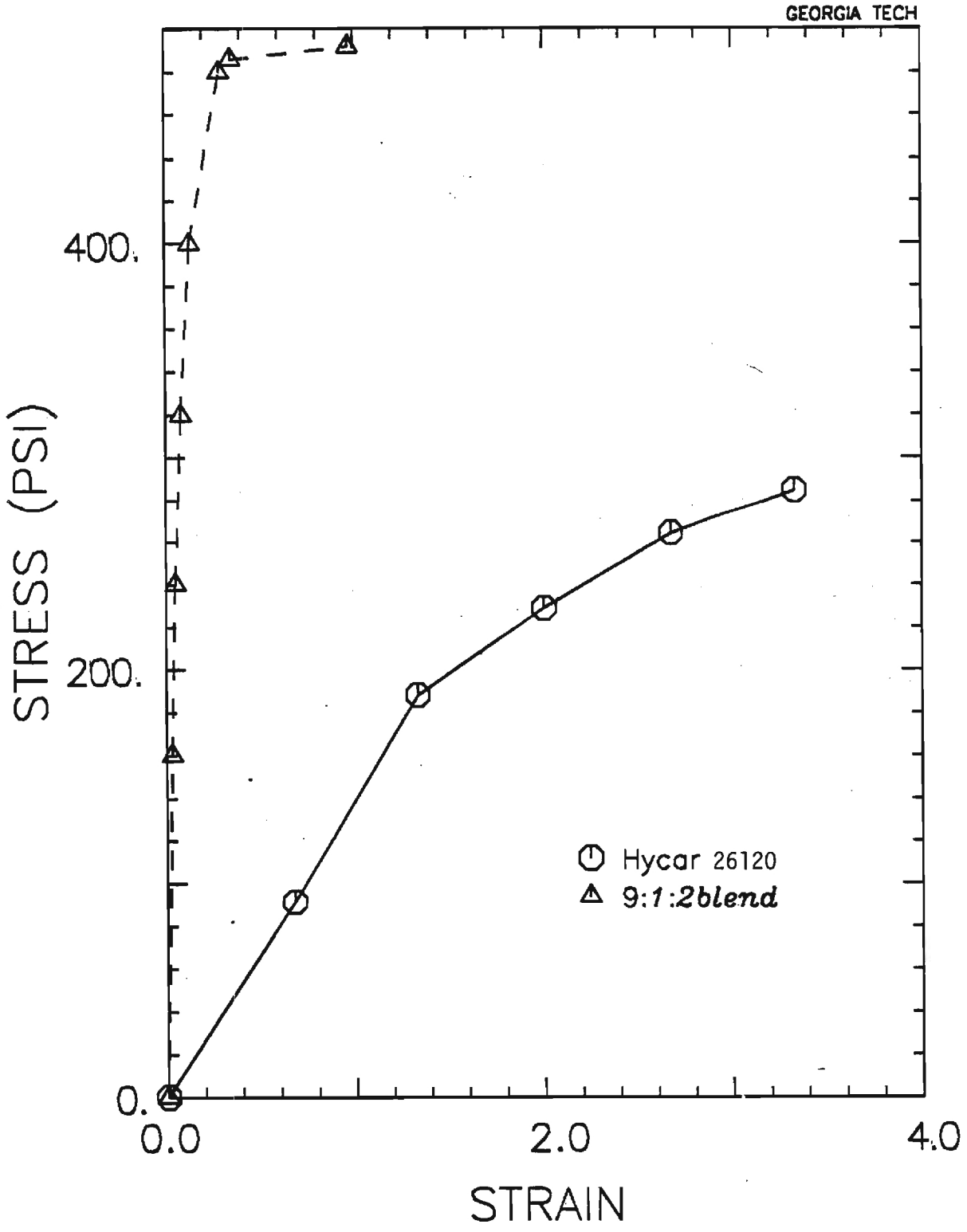
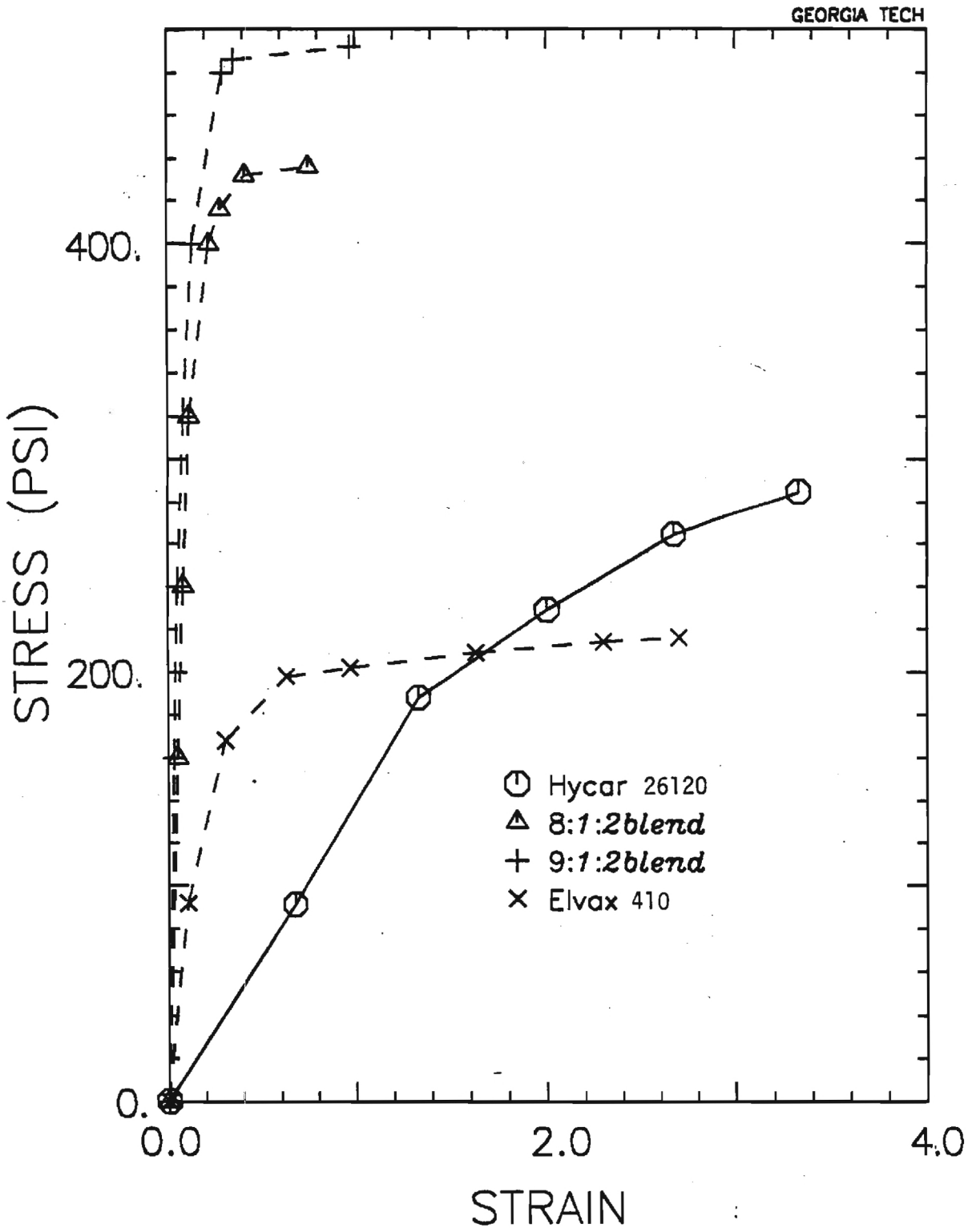


Fig. 15

Film Stress/Strain Curve Comparisons of Blended and Virgin Resins



The film property comparisons for the 7:1:2, 8:1:2 and 9:1:2 blends with the Hycar were not favorable (Figs. 12-14, Table 5). Even small amounts of the AC-400 and AC-580 materials blended with the Elvax 410 caused brittleness to develop, with extremely high initial moduli and ultimate tenacities in the blended materials, and with extremely low elongations-to-break. Fig. 15 gives a direct comparison of the stress/strain curves of the 8:1:2 and 9:1:2 blends with virgin Elvax 410 and the Hycar 26210.

Despite the lack of promise exhibited by the blends, it was decided to spray one of the powders on the polyester/cotton sheeting material to assess the aesthetic qualities. The 7:1:2 blend was merged in the melt with a small quantity of a blue tracer pigment. The blend did not cryogenically grind as well as earlier binders, and the particles were irregularly shaped. The cut retained on a 120-mesh screen was fed into the fluidized bed hopper of the hand-held Nordson gun system, sprayed onto the fabric positioned on a wooden embroidery hoop and the binder flowed at various times/temperatures in a static air oven. Table 6 details the cured fabric appearance/aesthetic properties.

Good flow was obtained at 155°C/30 sec., with total penetration of the resin system (bleed through) and 360° encasement of individual yarns. Around the pigmented area, an oil or wax material could be seen diffusing away from the area. The material was likely one of the low viscosity blend components (AC-400 or AC-580) separating from the melt and flowing with greater ease through the textile structure.

Based on the cumulative research results, the melt blend

Table 6. Time/Temperature Study with 7:1:2 Blend Resin on Polyester/Cotton Sheeting

Oven Temp. (°C)	Dwell Time (sec.)	Fabric Appearance/Aesthetics (Observations)
140°C	30 sec.	Heathered, melt but no flow, rough, no waxing or bleed-thru.
	35 sec.	Heathered, flow better, some bleed thru, no waxing. Not as rough.
	40 sec.	Heathered, some waxing. Fabric seems somewhat stiffer, smoothest yet.
150°C	35 sec.	No big difference from 30 sec. at 140°C.
155°C	30 sec.	Similar to 40 sec. at 140°C
	60 sec.	Total penetration into fabric. Bleed thru and waxing both very evident. Windowing not apparent, fabric stiff, but feels soft.

approach was abandoned, and the virgin Elvax 410 was chosen as the primary candidate for textile toner development. Although concern remained with the high melt viscosity of the material, it gave the best choice of properties (grindability, film properties, clarity, textile properties, etc.) of any of the tested formulations.

XEROGRAPHY PRINTING OF A POLYESTER/COTTON SHEETING FABRIC

Research was concentrated initially on achieving clear, well-defined prints with no "edge effects" on the polyester/cotton sheeting utilizing the Haloid process (Figs. 7-8). Two toner/carrier systems designed for paper xerography were first investigated:

- o Red Toner No. 22-144, Haloid Xerox, Inc.
- o Black Toner Type 10, Xerox Corp.

Successful prints were achieved using the two paper toners separately and in combination. The samples were then subjected to crock fastness testing (wet and dry, by AATCC-TM8-1972).

The results are shown in Table 7. The data is reported according to the AATCC scale (5 = no apparent color transfer; 1 = severe color transfer). As a general rule, textile firms reject printed fabrics as "second quality" that rate below a 4 rating on the dry/wet crock scales.

As seen from Table 7, the paper toners failed to meet the industry standard. The resins of the paper toner were likely poly(styrene-co-acrylic) materials, common within the paper xerography industry. Not designed to meet textile fastness requirements, the results of Table 7 were not surprising, and reinforced the need for development of new materials.

Table 7.

Wet/Dry Crockfastness Test Results on Xerography-Printed Polyester/Cotton Sheeting Fabric (Paper Toners, by AATCC-TM 8-1972)

Paper Toner	Dry Crock Rating	Wet Crock Rating
Red (Haloid)	3-4	3
Black (Xerox)	2-3	1

The "carrier" component of the two developer systems above consisted of glass (silica) beads. The developer systems were washed with acetone to dissolve the toner, leaving behind the glass carrier beads. These beads were used to screen new candidate resins and dyes as to the triboelectric charge attraction between the carrier and the binder materials. The cleaned beads were placed in clear vials with a small amount of powdered materials added. The vials were then tumbled to simulate the Haloid cascade step (Fig. 8) and a visual observation made as to the coverage of the material powder on the bead, and the affinity to the bead surface due to the charge build-up.

Table 8 exhibits the screening results. The yellow epoxy powder was later determined to include inorganic pigments and fillers, which are detrimental in terms of the material's overall dielectric constant (and thus triboelectric behavior in xerography printing). Marginal prints were obtained with this powder. The sublimable disperse dyes, which were envisioned as free-standing toners for xerography printing of 100% polyester woven fabrics, had no affinity for the glass beads.

A platen press-out film was made from a Kodak magenta toner. The film was extremely brittle, and broke into sections when removal was attempted from the aluminum foil used as a platen press base, prohibiting stress/strain analysis. A second blue toner material supplied by Hunt Chemical Company as a candidate gave similar film behavior. Some of the Hunt material was hand-sprinkled on the polyester/cotton sheeting and cured at 150°C for two minutes. The wet and dry crock ratings by the standard AATCC test were 1 and 4, respectively.

Table 8. Coverage/Affinity of Various Toner Material Candidates with Glass Carrier Beads

Material	Manufacturer	Coverage/Affinity Observation	
		Bead A[a]	Bead B[b]
Yellow Epoxy Resin	Armstrong Powders	No	Yes
Yellow Polyester Resin	Armstrong Powders	Marginal	No
Magenta Toner[c]	Eastman Kodak	No	--
Blue Disperse Dye	Ciba-Geigy	No	No
Yellow Disperse Dye	Ciba-Geigy	No	No
Red Disperse Dye	Ciba-Geigy	No	No

[a] Isolated from black paper toner type 10, Xerox Corporation.

[b] Isolated from red paper toner type 22-146, Haloid Xerox, Inc.

[c] Colored paper toner currently used in Kodak's color copier system.

Based on the results, Haloid batch printing trials were discontinued, contingent upon development of a suitable carrier for the poly(ethylene-co-vinyl acetate) resin identified earlier as the primary candidate (see previous section), as well as carriers for readily-sublimable blue disperse dye supplied by Ciba-Geigy Company. The following four powder samples were ground and sent to the Hunt Chemical Company of New Jersey, a key player in xerography developer systems, for the carrier research:

1. Elvax 410 from duPont, poly(ethylene-co-vinyl acetate), melt blended with 5% by weight "phthalo blue" pigment (Wiley mill-cryogenic grind-air mill-sieve sequence).
2. MU-760 analog to Elvax 410 from U.S. Industries, but with higher film properties; pigmented and prepared as was Elvax.
3. Ground/sieved blue disperse dye cake from Ciba-Geigy (>5μ average particle size).
4. Ground/sieved blue disperse dye diluted with lignin sulfonate filler/dispersing agent; same dye as (3), from Ciba-Geigy (>5 μ).

The Hunt technical staff is evaluating physical properties of the four toner systems (dielectric, triboelectric etc.), and will supply Georgia Tech with compatible carriers for the toners. Batch printing on woven 100% polyester (disperse dye toners) and 50/50 polyester/cotton (pigment/resin toners) will be resumed on the Haloid copier when the first total developer system is supplied by Hunt.

In the interim, paper toner/carrier systems, which have given clear prints on the Haloid process but which have poor

textile fastness properties, are being utilized to develop a fully - continuous fabric xerography system. According to Hunt Chemical, different hardware/developer systems will operate equally well on the same photoconductive (in this case, selenium) surface. Therefore, to reduce project down-time while Hunt is researching carriers for the textile toners, the paper developer systems are being utilized for hardware system development. Translation from the paper to the textile developer systems when the latter become available should be straightfoward.

A review was made of commercial paper xerography copiers, in conjunction with Hunt Chemical's staff, that would present the highest probability of success in conversion to a textile printing system. A Xerox Model 3100 copier was eventually recommended, and two units were isolated from State Surplus.

One of the two Xerox copiers was methodically disassembled to obtain a working knowledge of the model. The second copier was put into operation to obtain information on each of the components of the copier. It was determined that each component could be isolated and controlled manually, thus allowing for optimization of each phase of operation of the copier.

In order to construct a control system for the copier, the high voltage power supplies were tested and the relationship between the test point voltages and the output voltages were determined. The magnetic brush system used a twenty volt DC power source. Using a variable 0 to 20 volt power supply enabled adjustment of the brush speed. The light source was useable as

delivered. Four digital displays were purchased and arranged to indicate the voltage of the corona devices.

A schematic diagram of the Xerox 3100 copier is shown in Figure 16. The lay-out facilitates conversion from paper to fabric feed, with the paper cassette to be replaced by a feed roll system for the beamed fabric.

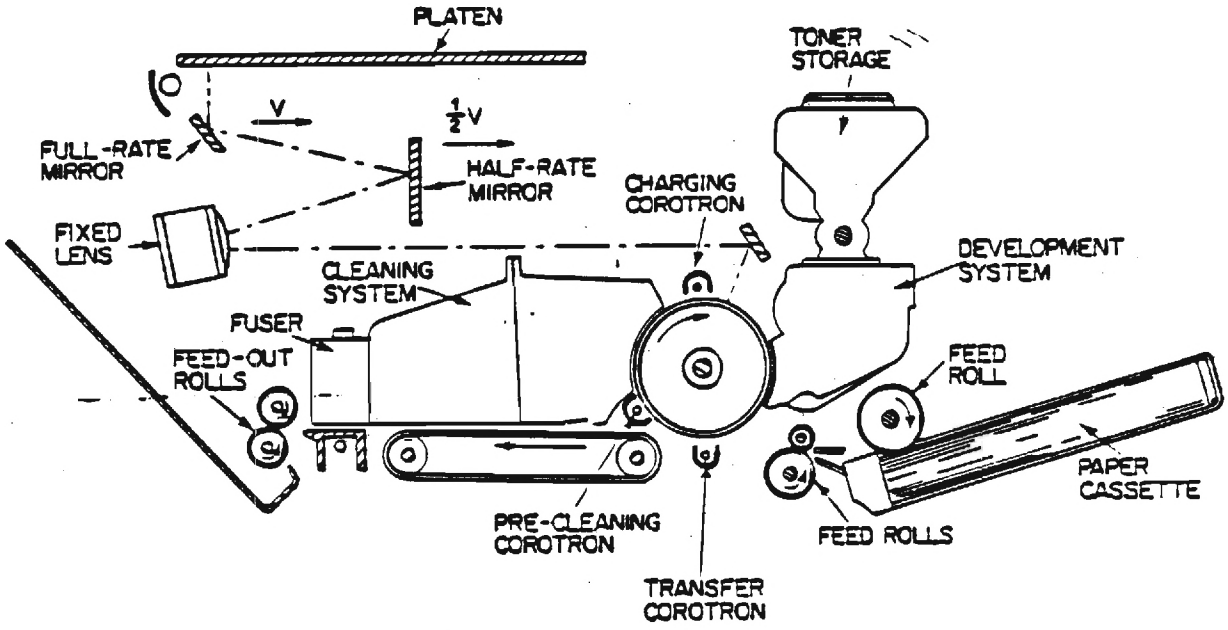
CHEMICAL BINDING OF A POLYESTER NONWOVEN

The polyester product, formed by an proprietary entanglement process, was composed of short staple polyester fibers, and was targeted for mattress pad covers. The participating company relayed the following information about the nonwoven and its current processing:

- Currently formed/dried, then shipped to a second location for chemical binding.
- Current binding process is wet pad-nip-cure sequence.
- Base weight of fabric is 1.3 oz/yd^2, while bound fabric is 1.05 oz/yd^2, indicating process stretch.
- Currently use an emulsion-based poly(vinyl acetate-co-acrylate) binder with melamine-based cross-linker.
- Specify 6.1% (of final product weight) solids add-on (SAO), but recent chemical analysis gave actual 7.3-7.5% SAO.
- Concerns about current product:
    a. "Unsquare" fabric properties (4.6:1 in tensile strength, MD:CD, plant-bound standard, GT measurements)
    b. Stiffness of material
    c. Must maintain low pilling on wash test

Fig. 16

Schematic Diagram of the Xerox 3100 Copier



Both bound and unbound rolls of nonwoven fabric were obtained from the participating plant, and the continuous Nordson spray booth system (Fig. 2) was evaluated as to settings required to achieve various pick-up levels. The first binder studied with the booth system was FA-252, Eastman's modified polyester (0-80 μ (fine grade) powder particle size range). This material, designed as an adhesive for fuseable interlining materials (Table 9), exhibited quite different film stress/strain properties from the materials considered as print binders:

- Linear Copolyester-Based
- Powder Used in Fuseable Interfacing Materials.
- T_m = 110 °C(230°F)
- Fusing Temperature = 120-150°C (250-302°F)
- $\eta_m = 4.8 \cdot 10^6$ Cps at 136°C
- Hand = Good
- Clarity = Translucent
- Drycleanable = Yes
- UV Stability = Good

Fig. 17 compares the stress/strain curve of the FA-252 with the "standard" print binder, Hycar 26120. Although the elongation-to-break was similar for the two materials, the FA-252 exhibited a much higher initial modulus, sharper yield point, and a three-fold higher breaking tenacity.

A carefully-controlled, multi-variable experiment was conducted on binding of the polyester nonowoven fabric through SOS processing. The Nordson continuous powder spray booth without electrostatics was utilized for the experiment, with the Eastman FA- 252 as the binder resin. The nonwoven fabrics were

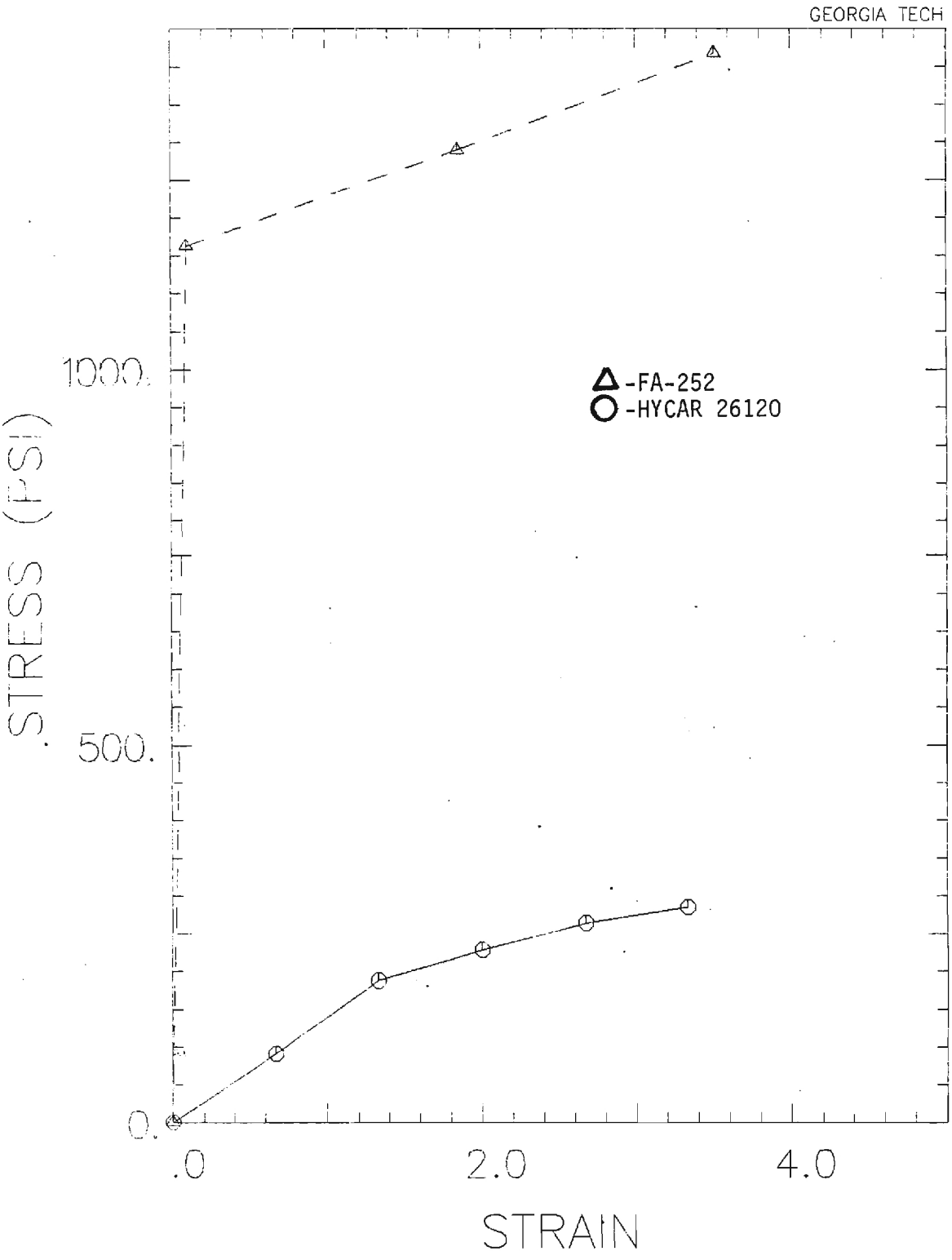


Fig. 17

Stress/Strain Characteristics of FA-252 NW Binder Compared to Hycar 26120 Print Binder

mounted under slight tension on wooden circular embroidery hoops, and were run through the Nordson unit in multiples on the belt at 100 fpm. Cure/flow was effected by a batch oven. The set of experiments was conducted on the plant unbonded nonwoven fabric, C-9462. Four levels of binder were applied, and three oven curing temperatures/two dwell times evaluated. The "control" fabric was C-5522, finished at the plant by the wet process. The "blank" fabric was C-9462, with no binder applied, but placed in a 200°C oven for 60 seconds to insure that any temperature effects on product properties were achieved.

The data matrices are shown in Tables 9 and 10. The code used is:

A1	Sample #1
3.7%	solids pick-up
oven T (°C):160	oven T(°C), 30 seconds at temperature
sts: 1421	breaking strength (psi)
stn: 0.40	strain to break (dimensionless)
mod: 3857	initial modulus (psi)
M.b.: 39.2	Mullen burst pressure (lbs)

The standard nonwoven Strip Tear Test (ASTM D-1682) was used to determine the mechanical properties on an Instron tester. Based on the Table 9 data (particularly sample D5), the higher-level solids runs of Table 10 were conducted at a set 200°C/30 sec. cure cycle.

Table 11 summarizes the most significant data of Tables 9-10, while Figures 18 and 19 graphically show the differences in

Table 9. Mechanical Properties of SOS-Bound (3.7-8.0% Solids Pickup) vs. Plant-Bound Nonwovens

MACHINE DIRECTION

A1	A2	A3	A4	A5	A6
3.7%	3.7%	3.7%	3.7%	3.7%	3.7%
160, 30"	160, 60"	180, 30"	180, 60"	200, 30"	200, 60"
sts: 1421	sts: 1443	sts: 1506	sts: 1379	sts: 1432	sts: 1467
stn: 0.40	stn: 0.45	stn: 0.48	stn: 0.45	stn: 0.45	stn: 0.40
mod: 3857	mod: 3714	mod: 5263	mod: 2813	mod: 4125	mod: 4333
M.b: 39.2	M.b: 33.9	M.b: 35.9	M.b: 32.8	M.b: 33.8	M.b: 39.6

B1	B2	B3	B4	B5	B6
4.9%	4.9%	4.9%	4.9%	4.9%	4.9%
160, 30"	160, 60"	180, 30"	180, 60"	200, 30"	200, 60"
sts: 1407	sts: 1317	sts: 1349	sts: 1500	sts: 1547	sts: 1540
stn: 0.40	stn: 0.41	stn: 0.40	stn: 0.32	stn: 0.35	stn: 0.32
mod: 5000	mod: 4363	mod: 3413	mod: 7071	mod: 4800	mod: 5167
M.b: 34.0	M.b: 39.3	M.b: 41.4	M.b: 37.1	M.b: 34.1	M.b: 34.1

C1	C2	C3	C4	C5	C6
6.6%	6.6%	6.6%	6.6%	6.6%	6.6%
160, 30"	160, 60"	180, 30"	180, 60"	200, 30"	200, 60"
sts: 1600	sts: 1500	sts: 1392	sts: 1588	sts: 1523	sts: 1580
stn: 0.27	stn: 0.30	stn: 0.50	stn: 0.34	stn: 0.32	stn: 0.32
mod: 6750	mod: 4650	mod: 4250	mod: 5833	mod: 7385	mod: 7692
M.b: 38.4	M.b: 41.9	M.b: 40.1	M.b: 36.1	M.b: 31.7	M.b: 37.8

D1	D2	D3	D4	D5	D6
8.0%	8.0%	8.0%	8.0%	8.0%	8.0%
sts: 1523	sts: 1454	sts: 1488	sts: 1544	sts: 1785	sts: 1568
stn: 0.40	stn: 0.34	stn: 0.35	stn: 0.40	stn: 0.38	stn: 0.30
mod: 6000	mod: 6188	mod: 5813	mod: 7583	mod: 7385	mod: 7750
M.b: 37.7	M.b: 39.4	M.b: 40.6	M.b: 42.7	M.b: 42.4	M.b: 39.7

Table 9 (Con't)

CROSS-MACHINE DIRECTION

A1	A2	A3	A4	A5	A6
3.7%	3.7%	3.7%	3.7%	3.7%	3.7%
160, 30"	160, 60"	180, 30"	180, 60"	200, 30"	200, 60"
sts: 565	sts: 632	sts: 555	sts: 517	sts: 716	sts: 662
stn: 1.15	stn: 1.00	stn: 1.20	stn: 1.10	stn: 1.10	stn: 1.25
mod: 330	mod: 394	mod: 351	mod: 360	mod: 615	mod: 215
M.b: 39.2	M.b: 33.9	M.b: 35.9	M.b: 32.8	M.b: 33.8	M.b: 39.6

B1	B2	B3	B4	B5	B6
4.9%	4.9%	4.9%	4.9%	4.9%	4.9%
160, 30"	160, 60"	180, 30"	180, 60"	200, 30"	200, 60"
sts: 568	sts: 552	sts: 662	sts: 792	sts: 750	sts: 620
stn: 1.35	stn: 1.20	stn: 1.10	stn: 1.20	stn: 1.20	stn: 1.20
mod: 273	mod: 334	mod: 480	mod: 517	mod: 506	mod: 458
M.b: 34.0	M.b: 39.3	M.b: 41.4	M.b: 37.1	M.b: 34.1	M.b: 34.1

C1	C2	C3	C4	C5	C6
6.6%	6.6%	6.6%	6.6%	6.6%	6.6%
160, 30"	160, 60"	180, 30"	180, 60"	200, 30"	200, 60"
sts: 626	sts: 634	sts: 633	sts: 571	sts: 583	sts: 617
stn: 1.10	stn: 1.20	stn: 1.10	stn: 1.20	stn: 1.10	stn: 1.30
mod: 403	mod: 339	mod: 450	mod: 578	mod: 372	mod: 410
M.b: 38.4	M.b: 41.9	M.b: 40.1	M.b: 36.1	M.b: 31.7	M.b: 37.8

D1	D2	D3	D4	D5	D6
8.0%	8.0%	8.0%	8.0%	8.0%	8.0%
160, 30"	160, 60"	180, 30"	180, 60"	200, 30"	200, 60"
sts: 643	sts: 722	sts: 602	sts: 644	sts: 821	sts: 750
stn: 1.25	stn: 1.25	stn: 1.10	stn: 1.30	stn: 1.25	stn: 1.20
mod: 298	mod: 509	mod: 353	mod: 396	mod: 517	mod: 568
M.b: 37.7	M.b: 39.4	M.b: 40.6	M.b: 42.7	M.b: 42.4	M.b: 39.7

Table 9 (Con't)

CONTROL FABRIC (C-5522)
400 F, tenter frame

MACHINE DIRECTION	CROSS-MACHINE DIRECTION
stress: 1752	stress: 382
strain: Ø.17	strain: Ø.32
modulus: 22,45Ø	modulus: 35Ø
Mullen burst: 29.4	Mullen burst: 29.4

BLANK FABRIC (C-9462)
200 C, 60 seconds

MACHINE DIRECTION	CROSS-MACHINE DIRECTION
stress: 1447	stress: 53Ø
strain: Ø.45	strain: 1.2Ø
modulus: 3357	modulus: 294
Mullen burst: 38.5	Mullen burst: 38.5

Table 1Ø. Mechanical Properties of SOS-Bound (1Ø.1-24.3% Solids Pickup) vs. Plant-Bound Nonwovens

	MACHINE DIRECTION	CROSS-MACHINE DIRECTION
E	stress:1526.4	stress: 586.1
1Ø.1%	strain: Ø.26	strain: 1.12
	modulus:1Ø,975	modulus: 383
F	stress:1732.5	stress: 763.8
12.2%	strain: Ø.32	strain: 1.19
	modulus: 964Ø	modulus: 571
G	stress:1792.8	stress: 699.9
14.2%	strain: Ø.32	strain: 1.16
	modulus: 952Ø	modulus: 595
H	stress:1648.3	stress: 819.7
16.2%	strain: Ø.33	strain: 1.16
	modulus: 88ØØ	modulus: 782
I	stress:1714.6	stress: 762.5
24.3%	strain: Ø.3Ø	strain: Ø.99
	modulus:1Ø,625	modulus: 815

Table 11.

Summary of Multi-Variable FA-252 Binder Study Data
(Cure: 200°C /30 sec.)

Sample	Solids (%)	Mullen Burst (lbs)	Machine Direction Properties			Wash/Pilling Ratings Face Side (5=Exc.,0=Poor)
			Stress (Psi)	Strain	Modulus (Psi)	
Standard[a]	7.3-7.5[b]	29.4	1752	0.17	22,450	5
Blank (Unbound)	0	38.5	1447	0.45	3357	3
A5	3.7	33.8	1432	0.45	4125	4-5
B5	4.9	34.1	1547	0.35	4800	-
C5	6.6	31.7	1523	0.32	7385	-
D5	8.0	42.4	1785	0.38	7385	5
E	10.1	-	1526	0.26	10,975	-
I	24.3	-	1714	0.30	10,625	5

a. Plant bound/cured by aqueous pad-nip cure process (400°F tenter).

b. Recent plant analysis on production material.

Table 11. (Con't)

Summary of Multi-Variable FA-252 Binder Study Data
(Cure: 200°C/30 sec.)

Sample	Solids (%)	Mullen Burst (lbs)	Cross-Direction Properties		
			Stress (Psi)	Strain	Modulus (Psi)
Standard[a]	7.3-7.5[b].	29.4	382	0.32	350
Blank (Unbound)	0	38.5	644	1.23	502
A5	3.7	33.8	716	1.10	615
B5	4.9	34.1	750	1.20	506
C5	6.6	31.7	563	1.10	372
D5	8.0	42.4	821	1.25	517
E	10.1	-	586	1.12	383
I	24.3	-	762	0.99	815

a. Plant bound/cured by aqueous pad-nip cure process (400°F tenter).

b. Recent plant analysis on production material.

