GEORGIA INSTITUTE OF TECHNOLOG	GY	OFFICE OF CONTRACT ADMINISTRATIC	N
PROJ	ECT ADMINISTRA	TION DATA SHEET	
		× ORIGINAL REVISION NO.	
Project No E-27-633		GTRI/SCK DATE 8 / 17 / 84	
Project Director: Dr. F. L. Co	ok	School XXK Textile Engineering	
Sponsor: DOE, Oak Ridge, Te	nn.		_
Type Agreement: Grant No. DE-	FG05-84CE40702		
Award Period: From 8/1/84	To 7/31/8	(Performance) 10/29/86 (Beports)	
Sponsor Amount:	This Change		
Estimated: \$ 225,	000	\$ 542.131	
Eunded: \$ 225	000	\$ 225 000 (Through 7/31/85)	_
Cost Sharing Amount: \$ 9-035	<u></u>	Cost Sharing No: E-27-324	
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ADMINISTRATIVE DATA 1) Sponsor Technical Contact:	OCA Contact	2) Sponsor Admin/Contractual Matters:	
Dr. B. G. Massey		Ms Joyce C Carringer	
Conservation and Renewal		Contract Management Preach	
Freeze Industrial Pressen	CE 12	Brownerst & Contracts Divided on	
Energy Industrial Programs	, CE-12	Procurement & Contracts Division	
U.S. DOE		U. S. DOE	_
Washington, D. C. 20585		P. O. Box E	-
(202) 252-2079		Oak Ridge, Tenn. 37831 (202) 57	<u>6-75</u>
Defense Priority Rating: <u>N/A</u>	(or)	Military Security Classification: <u>N/A</u> Company/Industrial Proprietary: <u>N/A</u>	=
RESTRICTIONS See Attached N/A Travel: Foreign travel must have prior a	Supplemental Inform pproval – Contact OC	mation Sheet for Additional Requirements. A in each case. Domestic travel requires sponsor	
approval where total will exceed	greater of \$500 or 1	25% of approved proposal budget category.	
Equipment: Title vests withGIT;	However, none p	roposed.	
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	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 WITHO	UT USE LIQUIDS:PROOF CONCEPTS POWE
Effective Completion Date 890301 (Performanc	e) 890601 (Reports)
Closeout Actions Required:	Date Y/N Submitted
Final Invoice or Copy of Final Invoice	Ŷ
Final Report of Inventions and/or Subcon	tracts Y
Government Property Inventory & Related	Certificate Y 900315
Classified Material Certificate	N
Actease and Assignment	N
CommentsSF 269, SF 270 AND SF 272 IN FIN FINANCIAL RECORDS.	AL FORM REQUIRED TO CLOSE OUT THE
Subproject Under Main Project No	
Continues Project No	
Distribution Required:	
Project Director	Y
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Report Coordinator (OCA)	Y
GTRC	Y
Project File	Y
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## APPLICATION OF CHEMICALS TO SUBSTRATES WITHOUT THE USE OF LIQUIDS: PROOF OF CONCEPTS FOR POWDER SPRAY GUN AND FLUIDIZED BED SOLID-ON-SOLID (SOS) PROCESSING OF TEXTILES, AND CONTINUED RESEARCH IN TEXTILE XEROGRAPHY PRINTING, SOLID SHADE COLORATION AND ELECTROSTATIC LIQUID SPRAY SOS FINISHING OF FABRICS

Continuation of

DOE Grant No. DE-FG05-84CE40702

Georgia Tech Project No. E-27-633

#### FINAL TECHNICAL REPORT

October 1, 1986-July 1, 1989

Submitted to the

#### DEPARTMENT OF ENERGY

by

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Atlanta, Georgia 30332 September 28, 1990

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#### INTRODUCTION

The first two years of research under DOE Contract No. DE-FG05-84CE40702 were devoted toward developing processes whereby certain chemicals could be applied to textiles without the use of water, mainly concentrating on powder deposition techniques (1). The approach was to identify powder-based processes in other industry sectors (mainly the metals and paper industries) that possessed the potential to be adapted to continuous textile manufacturing lines. The adapted textile processes were classified under the general category of **solid-on-solid (SOS) processes**, since no liquid water was required, and 100% of the chemical materials applied to the substrate remained with it into final product manufacture.

The current research focused on several areas of chemical treatment:

- . Yarn slashing
- . Textile xerography printing
- . Binding of nonwovens
- . Fluoropolymer barrier finishing
- . Liquid spray finishing

Several of these areas were sufficiently developed in the first phase to allow full-scale, proof-of-concept trials to be conducted at industrial sites in the third and fourth years of the project. Other areas were identified and preliminary investigations

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conducted in the first phase, but were largely left for full development in the reported phase, e.g., liquid spray finishing of 100% solids formulations.

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The SOS yarn slashing process was based on an electrostatic fluidized bed system built by Electrostatic Technologies, Inc. (ETI) of Connecticut. Used for years to apply coatings to continuous wires, the fluidized bed system placed a charge on the size powder, which then was attracted to the surfaces of 50/50 polyester/cotton yarns slated for sheeting production that were traversed through the fluidized powder cloud as a warp sheet. Melting of the thermoplastic size to form the desired plastic film around the yarn bundle and individual surface fibers completed the SOS slashing process. In the reported research, the size formulation was optimized, final development runs conducted at Georgia Tech, and the slashing process scaled up to a 60-end warp level in a proof-of-concept trial conducted at the West Point Pepperell Research Center in Shawmut, AL. Weaving trials were conducted with the SOS- and conventional-slashed warps to determine the weaving performance of the trial yarns versus those from plant production.

The textile xerography process, which had been successfully demonstrated in a continuous mode with single-color, beam-to-beam printing of 8.5 in.-wide sheeting fabric (1), was scaled up to a three-foot wide level with the Xerox Model 2510, optical-based copier. A three-applicator, continuous line was developed in which complex prints using green, red and blue toners were produced on the same sheeting fabric (one-, two- and three-color overlap

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prints).

Development research was completed in two areas of powder spraygun deposition to continuous-moving nonwoven textiles: chemical binding of a 100% polyester nonwoven slated for mattress pad covers, and fluoropolymer barrier finishing of a 100% polypropylene nonwoven utilized in hospital operating room environments as surgical gowns, covers and drapes. Built around the Nordson powder spraygun booth system adapted from the metals industry, the SOS process was demonstrated in two successful proofof-concept trials on a 60-in. wide production line at Nordson's Amherst, Ohio Research Laboratories. Line speeds of 200 yards per minute were successfully achieved, producing fabrics of desired properties in both nonwoven processing areas.

The failure to successfully produce quality solid shades on sheeting fabric with the Nordson powder spraygun system (1) led to research in adapting another Nordson development, the electrostatic liquid spraygun process, to the application of pigmented, liquid binder systems to textiles. By using segmental urethane oligomers possessing UV-curable acrylate functionalities, the liquid spraygun process allowed uniform, dark green coloration of the 50/50 polyester/cotton sheeting fabric in a continuous mode.

The incorporation of the 100% solids liquid spraygun technology into the SOS research then allowed expansion of the scope of the project into intrafiber finishing of textile fabrics. In particular, two types of finishes that penetrate the solid state structure of cellulosic fibers and reacts with the hydroxyl functional group of the substrate to produce a reacted, permanent

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finish were investigated: reactive silicone softeners and durable press resins. The softeners, applicable as neat liquids within the viscosity limitations of the Nordson spraygun system, gave excellent fabric performance on the standard polyester/cotton sheeting fabric, and were impervious to washing. The methylated derivative of the standard durable press resin common in the industry, dimethyloldihydroxyethylene urea (DMDHEU), was the material of preference in these studies since MEDMDHEU was a viscous liquid in the pure state versus a glassy state for the parent chemical. A combined finish formulation incorporating both durable press and thermal responsive chemicals was developed to provide a sprayable composition of the proper (<600 cps) viscosity. The SOS-finished fabrics had the combined durable press/thermal responsiveness characteristics of Polytherm<sup>c</sup> materials developed at the Southern Regional Research Center of the USDA from a wet padnip-cure process.

#### EXPERIMENTAL RESULTS AND DISCUSSION

#### I. SLASHING OF POLYESTER/COTTON STAPLE BLEND YARN

In preparation for the upcoming proof-of-concept trials at West Point Pepperell's Research Center, further research was conducted on "fine-tuning" the optimized, melt-blended size formulation (60/40 Eastman WD/adipic acid, with ester interchange allowed to take place in the melt). Henceforth, the size will be referred to as 60/40 WD/AA. Improving cross-warp uniformity on a 10-end sheet was also a priority.

A system for slashing up to 10 yarn ends was designed and constructed from UNISTRUT framing and fiber handling components donated by West Point Foundries (Figure 1). The fluidized bed applicator (five inch square bed size) was produced and supplied by Electrostatic Technologies, Inc., Model 400-A (Figures 2 and 3). The line's creel enabled relatively accurate adjustment of the tension of each fiber. Combs were included to vary the distance between the fibers at various critical points. A section beam was mounted of a Leesona winder for take-up. The earlier tube furnace was replaced with a Lindberg oven, and combs were inserted to control the yarn spacing before and after the fluidized bed and oven.

The grooved Teflon wiping blocks, inserted earlier in the line to smooth out the size film on the yarn surface and avoid excess spots, were replaced by two rolls with deeper grooves and with the grooves of the two units aligned with each other. The changed configuration kept the yarns separated and tracking properly





# FIG. 1

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FIG. 3. SCHEMATIC OF ETI ELECTROSTATIC FLUIDIZED BED

through the Lindberg oven and onto the take-up roll.

After reestablishing temperature profiles for the oven and the new wipe rolls, five yarns were strung and processed through the modified SOS slashing line with the 60/40 WD/AA size formulation. The initial solids pickup (SPU) was 14.3%. By the end of the run, SPU had dropped to 4.7%, indicating the necessity on long runs of having a feed system incorporated in the process to replenish the bed level in a concise manner. Line conditions used for the run were:

Oven:215CVoltage:39kVWipe Blocks:215, 220C Amps:unknownVibrator:onYarn Height:3" above bedPowder:NB 41879A LotParticle Size: >75 microns

Yarns from the run, coded 439-79A-34 lot and in the 12-14% SPU range, were subjected to Ruti Webtester abrasion cycles to assess resistance to breakage. The results, shown in Table 1, gave a wide range of average cycles for all breaks across the warp, with yarn position 5 giving the best results (6663 aver. cycles) and position 2 the poorest (1505). Steady-state application across the fiveyarn warp had obviously not been achieved in the first run.

The run was repeated, with several variable changes from the first attempt:

Voltage: 35kV Line speed: 2.6 ypm

SPU: 13.7-29.5%

The yarn samples, coded 439-79A-36B, gave higher average cycles to break (max. of 8867), but still showed considerable variation both in SPU and average cycles to break across the five-component warp

Air Pressure: <2 scfm

#### TABLE 1.

#### RUTI WEBTESTER RESULTS FROM 5-END RUN SAMPLE 439-79A-34<sup>a</sup>.

			YARN NUMBER		
	<u>1</u>	<u>2</u>	<u>3</u>	4	<u>5</u>
Total Number Tests Run	1	2	2	2	2
Min. Cycles at 1st Break	1003	885	2246	2989	2568
Max. Cycles at 10th Break	2251	2091	5072	5111	10460
Average Cycles 6th Break	1823	1564	3784	4338	7178
Average Cycles All Breaks	1773	1505	3670	4228	6663
Cycles/lst Hair Clump	70	158	55	132	94
Total Clumps	47	48	22	28	13
Cycles/1st Quasi- Break	1000	1358	2660	4090	2858
Total Quasi- Breaks	3	1	2	1	5

<sup>a</sup> The machine settings were: pivot setting = 3mm, cyclical elongation = 0.5%, weight tensioning = 10 g, cycles/min=400, and yarn tensioning = 7.8N. Yarn 1 was run through the slahsing line closest to the wall, and yarn 5 closest to the middle of the room. The % pick-up was 14.3 initially, but drifted down to 4.7% at the end of the run. Yarn with pick-ups ranging from 12-14% were tested here.

(Table 2). To check reproducibility of the Ruti Webtester technique, the yarn positions 1 and 5 were retested. In comparing Tables 2 and 3, the results for Yarn 1 were reasonably close on average cycles to break (3196 vs. 4533), but Yarn 5, at the much SPU (29.2% vs. 13.7%), higher exhibited poor Webtester reproducibility from the first to second tests (7534 vs. 11693 aver. cycles to break). By comparison, the average cycles to break for the commercially-sized standard warp from West Point Pepperell (WPP) with ring spun, 50/50 polyester/cotton construction (PVAbased size) was around 17,000.

During this time period, while negotiating with researchers from WPP and West Point Foundry & Machine Co. on the arrangements for the proof-of-concept trials, the team discovered that the partner plant (Lanier, in Shawmut, Ala.) was in the process of switching spinning technologies from ring to air jet (Murata). Air jet-spun yarn has a quite different internal structure from ringspun yarn, with the polyester/cotton distribution altered, alignment of fibers along the fiber axis inferior with more hooks (fiber entanglements), and generally giving a yarn with lower mechanical and abrasion resistant properties. In short, the switch to air jet technology increases production speeds by '2X, but results in an inferior yarn construction.

Although late in the SOS research and approaching the target time period for scaling up the slashing process at WPP, the team decided to redirect the investigation toward the company's new focus and production, air jet spun staple yarns. Samples of asspun yarns were collected from the Lanier plant (34's cotton count,

## TABLE 2.

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#### RUTI WEBTESTER RESULTS FROM 5-END RUN SAMPLE 439-79A-36B

			YARN NUMBER		
	1	2	3	4	5
50 Yard Weight	0.8834	0.9687	0.9795	1.0066	1.0038
<pre>% Pick-Up</pre>	13.7	24.7	24.9	29.5	29.2
Average Cycles 6th Break	3405	6395	9562	5269	8085
Average Cycles All Breaks	3196	6100	8867	4476	7534
Cycles/lst Hair Clump	64	156	84	47	74
Total Clumps	89	42	29	57	52
Cycles/lst Quasi-Break	800	1570	1505	912	1996
Total Quasi- Breaks	4	7	5	8	11

The machine settings were: pivot setting = 3mm, cyclical elongation = 0.5%, weight tensioning = 10g, cycles/min = 400, and yarn tensioning = 7.8N. Yarn 1 was run through the slashing line closest to the wall, and yarn 5 closest to the middle of the room. The pick-up was 13.8% when checked by weighing a 5 yard hank of 5 ends at the end of the run. The individual pick-up weights were checked on 50 yard lengths taken between the 2nd and 3rd tests. A total of 4 tests were run on each yard.

## TABLE 3.

## RUTI WEBTESTER RESULTS FROM SAMPLE 439-79A-36B REPETITION OF TESTS

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.

		YARN NUMBER
	1	<u>5</u>
50 Yard Weight	0.8834	1.0038
% Pick-Up	13.7	29.2
Average Cycles 6th Break	4589	12026
Average Cycles All Breaks	4533	11693
Cycles/lst Hair Clump	76	156
Total Clumps	19	20
Cycles/lst Quasi-Break	3125	1107
Total Quasi-Breaks	3	4

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50% duPont Type 106 (1.42 denier per filament, dpf)/50% cotton), and standard samples of the same yarn slashed by the PVA plant process were also isolated to provide a new baseline for the research program.

The Ruti Webtester results for the new yarn construction slashed by the plant process, shown in Table 4, gave the first indication that the air jet spun yarn was much more susceptible to abrasion breaks than the earlier ring spun construction. The highest average cycles to break, exhibited by Yarn 4 in the warp, was 4379, matching the poorest performers of previous SOS runs (compare Table 2). The lowest numbers in the group, given by Yarn 2 in the warp, dropped to 2280 average cycles to break, a level not reached in any of the earlier ring spun yarn warps with the SOS process. Compared to the <sup>-</sup>17,000 cycles to break exhibited by plant-sized, ring spun yarn, the air jet spun product was obviously inferior in abrasion resistance.

Mechanical and SPU analyses were then conducted on the air jet spun yarns (Table 5). The yarn strengths were running approximately 15% lower than observed earlier with the ring spun product, and the size SPU appeared high (17.1%). The general rule of thumb for ring spun yarn SPU is 10-12%.

A series of runs were conducted on the SOS slashing line with the air jet spun yarn to reestablish optimum variable settings in the process, to maximum Ruti Webtester results, and in particular, to optimize average cycles to break on the warp yarns. Both the standard 60/40 and a new 65/35 WD/AA size formulations were used. Run conditions and Webtester results are detailed in Table 6, along

## TABLE 4.

## RUTI WEBTESTER RESULTS PLANT SIZED AIR SPUN YARN

	1	<u>2</u>	<u>3</u>	4	<u>Average</u>
50 Yard Weight	0.925	0.925	0.925	0.925	
<pre>% Pick-Up</pre>	17.1	17.1	17.1	17.1	
Number Cycles 6th Break	3735	2335	2971	4391	3358
Number Cycles All Breaks	3714	2280	2949	4379	3331
Cycles/lst Hair Clump		1396			
Total Clumps	0	2	0	4	
Cycles/lst Quasi-Break	2581	1569	2525	3218	
Total Quasi- Breaks	.7	8	8	7	

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## TABLE 5.

## ANALYSES OF LANIER MURATA AIR JET SPUN WARP YARN

<u>Property</u>	Yarn	
	<u>Unsized</u>	<u>Sized</u>
Breaking Load	270.6 gm <sup>a.</sup>	318.6 gm <sup>b.</sup>
Breaking Elongation	8.68 <sup>a.</sup>	7.4 <sup>b</sup> ·
Solids Level	0.0%	17.1%
	(34.2 cotton count)	(29.2 cotton count)

<sup>a.</sup> Average of 100 readings from one package.

<sup>b.</sup> Average of 100 readings from two skeins (from separate sized warps).

#### TABLE 6.

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## SUMMARY OF RUTI WEBTESTER RESULTS FOR SOS SLASHING OPTIMIZATION

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No. IO Type Size Conditions Pick-up C (%) 1 Unsized Ring 2 Unsized Air 3 Plant Sized Ring PVA conventional process 17.0 1 4 Burlington Ring ? hot melt slashing, 5 rpm 11.4 7 rpm 18.8 5 Plant Sized Air PVA conventional process 13-14 17 1	ycles Load (#) (gms) 1340 346 692 264 16984 377 1480 1766 1948 322 3331 317 3455 3566 6050	Elong. (%) 8.9 8.8 7.5 7.8 7.8 7.6
(%)          1 Unsized       Ring         2 Unsized       Air         3 Plant Sized       Ring       PVA         4 Burlington       Ring       ?         5 Plant Sized       Air       PVA         5 Plant Sized       Air       PVA         1 Unsized       Air       PVA         1 Unsized       Ring       ?         1 Unsized       Ring       ?         1 Unsized       Ring       ?         1 Unsized       Air       ?         1 Unsized	(#) (gms) 1340 346 692 264 16984 377 1480 1766 1948 322 3331 317 3455 3566 6050 1773	(%) 8.9 8.8 7.5 7.8 7.6
1 Unsized Ring 2 Unsized Air 3 Plant Sized Ring PVA conventional process 17.0 : 4 Burlington Ring ? hot melt slashing, 5 rpm 11.4 7 rpm 18.8 5 Plant Sized Air PVA conventional process 13-14 17 1	1340       346         692       264         16984       377         1480       1766         1948       322         3331       317         3455       3566         6050       1773	8.9 8.8 7.5 7.8 7.6
2 Unsized Air 3 Plant Sized Ring PVA conventional process 17.0 : 4 Burlington Ring ? hot melt slashing, 5 rpm 11.4 7 rpm 18.8 5 Plant Sized Air PVA conventional process 13-14 17 1	692       264         16984       377         1480       1766         1948       322         3331       317         3455       3566         6050       1773	8.8 7.5 7.8 7.6
3 Plant Sized Ring PVA conventional process       17.0         4 Burlington Ring ?       hot melt slashing, 5 rpm       11.4         7 rpm       18.8         5 Plant Sized Air       PVA conventional process       13-14         17.0       17.0       17.0	16984     377       1480     1766       1948     322       3331     317       3455     3566       6050     1773	7.5 7.8 7.6
4 Burlington Ring ? hot melt slashing, 5 rpm 11.4 7 rpm 18.8 5 Plant Sized Air PVA conventional process 13-14 17 1	1480 1766 1948 322 3331 317 3455 3566 6050	7.8 7.6
7 rpm 18.8 5 Plant Sized Air PVA conventional process 13-14 17 1	1766 1948 322 3331 317 3455 3566 6050	7.8 7.6
5 Plant Sized Air PVA conventional process 13-14	1948     322       3331     317       3455     3566       6050     1773	7.8 7.6
17 1	3331 317 3455 3566 6050	7.6
17.1	3455 3566 6050	
6 418-79A-96 Ring 60/40 1 end, 33 kV, 1.6 ypm 12.6 400F oven, 270C rolls	3566 6050 1773	
7 439-79A-27R Ring 60/40 2 ends, 31.5 kV, 1.6 ypm 11.0	6050	
400F oven, 270C rolls	6050	
8 439-79A-27L Ring 60/40 2 ends, 31.5 kV, 1.6 ypm 16.3	1773	
400F oven, 270C rolls	1773	
9 439-79A-34 Ring 60/40 5 ends, 39 kV, 2.5 ypm	1773	
200C oven, 220C rolls 12-14 #1		
#2	1505	
#3	3670	
#4	4228	
#5	6663	
10 439-79A-34B	3196	
200C oven, 220C rolls	4533	
#2	6100	
#3	8867	
#4	4476	
· *5	7534	
1	1693	
11 439-79A-36C Air 60/40 5 ends, 32.5 kV 9.6 #4	680	
12 439-79A-40 Ring 60/40 5 ends. 22.5 kV. 2 ypm 10.6 #4	1821	
11.7 #5	2576	
13 439-51A-52A Ring 60/40 1 end. 35 kV. 2 ypm 19.4	5329	
240C oven. 220C rolls		
14 439-31A-52B Air 60/40 1 end. 35 kV. 2 ypm 10.00	316	
240C oven. 220C rolls		
15 439-51A-53A Air 60/40 1 end. 37.5 kV. 0.95 vpm 17.5	790	
244C oven. 240C rolls 27.4	3575 333	5.9
16 439-51B-57 Air 65/35 1 end.40 kV.0.99 vom 12.7	859	
230C oven. 250C rolls		
17 439-51B-55A Air 65/35 1 end.40 kV.0.95 vom 14.1	1114	
210C oven. 260C rolls		

## TABLE 6.

## (con't.) SUMMARY OF RUTI WEBTESTER RESULTS FOR SOS SLASHING OPTIMIZATION

Entry	Sample	Yarn	C / -	Run	Solids	Avg.	Break	
NO.	10	туре	Size	Conditions	Pick-up (%)	Cycles (#)	Load (gms)	Elong. (%)
18 43	9-518-55R	Ring	65/35	1 end,40kV,1.66 ypm	14.3	5350		
				210C oven, 260C rolls				
19 43	9-51 <b>8-</b> 57	Ring	65/ <b>3</b> 5	1 end,40kV,1.83 ypm	16.7	7712	386	7.4
				210C oven, 270C rolls	19.2	13289		
20 43	9-518-58A	Ring	65/35	1 end,40kV, 2.5 ypm	21.0	2971	385	5.8
				cold oven, 270C rolls				
21 43	9-518-588	Ring	65/35	1 end,40kV, 2.5 ypm	17.4	2678		
				cold oven, 270C rolls-				
				serpentine config.				
22 43	9~51B-58C	Air	65/35	1 end,40kV, 0.95 ypm	18.2	1215		
				cold oven, 270C rolls-				
				serpentine config.				
23 43	9-518- <b>59A</b>	Air	65/35	1 end,40kV, 2.5 ypm	21.7	712		
				cold oven, 270C rolls-	16.9	650		
				serpentine config.	17.8	1551		
					17.8	695		
24 43	9-51 <b>8-5</b> 98	Air	65/35	1 end,40kV, 2.5 ypm	14.6	1104		
				cold oven, 270C rolls-	12.9	996		
				serpentine config.	17.0	1373		
					13.0	1153		
25 43	9-51B-62	Air	65/35	1 end,40kV, 2.3 ypm	26.2	1095		
				205C oven, 230C rolls-	25.9	2938		
				serpentine config.	24.6	2254		
26 43	9-518-63	Air	65/35	1 end,40kV, 2.3 ypm	10,8	690		
				205C oven, 270C rolls-	11.3	621		
				serpentine config.	12.1	9 <b>64</b>		
					13.1	487		
					14.3	1565		
					15.0	1505		
					17.0	1364		
					. 19.1	900		

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with the results for freshly-isolated samples of both ring and air jet spun yarns from the Lanier Plant of WPP. The unsized air jet spun yarn withstood only 50% of the abrasion cycles endured by the ring spun yarn before breakage on average, and the sized yarn compared even worse (at comparable SPU levels (17%), 3331 average cycles to break for air jet spun vs. 16984 for ring spun yarns). Higher SPU levels on the SOS process with the air jet spun yarn gave abrasion resistance comparable to the plant-slashed analog, e.g., 3575 average cycles to break for entry 16 of Table 6, coded Run 439-51A-53A and possessing a 27.4 SPU. Tensile properties were also similar (333 gm breaking load vs. 317, 5.9% elongation to break vs. 7.6). However, the extra 10% of SPU was necessary to achieve a match in properties, as evidenced by the test results for the 17.5% SPU sample from the same run (790 average cycles to break). Indication that process variables were beginning to come into optimization range came from entry no. 19 of Table 6, where the 65/35 WD/AA formulation yielded a sized, ring spun yarn exhibiting 13,289 average cycles to break at a 19.2% SPU level.

The major results of the optimization study are summarized in Table 7. The decision was reached to switch from the five inch ETI fluidized bed applicator that had been used exclusively in the research up to this point to the larger unit that was to be used in the subsequent proof-of-concept trials at WPP (12" x 14" bed size). The Lindberg oven was replaced by a constructed oven incorporating three heating zones and real-time temperature controllers, also slated for incorporation in the WPP line (Figure 4). The creel was moved to the right of the applicator and the oven to the left,

## TABLE 7.

## RUTI WEBTESTER COMPARISONS OF SIZED YARNS

Sample <u>No.</u>	Yarn <u>Type</u>	<u>Size</u>	<u>Source</u>	Solids Pick-up <u>(%)</u>	Avg. Cycles on Webtester <u>(#)</u>	Breaking Load <u>(gms)</u>	Elong. To Break <u>(%)</u>
1	Ring Spun	PVA	Lanier Plant	17	16,984	377	7.5
2	Air Jet Spun	PVA	Lanier Plant	13-14	1,948	322	7.8
3	Air Jet Spun	PVA	Lanier Plant	17.1	3,331	317	7.6
4	Air Jet Spun	WD/AA	GT(SOS)	27.4	3,575	333	5.9


representing the same right-to-left process flow as was necessary in the WPP Research Center. These changes were made in hopes of better optimizing the process line with the tighter-controlled fluidized bed system and oven, and to check out the major components of the proof-of-concept trial line before moving to the plant site.