Fig. 18

Mechanical Properties of C-9462 Nonwoven Fabric at Different Solids Loading (200°C/30 Sec. Set Cure): Machine Direction

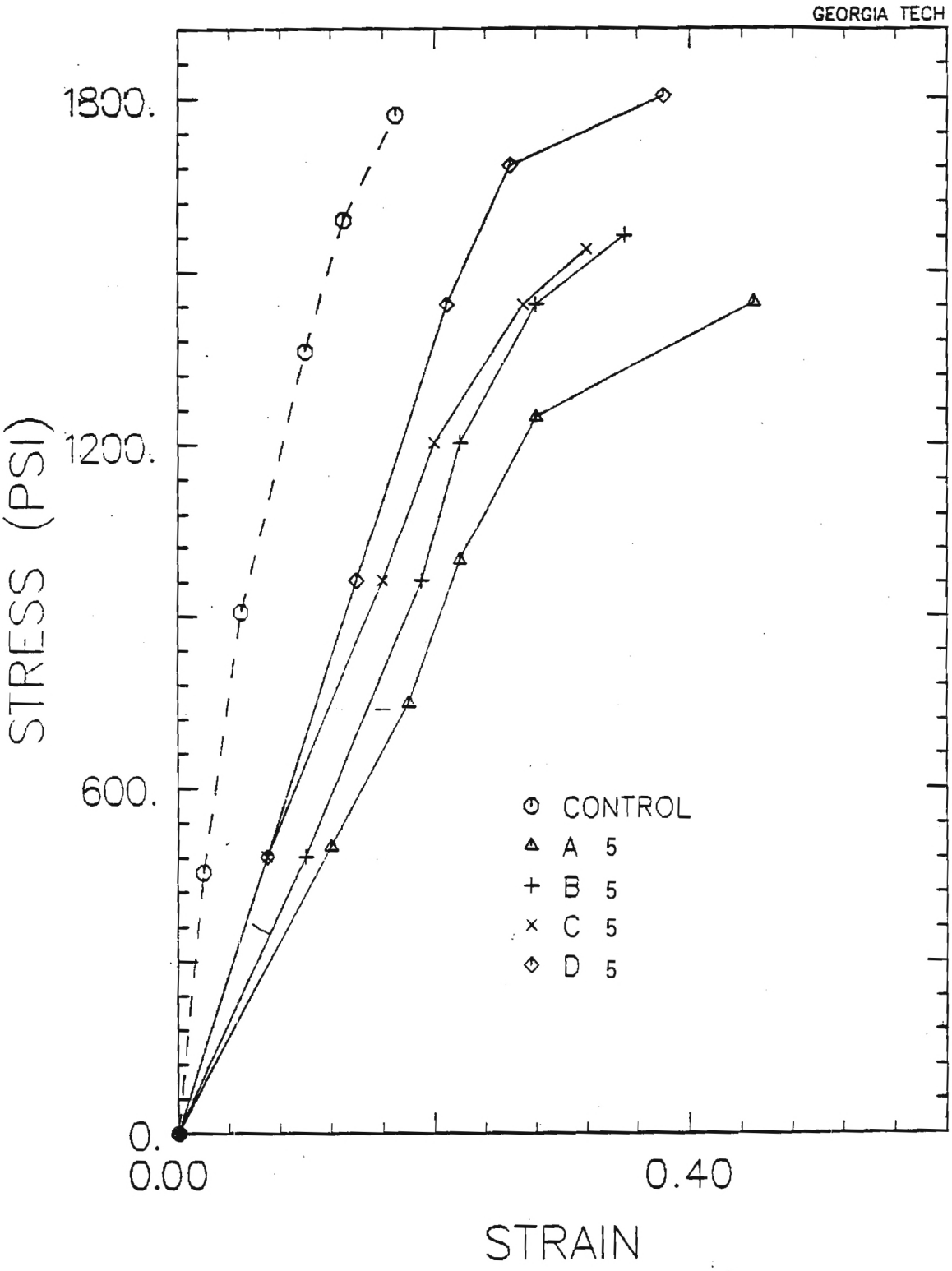
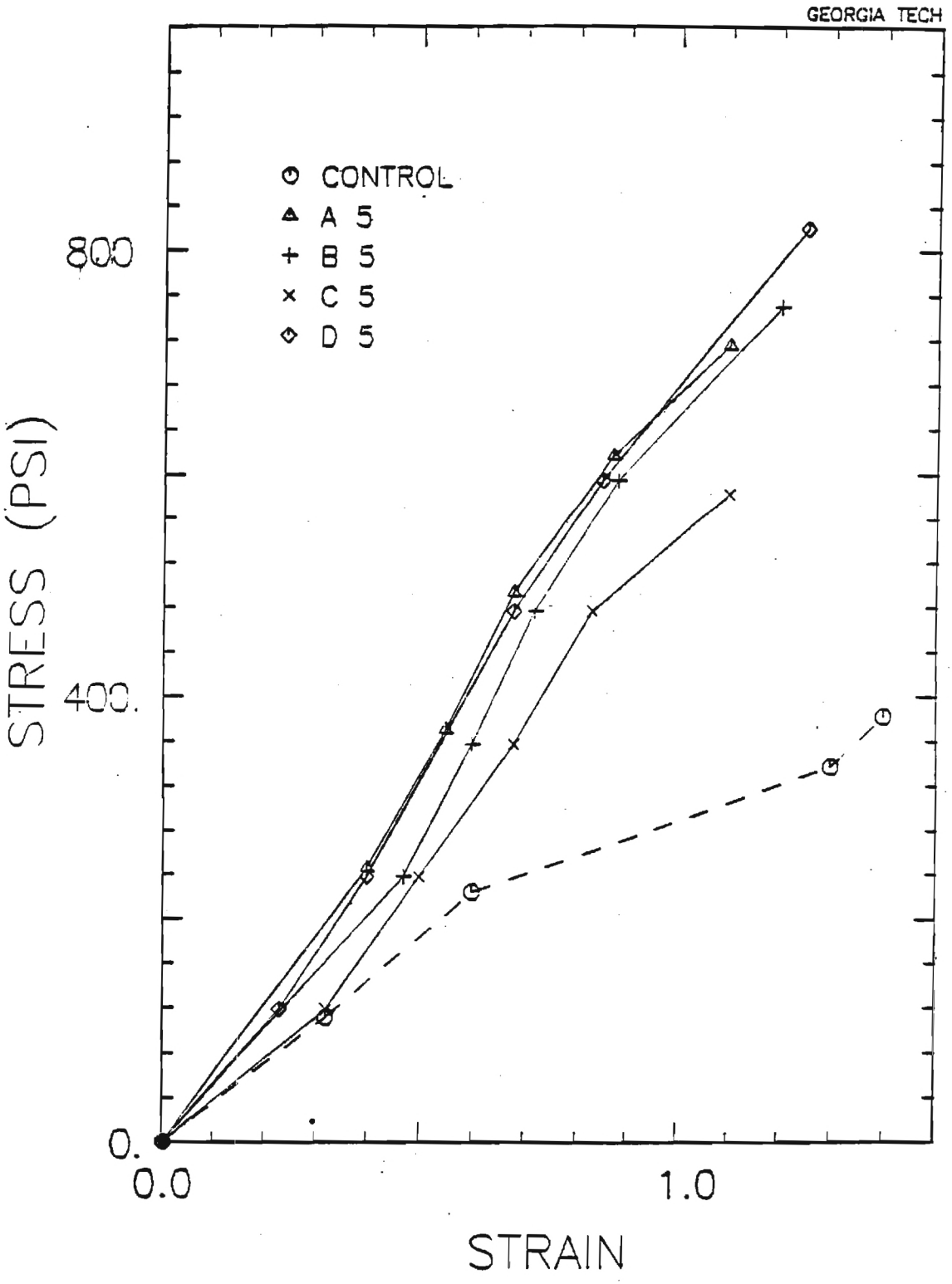


Fig. 19

Mechanical Properties of C-9462 Nonwoven Fabric at
Different Solids Loading (200°C/30 Sec. Set Cure): Cross-Machine Direction



the stress/strain curves in machine (MD) and cross-machine (CD) directions of the nonwoven at the defined optimum SOS conditions (solid lines):

Sample No.:	D5
Solids Pickup:	8.0%
Cure Temp.:	200°C
Cure Time:	30 sec.

The dotted lines on the plots correspond to the plant-bound standard, produced in a wet pad-nip-tenter oven process (poly(vinyl acetate-co-acrylic) binder). The base weight of the plant-bound standard was 1.05 oz/yd² due to stretching in the wet pad/tenter clip process (down from 1.3 oz./yd² unbound). No stretching took place on the SOS studies (minimal tension on the fabrics), and thus the density differences in SOS-bound vs. plant-bound fabrics must be considered when viewing Figures 18-19.

The data in Figure 18 were replotted in Figure 20 with stress expressed as pounds force per unit area/pound mass of fabric per unit area instead of psi to take in consideration the difference in fabric weight per unit area between plant-bound and SOS-bound fabrics. The conversion was effected by the relation:

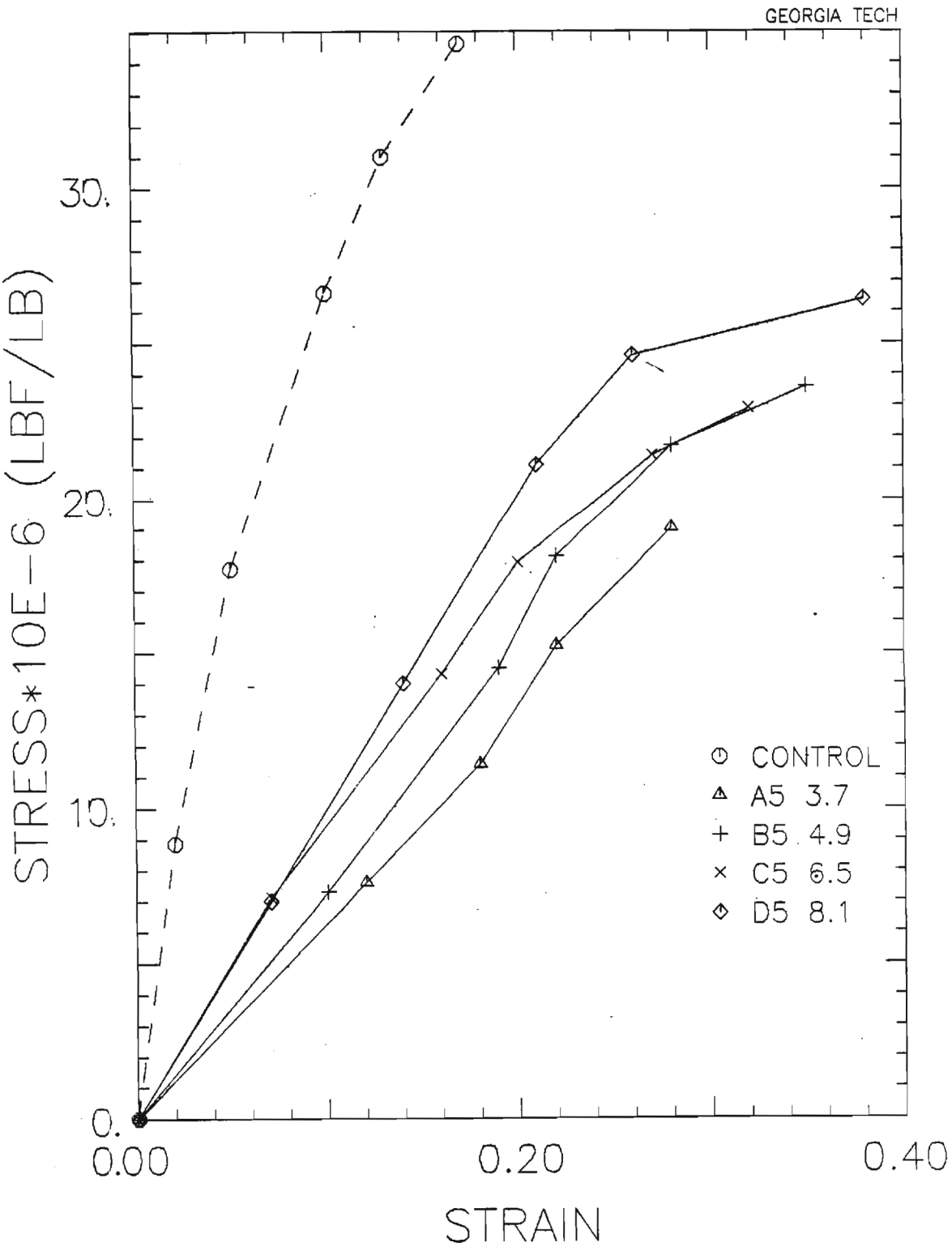
$$\text{Force } \left(\frac{\text{lbsf}}{\text{in}^2}\right) \div \text{Fabric Weight } \left(\frac{\text{lbs m}}{\text{in}^2}\right)$$

$$= \text{Stress } \left(\frac{\text{lbsf}}{\text{lbsm}}\right)$$

Fabric thickness was measured individually for each fabric in determining the in² factor (fabric strip width in the Instron jaws x thickness in inches of the fabric), as Appendix 2 shows that

Fig. 20

Data in Fig. 18 Replotted with Stress Expressed as LB_F/LB_M



the thickness was a clear function of solids pick-up level.

The effect of the stress unit change was to shift the SOS data plots downward in relation to the plant-bound control (Fig. 20). The key property, however, was considered to be the ultimate breaking stress of the final, bound product, regardless of the comparative weights per unit area. The psi unit was therefore considered a more accurate comparison of the plant and SOS fabric properties.

The data showed that the 8.0% SOS fabric (D5) in the MD approached the tensile strength of the plant standard, with a higher strain to break and a lower modulus (less stiff, Fig. 18). The CD mechanical properties were far superior to those of the plant standard, yielding a "squarer" fabric in terms of the two-dimensional properties (e.g., in tensile strength, 2.2:1 ratio vs. 4.6:1, MD:CD, D5 sample vs. plant standard). The SOS-produced fabric was softer, and gave a Mullen burst pressure 1.44X that of the standard.

Using the modified wash test prescribed by the plant for assessing pilling (5 rating: no pilling; 1 rating: very severe, by ASTM D-3512 pictures, Appendix 3), the following results were obtained:

Unbound (greige) fabric: 3

Plant-Bound Control: 5

SOS, 3.7% SPU Sample: 5 (slightly fuzzier than standard)

SOS, 8.0% SPU Sample: 5 (equivalent to standard)

SOS, 24.3% SPU Sample: 5 (less fuzzy)

Thus the optimized conditions (sample D5) gave equivalent pilling

properties on the wash test to the plant-bound standard.

Mechanical properties steadily improved from a low of 3.7% solids pickup (SPU) to 8.0%. However, as shown in Table 10 and Figures 21-22, increasing the SPU up to 24.3% gave practically no improvement in MD properties, and little in the CD direction.

Photographs of the plant-bound standard showed considerable fiber-bridging by binder film (Figure 23). In fact, the individual fibers appeared to be "sized" with the fiber, i.e., encased. Both factors lead to the high stiffness and recovery from deformation observed in the CD direction of the standard.

By contrast, the FA-252 gave a more globular formation on melting, showing limited flowability at 200°C as a reflection of its high melt viscosity (low 10^5 Cps at 200°C, Table 4, Fig. 24). The dramatic improvement in mechanical properties over the unbound nonwoven, however, indicated that statistical, effective fiber junction binding was indeed taking place.

Some concern existed that the properties of Sample D5 (Table 9) were out of context with those of the surrounding samples in the matrix (D4, C5, D6). As a check, C-9462 fabric was then resprayed with FA-252 on the Nordson line at 7.9% and 9.9% deposition levels, and the fabrics cured at 200°C for 30 and 60 seconds.

The strip test data is shown in Table 12 and Figures 25-26. The unbound material's properties (not cure-cycle treated) are also shown in the figures, demonstrating the anomalous CD properties. The fabric collected from the repeat SOS runs showed substantially lower breaking tenacity than the Table 10-11 samples under similar application and flow/cure conditions.

Fig. 21

Mechanical Properties of C-9462 Nonwoven Fabric at Higher Solids Loading (200°C/30 Sec. Set Cure): Machine Direction

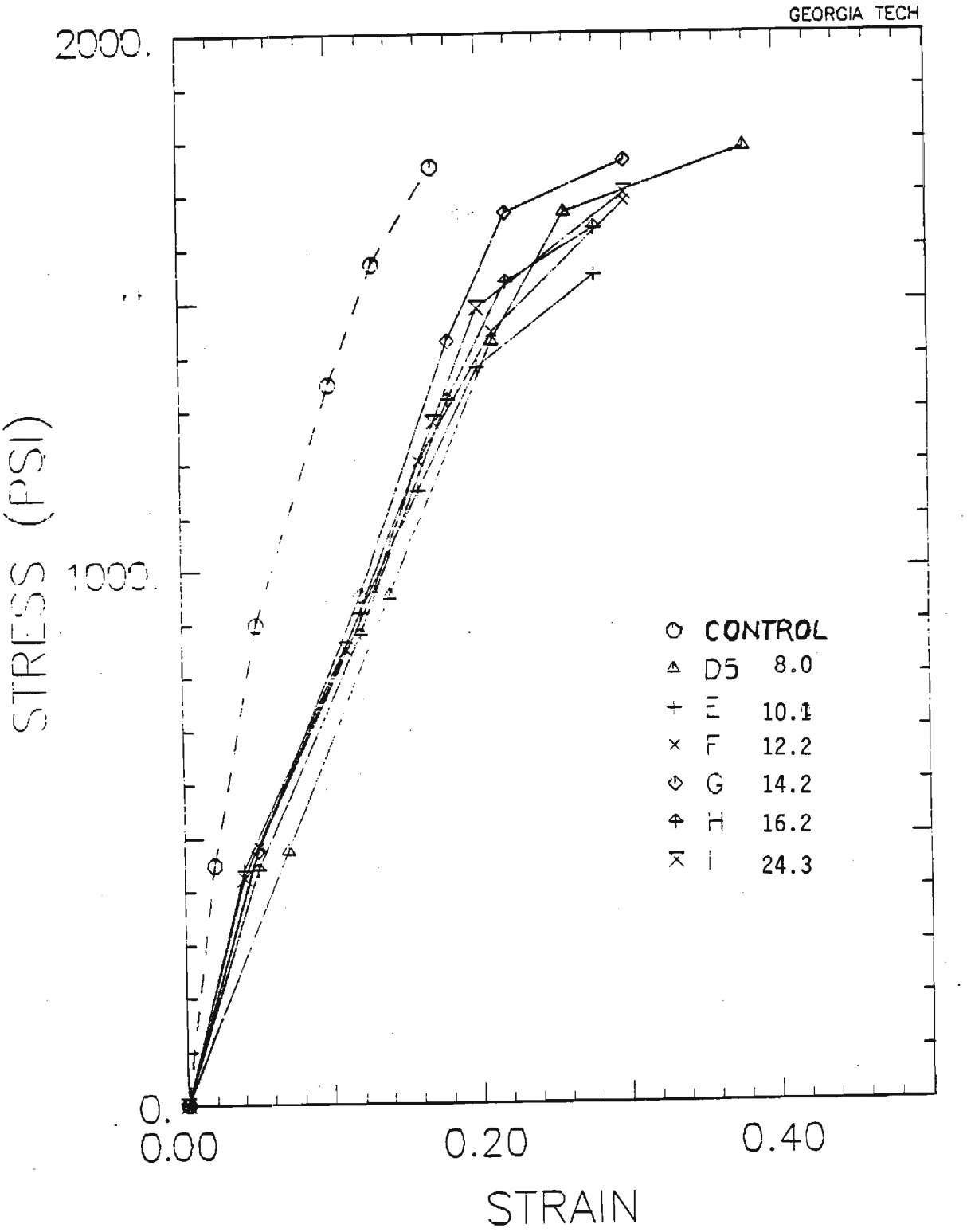
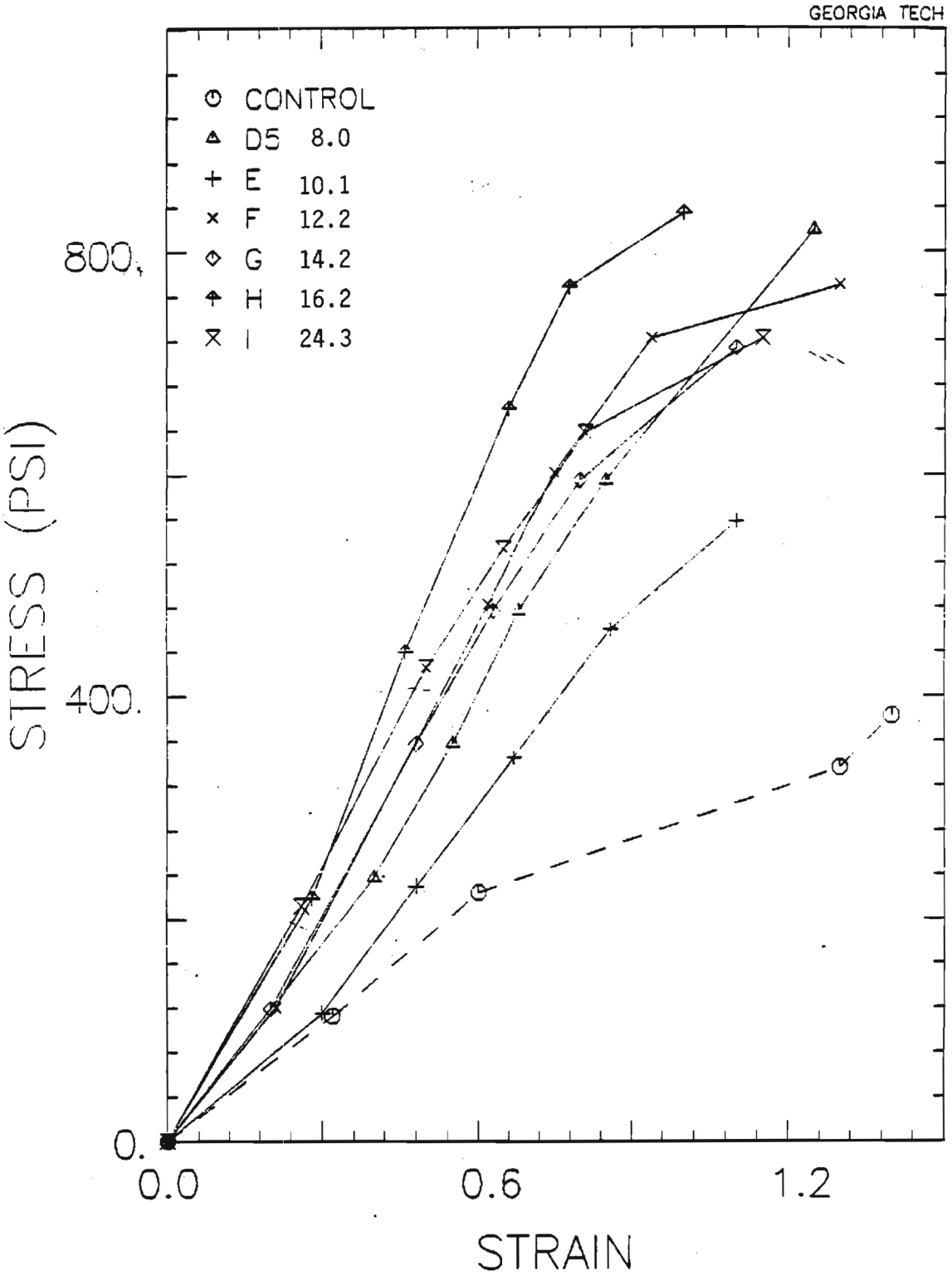


Fig. 22

Mechanical Properties of C-9462 Nonwoven Fabric at Higher Solids Loading (200°C/30 Sec. Set Cure): Cross-Machine Direction



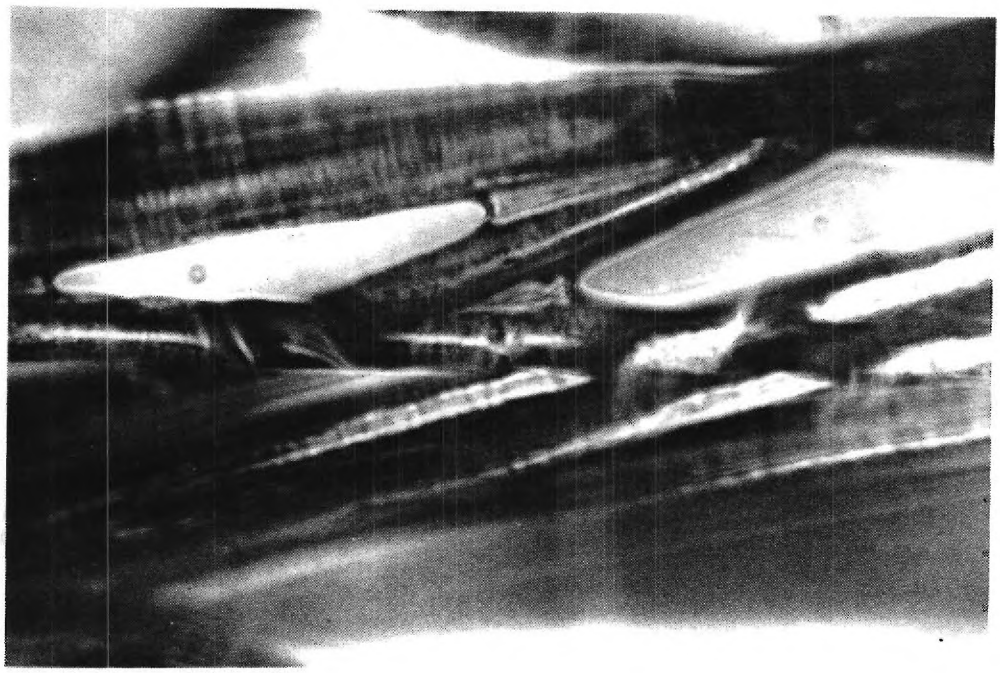


Fig. 23

Photograph of Plant - Bound Chicopee Nonwoven

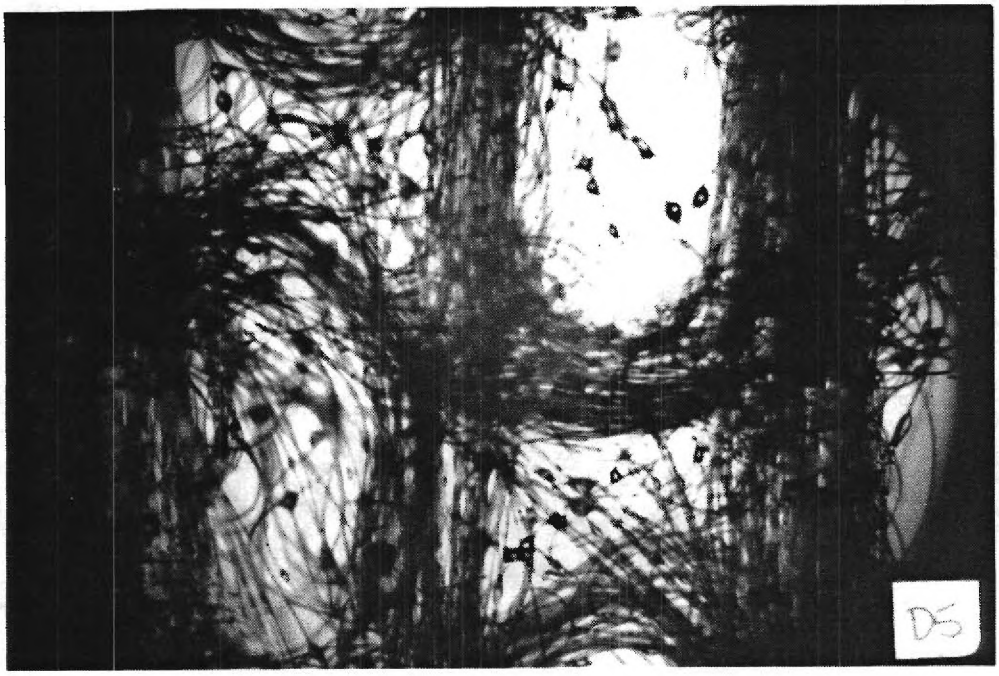


Fig. 24

Photograph of SOS - Bound Chicopee Nonwoven
(FA-252 Resin, D5 Sample, 10% SPU)

Table 12. Second Trial Mechanical Data of Nonwoven Fabrics Bound by FA-252 in the SOS Process

Sample (% pickup)	MD			CD		
	Stress (psi)	Strain	Mod. (psi)	Stress (psi)	Strain	Mod. (psi)
D5[a] (8.Ø)	1785	Ø.38	7385	821	1.25	517
A1[b] (7.9)	1256	Ø.35	6533	683	Ø.96	7Ø7
D6[b] (8.Ø)	1568	Ø.3Ø	775Ø	75Ø	1.2Ø	568
A3[b] (7.9)	1237	Ø.28	7952	553	1.ØØ	627
E[a] (1Ø.1)	1526	Ø.26	7385	586	1.12	383
B1[a] (9.9)	137Ø	Ø.28	7764	578	Ø.86	755
B2[b] (9.9)	129Ø	Ø.3Ø	7913	572	Ø.9Ø	774

[a] Cured at 2ØØ°C/3Ø seconds.

[b] Cured at 2ØØ°C/6Ø seconds.

Fig. 25

Repeat Trial Results with SOS Binding of Nonwovens by FA-252: Machine Direction

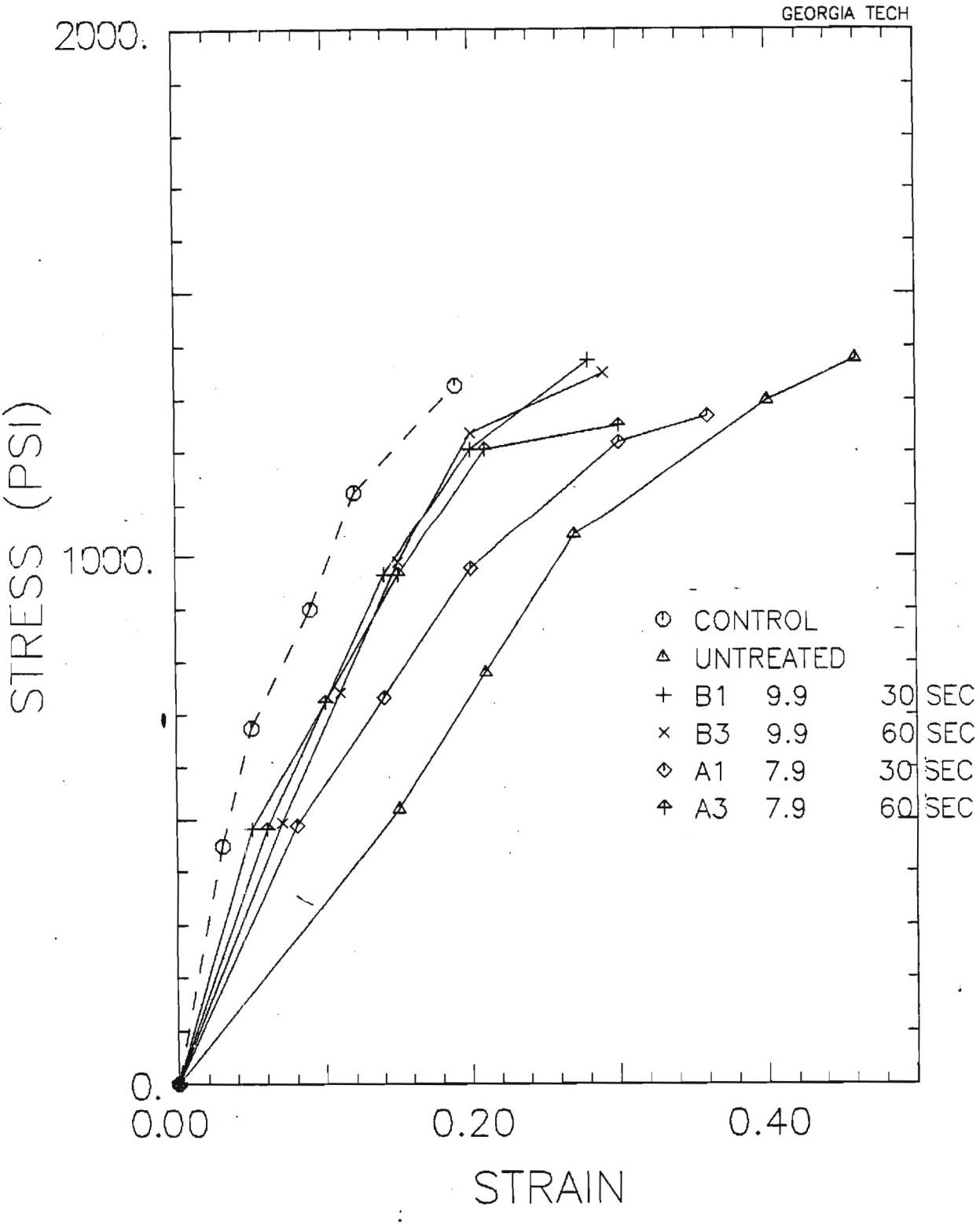
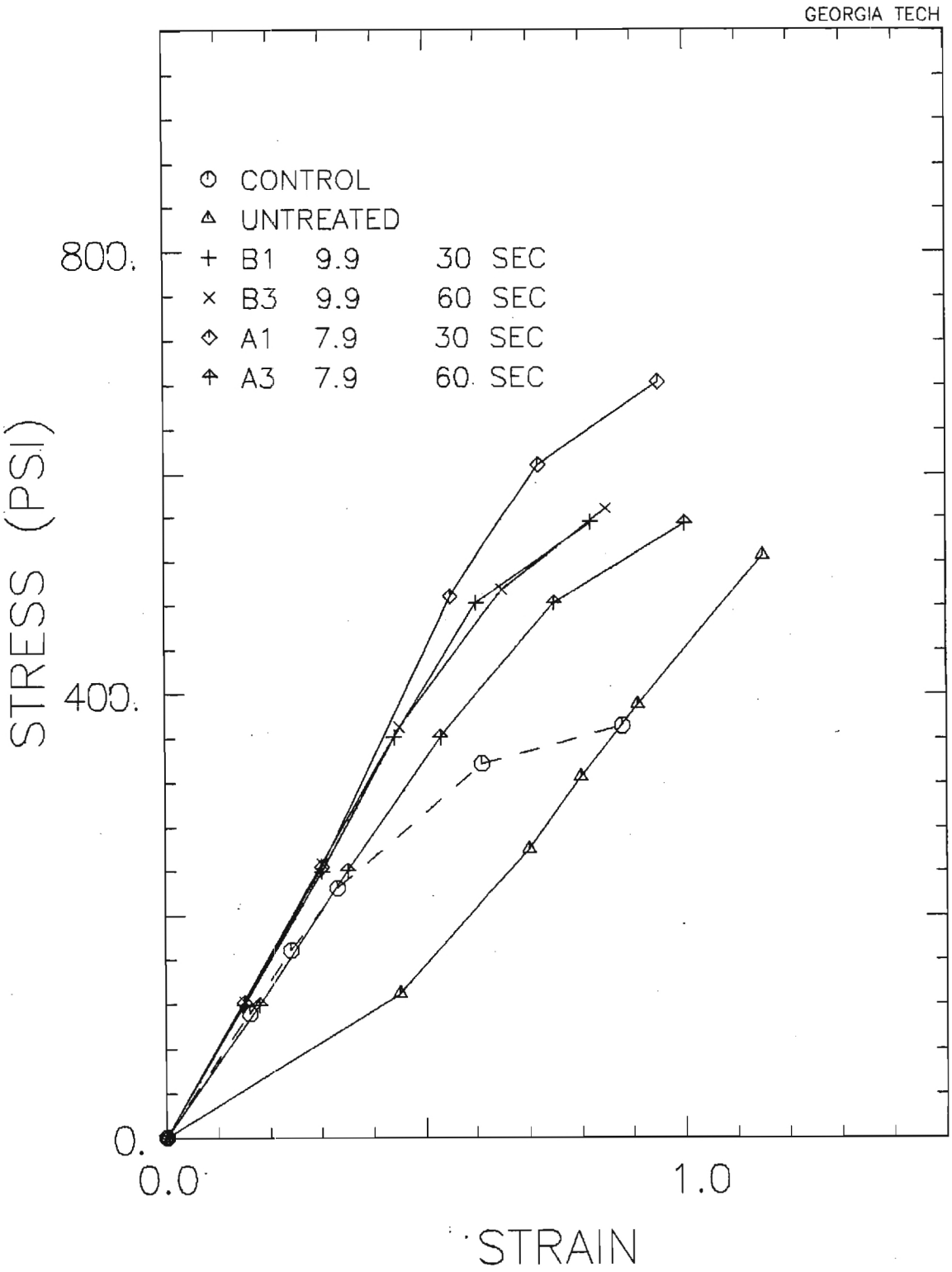


Fig. 26
Repeat Trial Results with SOS Binding of
Nonwovens by FA-252: Cross-Machine Direction



Several reasons were postulated to explain the drop in properties:

1. Poor reproducibility of the process.
2. Instron differences (the samples from the two sets were torn on two different machines).
3. Nonuniformities in the as-formed, entangled nonwoven fabric, leading to "weak spots" at various points within the supplied roll.

The first theory was not given credence, as the line had been thoroughly evaluated for deposition level vs. settings with the FA-252 powder before beginning the fabric trials. The second theory was therefore checked.

The control (plant-bound) fabric was tested on both the Instron Model 1130 and Model 1125 testers using the same load cell. The results, shown in Table 13, revealed that the difference in machine testers was insignificant, although the fabric showed a substantially lower breaking strength (MD) than earlier (1375 psi vs. 1752 psi, Table 9).

The two-Instron experiment, however, failed to consider that the samples were taken from a relatively small length of the fabric on the supplied roll, compared to the long lengths of the unbound fabric consumed by the SOS trials in the time period between the Table 10-11 and Table 12 runs. Theory 3, therefore, was not disproved by the experiment. Through-roll testing on both the bound/unbound fabrics was thus planned for Year 3 to determine the uniformity of properties as a function of length and depth into the rolls.

Table 13. Mechanical Properties of Plant-Bound Nonwoven on Different Instrons

Instron Model (No.)	MD		CD	
	Stress (psi)	Strain	Stress (psi)	Strain
113Ø	1375	Ø.19	316	1.16
1125	1325	Ø.19	365	Ø.88

In summary, the SOS process with FA-252 gave an acceptable product at 8% SAO level from a mechanical property viewpoint, with pilling on washing equivalent to the plant-bound product. The flow of the polymer, however, was not as fluid as desired, reflecting its high melt viscosity (Table 4, 4.8×10^6 Cps at 136°C). Although statistically-random "spot-welding" of fiber was adequate to give the desired mechanical properties (Figures 18-19, 24), a smooth junction film binding such as exhibited by the plant binder (Fig. 23) was not being achieved. Research was therefore initiated to reduce the viscosity of the FA-252 polyester by blending/interchanging it in the melt with various modifiers.

In Year 1, preliminary studies on reaction melt modification of FA-252 had been conducted with three materials:

a. MFP - a commercially available melt modifier of proprietary composition.

b. Terephthalic acid - an aromatic, difunctional acid.

c. Sebacic acid - an aliphatic, difunctional acid.

Conditions were 200°C/~5 min. in the melt. The results are reported in Tables 14-16.

As shown, the melt viscosity of FA-252 was reduced, but not to the desired low 10^3 - 10^2 Cps region. Only one case (20% sebacic acid at 20% loading) gave a reading in the high 10^3 Cps range (9.2×10^3). The high loading level, however, was considered at this time to be unacceptable from a film property viewpoint.

Later slashing formulation research with Eastman WD (also a modified polyester), however, revealed that high loading (30-40%)

Table 14.

FA-252 Modified by MFP: Melt Viscosities at 200°C in Cps

Composition	% MFP Additive Level			
	0.0	0.5	1.0	10.0
Upigmented FA-252	8.8×10^4	1.1×10^5	5.8×10^4	1.1×10^5
10% Pigmented FA-252	5.4×10^5	6.4×10^4	1.4×10^5	3.2×10^5
	—	2.3×10^5	—	—
2.5% Pigmented FA-252	—	1.6×10^5	1.4×10^5	—

Table 15.

Unpigmented FA-252 Modified by Terephthalic Acid:

Melt Viscosities at 200°C

T - Acid Level (% by Weight)	Viscosity (Cps)
1	7.5×10^4
5	6.6×10^4
10	6.5×10^4

Table 16.

Unpigmented FA-252 Modified by

Sebacic Acid: Melt Viscosities at 200°C

Sebacic Acid Level (% by wt.)	Viscosity (Cps)
1%	6.0×10^4
10%	1.8×10^4
20%	9.2×10^3

and much larger melt hold times (>15 minutes) gave viscosity ranges in the 10^2-low 10^3 Cps range utilizing adipic acid (see slashing section). Further research was therefore conducted on FA-252 with the following melt modifiers:

Poly (adipic anhydride), PADA

Adipic Acid

Sebacic Acid

Diacid Blends with: Polyethylene Glycol (PEG)

Cyclohexanedimethanol (CHDM)

The conditions in the Brookfield viscometer were 200°C, and viscosity was followed as a function of time in the melt.

The formulations giving the most dramatic drops in viscosity with time are plotted in Fig. 27. FA-252 alone was shown to give little change in melt viscosity at 200°C/20 minutes, confirming its good thermal stability under the experimental conditions. With the melt modifiers in the system reactive with the FA-252 polyester via transesterification mechanisms, viscosities dropped off in varying degrees with time, depending on formulation. Interestingly, the FA-252/10% PEG 3400/5% sebacic acid formulation gave an extremely low well of melt viscosity (~200 Cps at 14 minutes), and then began to climb again, indicating cross-linking of the backbone as a secondary, slower reaction compared to transesterification.

Films were then platen pressed from the Fig. 27 formulations. All of the films gave increased cloudiness and were more brittle compared to the virgin polymer. Fig 28 details the film stress/strain characteristics of a FA-252/30%

Fig. 27

Melt Viscosity vs. Time for Formulations of FA-252 with Various Modifiers

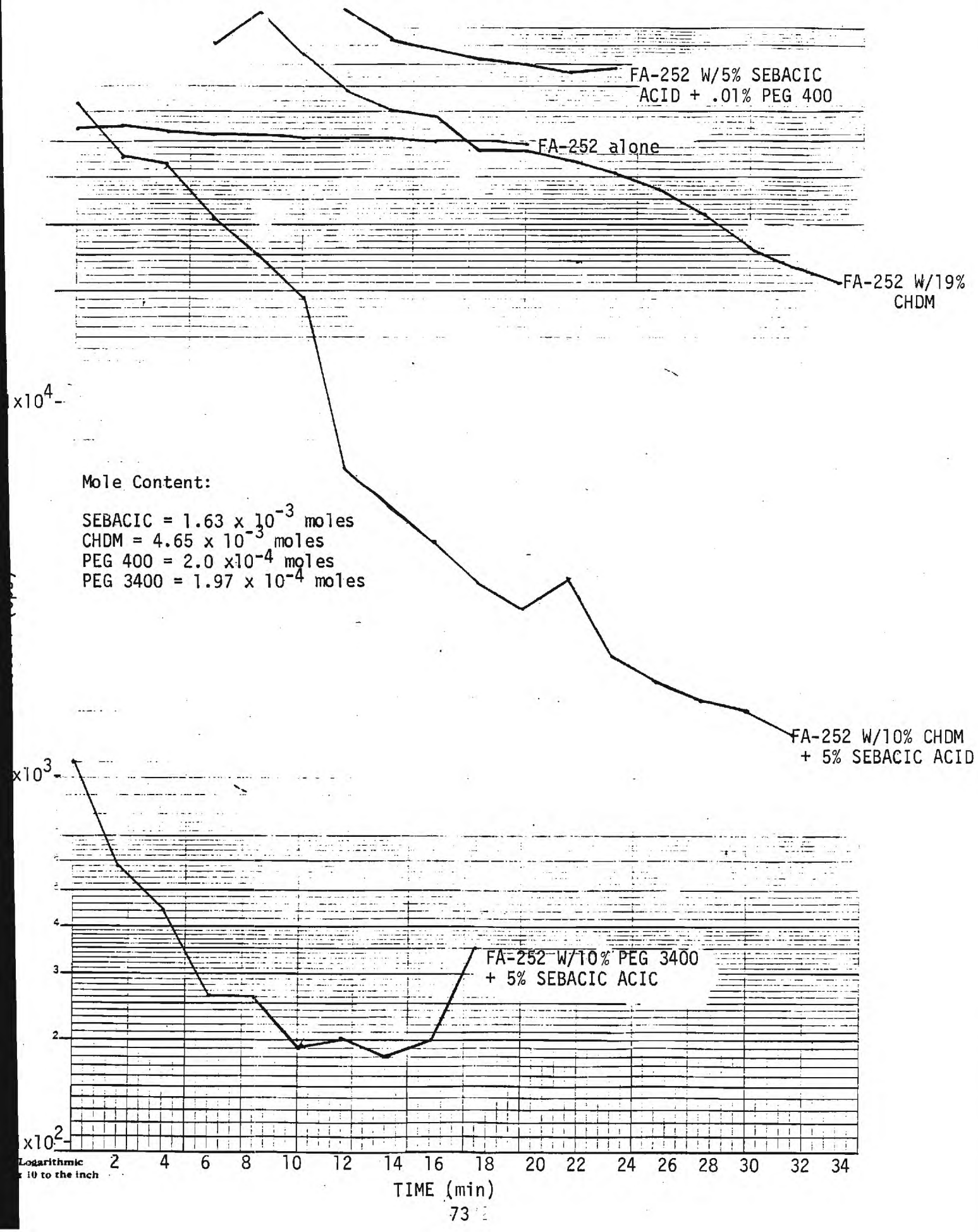
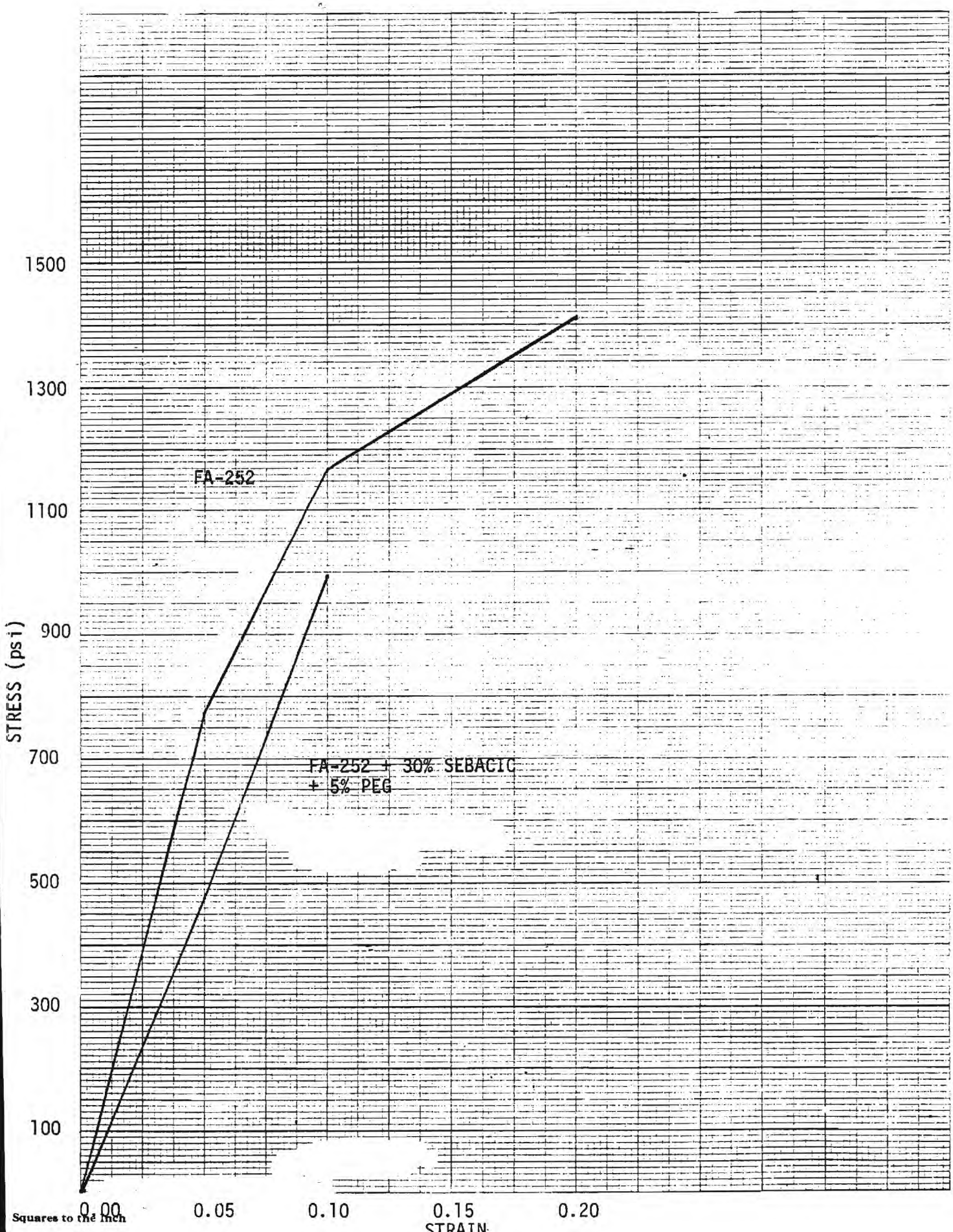


Fig. 28

Film Stress/Strain Behavior of FA-252 vs. a Modified Blend



sebacic acid/5% PEG composition. The stress/strain curve reflects the increase in brittleness compared to FA-252, with a high initial modulus, brittle failure with no yield point and a low strain-to-break/lower breaking tenacity. For comparison, the plot for unmodified FA-252 film is also shown.

The simple organic modifiers were ruled unacceptable, based on the detrimental effects to the film properties. Tennessee Eastman then agreed to supply the research group with the two potential melt modifiers described in Table 17. The FA-252 prepolymer and FA-250 were tested for viscosity on the Brookfield, along with several blends of varying proportions between the prepolymer and the FA-252.

The results are detailed in Table 18. The prepolymer exhibited an extremely low melt viscosity at 160°C (115 Cps), indicative of its low molecular weight. A 50/50 blend of the prepolymer with the FA-252 gave a viscosity in the targeted range ($1.875 \cdot 10^3$ Cps), although additional clouding was evident. The blend viscosity was proportionate to the relative quantities of long- and short-chain FA-252, returning to the 10^5 Cps range at a 90:10 FA-252:prepolymer ratio.

The FA-250 in the virgin state possessed a high melt viscosity ($1.68 \cdot 10^5$ Cps at 12 min.) which reduced on holding for 20 min. to $7.3 \cdot 10^4$ Cps. Blending of the FA-250 with the prepolymer and with FA-252 failed due to melt incompatability of the two polymers, i.e., phase separation.

The results were encouraging in terms of melt viscosity modification of the FA-252 with a totally-compatible material (its own prepolymer). The additional clouding eliminated the

Table 17. FA-252 Melt Modifiers Supplied by Tennessee Eastman

A. FA-252 prepolymer, or oligomer, from the sample port of the reactor.

B. FA-250, a pilot-scale product that was marketed as a lower-melt viscosity alternative to FA-252 (similar, but different, backbone).

Table 18.

Melt Viscosity of FA-252 Blended with Varying Proportions of Prepolymer

Sample Blend	Min. Viscosity[a]	Brookfield Cell	Time In Melt
252/252P 50/50	1875 cps.	LVT	4 min.
252/252P 60/40	10800 cps.	"	3 min.
252/252P 80/20	27600 cps.	"	4 min.
252/252P 90/10	134000 cps.	"	14 min.
FA-250	73000 cps.	LVT at .3 rpm	20 min.
	168000 cps.	HBT at 10 rpm	" "
252 Prepolymer	115 cps.	LVT	7 min.

[a]. Tests conducted at an isothermal 160°C.

blend as a print binder candidate, but did not prohibit its use in the colorless binding of the nonwoven. FA-250 proved not to be a viable candidate, since its viscosity in the virgin state was well above the 10^2 - 10^3 Cps range, and it was melt incompatible with the FA-252 prepolymer.

A similar spray/bind evaluation was then conducted with the nonwoven binder candidate from H.B. Fuller Co., IF-3237. The modified epoxy, melt blended before grinding with a blue "tracer" pigment, was supplied in powder form by Fuller, and was utilized as obtained (40-70 μ in particle size range).

Samples of the unbonded C-9462 nonwoven were sprayed on the Nordson spray booth system at various pick-up levels with the IF-3237 resin. The blue powder was deposited at seven weight levels, and the spraying conditions were held constant at: web speed=100 fpm; fluidizing air= 7 psi; atomizing air= 8 psi; up amp. air= 12-13 psi; and down amp air= 40 psi. The flow rate was modified to achieve the various levels of deposition. The oven conditions were held constant at 200°C with a 30 second dwell time.

Although the powder fluidized well, there was noticeable non-uniformity in the spray pattern. The non-uniformity was most obvious at the low deposition levels, but was present in all cases. The condition may have been visible for the first time due to the pigmentation present, or it may have been restricted to the IF-3237. A large color difference also existed between the face and back sides of the fabric. The back side was much lighter, indicating less powder clinging to the back.

Photos were taken of each sample, evaluated for polymer flow and compared to the results seen from the Instron data. The differences seen in the photos related to level of powder deposition. The flow was good along fibers and at cross-over points, and looked to be the same in all samples. Figure 29 exhibits the 8.4% sample under transmitted light, and Figure 30 one of the fabrics under reflected light.

The IF-3237 binder showed much better flow to, and coverage of, fiber cross-over (junction) points than did the earlier FA-252 binder (compare Figures 29-30 with Figure 24). The IF-3237 appeared to have a more compatible surface tension relationship with the polyester fiber in the melt stage, and flowed to a greater extent than had the FA-252.

Mechanical properties were obtained on the Instron tester as with the earlier FA-252 bound samples. The results are contained in Table 19, and Figures 31 and 32 show the plotted stress/strain curves versus the plant-bound control.

None of the samples approached the 1752 psi tenacity of the plant-bound control fabric, with the 5.2% loading giving the highest machine direction strength (1548 psi., Sample B). As with the FA-252, a peak was reached in tensile strength with solids add-on, with a decline in the MD property beyond the 5.2% loading. The CD tenacity, however, did reach its highest value at the highest loading (709 psi at 24.4% loading).

The modulus of Sample B was substantially lower than the plant control in the MD direction (7404 psi vs. 22,450 psi), but compared favorably with FA-252's Sample D5 of Table 11 (7407 psi vs. 7385). Initial modulus for both the SOS samples were higher

Fig. 29

Fabric C-9462 SOS-Sprayed with 8.4% IF-3237 Binder
($200^{o}C$/30 sec. Cure, Transmitted Light)

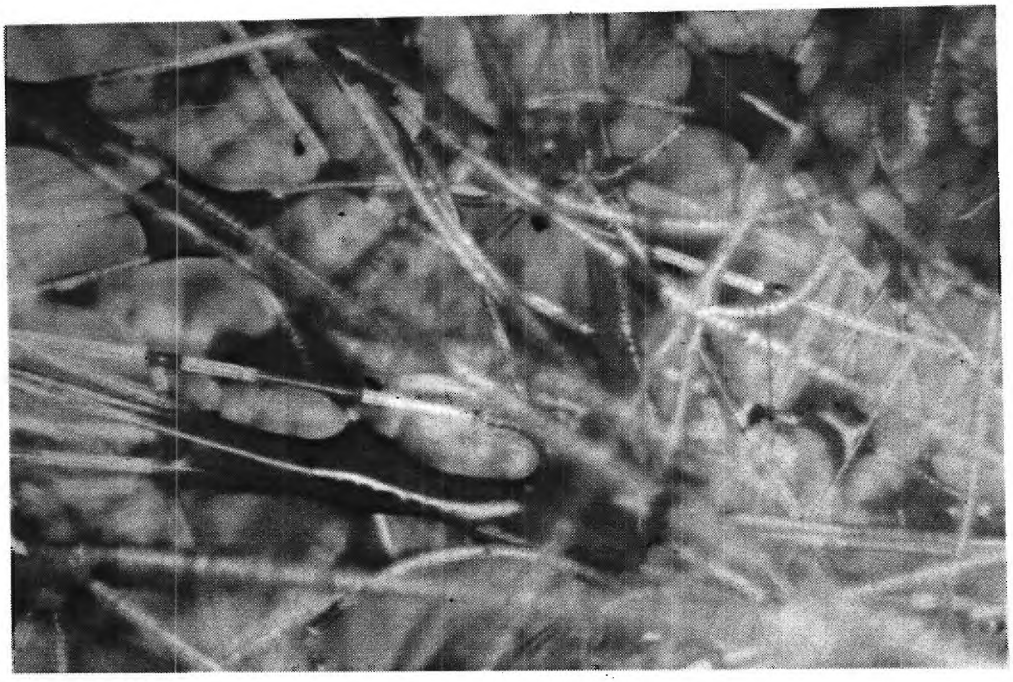


Fig. 30

Fabric C-9462 SOS-Sprayed with IF-3237 Binder
(200°C/30 sec. Cure, Reflected Light)

Table 19

Ultimate Mechanical Properties of C-9462 Fabric Bound With IF-3237 at Different Loadings (200°C/30 Sec. Cure)

MACHINE DIRECTION

Sample ID	Binder (%)	Breaking Stress (psi)	Breaking Strain	Initial Modulus (psi)	Mullen Burst (lbs)
Blank[a]	0.0	1369	0.41	3652	24.2
Control[b]	6.0	1752	0.17	22,450	29.4
A	3.4	1530	0.32	6299	33.3
B	5.2	1548	0.32	7407	38.1
C	6.7	1397	0.25	8246	33.6
D	8.4	1434	0.28	8484	34.1
E	12.1	1443	0.21	13580	30.1
F	15.7	1388	0.18	14007	28.0
G	24.4	1359	0.17	15646	29.5

CROSS-MACHINE DIRECTION

Sample ID	Binder (%)	Breaking Stress (psi)	Breaking Strain	Initial Modulus (psi)	Mullen Burst (lbs)
Blank[a]	0.0	644	1.23	502	24.2
Control[b]	6.0	382	0.32	350	29.4
A	3.4	533	1.20	522	33.3
B	5.2	673	1.10	710	38.1
C	6.7	655	1.00	697	33.6
D	8.4	534	1.20	608	34.1
E	12.1	633	1.07	742	30.1
F	15.7	642	0.93	780	28.0
G	24.4	709	0.92	903	29.5

[a] Unbound plant (greige) fabric.

[b] Plant-bound fabric (wet processed).

FIGURE 31

Mechanical Properties of C-9462 Nonwoven Fabric at Different Solids Loading (200°C/30 sec. Set Cure): Machine Direction

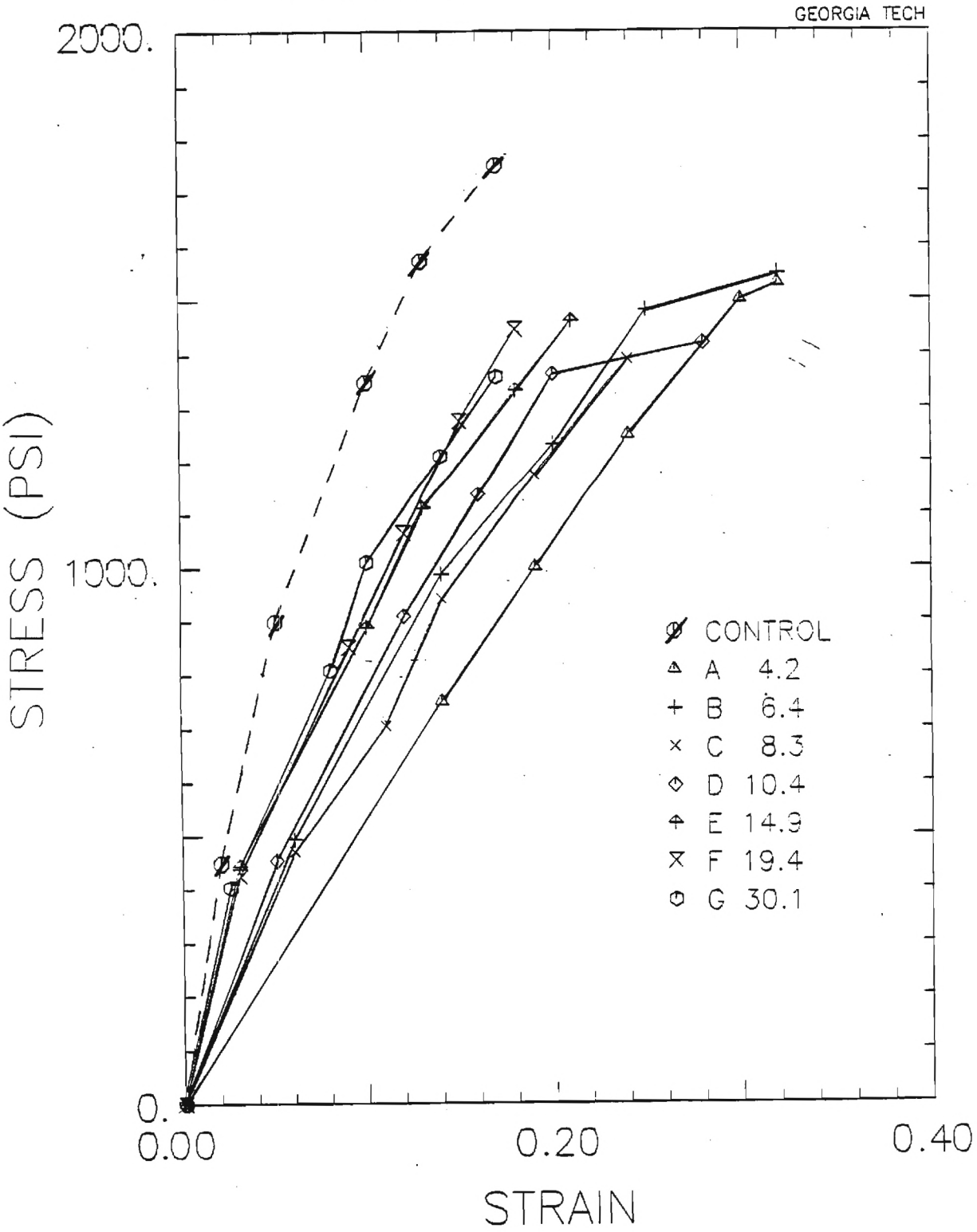
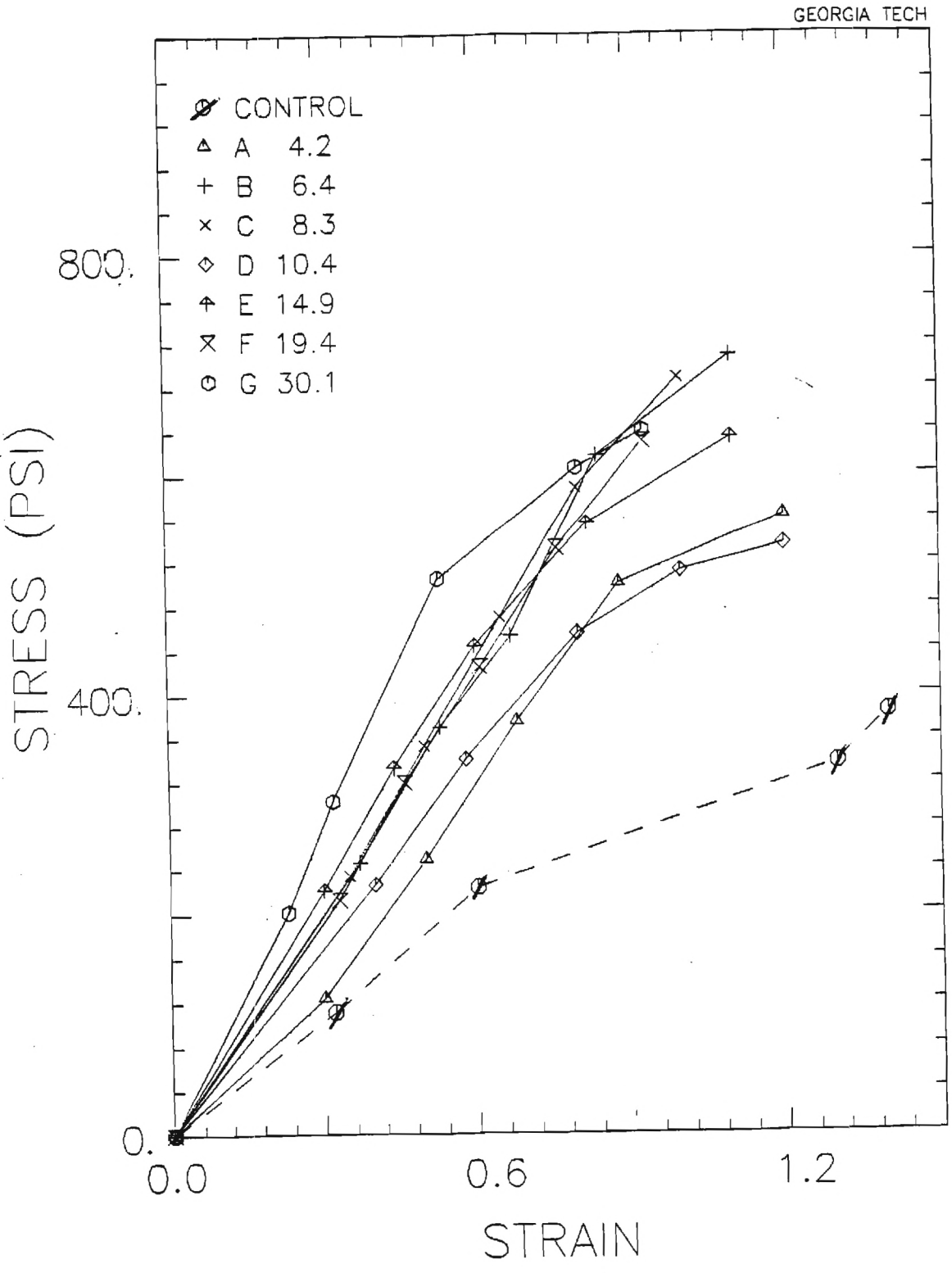


FIGURE 32

Mechanical Properties of C-9462 Nonwoven Fabric at Different Solids Loading (200°C/30 sec. Set Cure): Cross-Machine Direction



(~2X) than that of the plant control in the CD. The lower modulus in the MD gave the SOS fabrics a softer "hand" than the stiff plant-bound standard. Sample B of Table 19 gave a Mullen burst test pressure 1.3X that of the plant-bound standard, and all of the IF-3237-bound fabrics gave a wash/pilling rating of "5".

In summary, the H.B. Fuller IF-3237 resin was also concluded to be a viable candidate for binding of the polyester nonwoven. The major advantage over Eastman's FA-252 was better melt flow characteristics; however, the ultimate tensile strength of the bound fabrics were lower for the IF-3237 (13% below the top FA-252 reading, Sample D5). Mullen burst performance was similar for fabrics bound by both resins, and wash/pilling ratings of "5" were obtained at optimized levels of both.

A third binder, H.B. Fuller's PS-1 (a heat-activated, self-crosslinking, polystyrene-based copolymer) was then evaluated, with defined properties:

T_m = 176°C

η = 3.8×10^6 Cps at 200°C

particle size: 75% 125 microns, some 400 microns

press-out: transparent, rigid film, good flow

Samples of the C-9462 nonwoven were sprayed at various levels with the PS-1 resin. The deposition levels tested were 3.6, 4.9, 6.8 and 8.7%. Powder levels above 10.8% could not be achieved. Increasing the flow pressure actually decreased the pick-up, apparently due to segregation of the paticles within the bed, or selective size particle spraying. Conditions held constant were: web speed=100 fpm; fluidizing air=48 psi;

atomizing air= 8 psi, up amp= 5 psi, and down amp= 40 psi. Flow pressure varied from 4-5.5 psi. The oven conditions were 200°C for 30 seconds.

The powder particles were visually observed to have remained spherical and on top of the fibers after fusing in the oven. The reason may have been a surface tension/wetting problem, or that the melt viscosity (3.8×10^6 Cps) was too high to permit flow. To rule out particle size as a problem, the powder was reground using the Trost air mill. The throughput was good ~ 2.4 lbs/hr) on the small unit. The powder was finer after grinding, but not significantly.

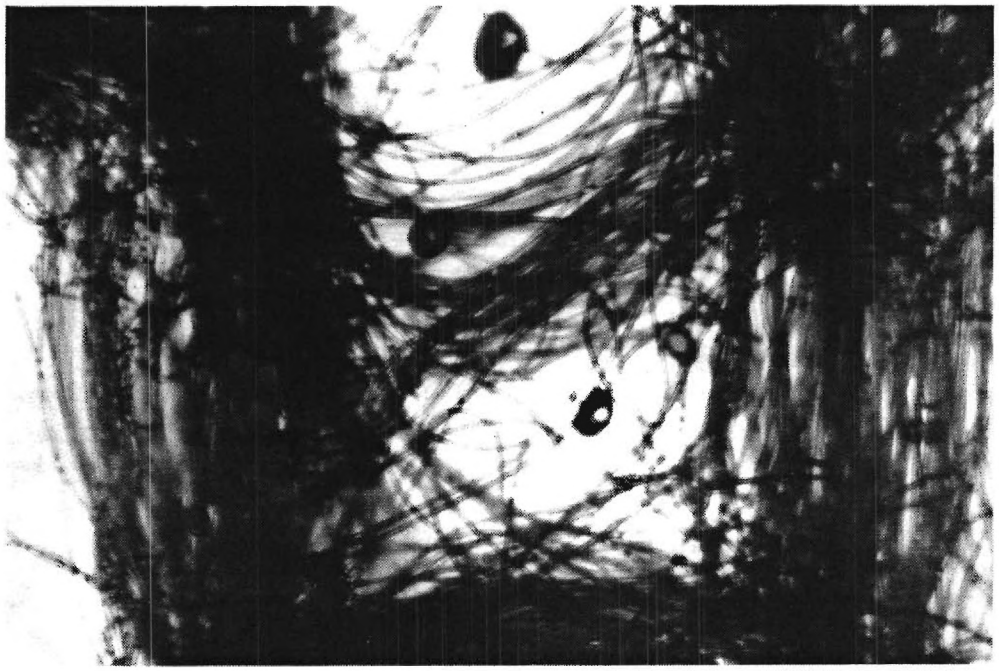
The air milled powder still required 45 psi air to fluidize on the Nordson unit. The powder coming out of the spray nozzle appeared to be predominantly the larger-sized particles. Only two samples, at 5.7% pick-up, were made. One sample was cured at 200°C for 1 min., and the other at 200° C for 2 min. In both cases, the polymer still stood on top of the fiber, and could be seen as glittery "dots" when viewing the fabric at an angle. PS-1 powder sprinkled on Mylar polyester film and cured in the oven also showed this behavior.

The problem appeared to be the wetting/flow properties of the PS-1. Photos were taken and showed very little flow (Fig. 33).

When the original-sprayed (not air milled) PS-1-bound fabric strips were placed under tension on the Instron, some of the powder particles were observed to release from the fiber surfaces and physically separate from the fabric. The release was

Fig. 33

Nonwoven Fabric C-9462 SOS-Bound with PS-1 Resin at 8.7% SAO (cure: 200°C/1 min.).



attributed to poor surface adhesion between the PS-1 and the polyester fiber.

The results of the Instron tests on the original sprayed fabrics are contained in Table 20. The breaking load was actually lower in the MD than that of the unbound (blank) fabric past a 3.6% SAO level (< 1447 psi.). Strength in the CD was slightly less than that of the unbound fabric (< 644 psi.). Mullen burst pressures were higher than the standard fabric, but comparable to the unbound fabric.

Comparative stress/strain curves are shown in Figs. 34-35. The visual plots re-emphasize the wide discrepancy in MD properties between the control (plant bound) fabric and the PS-1 samples. A ~2X improvement in CD tenacity may have been more a reflection of the poor CD properties of the control than the enhanced properties of the SOS-bound samples.

Samples B1 thru D1 gave a wash/pilling rating of "5" (Table 20). However, all three were noted to be "fuzzier" than the standard (plant bound) fabric after the procedure.

The accumlated results, both mechanical as well as visual, indicated that PS-1 was not a viable candidate for SOS binding of nonwovens. The material was therefore deleted from active consideration, leaving FA-252 and IF-3237 as the resins of choice for further research and proof-of-concept in Year 3.

Table 20

Ultimate Mechanical Properties of C-9462 Fabric SOS Bound by PS-1 Resin at Different Loadings (200°C/ 30 sec. Cure).

Sample	Solids (%)	Mullen Burst (lbs)	Machine Direction Properties			Wash/Pilling Ratings Face Side (5=Exc.,0=Poor)
			Stress (Psi)	Strain	Modulus (Psi)	
Standard[a]	7.3-7.5[b]	29.4	1752	0.17	22,450	5
Blank (Unbound)	0	38.5	1447	0.45	3357	3
A1	3.6	36.2	1515	0.33	6150	4
B1	4.9	39.8	1345	0.32	5623	5
C1	6.8	33.7	1321	0.35	4762	5
D1	8.7	36.9	1410	0.44	3795	5

Table 20 con't.