Another change incorporated into the overall scheme was to add air milling to the grinding sequence of the melt-interchanged, 65:35 WD/AA size formulation. After cooling the prepared size from the melt, the sequence thus constituted in order Wiley mill (gross) grinding-cryogenic mill grinding-air mill grinding. The air mill addition, made possible through a donation by H.B. Fuller Co., resulted in a much higher fraction of the powder in the 0-75 micron particle size range (Figure 5). The larger fraction of smaller particles was theorized to provide better penetration of the fine fiber structure at the surface of the staple yarns and lead to more efficient and uniform size film deposition, a postulate proved correct by higher Webtester performances in subsequent runs.

A number of 10-end warp trials were conducted on the final Tech line configuration with the open end spun yarn and the finer size formulation particle size. The run conditions are shown in Table 8 (65:35 WD/AA formulation). Table 9 gives the Ruti Webtester results and mechanical properties of yarns sized with the regular (cryogenic ground only) size, while Table 10 details the same data for the optimized formulation ground with the entire sequence (air milling added). At an average of only 12.5% SPU, the more finely-ground powder out-performed its cousin at a higher

FIG. 5. COMPARISON OF PARTICLE SIZE DISTRIBUTIONS OBTAINABLE WITH AIR MILLED INCORPORATION (65:35 WD/AA)



# TABLE 8.

TEN END WAP RUN CONDITIONS (FINAL LINE CONFIGURATION)

Size: 65:35	Eastman WD/Adipic Acid
Fluidized Bed:	40 KV Voltage 7 C <b>FM</b> Air
Oven:	200°C
Rolls:	250°C
Speed:	5 YPM

#### TABLE 9.

#### RUTI WEBTESTER RESULTS AND MECHANICAL PROPERTIES FROM INDUSTRIAL TEN END RUN (FINAL LINE CONFIGURATION)<sup>a</sup>.

	PICK-UP	BREAKING	ELONGATION	RUIT TEST
<u>END</u>	<u>(%)</u>	(LOAD(g)	<u>(%)</u>	(CYCLES)
1	14.4	365	5.7	1470
2	23.5	-	-	2680
3	23.4	-	-	-
4	17.3	380	6.4	-
5	22.8	-	•	-
6	12.4	348	5.8	-
7	16.5	360	5.8	1996
8	9.1	-	-	-
9	8.0	-	-	1748
10	10.3	-	-	-
AV	15.8	363	5.9	1974

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<sup>a.</sup>Powder obtained via Wiley Mill-Cryogenic Mill Grinding Only.

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## TABLE 10.

#### RUTI WEBTESTER RESULTS AND MECHANICAL PROPERTIES FROM OPTIMIZED TEN END RUN (FINAL LINE CONFIGURATION)<sup>a</sup>.

<u>End</u>	<u>Pick-Up (%)</u>	Breaking Load (g)	Elongation (%)	Ruti Test <u>(Cycles)</u>
1	8.2			
2	12.4			
3	13.0	378	6.0	
4	11.3			
5	20.1			
6	11.3			
7	16.6	360	6.1	3901
8	10.1			
9	12.9	338	6.1	3523
10	8.7			
AV	12.5	359	6.1	3712

Powder obtained via Wiley Mill - cryogenic mill - air mill grinding sequence.

average SPU (15.8%), enduring 3712 average cycles to break on the Webtester vs. 1974. Breaking loads and elongations were about the same for both runs. The optimized product gave test results very comparable to those obtained with the plant sized, air jet spun yarn (see Table 7). The decision was made to discontinue the Tech trials and devote all energies to preparations for the WPP proofof-concept trials at Shawmut, Alabama.

After several visits to the WPP site, the decision was reached to conduct the weaving trials on the SOS-slashed warp on Georgia Tech's Sulzer TW-11, Series 30,000 Projectile Weaving Machine. The Sulzer was similar to the same brand/vintage weaving machines in place in the partner Lanier Plant. The only modification required was a gear change to speed the production rate up from 250 to 280 picks per minute (ppm) to match the slowest plant speeds.

The targeted fabric, again designed to match plant product, was specified as:

Murata Air Jet Spun Yarn, 50:50 Blend 60 x 60 Construction 60" Wide Greige Width 62" Width Between Beam Flanges 3600 Warp Ends Required

Two appropriate beams were borrowed from American Scholze, and one shipped to the Lanier Plant to fill with plant-slashed yarn of the same construction to use as a standard on the Tech loom. A new reed/harness system for the loom was donated by Steel Heddle Co. to allow generation of the targeted fabric (29.04 dents per inch, 1800 total dents on the reed).

The slashing sequences for the SOS and conventional processes are outlined in Figures 6 and 7, respectively. Considerable thought went into the various directional changes of the yarn as it progressed through the SOS vs. conventional processes, as these can affect twist and twist direction with respect to the yarn axis.

The conventional pilot slashing line at WPP was utilized with the target warp sheet (60 ends). Components used included the creel, entrance comb, exit comb and take-up. Figure 8 provides the flow diagram of the WPP line modified with the SOS fluidized bed applicator and three-zone oven. The steam can section was simply bypassed with the SOS warp by guiding the sheet between the can banks. Utility requirements for the modified line were established (Table 11), and greige yarn was isolated from the same merge lot at the Lanier Plant in sufficient quantity to satisfy both the plantslashed as well as SOS-slashed warps. The quantity, due to several problems arising during section beam formation (see Figures 6-7), proved insufficient, and a second lot of plant greige yarn had to be secured in order to complete the section beam formation for feeding the SOS line.

To ascertain the effect of variable introduction by using two greige yarn feed lots to supply the SOS line, mechanical properties were determined in a statistical fashion for yarn samples from each lot (Table 12). Feed Batch A corresponded to the initial batch of isolated greige yarn that was utilized to complete the conventional (standard) slashing run, and for 71% of the SOS slashing run. Feed Batch B was the second lot of yarn secured from the Lanier Plant.

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FLOW DIAGRAM FOR WPP-SOS SLASHING PROCESS



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## TABLE 11.

# UTILITY REQUIREMENTS FOR SOS SLASHING LINE

# <u>Electricity</u>

	<u>115_Volts</u>	AC60 Hz	
Coating Machine	1. 2 Amps		
Collector	2. 15 Amps		
Air Dryer	3. 10 Amps		
	240 Volts	AC 60 Hz	Single Phase
Oven	1.72 Amps	preferably	100 Amps
Wipe Bars	2.52 Amps		

# <u>Compresses Air</u>

12 SCFM @ 85 to 100 PSI

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\* Note - A good earth ground is also needed.

#### TABLE 12.

#### TENSILE PROPERTIES OF SOS LINE FEED YARNS

Test Method: 30 single yarn samples from each batch, taken from three different positions in the spool 2' apart.

Property	Feed Batch A <u>(1-25 Section Beams)</u>	Feed Batch B <u>(26-35 Section Beams)</u>
Denier	145	144
Tenacity (gpd)	1.73	1.43
(Coefficient of Variation, C.V.)	(14.7%)	(21.2%)
Breaking Elongation %	8.5	7.9
(C.V.)	(10.6%)	(17.6%)
Initial Modulus (gpd)	2.10	1.88
(C.V.)	(19.6%)	(17.3%)

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coefficient of variation (%C.V.) between the two lots was surprising, with the Feed Batch B yarns proving to be considerably weaker than the A batch (0.3 gpd less tenacity, only 83% as strong as A), and the %C.V. much wider. Breaking elongation and initial modulus were also lower for the Batch B yarns, and the distribution of breaks was wider (compare Figures 9 and 10). Some breaks occurred in the ~100 gm range for Batch B yarns, whereas the lowest breaks in the Batch A yarns occurred at the ~160 gm range.

The Lanier Plant had only recently switched production from ring spinning to air jet spinning, and quality on the overall plant product had not reached previous levels of control during the transition period. The discrepancy in mechanical properties between the Feed Batch A and B yarns eventually proved to have a negative impact on slashed yarn properties in weaving trials.

West Point Foundry & Machine Co. designed and built 31 section beams for takeup of the SOS slashed yarn that incorporated a one inch space between the section beam heads for laydown of the 60 yarn ends. The composite support discs were reinforced when mounted on the beam axis by two metal discs of the same diameter cut from conventional beam heads. The narrow width between the beam heads required development of a technique for initiating takeup of the 60 end sheet (which was condensed down to a one inch tape through combs) on the center hub. The beam heads were made in sufficient diameter to hold ~1000 yards each of slashed yarn.

The conventionally-slashed standard was prepared by WPP employees at the Research Center, following the procedure detailed in Appendix 2. The PVA size mix was obtained from the Lanier Plant



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BREAK DISTRIBUTION FOR FEED BATCH B YARNS (SECTION BEAMS 26-35) FIG. 10.

Breaking Load (gms)

production stock, and consisted of recycled PVA from the plant's ultrafiltration recovery unit. Blue tint was added to the size formulation to distinguish the PVA-slashed material.

The SPU of the conventionally-slashed yarn was higher than that normally obtained in plant production, indicating translation difficulties with the pilot (three-foot wide) Research Center line and its limited speed and controls (Tables 13 and 14). The yarn produced to be а 35's cotton count yarn by plant was specifications, but measurements of the greige yarn at Georgia Tech yielded a consistent value of 36.6's cotton count. As shown in Table 13, the size SPU was high using either yarn count, running in the >20% range. The Quality Control Laboratory of WPP reported a 14.5-15.1% SPU range for the same yarn pulled from plant production Additional property/performance enhancement was (Table 14). forecast for the conventionally-slashed yarn from the Research Center as a result of the additional size loading vis-a-vis plant production.

The run conditions set for the SOS trials are reviewed in Table 15. The conditions were based on the development work detailed earlier, and with the late change from ring spun to air jet spun yarn, were heavily dependent on the 10-end warp runs conducted at Georgia Tech with the trial fluidized bed applicator and curing oven.

The proof-of-concept trials were conducted over a two week period at the WPP Research Center utilizing one team/shift in order to operate around the clock. Each team consisted of a faculty member, a research associate, a WPP technician and a WPP liaison

## TABLE 13.

#### ANALYSIS OF CONVENTIONAL-SIZED (BLUE) YARN SPU LEVEL

<u>Sample (#)</u>	<u>Based on 35's</u>	<u>Based on 36.6's</u>
1	21.8	27.3
2	23.1	28.7
3	25.4	31.1
4	24.2	29.9
5	21.2	26.8
6	21.6	27.2
7	21.7	27.2
8	21.7	27.2
9	21.6	27.2
10	21.4	26.9

#### TABLE 14.

#### WPP DESIZE RESULTS OF LANIER PLANT - SLASHED YARN

- Yarn: 35.0's 50/50 polyester/cotton AJS yarn from Lanier
- Size: Lanier's reclaimed PVA mix
- Method: Boil 30 minutes in 0.1 N HCl solution (old ASTM D-334-60 method)

Add-On (1000 yds per doff)

lst	Doff	14.8%
2nd	Doff	14.5%
3rd	Doff	15. <b>1</b> %
4th	Doff	14.6%
5th	Doff	14.8%

#### TABLE 15.

# SOS TRIAL RUN CONDITIONS

Size 65:35	Eastman WD/Adipic Acid
Fluidized Bed:	75 KV Voltage 12 CFM Air
Oven:	200°C
Rolls:	250°C
Speed:	5-8 YPM

person from its research staff. To reach a standard procedure, a line was marked on the inside of the fluidized bed plastic cover so that additional powder could be added by hand as the bed depleted. Line speed was initially targeted for the 5-8 ypm range, with plans to raise the speed provided the targeted SPU (15%) could be maintained within the limitations of the ETI fluidized bed system (voltages, air flows, powder charge capabilities, etc.). SPU was checked at the start of each section beam, and after doffing each run end.

A total of 35 section beams were accumulated during the two week trial. In the middle of the Beam 27 run, the facility's compressed air supply failed due a freeze-up problem, which took approximately one hour of run time to correct. The air pressure to the unit dropped below the 12 cfm target during this period, and fluctuated sporadically. Beam 27 was retained and used in the final warp beam, a decision which proved to be unwise, as detailed later in this report. With the exception of the system failure during Beam 27, only minor problems were encountered and mastered during the 35 runs, mainly dealing with technique development on restarting an end that broke during a run, starting the condensed warp on the takeup section beam, finding and maintaining the correct yarn tension on the warp to prevent "tracking" of the yarns out of the grooves on the heated Teflon rolls, etc.

The close proximity of the yarns in the heating zone occasionally resulting in "twinning", where two adjacent ends would touch and stick together as the size melted and flowed. Generally, keeping proper tension on the yarns and use of a comb (one end per

dent) immediately after the heated roll at the oven exit split the yarns back apart, although occasionally operator intervention was necessary to segregate two ends.

Table 16 details SPU at both the start and end of each of the 35 section beams. The data provides no information on SPU in the bulk of the beams between start and finish, and the reliability of the figures was considered better for the reading at the end of runs versus the beginning, as attainment of steady-state conditions was suspect in the early stages of each beam's accumulation. Several of the measurements caused considerable concern in terms of low SPU, and in particular those beams where either the initial or ending measurement showed SPU's in the single digits (Beams 2, 3, 5, 6, 24, 25, and 30). The scatter in the data also indicated that steady state was not being maintained consistently throughout the trial. The perturbation in cloud density, size and configuration visible upon hand addition of size to periodically replenish the bed was suspected as being the cause.

To check cross-bed uniformity of deposition, various ends of Beam 25 were analyzed over 150 yard lengths for SPU, and the averaged results for each end are shown in Table 17. The results were more uniform than had been achieved in the Tech preliminary runs, but indicated a heavier concentration of size and resulting SPU in the center of the bed (End 31, 23.4% SPU).

Beam 31, which possessed two broken ends that ran "wild" through the section beam and was thus unsuitable for use in loom beam formation, was fully sacrificed for testing to determine through-beam SPU uniformity. The results are graphed in Figure 11.

# TABLE 16.

# **%** SPU AT BEGINNING AND END OF GENERATED BEAMS

<u>Beam</u>	<u>Start</u>	End	<u>Beam</u>	<u>Start</u>	<u>End</u>
1	13.5	12.0	19	12.0	12.3
2	12.0	9.2	20	12.3	13.0
3	9.2	14.4	21	13.0	16.6
4	14.4	12.3	22	16.6	21.3
5	12.0	8.4	24	13.7	9.2
6	8.4	12.6	25	9.6	10.3
7	20.7	15.9	26	14.6	16.1
8		11.3	27*	16.1	20.5
10	14.0	14.3	28	14.5	18.0
11	14.3		30	11.3	9.4
12	21.0	11.4	32	16.2	17.0
13	11.4	10.9	33	17.0	20.5
16		23.5	34	20.5	
17		11.5			
18	12.6	12.0			

\*Beam 27 - Failure of Air Supply

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# TABLE 17.

ACROSS-WARP SPU UNIFORMITY OF SOS BEAM 25 (150 YARD SAMPLES)

End	SPU %
11	19.1%
31	23.4%
51	17.4%
60	17.9%

The data showed conclusively that steady-state deposition was being periodically disturbed during the run, with high and low SPU peaks occurring roughly every 60 -70 yards. Highest SPU recorded was 36.6% and low was 6.9%, for a raw range of 29.7% SPU difference for yarns at various points in the beam. The section of the beam from 460 to 500 yards from the end was also disturbing, with the 40-yard section falling below the 10% threshold level. High, medium and low SPU groupings from Figure 11 are collected in Table 18.

Figure 12 connects the maxima and minima points of Figure 11, emphasizing the close periodicity of the high and low range values as a function of distance into the beam. Figure 13 graphs the elapsed time between peaks of Figure 11 versus peak number. The graph deviates around the seven minute mark, which corresponded to the approximate time required under the run conditions to deplete the fluidized bed to a point below the marked add line, and thus to the time that powder was added to the applicator. From the data, the adds were dramatically altering the cloud density, greatly increasing the yarn SPU for the warp sections passing through the applicator during the add period. The bed settled down over the next few minutes, giving a gradual drop in SPU until a valley (steady state) was reached (Figures 11-12). Shortly after reaching steady state, the bed volume would reach a depletion point to carry it below the add line, and the cycle was repeated as an add was In the 460-500 yard range, a broken end requiring immediate made. operator attention delayed the addition of powder by several minutes from the norm, allowing the powder level to drop considerably below the add line and resulting in a 40-yard section



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FIG. 11.

# TABLE 18.

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#### SPU GROUPINGS FROM THROUGHOUT SOS BEAM 31

SPU <u>REGAIN</u>	SAMPLE <u>NO.</u>		SPU 
High	3		36.6
	40		35.0
	32		32.9
	27		32.1
	2		<u>30.6</u>
		Average	33.4
Medium	26		20.2
	31		19.9
	29		19.5
	21		19
	12		<u>18.9</u>
		Average	19.5
Low	47		9.1
	49		9.1
	28		8.8
	46		<u>6.9</u>
		Average	8.5

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FIG. 12. MAXIMA AND MINIMA OF FIGURE 11AS A FUNCTION OF BEAM LOCATION

DISTANCE INTO BEAM (yds x 10)



FIG. 13. ELAPSED TIME BETWEEN PEAKS OF FIGURES 11-12

with subnormal SPU. The evidence clearly indicated that the practice of hand-addition of powder during the runs had resulted in beams with widely-fluctuating yarn SPU levels throughout the diameters, culminating in a critical loss of control in yarn quality and properties.

Table 19 compares Ruti Webtester results of greige, SOS sized and conventionally sized yarns from the WPP Research Center trials. As expected from Figures 11-12, the SOS yarns gave widely varying performances on the Webtester, ranging from 302 average cycles to break to 614, and all uniformly low. The conventionally sized yarn with its high SPU (Table 13) gave a corresponding high Webtester result of 7144 average cycles to break.

Mechanical properties of the conventionally sized yarns were also much higher than the corresponding SOS slashed materials (Table 20), ranging from 0.10 gpd in comparison to the Feed Lot A yarns to 0.19 gpd versus the Feed Lot B yarns. As noted earlier, the Feed Lot B yarns were, on average, 0.30 gpd weaker than the Feed Lot A yarns directly from the Lanier Plant, a deficiency not completely eliminated by the added size.

The degree of "hairiness" of the SOS sized yarns was good, comparing favorably to the conventionally slashed standard and actually out-performing it in terms of a lower population of >1.0 mm fray lengths (Toray Fray Counter data, Figure 14). The wiping action provided by the grooves in the heated Teflon rolls had performed well in laying down the hairs along the yarn axis, and the avoidance of bust rods at the end of the line (necessary in the conventional process to break the bridging PVA film between yarns

## TABLE 19.

# RUTI WEBTESTER RESULTS FOR CONVENTIONAL SOS SIZED YARNS (AVERAGE CYCLES TO BREAK)

<u>Beams 26-36</u>

	SOS	Conv.		SOS
Unsized	Sized	Sized	Unsized	Sized
329	499 (#4)	7144	456	302 (#32)
	302 (#14)			614 (#31)

<u>Beams 1-26</u>

#### TABLE 20.

# MECHANICAL PROPERTIES OF GREIGE, SOS SLASHED AND CONVENTIONALLY COMBED YARNS

		<u>Lot_A</u>	
	<u>As Spun</u>		<u>SOS Sized</u>
Breaking Load	251 g		326 g
Tenacity	1.73 g/d		1.86 g/d
Elongation	8.5%		7.1%
Initial Mod.	2.10 g/d		2.71 g/d
		<u>Lot B</u>	
	<u>As Spun</u>		SOS Sized
Breaking Load	206 g		322 g
Tenacity	1.43 g/d		1.77 g/d
Elongation	7.9%		6.2%
Init. Mod.	1.88 g/d		2.63 g/d
		Conventional <u>Sized</u>	
	<u>As Spun</u>		<u>SOS_Sized</u>
Breaking Load	264 g		341 g
Tenacity			1.96 g/d
Elongation	8.8%		7.1%
Init. Mod.	•••		2.94 g/d

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FIG. 14. TORAY FRAY COUNTER DATA COMPARING YARN HAIRINESS

and sheets) also aided in producing a less hairy yarn.

In preparation for weaving trials, 30 of the available section beams of SOS slashed yarns were selected for rebeaming on two 20inch aluminum section beams (900 ends each) that in turn were combined in a rebeaming operation to give a single, 20-inch wide section beam of 1800 ends. Finally, the white SOS yarn was merged with the blue, conventionally slashed yarn on a 3600-end loom beam so that both yarns would experience the same abrasion, tension and forces progressing through the Sulzer projectile loom, i.e., develop a true "head-to-head" weaving performance comparison between the two yarns (see Figure 6). All of the beaming operations were performed on a warper at the facilities of West Point Foundry & Machinery Co. in West Point, Georgia.

In the initial consolidation from the one inch section beams, significant problems were encountered in smooth pull-up of the SOS yarns from the spools. Frequent "cross-over" of adjacent yarns was evident throughout the beams, which caused breakage when the crossed points reached the adjustable rake-comb at the end of the warper. In addition, the yarns exhibited substantial "sticking", with substantial force required to separate the individual ends as they came up on the beam hub.

The cross-over problem was eventually traced to the course rake-comb used at the end of the WPP slasher line at the Research Center. Since substantial reduction of the warp width (from 15 inches to one inch) was necessary in a relatively short distance (approximately four feet), a comb with 30 dents/inch was used to guide the yarn ends into the adjustable rake-comb, which was coarse

(10 dents/inch, Figure 8). To achieve the funneling of the 60 ends into a one inch tape capable of take-up on the section beam, as many as six ends of yarn were strung in a single dent of the rakecomb by the WPP technicians. With the natural twisting action of the take-up, the yarn tension utilized and the tendency of a single end to "wander" in the space between the two metal pins defining each dent, the various ends tended to cross.

The sticky nature of the yarns was suspected to be moisturerelated, as some unreacted adipic acid was trapped in the size upon cooldown from the melt in the transesterification process. Although the percentage of AA per unit mass of the size was not quantitatively determined, collection of a white powder on the exterior, cool part of the oven was observed. With AA's tendency to sublime, and with the inability of higher molecular weight transesterification product to pass into the vapor phase without degrading, the condensed powder was suspected of being the diacid. Subsequent Infrared Spectrophotometric Analysis of a sample of the sublimate confirmed it as adipic acid. With its two organic acid functionalities, AA is hygroscopic in nature, capable of forming multiple hydrates with water molecules from air.

The tacky nature of the size was reemphasized a few months after the WPP trial terminated with a sample of the material that had been stored in a tightly-sealed, one gallon plastic jar. The fine powder had solidified into a solid block upon sitting, indicating a long-term storage problem with the size in addition to the yarn sticking problem caused by its tacky nature.

The stacking order of the one-inch section beams for the

initial 20-inch section beam formation is shown in Figure 15. Accounting of the beam stacking order became important later in correlating areas of the final loom beam that gave frequent breaks in the weaving trial.

After considerable experimentation, it was discovered that a rod placed between the two yarn sheets exiting the stacked section beams and thrust up and down in a "beating" motion successfully separated stuck yarn ends as they came up, and also helped uncross twinned yarns. Eventually, using a side-arm lab flask shaker with an oscillating arc control and outfitted with an aluminum beater paddle, the process was motorized. The formation of the two SOS section beams then proceeded smoothly, with far fewer problems encountered on rebeaming to the single, 1800-end section beam (Figure 6). All of the beaming operations with the conventionally slashed yarn went smoothly with no added features to the warper line.

The initial weaving trial on the Sulzer TW 11 Projectile Loom was conducted with the beam of yarn slashed at the Lanier Plant on the commercial process line. To increase the effect of size problems and decrease the effect of "thin yarn spots", the technician closed the stroke on the harnesses and adjusted to a low cloth support setting. Out of 1.996MM picks performed during the trial, only 452 loom stops were recorded, corresponding to a rate of stops of only 22.65/100K picks. Only 102 of the stops were deemed to be size-related by the operator, corresponding to 5.11 stops/100K picks, or 22.6% of the total stops. Size-related stops included: size oven fly; stuck ends; lint balls; and cut-out ends.

#### FIG. 15.

## SPACING ORDER OF ONE-INCH SECTION BEAMS TO FORM TWO 20-INCH BEAMS

<u>Take Up Beam</u>

<u>Left</u> <u>Front</u> <u>Right</u>

Bottom Beam

33-32-35-34-30-28-27-26-25-24-22-21-20-19-18

<u>Top Beam</u>

17-16-14-13-12-11-10-8-7-6-5-4-3-2-1

#### <u>Rear</u>

The following section beam numbers were missing: #31: 2 broken ends @ ~400 yds. (475 yds. short of end), used as spare #29, 23, 15, 9: Rerun

Beam numbers 32 and 33 had one broken end, each beam:

#32: Break @ 685 yds. (190 yds short of end)

#33: Break @ ~400 yds. (475 yds. short of end)

The continuation yarn was marked with a black marker to denote the break area.
Slashing process-related stops were attributed to 38 of the total number, accounting to 1.90/100K picks rate (8.4% of total breaks). These stops included: ends run out; ends run in; hard size strips; and replaced ends.

Yarn quality-related stops, which included only thin spots in individual warp yarns as the cause of breakage, accounted for 238 of the total (52.7%, 11.92/100K picks). In discussing the percentages with industry experts, the frequency of yarn qualityrelated breaks was considered high, and again was an indicator of WPP's recent conversion to air jet spinning and its struggle to regain previous levels of quality control over product from the new machines.

Figure 16 shows that most of the warp breaks that could be attributed to one of the three central causes were bunched around the center of the loom beam (25-40 inches from the left selvage). The extraordinary peak jump at the 26 inch position indicates an off-quality package(s) of considerably weaker strength than its neighbors, resulting in a warp section that failed <sup>2</sup>X more frequently than the next weakest section at the 33 inch position.

The propensity for yarns to break mainly in the center of the warp could not be readily explained. Possibilities included:

1. Extra tension in the center of the loom, or in the warp from earlier beaming operations.

2. A weak section of warp caused by packages from a single machine producing off-quality yarn being creeled together at the warper.

3. Insufficient size mixing or uneven size application

FIG. 16 NUMBER OF BREAKS ACROSS THE LOOM WARP AS A FUNCTION OF DISTANCE FROM THE LEFT SELVAGE



FIG. 17



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## TABLE 21.

## WPP DESIZE ANALYSIS-SPU DETERMINATION ON TWO YARNS

	BREAKING STRENGTH <sup>a</sup>	ELONGATION TO BREAK	ACID DESIZE (SPU)
Conventional Sized Yarns (Beams #3 & #4)	12.1 oz.	5.7%	14.8%
SOS Sized Yarn (Section Beams #5 <sup>b.</sup> & #10 <sup>c.</sup> )	11.6 g.	6.6%	9.3%

<sup>a.</sup> 40 breaks on Uster Single Strand Tester.
<sup>b.</sup> 8.7% SPU for Beam 5.
<sup>c.</sup> 10.3% SPU from Beam 10.

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FIG. 18. STOP DATA FOR BATCH A FEED YARNS





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causing a section of warp to be improperly slashed.

The list is not all-inclusive, and the effect may have been synergistic between several of the possibilities operating simultaneously.

Figure 17 summarizes all of the warp breaks that occurred, again as a function of the distance from the left selvage. A total of 74 breaks (16.4%) could not be clearly attributed to one of the three main categories of break causes, and were thus relegated to miscellaneous status.