Sample	Solids (%)	Mullen Burst (lbs)	Cross Direction Properties		
			Stress (Psi)	Strain	Modulus (Psi)
Standard[a]	7.3-7.5[b]	29.4	382	0.32	350
Blank (Unbound)	0	38.5	644	1.23	502
A1	3.4	36.2	654	1.18	528
B1	4.9	39.8	631	1.20	569
C1	6.8	33.7	611	1.25	555
D1	8.7	36.9	620	1.20	460

Fig 34

Mechanical Properties of C-9462 Nonwoven Fabric at Different PS-1 Loadings (200°C/30 Sec. Cure): Machine Direction

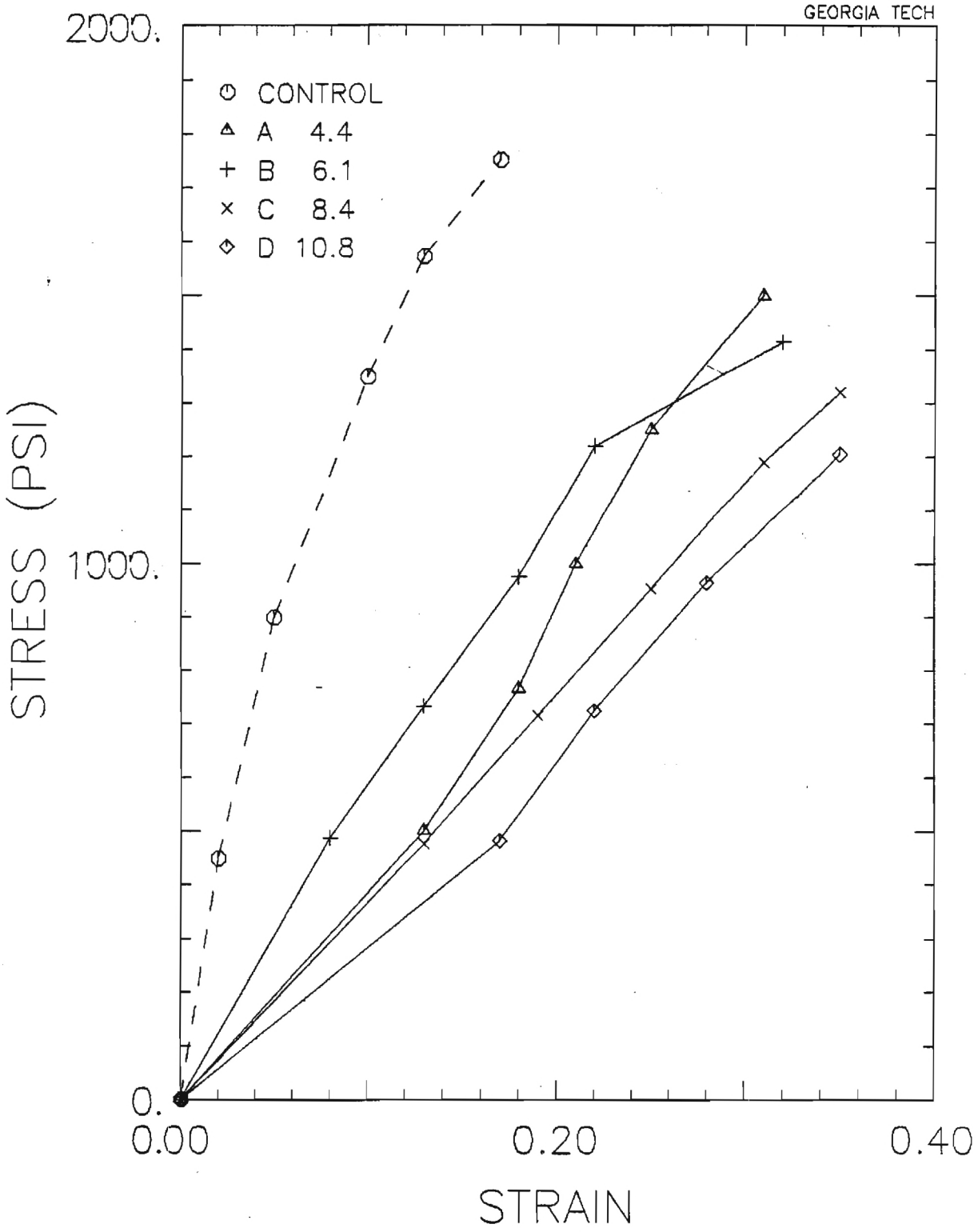
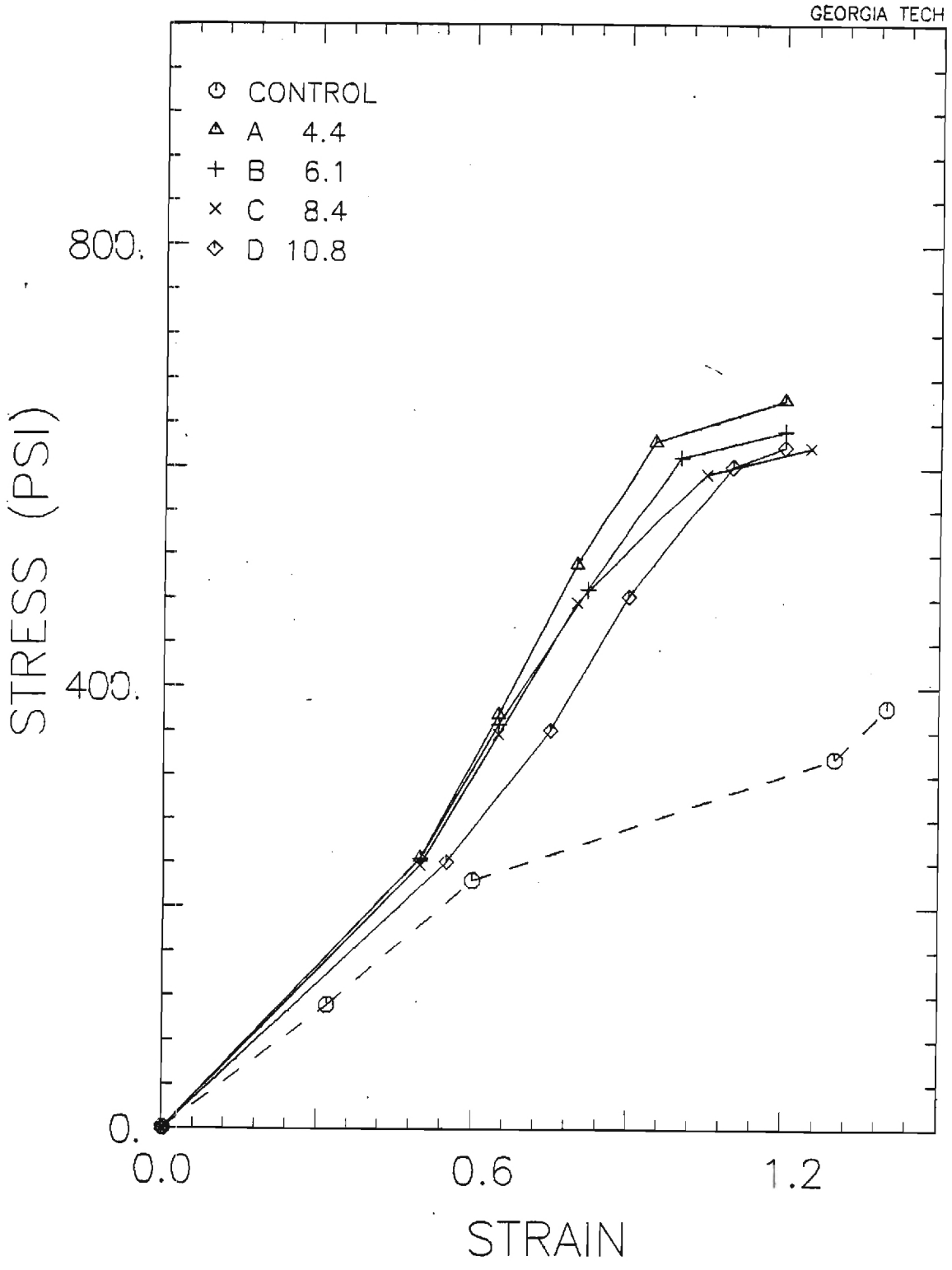


Fig 35

Mechanical Properties of C-9462 Nonwoven Fabric at Different PS-1 Loadings (200°C/30 Sec. Cure): Cross Machine Direction



FLUOROPOLYMER FINISHING OF A POLYPROPYLENE NONWOVEN

The initial candidate for fluoropolymer (FP) finishing of the polypropylene (PP) nonwoven investigated in Year 1 was the 3M Company's FC-214. The FP, supplied to the textile industry in 30% solids emulsion form, was designed for finishing of upholstery and drapery fabrics with properties:

Tm = 100°C (212°F)

Cure Temperature = 150°C (281°F)

Clarity = Clear, colorless when cured

Drycleanable = Yes

UV Stability = Good

The attractiveness of the FC-214 lay in the ability to isolate the (grindable) solid from the commercial emulsion by addition of absolute ethanol, followed by filtration. The FP was not molecular engineered, however, for the particular product area of the PP nonwoven.

The 3M Company notified the research team that a solvent stage in the production of several fluoropolymers had been identified that allowed isolation of solid polymer, i.e., emulsion breakdown was not necessary. Four new candidates were subsequently supplied to Georgia Tech for the SOS research:

Code	End-Use
L - 9XX2	PP Nonwoven, W/Additive
L - 9XX1	PP Nonwoven, W/O Additive
L - 9694	Resembles Carpet Finish
L - 9692	Textile Rainwear

The chemical structures of the supplied polymers were proprietary to the 3M Co.. The L - 9XX1 composition was the same as that currently applied by the participating nonwoven plant, and L - 9XX2 was the same FP compounded with the correct proportion of an additive. The L - 9692 FP was designed for rainwear, but was grindable to a free-flowing powder. L - 9694 was better suited for carpets than textiles (Table 21).

After general physical properties were defined for the new candidates, the Dot Adhesion Test (Table 2) was utilized to determine the affinity for Mylar polyester fiber. The results are displayed in Table 22. Earlier FC-214 results are shown for comparison. Only the L - 9XX1 FP gave any adhesion to the Mylar.

The L - 9XX1 and L - 9694 compositions were next evaluated. After grinding on the Bantam Mill at ambient temperature, the L - 9694 powder passed through a 200 mesh screen (< 125 μ particles). The powder fluidized well, and was sprayed using the small Nordson feed bed (due to the limited amount of FP available) and the pneumatic spray gun without electrostatics. The PP nonwoven fabric was mounted on wooden embroidery hoops as described for the earlier NW binder research, and the line speed was set at 100 fpm. A total of four SAO's were sprayed: 1.7%, 2.0%, 0.5% and 2.0%.

The 3M Kit Test utilized in the earlier FC-214 research was used to determine the barrier properties of the material. The method involves dropping a specific mixture of oil/solvents on the surface and determining penetration. Appendix 4 details 12 liquid mixtures used in the test, and Table 23 details the test.

Table 21

Additional FP Candidates for SOS Research

L - 9XX1

A high fluorine content fluoropolymer that has repellancy and soil release properties. White, fluffy powder which smoothes out after blending with 1% fumed silica.

Tm = 71°C (158°F)

Particle Size = 12-130 μ, largest fraction 60-80 μ

Adhesion = 99% on Mylar

L - 9694

Fluorochemical that is very substantive to synthetic fibers and provides good soil repellancy. Tan solid which ground to off-white powder.

Tm = 105°C(221°F)

Particle Size = 10-80 μ, fraction 40-50 μ

Adhesion = 0% on Mylar

L - 9692

A high fluorine content, acrylate - type polymer that has very high repellancy. White, free-flowing powder.

Tm = 85°C (185°F)

Particle Size = 5-160 μ, largest fraction 15-20 μ

Adhesion = 0% on Mylar

Table 22

Performance of Various FP Candidates in

DOT Adhesion Test [a] (DAT)

Material	Original Dots (#)	Removed by Tape (#)	Removed by Flexing (#)	Adhesion (% of Dots
L - 9XX1	100[b]	-	-	0
L - 9694	100	1	0	99
L - 9692	100[b]	-	-	0

a. See Table 2 for details of the DAT.

b. All of the solidified FP actually fell from the Mylar film as the film was raised to the verticle position before tape was applied, i.e., no apparent adhesion.

Table 23

Kit Test Method for Determining
FP Barrier Properties

1. Place drop on substrate from 1 in.
2. After 15 sec., remove excess with Kimwipe.
3. Examine area.
4. Failure: Darkening caused by penetration

   Pass: No darkening
5. Report: Kit Rating (highest numbered solution that passes)

   Average: 5 to nearest 0.5

Designed more for plant quality control testing than specific testing, the higher the Kit Number attained by a sample, the more efficient the barrier properties.

The performance of the L - 9694 - sprayed fabrics is spotlighted in Table 24, along with that of the control (plant-finished) fabric. The performance of the SOS fabrics was well below that of the plant-finished control (which used the L-9XX1 FP).

The L-9XX1 was ground on the Bantam, and sieving of the powder yielded the largest fraction on 120 mesh screen (>125 μ). Blending with 1% Cab-O-Sil flow modifier did not prevent the suspected clumping. The isolated powder would not fluidize on the small Nordson feed bed, preferring instead to "channel". The fraction passing through a 200 mesh screen was then placed in the cleaned bed, and exhibited the same channeling behavior.

A Coulter Counter Model TA&A, located at Armstrong Powder Co., Warsaw, Indiana was used to determine the particle size distribution of the three fluoropolymers sprayed in the Nordson units. The Coulter Counter plots of the powders retained on a 400 mesh screen are shown in Figures 36 - 38, with Fig. 39 giving for comparison the distribution for the supplied Eastman FA-252 polyester powder. The L-9692 FP gave a much flatter and broader size distribution than the other three materials.

The 3M Co. at this point agreed to supply the research team with L-9XX1 FP (the plant material) blended with the normal additive. The reformulated material was coded L-9XX2.

Table 24

Performance of L - 9694 Sprayed Fabrics in the 3M Kit Test

Sample	SAO (%)	3M Kit Number (Passed)
A	1.7	4
B	2.Ø	2
C	Ø.5	-
D	2.Ø	3
Control (Plant Fin.)	—	7[a.]

a. Wet at No. 7, but not soaked through.

FIG. 36

Coulter Counter Particle Size Distribution of L-9XXT FP,

400 Mesh-Retained Sample

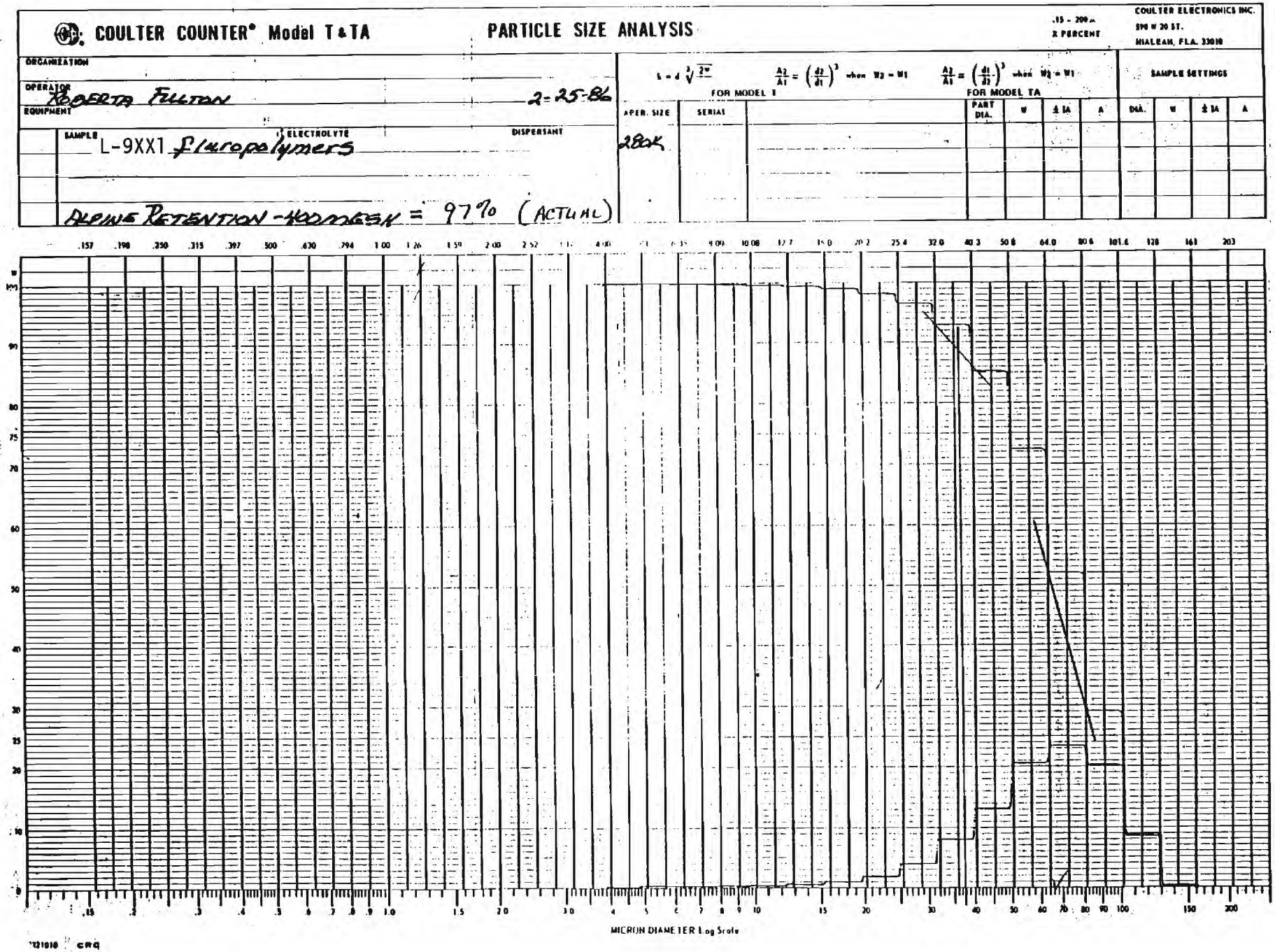


FIG. 37

Coulter Counter Particle Size Distribution of L-9694

400 Mesh-Retained Sample

COULTER COUNTER® Model T&TA — PARTICLE SIZE ANALYSIS — .15 – 200 μ — % PERCENT

COULTER ELECTRONICS INC.
590 W 20 ST.
HIALEAH, FLA. 33010

ORGANIZATION

OPERATOR ROBERTA FULTON 2-25-86

EQUIPMENT

SAMPLE L9694 fluoropolymer ELECTROLYTE DISPERSANT

ALPINE RETENTION · 400 MESH = 81%

$k = d\sqrt[3]{2^n}$ FOR MODEL T $\frac{A_2}{A_1} = \left(\frac{d_2}{d_1}\right)^3$ when $W_2 = W_1$ $\frac{A_2}{A_1} = \left(\frac{d_1}{d_2}\right)^3$ when $W_2 = W_1$ FOR MODEL TA

APER. SIZE	SERIAL		PART DIA.	W	±tA	A
284K						

SAMPLE SETTINGS	DIA.	W	±tA	A
	—	—	—	

.157 .198 .250 .315 .397 .500 .630 .794 1.00 1.26 1.59 2.00 2.52 3.17 4.00 5.04 6.35 8.00 10.08 12.7 16.0 20.2 25.4 32.0 40.3 50.8 64.0 80.6 101.6 128 161 203

W 99 95 90 80 75 70 60 50 40 30 25 20 10 0

.15 .2 .3 .4 .5 .6 .7 .8 .9 1.0 1.5 2.0 3.0 4 5 6 7 8 9 10 15 20 30 40 50 60 70 80 90 100 150 200

MICRON DIAMETER Log Scale

FIG. 38

Coulter Counter Particle Size Distribution of L-9692

400 Mesh-Retained Sample

COULTER COUNTER® Model T&TA — PARTICLE SIZE ANALYSIS

.15 - 200 μ
X PERCENT

COULTER ELECTRONICS INC.
590 W 20 ST.
HIALEAH, FLA. 33010

ORGANIZATION

OPERATOR ROBERTA FULTON 2-25-86

EQUIPMENT

SAMPLE L9692 Fluoropolymers ELECTROLYTE DISPERSANT

ALPINE RETENTION - 400 MESH = 41% (ACTUAL)

$L = d\sqrt[3]{2^n}$ $\frac{A_2}{A_1} = \left(\frac{d_2}{d_1}\right)^3$ when $W_2 = W_1$ $\frac{A_2}{A_1} \propto \left(\frac{d_2}{d_1}\right)^3$ when $W_2 \neq W_1$

FOR MODEL T FOR MODEL TA

APER SIZE	SERIAL	PART DIA.	W	± IA	A
2BOK					

SAMPLE SETTINGS DIA.	W	± IA	A

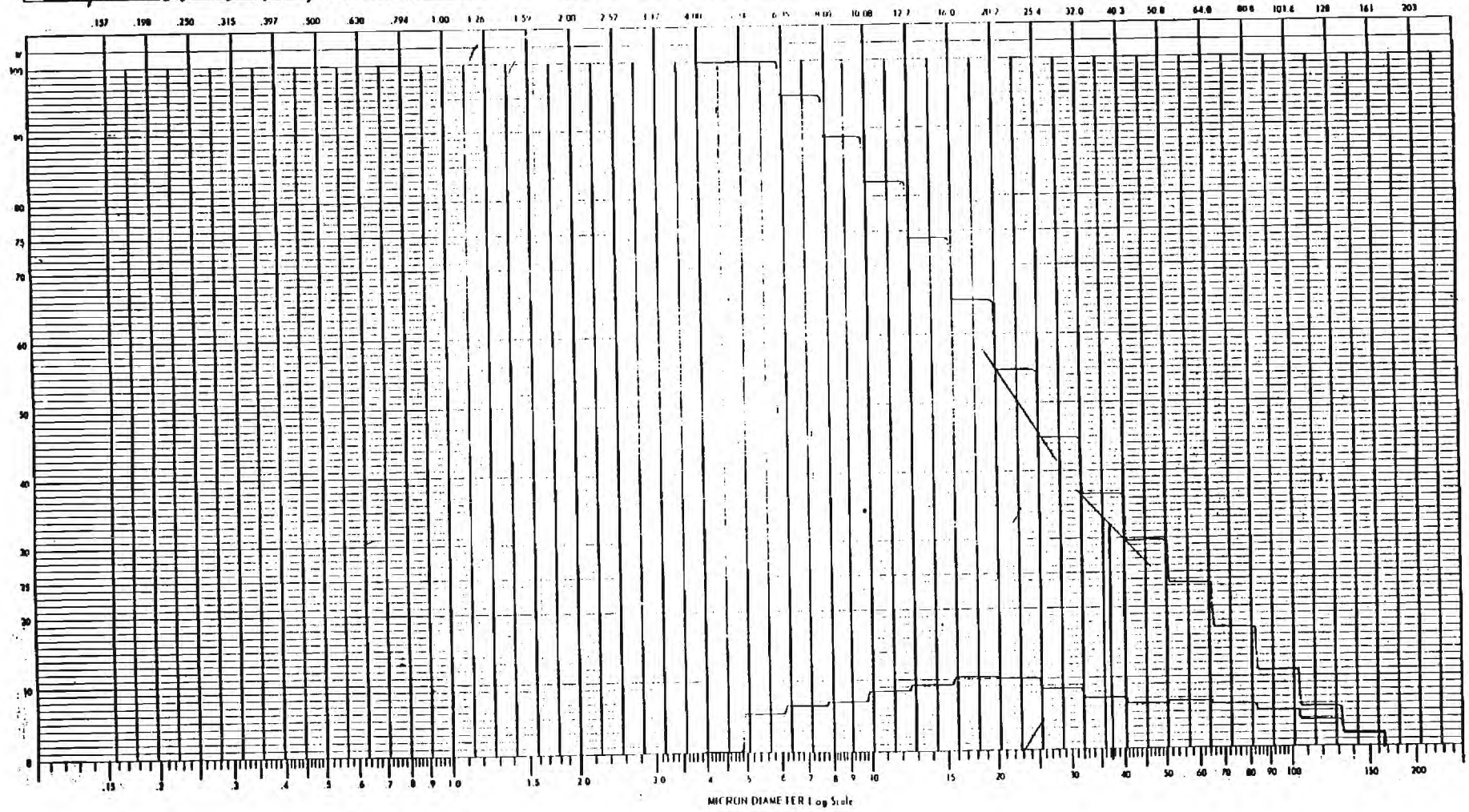
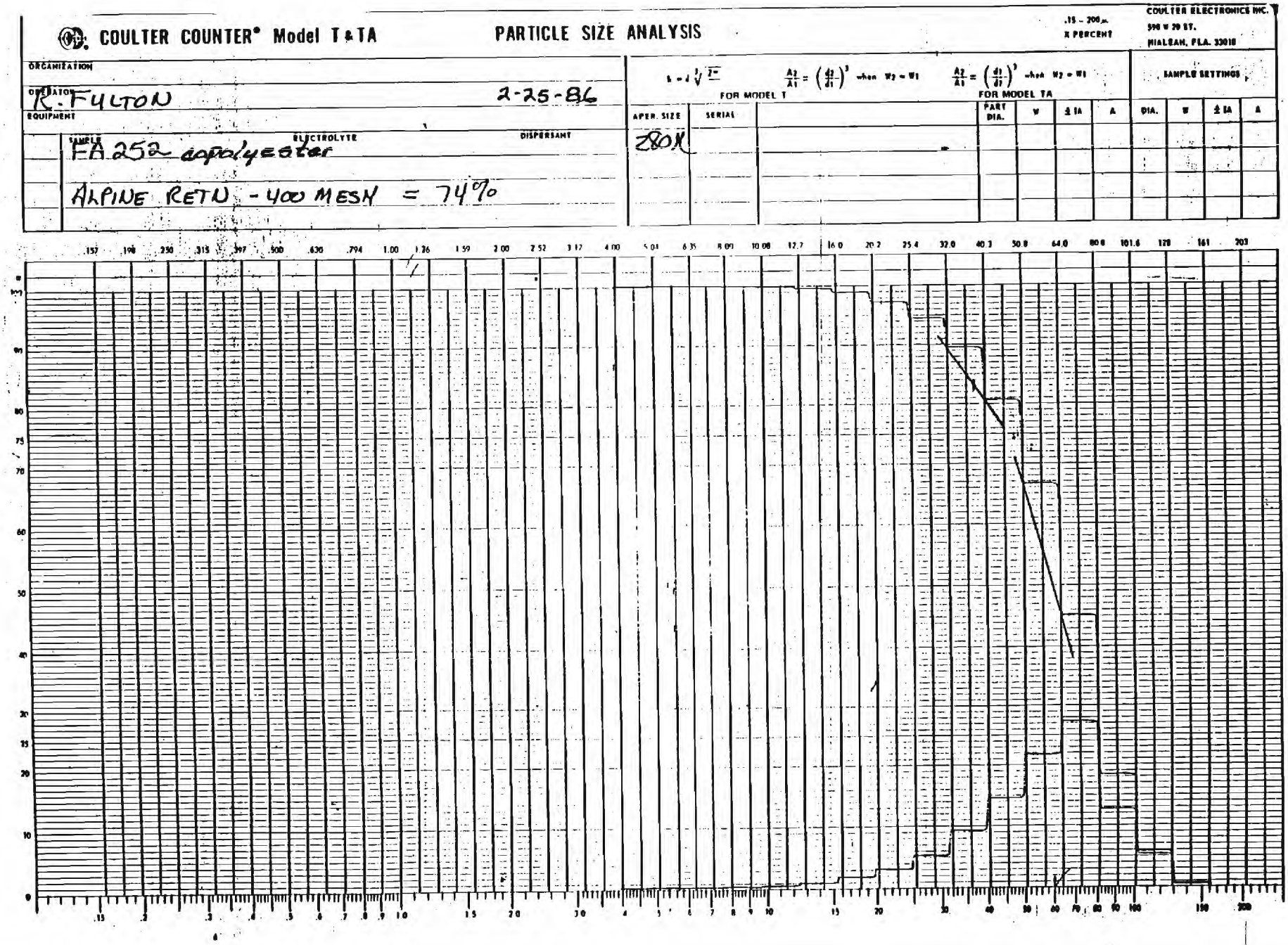


FIG. 39

Coulter Counter Particle Size Distribution FA-252 Polyester,
400 Mesh-Retained Sample



After cryogenic grinding and sieving, initial attempts to fluidize L-9XX2 failed. The spray-dried emulsion solids were placed in the large Nordson hopper and the fluidizing air pressure raised, still with no fluidization. Due to the "lumpy" nature of the powder, moisture was suspected. The powder was left in the bed with the fluidizing air on overnight to dry the powder. The next morning, there was no improvement seen in the powder behavior.

Grinding the powder in the air mill was then attempted. The process was particularly inefficient because the powder did not self-feed into the mill from the vibrating feed trough. The powder had to be hand fed into the mill. Grinding was halted once enough powder had been ground to cover the bottom of the small fluidized bed hopper and fluidization was rechecked. No improvement was evident.

Personnel at 3M offered that the addition of Cab-O-Sil should cause no problems with the efficiency of the fluoropolymer. A finely-ground calcium carbonate was also suggested. Nordson Co. suggested changing the pump parts of the unit to low flow components. Experiments using only one pump, if the flow rate was still too high, were also suggested.

The L-9XX2 FP was next reground on the air mill, vacuum dried (24 in. Hg, 50°C) and levels of 1%/2%/3% (by weight) of Cab-O-Sil blended with separate portions of the powder. Fluidization was attempted with the six inch Nordson bed with and without vibration. Addition of > 2% Cab-O-Sil, coupled with bed vibration, was necessary to achieve minimal bed fluidization.

The obvious hydrophilic nature of the L-9XX2 FP was a critical barrier to its use in a SOS mode. Further research in Year 3 was planned to attempt circumvention of the hydroscopic nature of the material, along with the attendent powder clumping/channeling problems.

SLASHING OF A POLYESTER/COTTON STAPLE BLEND YARN

In addition to the two SOS size polymer candidates defined in Year 1 (Eastman WD and Carboset 525), research was also conducted in Year 2 on a proprietary size supplied by Burlington Industries that had been previously used in Hot Melt Slashing research (henceforth referred to as HMS size). Flakes of the size were ground on the Bantam apparatus at room temperature and sieved to give three fractions:

a. 66% retained on 60 mesh screen

b. 29% retained on 120 mesh screen

c. 5% finer than 120 mesh screen

Fraction a was chilled overnight on dry ice and reground to give three new fractions:

d. 36% retained on 60 mesh screen

e. 50% retained on 120 mesh screen

f. 6% finer than 120 mesh

The 125-250 μ particle size sample was utilized in the ETI Fluidized Bed (5" x 5") to evaluate the HMS material as to solids pick-up (SPU) on the (single) polyester/cotton staple yarn as a function of line speed and voltage setting. No Cab-O-Sil flow

modifier was added, and a vibrator was necessary to fluidize the bed. The initial procedure for operation of the ETI Slashing line is contained in Appendix 5.

Set line (Fig. 6) conditions were:

Ground Teflon Wiping Block: 80% of 120 V setting

Air Flow: 2 psi

Oven T: 220°C (425°F)

A SPU of 15-20% was sought. The investigated line speeds and charged plate voltage settings are shown in Table 25. For reference on oven flow, correlation between line speed and oven dwell time for the system was:

Line Speed (ypm)	Oven Dwell Time (sec.)
12	1.8
10	2.17
5	4.3
4	5.4
2	10.8
1	22

The Table 25 data show the extreme flexibility of the ETI system in controlling SPU by line speed and/or voltage setting. Realistically, with the high melt viscosities of the investigated polymers, oven dwell times of less than 5-6 seconds would not give adequate flow of the size to produce a uniform film. In the Table 25 study, samples run at 12 ypm presented a rough, "sandpaper" surface texture, indicating incomplete melting/flow of powder particles.

Using a stereomicroscope outfitted with a 35 mm camera, four of the HMS-sized yarns were examined over SPU's in the 15-75% range. All of the yarns appeared "hairier" than any of the yarns sized with the melt-blended Eastman WD/adipic acid or Carboset

Table 25. SPU as a Function of Line Speed and Plate Voltage Setting: HMS Size

Line Speed (ypm)	Plate Setting (RV)	SPU (% by Weight)
2.Ø	Ø	23
2.Ø	1Ø	26
2.6	1Ø	3
3.3	3Ø	18
4.Ø	3Ø	12
5.45	35	16
6.Ø	35	9
6.7	35	11
5.45[a].	4Ø	36
5.45[a].	5Ø	75
5.45[a].	4Ø	16
5.45[a].	5Ø	46
6.7	5Ø	26
7.5	5Ø	18
7.5	6Ø	67
1Ø.Ø	6Ø	3Ø
12.Ø	6Ø	15
7.5	5Ø	18
7.5	6Ø	67
1Ø.Ø	6Ø	67
	7Ø	47

a. Repeat runs conducted on different days with modified sprocket/chain systems on the take-up winder.

525/ adipic acid formulations. Particles that had not completely flowed were visible, but not to a greater extent than was observed with the 60/40 and 40/60 polymer blends. Degree of encapsulation was difficult to determine as the transparent size was difficult to distinguish on the white yarn.

The solidified HMS size left on the inside wall of a glass beaker after melt pouring was used to conduct a mock desizing feasibility study. The film was released from the interface by hot water, but the size did not dissolve. Warm water containing ammonium hydroxide, however, solubilized the HMS size.

Melt viscosities were determined with the Brookfield Viscometer for the HMS and two melt-blended formulations (Table 26). All three of the sizes were in the desired 10^2 - 10^3 Cps range.

Since the HMS size offered no clear advantages over the melt blended size formulations, and the "hairiness" of the resulting yarns was more pronounced, research was concentrated on the blended materials. Efforts were directed toward optimizing the two candidate systems so that a decision could be reached on the most viable candidate for scale up.

A study of SPU vs. line speed was conducted with the 40/60 Carboset 525/adipic acid powder (125-250μ particle size fraction) using no electrostatics, i.e., the voltage setting was left off. Cab-O-Sil flow modifier was physically blended with the powder at a 0.5% by weight level using a Hobart blender. The bed vibrator was used, the air flow set at 2 psi and the heating elements of the teflon wiping block powered at 80% of full voltage. Oven temperature was also varied.

Table 26. Melt Viscosities of HMS and Melt-Blended Sizes

Size	Temp. (ºC	Viscosity (Cps)
HMS	191	475
40/60 Carboset 525/ Adipic Acid	160	2800
40/60 Carboset 525/ Adipic Acid	180	1060
60/40 Eastman WD/ Adipic Acid	160	2600
60/40 Eastman WD/ Adipic Acid	170	1220

Table 27 and Figure 40 detail the results. Duplicate runs on different days under the same conditions and 3.3 ypm line speed gave close correlation on SPU (213 vs. 205%). At set line speed (4.6 ypm) with increasing oven temperature, the apparent SPU decreased. Visually, "smoking" occurred at the exit of the oven at $T \geq 232°C$ (450°F). The smoke was suspected of being unreacted, entrained adipic acid, which sublimes at 205°C. Since the line speed and bed conditions were held constant, mass loss due to adipic acid evolution was substantial, with a drop from 61% SPU to 13% in the 216°-246°C temperature range.

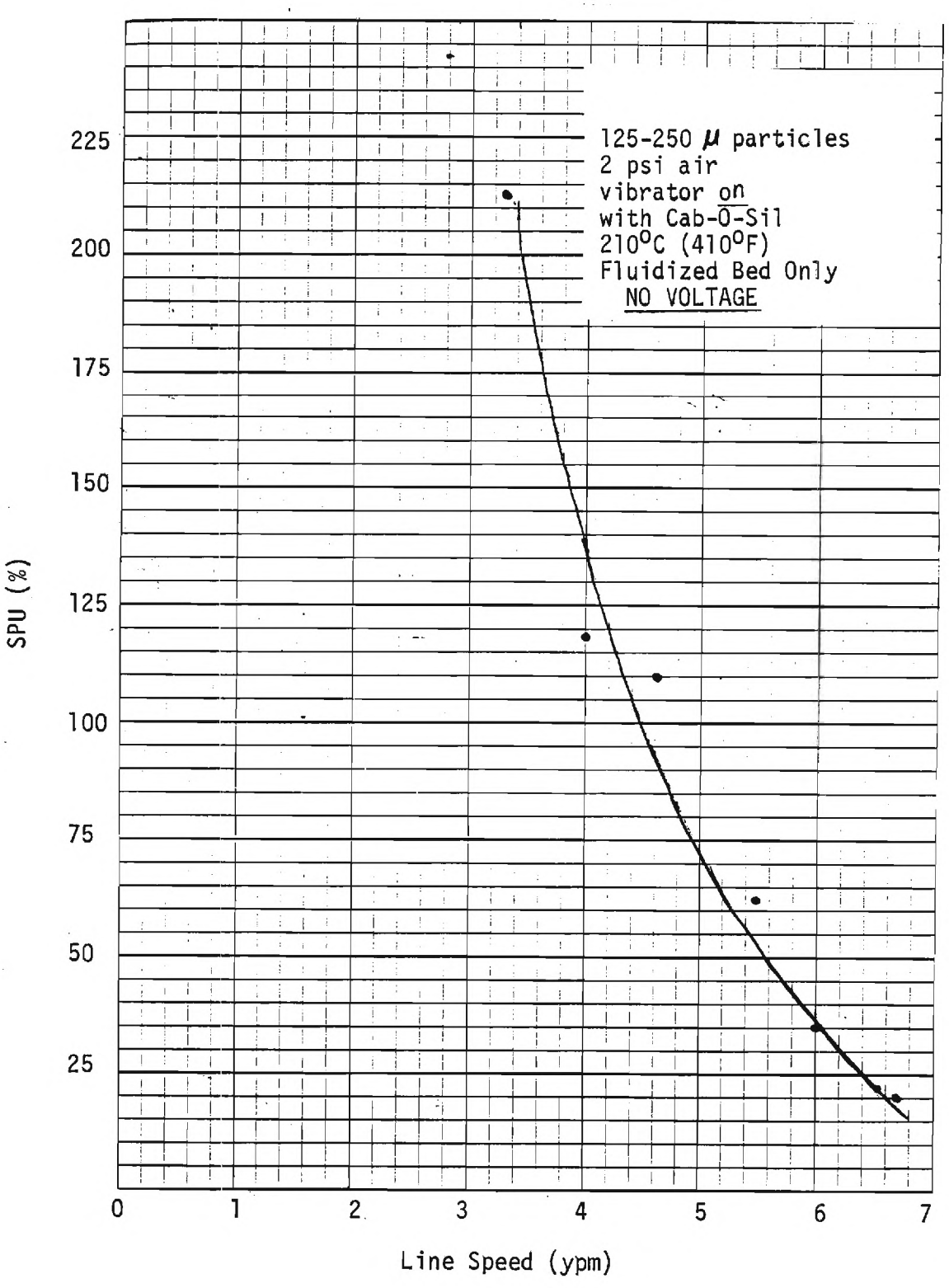
Stereomicrographs revealed that increasing the oven temperature gave no visual improvement of film formation/polymer flow on the yarns, and no improvement in degree of hairiness. The decrease in SPU with increasing oven temperature was also evident by the micrographs. For comparison, stereomicrographs were obtained of 60/40 Eastman WD/adipic acid - sized yarn samples. The 51% SPU sample showed more partially- melted particles than had the 40/60 Carboset 525/adipic acid samples, and the yarn was more encapsulated than with the 18% sample. The 51% SPU sample had more long hairs than the 18% SPU sample, while the reverse was true for short hairs.