Prior to beginning the weaving trial on the combined SOS/conventionally slashed yarn loom beam, samples were submitted to the WPP quality control lab to determine the SPU by its acid desize procedure (specific to PVA and PVA/starch blend size formulations). The results, shown in Table 21, gave a near-normal 14.8% SPU for the blue, conventionally-slashed yarn, while the SOSsized yarn contained only a 9.3% average SPU loading. One caution, however, was the ability of the acid desize chemicals used in the WPP procedure to remove the 65:35 WD/AA formulation from the yarn.

A total of 114K picks were conducted on the weaving trial with the mixed SOS- and conventionally-slashed yarn beam. The SOSslashed warp yarn break data as a function of distance from the selvage are detailed in Figures 18-19. The former deals with the warp yarns originating from the Batch A feed yarns from the Lanier Plant, and the latter with the Batch B feed yarns that had been shown to possess inferior mechanical properties to those of Batch A. Table 22 summarizes the stop data for the trial.

In comparing the plots to Figures 16-17, breaks were more

## TABLE 22.

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STOP DATA FOR MIXED LOOM BEAM CONTAINING SOS- AND CONVENTIONALLY - SLASHED YARNS

TOTAL PICKS ACHIEVED:	114К
A STOPS:	151
B STOPS:	166
TOTAL STOPS:	317
RATE OF TOTAL STOPS:	278/100K PICKS
RATE OF A STOPS:	132/100K PICKS
RATE OF B STOPS:	145/100K PICKS

numerous than with the plant-slashed beam, and more generally distributed across the beam width. Rarely were 1000 picks achieved continuously without a break during the trial. From Figure 15 and with each section beam contributing a one-inch coverage to the loom beam, specific problem section beams could be easily identified and correlated with high peaks in the plots. For example, Section Beam 27, which had been accumulated during the compressed air pressure drop at the trial (see earlier discussion), was located 5-6 inches from the left selvage, corresponding to the pronounced 18-break peak in Figure 19 (the highest of any SOS yarn section on the loom beam). As expected from the greige yarns' mechanical properties, Batch A outperformed Batch B, but neither compared to the excellent weavability of the plant-sized or conventionally-sized yarns. For example, the rate of total stops for the SOS-sized yarns was tenfold greater than that for the plant-slashed material (278 stops/100K picks vs. 22.7 stops/100K picks). Yarn breakage on the mixed beam due to the blue-tinted, conventionally-slashed standard material was so infrequent as to be considered negligible, compared to the frequent stops precipitated by the SOS-slashed yarns. The beam was performing so poorly that the weaving trial was terminated after 114K total picks, compared to 1.996MM picks completed with the plant-slashed beam.

Two problems thus arose in the proof-of-concept trial that could not be foreseen in the small-scale research:

1. Normal slashing speeds could not be achieved within the limitations of the utilized ETI fluidized bed applicator system.

2. The melt-interchanged, 65:35 WD/AA size formulation did not possess sufficient film properties to provide excellent weavability on the Sulzer Projectile Loom, and its tacky nature facilitated sticking of slashed product while preventing long-term storage of the powdered size.

### **II. TEXTILE COLOR XEROGRAPHY**

Over the past few years, the textile industry has moved toward quick response, just-in-time delivery and shorter process runs to facilitate frequent style and color changes. Reduced process efficiency accompanies short runs unless changeover down time is small. Processes such as continuous carpet dyeing have been modified to minimize time required for changeover. However, current fabric printing systems do not lend themselves to rapid changeover.

Rotor screen printing, currently the predominant method of fabric printing, has several disadvantages. Color and pattern changes require long process times, with screen production slow and expensive. Screens have relatively short lives and require considerable storage space when not being used. Thus a new technology for fabric printing is needed that will permit frequent style and color changes with a minimum of down time for changeover, and which allows storage of design information. Xerographic printing has the potential of meeting these requirements.

Technologies eliminating water from fabric printing while simultaneously relieving response time and information storage problems associated with screen printing were considered. Two major candidates were ink jet printing and electrophotography. Although ink jet printing has promise, disadvantages are also associated with it. The major one is that it is liquid-based, relying on solubilized dyes, which eliminated it as a candidate for the DOE project. Other disadvantages include: high resolution

needed for apparel is probably unattainable; insoluble dyes such as disperse dyes and pigments are not compatible with the technique; dyes used in the process must have the proper textile characteristics and influence on rheology; and the use of three primaries to produce color is probably not attainable.

Electrophotography involves the formation of a latent image and transforming it into a visible image or print. Two types of electrophotography were considered for fabric printing: direct imaging on the fabric and xerography. Direct imaging eliminates some of the steps of xerography, but presents two problems not associated with it. Preliminary tests indicated that cottoncontaining fabrics, under standard conditions of 65% relative humidity and 20°C, dissipate charge very rapidly. Thus the time available for developing the charged pattern is extremely short. Since much of the fabric to be printed contains cotton, the charge dissipation problem would have to be solved, perhaps by bone-drying the fabric, an energy-intensive, expensive process. The other problem is related to the nature of the fabric surface. Developing the positive print image without the entrapment of toner in uncharged regions of the fabric would be difficult. Due to these potential problems, and with xerography being an already highlydeveloped technology for paper printing, it was selected as the method of choice.

Xerography has several potential advantages for printing fabrics. Information storage can be either optical or computerized, eliminating the need for large storage space for wire screens. Another is the potential for producing color using three

primaries. Since the system can be computerized, fast style and color changeover are possible. The resolution needed for printing apparel fabric should be attainable. Image development can be achieved without the use of solvents. Finally, pigments which are generally much less expensive than dyes and offer better lightfastness and other properties can be used for coloration, and the process avoids a final washing/drying step.

Fabric xerographic printing has requirements beyond those for paper printing. Xerographic paper printing systems have been designed primarily for operating in the batch mode (a single sheet is normally printed) and for fairly narrow widths (usually 8.5 inches). Fabric printing systems will need to print much wider webs in a continuous mode. Toner requirements for paper printing are quite different from those for textile applications, with the binders normally consisting of styrene/acrylate copolymers exhibiting poor adhesion to textile fibers and dry cleaning solvent fastness.

The basic steps of xerography are illustrated in Figure 20. Metal that is electrically grounded is coated with a layer of photoconductor (PC). The first step involves charging the surface of the PC which will hold a charge in the dark. The charging is usually accomplished by passing a corona over the PC surface. The second step involves producing a "latent", or negative, electrostatic image by exposing the PC to light. Since light causes the PC to become conductive, charge is drained from the surface in regions that are exposed. This step is usually accomplished by reflecting light from an original or passing light











3. DEVELOP

4. TRANSFER



5. FIX



# 6. CLEAN

FIG. 20

BASIC STEPS IN XEROGRAPHY

through a transparency onto the PC surface. A laser driven by a computer can also be used to produce the image. The third step is developing the latent image by placing toner (colorant plus binder) in regions where electrostatic charge is located. Development involves the use of a developer system composed of carrier bead and toner (melt blended binder-pigment). The carrier bead, which consists of metal shot or wire filings coated with a polymer film (often on epoxy), is much larger in size than the toner particles. The triboelectric characteristics of the toner and carrier are such that when they are thoroughly mixed, they become oppositely charged and attract each other. The carrier is oppositely charged from the When carrier which holds toner on its surface is PC surface. brought into contact with the PC, the toner is attracted to charged regions of the PC. Transfer of toner to these regions on the PC develops the positive print image.

The fourth step is the transferring of the developed image to the substrate being printed. The substrate is brought into contact with the PC, and the back is strongly charged, usually by employing a corona, so that the toner transfers to the surface of the substrate. The fifth step is fixing the toner to the substrate. The temperature of the toner is raised, causing the resin binder to flow. Pressure is often used as well as a heat source. Following fixation, the surface of the PC is cleaned and the process is repeated.

### PHASE I

The objectives of Phase I were to investigate on a bench scale the technical feasibility of using basic xerography to print woven

fabrics and to identify binder materials for toners meeting textile requirements. The results of Phase I were reported in the first Final Report of DOE Project Number DE-FG05-84CE40702.

#### PHASE II

The objectives of Phase II were to continue the development of a suitable toner for xerographic printing of polyester/cotton sheeting fabric, and to demonstrate the continuous xerographic printing of fabric. A beam-to-beam printing process with a single color was developed.

After over 60 commercially available materials as well as melt-blended combinations were screened as potential binders for fabric toners, Elvax 410 (a polyethylene-co-vinyl acetate produced by E. I. du Pont Co.) was selected as the primary candidate. Elvax 410 had good film forming and adhesive properties while exhibiting stress/strain properties reasonably in the range of those of the standard, Hycar 26120 resin. The major problem associated with Elvax was that its melt viscosity (~10<sup>6</sup> cps) was appreciably higher than the target value (approximately 1000 cps) believed to be needed for good flowability. Blends of Elvax 410 with several other materials were made in an attempt to reduce its melt viscosity while maintaining desirable binder film properties. Materials used in the blending tests included various Allied Chemical Co. copolymers (of the ethylene or ethylene-acrylic acid copolymer type, but with lower molecular weight). The results of the melt viscosity and dot adhesion tests are shown in Table 23. Two of the blends (Samples 5 and 6) at a 1:1 weight ratio gave

promising melt viscosities, while yielding adhesion results comparable to virgin Elvax 410. Four of the other blends (samples 8-11) had melt viscosities in the 3000 to 6000 cps range and acceptable adhesion results.

The stress/strain behavior of selected Elvax 410 blends were compared with those of virgin Elvax 410 and the standard, Hycar 26120 resin. In Figure 21, virgin Elvax 410 is compared with the standard. The film produced from Hycar 26120 resin had a low initial modulus, an extremely high elongation-to-break and a low tenacity. Of the candidate binders, Elvax 410 came closest to duplicating the stress/strain properties of the standard. Although Elvax 410 has a higher initial modulus than the standard, its elongation to break and breaking strength were similar to those for the standard.

The film properties of the blends did not compare favorably with those of the Hycar 26120. For example, the stress/strain plot for 8:1:2 and 9:1:2 blends (Samples 12 and 13 in Table 23) are shown in Figure 21. Even small amounts of AC-400 and AC-580 blended with Elvax 410 caused brittleness, resulting in high initial moduli and breaking strengths, but extremely low elongations-to-break. Based on the results, the melt blend approach was abandoned and the virgin Elvax 410 was chosen as the primary candidate for textile toner development.

A review of commercial paper xerography copiers was made to identify a copier that could be most easily modified for continuously printing fabric. Hunt Chemical Company, a paper toner manufacturer, recommended a Xerox Model 3100 copier. The layout of

Table 23. RESULTS OF MELT VISCOSITY AND DOT ADHESION TESTS WITH VARIOUS RESINS AND THEIR BLENDS

Sample	Compo	osition		Viscosity	Total Adhesion
Number	<u>Ratio</u>	<u>Material</u>		(CPS)	<u>(</u> &)
1		ELVAX 410ª		6.3x106 <sup>6</sup>	89
2		AC-400 <sup>b</sup>		610	35
3		AC-580°		650	42
4		AC-629 <sup>d</sup>		200	12
5	1:1	410 <sup>a</sup> :400 <sup>b</sup>		2600	100
6	1:1	410:580°		1910	88
7	1:1	410:629 <sup>d</sup>		2870	9
8	5:4:1	410:400:580	3150		97
9	6:2:2	410:400:580	3500		88
10	7:2:1	410:400:580	5150		84
11	7:1:2	410:400:580	6010		83
12	8:1:2	410:400:580	9000		
13	9:1:2	410:400:580	10,000	)	

a. A poly(ethylene-co-vinyl acetate), duPont.

b. A poly(ethylene-co-vinyl acetate), Allied.

c. A poly(ethylene-co-vinyl acrylic acid), Allied.

d. An oxidized polyethylene, Allied.

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e. Measured using a Model LVT Brookfield Viscometer, Spindle 4, at 200°C.

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FILM STRESS/STRAIN CURVE COMPARISONS OF BLEND AND VIRGIN RESINS

FIG. 21

STRAIN.

the copier facilitated conversion from paper feed to fabric feed which would allow continuous printing of 8-1/2 inch wide fabric.

One Xerox Model 3100 copiers was used to obtain the operational characteristics of the unit. Another was modified to allow manual control of major components of the system and to permit continuous operation for fabric printing. Under manual control, it was possible to vary copier parameters during testing.

The following modifications were made to the Xerox 3100 copier: installation of variable speed motor to allow control of fabric speed; rewiring of exposure light; installation of a variable voltage power supply to allow varying magnetic brush speed; installation of controls for fusing system; installation of let off and take up rolls for the fabric; and attachment of power supplies to four corotrons and the developer cage.

Paper developer systems (toner plus carrier) compatible with the Xerox 3100 copier were used initially to demonstrate continuous xerographic printing of fabric. While the continuous xerographic fabric printing system was being constructed and demonstrated, work continued to fine-tune a textile developer system which would satisfy textile printing requirements. Textile toners for polyester/cotton blend fabric and 100% polyester fabric were sought. The toner for polyester/cotton blend fabric consisted of a binder plus pigment, while toner for 100% polyester fabric was sublimable dye without binder. The concept was to use heat to transfer the sublimable dye into the polyester so that binder would not be needed.

Based on the results of Phase I, poly(ethylene-co-vinyl

acetate) resin (EVA) was selected as the primary binder candidate for the toner for polyester/cotton blend fabrics. Two types of poly(ethylene co-vinyl acetate) resin (EVA) were used. In addition Elvax 410, MU 760 (another polyethylene-co-vinyl acetate, 82:18 ratio, which is similar to Elvax 410, but produced by U.S. Industries) was also investigated. Neither of the two resins were produced commercially with pigment, and pigmented samples could not be obtained. Attempts were made to produce toner by melt blending of the resins with 5% by weight of phthalocyanine blue pigment and grinding. The blends were processed through a Wiley mill, a cryogenic grinder, an air mill and then sieved.

Candidate toners for 100% polyester fabric were produced two ways. One was produced by grinding and sieving blue disperse dye cake obtained from Ciba-Geigy. The other was made by grinding and sieving blue disperse dye diluted with lignin sulfonate filler/dispersing agent. The chromophore in both samples was the same.

Production of developer systems using the textile toners required finding a carrier particle compatible with the toners. Samples of the four toners described above were evaluated by Hunt Chemical Company in an effort to find a suitable carrier.

The physical properties of the non-polar, four candidate toners were evaluated by Hunt Chemical Company. The triboelectric properties of the non-polar disperse dye samples were such that very little charge was generated when the material was thoroughly mixed with various carriers. Thus a developer system could not be made using the Ciba-Geigy blue disperse dye as toner with available

carrier particles compatible with the Xerox Model 3100 copier.

Hunt Chemical's studies revealed that the particle size of the melt-blended EVA/pigment was too large and contained fiber-like material even though the material had been processed through several grinding operations. Subsequent attempts to grind the material were unsuccessful. Apparently, the material is too amorphous and tends to fibrillate instead of fragmenting into small particles even under cryogenic conditions.

Hunt Chemical attempted to produce an EVA-based toner by spray drying an emulsion produced by Pierce and Stevens. The first attempt was unsuccessful because particle formation did not occur. Instead, the material coated the inside of the dryer. A second attempt was made using EVA emulsion loaded with wax, silica and pigment. Although spray drying resulting in the formation of small particles, the triboelectric properties of the EVA toner were such that very little charge was generated when the toner was mixed with various carriers. Generation of a developer system was thus not possible from the spray-dryed, filled EVA emulsion obtained from Pierce and Steven.

Toner could also not be produced from the standard Hycar 26120 by grinding due to the highly-amorphous nature of the material, and thus it was not given serious consideration as a xerographic toner in Phase I. During Phase II, Hunt Chemical Company proposed that a toner be produced from the spray drying. Hycar 26120, which is an emulsion containing complex acrylic polymers, was loaded with silica and pigment and spray dried. Samples of toner were produced; however, the complex acrylic resin proved to be a

thermoset material and decomposed before it melted. It was thus deemed not suitable as a toner resin in textile xerography.

One candidate toner was found by serendipity. Xerography printing was simultaneously being investigated as a method of pattern bonding nonwovens. Kativo, which is a modified epoxy produced by H. B. Fuller, was being tested as a candidate toner for binding nonwovens. Pigmented Kativo was available in a range of colors, the first tested containing phthalocyanine blue pigment. The triboelectric properties of the Kativo were such that sufficient charge was generated on its surface when it was mixed with carrier supplied by Hunt Chemical Company. A developer system was made using the blue Kativo by sieving it through a 325 mesh screen and mixing it with carrier. Red Kativo containing no phthalocynanine pigment was next used as the toner component of the The red Kativo contained two pigments, Naphthol developer. Acrylamide CI Red Pigment PR and Pyrazolone Orange CI Pigment PO34.

Continuous runs on the modified Xerox 3100 Copier were made using the blue and red Kativo toners. Single-color prints were made on fabric having a width of approximately 8-1/2 inches. Process speeds ranged from four to nine feet per minute. Runs lasting up to approximately 10 minutes were made. Samples produced during the optimized continuous runs were heat-treated for ten minutes in an oven at a temperature of 150°C. Crockfastness tests (AATCC Crockmeter Method Number 8-1981) were then run on the prints.

Crockfastness test results are summarized in Table 24. The dry crockfastness of the blue Kativo prints was excellent, with all

samples exhibiting having ratings of 5, but the wet crockfastness was not as good. Sample ratings for the wet crockfastness test ranged from 3 to 5 depending on the size of the printed area: the smaller the area, the higher the rating. The crockfastness ratings of the red Kativo toner were lower than those for the blue Kativo. The crockfastness ratings for dry specimens were 4, while ratings for wet samples range from 3 to 4. Although the wet crockfastness of the Kativo prints was not as good as would have been desired, the wet crockfastness ratings were as good as those for standard red and black screen prints from the textile supplier.

### PHASE III

Phase III involved scaling up to continuous 36-inch wide, three-color textile printing. One of the objectives was to demonstrate a xerographic process for printing 36-inch wide, polyester/cotton sheeting fabric. The prints were to contain three colors, with regions having single, two and three overlapping colors. The second objective was to continue development of a toner meeting textile requirements.

During the previous two phases of the project, Hunt Chemical Company had matched toner and carrier for the project. For Phase III, the decision was made to develop in-house capability for making electrical measurements of toner and carrier materials to arrive at optimum textile developer combinations. The following three measurement systems for characterizing toner were either built or purchased: 1.) electrical measurement apparatus for the evaluation of carrier materials; 2.) triboelectric apparatus to

Table 24.	WET/DRY CROCKFASTNESS TONER	TEST <sup>a.</sup> RESULTS WITH TEXTILES	XEROGRAPHY
Specimen	Dry Crock Rating	Wet Crock Rating	
Xerographic Prints Blue Kativo Red Kativo	5 4	3-5 3-4	
Screen Prints <sup>b.</sup> Red Black Blue	4 - 5 4 - 5 5	3 3 4 - 5	

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a. Tests were performed according to AATCC-TM-8-1981. b. Control obtained from a leading sheeting manufacturer.

evaluate the charge generating characteristics of toner/carrier combinations; and 3.) an impedance bridge and sample cell for dielectric measurements of toner and carrier candidate materials.

Since building a 36-inch wide copier in-house would have been very difficult and prohibitively costly, a survey was made to locate an existing machine of proper width that could be modified for continuous runs. Several 36-inch wide copiers were identified on the international market, but most were prohibitively expensive, costing from \$50,000 to more than \$100,000. One of the copiers, the optical-based Xerox Model 2510, was priced in the \$3000 to \$4000 range. An evaluation of the Xerox 2510 copier indicated that it could be modified and used for continuous printing of fabric. 2510 copier is a batch-type machine that can make The Xerox reproductions having widths up to 36 inches. Basically a blue print reproduction machine, it is best suited for printing lines. Solid areas are difficult to print with the Xerox 2510 copier, and some difficulty in printing dark, solid areas with the unit was anticipated. However, the Xerox 2510 was suitable for demonstrating the continuous xerographic printing of 36-inch wide fabric.

Since the Xerox 2510 is a batch-type machine for making onecolor prints, several modifications were necessary to allow continuous operation. Three of the machines were used in tandem in order to produce three-color prints. The Xerox Corporation cooperated in this phase of the project, providing assistance and advice in setting up and conducting the demonstration. A schematic of the set up is shown in Figure 22. Modifications were made to

facilitate continuous feeding of fabric through the copiers and to allow operator control of copier functions. In addition, takeup and let-off rollers were added to the arrangement.

The Xerox 2510 utilizes an optical system in copying an original. The initial system for making three-color prints used three original image designs made in continuous loops with one mounted on each machine, as illustrated in Figure 23. This arrangement proved unsatisfactory, as the pattern printed inconsistently. The images were friction-driven and slippage occurred. The loops could also not be made with exactly the same lengths, and the relative positions of the loops thus changed with run time.

The problem was solved by making one continuous loop image that passed through all three copiers, as shown in Figure 24. Slippage was not a problem since the image shifted as a unit. This arrangement proved satisfactory for the demonstration and for producing prints for textile testing; however, it would not be acceptable for a commercial machine. Commercial systems would be more complicated, with the images produced by lasers driven by computers. Problems involving color registration would have to be addressed, but should be solvable.

A Wild Macroscope was used to make an optically examine xerographic prints produced using paper and experimental textile toners. The xerographic prints were compared with screen prints produced commercially on the same sheeting fabric used for the xerographic prints. After making a straight cut of the 50/50 polyester/cotton sheeting fabric, slides of the top and side views



FIG. 22

CONTINUOUS 36-INCH WIDE XEROGRAPHIC PRINTING LINE



FIG. 23

INITIAL IMAGING CONFIGURATION



FIG. 24

FINAL IMAGING CONFIGURATION

of the fabric were taken. The magnification of the pictures varied from 25 to 50X. The slides illustrated the details on a microscopic scale of the nature of flow of toner materials over the fabric surface. Effects of fusion temperature and of crocking were also illustrated.

The primary materials used in this phase of the project were: paper toners compatible with the Xerox 2510; candidate toners for textile applications; carriers used to produce 2510-specific developer systems with the toners; and additives to enhance the properties of the developer systems. The paper toners (Xerox 1025 toner, styrene-acrylate copolymer resin) were provided by the Xerox Corporation and were compatible with the Xerox 2510 copier. Three colors of Xerox 1025 paper toners (red, blue and green) were matched with carrier to produce developer systems for testing. Since the paper toners had been developed for operation in the systems closely matching the Xerox 2510 copier, the required textile properties were not generated, but they were useful in demonstrating the feasibility of continuously printing fabric.

The materials considered as candidates for textile toners in this phase of the project included Kativo, the modified epoxy produced by H. B. Fuller, and two types of poly(ethylene-co-vinyl acetate) produced by U. S. Industries. Since pigmented Kativo was commercially available, it qualified as a toner if it could be matched with a carrier. Kativo with several different colors of pigments were tested with carrier for triboelectric characteristics. Three colors (red, blue and green) were selected for the Kativo® printing tests. The pigments contained in the

toners were: red toner-Naphthol Acrylamide CI Pigment #PR 170 and Pyrazole Orange Pigment #PO 34 (blend); blue toner-Iron Blue #27, CI Pigment #77510; and green toner-Iron Blue #27, CI Pigment #77510 and Diarylide Yellow #14, CI Pigment #21095.

The two U. S. Industries poly(ethylene-co-vinyl acetate) resins were considered, MU 760 and FE 532. The ratios of ethylene to vinyl acetate for the two materials were 82:18 and 91:9, respectively. These EVA materials were not commercially available with pigment, and samples containing pigment could not be obtained from the manufacturer. Since attempts to produce pigmented samples via melt blending/grinding and pigmenting emulsions followed by spray drying were unsuccessful (see earlier discussions), the decision was made to stain the samples using Cibacet Blue Disperse Dye to visually follow the print location. Although the fastness properties of the stained materials would not be meaningful, the tactile properties of the prints could be assessed.

Eight different types of carriers were tested during this phase of the investigation. These carriers, which were supplied by Hunt Chemical Company and the Xerox Corporation, had electrical resistivities ranging from low to high values. The low resistivity carriers are used to best advantage for area printing, whereas the high resistivity materials are well suited for line reproduction.

An additive, CAB-O-SIL<sup>R</sup>, was used to prevent the polymer particles from blocking and to enhance triboelectric charging. CAB-O-SIL<sup>R</sup> is fused silica having a chain-like structure that tends to keep the toner particles separated.

The substrates used for the 36-inch wide continuous

xerographic printing tests were rolls of paper and 50/50 polyester/cotton sheeting fabric. The paper was used to set up the three Xerox 2510 copiers in series to run continuously to assure the machines were functioning properly. Once the line was operational, the fabric was used with paper toners, and finally textile toners were incorporated.

A number of tests were run to characterize toner and carrier, the most useful ones characterizing the triboelectric properties. The results were used to assess the compatibility of different materials for attaining good quality xerographic prints. The test consists of placing a small sample (approximately three grams) of well-mixed toner and carrier in a small stainless steel cylindrical The mixing causes the toner and carrier to rub against each cage. other and become oppositely charged. The cage or holder has 400 mesh stainless steel screens at its end walls. The holes in the screens are large enough to permit the toner particles to leave the cage, but are small enough to prevent the carrier particles from When dry nitrogen is blown back and forth across the exiting. cage, the charged toner particles are blown away and the charged carrier particles remain inside. The outside of the cage is oppositely charged from the carrier. An electrometer lead attached to the support for the metal cage permits the recording of the electrical charge that has been stored on the cage as a result of the friction between the particles of toner and carrier.

The polarity of the photoconductor drum used in the Xerox 2510 copier is positive. Thus the desired polarity to be developed on toner for use with this copier is negative. The carrier

particles remaining in the cage after the triboelectric test will be positively charged, and the electrometer will indicate the magnitude of this charge. The results of the test is referred to as the triboelectric number, and its units are microcoulombs per gram of toner.

Four types of continuous xerographic tests were conducted: paper toners on paper, using Xerox 1025 toner (red, green and blue) with Xerox 2510 carrier; paper toners on fabric, using the same developer system as in the initial runs; Kativo® toners (red, green and blue) on fabric, using Kativo® mixed with Xerox 2510 carrier as the developer system; and FE 532 toner on fabric, using FE 532 tinted with blue disperse dye and mixed with Xerox 2510 carrier. The prints made during the first three types of tests contained three single colors as well as regions having two and three overlapping shades in all the possible combinations. The fourth test produced single-color prints.

Several tests were performed to evaluate the quality of the prints produced using xerography: wet/dry crockfastness; colorfastness to laundering; colorfastness to chlorine; colorfastness to dry cleaning; colorfastness to light; flammability ; and stiffness of print. All of the tests were performed on the Kativo prints, but the tests were not performed on the FE 532 prints since they did not contain pigment. The FE 532 prints were compared visually with the Kativo prints for print quality.

Wet and dry crockfastness tests were performed on the prints according to AATCC Test Method 116-1983. The amount of color transferred from the specimen under investigation was evaluated by

means of the AATCC Gray Scale for Staining.

The color fastness to laundering of the treated fabrics, when subjected to accelerated conditions of home laundering, was evaluated according to AATCC Test Method 61-1986. The test conditions of Test No. 3A were employed. The color change of the test specimens were evaluated using the Gray Scale for Staining and the Gray Scale for Color Change.