At this point in the research, several modifications were made to the basic SOS slashing line (Fig. 6). In the initial construction of the wipe roll, a small hole for the yarn was drilled lengthwise through a 1 1/4" DIA x 1 1/2" L Teflon cylinder. The block was then surrounded with heating tape and connected to a Variac power source. In the modified

Table 27. SPU as a Function of Line Speed: 40/60 Carboset 525/Adipic Acid with 0.5% by Weight Cab-O-Sil.

Line Speed (ypm)	Oven Temp. (°C)	(°F)	SPU (% by Weight)
3.3	210	410	213
3.3	210	410	205
4.0	210	410	118
4.6	210	410	110
5.45	210	410	62
6.0	210	410	35
6.7	210	410	21
4.6	210	410	110
4.6	216	420	61
4.6	232	450	25
4.6	246	475	13

Figure 40. Relation of SPU to Line Speed in 40/60 Carboset 525/Adipic Acid Size Application



arrangement, five grooves were cut around the diameter of the outer surfaces of two 1 1/4" DIA x 4 3/8" L Teflon cylinders. The blocks were surrounded with heating tape connected to Variacs and were located as near as possible to the oven exit. The physical line arrangements with the original (single-wipe) and the modified (double wipe) Teflon rollers is shown in Fig. 41. Using a thermocouple inserted into the grooves of the double-wipe system's rolls, 80% and 100% voltage settings on the Variacs produced surface temperatures of 136° and 170°C, respectively.

A copper outer tube/pyrex inner tube system was also installed in the oven to give more uniform heating inside of the thermal zone. With the oven controller set at 210°C (410°F), the temperature/location in oven profile shown in Fig. 42 was generated by pulling a thermocouple connected to a meter through the thermal zone. The temperature was $\geq$ 200°C over an eight inch span of the thermal zone.

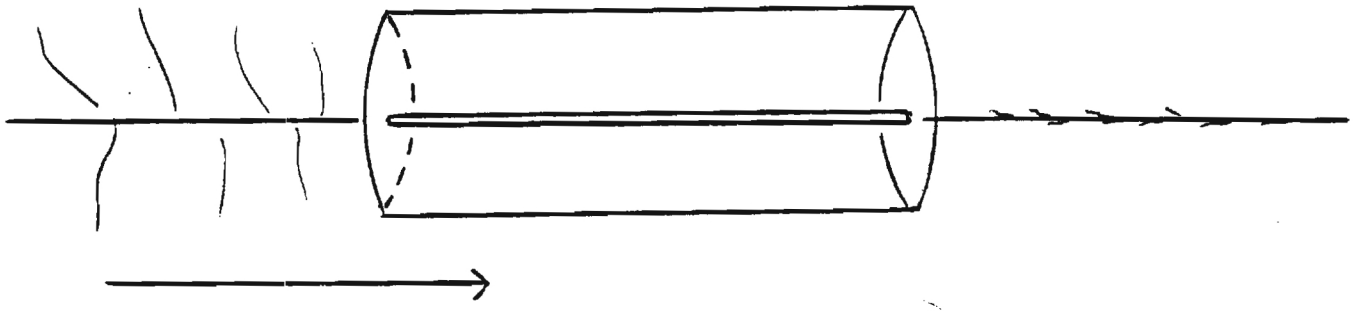
After the line modifications were made, long runs were attempted with both the 40/60 Carboset 525/adipic acid and 60/40 Eastman WD/adipic acid formulations (15% SPU level sought). The first long, single-yarn run was conducted with the 40/60 Carboset 525/adipic acid powder physically blended with 0.5% (by weight) Cab-O-Sil. The following run conditions were used:

Plate Voltage:	0 kV
Air Pressure:	2 psi
Line Speed:	6.7 ypm
Oven Temp:	210°C (410°F)
Vibrator:	On
Wiper System:	Two Roll, 170°C
Powder:	125-250μ fraction

Visually, the yarn appeared to pick up little powder when passing through the bed. SPU measurements could detect no gain in weight

FIGURE 41. Physical Line Arrangement of Single and Double Wipe Teflon Roll Systems

SINGLE WIPE



DOUBLE WIPE

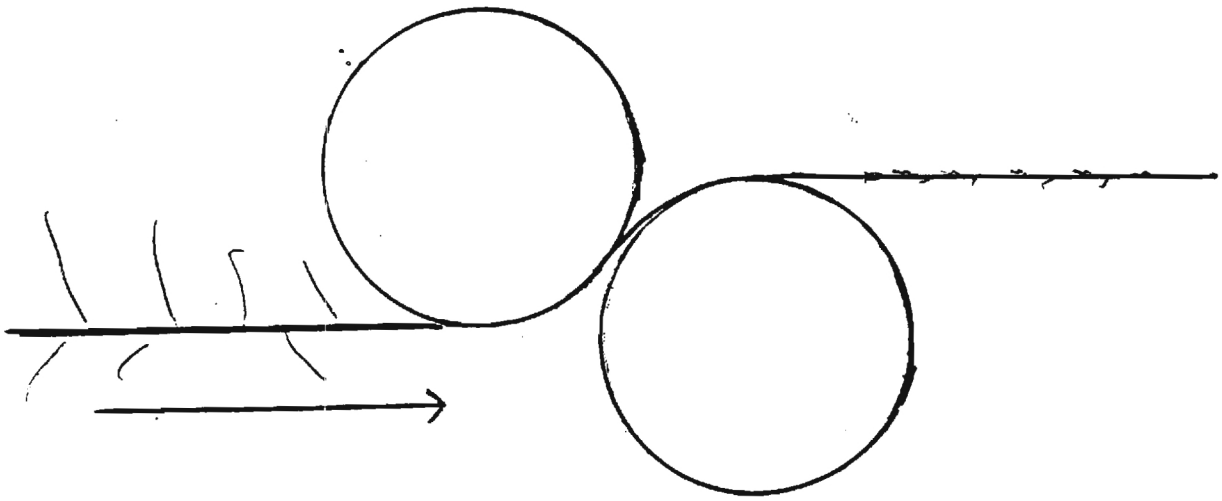
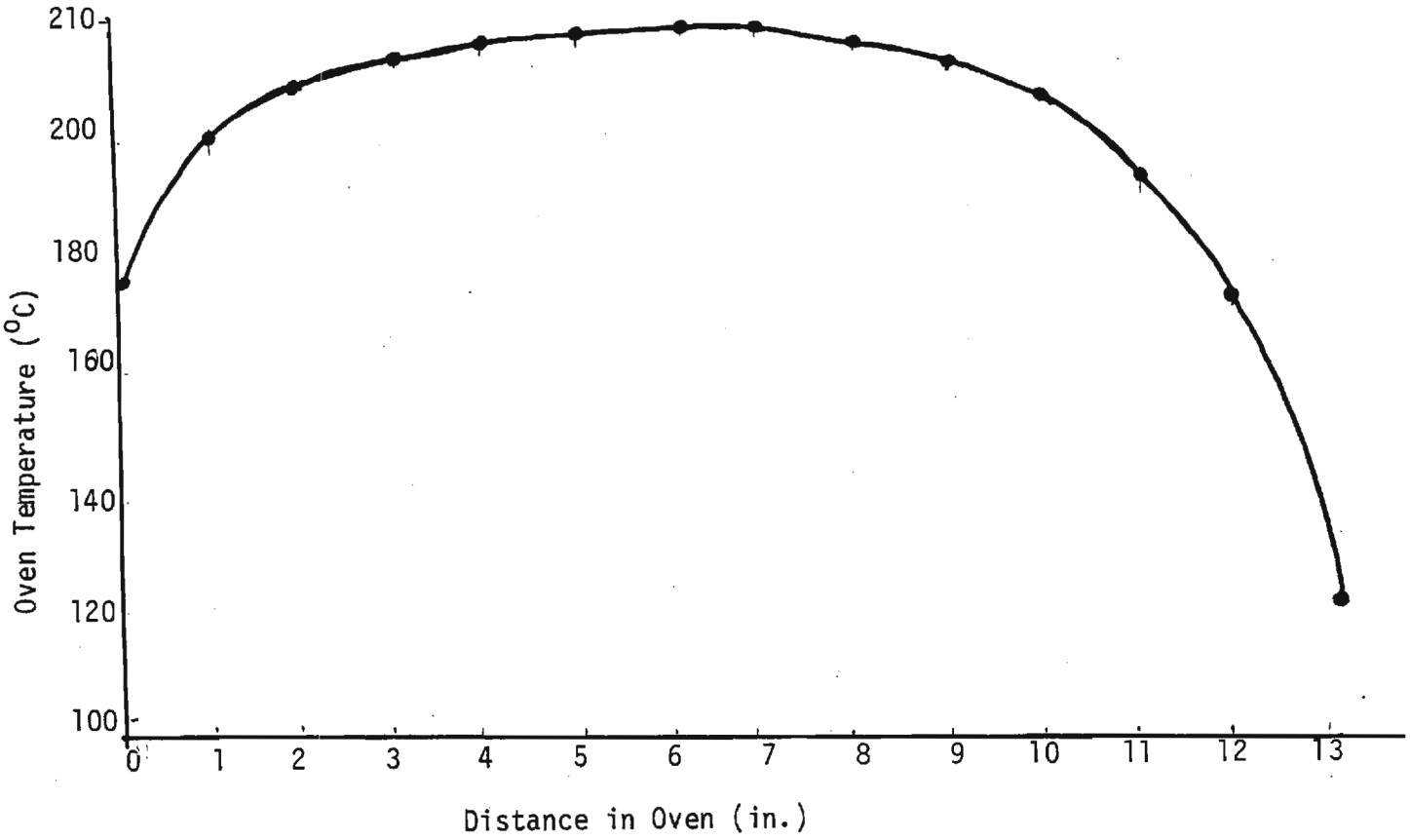


Figure 42 . Thermal Profile of the Modified Oven System



compared to the greige yarn.

A Toray Fray Counter was utilized to quantitatively determine the degree of hairiness of the yarn produced in the long run with the 40/60 Carboset 525/adipic acid size (test period: 60 sec.; running rate: 8 ypm). The standard plot of number of frays/yd. vs. fray length (mm) is shown in Fig 43. For comparison, Fig. 44 gives the fray data for the greige ring spun yarn and also the plant-sized (PVA) yarn.

A similar long run on ring-spun yarn was then conducted with the 60/40 Eastman WD/adipic acid formulation, including 0.5% Cab-O-Sil. The line conditions were the same as for the previous run except the following:

Plate Voltage:	45 kV
Line Speed:	2.5 ypm
Vibrator:	Off

The SPU level was measured at 9%.

The fray plot for the WD-sized yarn is shown in Fig. 45. The slashed yarn showed a higher population of short hairs than the Carboset-sized sample ($\leq$ 0.5 m length range, Fig. 45 vs. Fig. 43).

A Ruti Webtester was then used to evaluate the abrasion resistance of the two sized yarns, and as an indication of weavability. Four to seven sets of yarns were run on the machine, with 10 yarns/run, and the cycles-to-break recorded. The raw data is reported in Table 28 for the Carboset-sized sample, and in Table 29 for the WD sized yarn. Break 6 average was 1090 cycles for the former yarns, and 6392 for the latter. Overall, the Carboset-sized samples gave a 1032 average cycles-

Figure 43 . Fray Data from Long 40/60 Carboset 525/Adipic Acid Run

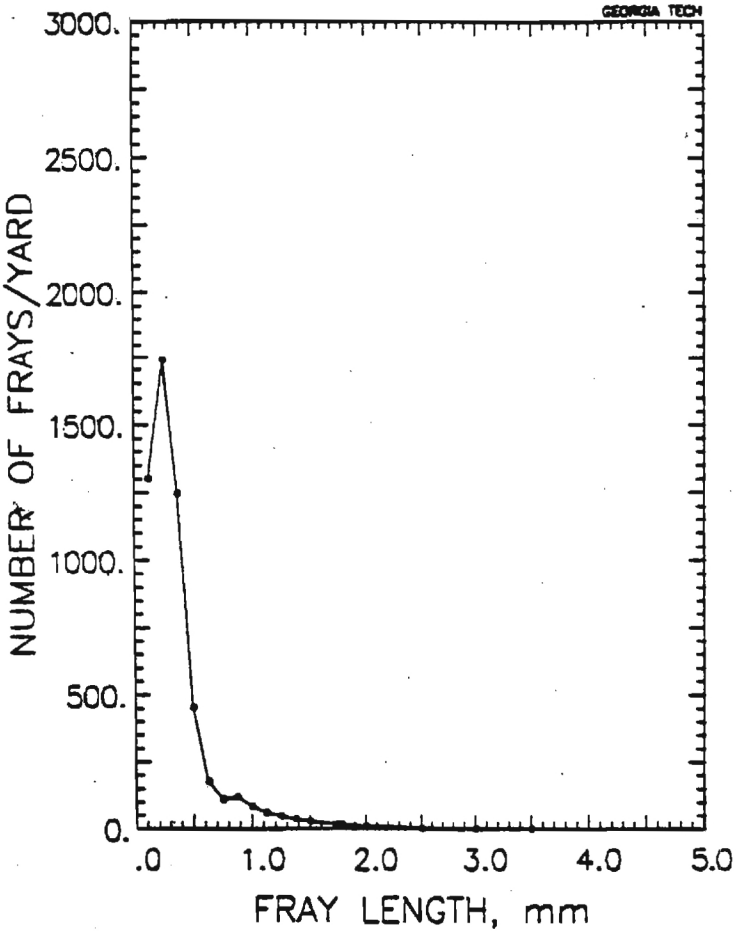


Figure 44. Fray Data for Greige and Plant-Sized (PVA) Ring Spun Yarns

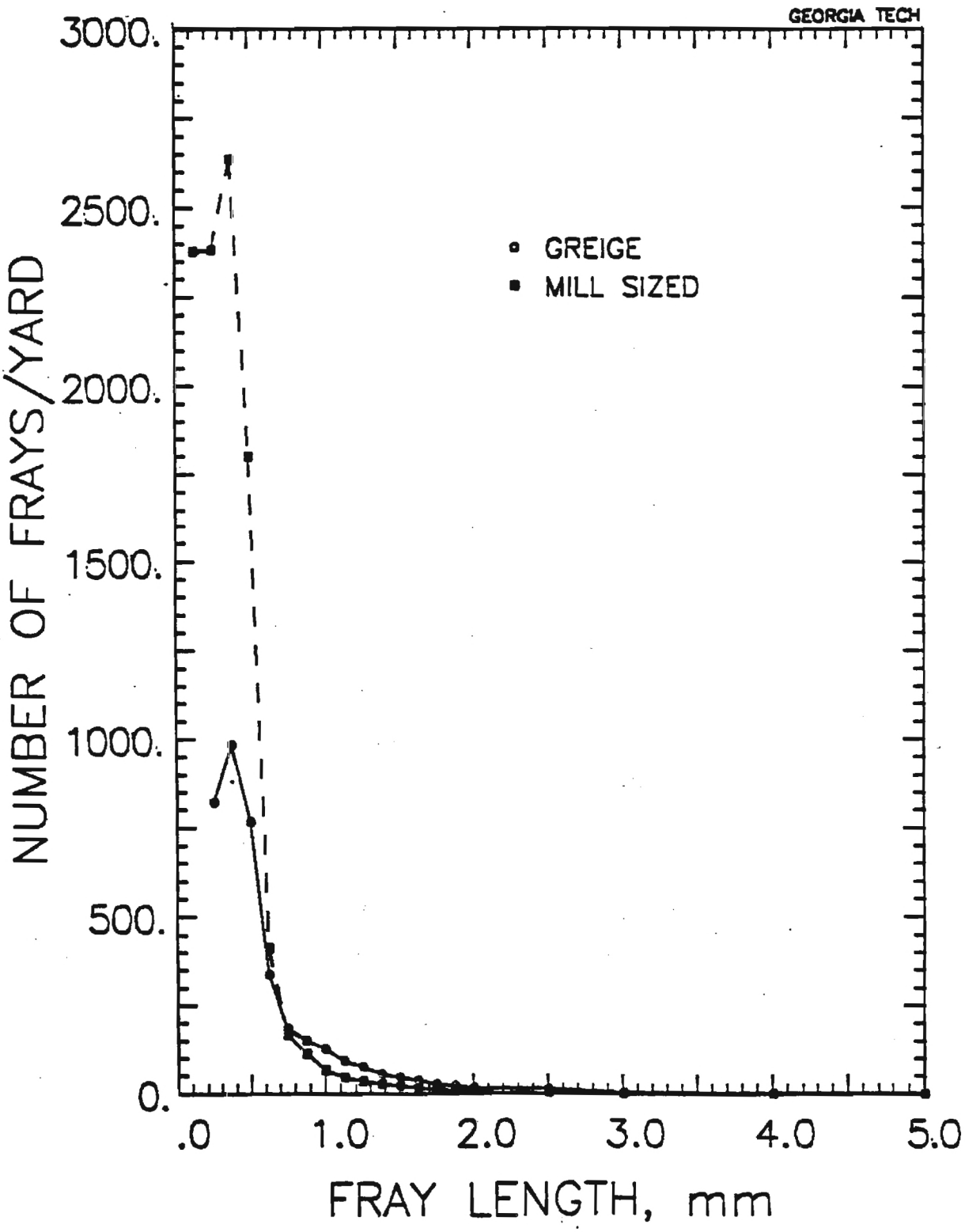


Figure 45. Fray Data from Long 60/40 Eastman WD/ Adipic Acid Run

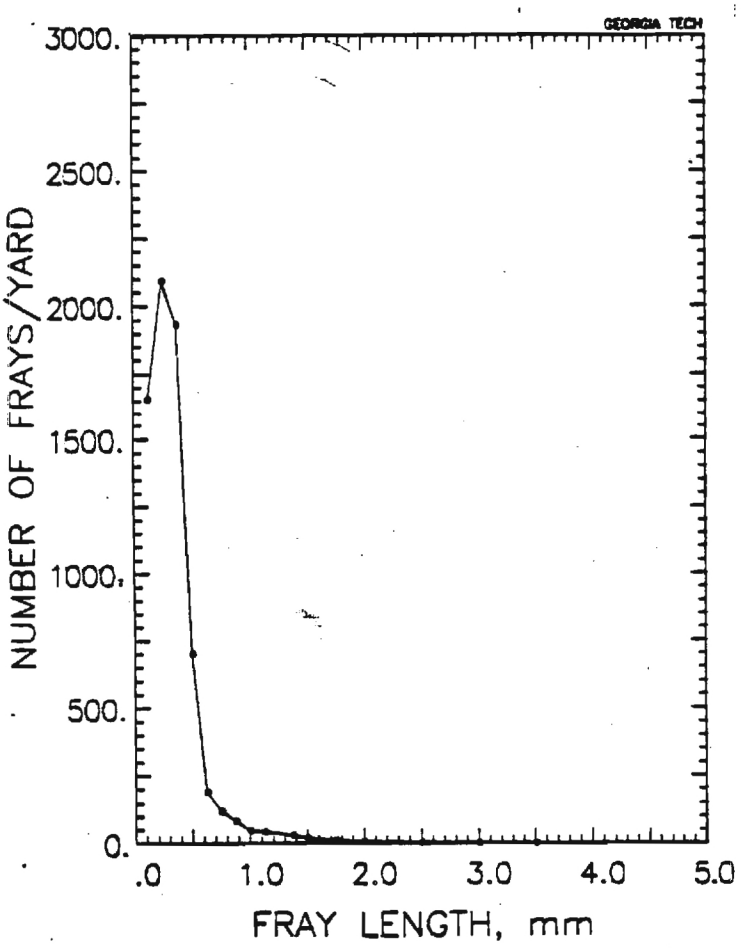


Table 28. Ruti Webtester Data for Long 40/60 Carboset 525/Adipic Acid Run

Ruti-Webtester Conditions Sample Code: 405-90-A-418-25-A
Pivot: 3.0
Cyclic elongation: 0.5%
Wt Tensioning: 10 gm
Cycles/minute: 400
Yarn tensioning: 7.8 N

Break No.	SET 1	2	3	4	
1	360	849	707	715	
2	759	857	725	830	
3	895	890	845	1073	
4	902	922	906	1107	
5	906	1132	930	1135	
6	1090	1187	945	1139	- Break 6 Aver: 1090
7	1099	1216	1077	1161	
8	1107	1387	1086	1165	
9	1124	1404	1165	1283	
10	1201	1518	1168	1356	
AVER	944	1136	955	1096	- Overall Aver: 1032
QUASI	707	730	---	505	
HAIR CLUMP	387	90	140	130	

Table 29. Ruti Webtester Data for Long 60/40 Eastman WD/Adipic Acid Run

Ruti-Webtester Conditions Sample Code 405-90-A-418-25-B
Pivot (abrading pins): 3.0
Cyclic elongation: 0.5%
Wt tensioning: 10 gm
Cycles/minute: 400
Yarn tensioning: 7.8 N

	SET							
Break No.	1	2	3	4	5	6	7	
1	4921	4082	3528	6931	4195	2419	2197	
2	6440	6505	3859	7087	4329	4263	2391	
3	7139	6583	4236	7862	4431	4348	2586	
4	8434	6606	5661	8548	4497	4479	2661	
5	8671	6838	6228	8704	4626	4543	2702	
6	8845	7217	6693	8827	4647	5692	2820	- Break 6 Aver: 6392
7	9037	7378	6696	9034	4658	5933	2998	
8	9543	7408	6763	9176	4690	6044	3054	
9	9974	7422	6950	9205	4879	6089	3140	
10	10845	7490	7127	9646	5152	6151	3170	
AVER	8385	6754	5774	8502	4610	4996	2772	- Overall Aver: 5099
QUASI	---	5570	---	---	3000	---	810	
HAIR CLUMP	170	700	370	350	140	170	145	

to-break, while the WD-sized samples averaged 5099 cycles-to-break. Although consistency was poor (e.g., compare Run 1 results with Run 7), the WD-sized yarns were clearly more abrasion-resistant than the Carboset-sized yarns. Questions remained, however, as to the amount of size deposited on the latter yarns (zero SPU measured).

The decision was reached to repeat the slashing run with the 40/60 Carboset 525/adipic acid/0.5% Cab-O-Sil formulation, with the following changes in the line conditions from the initial size run:

Line Speed:	6.75 ypm
Air thru Bed:	~ 1 hour prior to start-up
Powder Add:	at start-up

Conditioned, sized yarn gave a 20% SPU level. Two separate samples were then checked at different points in the take-up package, giving 60% and 13.6% SPU, respectively. Uniform deposition with time was obviously not being achieved on the single yarn line at this point in the research.

The tabulated data for the Toray fray counter is detailed in Table 30. The population of short hairs increased significantly over the previous run (e.g., 1248 frays/yd of 0.375 mm length in Run 1, compared to 3338 frays/yd in Run 2). The same held true in comparison to the WD-sized yarn (1933 frays/yd at 0.375 mm vs. 3338 frays/yd).

Ruti Webtester results for the repeat run are given in Table 31. The average cycles-to-break at the Break 6 point as well as the overall average improved ~3X over the initial Carboset 525 run, although the average breaks were only ~0.5X those obtained with the WD-sized yarn (compare Tables 29, 30 and 31).

Table 30. Fray Counter Data for Repeat of Long 40/60 Carboset 525/Adipic Acid Run

Toray Fray Counter Conditions Sample 405-90-A-418-30-A
Test Period: 60 sec
Running Rate: 8 ypm

Fray Length (mm)	Frays (#)	Aver. Frays (#)	Fray Density (#/yd.)
0.125	21194	21465	2683
	21735		
0.250	24749	24207	3026
	23664		
0.375	26012	26704	3338
	27395		
0.500	24799	25425	3178
	26050		
0.625	14668	16022	2003
	17375		
0.750	2886	3814	477
	4742		
0.875	366	476	59
	585		
1.000	117	168	21
	219		
1.125	44	56	7
	68		
1.250	47	55	6.8
	62		
1.375	88	84	10.4
	79		
1.500	11	12	1.5
	13		
2.000	13	13.5	1.7
	14		
3.000	0	0	0
	0		

Table 31. Ruti Webtester Data for Repeat of Long 40/60 Carboset 525/Adipic Acid Run

Ruti-Webtester Conditions Sample Code: 405-90-A-418-25-A
Pivot: 3.0
Cyclic elongation: 0.5%
Wt Tensioning: 10 gm
Cycles/minute: 400
Yarn tensioning: 7.8 N

Break No.	SET					
	1	2	3	4	5	
1	1761	1962	1700	2838	2145	
2	2126	2676	2981	2939	2226	
3	2188	2782	3064	3186	2530	
4	2834	2979	3171	3621	3047	
5	2838	3364	3518	3842	3163	
6	2871	3498	3760	3884	3215	- Break 6
7	2925	3522	4293	3968	3628	Aver: 3446
8	3254	3672	4374	3990	3656	
9	3269	3684	4733	4006	3748	
10	3298	3704	4952	4036	3967	
AVER	2742	3185	3655	3631	3133	- Overall Aver: 3269
QUASI	1910	3010	---	---	1800	

Consistency on the sets were good, going from a minimum average of 2742 cycles to a maximum of 3655 cycles. The procedural changes thus improved the powder pickup, with a corresponding improvement in abrasion resistance.

The uneveness of powder SPU down the single-yarn length was suspected of originating in change of powder bed depth with run time. The previous 40/60 Carboset 525/adipic acid run was repeated at two starting bed depths: 0.5 and 1.0 inches. Aproximately 300 yards of sized, ring-spun yarn was collected at each bed depth. Percent pick-up was checked on successive ten-yard lengths throughout the two packages, and SPU's were plotted as a function of location in the packages.

The results are shown in Fig. 46. Bed depth exhibited no measurable loss during the 300 yard runs. SPU variations with package location was periodic for both bed depths, and the periodicities synchronized well for the two runs.

With such short yarn samples being measured to determine weight change, the variation of the greige yarn denier itself, arising from variations in the yarn formation processes, was suspected of contributing to the perceived variation in SPU. Utilizing a Uster Eveness Tester, percent coefficients of variation (% CV's) as high as 30% were quantified with the greige, ring-spun yarn.

Plotting the weight of 10 yard samples of greige yarn against package location, Fig. 47 was obtained. Periodicity in the plot follows a similar pattern to that shown earlier for the SPU vs. package location (compare Figs. 46 and 47). Fluctuation

Figure 46. Variation of SPU with Position in Package of 300 Yard, 40/60 Carboset 525/Adipic Acid Run

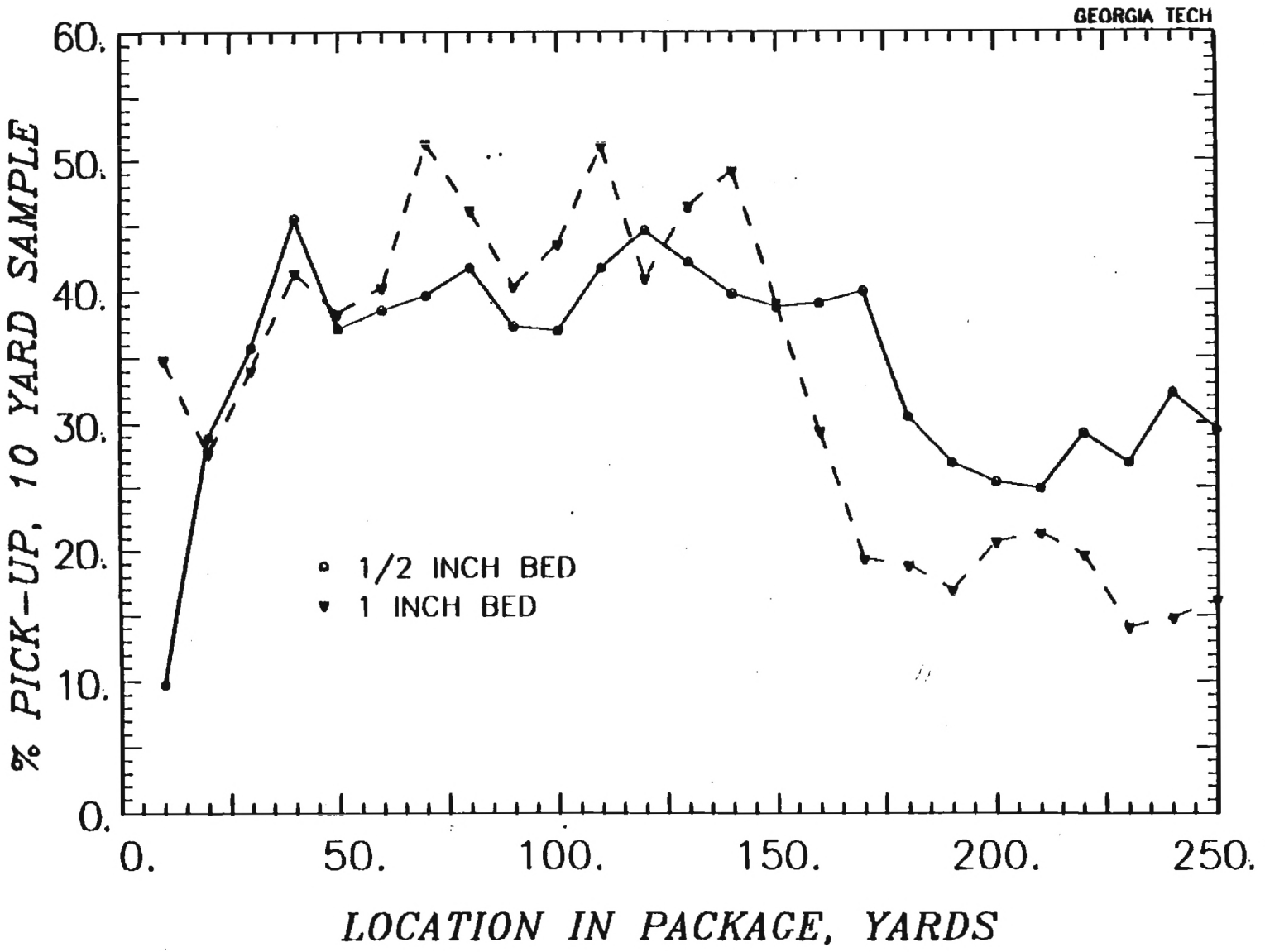
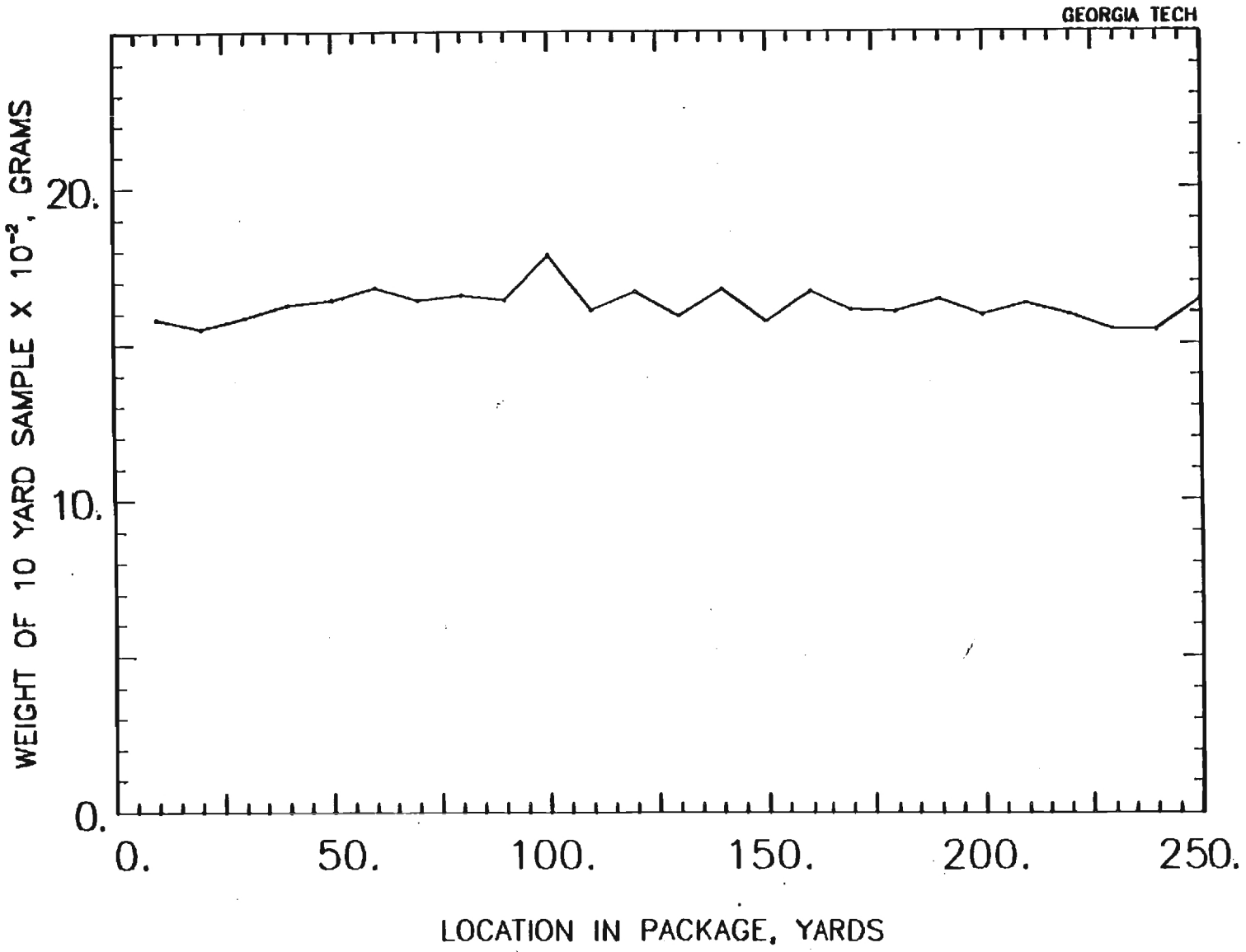


Figure 47. Variation of Greige, Ring Spun Yarn Weight with Location in the Package.



in greige yarn uniformity thus contributed to the perceived fluctuation of SPU vis-a-vis the way in which measurements were made, although the magnitude of the yarn weight fluctuation did not fully account for the total magnitude of SPU fluctuation.

Conversation with ETI indicated that large fluctuations occurred in the fluidized bed until flow rates were stabilized (~ 5 minutes). The recommendation was made that dry air should be flowed through both the size powder and the porous plate 1-2 hours before start-up of the yarn flow to stabilize the system, dry the powder and reach true bed steady-state conditions.

Comparative slashing runs were conducted with 70/30 and 60/40 melt blends of Eastman WD/adipic acid with 0.5% Cab-O-Sil. Dry air was run through the bed for one hour before start-up, followed by an additional hour through the bed/powder. SPU level was checked every 25 minutes for both runs, and was determined on samples: a) directly off the line; and b) after 24 hour conditioning in a standard textile testing lab (75% $\pm$ 2% R.H., 70°F $\pm$ 2°F temp.).

The SPU results are contained in Table 32. The 60/40 blend ran excellent, with good bed stability. The 70/30 blend, however, gave an unstable bed, with extreme variation (both visual and measured) in powder SPU. Both blends showed an apparent SPU change on conditioning under the testing lab environment. The sized yarn was thus susceptible to regain change, depending on the R.H./T conditions of the room.

The Toray fray plot for the two yarns are shown in Figs. 48 and 49. The 70/30 plot exhibited a higher peak in the short ($\leq$ 0.5 mm) fray length region, and also exhibited a higher

Table 32. SPU of On-Line and Conditioned Yarn Samples from Comparative Eastman WD/Adipic Acid Runs

Blend	Sample Wt.[a] (On-Line, gm)	SPU (%)	Sample Wt.[a] (Conditioned, gm)	SPU (%)
60/40	0.1720	5.0	0.17570	7.3
60/40	0.1700	4.0	0.17288	5.6
60/40	9.1870	14.2	0.19093	16.6
70/30	0.1890	15.4	0.19211	17.0
70/30	0.1740	6.3	0.17743	8.4
70/30	0.1945	18.8	0.19925	21.7
70/30	0.1720	5.0	0.17567	7.3
70/30	0.2070	26.0	0.21310	30.2
70/30	0.1865	14.0	0.19142	16.9
70/30	0.1663	1.6	0.16914	3.3

[a]. Based on 10 yard length.

Figure 48. Toray Fray Plot for Comparative 60/40 Eastman WD/Adipic Acid Run

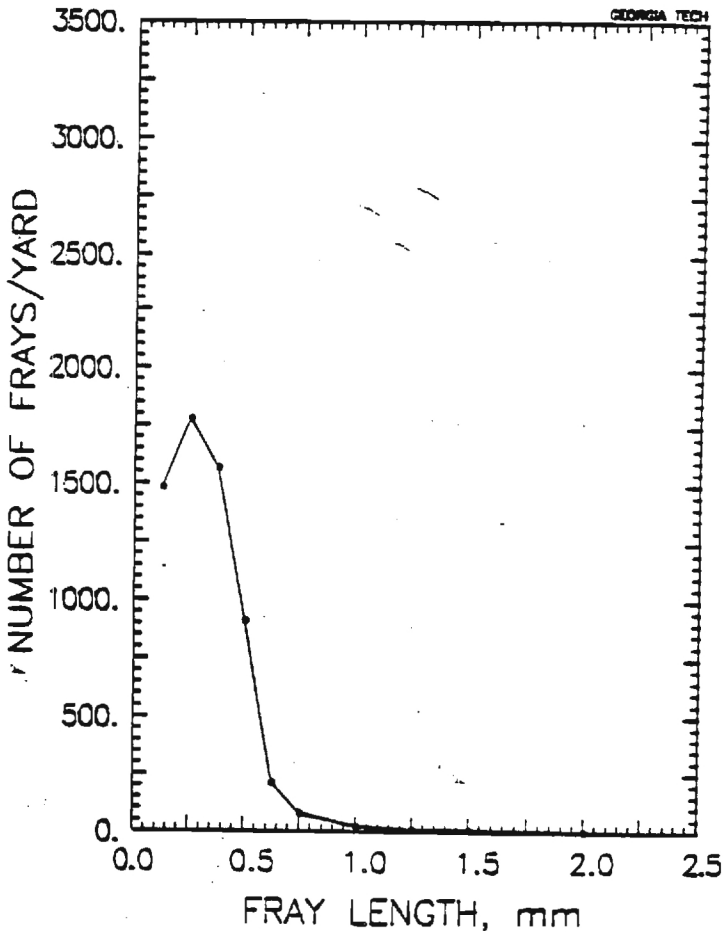
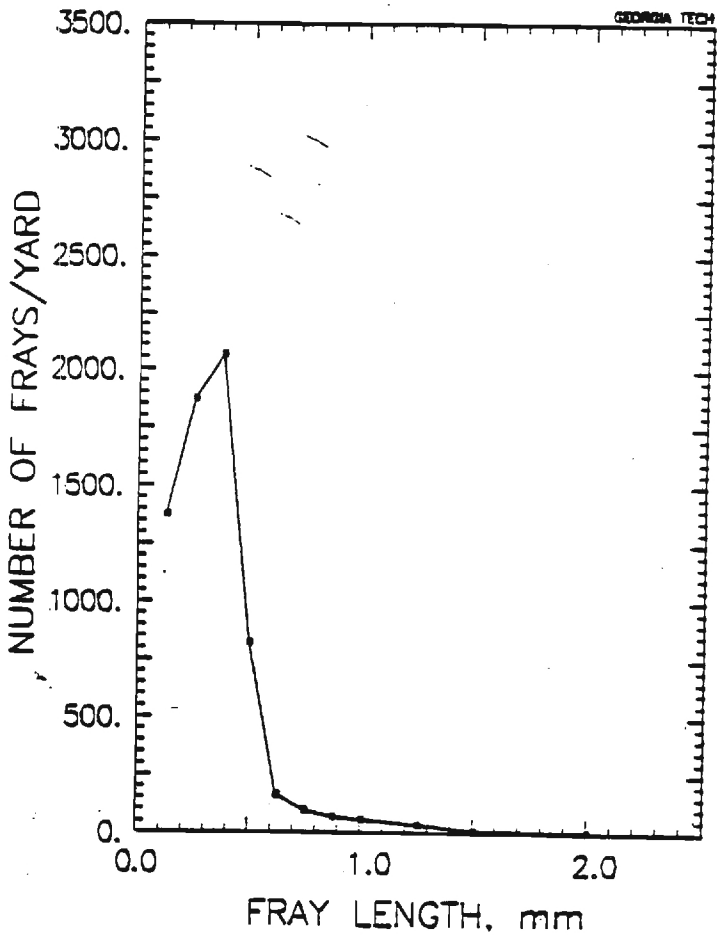


Figure 49. Toray Fray Plot for Comparative 70/30 Eastman WD/Adipic Acid Run



population of hairs in the 0.5 - 1.0 mm range. For comparison, Fig. 50 gives the fray plot for an earlier run with the 60/40 blend using both double- and single-wipes on the Teflon rolls. The double wipe system was used for both of the comparative runs. The 60/40 sample thus gave good fray correlation with the earlier run.

Ruti Webtester results for the two comparative runs are detailed in Tables 33 and 34. The 60/40 blend gave superior uniformity in the test, and ~2X higher average cycles-to-break.

SEM's were obtained for sized yarns from the two comparative runs. Observations of both yarns were similar:

1. Individual powder "particles", i.e., unflowed material, were apparent and appeared large compared to the fiber DIA (~ 2:1 ratio).
2. Some evidence of wetting followed by spreading along the fiber axis was observed.
3. Spreading appeared due to the wiping block action.
4. No film "bridging" was observed between fibers.
5. No total yarn encapsulation was found.

For base analysis, the commercially sized (PVA) yarn was also examined under SEM magnification. The plant-sized yarn was also not totally encapsulated, although individual fibers appeared to be. "Bridging" of PVA size film between individual fibers was prominent, despite the use of "bust rods" in the commercial (wet) process.

From the cumulative data, the conclusion was reached that the 70/30 Eastman WD/adipic acid gave inferior fluidization/SPU/

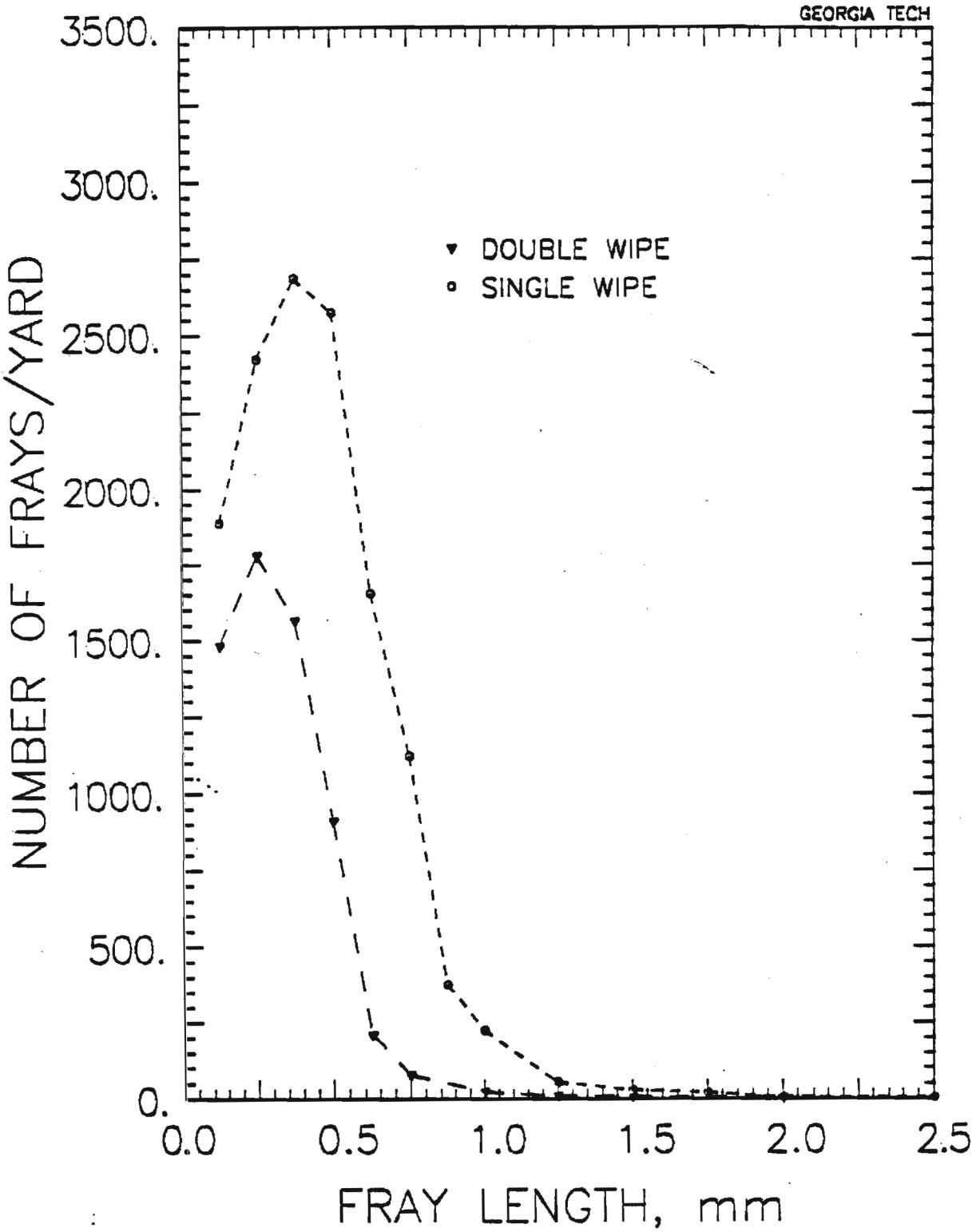


Figure 50. Fray Plot of Yarn Samples Generated with Double and Single Wipe Teflon Rolls: 60/40 Eastman WD/Adipic Acid

Table 33. Ruti Webtester Results for the Comparative 60/40 Eastman WD/Adipic Acid Run

Break No.	STEP Run 1	Run 2	Run 3	Run 4		
1	7996	7160	5839	6461		
2	8139	7517	6117	6855		
3	8162	7570	7025	7000		
4	8221	7641	7787	7034		Break 6
5	8350	7645	8177	7265		Average:
6	8360	7647	8610	7295	----	7978
7	8373	7883	8996	7305		
8	8414	7934	9030	7671		
9	8555	8353	9050	7784		
10	8593	8671	9130	7905		Overall Average:
Av.	8316	7802	7976	7258	----	7838
Hair Ball	1300	1070	1300	1600		
Quasi Break	-	-	2720	4560		

Table 34. Ruti Webtester Results for the Comparative 70/30 Eastman WD/Adipic Acid Run

Break No.	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	
1	1644	1503	3018	2646	1929	3026	
2	2413	3399	3192	2654	1969	3141	
3	2451	3518	3319	3309	2018	3155	
4	2787	3809	5153	4297	2651	3453	Break 6
5	2964	3911	6460	4312	2820	3491	Average:
6	3014	3995	6988	4826	3125	3642	-- 3444
7	3199	4016	7004	5294	3178	3654	
8	3234	4018	7147	5388	3324	4080	
9	3301	4778	7157	5590	3388	4428	
10	3331	4908	7158	6436	3681	4435	Overall Average:
Av.	2834	3792	5660	4475	2798	3651	-- 3868
Hair Ball	630	388	394	530	280	280	
Quasi Break	2730	-	4634	4130	225	2010	

final product properties to the 60/40 blend. The research was therefore concentrated on improving the performance of the 60/40 blend.

Researchers at Burlington Industries suggested addition of stearic acid to the 60/40 Eastman WD/adipic acid blend in the melt stage to provide greater lubricity to the size. A five-pound melt blend of 90% (by weight) 60/40 Eastman WD/adipic acid and 10% stearic acid (M.P. 69-70°C, density 0.847 gm/cm^3) was made by first combining the initial two ingredients, holding at the blend temperature for the prescribed period, and then adding the stearic acid. In pouring of the final melt for cooling, some phase separation was observed. The sample also appeared to solidify slower than earlier 60/40 melt blends.

The solidified size was frozen with dry ice, and poured quickly through the Wiley mill. The sample was then cryogenically ground twice, yielding a fine, powdery product. In comparison, the unmodified 60/40 blend had given a course, granular powder on cryogenic grinding that aggregated with shelf time unless 0.5% of Cab-O-Sil was added. On conventional sieving, the following cuts were obtained:

1. retained on 60 mesh (> 250 μ): 20%
2. retained on 120 mesh (125 - 250 μ): 77%
3. finer than 120 mesh (< 125 μ): 3%

The powder was then resieved using a vacuum over short periods in the shaker to reduce screen clogging. After several sievings (without regrinding), the results were:

1. retained on 60 mesh (> 250 μ): 3.8%
2. retained on 120 mesh (125-250 μ): 20.5%

3. retained on 170 mesh (88-250 μ): 47.1%

4. retained on 200 mesh (75-88 μ): 26.0%

5. finer than 200 mesh (< 75 μ): 2.6%

Attempts were made to apply the stearic-acid modified, blended size to the ring spun yarns using the 125-250 μ - range particles. The powder bed exhibited severe channeling without use of a vibrator. Vibrator addition resulted in visible "bubbling" of the sample, but no true cloud formation was attained with an air flow rate up to 8 psi. When yarn was passed through the chamber ~ 6 in. above the bed base (the normal location) no powder SPU was observed. The yarn was lowered to ~2.75 in. above the bed base, and after several attempts, the standard on-line weighing method revealed a 14.5% SPU level. The adjusted line conditions were:

Voltage:	400 kV
Current:	5 μ A
Air Flow:	8 psi
Line Speed:	1.15 ypm
Yarn Position:	2.75 in above base

SPU appeared uniform initially, but was suddenly and unexplainably lost at the 15 min. point, and could not be regenerated. The run was terminated.

The 75-90 μ powder fraction was then added to the empty bed. The powder fluidized for ~ 30 sec, then suddenly channeled, even with use of the vibrator. Addition of 0.5% fused silica and raising of the air pressure to 8 psi gave some improvement. Trials were made under the following range of

conditions:

Voltage:	42-52 kV
Current:	5-25 μ A
Air Flow:	8 psi
Line Speed:	0.5-1.18 ypm
Yarn Position:	2.75 in. above base

Little or no yarn SPU was observed. Some improvement occurred on grounding the oven, but insufficient to warrant testing the yarn. Efforts on the stearic acid-modified 60/40 Eastman WD/adipic acid size were therefore suspended.

Several changes were made in the slashing line system at this point (Fig. 6). An air pressure regulator was installed between the air dryer and the initial air inlet to the fluidized bed. A flow meter was also installed in the line. A copper rod attached to an earth rod with 14 guage wire was installed to fully ground the system. A Sola voltage regulator was also installed on the power feed line. A 3M Co. Static Eliminator Bar (SEB) Model 210 was placed just before the fluidized bed to discharge the moving yarn before powder application. The SEB contained a radioactive source, and required no electricity. The unit reduced both positive and negative yarn charges to near zero levels, and thus negated potential size SPU problems as the moving yarn attained static charge due to triboelectric and/or environmental influences.

A new wipe roll heater system was also designed and built by incorporating two Chromalox cartridge heaters (750 W, 107 W/in , 5 in. L x 0.5 in. DIA) into two aluminum rods (5 in. L x 0.75 in. DIA). The heater systems were then placed in two Teflon rods (8 in. L x 1.33 in DIA). The wiper systems were controlled by

implanted thermo-couples wired to separate digital units. The Teflon surfaces were ground with 20 indentions around the diameter of the rods to provide yarn registration. The new design provided:

1. Higher temperature at roll/yarn contact
2. More uniform heating
3. Multiple end capability
4. More accurate control/reading of contact temperature.

To check application mode, Burlington Industries applied the developed SOS size (60/40 Eastman WD/adipic acid) to the project's ring spun yarn by two proprietary Hot Melt (HM) slashing techniques. The variations used are detailed in Table 35.

The yarns were physically similar in feel to the SOS-slashed materials, but were very "twist-lively". The commercially-sized yarn (PVA) had exhibited little twist liveliness.

Ruti Webtester results for the HM yarns IB and IIB are shown in Tables 36-37. The overall average cycles-to-break were low (1475 and 875, respectively). Comparison to other materials is made in Table 38. The SOS-slashed yarns possessed superior abrasion resistance to the HM-slashed yarns using the same size blend formulation. However, the performance was only ~ 0.5X that of plant-slashed yarn.

To check performance of the Ruti Webtester machine with time, plant-sized, ring-spun yarn was subjected to repeated testing at four cycles/min. speeds. The size on the yarn consisted mainly of PVA, with a small percentage of wax. The initial testing of the yarn had occurred in July, 1985 (see results, Table 38), and the plant-sized yarn had been stored in a

Table 35. Variations Used in Hot Melt Slashing

Set	Method Code	Line Speed(ypm)	SPU (%)
IA	HM-48M	400	10.5
IB	"	"	"
IIA	HM-48M2	"	10.25
IIB	"	"	"

Table 36. Ruti Webtester Performance of HM-Slashed Yarn IB: 60/40 Eastman WD/Adipic Acid Size (B.I.)

Conditions:

Pivot setting:	3.0 mm
Cyclical elongation:	0.5 %
Weight tensioning:	10 gm
Cycles/minute:	400
Yarn tensioning:	7.8 N

Break No.	STEP Run 1	Run 2	Run 3	Run 4		
1	1030	961	505	481		
2	1240	1044	515	845		
3	1723	1204	623	972		
4	1964	1728	667	982		Break 6
5	1976	1875	855	1098		Average:
6	2073	2125	874	1140	---	1553
7	2155	2250	907	1148		
8	2219	2491	1208	1156		
9	2318	2657	1453	1170		
10	2334	3962	1674	1407		Overall Average:
Av.	1903	2030	928	1040	--	1475
Hair Clump	200	135	98	105		
Quasi Break	-	-	-	-		

Table 37. Ruti Webtester Performance of HM-Slashed Yarn IIB: 60/40 Eastman WD/Adipic Acid Size (B.I.)

Conditions:

Pivot setting:	3.0 mm
Cyclical elongation:	0.5 %
Weight tensioning:	10 gm
Cycles/minute:	400
Yarn tensioning:	7.8 N

Break No.	STEP Run 1	Run 2	Run 3	Run 4		
1	492	440	382	297		
2	515	769	482	524		
3	566	1002	568	674		
4	636	1012	592	736		Break 6
5	696	1052	613	861		Average:
6	879	1101	642	893	---	879
7	927	1205	769	1018		
8	964	1471	848	1040		
9	1006	1512	907	1256		
10	1034	1680	984	1945		Overall Average:
Av.	772	1124	679	924	--	875
Hair Clump	80	96	91	93		
Quasi Break	-	-	-	-		

Table 38. Comparison of Ruti Webtester Performance of Various Ring Spun Yarns

Application Method	Break 6 Aver. (# cycles/break)	Overall Aver. (# cycles/break)
Commercial (PVA)[a]	17008	16971
Greige (Unsized)	1396	1340
SOS (60/40 Eastman WD/ Adipic Acid) Sample[b]	7978	7838
HMS (60/40 Eastman WD/ Adipic Acid) Sample IB[c]	1553	1475
HMS (60/40 Eastman/WD Adipic Acid) Sample IIB[d]	874	875

a. Wet process, ~ 14% SPU

b. Best run to date, NB Code 405-79-A-418-44-A, 10-20% SPU

c. Run by Burlington Ind., 10.5% SPU

d. Run by B.I., 10.25% SPU

controlled environment in the School of Textile Engineering in the interim seven months between tests.

The Webtester performance of the commercial yarn in the repeat test is contained in Table 39. The average cycles-to-break had decreased ~2 - 2.5X compared to the earlier test. Discussions with West Point Pepperell researchers revealed that embrittlement of the plant size could occur over long time periods, probably due to slow crystallization of the PVA and/or minor wax components of the size. The second Webtester results were therefore invalidated, and the "old" yarn standard lot was discarded.

Toray fray counts were obtained for the HM-slashed yarn samples IB and IIB. The tabulated results are shown in Tables 40 and 41. HM Method I gave the lowest fray count. For comparison, the fray count for greige and plant-sized (PVA) ring spun yarns is shown in Fig 51.

The HM-sized yarns were less "hairy" than the plant-sized sample in the $\leq$ 0.5 mm range. The greige yarn was less hairy than any of the sized samples. For the plant sized yarn, the increased hairiness over the greige yarn was due to the "bust rod" action at the end of the wet slasher that breaks the PVA film bridge between yarns. For SOS slashed yarns, the hairs may have been "raised" from the main body of the yarn in the charged powder bed, and the hairs were not completely laid down in the wipe roll step. The reason for the increased HM yarns hairiness vs. the greige yarn was unclear, as the details of the two HM Burlington Industries processes were not revealed.

Table 39. Ruti Webtester Performance of Standard, Plant-Sized Yarn Nine Months after Initial Testing

Conditions:
Pivot setting: 3.0mm
Cyclical elongation: 0.5%
Weight tensioning: 10 g
Yarn tensioning: 7.8 N
Speeds: 200, 300, 400 and 500 cycles/min.

	SET/SPEED							
	200 cpm		300 cpm		400 cpm		500 cpm	
	1	2	1	2	1	2	1	2
1	7713	8065	9052	7462	7180	5720	5644	7199
2	7791	8068	9101	7557	7199	5766	5798	7236
3	7815	8103	9112	7579	7215	5814	5803	7316
4	7832	8113	9117	7586	7302	5873	5897	7364
5	7834	8117	9123	7590	7366	5876	5970	7373
6	7866	8121	9147	7625	7378	5909	5980	7387
7	7913	8133	9161	7634	7457	5923	6011	7408
8	7918	8288	9171	7649	7557	5962	6039	7413
9	7924	8339	9179	7699	7586	5967	6052	7465
10	7929	8344	9182	7703	7632	5991	6061	7488
Ave	7854	8169	9135	7608	7387	5880	5926	7365
Hair Clump	3195	2718	3200	3080	3250	2700	2920	3850
Quasi Break	3510	3010	4350	3375	3490	3842	3700	4375

Table 40. Toray Fray Data for HM-Slashed Yarn IB: 60/40 Eastman WD/Adipic Acid Size (10.5% SPU)

Toray Conditions:
Test Period - 60 sec.
Running Rate - 8 ypm.

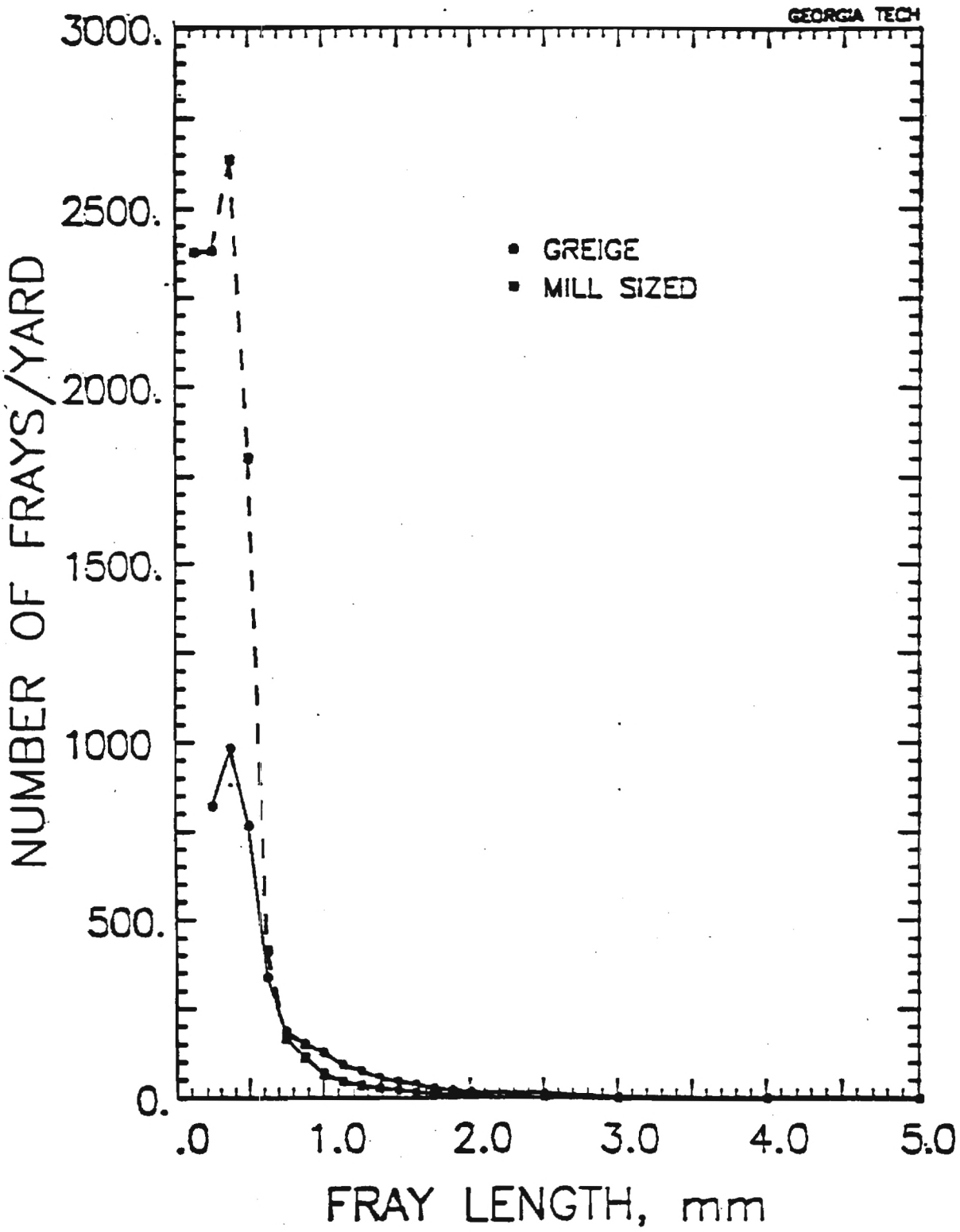
Fray Length (mm)	No. of Frays (#)	Aver. (#)	No. of Frays/ Yd.
0.125	11472		
	10385		
	10489	10782	1348
0.250	14801		
	14583		
	15901	15095	1887
0.375	16104		
	14844		
	14009	15002	1875
0.500	6000		
	5910		
	5833	5914	739
0.625	721		
	778		
	747	749	94
0.750	293		
	265		
	296	285	36
0.875	175		
	225		
	185	195	24
1.000	135		
	120		
	99	118	15
1.250	65		
	61		
	64	63	7.9
1.500	42		
	40		
	39	40	5.04

Table 41. Toray Fray Data for HM-Slashed Yarn IIB: 60/40 Eastman WD/Adipic Acid Size (10.25% SPU)

Toray Conditions:
Test Period - 60 sec.
Running Rate - 8 ypm.

Fray Length (mm)	No. of Frays (#)	Aver. (#)	No. of Frays/ Yd.
0.125	12022		
	12628		
	12485	12378	1547
0.250	17403		
	16972		
	16013	16796	2099
0.375	15658		
	15910		
	15841	15803	1975
0.500	6166		
	6075		
	5856	6032	754
0.625	864		
	810		
	893	856	107
0.750	275		
	308		
	264	282	35
0.875	188		
	173		
	183	181	23
1.000	140		
	116		
	103	120	15
1.250	74		
	63		
	58	65	8.1
1.500	21		
	39		
	37	32	4.04

Fig. 51. Toray Fray Data Plot for Greige and Plant Sized (PVA) Ring Spun Yarns.



Melt modifications to Eastman WD polyester had consisted of organic diacid interchange reactions up to this point in the research. Melt blending with two long-chain polyethylene glycols (PEG's) of low/high molecular weights (MW's) was undertaken to assess changes in sized yarn properties:

1. A 3400 MW polyethylene glycol from Polysciences (white, waxy, solid particles).
2. A 400 MW polyethylene glycol from Fisher Scientific (clear liquid).

The procedure used for the melt blending is detailed in Appendix 6.

The various melt formulations are given in Table 42. One formulation (No. 6) included both 3400 MW PEG and adipic acid. Formulation 7 substituted Eastman 252, the modified PET-based binder used earlier in the nonwovens research, for Eastman WD.

The Eastman WD/3400 PEG blend turned white when power mixing was started. A gas was evolved during the mixing, as evidenced by bubbling. Three layers formed in the container when stirring was stopped:

Top Layer: Solid Polymer
Middle Layer: Trapped Gas
Lower Layer: Melted Mixture

All of the samples were soluble in boiling water.

Melt viscosities, drop test results and film characteristics/ appearances are detailed in Tables 43-45. The 400 PEG material gave a very tacky melt blend with no film formation (Table 43). The 3400 PEG blend was a fiber-former at 85/15 and 75/25 levels,

Table 42. Melt Blended Formulations of PEG's with Eastman Polyesters

Formulation	Blend (by parts weight)
Eastman WD/3400 Polyethylene Glycol	90/10 85/15 75/25
Eastman WD/400 Polyethylene Glycol	90/10 75/25
Eastman WD/3400 PEG/Adipic Acid	80/10/10
Eastman 252/3400 Polyethylene Glycol	75/25

Table 43. Melt and Film Properties of Various Eastman WD/400 PEG Blends

90/10 Blend Sample No. 418-76-A	75/25 Blend Sample No. 418-70-A
Viscosity-dropped steadily over 1 hour initial = 55,000 Cps 1 hour = 22,400 Cps	Viscosity-dropped steadily over 1 hour inital = 26,400 Cps 1 hour = 9,500 Cps
Raised T to 200°C over next hour to drop viscosity to 5000 cps.	Raised T to 180°C, dropped easily to 5000 cps.
Could not do drop test	Drop Test-Sample never set; sticky tacky gel remained after 1 week. Drop test sample destroyed
Sample is very flexible Very tough Similar to 85/15 (3400) although this sample is less tacky and less moldable	No press out - too tacky
	Characteristics - See drop test Much easier to blend because of liquid glycol.

Table 44. Melt and Film Properties of Various Eastman WD/ 3400 PEG Blends

90/10 Blend Sample No. 418-68-A	85/15 Blend Sample No: 418-73-A	75/25 Blend Sample No: 418-67-A
Viscosity was very high = held T @ 170°C rather than standard 160°C initial = 420,000 Cps 1 hour = 303,000 Cps No Drop Test No Press Out Characteristics Same color as WD alone Very tough and elastic Very good elasticity	Viscosity initial = 640,000 Cps 1 hour = 156,00 Cps Raised T to drop further Temperature steadily increased over next 70 minutes while viscosity steadily dropped to 5000 Cps Drop Test 100 adhered 0 removed Press Out Very good adherence to foil Very good flexibility One small piece removed was very flexible and saran-like Very good elastic recovery Characteristics Very pliable, deforms readily around any object (like an ink pen) Surface feels plastic-slightly tacky like saran Fibrous	Viscosity dropped steadily over 1 hour Raised T to 180°C; dropped to 5000 Cps Drop Test 99 adhered 1 removed Characteristics Sample readily formed; elastic, flexible fibers rather than drops. Sample turned white upon cooling Press Out Attempt Formed very good adhesive between Teflon sheets When pulled apart observed perfect adhesive strands between sheets

Table 45. Melt and Film Properties of Various Polyester/PEG Blends

Eastman WD/3400 PEG/Adipic Acid 80/10/10 Blend Sample No: 418-75-A	Eastman 252/3400 PEG 75/25 Blend Sample No: 418-69-A
Viscosity-dropped very quickly in less than 1 hour (25 minutes) initial = 42,000 Cps 25 minutes = 5000 Cps	Viscosity-dropped readily in 20 minutes @ 140°C initial = 22,000 Cps 20 minutes = 540 Cps
Drop Test 100 adhered 0 removed	No Drop Test - 2 components separated on cooling on pipette dropper
Press Out 110°C, 10 pressure, 1 minute; could not remove from foil - seems very flexible	Press Out 140°C, 10 pressure, 1 minute; adhered well to aluminum foil. Small film sample torn away, definitely showed 2 component film
Characteristics Set up quickly - much faster than WD/glycol alone. Turned vanilla white upon cooling Good flexibility, very elastic and very tough. Surface is smooth-almost silky feel. Seems much better than any previous WD blend. Will attempt to grind.	Characteristics Sample remained cloudy while mixing, unlike WD/glycols which became clear. Possibly suspended 252 in glycol? Final sample was less elastic than WD samples. When small strands were drawn, glycol popped out, crystallized readily. White color when cool, slightly tacky/waxy feel.

with good adhesion on the drop tests and to Teflon (the films could not be removed, Table 44). Adipic acid inclusion to the Eastman WD/3400 PEG blend (80/10/10) also produced a flexible film with good adhesive properties (Table 45). However, the Eastman 252 binder was not melt-compatible with the 3400 PEG, giving relatively poor film properties when compared to the Eastman WD/adipic acid blend. The tri-component blend (Table 45) did not cryogenically grind to a free-flowing powder on the Bantam equipment. No advantages were seen with the blends.

From earlier SOS slashing line runs, the powder coated yarn was not fully fusing at a wipe roll setting of 230°C, i.e., a continuous film with good interfiber/hair adhesion was not achieved. The heat transfer from the internal aluminum core through the teflon outer layer of the composite wipe roll was apparently not an efficient process. The melting oven was therefore set at 210°C, and surface temperatures of the dual wipe rolls were determined at thermocouple set points of 225-250-275-300°C. Fig. 52 shows the two points that the surface temperatures were measured at the various settings.

Table 46 details the surface temperatures achieved at different set points, while Fig. 53 shows the linear relationship between contact temperature and set temperature for the two measurement points. A block set temperature of 300°C was an upper boundary, as poly (tetrafluoroethylene), or Teflon, has a melting temperature of 327°C, and a useful mechanical property limit of 260°C. Based on the data and the size formulation melting point, set points in the 250-275°C range for the two wipe roll systems appeared justified.