The color fastness to chlorine was evaluated using AATCC Test Method 61-1986. Test conditions were from Procedure No. 5A. The color change of the test specimens was evaluated using the Gray Scale for Color Change and the Gray Scale for Staining.

The color fastness of the treated fabrics to drycleaning solvent was evaluated according to AATCC Test Method 132-1985 with one exception: the steel disks were replaced by stainless steel balls. The effect of perchloroethylene on the color of the test specimen was classified by the AATCC Gray Scale for Color Change.

The color fastness of the prints to light was determined by AATCC Test Method 16E-1979 (Xenon light). The samples were exposed for 45 and 65 hours (because of no significant color change after 20 hours). The 65 hour exposure was the upper limit of testing. The black panel temperature was 117F, and the relative humidity was 15%. The results were rated by the Gray Scale for Color Change.

The flammability of the printed fabrics was evaluated by ASTM Test Method D1230-61. The time of flame spread was noted, as well as the number of fabric ignition attempts. The results were categorized according to three classes of flammability, defined in

the test method.

The tests to characterize the triboelectric properties of toner and carrier were useful in assessing the compatibility of toner and carrier. The Xerox 1025 paper and textile toner candidates were tested with a number of different carriers. Xerox 2510 carrier was selected for the printing tests, as it gave acceptable triboelectric numbers and was readily available. Table 25 results assess the potential for printing with commercial paper toners designed for the Xerox 1025 and Xerox 2510 copiers. The triboelectric numbers for these toners were sufficiently high that no charge enhancer was needed. The initial continuous runs were made using a composition consisting of 2% (by weight) of toner and 98% of carrier.

The triboelectric numbers for developer systems produced from red, green and blue Kativos mixed with Xerox 2510 carrier are given in Table 26. The effect of low concentrations of a charge enhancer, CAB-O-SIL<sup>R</sup>, on the triboelectric number is shown. The addition of CAB-O-SIL<sup>R</sup> at a level of approximately 1% was needed to obtain sufficiently high triboelectric numbers for successful printing on the polyester/cotton sheeting fabric. Higher levels of CAB-O-SIL<sup>®</sup> were not used because the triboelectric numbers were not appreciably changed with increasing concentration. Toner properties can also be negatively affected by high levels of additives. For the initial runs, the Kativo toners were sieved to a particle size of approximate 45 microns and mixed with CAB-O-SIL<sup>R</sup>. The developer system consisted of 2% of the toner mixture and 98% of the Xerox 2510 carrier.
Tests with the EVA toners revealed that  $CAB-O-SIL^R$  was needed not only as a charge enhancer, but also as an anti-blocking agent. Without the addition of  $CAB-O-SIL^R$ , the FE 532 and MU 760 materials blocked to the extent that sieving was impossible. When  $CAB-O-SIL^R$ was added, the material could be sieved and triboelectric tests performed. The triboelectric test results for the EVA toners with 1% additive are given in Table 27. The triboelectric number for FE 532 was very high with only 1%  $CAB-O-SIL^R$  added (+40.5). A higher level of additive was required to obtain a corresponding triboelectric value for the MU 760. FE 532 was therefore selected as the EVA material of choice for the textile xerography tests.

The initial continuous runs were made using the Xerox 2510 paper toners and 36-inch wide, white paper. The pattern definition was satisfactory; however, the depth of color of large solid areas was only fair (light shades). Solid areas are difficult to print with the Xerox 2510 copier since it was designed to be a blue print (line) copier, i.e., the toner delivery system on the copier was not designed to deliver large quantities of toner required for printing solid regions.

The next step involved using the paper toners to print on fabric. The pattern definition was acceptable, but the depth of shade again was only fair. In an effort to improve the depth of shade, the concentration of the paper toner in the developer mixture was increased to approximately 3% by adding toner to the developer tray. The depth of color was improved without a sacrifice in pattern definition (medium shades); however, results were not consistent from shade-to-shade. The depth of shade

# Table 25. TRIBOELECTRIC NUMBERS FOR XEROX 1025 TONERS WITH XEROX 2510 CARRIER

	<u>Triboelectric Numbers</u>
<u>Toner Color</u>	(µC per g)
Red	26.2
Blue	16.8
Green	22.2

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Table 26.	THE	EFFECT	OF	CAB-O-S	IL <sup>K</sup> C	N T	ΓHE	TRIBOELECTRIC	NUMBERS	FOR	KATIVO
	TONE	RS AND	XERC	X 2510	CARRI	IER					

Toner Color	Triboelectric Numbers (µC per_g) CAB-O-SIL <sup>R</sup> (%)						
	0	1.0	1,5	2.0			
Red	+4.1	+7.0	+6.5	+7.8			
Green	+2.3	+9.2	+2.9	+6.2			
Blue	+0.6	+8.2	+9.1	+6.7			

decreased in the order green, red and blue. Since this was the order in which the printing was conducted on the continuous line, it was hypothesized that the fabric was being dried during contact with the fuser drum, changing the fabric's dielectric properties. As a result, less toner was being picked up in subsequent copiers. However, in tests with moist air forced across the fabric between the copiers, the shade did not change significantly.

Microscopic examination of the printed areas indicated that increased deposition of toner would darken the shade. The toner concentration of toner was increased to approximately 4%, and a run made. The color was darker, but the inside of the copier was covered with toner. At this level of toner, the carrier was not able to control it, and considerable dusting resulted.

In the next series of tests, the Kativo toners were used to print on fabric. Based on the results with the paper toners, a 3% toner concentration was used. In the initial runs, large regions of heavy toner deposition resulted in streaking on the fabric, especially with the red Kativo. "Echo" images were observed as faint repeated images printed behind a designated pattern. In addition, excessive dusting of toner occurred in the inside of the copier.

The problems encountered with the Kativo toners were discussed with representatives of the Xerox Engineering Corporation, and they advised that the "echo" images observed in Kativo prints were likely caused by a toner fusing problem, known as "hot offset". This phenomenon occurs when the toner is not elastic enough to "snap back" to the substrate from the fuser drum. On subsequent

Table 27.	THE EFFECT	OF CAB-O-SIL	ON THE TR	IBOELECTRIC NUMBERS	FOR TWO U.S.
	INDUSTRIES	EVA TONERS ST	TAINED WITH	DISPERSE DYE	

		Tribo	electri <u>(μC pe</u>	c Number rg)	-S				
Toner		CAB-O-SI1 <sup>R</sup> (%)							
	0	0.5	1.0	1.5	2.0	5.0			
FE 532 MU 760	-	+2.7	+40.5 +5.6	+39,9 -	+18.8 +17.5	+38.2			

revolutions of the fuser drum, the toner left on it is deposited on the substrate, resulting in faint ghost images. Factors that can contribute to hot offsetting include: lack of oil release on the fuser drum; too high a temperature for the fuser drum; and poor elastic properties of the toner. Since the elastic properties of the toner could not be changed, efforts were made to adjust the oil release fluids and fuser drum temperature.

The wiping mechanism of the fuser assembly applies oil to the fuser drum to aid in the release of molten toner. Examination of the wiping mechanisms revealed that the wicks were coated with polymer, impairing lubrication. Replacing the old wicks with new wicks loaded with oil improved the short-term wiping performance of the mechanism, but toner quickly built up on the wicks, hindering subsequent wiping. Obviously, modifications to keep toner on the substrate would be preferable. Noncontact approaches to fixing the toner could be used; however, developing a new, indirect heating system for fixing toner on the Xerox 2510 was beyond the scope of the research project.

Another factor contributing to the offsetting problem was that the fuser temperature was set too high. The fuser roll temperature on the Xerox 2510 could be set between approximately 120 and 160°C. This was too high for Kativo toner which has a melting point of approximately 95°C. The Xerox 2510 copier was modified to lower fuser roll temperatures. For settings at and below 90°C, "cold offsetting" was observed where toner was not completely fused onto the fabric or the fuser drum. When the fuser temperature was increased above 100°C, hot offsetting worsened with increasing

temperature. The test results indicated that the optimal temperature for minimizing offsetting was about 100°C.

Since excessive dusting of toner inside the copier had occurred when toner concentration was 3%, carrier was added to the developer system to reduce the toner concentration to approximately 2%. Prints at this concentration were of better quality, but were lighter in depth. Increasing toner concentration back to 3% resulted in hot offset of toner and a return of the dusting problem. Although the carrier was able to control the paper toner at a concentration of 3%, it was unable to control the Kativo toner at the higher concentration. One possible reason was that the Kativo toner was not as well matched with the carrier as the paper toner, as indicated by the triboelectric numbers. Another was that the Kativo toner particles were too large. The Kativo toner had been sieved using a mesh screen with openings of approximately 45 The screen selection was based on information in the microns. literature indicating acceptable toner particle size ranged from 5 to 45 microns; however, according to xerography experts, the typical range of toner particle size is from 5 to 20 microns, with the distribution slanted toward the 5 micron end.

Samples of each of the three Kativo toners were sent to a laboratory for particle size analysis with a Coulter Counter. The mean particle size for the green, blue and red Kativo toners were 18, 32 and 51 microns, respectively. The percentage of the particles falling in the desirable range was 41, 17, and 6 for the green, blue and red toners, respectively. The particle size was indeed too large for good xerographic performance.

The toners were then air milled to reduce the particle size. Due to time constraints of the project and the unavailability of a mesh screen with openings smaller than 45 microns, the ground toner was collected and loaded into the copier. Evidently, the particle size had been reduced, since the prints exhibited better print quality and depth of shade. When toner concentration was increased to 3%, dusting of toner inside the copier was also reduced. The higher toner concentration gave darker prints.

After all the process development changes were made, a printing run was conducted with polyester/cotton fabric and the three colors of Kativo toner. Although the offsetting problem was never totally eliminated, three-color prints (including two- and three-color overlap prints) suitable for textile testing were produced.

The results of the textile property tests are summarized in Table 28. The flammability test results indicated that no significant differences existed in the flammability of the Kativo printed samples and unprinted fabric. The crockfastness results of all three of the Kativo toners were acceptable. When compared to control (screen printed fabric obtained from a leading sheeting manufacturer), the Kativo toners exhibited slightly higher ratings for the wet crockfastness test. The color fastness of the Kativo prints to laundering, chlorine and dry cleaning was excellent. With the exception of the green, the color fastness of the Kativo prints to light was acceptable, but inferior to the control. Since the red Kativo print was rated excellent after 65 hours of exposure, the problem with the green should be solvable by changing

the green pigment.

The results of flexural rigidity tests are summarized in Table 29. The stiffness of the Kativo printed fabric was much higher than the unprinted fabric, presenting a potential problem that should be further studied. Sufficient plant-printed control was not available for comparative flexural rigidity tests.

Since the triboelectric number for FE 532 (EVA) toner was very high with only 1% CAB-O-SIL<sup>R</sup> additive, it was selected over MU 760 toner for producing xerographic prints. The FE 532 toner, which had been stained using blue disperse dye, was loaded into a unmodified Xerox 2510 copier and prints made. Colorfastness tests were not performed on the prints because a commercial toner would contain pigment, not disperse dye, and thus the results would not be meaningful.

The FE 532 EVA toner xerographically outperformed the Kativo toner. Although the prints were light in color due to the small amount of disperse dye absorbed by the resin, the clarity and definition of the prints were superior to those produced using Kativo toner. The aesthetic properties of the prints were also qualitatively judged superior to the Kativo prints.

The results of the optical microscopic examination revealed that the wetting and penetration obtained with the Kativo and FE 532 toners were similar to those obtained with conventional printing. Photographs taken of the xerographic prints produced using paper toners showed that the individual toner particles, although adhering loosely to the fabric, do not wet the individual fibers. Similar results were obtained for several types of paper

	CROCKFASTNESS				CHLOR	INE <sup>c</sup>	DRY CLEANING			
SPECIMEN	DRY	WET	COLOR <u>CHANGE</u>	STAINING	COLOR CHANGE	STAINING	COLOR CHANGE	20 <u>,HR</u> ,	45 <u>HR</u> .	65 <u>HR</u> ,
KATIVO TONER										
RED	4	5	5	4.5	5	5	5	5	5	5
GREEN	4.5	5	5	5	5	5	5	5	3.5	3
BLUE	5	5	5	4.5	5	5	5	5	4.5	4
2-COLOR	4	4	5	4.5	5	5	5	5	4	4
3-COLOR	4	4	5	4.5	5	5	5	5	4	4
SCREEN PRINT										
BLUE	5	4.5							5.0	5.0
BLACK	4.5	3.0							5.0	5.0
RED	4.5	3.0							5.0	5.0

#### TABLE 28. RESULTS OF FASTNESS TESTS FOR PRINTS PRODUCED WITH KATIVO TONER

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 a.
 AATCC
 TEST METHOD 116-1983

 b.
 AATCC
 TEST METHOD 61-1986

 c.
 AATCC
 TEST METHOD 61-1986

 d.
 AATCC
 TEST METHOD 132-1985

 e.
 AATCC
 TEST METHOD 16E-1979

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produced by a leading sheeting manufacturer f.

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toners, including those for the batch Haloid system and three colors of Xerox 1025 toner. In these cases, no toner material penetrated into the interior of the fibers. Contrastingly, the wetting of both polyester and cotton fibers by the Kativo toners of all three colors was excellent. The toner material was observed on top of the fabric as well as in denoting penetration cut regions. With heavy toner concentrations, bridging of the spaces between fibers was also evident.

The FE 532 material, stained with disperse dye, also had excellent fiber wetting properties. The cross-sectional view indicated a complete penetration of the fibers. The thickness of the deposited material appeared to be less than for the Kativo toner.

The plant-printed control fabric, was also examined under the microscope. The wetting and fabric surface penetration was similar to that observed for the Kativo and FE 532 toners.

#### TABLE 29. STIFFNESS OF KATIVO PRINTS

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	FLEXURAL RIGIDITY (mg-cm)					
<u>Specimen</u>	<u>Mean</u>	<u>S,D,</u> *				
1-Color	354	.132				
2-Color	365	124				
3-Color	352	196				
Control	194	20				

\* The large deviations between values resulted from inconsistent coverage of Kativo patterns along the entire specimen length.

#### III. POWDER BINDING OF NONWOVEN FABRICS

The developmental research was conducted on a polyester nonwoven fabric marketed commercially as the outer surface of a mattress pad cover product. Currently, the material is chemically bound by a wet pad-nip-cure process utilizing a modified polyvinyl acetate-based resin supplied in emulsion form (7.3-7.5% SPU). The product, composed of extremely short polyester fibers, is formed by a proprietary entanglement process, and was coded C-9462. The fabric is formed/dried at one location, and then shipped in roll form approximately 250 miles to a second company location for chemical binding. Base weight of the greige fabric is 1.3 oz/yd<sup>2</sup>, while the stretched-down, bound fabric is 1.05 oz/yd<sup>2</sup>. Further details on the base fabric properties are contained in the initial Final Report submitted on DOE Contract No. DE-FG05-84CE40702.

During the same previous reporting period, two powder resins were identified as prime candidates for binding the nonwoven:

1. H. B. Fuller's IF-3237, Kativo Line - A modified epoxy, the IF-3237 flows like a true thermoplastic, but slow curing kinetics take place during the flow, effecting cross-links and molecular weight increases that render the final material a thermoset. The resin began flowing in the 90-100C range, and showed excellent wettability and junction-point migration on the individual polyester fibers during the flow phase.

Mechanical properties of the bound fabrics were not as high as those achieved with the second resin candidate, Eastman's FA-252.

2. Eastman's FA-252 - A modified polyester built around polyethylene terephthalate, the FA-252 is a true thermoplastic with a 120-150C fusing temperature and a higher-than-desired melt viscosity (4.8 x  $10^6$  Cps at 136C). The flow characteristics on the polyester fibers had been shown to be poor (probably because of the high viscosity), with the material giving statistically-random "spot welding" of the fiber cross-over points. However, higher mechanical properties of the bound nonwoven were achieved with the FA-252 than the better-flowing IF-3237 material.

In preparation for proof-of-concept trials, the C-9462 fabric was again run through the SOS process under previously-optimized line conditions to check reproducibility of the system. The Nordson Corporation Lab Spraygun Booth Assembly (20 in. width, Fig. 25) used in the developmental research was again employed, with run conditions set as nearly equal as possible to the earlier runs. SPU levels of 8.1 and 10% were utilized with the FA-252 resin.

The mechanical properties of the bound fabrics obtained in the repeat, optimized runs were uniformly lower than those achieved in the earlier trials (Table 30). After checking to insure that no variables had entered the process, that all optimized settings were properly incorporated, and after discussions with plant personnel, uniformity problems in the roll of greige nonwoven fabric supplied by the plant partner were suspected as the cause of the anomaly.



Fig. 25

Schematic of Nordson Spray Gun Booth Assembly



# TABLE 30.

REPEAT OF OPTIMIZED FA-252 BINDING TRIALS ON C-9462 NW FABRIC

SPU	SPU Cure Cycle		Fabric		Initial			Repeat	
Level	Temp. <u>(°C)</u>	Time <u>(sec)</u>	Direction	θ <u>(psi)</u>	E	Mod <u>(psi)</u>	θ <u>(psi)</u>	E	Mod <u>(psi)</u>
8.1	200	30	MD	1785	0.38	7 <b>38</b> 5	1256	0.35	6533
n	11	Ħ	CD	821	1.25	517	683	0.96	707
8.1	200	60	MD	1568	0.38	7750	1237	0.28	7952
	Ħ	ti	CD	750	1.2	568	553	1.0	627
10	200	30	MD	1526	0.26	7385	1350	0.28	7764
n	17	11	CD ·	586	1.12	383	578	0.86	755
Blank	200	30	MD	1496	0.42	4213			
n	tt	n	CD	610	1.3			<del>-</del> -	

As a first check of the nonuniformity theory, 19 fabric samples were taken from three different sections of a role of bound C-9462 (control) fabric from the partner's plant facility and tested for machine direction (MD) mechanical properties. The variation of average breaking load values for the three sets are detailed in Table 31. Surprisingly, the sets from the same roll varied as much as 5.6 lbs (56%). More detailed through-roll uniformity studies on both control as well as greige fabrics were deemed necessary by the data.

The 96 in.-wide control fabric properties were first tested every two yards down a total of 14 yards (52 feet) of a roll. Across the width, three samples were taken at each position: left selvage, center and right selvage. With each fabric sample, five break tests were conducted in both MD and cross-direction (CD) for a total of 10 breaks/sample, and 30 breaks/position in the roll.

The accumulated mechanical data, detailed in Table 32, includes the breaking strength in pounds force, along with the coefficient of variation (% CV) for the individual values. In four of the six positions, the fabric was weaker in the center than at either of the selvages. The % CV's were high in a number of cases, e.g., 14.8% and 14.0% values for 5-Right and 6-Center, respectively. The data showed conclusively that the plant-bound fabric did indeed possess substantial nonuniformity in mechanical properties down a fairly short section of roll.

Table 33 converts the breaking stress data from pounds to psi to provide easier correlation with the Table 30 values. In all cases, an average sheet thickness of 0.0089 in. was used to

#### TABLE 31.

## VARIATION OF BREAKING LOAD VALUES OF CONTROL FABRIC

Set #	Average Breaking Load
1	15.6
2	10.0
3	11.8

# TABLE 32.

				CD				
Position		Breakin	g	Breaki	ng	Brea	king	Breaking
2	#)	<u>Load</u>		<u>Strain</u>		Load	-	<u>Strain</u>
			CV		CV		CV	CV
		<u>(1bs)</u>	<u>(%)</u>		<u>(</u> %)	<u>(1bs)</u>	<u>(</u> %)	<u>(%)</u>
1.	Right	12.6	11.7	.3	16.64	3.16	20.4	1.12 7.5
	Center	9.56	9.1	.22	11.2	3.76	5.5	.96 9.3
	Left	11.62	9.8	.22	11.2	3.03	10.2	1.22 6.1
2.	Right	10.84	6.3	.2	4.3	3.3	10.6	1.26 4.0
	Center	9.62	4.0	.21	10.2	3.23	11.9	.93 13.6
	Left	12.12	6.4	.22	1.25	3.98	8.6	1.34 4.0
3.	Right	12.36	7.0	.21	10.6	3.28	15.9	1.13 15.2
	Center	9.68	5.3	.26	16.6	3.07	17.7	.98 15.4
	Left	12.68	4.4	. 2	0	3.06	10.9	1.15 9.7
4.	Right	14.4	5.0	.21	5.2	3.5	13.4	1.16 17.1
	Center	10.66	4.2	. 23	9.8	2.8	13.6	.91 15.7
	Left	13.32	10.8	.24	17.6	3.56	8.1	1.26 12.0
5.	Right	10.9	14.8	.22	10.6	2.96	8.8	1.08 16.6
	Center	10.96	5.2	.21	10.3	2.96	19.0	.98 8.5
	Left	11.68	5.2	.21	10.3	2.96	19.0	.98 8.5
6.	Right	9.3	9.1	.21	10.6	3.35	10.5	1.1 12.8
	Center	10.48	14.0	. 20	4.4	3.28	12.8	.98 19.6
	Left	12.2	1.2	.2	0	3.08	19.9	1.2 8.3

#### PROPERTIES DOWN A 14 YARD LENGTH OF CONTROL FABRIC ROLL

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#### TABLE 33.

## CONTROL FABRIC BREAKING STRESS VARIATION<sup>a</sup>

	Position <u>(#)</u>	<u>Breaking</u> MD <u>(psi)</u>	<u>Stress</u> CD <u>(psi)</u>
1	Right	1415.7	355.0
1	Center	1071.9	422.5
1	Left	1305.6	340.4
2	Right	1218.0	370.8
2	Center	1018.0	362.9
2	Left	1361.8	447.2 MAX
3	Right	1388.8	368.5
3	Center	1087.6	344.9
3	Left	1424.7	343.8
4	Right	1618.0 MAX	393.3
4	Center	1197.8	314.6 MIN
4	Left	1496.6	400.0
5	Right	1224.7	330.3
5	Center	1231.5	330.3
5	Left	1312.4	348.3
6	Right	1044.9 MIN	376.4
6	Center	1177.5	368.5
6	Left	1370.8	346.1

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calculate the breaking stress. The Table 33 variations indicated that the anomalies observed in Table 30 could easily have been due to nonuniformities in the two sections of roll fabric used in the optimized trials rather than process variation.

The question then arose as to the root cause of the nonuniformity in the plant-bound material: was it due totally to nonuniform properties inherent in the proprietary formation process, or to nonuniform application/final location of the chemical binder in the wet finishing process? The position variation study was repeated with the greige, unbound fabric, and the results recorded in Table 34. The fabric thickness used was a uniform 0.0071 in., reflecting the thinner cross-section of the substrate without binder film. Substantial variation was seen in the average break values, but the % CV's were not as high as observed in several cases in Table 33. By wetting the fabric and subjecting it to stresses on the tenter line in curing, weak points in the structure introduced during the formation process were apparently accentuated, producing even wider variations in the final mechanical properties of the plant product.

The through-roll uniformity studies thus revealed another possible advantage for SOS powder binding over the conventional wet process. The fabric is not wet out in the SOS process, and is subjected to much lower tensions in the dry state as it is propelled through the process (only enough tension is required to keep the fabric from sagging or buckling, i.e., a smooth surface must be presented to the spray gun pattern). Consequently, weaker sections of fabric originating in the formation process should not

#### TABLE 34.

Pos.	Breaking (lbs)	Load CV (%)	Breaking	Strain CV (%)	Breaking Stress (psi)	Breaking (Ibs)	Load CV (%)	Breaking	Strain CV (%)	Breaking Stress (psi)
1 ·	10.52	6.1	.46	11.9	1481.7	4.14	20.6	1.28	3.5	533.1
2	10.44	8.5	, .48	11.9	1470.4	4.88	6.1	1.25	4.6	687.3
3	10.86	9.1	.6	8.8	1529.6	4.18	15.7	1.34	8.5	588.7
4	11.04	1.9	.47	12.1	1554.9	4.42	17.6	1.26	7.1	622.5
5	11.52	11.6	.67	12.1	1622.5	4.52	7.8	1.2	0	636.6
6	9.82	5.8	.43	6.4	1383.1	4.85	10.8	1.33	7.23	683.1

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## TENSILE TESTING OF UNFINISHED NON-WOVEN FABRIC<sup>a,b</sup>

a. Stress values calculated with a fixed fabric thickness value of .0071\*.

b. Position to Position Variation - (Samples every 2 yards)

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be worsened in relative mechanical properties to the rest of the structure by the SOS process.

Also in preparation for the proof-of-concept trials to be conducted at Nordson Corp., the optimized IF-3237 spray gun trials were repeated to assess reproducibility. To determine the effects (if any) in curing of the resin in a batch oven that required opening of the door to insert the sample, precipitating a drop in environment temperature and injecting uncertainty as to when the material reached Tmax, both slotted and hinged-door ovens were utilized in the repeat study. The former was developed by cutting a rectangular slot in the oven door of sufficient size to slide in the embroidery hoop-mounted fabrics containing the sprayed-on, powdered resin. The opening was fitted with a spring-loaded flap door that allowed minimal heat loss as the fabric assembly was slid through the slot and onto a metal grate shelf in the oven, i.e., much as a pizza is handled in a commercial food operation.

Three SPU levels were utilized in the study: 3.8, 5.4 and 6.9%. The MD/CD mechanical properties are exhibited in Table 35, with properties from both the old (hinged oven) and new (slide slot oven) methods highlighted. Figures 26 and 27 visually compare the data from the old/new methods in both MD and CD directions. The results followed the same pattern as seen in the earlier optimized run in that the highest MD breaking strengths were obtained with the lower SPU (1466 psi, 3.8%). The highest MD/CD breaking strength data (1466/719 psi) compared favorably with the maxima achieved in the earlier research (1370/578 psi). The values for fabrics cured by the old/new methods gave no clear pattern with

#### TABLE 35. COMPARISON OF OVEN CURING TECHNIQUES FOR OPTIMIZED RUN OF IF-3237, SOS SPRAY-BOUND FABRIC

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	âver joe	STD FEV	AVEP43E	BTB DEV
S.8 X ELIDE CVEN MD	(465.95	-3,51	.33	.û4
3.8 % SLIDE OVEN SD	719.12	91.92	1,14	,04
THE A DIE OVEN YO	1443.77	74,43		.13
3.8 % GLD OVEN CD	197,95	17,48	1.20	.08
5.4 % SLIDE OVEN MD	: :379,E3	52,21		11 <sup>2</sup>
5.4 % SLIDE 0125 ID	_53,49	66.35	· 1.15	.Û£
E.A.N GID GVEN KQ	: 1354.77 :	45,57		, ð5
5.4 % 0-10 CMEN 10	: 579.07 :	89.65	1.12	.97
E.º X SLIDE OVEN MD	1518,28 	100.09	.21	.02
ELP N SLIDE OVEN CD	: 365.59	40.59	. 94 ;	.08
a, 9 % Olo dven Mi	: 1413.64 : :	93.87	.26	<b>,</b> (†4
5.9 % CID 3VEN Tr	519.22 1	124.01		, G9





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frequent reversals, and no solid evidence existed that the slide slot oven provided any advantages over the hinged oven.