Fig. 52. Wipe Roll Surface Temperature Measurement Points

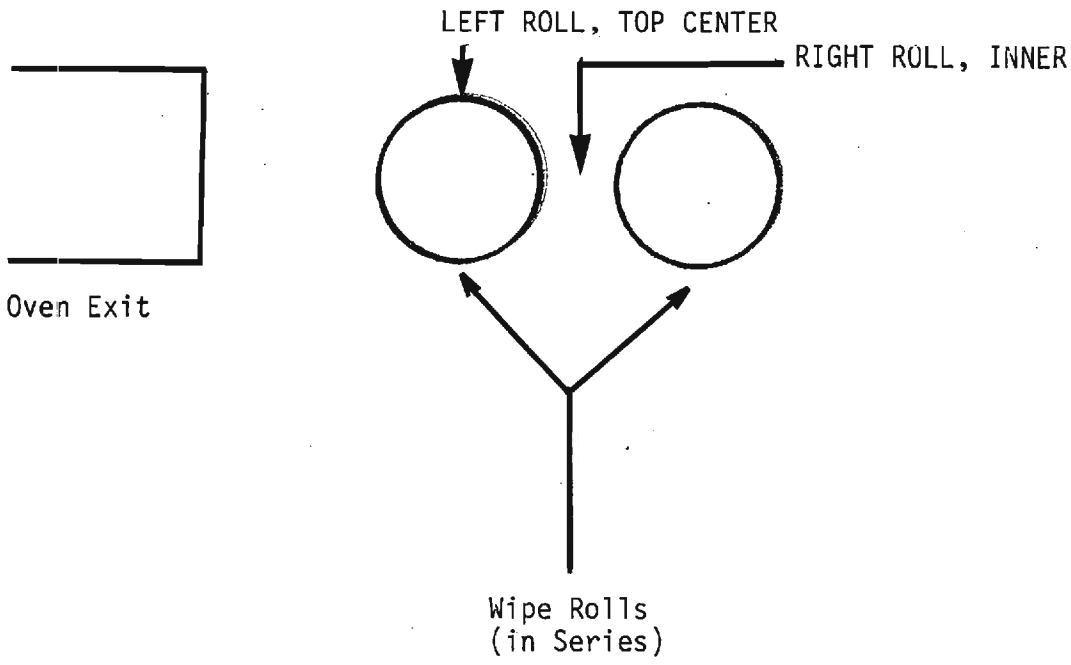
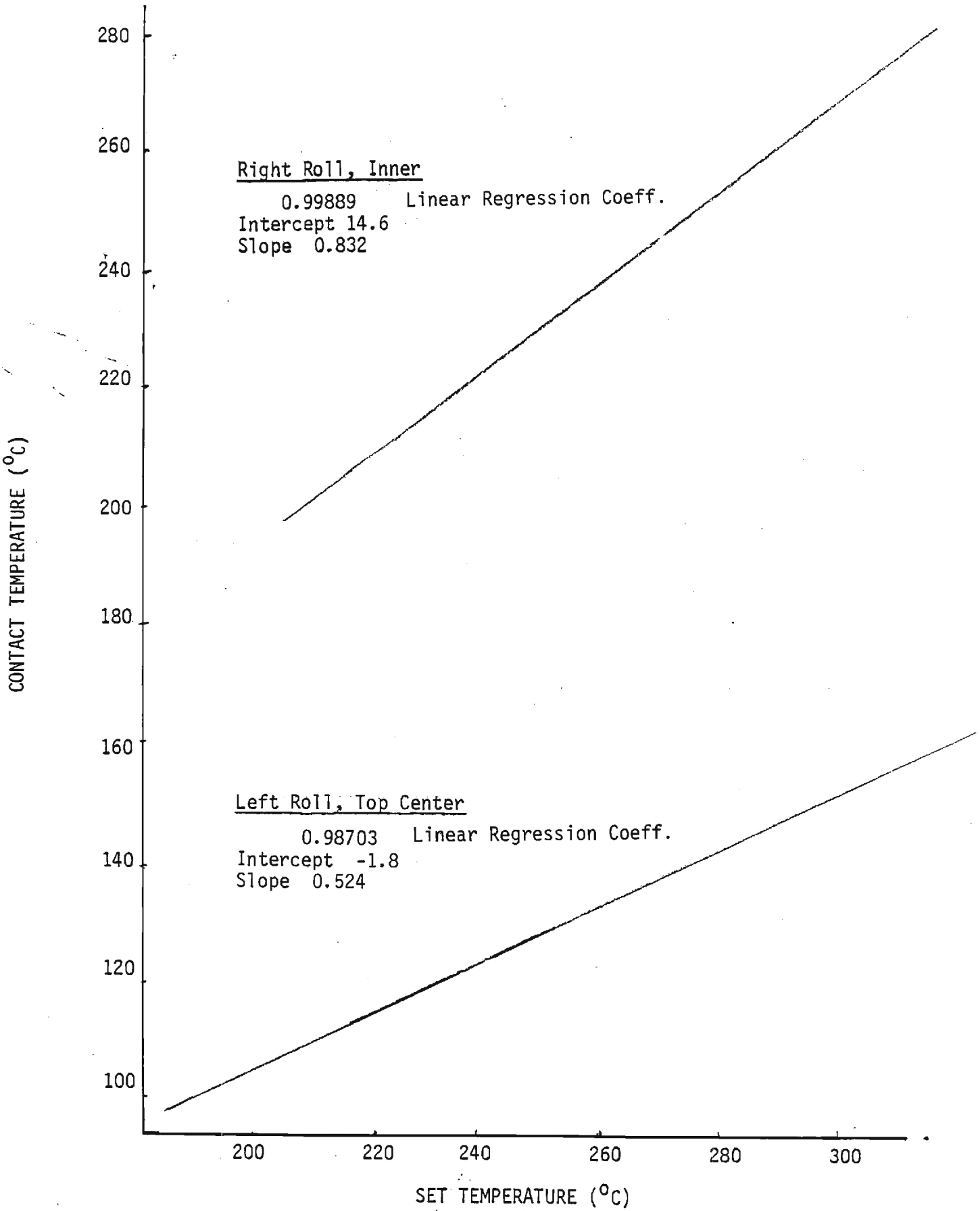


Table 46. Surface Temperatures of Two Wipe Rolls at Fixed Measurement Points

Block Set	Left Top Center	Inner Right Roll
225°C	118°C	203°C
250°C	128°C	221°C
275°C	139°C	243°C
300°C	158°C	265°C

Fig. 53. Relationship Between Contact Temperature and and Set Temperature on the Dual Wipe Roll System



A fresh batch of 60/40 Eastman WD/adipic acid was melt blended and ground in succession with the Wiley mill (twice), Bantam cryogenic grinder (twice) and an air jet mill (once). The latter device impinges the particles under extremely high air flows, fracturing the particles as they collide. Sieving yielded the following fractions:

Retained on 60 mesh	(>250 μ):	11.6gm	(0.7%)
Retained on 120 mesh	(125-250μ):	61.6 gm	(3.5%)
Retained on 170 mesh	(88-125μ):	82.4 gm	(4.8%)
Retained on 200 mesh	(75-88 μ):	61.1 gm	(3.5%)
Passed thru 200 mesh	(<75 μ):	1512.3 gm	(87.5%)

The sieved powder passing through the 200 mesh screen was very fine, showing a tendency to clump, but broke apart easily.

Two runs on the SOS slashing line were conducted with the 0-75 μ fraction of powder. The bed did not fluidize in a uniform fashion (tended to "geyser" and channel). Set conditions for the two runs were:

Vibrator:	On
Oven T:	410 °F (210°C)
Yarn Height:	3 in. above base
Plate Voltage:	32.5 kV
Line Speed:	2.2 ypm

The variable in the two runs was the temperature of the two wipe rolls:

	Roll 1 Set Point (°C)	Temp. Contact	Roll 2 Set Point (°C)	Temp. Contact	NB Code
Run A	273	142	273	244	418-79-A-80-B
Run B	260	135	260	231	418-79-A-81-B

The surface temperatures were measured at the points shown earlier in Fig 52. Average SPU's for the two yarns (50 yd, conditioned weight) were 11.6% for Run A, and 7.9% for Run B.

The yarn conducted under Run A conditions discolored

somewhat, but the size appeared uniformily applied. No discoloration was noted in Run B, but the size did not appear as uniformily applied as with the higher set temperatures. The size also was not well adhered to the fibers in Run B.

Tabulated Toray Fray Counter data for the yarns are shown in Tables 47 and 48. The Run A yarns exhibited a higher fray count in the $\leq$ 0.500 mm length range, but a lower count in the > 0.625 mm range. The peak count for Run A yarns was at 0.375 mm (2814 frays/yd.), compared to 0.500 mm for Run B yarns (2297 frays/ yd.).

Ruti Webtester results for the two runs are detailed in Tables 49 and 50. The Run A yarns were superior to the Run B yarns in abrasion resistance (10,225 cycles-to-break average vs. only 4,450 cycles), but were still considerably below the ~16,000 cycles observed for plant-slashed, ring-spun yarns. The SPU's were lower than for the plant-sized yarn (11.6% (A) and 7.9% (B) vs. 14-17% for the plant yarns) which accounted for a portion of the Webtester differences, as well as A-B differences.

SEM scans revealed little visual difference between yarn samples from Runs A and B. The cotton fibers appeared to have collected more powder than the polyester fibers. Although some evidence of size flow existed, a larger fraction of the size did not show good flow. An image analyzer microscope was also used to peruse the fibers but with less success, as the colorless size did not highlight well under either transmitted or reflected light.

Additional optimization runs were conducted with the 60/40 Eastman WD-adipic acid blend under the following line conditions:

Table 47. Toray Fray Counter Data for Run A Yarns: 273°C Wipe Roll Settings

Toray Conditions:
Test Period - 60 sec.
Running Rate - 8 ypm.

Fray Length (mm)	No. of Frays (#)	Aver. (#)	No. of Frays/ Yd.
0.125	11407		1426
0.250	19324		
	18094	18709	2339
0.375	22512		2814
0.500	19861		
	21234	20548	2568
0.625	10345		1293
0.750	2825		353
0.875	1299		162
1.000	579		72
1.125	508		63.5
1.250	332		41.5
1.500	173		21.6
1.750	123		15.38
2.000	73		9.13
3.000	6		0.75

Table 48. Toray Fray Counter Data for Run B Yarns: 260°C Wipe Roll Settings

Toray Conditions:
Test Period - 60 sec.
Running Rate - 8 ypm.

Fray Length (mm)	No. of Frays (#)	Aver. (#)	No. of Frays/ Yd.
0.125	10397		1300
0.250	8684		
	8124	8404	1051
0.375	13557		1695
0.500	19545		
	17201	18873	2297
0.625	18205		2276
0.750	9781		
	6105	7943	993
1.000	877		110
1.250	497		62
1.500	244		31
1.750	173		21.6
2.000	82		10.25
3.000	15		1.88

Table 49. Ruti Webtester Results on Yarns from Run A: 273°C Wipe Roll Setting

Conditions:

Pivot setting:	3.0 mm
Cyclical elongation:	0.5 %
Weight tensioning:	10 gm
Cycles/minute:	400
Yarn tensioning:	7.8 N

Break No.	STEP Run 1	Run 2	Run 3	Run 4	Run 5	
1	2214	9195	6278	5986	7127	
2	9507	10133	6293	5994	8112	
3	9512	10298	6642	6529	8169	
4	9591	10648	8108	7869	8390	
5	10217	10738	10601	8047	9029	Break 6
6	11389	10874	14368	8895	9245	Aver:
7	12207	11057	15224	9610	9953	10954[a]
8	12349	11189	17462	9914	12265	
9	12380	11356	17778	10756	12271	
10	12412	11400	18384	10809	12455	
Av.	10178	10689	12114	8441	9702	Overall Aver:
Hair Clump	700	6850	2632	3760	4060	10225[a]
Quasi Break	7300	10150	6230	4440	8400	

[a] First three breaks of Runs 3 and 4 were consecutive yarns in the package; assume uniformity problem existed.

Table 50. Ruti Webtester Results on Yarns from Run B: 260°C Wipe Roll Setting

Conditions:

Pivot setting:	3.0 mm
Cyclical elongation:	0.5 %
Weight tensioning:	10 gm
Cycles/minute:	400
Yarn tensioning:	7.8 N

Break No.	STEP Run 1	Run 2	Run 3	Run 4	
1	3647	2593	3571	2987	
2	4906	2883	3657	3827	
3	5138	3242	3929	4141	
4	5217	3331	4016	4683	Break 6
5	5667	3729	4108	4991	Aver:
6	5722	3746	4155	5077	4675
7	5744	3749	4236	5244	
8	5816	4076	4245	5288	
9	5874	4555	4250	5366	
10	5968	4898	4263	5424	Overall Aver:
Av.	5374	3680	4043	4703	4450
Hair Clump	450	275	325	550	
Quasi Break	4630	3140	3150	2920	

Run C:

Vibrator:	ON
Oven T:	410°F (210°C)
Wipe Block 1 Contact T:	140°C
Wipe Block 2 Contact T:	240°C
Yarn Height:	3 in. above base
Plate Voltage:	37.5 kV
Line Speed:	2.2 ypm
NB Code:	418-79-A-86-C

Run D: As above, except:

Oven T:	400°F (205°C)
Wipe Block 1 Contact T:	136°C
Wipe Block 2 Contact T:	238°C
Plate Voltage:	40 kV
Line Speed:	1.6 ypm
NB Code:	418-79-A-89-D

Average SPU's were measured for (conditioned) yarns from Runs C and D at 10.7 and 14.6%, repectively.

Fray Counter data for Runs C and D are plotted in Figures 54 and 55. Run D conditions gave a "hairier" yarn than did Run C conditions on the plant slashing process. Run C yarns were less "hairy" than the conventionally-sized yarn in the $\leq$ 0.500 mm fray length range, and exhibited comparable hairiness in the >0.500 mm range.

Ruti Webtester data for Runs C and D are listed in Tables 51 and 52. The Run D yarns gave a ~ 2X higher cycles-to-break average than the Run C yarns, reflecting their higher SPU (14.6% D vs. 10.7% C).

Based on the data, Run D was repeated with the following line changes:

Run E:

Wipe Block 1 Contact T:	151°C
Wipe Block 2 Contact T:	232°C
Plate Voltage:	41.3 kV
NB Code:	418-79-A-93

SPU of conditioned yarn was found to be 13% (average). Two yarn breaks occurred during the run, both at the wipe block section of

Fig. 54. Toray Fray Counter Data Plot for Optimization Run C Yarns

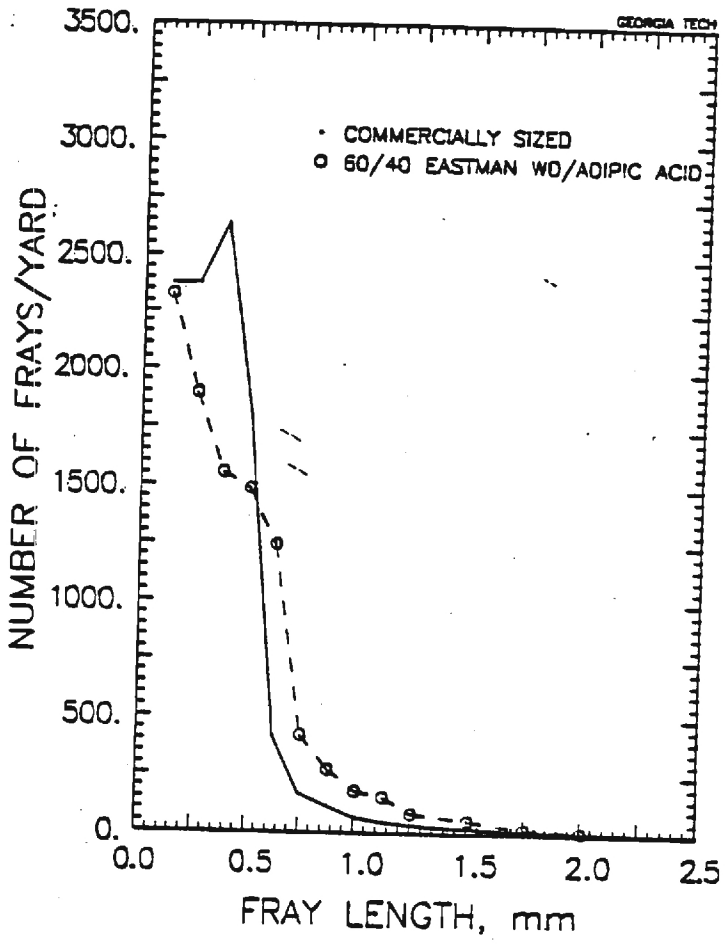


Fig. 55. Toray Fray Counter Data Plot for Optimization Run D Yarns

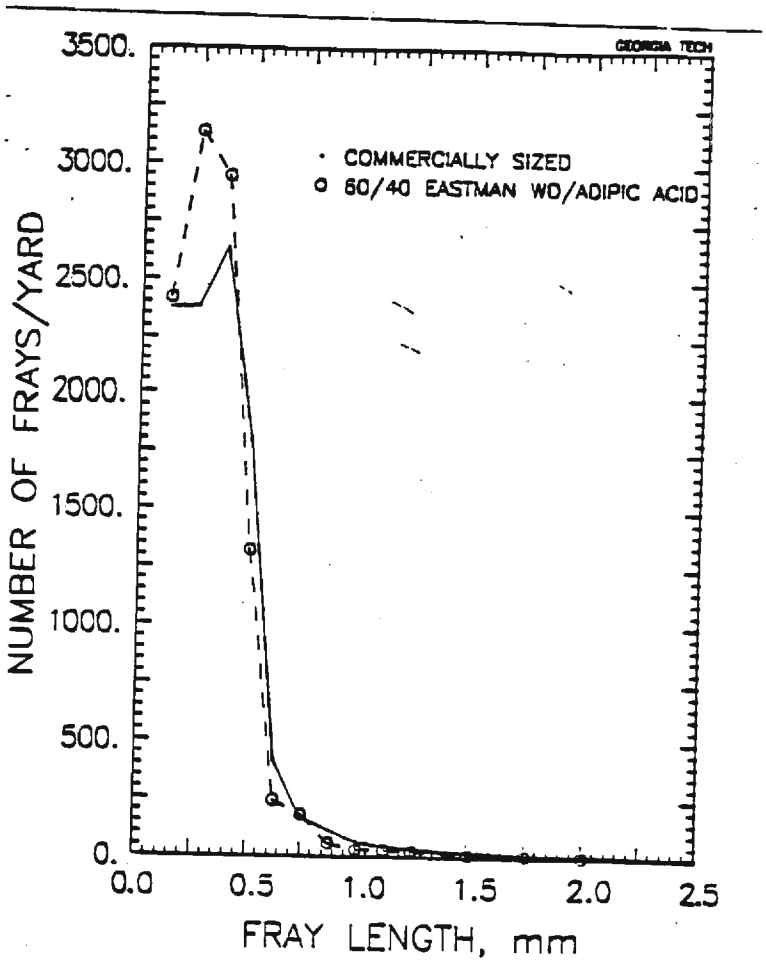


Table 51. Ruti Webtester Data for Yarns Generated in Optimization Run C

Conditions:

Pivot setting:	3.0 mm
Cyclical elongation:	0.5 %
Weight tensioning:	10 gm
Cycles/minute:	400
Yarn tensioning:	7.8 N

Break No.	STEP Run 1	Run 2	Run 3	Run 4	Run 5	
1	4341	2910	6254	3722	3920	
2	4437	3584	6702	3886	4169	
3	4465	4131	7105	4609	4292	
4	4800	5760	7109	5609	4303	Break 6
5	5538	9718	7117	5731	4369	Aver:
6	5672	10107	7131	7481	4380	6954
7	10293	7141	7499	5985	5538	
8	5787	10815	7411	7546	6138	
9	5789	10929	5716	7680	6170	
10	5801	11181	7860	9305	6214	Overall Aver:
Av.	5238	7943	7339	6307	4995	6324
Hair Clump	170	2600	3780	675	385	
Quasi Break	3870	2860	3720	3966	4200	

Table 52. Ruti Webtester Data for Yarns Generated in Optimization Run D

Conditions:

Pivot setting:	3.0 mm
Cyclical elongation:	0.5 %
Weight tensioning:	10 gm
Cycles/minute:	400
Yarn tensioning:	7.8 N

Break No.	STEP Run 1	Run 2	Run 3	Run 4	Run 5	
1	2067	2190	3467	9468	5803	
2	5466	6617	3975	9829	5972	
3	5709	10604	5066	13747	8526	
4	6090	11117	5141	14767	9941	Break 6
5	7630	11534	5889	14772	13136	Aver:
6	7713	12466	9685	14784	14432	11816
7	7810	12504	9794	14843	14796	
8	10422	12596	10146	15241	14847	
9	10431	18328	10250	15665	20340	
10	25760	18484	10288	16321	23833	Overall Aver:
Av.	8910	11644	7370	13944	13163	1106
Hair Clump	270		725	1401	270	
Quasi Break		18015	5111	14670	14200	

the line.

The Toray Fray Counter plot for Optimization Run E is exhibited in Fig. 56. Although the plot matches the commercially-sized material at $\geq$ 0.5 mm fray length, a high count of 3613 frays/yd was recorded at the 0.250 mm fray length (off-scale on the ordinate of the plot).

Ruti Webtester data for Run E yarns revealed a Break 6 average of 10834 and the overall average of 10064 cycles-to-break, both close to those of Run D and indicating the repeatability of the process. The resulting yarn properties reflected a small difference in SPU and wipe roll temperatures:

Run	SPU (%)	1 (°C)	2 (°C)	Aver. Cycles to Break (#)
D	14.6	136	238	11006
E	13.0	151	232	10064

On the ring spun yarn, SPU level probably had the greatest influence on Ruti Webtester performance.

Several conclusions were drawn at this stage of the research:

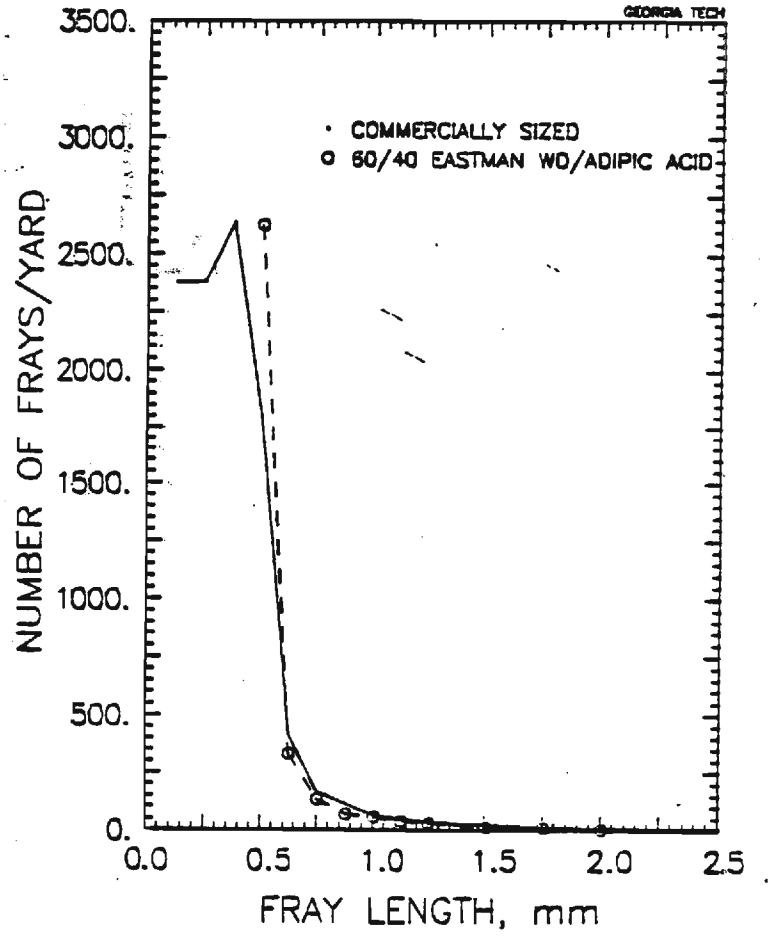
1. Peak Ruti Webtester properties had been reached with the 60/40 Eastman WD-adipic acid formulation.

2. "Blocking" of the formulation had been observed.

Further research was then undertaken to modify the basic 60/40 formulation with third components to improve film strength, specifically stearic acid and 8000 MW PEG.

Various blends with stearic acid were first formulated in the thermal cell of a Brookfield viscometer at 160°C, and the viscosity monitored at 5 minute intervals until a level of ~1000

Fig. 56. Toray Fray Counter Data Plot for Optimization Run E Yarn



Cps was reached. Data is contained in Table 53. Increasing the Eastman WD content at a low stearic acid introduction led to a more fibrous material that was stiffer. Figures 57-60 exhibit the relationship of the melt viscosity vs. time for four blended formulations. Tables 54-57 relay the mechanical properties of the four formulations tested. A major change in the film mechanical properties occurred at the 70/28/2 blend level, where the % elongation-to-break increased by ~ 6X over the other formulations. All of the samples turned from cloudy to opaque on stressing on the Instron, i.e., they crystallized in the draw region.

Similar experiments were conducted with Eastman WD/adipic acid/8000 MW-polyethylene glycol blends. Data is contained in Table 58, and viscosity vs. time plots in Figs. 61-63 for the blends exhibiting melt homogeneity. Film properties, however, were not promising (see comments, Table 58), and the research effort returned once again to the 60/40 Eastman WD/adipic acid formulation.

Using the following conditions and the modified line procedures, Optimization Run F was conducted with the blended size:

Size blend:	60/40 Eastman WD/adipic acid
Vibrator:	On
Oven T:	400°F (205°C)
Wipe Blocks:	294° (1)/127°C
	240°C (2)/235°C
Yarn Height:	3 in. above base
Plate Voltage:	32.5 kV
Line Speed:	1.6 ypm
Amps:	2.5 μ A

The average measured SPU was 14.2%. Toray Fray Counter data on the generated yarn is detailed in Table 59, and the Ruti

Table 53. Results of Blending Various Weight Proportions of Eastman WD/Adipic Acid/Stearic Acid

NB Sample	Blend[a] Ratio	Time Until η = 1000cps	Drop Test (% adhesion)	Comment
439-2-A	54/36/10	40 min.	65	properties destroyed little or no elasticity poor adhesion
439-3-A	60/38/2	90 min.	100	better flex and film properties than 439-2-A v. similar to std. 60/40 les blocking observed
439-5-A	65/33/2	130 min.	99	better film properties than 439-3-A somewhat fibrous
439-7-A	70/29/2	160 min.	99	very fibrous, most fibers were removed during drop test, less elasticity than PEG samples

[a] Ratios given as: Eastman WD/Adipic Acid/Stearic Acid

Fig. 57. Melt Viscosity/Time Relationship for a 54/35/10 Blend of Eastman WD/Adipic Acid/Stearic Acid (160°C)

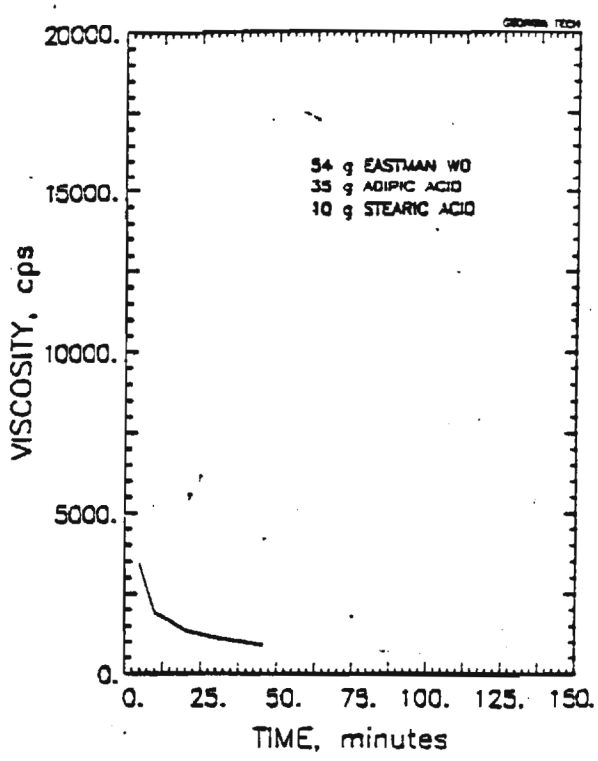


Fig. 58. Melt Viscosity/Time Relationship for a 60/38/2 Blend of Eastman WD/Adipic Acid/Stearic Acid (160°C)

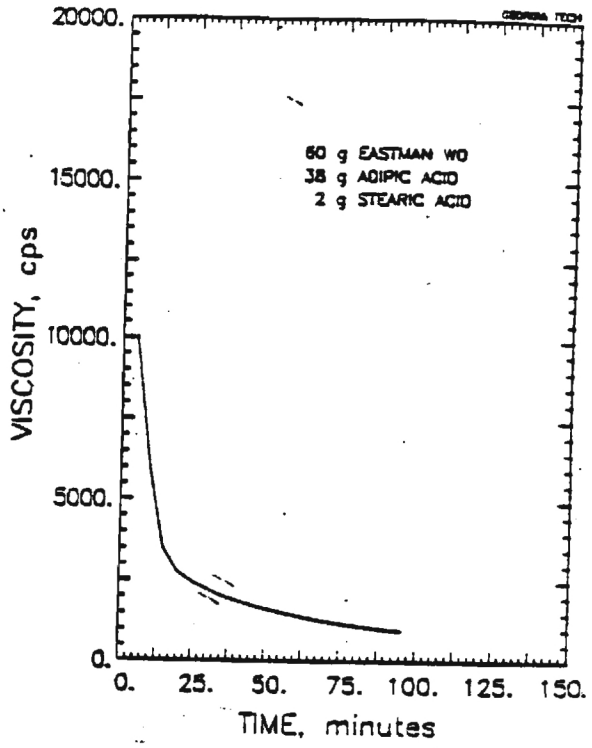


Fig. 59. Melt Viscosity/Time Relationship for a 65/33/2 Blend of Eastman WD/Adipic Acid/Stearic Acid (160°C)

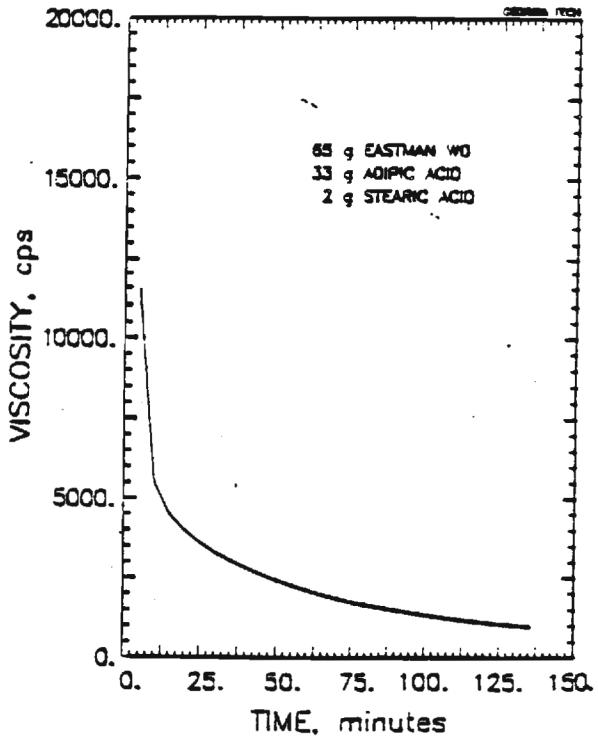


Fig. 60. Melt Viscosity/Time Relationship for a 70/28/2 Blend of Eastman WD/Adipic Acid/Stearic Acid (160°C)

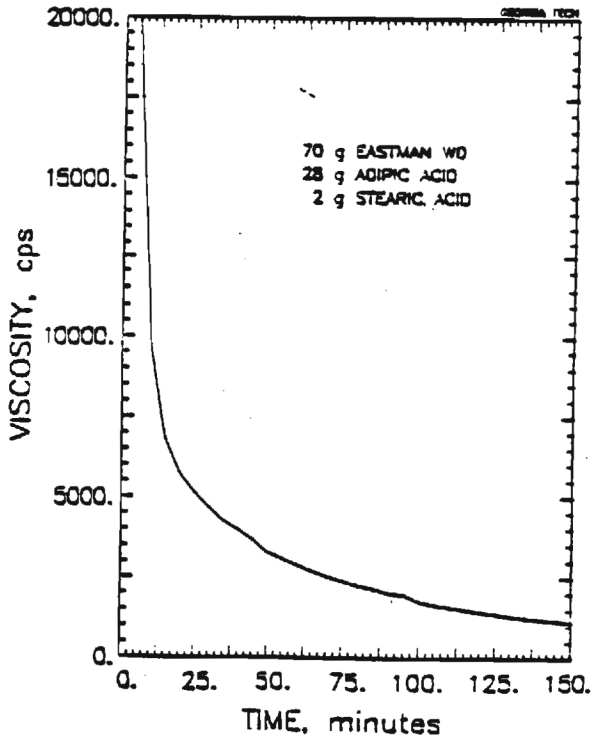


Table 54. Mechanical Properties of Films Cast From 54/36/10 Blend of Eastman WD/Adipic Acid/ Stearic Acid (160°C)

Sample	Load at Break (g)	(lb)	Break Factor (lb/in)	Tensile Strength (lb/in^2)	Elong. Break @ (%)	Stress @ 10% Elong.
B	1310	2.9	1.45	369	61.3	337
C	860	1.9	0.95	242	32.5	112
D	1475	3.3	1.65	420	62.5	376
AVE	1215	2.7	1.35	344	52.1	275

Table 55. Mechanical Properties of Films Cast From 60/38/2 Blend of Eastman WD/Adipic Acid/ Stearic Acid (160°C)

Sample	Load at Break (g)	(lb)	Break Factor (lb/in)	Tensile Strength (psi)	Elong. Break @ (%)	Stress @ 10% Elong. (psi)
A	3357	7.4	3.7	489	53	489
B	3538	7.8	3.9	516	44	516
C	3629	8.0	4.0	529	54	529
D	4309	9.5	4.75	628	46	628
E	2517	5.55	2.78	367	70	367
F	2495	5.5	2.75	364	115	364
AVE	3308	7.3	3.65	482	64	482

Table 56. Mechanical Properties of Films Cast From 65/33/2 Blend of Eastman WD/Adipic Acid/ Stearic Acid (160°C)

Sample	Load at Break (g)	Break (lb)	Break Factor (lb/in)	Tensile Strength (psi)	Elong. Break @ (%)	Stress @ 10% Elong. (psi)
A	1859	4.1	2.05	489	60	477
B	1519	3.35	1.68	400	50	394
C	953	2.1	1.05	251	113	227
D	1451	3.2	1.6	382	71	382
E	1111	2.45	1.23	292	68	292
F	861	1.9	.95	227	94	221
AVE	1291	2.85	1.43	340	76	332

Table 57. Mechanical Properties of Films Cast From 70/28/2 Blend of Eastman WD/Adipic Acid/ Stearic Acid (160°C)

Sample	Load at Break (g)	Break (lb)	Break Factor (lb/in)	Tensile Strength (psi)	Elong. Break @ (%)	Stress @ 10% Elong. (psi)
A	1905	4.2	2.4	334	318	550
B	2404	5.3	2.65	421	421	342
C	2994	6.6	3.3	524	517	362
D	3629	8.0	4.0	636	620	550
E						
F	4037	8.9	4.45	707	302	660
AVE	2994	6.6	3.36	524	436	493

All samples in 439-7-A series crystallized (turned from cloudy to white) while being stressed in Instron.

Table 58. Results of Blending Various Weight Proportions of Eastman WD/Adipic Acid/8000 MW Polyethylene Glycol

NB Sample	Ratio[a]	Time Until η = 1000 Cps	Drop Test	Comment
439-9-A	60/0/40	@ 125 min η = 121000	none	slow to solidify, 2-componet mixture.
439-12-A	70/15/15	@ 180 min η = 3350	100% adhesion	very fibrous, good film former, slow to solidify, good elongation & recovery
439-14-A	60/20/20	@ 160 min η = 1950	99%	good flexibility, low strength, slightly tacky
439-16-A	65/25/10	@ 160 min η = 1508	100%	readily formed drops unlike 439-12-A & 439-14-A very flexible, fibrous, some tackiness, better film properties than 439-14-A

[a] Ratios given as: Eastman WD/Adipic Acid/8000 MW PEG

Fig. 61. Melt Viscosity/Time Relationship for a 70/15/15 Blend of Eastman WD/Adipic Acid/8000 MW PEG

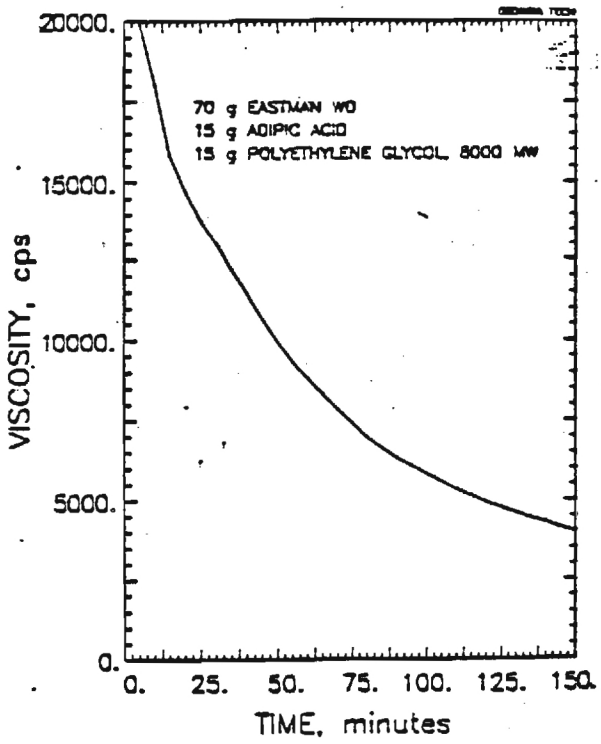


Fig. 62. Melt Viscosity/Time Relationship for a 60/20/20 Blend of Eastman WD/Adipic Acid/8000 MW PEG

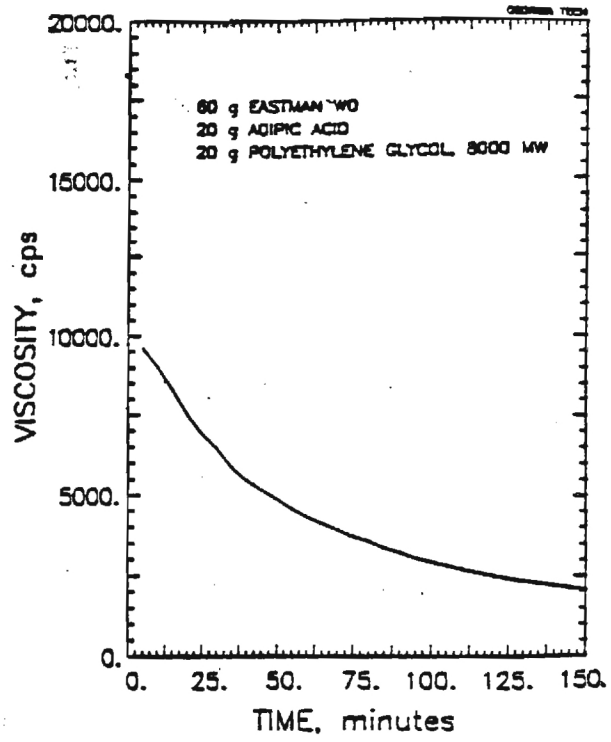


Fig. 63. Melt Viscosity/Time Relationship for a 65/25/10 Blend of Eastman WD/Adipic Acid/8000 MW PEG

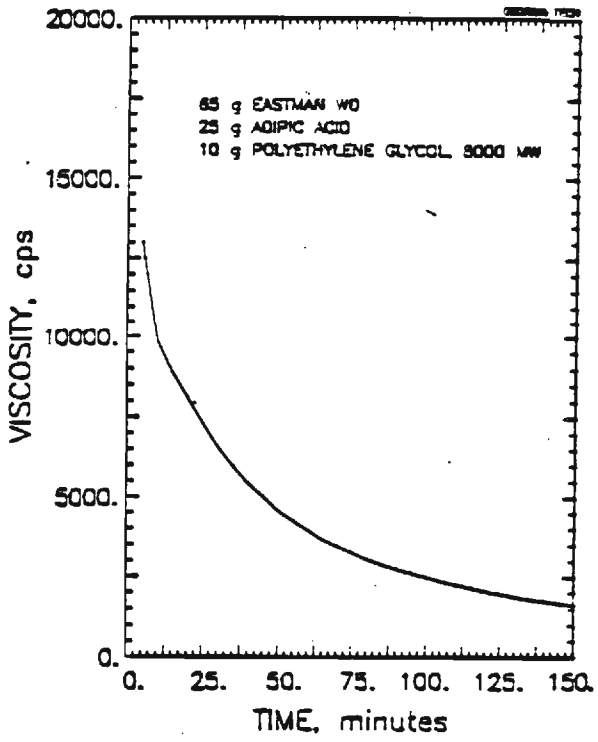


Table 59. Toray Fray Counter Data for Yarn Produced in Optimization Run F.[a]

Fray Length (mm)	No. of Frays (#)	Aver. (#)	No. of Frays/ Yd.
0.125	16790		2099
0.250	12164		1521
0.375	11792		1474
0.500	12262		
	12215	12239	1530
0.625	12646		1581
0.750	13298		1662
0.875	12414		1552
1.000	9224		1153
1.125	5462		683
1.500	873		109
2.000	250		31

[a] Yarn NB Code 418-79-A-96

Webtester data in Table 60.