The research team concluded at this point that the groundwork was complete for conducting large-scale, proof-of-concept trials on the SOS nonwoven binding process at Nordson's Amherst, Ohio facility. Constructed to simulate full-width textile processes, a brief schematic of the Nordson application line is shown in Figure 28. A full, 60 in. wide fabric was mounted on a wooden rectangular frame with staples (5 ft. x 7 ft.), and propelled through the spray booth by a screw drive system up to 200 yards per minute linear speed. Two spray guns were required to cover the full area, with the pattern spreading over a 30 in. diameter from each gun. After a "dead" area between the spray booth exit and oven entrance for slowing down the carrier (~12 ft.), the material entered a fivefoot zone heated by infrared (IR) quartz tubes.

Considerable attention was paid to the IR oven at the start of the trial, as all previous work had been conducted on batch, convection ovens, and the curing rate and uniformity differences between the two types of systems were unknown. Heating/temperature uniformity across the width and down the length of the IR oven was of special concern, as the system was controlled by the output of a single sensor located at the end corner of the rectangular box area. After replacing clogged fan air filters to insure a uniform flow of air around the oven box, and by making two passes through the unit and rotating the wooden carrier 180 degrees before the second entrance, reasonable uniformity of curing was attained across the width and down the length of each fabric sample. The

FIG. 28. SCHEMATIC OF NORDSON TEXTILE POWDER APPLICATION LINE



two passes, each consisting of a forward and reverse traversal through the heated zone, thus constituted four complete heating times for each fabric cure, and roughly simulated the total heating time experienced by the developmental fabrics in the GT small-scale research.

Sieve analyses of the materials to be used in the Nordson trials were performed by H. B. Fuller Co. personnel (Figures 29-32). Figure 29 shows that little of the medium grade of the FA-252 resin passed through the 200 mesh screen, with the bulk of it requiring 50-200 mesh range screens to filter through. Approximately 39% of the fine grade passed through the 200 mesh screen (Fig. 30), not as much as the blue-pigmented IF-3237 (Fig. 31), but within close proximity in terms of ground powder particle size range. The unpigmented version of the IF-3237, coded IF-1977, showed a much broader particle size distribution, with significant percentages of mass retained even on 40 and 50 mesh screens (12.8 and 22.34%, respectively, Fig. 32). The unpigmented material possessed a slight yellow, generic tint, a result of side reactions in the polymerization process building up a weak chromophore to visible light in the blue end of the spectrum.

The fabric roll supplied by the plant partner for the Nordson trials was the closest construction in production to that utilized in the two-year developmental study (coded C-5716, 67" wide, 500 yards length, base weight of 1.6  $oz/yd^2$ ). The binding runs were coded as:

"F" Series - Utilized the blue pigmented, regular grade of
 H. B. Fuller's IF-3237 modified epoxy resin.

# FIG. 29. SIEVE ANALYSIS OF MEDIUM FINENESS GRADE OF FA-252 POWDER



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Parcentage

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# FIG. 32. SIEVE ANALYSIS OF CLEAR (UNPIGMENTED) VERSION OF IF-3237 POWDER (CODED IF-1977)



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2. "E" Series - Utilized the medium fineness grade of Eastman's FA-252 modified polyester resin.

3. "EZ" Series - Utilized the fine grade of the FA-252 resin.
4. "FB" Series - A repeat of the "F" series, utilizing a two-spraygun arrangement in the booth.

5. "FB-S" Series - A repeat of the 5% SPU/200 ypm "FB" series runs.

6. "FY" Series - Utilized the "clear" (unpigmented) version

of the Fuller IF-3237 that exhibited a slight yellow tint. In the mainline series, application speeds of 150 and 200 yards per minute (ypm) were utilized, along with three SPU loadings (light, heavy and medium). Table 36 lists the various series, along with the percentage SPU of powder for each run and the average SPU for each series. Cure conditions were optimized for the initial "F" series at 30 ft/min speed through the three foot heating zone, four passes, with a Tmax of 384F and a 180 degree reversal of the carrier after one forward/reverse transversal of the zone (24 sec. total fabric dwell time in the IR oven). The area of treated fabric measuring from the edges of the wooden carrier was 59.5 in. wide x 83 in. long.

The first nine runs of the "F" (initial) series were used to zone into the proper line settings (air flow rates, delivery pump pressures, oven conditions, etc.). A one-foot cardboard square covered in black velvet fabric was tared and placed in the center of the fabric rectangle prior to each run, and the weight of the applied powder determined directly by weighing the square after the sled passed through the spray booth and before it entered the oven,

TABLE 36.	SOS NONWOVEN SPRAY BINDING SERIES AND AVERAGE SPU'S	

...

SAMPLE # COMPANY	ACTUAL PERCENT	SPEED YPM	AVERAGE PERCENT	NOTES	
10 F	11.1%	200	10.7%		
11 F	10.3%	200		:	
12 F	11.5%	200			
13 F	11.1%	i 200			
14 F	10.5%	200			
15 F	10.3%	200			
16 F	11.1%	200			
17 F.	11.5%	200			
18 F	10.1%	200			
21 F	9.3%	200			
25 F	7.3%	200	7.7%		
26 F	7.7%	200			
27 F	8.5%	200			
29 F	8.3%	200			
30 F	8.7%	200			
31 F	6.9/	200			
32 F	7.9%	200			
33 F	7.5%	200			
34 F	6.5%	200			
35 F	7.3%	200			
36 F	4.4%	200	4.8%		
37 F	5.2%	200			
38 F	5.2%	200			
39 F	5.0%	200			
40 1	4.4%	200			
41 1	4.8%	200			
42 1	4.27	200			
43 1	4.8%	200			
	5.0%	200			
45 F		200		approx. 54	
46 F	5.2%	150	4.5%		
47 F		150		approx. 5%	
49 F	4.4%	150			
48 1		150		approx. 57	
50 F	4.2%	150			
51 F	4.6%	150			
52 F	4.97	150			
53 F	4.67	150			
04 F	4.0/	150			
55 M	4.1%	150			
56 F	8.3%	150	7.7%		
57 F	8.2%	150			
58 F 🦯	7.2%	150			
59 F	6.8%	150		•	
<b>6</b> 0 F	8.4%	150			
SAMPLE #	COMPANY	ACTUAL PERCENT	SPEED YPM	AVERAGE PERCENT	NOTES
------------	-----------	--------------------------------	--------------	--------------------	-------
61	F	8.0%	150		
62	F	7.6%	150		
63	F	7.2%	150		
64	F	7 <b>. 0%</b>	150		
65	F	8.0%	150		
67	F	11.5%	150	10.8%	
68	F	9.5%	150		
69	F	11.17	150		
70	F 	10.5%	150		
71		10.97.	150		
72	r r	1 L = 1 /= 1 1 - 1 =/	150		
ت / ارت	r	1 I I I I I I 1 / S - EE 12	150		
74	F	10.0% 11.1%	150		
70	F	1 1 a 1/a 1/5 579/	150		
/0	Ľ.	d Var vi∕a	130		
1	E	10.7%	200	10.2%	
2	E	10.5%	200		
Э	Ε	10.1%	200		
4	E	9.3%	200		
5	E	9.7%	200		
6	E	11.1%	200		
7	E	9.9%	200		
8	E	9.9%	200		
9	E.	10.9%	200		
11	<u>t-</u>	°⊒ . 77.	200		
12	Ē	7.1%	200	7.7%	
13	E	7.3%	200		
14	Ε	7.0%	200	,	
15	E	8.3%	200		
17	E	8.2%	200		
18	E	7.1/	200		
19	E	7.9%	200		
20	E	7.5%	200		
. 21	E.	8.3%	200		
22	E	8.2%	200		
23	E	3.8%	200	4.7%	
24	E	5.8%	200		
25	E	3.8%	200		
26.	E.	5.2%	· 200		-
27	E	5.4%	200		
28	E	4.6%	200		
29	E	4.8/	200		
30	E	4.6%	200		
31	E E	5.0%	200		
32	E.	3.8%	200		

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SAMPLE # C		ACTUAL PERCENT	SPEED YPM	AVERAGE PERCENT	NOTES
33 E 34 E 35 E 36 E 37 E 39 E 40 E 41 E		5.8% 4.0% 4.2% 4.8% 5.0% 5.6% 5.0% 4.4%	150 150 150 150 150 150 150 150	4.7%	
42 E 43 E 44 E 45 E 46 E 47 E 48 E 49 E 51 E 51 E		4.2% 8.7% 7.4% 6.3% 8.2% 7.6% 8.0% 7.7% 8.1% 7.1% 7.5%	150 150 150 150 150 150 150 150 150	7.7%	
53 E 54 E 55 E 57 E 58 E 59 E 60 E 61 E		9.9% 10.1% 10.3% 10.9% 9.7% 10.3% 9.9% 9.5% 9.5%	150 150 150 150 150 150 150 150 150	10.0%	
1 E 2 E 3 E 5 E 5 E 8 E 9 E 10 E	Z Z Z Z Z Z Z Z Z Z Z Z Z Z	7.7% 7.1% 6.6% 7.7% 7.5% 6.9% 6.6% 6.9%	200 200 200 200 200 200 200 200 200 200	7.1%	
11 E 12 E 13 E 14 E	Z Z Z Z	5.2% 5.2% 5.6% 5.2%	200 200 200 200	5.4%	· .

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SAMPLE #	COMPANY	ACTUAL PERCENT	SPEED YPM	AVERAGE PERCENT	NOTES
15 16	EZ EZ	6.0% 5.4%	200 200		
17	EZ	5.2%	200		
18		5.2%	200		
20	E.2. E.7	5.0%	200		
20		س′⊥ ∎لب	200		
21	EZ	7.7%	150	7.9%	
22	EZ	8.7%	150		
23	EZ	8.3%	150		
24	EZ	7.0%	150		
25	EZ	7.7%	150		
26	EZ	8.1%	150		
27	EZ	7.7/	150		
28	EZ	7.4%	150		
29	L. Z	8.2%	150		
1	FB	4.5%	200	4.9%	
2	FB	5.2%	200		
Э	FB	5.0%	200		
4	FB	6.3%	200		
5	FB	4.4%	200		
6	FB	4.6%	200		
7	FB	3.5%	200		
8	FB	5.2%	200		
.Э	FB	5.0%	200		
10	FB		200	APPROX.	5%
11	FB	6.7%	200	7.6%	
12	FB	7.7%	200		
13	FB	7.3%	200		
14	FB	6.7%	200		
15	FB	8.3%	200		
16	FB	7.5%	200		
17	FB	8.9%	200		
18	FB	8.9%	200		
. 19	FB	7.5%	200		
20	FB	6.7%	200		
21	FB	10.0%	200	10.07	
22	FB	9.3%	200		
23	FB	9.5%	200		
24	FB	9.5%	200		
25	FB	9.5%	200		
26	FB	10.3%	200		
27	FB	10.9%	200		
28	FB	9.7%	200		
29	FB	10.3%	200		
30	FB	10.7%	200		

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SAMPLE #	COMPANY	ACTUAL PERCENT	SPEED YPM	AVERAGE PERCENT	NOTES
31	FB	10.3%	150	10.17	
32	FB	9.9%	150	1011/1	
āc	FB	10.3%	150		
34	FB	10.5%	150		
35	FB	9.7%	150		
36	FB	10.5%	150		
37	FB	9.7%	150		
38	FB	3.7%	150		
39	FB	9.3%	150		
40	FB	10.9%	150		
41	FB	6.7%	150	7.0%	
42	FB	7.7%	150		
43	FB	7.7%	150		
44	FB	6.7%	150		
45	FB	7.7%	150		
46	FB	6.0%	150		
47	FB	7.7%	150		
48	FB	6.3%	150		
49	F B	/.1%	150		
50	ΓB	6./%	150		
51	FB	5.0%	150	5.1%	
52	FB	4.8%	150		
53	F B F B	and a second	150	APPRUX.	5%
04 EE	F B E D	0.4% E 0%	150		
30 50	F B F D	U= 2/4 1 0*/	150		
57	r B F B	4.0/. 1 Q*/	150		
58	FB	4.0%	150		
59	FB	5.4%	150		
60	FB	5.8%	150		
61	FB-S	5.0%	200	5.3%	
62	FB-S	5.4%	200		
. 63	FB-S	5.6%	200		
64	FB-S	5.8%	200		
50	FB-5	4.5/	200		
65 67	r 6-3 50-0	5.47	200		
67 20	г Б-С ГЪ-С	니.47 전 전7	200		
	r Ding F Ding	+.+/. 5. CV	200		
70	FB-S	5.8%	200		
1	FY	6.8%	200	7.0%	
2	FY	6.5%	200		
3	FY	7.5%	200		• ·
4	FY	7.17	200		

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SAMPLE #	COMPANY	ACTUAL PERCENT	SPEED YPM	AVERAGE PERCENT	NOTES
5	FY	7.1%	200		
6	FY	6.5%	200		
8	FY	7.3%	200		
U 10		6./7. 7.7.	200		
10	l" Ť	/ • / /•	200		
11	FY	5.6%	200	5.5%	
12	F.Y	5.6%	200		
ت ا 1-1		J. 2/. 4 DV	200		
14		4.8%	200		
10		5.0%	200		
10	FV	5.6%	200		
19	/ I £≂V	5.27	200		
10	EV	5.2% 6.0%	200		
20	FV	5.0%	200		
EC.	1 1	0.0%	£ 97 W		
21	FΥ	9.7%	200	10.1%	
22	FY	9.3%	200		
23	FY	10.3%	200		
24	FY	10.5%	200		
25	FY	9.5%	200		
26	FY	8.9%	200		
27	FY	10.9%	200		
28	FY	10.3%	200		
29	FY	10.77	200		
30	ΓΥ	10.9%	200		
31	FΥ	7.5%	150	7.4%	
32	FY	7.3%	150		
33	FY	7.9%	150		
34	FY	6.9%	150		
35	FY	6.9%	150		
36	FY	6.9%	150		
37	FY	7.1%	150		
38	F Y	7.9%	150		
. යිත් දෙද	FY	8.5%	150		
40	F Y	7.97.	150		
41	łΥ	6.6%	150		
42	FY	5.4%	150	4.9%	
43	FY	5.9%	150		
44	FY	5.0%	150		-
45	FY	4.4%	150		
46	FY	4.8%	150		
47	FY	4.4%	150		
48	FY	4.8%	150		
49	FY	4.4%	150		
50	FY	4.8%	150		

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SAMPLE #	COMPANY	ACTUAL PERCENT	SPEED YPM	AVERAGE PERCENT	NOTES
51	FY	4.8%	150	ng mining mang sings sings lang same ang sings mang sings sings sings sings	
52	FY	10.3%	150	9.8%	
53	FY	10.1%	150		
54	FY	9.5%	150		
55	FY	9.3%	150		
56	FY	10.5%	150		
57	FY	10.0%	150		
58	FY	9.1%	150		
59	FY	9.1%	150		
60	FY	9.5%	150		
61	FY	10.9%	150		

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i.e., the sled was stopped in the "dead" region between the booth exit and the oven entrance, and the square removed and weighed. The accuracy of the method was questionable and did not adequately address powder SPU variation across the width of the web, but was the only mechanism available at the Nordson site for tracking the weight of added powder per unit area of fabric.

The three average SPU's achieved in the "F" series at 200 ypm were 4.8 (eight runs), 7.7 (nine runs) and 10.7% (ten runs). The standard nonwoven Strip Tear Test (ASTM D-1682) was used to determine the mechanical properties of the produced fabrics. Appendix 3. Figures 33-34 detail the averaged stress/strain plots for the three SPU levels in both MD and CD directions. In Fig. 33, only about 100 psi in tensile strength was gained by increasing from 4.8 to 7.7% SPU, with practically no change in progressing to the 10.7% loading. Mechanical properties in the CD were nearidentical for all three SPU's.

Higher average tenacities were achieved at the lower (150 ypm) speeds, as exhibited in Figures 35-36. SPU's achieved in this section of the "F" series were 4.5% (10 runs), 7.7% (10 runs) and 10.8% (10 runs). Properties improved with higher SPU, with the 10.8% loading giving the largest MD tenacity and modulus values and the lowest strain, the latter two indicating increased stiffness with added resin loading (Fig. 35). CD results were mixed, with again little difference in mechanical properties resulting from increased resin loading (Fig. 36).

Figures 37-42 directly compare the mechanical properties achieved at the three SPU levels and at the two linear line



#### FULLER COARSE-GRADE, 200YPM (M.D.)

FIG. 33. MD STRESS/STRAIN CURVES FOR "F" SERIES FABRICS OF VARIOUS SPU'S, 200 YPM (IF - 3237 RESIN)



FULLER COARSE-GRADE, 200YPM (CR.D.)

FIG. 34. CD STRESS/STRAIN CURVES FOR "F" SERIES FABRICS OF VARIOUS SPU'S, 200 YPM (IF - 3237 RESIN)



FULLER COARSE-GRADE, 150YPM (M.D.)

FIG. 35. MD STRESS/STRAIN CURVES FOR "F" SERIES FABRICS OF VARIOUS SPU'S, 150 YPM (IF-3237 RESIN)



FULLER COARSE-GRADE, 150YPM (CR.D.)

FIG. 36. CD STRESS/STRAIN CURVES FOR "F" SERIES FABRICS OF VARIOUS SPU'S, 150 YPM (IF - 3237 RESIN)



FULLER COARSE-GRADE (machine dir.)

FIG. 37. COMPARISON OF MD PROPERTIES AT LOW SPU AND TWO PROCESS SPEEDS (IF - 3237 RESIN)



FULLER COARSE-GRADE (cross dir.)

FIG. 38. COMPARISON OF CD PROPERTIES AT LOW SPU AND TWO PROCESS SPEEDS (IF - 3237 RESIN)



FULLER COARSE-GRADE (machine dir.)

FIG. 39. COMPARISON OF MD PROPERTIES AT MEDIUM SPU AND TWO PROCESS SPEEDS (IF - 3237 RESIN)



FULLER COARSE-GRADE (cross dir.)

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FIG. 40. COMPARISON OF CD PROPERTIES AT LOW SPU AND TWO PROCESS SPEEDS (IF - 3237 RESIN)



### FULLER COARSE-GRADE (machine dir.)

FIG. 41. COMPARISON OF MD PROPERTIES AT HIGH SPU AND TWO PROCESS SPEEEDS (IF - 3237 RESIN)



#### FULLER COARSE-GRADE (cross dir.)

FIG. 42. COMPARISON OF CD PROPERTIES AT HIGH SPU AND TWO PROCESS SPEEDS (IF -3237 RESIN)

application speeds. Figure 37 clearly shows an advantage at the 150 ypm speed in the MD direction for the lower SPU, a pattern that held to varying degrees at both higher SPU's (Figures 40 and 42, respectively). The two plots tended to converge in the CD.

Mullen burst pressure-to-rupture and pilling test results for all of the fabrics generated in the Nordson proof-of-concept trial are contained in Appendix 4. The Mullen Burst Tester employs a rubber bladder that forms a semi-hemisphere against the fabric sample as pressure from a working fluid builds against it, and the recorded value is the ultimate pressure required to rupture the fabric surface. A modified wash test procedure prescribed by the plant partner was used to assess pilling (5 rating: no pilling; 1 rating: very severe pilling, by ASTM D-3512 picture comparisons). As a baseline, the control (plant-bound) fabric exhibited a Mullen burst pressure (MBP) of 29.4 lbs, with a pilling rating of 5. The greige (unbound) fabric gave a MBP of 38.5 lbs (higher due to its stretchable nature in the unbound state) and a pilling rating of only 3.

The average MBP's were higher than the control for all the variations within the "F" series fabrics (42.8-44.6 lbs), but the average pilling ratings were also uniformly lower than the control (3.6-4.3). The highest average pilling rating was obtained with the 7.7% SPU at 150 ypm line speed. The MBP and pilling test results indicated the "softer" handle of the SOS-bound fabrics over the control, but also the looser binding of extremely short fibers within the fabric surface that generate pills.

In a similar fashion, the bound fabrics originating from the

"E" series of runs (medium fineness grade of Eastman's FA-252 resin) were evaluated as to mechanical properties (Figures 43-44). The 4.7% SPU level gave uniformly higher MD tenacities than the 7.7% SPU level, and the maximum tenacity achieved (~1400 psi) was lower than achieved in the developmental studies and with the control (1785 and 1752 (at 8% SPU) psi, respectively). The highest modulus (1860 psi) was also lower than that of the developmental fabric (7385 psi, again at 8% SPU). The CD stress/strain plots crossed, and showed little difference between the two SPU's. Further stress/strain plot comparisons for the various runs within the "E" series are contained in Appendix 5. The decision was reached to repeat the study at Nordson with the fine grade of FA-252 to determine if a smaller particle size distribution would improve ultimate properties.

"EZ" series of runs were conducted under the same The conditions as the "E" series above, but with the finer particle size distribution (compare Figures 29 and 30). Higher MD mechanical properties were achieved with the finer powder at both SPU's and line speeds (Figures 45-46). The ultimate tenacity was as high for the 5.4% SPU/200 ypm run as for the 7.9% SPU/150 ypm trial (~1550 psi), although the modulus was slightly lower. The fabrics were considerably more flexible than either the control or optimized developmental fabrics, with the highest modulus (2590 psi) corresponding to 11.5 and 35% of the moduli of the two compared fabrics, respectively. A factor that influenced the apparent moduli differences was the thicknesses of the initial research and plant trial fabrics, which were different but



EASTMAN SPRAYED at 200 YPM (M.D.)





EASTMAN SPRAYED at 200YPM (CR.D.)





EASTMAN FINE-GRADE (M.D.)

FIG. 45. COMPARISON OF MD PROPERTIES AT BOTH SPU'S AND SPEEDS (FINE FA-252 RESIN)



FIG. 46. COMPARISON OF CD MECHANICAL PROPERTIES AT LOW AND MEDIUM SPU'S (FA-252 RESIN)

necessarily included in the modulus calculation equation. As with the "E" series runs, little difference was observed in the CD mechanical properties with either speed or SPU changes (Fig. 46).

Mullen burst pressures were fine for both the "E" and "EZ" series of runs (Appendix 4), with average maxima attained at 10.1% SPU/200 ypm for the former (48.5 psi) and at 7.9% SPU/150 ypm for the latter (51.5 psi). All the averages were substantially above that of the control (29.4 psi), and were comparable to the optimized developmental fabric (42.4 psi).

Pilling test results were excellent for the "E2" series, running 4.8-5.0 on the average ratings at the three SPU levels (Appendix 4). The "E" series gave lower ratings (3.6-4.4 averages), in line with the "F" series. The smaller particle size of FA-252 utilized in the "E2" series again proved beneficial, with the statistical "spot welding" mechanism of the high viscosity resin operating more efficiently with a higher number of particles per unit area of fabric surface and a higher overall surface area of particles (and of subsequent resin melt pools) available. The more efficient utilization of powder resulted in tighter bonding of the fine, hair-like fibers at the fabric surface that normally roll up to produce pills.

In the next series of runs, "FB", the fine-particle IF-3237 modified epoxy resin was again used, but with a two-gun spray system in the Nordson booth. In the earlier series, questions had arisen as to the uniformity of powder deposition across the width of the booth and fabric, as high air pressures were required to expand the circular powder cloud radius so that the outer

circumference reached the edge of the fabric. Ten-run bursts were conducted at seven SPU/line speed combinations (Table 36). The final runs, which were a direct repeat, were coded as the "FB-S" series.

Figures 47-48 give the stress/strain plots for the three SPU levels utilized at 200 ypm line speed (4.9, 7.6 and 10%). The plots showed little difference between the 4.9 and 7.6% SPU levels, with the highest tenacity (~1100 psi) achieved with the lower level. Only slight property improvements were achieved in the CD plots with higher SPU levels (Fig. 48).

Figures 49-52 compare the mechanical properties of fabrics at similar SPU levels produced in the "FB" series of runs, but at the two linear line speeds. In the MD, fabrics produced at the 200 ypm speed gave higher tenacities than those from the 150 ypm speed runs, with a maxima of ~1150 psi (Fig. 49). As in the earlier runs with the IF-3237 resin, the lower SPU level (4.9-5.1%) proved optimum, with slightly lower properties obtained at the 7.0-7.6% levels (Fig. 51). As was often the case, the CD properties appeared to be independent of SPU level or line speed (Figures 50 and 52).

From Appendix 4, average Mullen burst pressures for the "FB" series fabrics ranged from 38.8-44.5 psi, well above that of the control and comparable to the optimized developmental fabric. Pilling test ratings averaged from 4.7-5.0, showing considerable improvement over the "F" series fabric results and demonstrating the more efficient short-fiber binding afforded by the two-gun arrangement and the finer particle size distribution.

FIG. 47. MD STRESS/STRAIN CHARACTERISTICS OF FABRICS PRODUCED AT THREE SPU LEVELS AND THE TWO-GUN ARRANGEMENT (FINE IF - 3237 RESIN) FB 200, 4.9/7.6/10%

MACHINE DIRECTION



FIG. 48. CD STRESS/STRAIN CHARACTERISTICS OF FABRICS PRODUCED AT THREE SPU LEVELS AND THE TWO-GUN ARRANGEMENT (FINE IF -3237 RESIN) FB 200, 4.9/7.6/10%

CROSS DIRECTION



FIG. 49. COMPARISON OF MD PROPERTIES FOR FABRICS BOUND AT LOW SPU LEVELS AND TWO LINE SPEEDS (FINE IF - 3237 RESIN)

FB 150, 5.1% VS. 200, 4.9%





## FIG. 50. COMPARISON OF CD PROPERTIES FOR FABRICS BOUND AT LOW SPU LEVELS AND TWO LINE SPEEDS (FINE IF - 3237 RESIN)

FB 150, 5.1% VS. 200, 4.9%

CROSS DIRECTION





FIG. 51. COMPARISON OF MD PROPERTIES FOR FABRICS BOUND AT MEDIUM SPU LEVELS AND TWO LINE SPEEDS (FINE IF - 3237 RESIN)

FB 150, 7.0% VS. 200, 7.6%





FIG. 52. COMPARISON OF CD PROPERTIES FOR FABRICS BOUND AT MEDIUM SPU LEVELS AND TWO LINE SPEEDS (FINE IF-3237 RESIN)

FB 150, 7.0% VS. 200, 7.6%





The final nonwoven binding trials were conducted with the unpigmented form of the IF-3237 resin (coded IF-1977 by H. B. Fuller Co.) that had a generic, slight yellow tint (see earlier discussion and Table 36). The resin was ground to specifications for the medium fineness grade of the FA-252 material (Figure 29), and the spray booth was converted back to the single gun arrangement so that a direct comparison in resulting fabric properties could be achieved between the "E" and "FY" series Figures 53-56 detail the MD and CD stress/strain products. behaviors of the medium- and heavy-SPU level fabrics produced at 150/200 ypm. The maximum average MD tenacity was achieved at the 7.4% SPU/150 ypm line speed conditions (~1150 psi), correlating closely with the results of the "FB" series with pigmented resin. Interestingly, the optimum speed at the higher (9.8-10.1%) SPU levels, but with a higher modulus and lower strain achieved at both line speeds, i.e., the fabric stiffened with higher SPU (Figure 55). The CD properties showed a moderate advantage to the 150 ypm speed in the 7.0-7.4% SPU range, but little significant difference in the 9.8-10.1% series (Figures 54 and 56). Further stress/strain plot comparisons for the "FY" series are shown in Appendix 6.