"Hairiness" of the slashed ring-spun yarn was comparable to the plant-sized standard (2099 frays/yd. max at 0.125 mm). Cycles-to-break averages on the Ruti Webtester were still low (3455 average). The decision was then reached to attempt to translate from a single yarn run through the 5 in. bed to two yarns run simultaneously (coded Dual Run A).

The configuration of the slashing line was basically unchanged from previous runs with the following exceptions:

- Two cones of yarn being unwound and both yarns passing over several tension bars prior to entering the bed.
- A divider placed in the guide for the Leesona so that the yarns could both be traverse-wound on the same package.
- A new bed vibrator was installed.

The line conditions used for the run were as follows:

Oven:	400°F	Voltage:	31.25 kV
Wipe Blocks:	270°/270°C Set	Amps:	2.5 milliamps
Vibrator:	ON	Yarn Height:	3" above bed
Powder:	60/40 WD/adipic acid (ref. NB 41879A), <74 microns		

The run time was approximately 2.3 hrs. for a yield of about 600 yds.

The slashed yarns were split out of the traverse wound package onto a warper and then backwound onto individual packages prior to testing. The 50 yd. pickup weights gave 16.3% SPU for the left yarn and 11.0% SPU for the right yarn. Considerable variation in SPU at different passage points through the fluidized powder cloud was thus observed in initial Dual Run A. Possible causes were:

Table 60. Ruti Webtester Data for Yarn Produced in Optimization Run F

Break No.	STEP Run 1	Run 2	Run 3	Run 4		
1	1487	1840	3649	1980		
2	2072	2403	4117	3147		
3	2093	2499	4374	3277		
4	2662	3075	4514	3322		Break 6
5	3072	3079	4539	3545		Average:
6	3157	3120	4559	3664	---	3625
7	3373	3607	4948	3557		
8	3381	3633	4966	3682		
9	3389	3640	5030	3719		
10	3406	3674	5059	3754		Overall Average:
Av.	2809	3057	4576	3376	---	3455
Hair Clumps	115	1100	120	150		
Quasi Break	2700	3600		2900		

- Uneven cloud distribution in the chamber.
- Different tensions on the two yarns (the degree of tension had been observed earlier to influence SPU).
- Uneven charging of the high-voltage plate.

Since earlier single-yarn runs had yielded the highest properties with the higher SPU's, the left yarn of Dual Run A was tested. Toray Fray Counter data is listed in Table 61, and Ruti Webtester data in Table 62.

The yarn was slightly "hairier" than that from the (single yarn) Optimization Run F (compare Table 61 with Table 59). The overall average cycles-to-break on the Webtester, however, was ~2X that of the earlier yarn, reflecting the differences in SPU (16.3% vs. 14.2%).

The right yarn of lower SPU (11.0%) was then checked. A peak of 2761 frays/yd. was obtained at 0.375 mm fray length on the Toray Fray Counter plot, and the average cycles-to-break for Break 6 and the overall average were 3758 and 3566, respectively, on the Ruti Webtester. The right yarn thus corresponded more closely in properties to the Optimization Run F yarn than did the left yarn (11.0% vs. 14.2% SPU, compare above Webtester results with those of Table 60 and Table 62). A plateau level of abrasion properties vs. SPU appeared to exist in the 11-14% range, which changed to a rapid rise in the $\geq$ 16% SPU range.

Research that remained for Year 3 before entering proof-of-concept, scale-up trials at the participating company site included:

Table 61. Toray Fray Counter Data for Left Yarn of Dual Run A [a]

Frays >mm	# Frays	Average	Frays/Yd.
0.125	16994		2124
0.250	11152		
	13331	12242	1530
0.375	10379		
	13893	12136	1517
0.500	10943		
	9266	10105	1263
0.625	10412		
	9725	10069	1259
0.750	3142		
	2598	2870	359
0.875	1061		133
1.000	696		87
1.125	445		
1.250	346		43
1.375	280		35
1.625	156		20
2.000	69		9
3.000	30		4

[a] Yarn NB Code 439-79-A-27L

Table 62. Ruti Webtester Data for Left Yarn of Dual Run A [a]

Break No.	STEP Run 1	Run 2	Run 3	Run 4		
1	1878	2570	2811	2109		
2	1925	3298	3185	3552		
3	5225	4578	3810	4200		
4	7020	4648	3866	4833		Break 6 Average:
5	8421	6031	4354	4943		
6	8499	6913	4922	5161	---	6374
7	8582	6934	5624	5186		
8	11255	6982	7222	7226		
9	11278	8500	7434	8449		
10	11999	9830	7978	8735		Overall Average:
Av.	7608	6028	5121	5443	---	6050
Hair Clumps	348	520	476	500		
Quasi Break	5100	3650	2750	5045		

[a] Yarn NB Code 439-79-A-27L

1. Optimization of the Eastman WD/adipic acid blend, with 60/40 and 65/35 being the only two remaining size candidates.
2. Improvement of "cross-bed" SPU uniformity.
3. Check-out of a larger (14 in.) bed system to be used in company trials with a 10-end warp sheet.
4. Design/building of feed creel/oven/wipe roll systems for 10-yarn warp studies, with the combined thermal system to also be used in the company trials.

The main concern in preparation for the company trials was attainment of cross-warp SPU uniformity, a major feature of the conventional wet process of slashing.

100% SOLID LIQUID SPRAY COLORATION OF A POLYESTER/COTTON SHEETING FABRIC

After approval by DOE (see Introduction), a graduate student was enlisted to conduct preliminary screening research on resin candidates that fit the 100% solid liquid material criteria:

- low MW oligomers existing in liquid phase at room temperature.
- possession of UV-cureable functional groups.
- production of cured films meeting textile binder properties (see p. 16 list and Hycar 26120 stress/strain plot, Fig 17).

A portable, conveyorized UV curing unit was purchased from Argus International to cure the films (Model PP7106). A visual view of the unit is contained in Fig. 64, and a side-view schematic in Fig. 65. The unit contained two mercury vapor UV

Fig. 64. Visual of Exterior of Argus International Model PP 7106 Conveyorized UV Processor

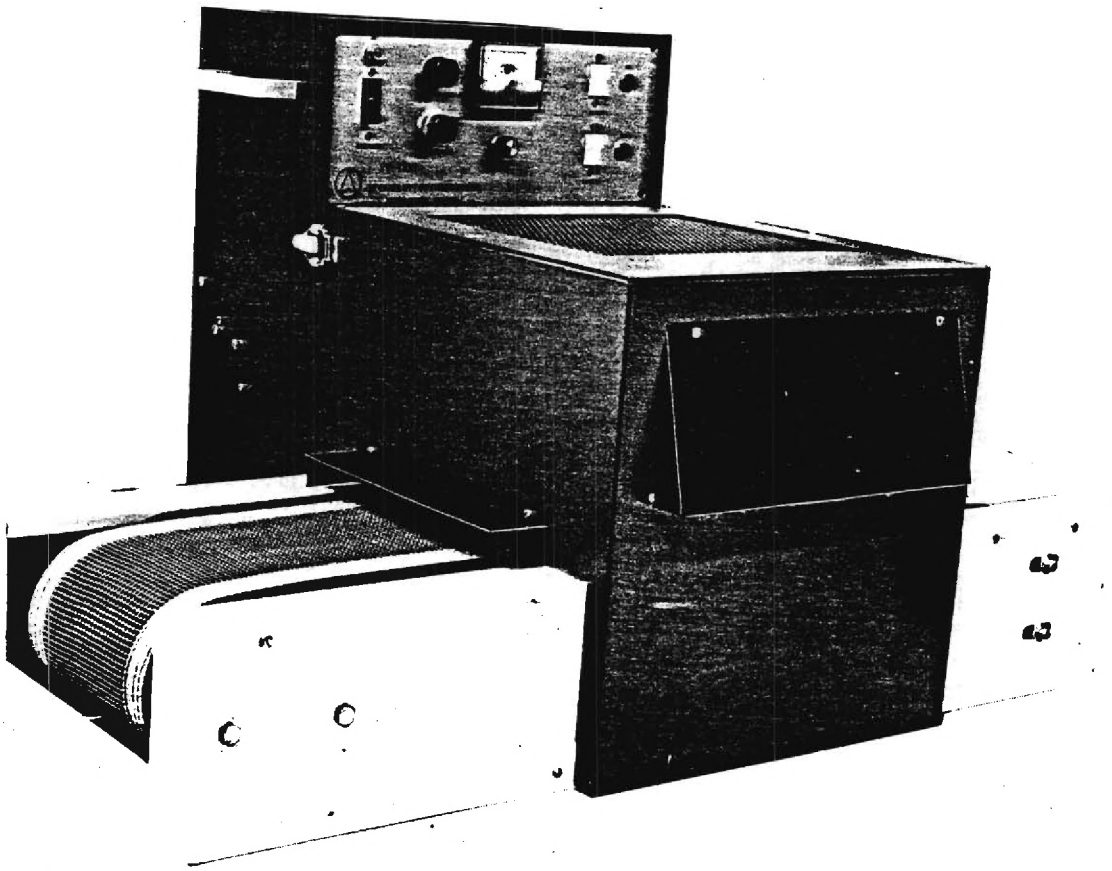
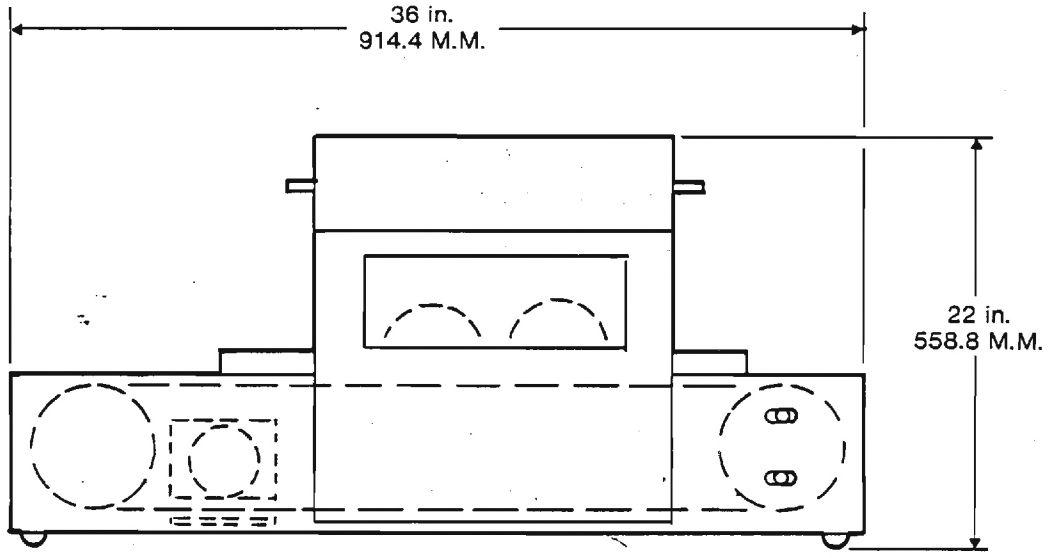
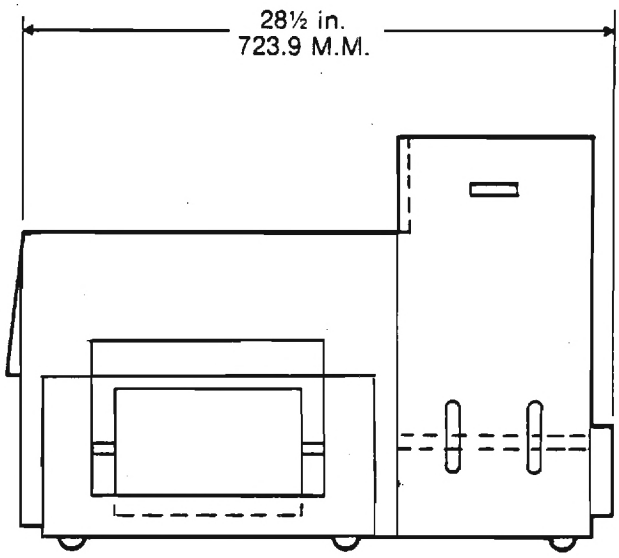


Fig. 65. Schematic and Specifications of Argus International Model PP7106 Conveyorized UV Processor





Specifications:

Lamp Type—200 watt per inch mercury vapor.
Lamps—Two (2)
Max. Curing Width—6" (150mm)
Service—120V Single Phase 50/60 Hz 20A
Venting/Exhaust—650 CFM (17.55 CMPM)
Speed Range—~~1[redacted]~~ 3-30 FPM
Approximate Net Weight—140 lbs. (64 Kg)

lamps of 200 W/in intensity and 180-400 nm spectral wavelength distribution. The reversible teflon-glass conveyor belt offered a speed range of 12-135 ypm. Fig. 66 gives the established relationship between dwell time in the UV zone and the conveyor belt speed of the Argus unit.

Films of resin-candidates were laid at a thickness of 3 mils on glass plates and cured at a set 5 sec dwell time on the Argus unit (both lamps employed). Table 63 reveals the resins utilized, additives employed and visual observations of the films produced.

Several of the more promising films, based on visual observations, were subjected to tensile testing on an Instron. The tensile data is contained in Table 64. For comparison purposes, the tensile properties of Hycar 26120, a complex poly-acrylate used extensively as a pigment binder in conventional wet printing, is also shown in the table (see Fig. 17).

Several of the films showed interesting mechanical correlations to the Hycar standard film, in particular Morton-Thiokol's ZM 1197. Although the elongation-to-break of the latter was ~ 0.4X of the Hycar film, the tensile strength was similar and the critical initial modulus (which largely determines flexibility, and hence aesthetic, properties) was even lower than Hycar.

The viscosities of the materials, however, were too high for the Nordson Liquid Spray System that was projected to be the application mode for fabric coloration. The Nordson system can spray liquids of $\leq$ 600 Cps at room temperature, with higher viscosities requiring heat for viscosity reduction before

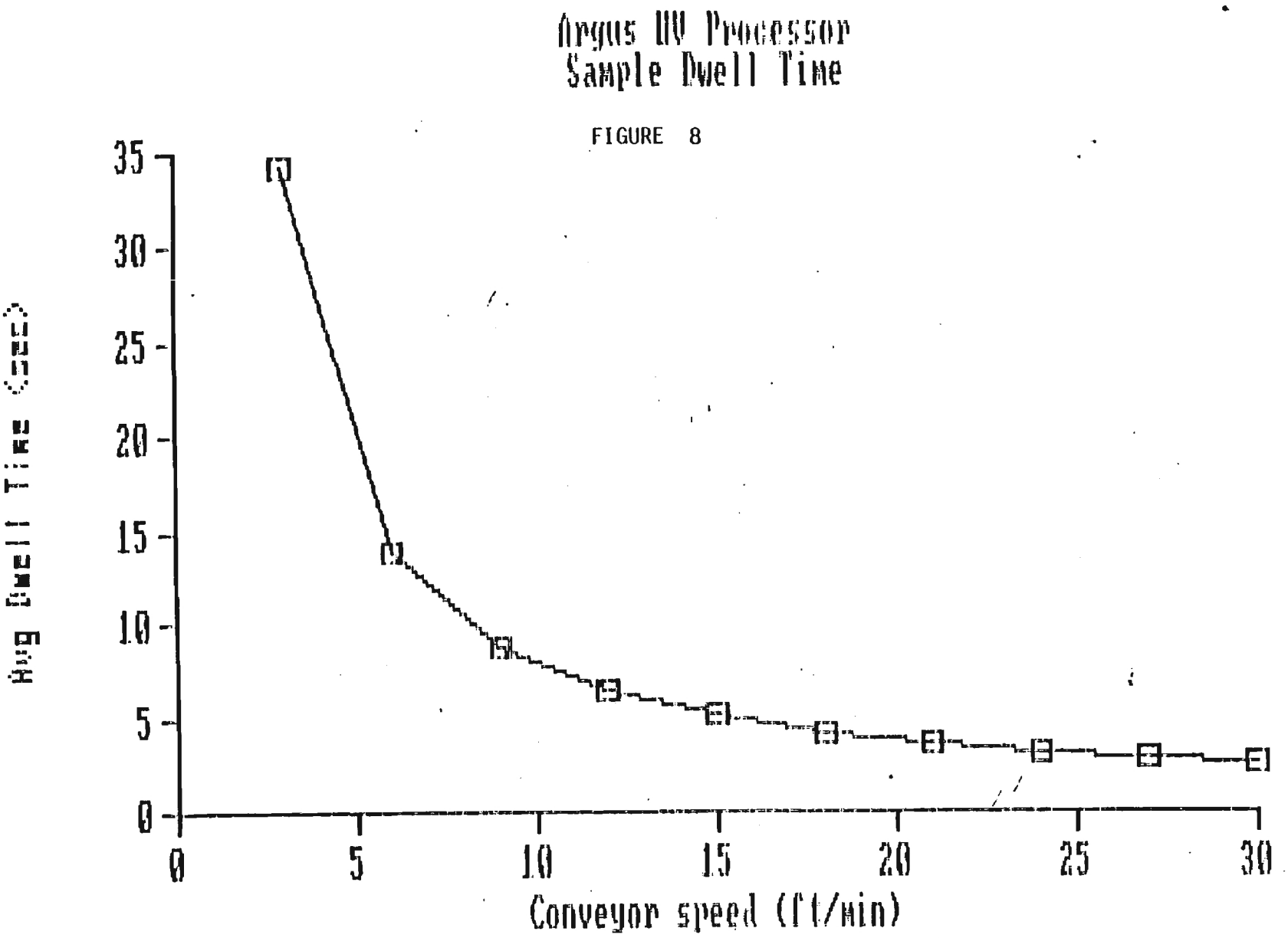


Fig. 66. Relationship Between Conveyor Speed and UV Cure Time in The Argus Unit

Table 63. Film Formation From Resins UV-Cured on the Argus Unit [a]

Resin	Additives	Viscosity(Lit.)	Comments/Observations on Films
Pedigree 140B RO-101 (P.D. George)	1% Irgacure-184 by weight	25,000 Cps @ 25°C	Flexible, stiff like cellophane (designed as optical fiber coating)
Photoglaze B3693-44 (Lord Corp.)	0% (blend of oligomer, monomer and photoinitiator)	1100-1800 Cps @25°C	Flexible, stiff like cellophane adhered to glass plate (may be heated to apply)
Uvithane 782 (Morton-Thiokol)	1% Irgacure-184	solid at room temp., low temp. melt	Soft, very flexible, extensible
ZM-1197 (Morton-Thiokol)	0% (blend of oligomer,monomer, photoinitiator, etc)	11,000 Cps @ 25°C	Adhered to glass, tears easily-possible signs of reaction in storage container (blended Uvithane 782)
Ebecryl 19-6230 (Radcure Spec.)	1% Irgacure 184	30,000-40,000 Cps @ 25°C	soft, flexible, tears easily collapses on itself
Chempot 19-4835 (Radcure Spec.)	1% Irgacure 184	4,000 Cps @ 60°C	stiff, soft slightly adhesive in nature
Chempot 19-4827 (Radcure Spec.)	1% Irgacure 184	3,500 Cps @ 60°C	stiff, soft, somewhat adhesive slightly extensible

[a] Dwell time in the Argus Unit was 5 sec. for all films (15 ft/min conveyor speed)

Table 64. Viscosities and Fiber Mechanical Properties of Selected UV-Cureable Resins

Trade Name (Supplier)	Viscosity	Tensile Strength (psi)	Tensile Elongation (%)	Tensile Modulus (psi)	Characteristics
Chempol 19-4327 (Radcure Specialities)	4500 Cps @ 140F	334	36.7	1173	Light Color Good Flexibility Good Adhesion
Ebecryl 19-6230 (Radcure Specialties)	30,000 Cps @ 25C	120	79	117	Very Light Color Good Adhesion Soft, Flexible
ZM 1197 (Morton Thiokol)	10,000 Cps @ 77F	300	100	50	Good Flexability Wash/Crock Fast
Chempol 19-4835[a] (Radcure Specialties)	5,000 Cps @ 140F	415	45.1	2381	Soft Added Adhesion Properties
Hycar 26120 Aqueous Emulsion (B.F.Goodrich)	75 Cps (50% Solids)	334.3	270.1	141.4	Standard (see Fig. 17)

[a] Oligomer dissolved in 10% tetraethylene glycol diacrylate diluent monomer.

application. In the commercial utilization of the Nordson spray system on metals with solvent-diluted or water-emulsified coatings, heating does not present problems. However, with the UV-cureable resins screened for SOS textile spraying, the highly-reactive functional groups were too sensitive for application of copious heat to reduce the viscosities. The search was thus continued for a lower-viscosity resin in the 100% solid form, i.e., requiring no diluent to reduce the viscosity.

A promising additive candidate, Sartomer's polyethylene glycol 200 dimethacrylate (possessing a RT viscosity in the <600 Cps range), was blended with Lord Corporation's B3693-44 Photoglaze product, a urethane-acrylate copolymer. The Photoglaze, marketed as a flexible vinyl coating, is formulated as a blend of oligomers/monomers/photoinitiators, and has a viscosity of 1100-1800 Cps at 77°F. It reportably can be heated to further reduce the viscosity., but no details on temperature vs. cure kinetics were available. Films were reported by Lord Corp. to be very flexible, chemically stable, and of good mechanical properties(2-3 ksi tensile strength, 40-80% elongation to break at 2 mil thickness). The postulate was that the Sartomer additive would further lower the Photoglaze viscosity to the desired ~600 Cps range, while introducing greater flexibility into the film (see Table 63). The product, marketed by Arco Specialty Chemicals, is claimed to be a "universal" monomeric diluent.

Thin films from the blended materials tended to "bead up" on teflon-coated release paper when drawn, and hence a 10 mil

thickness was necessary to produce a uniform film. An Argus unit conveyor belt speed of 20 ft./min. (3 sec dwell) and both lamps were used for curing.

The film effects of increasing the weight percentage of the Sartomer additive from 0.5 to 10% are detailed in Table 65. The blended formulations drew down well on glass, but the adherence was too great for removal for testing without rupture of the films. From Table 65, a two-pass cure (total of 6 sec exposure) was necessary to harden the blend at higher additive levels. The additive gave softer, more flexible films than obtainable with the unmodified Photoglaze.

Since Year 3 of the SOS project was to be directed toward proof-of-concept trials in the yarn slashing, nonwoven binding/finishing and xerography printing areas, no further research in the 100% solids liquid spray area was planned for that period. The preliminary work, however, revealed several materials that appeared to possess the necessary criteria for textile SOS applications via the Nordson electrostatic spray technology and UV curing, and set the framework for future research.

Table 65. UV-Cured Film Characteristics of Photoglaze B3693-44 Modified by Various Proportions of Sartomer Additive

Additive	Blend Component Weights (gm)	Observations/Comments
0.5%	0.025 Add.[a] 4.975 PG	good/no pronounced difference from unmodified film
1%	0.05 Add. 4.95 PG	softer, greater adhesion to substrate
2%	0.1 Add. 4.90 PG	soft, pliable film
5%	0.25 Add. 4.75 PG	must sit several seconds after irradiation to lose tackiness
10%	0.5 Add. 4.5 PG	ran twice under lamps to fully cure, no appreciable advantage to higher concentration

[a] Add. = Additive, PG = Photoglaze

CONCLUSIONS

RESIN BINDERS FOR XEROGRAPHY TONERS

After screening the film properties of a vast number of resins and blends, virgin Elvax 410, an 82% ethylene/18% vinyl acetate copolymer from E. I. duPont Co., was identified as the primary resin binder candidate for xerography toner development. Although concern remained over the high melt viscosity, the material gave the best choice of desired properties:

- grindability under cryogenic conditions to a free-flowing powder
- film mechanical properties reminiscent of the target material, Hycar 26120 (see Fig. 17)
- film clarity
- textile performance properties

The Elvax 410 was available from duPont as small pellets. Samples of the material were cryogenically ground twice on the Bantam equipment and passed three times through the Trost air mill. After sieving, the material was shipped to Olin-Hunt Chemical for matching with an appropriate carrier bead. Research in Year 3 is to be directed toward printing fabrics first on the Haloid batch xerography equipment, followed by adaptation to a continuous mode on a modified xerox machine (see next section). Demonstration of textile properties with the Elvax 410 resin (wet/dry crockfastness, light fastness, flexibility, etc.) will be a primary objective for the printed fabric produced in Year 3.

XEROGRAPHY PRINTING OF A POLYESTER/COTTON SHEETING FABRIC

Paper xerography developer systems were used to obtain initial fabric prints on the Haloid equipment. Clear, well-defined prints were obtained on 50/50 cotton/polyester sheeting fabric with two paper developer systems: Red Toner No 22-144 (Haloid Xerox, Inc.) and Black Toner Type 10 (Xerox Corp). The resins comprising the toner portion of the developer system, likely a poly (styrene-co-acrylic), failed to meet minimal textile fastness requirements.

The carrier beads from the paper developers were isolated by solvent removal of the toner, and several additional resins and disperse dyes were screened for triboelectric series compatability with the carrier beads (see Table 8). None of the screened materials were compatible, stressing the need for carrier bead development for textile-relevant resins.

In preparation for Year 3 research, four candidate resin materials were ground and sent to Olin-Hunt Chemical for carrier development:

- Pigmented Elvax 410
- Pigmented MU-760, a higher-performance analog to Elvax (U.S. Industries)
- Blue disperse dye cake
- Blue disperse dye cake with lignin sulfonate diluent

In the interim, a Xerox Model 3100 copier was defined as the machine of choice for continuous fabric xerography development (Fig. 16). Conversion of the copier (feed system, heater, electronics, etc.) was begun in preparation for Year 3.

CHEMICAL BINDING OF A POLYESTER NONWOVEN

Two resin candidates were identified as suitable for SOS binding of nonwoven: Eastman's FA-252 modified polyester and H.B. Fuller's IF-3237 modified epoxy. Both resins imparted machine direction mechanical properties to a polyester nonwoven that were comparable to those of the plant-processed standard. The binding actions of the two resins were quite different, with the FA-252 "spot-welding" the fabric while the IF-3237 exhibited good melt flow and concentration at the fiber junction points. The Nordson Spray Booth System performed well with both resins, giving uniform coverage of the fabric and consistent spray patterns. A particle size distribution of 0-75μ was optimized for the two resins.

Cross-machine direction properties of the SOS-bound fabrics were ~2X those of the plant-bound standard for both resins. Pilling test results were also comparable, and the SOS-bound fabrics were softer (less stiff) than the standard.

A third resin candidate, H.B. Fuller's PS-1 polystyrene-based product, was also evaluated. The resin particles formed poorly-adhered beads when melted on the surface of the polyester fiber, with little evident flow. Mechanical properties of PS-1 bound fabric were also poorer than those bound with FA-252 and IF-3237. The PS-1 resin was dropped as an active candidate.

Plans for Year 3 call for proving the concept of powder binding with the two identified resins on 60 in.-wide material. The trials will be conducted on a semi-continuous line at Nordson's Specialty Applications Lab in Amherst, Ohio.

FLUOROPOLYMER FINISHING OF A POLYPROPYLENE NONWOVEN

Several fluoropolymer (FP) candidates were sprayed on the PP nonwoven with the Nordson unit. L-9694, an FP normally used for nylon carpet applications, did not give acceptable barrier properties to the nonwoven when compared to the plant-finished standard (Table 24).

The L-9XX2 fluoropolymer, used in emulsion form with an additive by the participating plant partner, was isolated as a solid. The material was too hydrophilic for use as a powder, however, as the FP "clumped" and aggregated readily when exposed to air. Air mill grinding was inhibited by the inability to feed the material into the mill, and attempts to fluidize the resulting powder failed due to packing and resulting "channeling" behavior of the bed. Further research was planned for Year 3 to circumvent the hygroscopic nature of the material, along with the attendant powder clumping/channeling problems. Proof-of-concept trials on FP finishing were also planned on 60 in.-wide fabric at the Nordson facilities in Ohio.

SLASHING OF A POLYESTER/COTTON STAPLE BLEND YARN

A blended size for SOS slashing, 60/40 Eastman WD/adipic acid, was defined as the primary candidate. An electrostatic fluidized bed application system from Electrostatic Technologies, Inc., proved the best method for applying the size powder to 50/50 cotton/polyester yarns in a continuous mode. A grooved, heated Teflon roll was developed to smooth the melted size film as the yarn exited the oven, as well as to "lay down" the fiber hairs along the axis of the yarn.

Toray Fray Counter and Ruti Webtester data revealed that the SOS-slashed yarn from the optimized process were comparable in degree of hairiness and but not abrasion resistance to plant-slashed yarn standards. Preliminary dual-yarn runs on the EIT system revealed solids loading differences across the width of the bed, as well as some through-package nonuniformity. In preparation for proof-of-concept trials in Year 3, further research on "fine-tuning" the melt blend size proportions as well as improving uniformity on a 10-end warp sheet was planned.

100% SOLID LIQUID SPRAY COLORATION OF A POLYESTER/COTTON SHEETING FABRIC

Preliminary research was conducted to identify promising 100% solid liquid resin candidates for solid shade coloration of fabric. To be accomodated by the projected system, Nordson's Electrostatic Liquid Spray unit, blends of existing materials were investigated that showed promise of a < 600 Cps viscosity at RT. Blends of Lord Corporation's B3693-44 Photoglaze resin (a urethane-acrylate copolymer) with Sartomer's polyethylene glycol 200 dimethacrylate yielded UV-cureable films that had the proper visual and qualitative performance properties. Two passes through the UV-curing unit were necessary to cure the film. The preliminary research thus set the frame work for future research, projected for Year 4 of the effort.

APPENDICES

APPENDIX 1

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Appendix 2

Effect of Solids Pick-up Level on Measured Fabric Thickness

Sample	Solids (%)	Thickness Mils
Blank	0	7.7
Control	7.3-7.5	8.9
D-5	8.1	8.6
I	26.6	9.4

Appendix 3

Test Method For Washing and Pilling Evaluation

Washer: Chicopee uses a Kenmore machine; the School of Textile Engineering has a Whirlpool washing machine. The agitation level is set to HIGH, and the water level on MEDIUM. COLD water is used in this test.

Detergent: 90 mls. of the AATCC Standard Detergent is added to the washer after samples are loaded.

Samples: 10" x 10" samples are cut and labeled with a permanent marker. Up to 25 samples may be tested at the same time. If less than 25 samples are needed, the remainer of the load should be made up of 10" x 10" squares of the control fabric. These control squares are referred to as the "dummy load."

Washing Time: The specification calls for evaluation after the equivalent of 5 home laundry cycles. A cycle is considered to be a 15 minute wash. The test is run by washing for a total of 75 minutes and then allowing the samples to go through the rinse and spin cycles.

Note: Chicopee has modified their machine using a secondary timer to allow the total wash time to be set directly. Our machine is a standard home machine with a maximum wash time of 14 minutes. The machine must be manually reset to the beginning of the wash time, and the total wash time recorded independently.

Drying: The samples should all be removed from the washer and allowed to air dry before evaluation.

Pilling Evaluation: Samples are evaluated by visual comparison to the ASTM D-3512 photographic standards. These standards represent a rating scale of 5 to 1, where 5=no pilling, 4=slight, 3=moderate, 2=severe, and 1=very severe pilling.

Appendix 4

3M Test Kit

for Surface Oil Repellency

Kit Number	Volume Castor Oil* ml.	Volume Toluene* ml.	Volume Heptane* ml.
1	200	0	0
2	180	10	10
3	160	20	20
4	140	30	30
5	120	40	40
6	100	50	50
7	80	60	60
8	60	70	70
9	40	80	80
10	20	90	90
11	0	100	100
12	0	90	110

*All C.P. Grade

Major Value: Q.C. - Type Comparisons

APPENDIX 5

Initial Procedure for Operation of the ETI Single-Yarn Slashing Line

A. DIRECTIONS FOR STARTING UP THE ETI SLASHING LINE

1. Plug the air cleaner (yellow plug) into outlet.
2. Turn ON/OFF Switch for fluidized bed to "ON".
3. Plug vibrator (white cord) into outlet.
4. Put powder in bed.
5. Connect shroud and then tape shroud to bed to seal.
6. Turn air knob counter clockwise to make sure it is OFF.
7. Depress foot pedal (use weight).
8. Slowly turn air knob closkwise until powder fluidizes.
9. Let powder fluidize for about 1 hour.
10. String up yarn through bed and tape to oven exterior.
11. Turn on ovens and cartridge heaters by plugging them in. (try putting all oven plugs into poser strip and then using its switch).
12. After powder has run 1 hour, turn on voltage switch. Adjust voltage to 30 kV. Let run about 20 minutes.
13. String up oven and take-up. Check alignments. Turn on Leesona Winder using its switch, set line speed.
14. Run about 20 yards of yarn. Stop and reel off 10 yards to weigh. Adjust if not 10-14% pick-up.

B. DIRECTIONS FOR STOPPING ETI SLASHING LINE

1. Turn Leesona Winder OFF (can break yarn & let Leesona finish winding it up).

2. Turn off ovens, controller, vibrator and powder collector by unplugging them all.

3. Turn voltage to zero, then turn voltage switch OFF.

4. Turn air to zero, then take weight off of foot pedal.

5. Knock powder off of shroud, remove tape, and then dissassemble.

6. Scoop out powder - get as much out as possible.

7. Vacuum out hose to powder collector, then vacuum everything else.

8. Turn air for bed back on while vacuuming and leave on for a little while to blow out eporous plate.

9. Turn air back off.

10. Turn main power switch OFF.

Appendix 6. Procedure for Melt Blending PEG's with Eastman Polyesters

1. Melt (if applicable) glycol in beaker on hot plate.
2. Add Eastman WD or 252.
3. Stir until visibly blended.
4. Hold temperature at 160°C.
5. Read viscosity at 5 minute intervals for 1 hour or until viscosity reaches 5000 cps (whichever comes first).
6. If viscosity has not reached 5000 cps @ 1 hour, increase temperature until viscosity does reach 5000 Cps.
7. Conduct drop test.
8. Pour out on teflon sheet.

APPENDIX 7 TEXTILE WET PROCESS CONSUMPTION

Process	Energy Consumption Thermal BTU/lb.	Energy Consumption Electrical BTU/lb.	Processed Fiber lbs x 10^6	Energy Consumed BOE
Batch Dyeing				
Atmospheric Beck				
Carpet	13,300	330	605	1,400,000
Fabric	23,500	600	1,005	4,220,000
Hosiery	23,500	600	465	1,907,000
Jig	2,700	670	448	257,000
Pressure Beck	6,400	660	534	641,000
Jet	9,200	880	431	738,000
Package/beam	17,300	1,980	1,018	3,340,000
Stock	12,000	1,426	375	857,000
			Sub Total:	13,360,000
Continuous Processes				
Slashing	2,900	-	3,905	1,900,000
Fabric Dye Range	4,400	150	2,493	1,929,000
Finishing Range	7,800	660	3,207	4,612,000
Carpet Dye Range	12,300	1,300	351	826,000
Printer	9,300	230	1,018	1,656,000
Predryer				
Infra-red	900	-	7,528	1,152,000
Mechanical	-	200	4,895	166,000
Steam Cans	1,600	-	4,124	1,122,000
Dryers	2,100	200	16,471	6,532,000
		Sub Totals:	48,873	19,895,000
	Continuous Idle Time (.2 x 19,895,000)		=	3,979,000
		Totals	48,873	37,234,000