The Mullen burst pressures and pilling ratings for the "FY" series fabrics are compiled in Appendix 4. For bursts in which all the fabrics were tested, the MBP's ranges from 33.9-44.8 psi, with the maximum reading obtained at the 10.1% SPU/200 ypm conditions. Pilling ratings ranked from 3.8-4.3 on the average, below those achieved in the "FB" series with its dual-spraygun, finer-powder arrangement.

FIG. 53. COMPARISON OF MEDIUM SPU MD STRESS/STRAIN PROPERTIES OF "FY" SERIES FABRICS (IF - 1977 RESIN)

FY 150, 7.4% VS. 200, 7.0%





FIG. 54. COMPARISON OF MEDIUM SPU CD STRESS/STRAIN PROPERTIES OF "FY" SERIES FABRIC (IF - 1977 RESIN)

FY 150, 7.4% VS. 200, 7.0%

CROSS DIRECTION



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FIG. 55. COMPARISON OF HIGH SPU MD STRESS/STRAIN PROPERTIES OF "FY" SERIES FABRICS (IF - 1977 RESIN)

# FY 150, 9.8% VS. 200, 10.1%

MACHINE DIRECTION



FIG. 56. COMPARISON OF HIGH SPU MD STRESS/STRAIN PROPERTIES OF "FY" SERIES FABRICS (IF - 1977 RESIN)

FY 150, 9.8% VS. 200, 10.1%

CROSS DIRECTION


The final comparisons of stress/strain properties were made between the FA-252 and IF-3237 resin groups (Figures 57-60). Run conditions, SPU, particle size distribution, spraygun arrangement, etc., were held as constant as possible between these series, where the only major variables were the binder chemical composition and In Figures 57-58, the MD/CD stress/strain linear line speed. properties of the "FB" and "EZ" series fabrics (both sprayed with fine powder distributions at 5% SPU, the "FB" series at 150 ypm and the "EZ" series at 200 ypm) are directly compared. The Eastman FA-252 gave substantially higher tenacities in both fabric directions, as had been observed in the developmental research, with a slight advantage in added strain-to-break. The ~1100 psi tenacity in the CD was >3X that found for the control (plant-bound) fabric (382 psi), and topped the optimized developmental fabric's value by ~270 psi (821 psi). Coupled with its superior average Mullen burst pressure and pilling ratings (Appendix 4), the "EZ" series process proved superior to any conducted during the Nordson proof-ofconcept trials, even at the maximum 200 ypm linear line speed.

In Figures 59-60, the stress/strain behaviors of the "E" and "FY" series fabrics (medium fineness grade powders) are compared at the 8% SPU level, but with the Eastman resin applied at 150 ypm and the H. B. Fuller material at 200 ypm. Again, the FA-252 modified polyester significantly outperformed the IF-1977 (unpigmented IF-3237) modified epoxy in both fabric directions. Further stress/strain plot comparisons are made between the "E" and "FY" series in Appendix 7.

In all cases in the Nordson trial, fine powders outperformed

FIG. 57. DIRECT COMPARISON OF MD STRESS/STRAIN PROPERTIES FOR "FB" AND "EZ" SERIES FABRICS (5% SPU)

FB VS EZ, 5% PICK-UP

MACHINE DIRECTION



FIG. 58. DIRECT COMPARISON OF CD STRESS/STRAIN PROPERTIES FOR "FB" AND "EZ" SERIES FABRICS (5% SPU)

FB VS EZ, 5% PICK-UP

CROSS DIRECTION



FIG. 59. DIRECT COMPARISON OF MD STRESS/STRAIN PROPERTIES FOR "E" AND "FY" SERIES FABRICS (8% SPU)



MACHINE DIRECTION



FIG. 60. DIRECT COMPARISON OF CD STRESS/STRAIN PROPERTIES FOR "E" AND "FY" SERIES FABRICS (8% SPU)



CROSS DIRECTION



the medium fineness grades. The data confirmed the theory that a higher population of resin particles per unit area of fabric would lead to more efficient binding and better macropore penetration, and that the higher surface area of those finer particles would also result in better fiber wettability/junction binding.

#### IV. FLUOROPOLYMER FINISHING OF A POLYPROPYLENE NONWOVEN

The Year 2 developmental research in the area of applying powdered fluoropolymer finishes to a commercial polypropylene nonwoven to impart barrier properties in the final product (surgical gowns, drapes and covers) had ended with attempts to utilize the same material incorporated in the plant process, coded L-9XX2. Supplied by the 3M Co., the finish exhibited a high fluorine content with both repellency and soil release properties. The product was available as a white, fluffy powder which smoothed out after blending with 1% fumed silica. A proprietary additive was included in the base finish supplied to the plant partner, and the form of utilization in the commercial process was emulsion, with the application mode wet pad-pressure nip-cure. Physical properties of the fluoropolymer (FP) were:

> Tm = 71C (158F) Particle Size = 12-130 microns Largest Fraction = 60-80 microns Adhesion = 99% on Mylar film

A potential problem had arisen in use of the plant FP in a SOS mode in that the isolated powder was hydrophilic in nature, causing it to clump and readily aggregate when exposed to air. Air mill grinding was subsequently inhibited by the inability to feed the material into the mill before clumping took place.

The L-9XX2 FP was air mill-ground and vacuum dried at 24 in. Hg and 50C. CAB-O-SIL, a commercially-available fused silica flow

modifier, was added in 1%, 2% and 3% by weight increments to three portions of the FP, and fluidization of the powder was attempted in the six inch, square cross-section, Nordson feed bed. The degree of fluidization, which was determined qualitatively by both visual and tactile methods, was attempted both with and without vibration of the powder bed to assist dispersion of the powder. The 2% level of CAB-O-SIL was optimum for bed fluidization, and vibration was found to be essential.

A larger batch of the air mill-ground L-9XX2 FP was blended with 2% by weight CAB-O-SIL, and the final powder was fluidized with dry air for four days at 10 psi pressure. The FP fluidized much better after drying, but did not spray well when attempts were made to feed the fluidized material through the delivery gun of the pilot scale spray booth (see Figure 25). The powder tended to exit the spraygun very unevenly and in short bursts. A higher delivery air pressure was required than had been utilized in any of the nonwoven binding research (see Section III), and inspection of the spray nozzle and booth after spraying revealed that the tacky FP powder had formed a tenacious layer of stuck material on both. Based on the difficulties encountered in grinding, fluidizing and spraying the L-9XX2 plant FP, the decision was reached to eliminate it as a candidate for the proof-of-concept trials planned for the Amherst, Ohio Nordson Co. site.

The next FP candidate selected for study on the polypropylene substrate was coded FC-214, another 3M product that had performed well in earlier SOS carpet finishing trials. Normally applied to nylon upholstery and drapery fabrics, FC-214 was offered

commercially as a 30% solids emulsion. The formulation possessed the unique capability of precipitating out the FP as a solid when ethanol was added to the emulsion. The light tan, solid material was grindable to a fine, free-flowing powder, exhibited a Tm of 100C and a cure temperature of >150C, and produced a clear, colorless film with good drycleaning and light fastness properties.

As in the developmental research, the standard 3M kit test for surface oil repellency was utilized to determine the effectiveness of the FP barrier (Appendix 8). To develop a comparison base, the kit test was utilized to evaluate a section of plant fabric finished with the L-9XX2 FP. The top (outer) surface of the fabric passed through Solution Kit No. 8 of the 3M test, and the bottom (inner) surface passed Kit No. 7. The standard 90% isopropanol drop test, utilized on barrier fabrics to assess penetration of this common hospital chemical, gave a 3-4 rating on the plant control (5 rating max, see Appendix 9 for test procedure). The greige (unfinished) base polypropylene fabric failed both the oil and isopropanol drop tests, with complete penetration of the surface as soon as the Kit No. 1 oil or alcohol touched the fabric.

After cryogenic and air mill grinding of the isolated FC-214 material (0-75 microns particle size range), a number of continuous optimization trials were conducted at Georgia Tech on the 20-inch pilot Nordson spray booth system (single spraygun, no electrostatics employed). The sought SPU was 2%, which was in the range of the loading achieved in the plant process (1.7%).

The drop test results from the FC-214 optimized pilot runs are shown in Table 37. A curing temperature of 160C was defined, which

## TABLE 37.

# OIL AND ALCOHOL DROP TEST RESULTS FOR PLOT SCALE FC-214 RUNS

FINISH APP.	SPU (%)	CURE T/t (°C/min.)	KIT RATING	ISO7 RATING	ISO9 RATING
1-SIDE	2	160/2	5	0.0	0.0
11	2	160/3	6	0.0	0.0
11	2	160/4	6	5	0.0

was near the softening point of the polypropylene substrate fabric. A maximum 3M Kit Test of 6 was achieved, with 70% isopropanol rating only achieved after four minutes at the curing temperature (a 5). No 90% isopropanol ratings were achieved with the FC-214 FP under the optimized conditions.

Scanning electron microscopy was employed to visually ascertain the location and film formation characteristics of the FP under melt flow conditions. As a baseline, Figure 61 shows the unfinished nonwoven base fabric from the top (outer) view, while Figure 62 exhibits the plant control fabric finished with the emulsified L-9XX2 FP. In the magnified view of the control fabric, the FP film is evidenced by a crinkled surface nature, reminiscent of a capillary pattern in skin. Figure 63 visualizes the nonwoven fabrics from the 3 min/160C run of Table 37, while Figure 64 exhibits fibers from the 4 min/160C optimized pilot runs. Whereas good flow was achieved at the three minute cure time, the finish film was smooth, not crinkled as observed with the plant control. As seen clearly in the high magnification views of Figure 64, however, the crinkled, capillary-like film appearance was generated at the four minute cure time, which was also the point of maximum drop properties. Good fiber diameter coverage of the film was evident, as was cross-fiber bridging and migration to junction points (see last photograph of Fig. 64).

Based on the pilot scale runs, the decision was made to enter full scale, proof-of-concept trials with 3M's FC-214 FP at the Nordson test facility in Amherst, Ohio (see Section III for details of the test line, spraygun arrangements and facility). With the



FIG. 61

TOP VIEW OF UNTREATED (GREIGE) NONWOVEN FABRIC HEAT TREATED FOR TWO MINUTES AT  $156^{\circ}$ C.





FIG. 62. TOP VIEWS OF PLANT-FINISHED CONTROL FABRIC (L-9XX2FP)





FIG. 63

TOP VIEWS OF FABRIC FROM PILOT SOS PROCESS (FC-214 FP, 160°C/3 MIN. CURE)





# FIG. 64.

TOP VIEWS OF FABRICS FROM OPTIMIZED PLOT SOS PROCESS (FC-214 FP, 160°C/4 MIN. CURE)





# FIG. 64 - (CONT.)

TOP VIEWS OF FABRICS FROM OPTIMIZED PLOT SOS PROCESS (FC-214 FP, 160°C/4 MIN. CURE)

fine powder necessary to achieve a continuous film (0-75 microns range), the two spraygun arrangement was selected, with target cure conditions of 160C/2-4 min identified. Speeds of 150 and 200 ypm were again established goals, with SPU's of 1-1.75-2.5% sought. A 3M Kit Rating of 6-7 was targeted, coupled with an isopropanol 70% (ISO7) rating of 4-5 and an isopropanol 90% (ISO9) of anything above zero.

Curing the FP with the Nordson IR oven versus the convection oven used in the developmental research was again of concern. The "cure" of a FP finish does not involve chemical reactions, but rather an alignment of the molecule with the fiber surface such that the low surface energy, perfluorinated "tail" of the finish is pointed away from the interface (Fig. 65). Although the SEM's of the plant control and FC-214 fabrics showed similar film characteristics, they revealed nothing about the molecular alignment of the two FP's. Concern also arose from the inability to generate barrier properties on the pilot fabrics to ISO9 in that water deposition of the finish might facilitate the "tail" alignment versus melt film formation. However, literature in the area indicated that in the tenter frame cure of the emulsion formulation, the alignment does not occur until the water has been evaporated and the fabric/film approach the maximum temperature of the oven (2).

The fabric supplied by the plant partner had the following characteristics:

Roll	Label:	801-	-938635	-00
Base	Weight:	1.8	oz/sq.	yd.

#### FLUOROPOLYMER ACTION



CRITICAL: "TAIL" ALIGNMENT TO SURFACE

FIG. 65

PROPER ALIGNMENT OF FP TO FIBER SURFACE AFTER "CURE"

Width:	63 in.
Length:	1700 yds.
Composition:	100% Polypropylene
Process:	Web Entanglement/Thermal
	Bonding
Outlet:	Hospital Gowns, Drapes and
	Covers

The supplied fabric had a different pattern embossed in it by the heated thermal bonding roll, containing 156 small circles of partially melted area of fabric per square inch. By contrast, the development trial fabric exhibited a diamond-shaped, thermal bonded pattern of 200 diamonds/square inch. When the plant partner was questioned on the difference in thermal bonding patterns of the two fabrics, the research team was informed that both types of fabrics were routinely interchanged on the company's FP line with no change in barrier properties. Although the patterning and depth of cavity produced by the thermal bonding rolls in the two fabrics did not affect the performance of the wet process, it was subsequently discovered that they indeed influenced the distribution of meltflowable FP, and had a marked effect on the efficiency of the SOS spraygun process to generate the proper barrier properties.

Figure 66 details the particle size distribution of the ground FC-214 FP lot of powder used for the Nordson trials. The powder was classified as a fine grade, approximating the distributions for the resins utilized in the "EZ" and "FB" series of runs in the earlier nonwoven binding trials (see Section III).

A number of bursts were conducted through the SOS process at

i tartari <u>a</u>c \_ 30 -70 -50 -考〇 --34.73 30 -2).49 Ø 20 -7.44 7.05 \_\_\_\_\_ JQ ---3.79 39 p T 2.04 227 113 127-FD ť 5 ਼ 中日 Ð i. 76 20 25 30 <u>\_7</u>] -**1**-7 Ci() É ]=[ ] 200 270 500 Mesh Screen

SIEVE ANALYSIS OF THE FC-214 POWDER USED IN THE NORDSON FP TRIALS

FIG. 66

o .52 ₫/oc

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200 ypm, varying SPU and IR oven temperatures (Table 38). The 3M Kit Test was used as a quick screening mechanism to determine if any barrier properties had been generated. The eventual test results were inconsistent (Table 38), but several critical factors were discovered during the initial runs:

1. The poor cross-width control of the IR oven temperatures created difficulty in running the polypropylene fabric, with melting in certain spots evident at emitter temperatures >1095F and fabric temperatures of >260F.

In an early run, a too-low SPU was achieved, and to avoid 2. sacrificing the fabric sample and remounting a second one, the carrier was reversed and brought back into the spray booth from the "dead" area of the line, and an air gun was used to blow off the loose, unmelted powder. The carrier was then moved back to the starting section of the line, and propelled once again through the booth with the spraygun operating. The proper SPU was achieved, and after curing, higher kit test results were obtained. The theory was developed that force of the compressed air stream used to blow off the fabric had pushed finer powder particles down into the channels of the circular thermal bonding sections, allowing film flow throughout the channel. Microscopic examination of areas of earlier fabrics treated by the kit test revealed that failure (drop penetration) had indeed occurred at the thermal bonded points of the surface.

3. At the suggestion of 3M researchers who attended the trials as observers, sections sufficiently large to cover an

### TABLE 38.

### TEST PERFORMANCES OF FABRICS FINISHED UNDER NON-OPTIMIZED CONDITIONS (FC-214 FP)

RUN	UN SPU			ISOPROPANOL			
<b>#</b>	<u>.gms/ft<sup>2</sup></u>		<u>MAXIMUM KIT #</u>	<u>70</u> %	90%	BLOW OFFS	
2	.13	2.3	3	-	-	0	
3	.13	2.3	0	-	-	0	
4	.12	2.1	1	0	0	0	
5	.14	2.5	0	-	-	0	
6	.16	2.8	3	5	4	1	
7	.18	3.2	0	-	-	0	
8	.16	2.8	4	-	-	1	
9	.18	3.2	1	4	4	0	
10	.4	7.1	2	4	0	0	
11	.14	2.5	4	5	4	2	
12*	.14	2.5	< 3	0	0	0	
13	.15	2.6	4	-	-	0	
14	.15	2.6	5	5	5	2	
15	.15	2.6	0	0	0	0	
16	. 23	4.1	4	4	0	1	
18	.15	2.6	2	0	0	1	
22	.17	3.0	3	5	4	2	
23	. 14	2.5	1	0	0	1	

embroidery hoop were cut from the center of the fabrics after the standard double transversal of the IR oven by the carrier with 180 degree reversal (see Section III), and the mounted fabrics under slight tension (just enough to prevent sagging) were treated for an additional 10 minutes at 145F in a slotted convection oven. Immediately, higher kit test results were obtained, along with the beginnings of isopropanol barrier properties (Runs 14 and 22 of Table 38).

From these discoveries, an optimum FP SOS application process was developed as summarized in Table 39. An air knife placed across the width of the moving fabric just after the spray booth and/or a vacuum slot in the same line vicinity would have probably accomplished the same thermal bonding well penetration as the blow off procedure, but neither was available at the Nordson site, and project time did not permit retrofitting the line with these devices to test the theory.

A number of runs were then conducted under the optimized conditions, and Table 40 details the post curing and drop test procedures used to evaluate the resulting fabrics' barrier properties. Table 41 gives the actual test data. Five drops were added across the fabric samples from each 3M Solution Kit, and the number of droplets that showed no penetration are indicated under the 5-8 Kit Number columns. In the final four runs of the Nordson trials, a few drops were not penetrating even with Kit Number 8, and consistent "5" ratings were obtained in both the ISO7 and ISO9 drop tests. Although the SPU's were higher than targeted, running approximately 1% greater than that of the plant control, the

#### TABLE 39.

OPTIMIZED FP SOS APPLICATION PROCESS (FC-214)

OPTIMUM FLUOROPOLYMER APPLICATION

THREE SPRAY APPLICATIONS OF POWDER, FOLLOWED EACH TIME BY BLOWING POWDER OFF THE FABRIC WITH COMPRESSED AIR GUN

ONE FINAL SPRAY APPLICATION OF POWDER

PASSED THROUGH IR OVEN AT Tmax ON READOUT OF 260°F

A. 180° ROTATION OF FRAME

B. TWO PASSES THROUGH IR OVEN

POST-CURE: TEN MINUTES IN CONVECTION OVEN AT 145°C

•

#### TABLE 40.

POST CURING AND DROP TEST PROCEDURES USED WITH FABRICS FABRICS FROM OPTIMIZED RUNS (FC-214 FP)

- 1. Three sample squares were cut from each side & the center of the fabric from each finishing run.
- 2. The sample squares were placed in 9 inch embroidery hoops.
- 3. The samples were individually cured in an oven for 10 min. at 145 degrees C.
- 4. The samples were tested for surface oil repellency (3M Kit Test, TAPPI UM 557) and resistance to low surface tension liquids (isopropyl alcohol test)
  - 3M Kit Test: Five drops of each kit # solution were placed on the cured test specimens & allowed to stand for 15 sec. The drops were then swabbed, and the # of drops that remained on the fabric surface were recorded.
  - Isoprop Test: One drop each of 70% & 90% isopropyl alcohol was placed on the samples and allowed to remain for 5 min. The alcohol was then swabbed off and the remaining spot (if any) rated.

### TABLE 41.

### DROP TEST RESULTS FOR FABRICS GENERATED FROM THE OPTIMIZED SOS PROCESS (FC-214 FP)

SPU				K	it #	Isopr	Isopropanol	
<u>Run</u> #	<u>gms/ft</u>	<u> </u>	<u>5</u>	6	7_	8	<u>70</u> §	<u>908</u>
1	.14	2.5	5	5	5	0	-	-
19	.16	2.8	5	5	1	0	5	4
21	.16	2.8	5	5	2	0	4	0
24	.15	2.6	5	4	ο	0	5	0
25	.15	2.6	4	4	1	0	5	3
26	.15	2.6	5	5	1	0	5	5
27	.15	2.6	5	5	2	0	5	5
28	.14	2.5	5	5	0	0	5	4
29	.14	2.5	5	5	5	1	5	5
30	.17	3.0	5	5	5	2	5	5
31	.16	2.8	5	5	5	1	5	5
32	.17	3.0	5	5	5	1	-	-

consistency and level of barrier properties achieved with the FC-214 FP by the SOS powder spraygun process were outstanding.

The trial demonstrated for the first time that superior oil and isopropanol repellency properties could be imparted to a 100% polypropylene nonwoven utilizing a continuous, high-speed FP powder application/melt cure process. Although cumbersome due to the blowoff requirement, the SOS process demonstrated the potential for FP finishing using a waterless approach, with more sophisticated engineered processes possible to facilitate continuous processing at high linear speeds.

#### V. LIQUID SPRAY COLORATION OF POLYESTER/COTTON SHEETING FABRIC

Developmental research had determined that solid shade coloration of textile fabrics could not be achieved by spraygun application of a powder consisting of melt blended pigment/binder (1). Attention was then focused on developing UV-curable liquid resin/pigment systems of proper viscosity (<600 cps) to operate in the Nordson Corporation's electrostatic liquid spraygun system. A schematic of the Nordson assembly is shown in Figure 67. The electrostatic capability was a necessary feature of the Nordson system, as the like charge placed on the fine spray droplets prevented agglomeration into larger spheres via repulsion, and thus avoided spotting of the fabric.

A continuous, motor-driven belt system was built at Georgia Tech to allow transport of the 50:50 polyester/cotton sheeting fabric, mounted on a nine-inch diameter embroidery hoop under sufficient tension to prevent sagging, to traverse the booth and pass under the single spray gun at various linear speeds. By altering the spray gun control system setting and the belt speed, the amount of resin/pigment deposited per unit area of fabric was controlled.

The resins were cured to form the final film by passage through a Argus International Model PP-7106 Conveyorized UV Processor (1). The unit contained two mercury vapor UV lamps of

## FIG. 67

## NORDSON LIQUID SPRAY BOOTH SYSTEM



- 1. spray booth hood
- 2. spray gun/nozzle
- 3. conveyer assembly
- 4. heater
- 5. electrostatic system
- 6. piston pump
- 7. main air supply
- 8. fuild filter
- 9. fluid pressure gauge
- 10. hood exhaust

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. Figure 68 details the UV exposure dosage per unit area of fabric as a function of speed of transversal through the Argus unit. The investigated films and subsequent mounted fabrics that had been sprayed were passed through the oven as many times as necessary to fully cure (harden) the resin.

Based on the initial research (1), developmental work continued on identifying a resin/monomer diluent/photoinitiator system that gave a UV-curable, transparent film that was similar in mechanical properties to that of the selected standard resin, B. F. Goodrich Company's Hycar 26120. This resin, a complex acrylic that produces thermoset, highly amorphous, transparent films, is commonly used in wet screen printing operations as a flexible pigment binder of excellent aesthetic and fastness properties (see Section II). The stress/strain plot of a Hycar 26120 film is exhibited in Fig. 21.

After screening a number of commercially-available materials that possessed UV-sensitive functional groups, the research concentrated on two oligomeric products: Lord Corporation's Photoglaze U0283 and Interez Corporation's Novacure 6700. Photoglaze was an aliphatic urethane-acrylate segmental copolymer, while Novacure was an aromatic analog. Table 42 gives properties of the resins and their films.

Five diluent monomers were identified, and their chemical natures and properties are exhibited in Table 43. The monomer



# FIG. 68

FABRIC UV EXPOSURE VS. TRANSVERSAL SPEED THROUGH THE ARGUS UNIT

#### TABLE 42.

## SELECTED PROPERTIES OF UV - CURABLE OLIGOMERS AND THEIR FILMS

BRAND NAME	DESCRIPTION	QUALITATIVE <u>PROPERTIES</u>	VISCOSITY <u>(cp)</u>	<u>Tg</u>	% ELONGATION	TENSILE STRENGTH <u>(PSI)</u>
Photoglaze UO83	Urethane Acrylate	Very Flexible	1800	N/A	40-80 (2 mil)	2000- 35000
Novacure 6700	Aromatic Urethane Acrylate	Excellent Flexibility	6000 at 65°C	- 9°C	31	780

## TABLE 43.

## MONOMER SOLVENT CANDIDATES AND THEIR PROPERTIES

BRAND NAME	CHEMICAL NAME	QUALITATIVE <u>PROPERTIES</u>	MANUFACTURER
RC-20	(EOEOEA) Ethyoxyethoxy- ethylacrylate	Good Solvency Low Viscosity	Morton Thioko
ODA	Octyl/Decyl Acrylate	Good Solvency Low Viscosity	Interez
Photomer 4127	(NPGPDA) Propoxylated Neopentyglycol diacrylate	Flexible Low Tg Good elongation High tensile strength	Henkel
Photomer	(HDODA) 1,6-hexane diol diacrylate	Low Viscosity Contributes to flexibility	Henkel
Sartomer	PEG-200 Dimethacrylate	Good Solvency Low Viscosity	Arco

diluents, which were also curable on UV exposure, were necessary in the final sprayed formulations to modify the film properties of the two oligomers and bring them more in line with those of the standard (see later stress/strain plots).

Two photoinitiators were also selected for further evaluation (Table 44). These materials are common components of UV-curable resin formulations, being necessary free radical sources on exposure to accelerate the resin cure.

A Gardner Film Knife System from Pacific Scientific was used to lay films from investigated formulations on 12 in. x 9 in. x 1/8in. glass plates. The system allowed a film thickness range of 0-280 mils, with a six inch maximum width. A Federal Products Corp. Model 57B-1 micrometer was used to measure the thicknesses of resulting films within a  $\pm$  0.00001 mil accuracy. The procedures used to form, cure and test the films are contained in Appendix 10.

The stress/strain curve of the cured UO83 film with no monomer added is compared in Figure 69 with that of the Hycar 26120 standard film. The oligomer without monomer diluent thus yielded a film with too high an initial modulus, a weak yield behavior, a high tensile strength and a relatively low strain-to-break. The film was stiffer and stronger than the standard.

Henkel's Photomer 2127 NPGPDA monomer was added to the UO83 oligomer in a 70:30 resin:monomer ratio by weight, and the resulting film properties compared to those of UO83 alone and Hycar 26120 (Figure 70). The monomer incorporation actually worsened the film's relative properties to that of the neat resin and to Hycar, stiffening it considerably while doubling the tensile strength.

## TABLE 44.

## SELECTED PHOTOINITIATORS FOR UV-CURABLE FORMULATIONS

BRAND NAME	CHEMICAL NAME	MANUFACTURER
DEAP	2.2 diethoxycetophenone	Union Carbide
Irgacure 651	2.2 dimethoxy-2-phenylacetophenone	Ciba-Geigy





FIG. 69


EFFECT OF ADDING NPGPDA TO THE U083 FILM FORMULATION (70:30 RESIN:MONOMER RATIO)



Addition of the Sartomer PEG-200 monomer in the same weight ratio had a similar effect of the UO83 film properties (Figure 71). However, incorporation of RC-20, a diethoxyethyl acrylate, into the formulation at the same 70:30 ratio had the desired effect on the UO83 stress/strain plot, as evidenced in Figure 72. Although the strain-to-break was actually reduced by ~25%, the film's tensile strength was reduced dramatically, as was the initial modulus. A 15% RC-20 loading gave intermediate film properties (Figure 72).

Finally, the ODA monomers, consisting of mixed octyl and decyl acrylates, were blended into the UO83 formulation at 15 and 30% by weight levels. The mechanical properties of the resulting films are detailed in Figure 73. Although the stress/strain plot was modified in the direction of that of the Hycar standard, the improvements at comparable loadings were not as dramatic as the RC-20.

The Novacure 6700 oligomer was next investigated with the NPGPDA and ODA monomers. At 43% loadings by weight, the resulting films yielded the stress/strain properties shown in Figure 74. The 43% ODA loading gave a plot approaching that of the 70:30 UO83/RC-20 formulation, but was brittle in character with a much lower strain-to-break. The higher monomer diluent loading was necessary due to the higher film properties of the Novacure 6700 resin compared to the Photoglaze UO83.

Figure 75 allows a head-to-head comparison of the two oligomer candidates with the NPGPDA and ODA monomer diluents added to the formulations. In comparing Figure 75 with Figure 72 and reviewing the observations made of the films' qualitative characteristics







EFFECT OF ADDING RC-20 TO THE U083 FILM FORMULATION (70:30 RESIN:MONOMER RATIO)



EFFECT OF ADDING ODA TO THE U083 FILM FORMULATION (70:30 RESIN:MONOMER RATIO)





#### COMPARISON OF PHOTOGLAZE AND NOVACURE FILM PROPERTIES MODIFIED WITH ODA AND NPGDA MONOMERS



(Table 45), the decision was made to utilize the 70% Photoglaze UO83/30% RC-20 monomer diluent/1% Irgacure 184 photoinitiator formulation in the electrostatic liquid spray trials on fabric. The initiator was switched at the recommendation of both Lord Corporation and the Ciba Geigy Company from Type 651 (2,2-dimethoxy-2-phenylacetophenone) to Type 184 (1-benzoyl-cyclohexan-1-ol), as the latter produces less yellowing of the final film during UV cure. Any yellowing would potentially slant the shade of the colored fabric, especially with light hues.

After identifying the optimum resin system and before beginning the pilot scale continuous spray run, a proper pigment had to be isolated. Preferably, one with at least some level of solubility in the resin formulation was desired, with the remainder of the pigment finely dispersed in the liquid medium to avoid plugging of the fine exit holes in the spraygun nozzle. In a number of cases with common pigments, the colorants settled out of solution upon standing, indicative of poor dispersability. Of the ones tested, only Phthalocyanine Green Pigment from the Heubach Co. remained suspended in solution for long periods without addition of any auxiliary chemical to improve the dispersion stability.

In preparing the resin formulation for spraying, The Photoglaze UO83 oligomer (2126 gm, 64.2% by weight of final mixture) was heated in the dark to 150F for 45 minutes, reducing the viscosity to approximately 200 cps (the room temperature viscosity was 300 cps). The RC-20 diluent monomer (911 gm, 27.5%) was then slowly added and allowed to mix for 10 minutes. Phthalocyanine Green Pigment (241 gm, 7.3%) was next mixed in over

### TABLE 45.

# QUALITATIVE CHARACTERISTIC OF VARIOUS UV-CURED FILMS

57% Novacure 6700 - 43% Sartomer	Opalescent-flexible-film		
57% Novacure 6700 - 43% ODA	Clear & flexible film but brittle for cutting with knife		
70% UO83 - 30% ODA	Clear and flexible film but brittle for cutting with ink knife		

a 45 minute period with moderate agitation. Finally, the Irgacure 184 photoinitiator (34 gm, 1%) was added to complete the formulation. The final solution was stored at room temperature in the dark until the spray trial began.

Concern existed that excessive heat might be generated by the shear forces in the Nordson spraygun delivery system, initiating polymerization and seizing the pump. The viscosity profile in Figure 76 predicted the optimum processing temperature for the To be certain, a Brookfield thermocell and resin formulation. viscometer was used to follow the viscosity of the formulation as a function of time under isothermal conditions: 50, 70 and 100C. As determined from Figures 77-79, heating the formulation to 50C and 70C produced a significant drop in viscosity, while additional heating to 100C produced a relatively large growth rate in viscosity with time, apparently due to polymerization. The optimum processing temperature, generated by the heater supplied with the control system of the Nordson unit, was pegged at 70C. From Figure 78, the formulation was stable enough for safe processing for six hours at the operating temperature, with very little viscosity increase due to polymerization during this period.

Further details of the delivery pump operation and startup/shut-down procedures for the Nordson liquid spraygun system are provided in Appendix 11, while Appendix 12 shows the design details for the motor-driven belt system that was built to provide transport of the mounted fabric through the booth. The substrate was a standard 50/50 cotton/polyester, fully-prepared sheeting fabric isolated directly from the plant partner's production (119



τ.

Temperature (C)

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FIG. 77. ISOTHERMAL VISCOSITY PLOT FOR RESIN FORMULATION (50°C)

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Time (min.)

. . . . . . . . . . . . .

1.11

Viscosity (cps)

FIG. 78. ISOTHERMAL VISCOSITY PLOT FOR RESIN FORMULATION (70°C)

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211

Time (min.)

FIG. 79. ISOTHERMAL VISCOSITY PLOT FOR RESIN FORMULATION (100°C)

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:



Time (min.)

Viscosity (cps)

 $1^{2}$ 

gm/m<sup>2</sup> base weight). The fabrics were mounted under slight tension on nine inch diameter wooden embroidery hoops to prevent ripples and sagging as the booth was being traversed, and to simulate a tenter frame operation.

The control system and belt speed were synergized to produce an average SPU of 22% of the pigmented formulation. Several of the sprayed fabrics were predried in a conventional oven at 150C for 1.5 minutes before UV exposure to determine if a short thermal precure would avoid wicking problems (leading to unlevelness) of the low viscosity formulation.

All samples, including an unsprayed greige sample, were cured on the Argus UV unit in a second step, using both lamps for the exposure and operating at a belt speed setting of 25% of maximum. Five passes through the unit under these conditions were required for complete curing (defined as absence of tackiness).

Cured samples were conditioned for 24 hours at 70F and 65% relative humidity (standard textile testing conditions). The fabric was then neatly cut out of the inside edge of the embroidery hoop with a sharp razor blade, and the samples weighed in the conditioned atmosphere to check SPU.

The dark green-colored fabrics exhibited good levelness considering the crude drive system utilized to propel the mounted materials through the spray pattern. Microscopic evaluation of the fabric surfaces revealed the presence of solid pigment particles, as well as evidence from individual fibers protruding from yarns that true "dyeing", or molecular transport of solubilized pigment, had actually occurred to a certain extent (Figures 80-81). From



FIG. 80. SOS COLORED FABRICS AT 14X AND 40X MAGNIFICATIONS





FIG. 81.

SOS COLORED FABRIC AT 50X MAGNIFICATION AND COLORED YARN/FIBERS AT 64X MAGNIFICATION the visual evidence, the solid shade coloration aspect of the electrostatic spraygun process was judged to be good, and far superior to any of the results obtained in the earlier research attempting to achieve adequate shade coverage with the Nordson powder spraygun system (1).

Wet and dry crockfastness tests were performed on the samples according to AATCC Test Method 116-1983. The amount of color transferred from the samples to the white test fabric was evaluated by means of the AATCC Scales (Tables 46-47). The results, recorded in Table 48, reveal fair to good performance in dry crockfastness, but only fair to poor resistance to wet crocking. However, the wet crockfastness ratings were comparable on several of the fabrics to those achieved with screen printed fabrics supplied by the plant partner as controls ( control black and red shades gave a 3.0 average ranking, see Table 28). Improvement should be made in the pigment binding ability of the resin formulation before commercialization of the process (perhaps by increasing the cure time exposure) to raise the wet crockfastness ratings.

The SOS colored fabrics were subjected to accelerated conditions of home laundering according to AATCC Test Method 61-1986, with the particular conditions of Test No 3A employed. The color changes of the test specimens were evaluated using both gray scales for color change and stainings. The test results confirmed that the binding of the pigment by the binder formulation was not as efficient as desired, with poor ratings achieved for color change and only fair ratings for staining (Table 49).

By contrast, the colorfastness of the SOS colored fabrics to

## TABLE 46

# AATCC GRAY SCALE FOR COLOR CHANGE

Class	5	Negligible or no change as shown in gray scale step 5
Class	4.5	A change in color equivalent to gray scale step 4-5
Class	4	A change in color equivalent to gray scale step 4
Class	3.5	A change in color equivalent to gray scale step 3-4
Class	3	A change in color equivalent to gray scale step 3
Class	2.5	A change in color equivalent to gray scale step 2-3
Class	2	A change in color equivalent to gray scale step 2
Class	1.5	A change in color equivalent to gray scale step 1-2
Class	1	A change in color equivalent to gray scale step l

## TABLE 53

## FLAMMABILITY TEST PERFORMANCE OF SOS COLORED AND GREIGE FABRICS

<u>SPEC1</u>	IMEN	<u>IGNITION</u>	TIME (SEC.)	<u>RATING</u>
604		3x	16.5	Class 1
616		3x	16.5	Class 1
625		2 <b>x</b>	15.1	Class 1
634		2 <b>x</b>	14.8	Class 1
664		2 <b>x</b>	12.3	Class 1
500	Prepared Base Fabric	2x	13.0	Class 1
501	Prepared Base Fabric	2x	12.9	Class 1
502	Prepared Base Fabric	2x	13.7	Class 1

D1388-64. Both warp and filling directions were tested, with four determinations made per specimen (i.e., top and bottom of both ends). The overhang length was recorded to the nearest millimeter, and the flexural rigidity calculated (Table 54). The fabrics were stiffer than desired for a commercial sheeting product, with extreme warp values of >1250 mgm-cm obtained in three cases compared to only 192 mgm-cm for the greige (untreated) standard. By contrast, the Kativo prints generated in the earlier SOS xerography printing research from the same sheeting fabric exhibited warp values in the 352-365 mgm-cm range (Table 29). The high SPU loading of the formulation onto the fabric to achieve the desired dark green shade (22% average) was obviously a major contributor to the excessive stiffening. A higher concentration of pigment in the resin than the utilized 7.3% may have allowed achievement of the same dark shade, but at a much lower resin incorporation into the fabric structure.

The pigmented, UV-curable resin formulation was thus successfully and uniformily applied to the continuously-moving sheeting fabric by the Nordson electrostatic spraygun system, overcoming concerns regarding the applicability of the machine to the organic medium. The short thermal predrying step appeared to aid in the wicking of the formulation throughout the fabric structure, but more research is needed to further substantiate this observation. Additional investigations into the balance between pigment loading, photoinitiator concentration, formulation SPU and time of UV exposure should ensure more efficient resin curing (and thus improved color fastness properties) without limiting shades.

# TABLE 54

# RELATIVE STIFFNESS OF SOS COLORED AND PREPARED BASE FABRICS

SPECIMEN	DIRECTION	FLEXURAL RIGIDITY (MG-CM)
617	Warp	1,270
638	Warp	1,470
677	Warp	1,500
803	Warp	873
811	Warp	927
628	Filling	720
666	Filling	820
688	Filling	732
804	Filling	535
833	Filling	550
Prepared Base Fabric	Warp	192
Prepared Base Fabric	Filling	84.8

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#### VI. INTRAFIBER LIQUID SPRAY FINISHING PROCESSES

Once the Nordson electrostatic liquid spray system was in place and operational on application of the UV-curable resins (Section V), attention turned to development of SOS processes for intrafiber finishes to the applying same prepared, 50/50 polyester/cotton sheeting fabric utilized in the xerography printing and solid shade coloration research. Intrafiber finishes are those that actually penetrate the solid state structure of the fiber before curing, as opposed to interfiber finishes that reside on the fiber surface. Two areas of particular interest were reactive silicone finishes and permanent press (or durable press) resins.

Two reactive softeners manufactured by the Union Carbide Co. were identified as prime candidates for the initial electrostatic spray finishing trials. Ucarsil Magnasoft reactive finish was an older product line based on aminofunctional silicones and optimized to impart exceptional softness to a broad range of woven and nonwoven fabrics while minimizing yellowing. The material was available as a neat, 100% fluid in the proper viscosity range (250 cSt at 25C) to operate well in the Nordson spraygun system. The yellowing characteristics of aminofunctional silicones is well documented in the literature, but the Magnasoft product had one of the better reputations with the manufacturing industry in minimizing the yellowing problem. Table 55 lists the physical

# TABLE 55.

## PHYSICAL PROPERTIES OF THE UCARSIL MAGNASOFT REACTIVE SILICONE FINISH

Appearance	Clear Liquid
Emulsion Type	-
Color (GVS)	,1
Silicone Actives, %	100
Viscosity at 25°C, cSt.	250
Specific Gravity at 25/25°C	0.97
Refractive Ind <b>ex,</b> 25°C	1.40

properties of the permanent softener.

A newer reactive softener marketed by Union Carbide was Ucarsil T-29, an epoxyfunctional silicone that was claimed to avoid the yellowing problems associated with aminofunctional materials. The viscosity was higher than the Magnasoft line (700 cSt at 25C), but the material was stable to moderate temperatures and exhibited good viscosity reduction with temperature. Physical properties of the T-29 product are detailed in Table 56.

The viscosities of the two softeners as a function of temperature are exhibited in Figures 82-83. Both materials experienced a rapid drop in viscosity with temperature before it began to level off after 60C. The T-29 softener, with its less desirable viscosity from the standpoint of the Nordson machine's limitations (<600 cps), was stable for several hours under 90C isothermal conditions (Figure 84). Based on the data, the application temperatures were fixed at 60C and 90C for the Magnasoft and and T-29 softeners, respectively.

The application procedure was the same as that detailed earlier for the spraygun coloration of fabrics (Section V). Operating conditions for the Nordson system (Fig. 25) are detailed in Table 57. Curing conditions in a conventional convection oven were held at three minutes dwell time with three different temperatures: 170, 180 and 190C. Prepared base fabric was also carried through the curing cycle to generate a standard, as the softness/harshness of a fabric is affected by thermal conditioning.

Cured samples were conditioned for 24 hours at standard 70F/65% R.H. conditions, and cut from the embroidery hoop carrier

## TABLE 56.

## PHYSICAL PROPERTIES OF THE UCARSIL T-27 REACTIVE SILICONE FINISH

Properties	Units	Union Carbide UCARSIL T-27
Description		Epoxy-Functional Polysiloxane
Viscosity @ 25 deg C	cst cps	700 679
Specific Gravity		.97
Diluents		NONE
Flash Point	Deg. C	>200
Appearance		Amber Liquid
Density		8.1
°F		>400
Diluents Present		None



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FIG. 83. VISCOSITY BEHAVIOR OF NEAT T-27 FINISH AT VARIOUS TEMPERATURES

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FIG. 84. VISCOSITY OF UCARSIL T-29 REACTIVE SOFTENER UNDER 90°C ISOTHERMAL CONDITIONS

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## TABLE 57.

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# NORDSON LIQUID SPRAYGUN SYSTEM OPERATING CONDITIONS FOR REACTIVE SILICONE APPLICATIONS

RESIN	MAGNASOFT	UCARSIL T-29
TEMPERATURE (C)	60	90
ELECTROSTATIC OUTPUT (KV)	65	65
MAIN AIR PRESSURE (PSI)	100	100
FLUID AIR PRESSURE (PSI)	300	1200
NOZZLE NO.	48	15

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(see Section V). The average SPU for the two softeners, along with the SPU range, is recorded in Table 58.

finished fabrics were subjected to repeated home The launderings in accordance with AATCC Test Method 124-1984. Standard Test Condition Level IV was used for the wash (115F wash, cold water rinse). For the drying portion of the cycle, Level A -Tumble Drying was employed. Five cycles were performed on each Pilling tests were then conducted on both washed and sample. unwashed samples as well as the standard (ASTM Test Method D3512-76, Random Tumble Pilling Tester). An exception was made in that semi-circular samples were cut into halves, with one half pilled while the remaining piece was saved for pilling comparison. The data are exhibited in Tables 59-60. All of the fabrics, regardless of treatment, received the same 4 rating, with 5 being the maximum of the test procedure.

# TABLE 58.

### SPU OF FABRICS TREATED WITH REACTIVE SOFTENERS

RESIN	AVERAGE PERCENT	RANGE OF PICK UP
	PICK UP (%)	(%)
MAGNASOFT	2.11	0.72-3.26
UCARSIL T-29	2.33	1.25-3.00

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# TABLE 59.

### PILLING RESULTS FOR THE MAGNASOFT FINISHED FABRICS

RESIN	SAMPLE	RATING
MAGNASOFT	TREATED-UNWASHED	4
MAGNASOFT	TREATED-UNWASHED	4
MAGNASOFT	TREATED-WASHED	4
MAGNASOFT	TREATED-UNWASHED	4
MAGNASOFT	TREATED-UNWASHED	4
MAGNASOFT	TREATED-UNWASHED	4

## TABLE 60.

# PILLING RESULTS FOR THE T-29 FINISHED FABRICS

RESIN	SAMPLE	RATING
UCARSIL T-29	TREATED-WASHED	4
UCARSIL T-29	TREATED-WASHED	4
UCARSIL T-29	TREATED-UNWASHED	4

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To assess the critical softness factor of the finished fabrics, volunteer human subjects were asked to rate samples for best hand without the aid of visual observation of the surface appearance of texture. The individuals were told to base their judgement upon their own verbal criteria, e.g., softness, drape, stiffness, etc. Ratings were from 1 (best overall hand) to 4 (least desirable hand). Initial tests explored differences in curing temperature. Washed and unwashed treated samples, as well as washed and unwashed standard (unfinished) samples, were compared. Subsequent trials examined Magnasoft and T-29 finished fabrics in head-to-head comparisons.

From the statistical analysis of the raw data, curing temperature did not affect the degree of fabric softness (Table 61). In Table 62, the overall softness was graded on a three point scale: 3 for a first place rating, 2 for a second place rating and 1 for a third place rating. The F-test of confidence level of values in Table 62 revealed that the variance from any two of the cure temperatures was not significantly different at a 95% confidence limit, while the mean from two temperatures was not significantly different at a 99% confidence limit.

The test results for softener comparison of Magnasoft versus T-29 finished fabrics are listed in Table 63, and the overall rating of each softener is exhibited in Table 64. The data revealed that fabrics treated with each softener possessed a markedly improved hand over the standard (unfinished) fabric. The Magnasoft (aminofunctional silicone) product performed slightly

### TABLE 61.

BLIND AESTHETIC TEST RESULTS FOR OVERALL HAND OF SOS FINISHED FABRICS

RATING	TEMPERATURE (C)	OVERALL	MAGNASOFT	T-29	UNTREATED
	170	7	4	3	
<b>1ST</b>	180	3	3		
	190	4	4		
	170	1	1		
2ND	180	7	6	1	
	190	6	6		
	170	6	3	2	1
3RD	180	4	3	1	
	190	4	3	1	
### TABLE 62.

OVERALL STATISTICAL RATINGS FOR BEST HAND OF SOS FINISHED FABRICS

TEMPERATURE (C)	170	180	190
MEAN (X)	1.93	1.93	2.14
STANDARD DEVIATION	0.997	0.73	0.77
VARIANCE	0.995	0.533	0.593

### TABLE 63.

# SOFTENER TEST RESULTS COMPARING THE MAGNASOFT AND T-29 FINISHED FABRIC WITH THE STANDARD

RATING	TEMPERATURE	(C)	MAG	NASOFT	Т	-29	UNTREATED
			WASH	UNWASH	WASH	UNWASH	
1ST	170		6	2	5	-	1
	180		11	-	3	-	-
	190		11	1	2	-	-
	OVERALL		28	5	10	-	1
2ND	170		3	1	7	-	3
	180		2	-	8	-	4
	190		1	1	10	-	2
	OVERALL		6	2	25	-	9

### TABLE 64.

### OVERALL RATING FOR MAGNASOFT VERSUS T-29 FINISHED FABRICS

RESIN	TEMP. (C)	MEAN (X)	VARIANCE	STANDARD DEVIATION
MAGNASOFT	170	1.43	0.53	0.70
	180	1.86	0.13	0.36
	190	1.86	0.13	0.36
	OVERALL	1.42	0.67	0.79
UCARSIL	170	1.21	0.47	0.66
T-29	180	1.00	0.43	0.20
	190	1.00	0.43	0.20
	OVERALL	0.87	0.49	0.675

better than the T-29 (epoxyfunctional silicone) product in the blind evaluations. However, subjects were generally not able to confidently choose one softener-finished fabric over the other.

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The Nordson electrostatic liquid spray system was thus able to apply the neat, 100% softener fluids at low SPU in a continuous and efficient SOS mode. Both the Magnasoft and T-29 reactive softeners of the Union Carbide Co. adequately softened the 50/50 polyester/cotton standard sheeting fabric, and the finishes were fast to repeated launderings.

The second major area of interest in SOS intrafiber spraygun finishing was the application of durable press resins. The vast majority of cotton-containing fabrics produced in the U. S. are durable press finished, mainly with the reactive agent dimethyoldihydroxyethylene urea (DMDHEU) or its methylated or glycolated derivatives. The latter two resins were developed and commercialized to provide finishes exhibiting lowered release levels of free formaldehyde, a common problem with DMDHEU-treated fabrics due to the presence of unreacted (and subsequently cleaved) free methylol functional groups in the cured resin.

The C. H. Patrick Co. supplied the research team with waterbased formulations of DMDHEU and the two main derivatives. Upon isolation of the three resins from the commercial solutions, it was discovered that the DMDHEU was a glassy solid at room temperature and unsprayable in its undiluted form. The glycolated derivative, though a viscous liquid at room temperature in the pure form, was still unsuitable for spray applications. Apparently, during the water-based synthesis of the two materials, some crosslinking had

occurred to generate various higher molecular weight species that raised the viscosity of the isolated materials. Excessive hydrogen bonding through hydroxyl groups also contributed to the glassy nature of the pure DMDHEU.

The methylated DMDHEU derivative offered the best opportunity to develop a sprayable formulation with a durable press resin. Although well above the <600 cps limit imposed by the Nordson system's pumping capability, the material was flowable at room temperature, and was found to be highly soluble in commercial polyethylene glycols.

The latter fact became critical in developing a final A research team at the Southern Regional Research formulation. Center of the USDA in New Orleans, LA had been working several years to develop a process that would impart both permanent press and thermal responsiveness characteristics to cotton-containing fabrics. Eventually tabbed the Polytherm<sup>c</sup> process, the water-based formulation coupled polyethylene glycols (PEG's) of various molecular weights with unsubstituted DMDHEU resin and appropriate catalysts so that both major components reacted through the cellulose hydroxyl groups and with each other via dehydration to produce a permanent finish. The resulting Polytherm fabrics respond to the environment and body temperatures, i.e., when the body cools the fabric warms, and vice-versa. The durable press (DP) resins give carefree advantages to the resulting fabrics, but also prevent shrinkage of the structure upon wetting, a serious problem in early versions of Polytherm (PT) fabrics finished only with PEG's.

A waterless SOS process was envisioned, built around the methylated DMDHEU resin and a lower molecular weight (MW), and thus a lower viscosity, PEG. The DP resin, once solubilized in the PEG, could possibly be cured with the usual magnesium chloride/citric acid dehyration catalyst if the latter were soluble to sufficient concentration in the final formulation. Proper selection of the PEG would also provide a lower viscosity medium than provided with the neat methylated DMDHEU, affording sprayability within the Nordson system limitations. PEG's are linear complexing agents, or ligands, for metal ions, possessing the capability of incorporating additional electron density, e.g., from the Mg cation. As suspected, the MgCl<sub>2</sub>/citric acid catalyst system was indeed soluble in small, but adequate, concentrations in the PEG-solubilized DP formulation.

The developed formulation procedure involved first dissolving 192.6 gm of MgCl<sub>2</sub>/19.9 gm of citric acid (10:1 molar ratio) in 3.315 kgm of 400 MW commercial PEG with moderate (50C) heating. After cooling to room temperature, 729 gm of dried, neat methylated DMDHEU (MEDMDHEU) was added to the formulation with vigorous stirring. No water was added to the formulation, although due to the hydrophilic nature of the PEG, small quantities of adsorbed water of unknown concentration from the atmosphere were doubtlessly present in the formulations. Dry box techniques were not incorporated to rigorously exclude water from the mixture, however, as such an approach would be impractical in subsequent commercial SOS processes.

The final formulation gave a viscosity by Brookfield analysis

of ~720 cps at 25C, well above the Nordson pump limitation of 600 cps. The viscosity dropped nicely, however, as a function of temperature (Fig. 85), coming within the proper range at 40C. From the isothermal 40C data in Figure 86, the formulation was slowly reacting over a two-hour period, but the viscosity build-up was not excessive enough to be of concern with the Nordson system. Thermal curing conditions for the sprayed fabrics were defined as 165C/3 minutes in a standard convection oven.

Utilizing the standard Nordson liquid spraygun system arrangement (see Fig. 25 and Section V), the optimized formulation was applied to the moving, prepared 50/50 polyester/cotton base fabric mounted on nine-inch wooden embroidery hoops. The SPU's of the fabrics ranged from 21.5-47.3%, gauged to fall within the range optimized in the SRRC research and attained by varying the belt speed of the continuous drive system of the spray booth arrangement.

To ascertain the fastness properties of the treated fabrics, they were subjected to AATCC Test Method 124-1984, Level IV wash conditions (115F wash temperature, cold water rinse) and Level A drying conditions (tumble drying). The circular samples cut from the embroidery carrier hoops were halved, with one side subjected to 5 wash/dry cycles and the other left unwashed. Unfinished standard fabric was subjected to the same treatments as the SOS finished samples.

Wrinkle recovery angle tests were performed on the fabrics according to AATCC Test Method 66-1984. All samples were tested in both the warp and filling directions. Both washed and unwashed



#### FIG. 85. VISCOSITY OF THE POLYTHERM/DP RESIN FORMULATION AS A FUNCTION OF TEMPERATURE

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samples were examined. From Table 65, the wrinkle recovery behavior of the fabrics was greatly enhanced by the finishing treatment, with high values obtained in both the warp and filling directions comparable to those reported by the plant partner for normal production fabrics finished in a wet pad-nip-cure process with straight DMDHEU resins, and also to those finished by the SRRC PT process (Table 66).

With the lowering of the test numbers, the data indicated a slight decrease in wrinkle recovery angle characteristics of the treated fabric upon washing, but the drop range was comparable to those observed in the wash test with plant- and SRRC- finished fabrics, and was thus not a cause for concern on the fastness of the finish (Table 66). The increase from 21 to 48% SPU gave a corresponding increase in the warp and filling values, but the benefit was not considered worth the added chemical and loading costs (Table 65). The SRRC fabrics, finished in its wet process with 1000 and 1450 MW PEG's, showed little difference in wrinkle recovery angles with increasing MW, indicating that the test results were independent of the size PEG molecule incorporated in the PT/DP combined formulation.

Since the wrinkle recovery angle (WRA) characteristics of the fabrics were the key properties sought in the PT/DP finishing process, the test was used to determine the optimum SPU level for the SOS formulation. From the data in the lower half of Table 65, a SPU loading in the 35-37% range yielded the highest WRA numbers in the critical warp direction, and this range appeared to define the levelling point in WRA improvement.



## FIG. 86. 40°C ISOTHERMAL CHARACTERISTICS OF THE VISCOSITY OF THE POLYTHERM/DP FORMULATION

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### TABLE 65.

### WRINKLE RECOVERY ANGLE PERFORMANCE OF PT/PP FINISHED FABRICS

PERCENT PICK-UP	TREATMENT	WARP MEAN (DEGREE)	FILLING MEAN (DEGREE)
21%	WASHED	135.2	136.6
21%	UNWASHED	138.7	144.7
48%	WASHED	148.3	147.5
48%	UNWASHED	145.5	145.7
PREPARED BASE FABRIC	WASHED	119.3	116.8
PREPARED BASE FABRIC	UNWASHED	117.9	122.1

### CURE VS. PROPERTIES 3 MIN. CURE

TEMP. OF CURE ( <sup>0</sup> C)	% ADD ON	X WARP WRA ( <sup>0</sup> )
160	64.3 40.0	134.30 134.80
170	43.1 43.3	131.7 134.3
180	23.0 36.7	125.3 134.0
200	30.5 16.6	130.0 122.2

78% PEG MW400

17% Mc DMDHEU

5% MgC1<sub>2</sub>/CITRIC ACID

UNTREATED CONTROL -1050

### TABLE 66.

### WRINKLE RECOVERY ANGLE TEST COMPARISONS FOR PLANT FINISHED, SRRC FINISHED AND PREPARED BASE FABRICS

		WARP	FILLING
SPECIMEN	TREATMENT	MEAN (DEGREE)	MEAN (DEGREE)
PLANT CONTROL	WASHED	134.5	113.0
PLANT CONTROL	UNWASHED	143.5	135.5
SRRC (1000)	WASHED	118.0	130.5
SRRC (1000)	UNWASHED	125.5	126.5
SRRC (1450)	WASHED	118.5	138.5
SRRC (1450)	UNWASHED	126.5	128.5
PREPARED BASE FABRIC	WASHED	119.3	116.8
PREPARED BASE FABRIC	UNWASHED	117.9	122.1

Stiffness characteristics of the finished fabrics were determined by the Cantilever Beam Test, Option A, of ASTM Test Method D1388-64. Due to the unusual shape constraints of the specimen, only the warp direction was tested. Four determinations were made for each specimen, i.e., top and bottom of both ends, and the results averaged. The data, contained in Table 67, revealed that the prepared base fabric was much less stiff after washing than the unwashed standard (>50% drop in values). The unwashed, finished fabrics were comparable in stiffness to the washed standard, and much more flexible than the unwashed base fabric. Further reduction of the finished fabric stiffness was obtained by washing. Surprisingly, the 48% SPU-loaded fabrics were slightly less stiff than the 21% SPU fabrics, indicating the excellent softening capability of the PEG.

As evidenced by the data in Table 68, the plant- and SRRCfinished fabrics exhibited comparable or higher stiffnesses compared to the SOS-finished fabrics. The higher MW PEG fabric from SRRC indicated that stiffness increased as a function of PEG molecular size in its wet process.

To ensure that the strength of the SOS-finished fabrics had not been sacrificed in the processing, the breaking load and elongation of the materials were measured according to ASTM Test Method D1682-64 (one-inch cut strip method). As evidenced by the Table 69 data, a slight increase in strength was actually achieved upon SOS application and curing of the combined PT/DP formulation.

For viscosity lowering purposes, a much lower molecular weight PEG was utilized in the SOS studies than had been employed in the

### TABLE 67.

### STIFFNESSES OF SOS PT/PP FINISHED AND PREPARED BASE FABRICS

PERCENT PICK-UP	TREATMENT	FLEXURAL RIGIDITY (MG-CM)
48%	Washed	48.0
48%	Unwashed	83.8
21%	Washed	66.4
21%	Unwashed	91.8
Prepared Base Fabric	Washed	84.7
Prepared Base Fabric	Unwashed	209.0

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### TABLE 68.

### STIFFNESS VALUES FOR PLANT - AND SRRC - FINISHED FABRIC COMPARED TO PREPARED BASE FABRIC

SAMPLE	TREATMENT	FLEXURAL RIGIDITY
WPP	Washed	86.7
WPP	Unwashed	186
SRRC (1000)	Washed	156
SRRC (1000)	Unwashed	356
SRRC (1450)	Washed	245
SRRC (1450)	Unwashed	456
Prepared Base Fabric	Washed	84.7
Prepared Base Fabric	Unwashed	209

# TABLE 69.

### MECHANICAL PROPERTIES OF SOS PT/DP FINISHED AND PREPARED BASE FABRICS

SAMPLE	TREATMENT	BREAKING LOAD (LB)	APPARENT ELONGATION (%)
Prepared Base Fabric	Washed	56.8	18.0
Prepared Base Fabric	Unwashed	58.2	15.5
Treated	Washed	55.9	22.0
Treated	Unwashed	54.9	19.5

SRRC wet process research (400 vs. 1000 and 1450). The generation of Polytherm properties in the SOS-finished fabrics was thus in doubt, as SRRC researchers had postulated that the thermal response properties of the PEG arose from the ability of the bound chemical to coil (crystallize) and uncoil with the adsorption or release of heat, a trait dependent on the length of the PEG backbone chain. Differential Scanning Calorimetry (DSC) was employed by the SRRC team to quantify the temperature of the PEG phase transition and the total quantity of heat released or adsorbed at the transition temperature range.

The SOS-finished fabrics (21% SPU level) were compared by DSC with the same material finished at SRRC and with unfinished, prepared base fabric. The ranges employed for heating/cooling were -20C to 50C and the reverse. The DSC scans, conducted at 5C/min rates, are exhibited in Figures 87-94. As expected, the prepared base fabric showed no distinguishable DSC peaks on either heating or cooling cycles (Figures 87-89). The SOS PT/DP finished fabric exhibited a sharp endothermic peak at OC upon heating (Fig. 89), but did not show a corresponding exothermic peak on the cooling cycle (Fig. 90). The same fabric treated in the 1000 and 1450 PEG/DMDHEU process at SRRC yielded a broader endothermic peak, but at a much higher temperature (31 and 33C peak temperatures, respectively, Figures 91 and 93). Apparently, the peak temperature of the PEG phase transition was dependent on the molecular length of the backbone chain, a logical fact as the ability of a polymer to organize its structure is dependent on its molecular length. The SRRC-finished fabrics also showed no distinguishable DSC





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FIG. 88. COOLING DSC CYCLE FOR PREPARED BASE FABRIC

FIG. 89. HEATING DSC PLOT FOR SOS PT/DP FINISHED FABRIC (21% SPU)





FIG. 90. COOLING PSC PLOT FOR SOS PT/DP FINISHED FABRIC (21% SPU)

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FIG. 91. HEATING DSC PLOT FOR SRRC FINISHED FABRIC (1000 PEG)



# FIG. 92. COOLING DSC PLOT FOR SRRC FINISHED FABRIC (1000 PEG)



# FIG. 93. HEATING DSC PLOT FOR SRRC FINISHED FABRIC (1450 PEG)







exotherms on cooling, unlike fabrics of other fibers and constructions reported by it (Figures 92 and 94). The reasons why the 50/50 polyester/cotton fabric showed no analogous exothermic peak on the DSC cooling cycle were not readily apparent.

The critical data for the DSC studies are contained in Table The heat adsorption levels were similar in the SOS- and SRRC-70. finished fabrics, but the peak temperatures were separated by ~30C. Since the peak temperature for the SOS-finished finished fabric was considerably below that of room temperature (1.7C vs. 25C), the ability of the material to function in a true "Polytherm" fashion remained doubtful. Other benefits reported for the PEG incorporation by the SRRC research team (enhanced soil release, antistatic behavior, etc.) should, however, be present with the SOS-finished fabrics, although time restraints on the project did not permit complete analysis of the produced fabrics for additional physical and performance properties. The stiffness tests did, however, indicate the softening action of the PEG on the sheeting fabric.

In summary, the combined PEG/MEDMDHEU/catalyst formulation gave a reactive system that could be effectively applied in a continuous process to a polyester/cotton sheeting fabric with the Nordsen electrostatic liquid spraygun apparatus. The optimized formulation and procedure is summarized in Table 71. Acceptable permanent press, strength and stiffness properties were achieved on the fabrics produced with the SOS process, comparing favorably with plant- and SRRC- finished analogs. However, true "Polytherm" properties were not achieved in the desired 25-30C temperature

### TABLE 70.

CRITICAL DSC DATA FOR SOS-FINISHED, SRRC-FINISHED AND PREPARED BASE FABRICS

SPECIMEN	T <sub>M</sub> ( <sup>°</sup> C)	H <sub>F</sub> (CAL/G)	T <sub>c</sub> (°C)	H <sub>c</sub> (CAL/G)
SOS-Finished (21% SPU)	1.7	1.16		
SRRC - 1000	33	1.35		
SRRC - 1450	31	1.46		
Prepared Base Fabric				

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#### TABLE 71.

#### FORMULATION AND PROCEDURE FOR SOS PT/DP FINISHING OF BLEND SHEETING FABRIC

#### DP FORMULA

78% PEG (MW 400) 17% METHYLATED DMDHEU \* (DRIED PAT COREZ UM) 5% Mg Cl<sub>2</sub>/CITRIC ACID 10:1 MOLAR RATIO MgCl<sub>2</sub>/CITRIC ACID

\* C.H. PATRICK: MR. GEORGE LORIGAN 803-2440-4831

- 1. FABRIC IS CONDITIONED & WEIGHED AFTER BEING SIZED FOR A HOOP
- 2. SPRAYED W/DP FORMULA. OPTIMUM PICK-UP ~35-37% (P. 26)
- 2B. OPTIONAL: AFTER SPRAYING, FABRIC PASSED THROUGH SQUEEZE ROLLS AT ~1000 PSI.
- 3. CURED (BEST CURE LIKELY 2 MIN. AT ~165°C)
- 4. CONDITIONED AND WEIGHED
- 5. WRA TEST AATCC

range with the low chain length PEG employed (400). Further research will be required with higher MW PEG's and other materials (such as PEG/polypropylene glycol copolymer systems) that can yield stable formulations with heating to generate the proper viscosity range.

#### ENERGY ASPECTS OF SOS PROCESSES

Although commercial processes vary widely within the number of areas investigated in this research, a general comparison can be drawn between the developed SOS application processes utilizing dry powders, liquid resins and liquid finishes and the conventional water-based processes. Considerable energy is consumed in the latter in the heating up and evaporation of water, which must be accomplished before the treated fabric can reach curing temperatures. In the developed SOS chemical systems, the treated fabric only has to satisfy its inherent heat capacity to reach curing temperature, a far lower energy requirement than that required to effect the liquid-gas phase transition of water (~1000 BTU/pound).

Table 72 provides a general energy consumption comparison between a conventional wet pad/pressure nip/cure process and the analogous SOS process. The calculated energy savings are on the order of 4,075 BTU/lb. of processed textile, or a reduction in direct process energy of ~90%. Using this figure, the energy conservation potential of the seven SOS processes developed in the reported research is calculated as shown in Table 73. The total textile industry conservation potential upon SOS process incorporation is thus 7,530,000 BOE per year. This figure does not include other wet processes that are similar in nature to one or more of those for which an SOS analog was successfully developed,

### TABLE 72.

### GENERAL COMPARISON OF ENERGY CONSUMPTIONS IN CONVENTIONAL WET AND SOS CHEMICAL PROCESSES

	ENERGY	<b>REQUIREMENTS</b>
<u>STEP</u>	CONVENTIONAL WET PROCESS (BTU/1b.)	SOS <u>(BTU/lb.)</u>
Chemical Application	0	3
Predry	1990	N/A
Dry	598	N/A
Fixation/Curing	272	272
Wash	640	N/A
Dry	1900	N/A
Electrical Drives	<u>150</u>	<u>150</u>
TOTALS	4550	475

POTENTIAL ENERGY SAVINGS: 4075 BTU/1b.

#### **TABLE** 73.

#### ENERGY CONSERVATION POTENTIAL FOR SEVEN SOS PROCESSES DEVELOPED IN THE REPORTED RESEARCH

PROCESS	CONVENTIONAL PROCESS THERMAL CONSUMPTION <sup>C.</sup> <u>(BTU/Ib)</u>	CALCULATED SOS PROCESS CONSUMPTION <sup>a.</sup> <u>(BTU/Ib)</u>	CALCULATED SAVINGS <u>(BTU/Ib)</u>	POUNDAGE <u>(Ibs/year) X 10</u> ୍ର	ENERGY CONSERVATION POTENTIAL (BOE/year X10୍ର)
Yarn Slashing	2900	475	2475	3,905	1.67
Nonwoven Binding	<b>4550</b> <sup>a.</sup>	475	4075	<b>480</b> <sup>b.</sup>	0.34
Nonwoven Stain Repellent Finishing	4550 <sup>a.</sup>	475	4075	<b>200</b> <sup>b.</sup>	0.14
Xerographic Fabric Printing	9300	475	8825	1,018	1.55
Continuous Fabric Dyeing	4400	475	3925	20,493	1.69
Reactive Silicone Fabric	7800	475	7325	<b>96</b> <sup>d.</sup>	0.12
Durable Press Finishing	7800	475	7325	1,604 <sup>e</sup>	<u>2.02</u>
				TOTAL:	7.53 <sup>f</sup>

### TOTAL NATIONAL ENERGY CONSERVATION POTENTIAL OF FOUR SOS PROCESS AREAS: 7,530,000BOE/YEAR

- <sup>a.</sup> Calculated Consumption (Table 72)
- <sup>b.</sup> Poundages supplied by Kimberly-Clark Corp.
- <sup>c.</sup> Electrical consumption was calculated to be comparatively small and similar for both conventional and SOS processes (150-700 BTU/lb), and was thus neglected in the conservation calculations.
- d. Estimated as 3% of the total poundage of finished fabrics.
- <sup>e.</sup> Estimated as 50% of the total poundage of finished fabrics (these containing cellulosic fibers).
- <sup>f.</sup> Unless otherwise noted by footnote, the thermal consumption and poundage figures were taken directly from an earlier Georgia Tech report to DOE: F. L. Cook, W. W. Carr, W. C. Tincher, <u>et al.</u>, "Energy Conservation in the Textile Industry", Phase II Final Technical Report on DOE Contract No. EY-76-05-5099, School of Textile Engineeringand EngineeringExperimentStation, Georgia Institute of Technology, Atlanta, Georgia, October, 1976. A Barrel of Oil Equivalent (BOE) was defined as the standard 5.8 x 10<sup>-6</sup> BTU's.

e.g., the application of firming agents (interfiber finishes) to fabrics to stiffen up the material via the Nordson powder spraygun SOS technique.

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#### CONCLUSIONS

A 60-end warp was successfully slashed using a thermoplastic size formulation in proof-of-concept trials at West Point Pepperell's Research Center in Shawmut, AL. The optimized size utilized in the trials was a melt-interchanged blend of 65% Eastman WD/35% Adipic Acid. The proper SPU (>10%) could be held in the process only at speeds of <10 ypm, revealing a limitation with the ETI system at the maximum voltage capability. Weaving trials with the SOS-slashed yarns, conducted in a head-to-head manner with conventionally-slashed yarns on a Sulzer TW-11 projectile weaving machine, were unsuccessful, with the 65 WD/35 AA size of insufficient film properties to withstand the abrasion and tension forces imparted on the yarn by the weaving action. The size formulation's powder was also found to pack and solidify on storage due to its "tacky" and hydrophilic nature. The speed of application and size composition problems will have to be resolved in further developmental research before full-scale processes are possible.

Continous xerographic printing of sheeting fabric on a threefoot-wide, three-color continuous line was demonstrated. Complex prints of proper fastness properties and consisting of one-, twoand three-color overlap shades were produced. Pigmented Kativo resin manufactured by H. B. Fuller Co. (a modified epoxy), when mixed with 1% CAB-O-SIL fumed silica produced a toner material that

gave shades of medium depth with the Model 2510 copier and its carrier. A second resin candidate, FE 532 poly(ethylene-co-vinyl acetate) in a 91:9 monomer ratio, also gave good triboelectric series correlation with the carrier bead and adequate printing action, but no suitable procedure was defined for incorporating pigment into the EVA resin. Further research is required with the two toners to allow printing of dark shades, and to define a pigmentation process for the FE 532 resin.

Two proof-of-concept powder spraygun trials at the Nordson Corp. facility were highly successful, with Eastman's FA-252 modified polyester resin producing bound nonwoven polyester fabrics mechanical, pilling and Mullen burst pressure properties with comparable to those of the plant-bound standard. The hand of the SOS fabrics was superior to that of the stiff plant standard, evidenced by lower moduli and higher strains-to-break of the trial fabrics. Powder application speeds of 200 ypm (the upper limit of the 60 in. Nordson process line) were achieved, and indications were that the SOS powder spraygun technique could accomodate even higher line speeds without detrimental effects. A fine powder (<20 microns average particle size) was necessary with a two-gun booth arrangement to achieve optimum results. A second resin binder candidate, H. B. Fuller's IF-3237 modified epoxy resin, possessed excellent flow characteristics on the polyester fiber surfaces, but gave inferior mechanical properties on the fabric compared to the FA-252- and plant-bound materials.

On the same powder spraygun line at Nordson Corp., a 100% polypropylene nonwoven was successfully finished at 200 ypm with a

fluoropolymer from the 3M Co., coded FC-214, yielding adequate properties to oil/solvent barrier and isopropyl alcohol A mechanism to push the fine powder into the penetration. depressions made in the fabric at the thermally bonded points was necessary to avoid drop failures at these points. A compressed air qun was used in the plant trials, but future systems should be built around an air knife and/or a vacuum slot to penetrate the powder into the caverns of the thermal bonded fabric points. The process demonstrated for the first time the ability to properly barrier finish a polypropylene nonwoven substrate with a FP powder deposition/melt process.

Liquid spraygun processes were developed to apply three different finishes to polyester/cotton sheeting fabric in a continuous mode: pigmented UV-curable resins to generate solid shades, reactive silicone softeners and combined thermal responsive/durable press resin formulations. The levellness of shade provided by the solid shade formulation (Photoglaze UO83 oligomer/RC-20 monomer diluent/ Irgacure 184 photoinitiator) was good with the Nordson electrostatic liquid spraygun system, with the coloration due both to monomolecular penetration of dissolved Phthalocyanine Green Pigment and to entrained solid pigment The hand of the shaded fabric was only fair, with the particles. high SPU (22% average) contributing to excessive stiffening of the structure. Several fastness properties of the treated fabrics were also lower than desired, indicating incomplete curing of the resin Further research is required on the level of pigment binder. loading allowable with the resin formulation, and the degree of UV
exposure necessary to give complete curing. Combined thermal/UV curing should also be investigated as a mechanism to generate higher colorfastness properties in the treated fabrics.

Both intrafiber finishing processes gave fabrics with the desired properties. Liquid spray application of neat reactive silicones was an especially facile adaptation of the Nordson technology to textile processing, providing efficient application and curing to generate softened fabric properties. Both the Union Carbide products gave acceptable performances, although the material (aminofunctional) gave slightly better Magnasoft properties than the T-29 (epoxyfunctional) product. The targeted durable press properties were achieved with the combined PT/DP finish formulation, built around 400 PEG/MEDMDHEU/MgCl<sub>2</sub>-citric acid mixtures, but the "Polytherm" properties of the optimized formulation were not as good as those achieved by the SRRC team with the higher molecular weight PEG's out of water solution. Further developments are needed to identify polyglycols that will afford sprayable formulations of the proper viscosity, but will also be of sufficient molecular length to give the correct phase transition action to lead to thermal responsiveness.

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APPENDIX 1.

PARTS LIST FOR 31 SOS SECTION BEAMS

	ORDER NO.	EST P			DUNDRY /	AND M/	ACHINE CON	1PAN	14	INVOICE
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S O L D T O	•Georgia Account: Room 50 Atlanta	Instit s Payab , Knowl , Georg	ute of Tech le es Bldg. jia 30332	nology	S H P T O				u-	, 1907
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01	DTE NUMBER	COMMENTS				<b></b>			COMPLETE	PARTIAL
UANTITY	PART NUMB	ER N	4	DE	SCRIPTION		UNIT PRICE		TOTAL	
31 56 1 1	860930 860930C 860930D 860939E		Sample Loc Friction D Male Adapt Adapter Re	om Beam Discs ter	Plate		52.50 3.01	\$1	,627.5 198.6 40.4	50 56 40 40

UANTITY IHIPPED	PART NUMBER	•	DESCRIPTION	UNIT PRICE	TOTAL
31 56	860930 860930C		Sample Loom Beam Friction Discs	52.50 3.01	\$1,627.50 198.66
1	860930D 860939E		Male Adapter Adapter Retainer Plate		25.40
32	8660-930-A	-	Hubs - Partial MFC	15.00	480.00
1 2	861118		Adapter Ring	38.95	77.90
2			ADD 1/2 X 1/4 Keyway to bore of 31 Each loom beams 860930 Plywood Support Disc 20-3/4" O.D. X	FOR	142.60
2			5.92	FOR	20.00
2			Steel Blanks 6" O.D. X 1-1/2 Steel Blanks 5-1/4" OD X 1"	28.48	56.96
i	861209		Adapter		204.25
2 2 2	861210 861211		Ring - Lock Machining of existing 32" Loom Beam Plwyood support disc 20-3/4 X 5 920	38.95	77.90 354.85
2			X = 3/4		20.00
2			Rebeaming		29.52
			bar & base Crating and Hauling		39.85 118.00
				-	\$2 666 99
			Payment due before April 25, 1987		95,000.99
			and the second		
			· · ·	· ·	

#### APPENDIX 2.

PROCEDURE USED BY WPP TO PREPARE CONVENTIONALLY-SLASHED YARN STANDARD GA Tech Project Slashing Instructions For Conventional Trial Begin Slashing Monday, 1-5-87 35.0's 50/50 Poly/Cotton AJS Yarn from Lanier Slashing using <u>6</u> section beams of <u>60</u> each and <u>5,300</u> yards each Doff <u>5</u> beams, <u>360</u> each, <u>1050</u> yds. each.
Size Mix: Obtain about 30 gal. of Lanier's PVA size mix. Use 17 gal. in size box. Add 2 cups liquid blue tint to size box and stir.

Size Box Temp.: 180°F Squeeze Roll Pressure: 20 lbs Drying Caps: Use 3 case at 15 lbs steam pressure. Dancer Roll: Use 43 lbs total weight (27 gm/end tension) Front Stretch: 0%, use case drive Rear Stretch: 2% Slasher Speed: 9 yds/min.

Depending on performance and appearance, size mix may be changed and rolls cleaned after 2nd or 3rd doff. Slow to creep speed and dispose of yarn when draining size mix.

Obtain sample for desize test off each of the 5 slashed beams.

RE-BEAM THE 5 SLASHED BEAMS ONTO 1 LOOM BEAM, 1,800 ENDS, 1,000 YDS.

(The loom beam is to be combined with S.O.S. yarn onto a Sulzer beam at WP Foundry.)

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FULLER IF 3237 F SERIES GROUP AVERAGES

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AVERAGE			LŪÁD	STRAIN	MODULAS		ACTUAL		AVERAGE	PILLING			PERCENT	AVERAGE	LŨAD	STRAIN	HODULAS	PILLING
THICKNES	5 SAMPLE CO	DIR	AVG	AV6	AV6	DIR	PICKUP	SPEED	PICKUP	TEST	ÐIR	SPEED	PICKUP	THICKNESS	AVG	AVG	AV6	AV6
0.0099	UNTREATED	Ø	11.7	52	1114.77		9	0	0		1	NA	0	0.0099	11.7	52	1114.77	
0.0099	UNTREATED	1	9.5	86	<b>330.</b> 39		6	0	8		ß	NA	ñ	8.0099	9.5	86	338.39	
a 0400	15 5			15	1701 71		10 70		1 <b></b>									
0.0108		1	7.7	68 70	1321.31	1	10.57	200	10.77		1	200	10.7%	0.0114	10.2	74	744.53	ERR
0.0122 a atot	14 F (7 c	1	12.0	/ / / / / / / / / / / / / / / / / / /	007.87 (nn 5)	1	19.04	. 21910 . oao	10.74									
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9 2122	13 F	1	10.0	71 83	574 54	1	11.14	- 200 700	10.7%									
	18 F	•	10.5	03 74	728 85	1	10.17	200	10.7%									
0.0108	10 F	ſ	97	21 21	544 72	÷ F	11 17	- 200 - 200	12.7%									
B. 8119	16 F	ł	9.9	47	1334 55	1	11 17	200	18 77									
	10 T	f	10.6	92 80	505.00 505 70	1	10.77	200 200	10.7%									
a atat	21 F	1	2 t 9	69	701 30	1	19.3%	200	10.75									
0.0101 0.0177	14 F	A	14 3	45	1657 44	9	19 57	700	13 77		9	288	(9.77		14 5	4.1	2101 40	EDO
0.0112	18 F	A	14 5	रर	2551 29	9	10.34	200	10,7%		U	200	10.76	0.0117	14.3	41	2101,40	ENA
0.0111 0.9125	10 F	8	17.6	45	1599 19	) j	11 17	200	10.7%									
0.07E0 0.0108	15 F	a	15.3	41	2348 54	Ā	10 72	200	10.7%									
8.0100 8.0117	10 F	A	14 f	42	2155 18	ផ	10.3%	200	10.7%									
0.0113	12 F	3	17.6	19	2133.18	3	11 57	200	10.75									
0.0121	16 F	Ø	16.0	46	1829.28	a	11.12	200	10.7%									
	13 F	a	14 6	10	2379 24	a	11 57	200	10.7%									
0.0180 0.0191	21 F	a	11 8	39	2449.17	พื	9.37	200	10.7%									
	10 5	ø	13.3	10	2196-25	a	11.17	288	10.7%									
0.0105 0 0115	30 F	i	19.7	79	511.88	1	8.72	200	7.77		1	299	7 77	0 0111	10 3	<u>pa</u>	591 72	EDD
8 8112	30 F	1	9.7	73	557 23	1	6.9%	200	7 7 7		1	200	/ • / /•	0.0111	16.5	0.0	J01,/2	CUV
8.0198	26 F	i	19.5	82	686.77	1	7.7%	200	7.7%									
0.0118	34 F	:	18.6	75	510.93	1	6.52	200	7.72									
8,8186	32 F	1	10.5	80	394.57	1	7.9%	200	7.7%									
9.9129	35 F	1	19.2	86	365.57	1	7.3%	266	7.71									
0.0106	29 F	1	9.9	80	525.09	1	8.3%	200	7.7%									
0.0111	33 F	1	9.6	81	452.79	1	7.5%	200	7.7%									
0.0184	25 F	1	11.3	77	516.79	i	7.3%	200	7.7%									
0.0112	27 F	1	10.6	82	576.35	1	8.5%	200	7.7%									
9.0112	31 F	0	15.3	43	2198.17	0	6.9%	200	7.7%		0	280	7.7%	3.0111	14.8	42 (	2247.38	FRR
0,0120	35 F	0	14.9	42	2011.85	0	7.3%	290	7.7%									
0.0118	34 F	9	15.9	44	2099.63	0	6.5%	200	7.7%									
0.0115	30 F	6	13.7	42	1944.97	0	8.7%	290	7.7%									
0.0104	25 F	9	15.4	42	2563.19	8	7.3%	208	7.7%									
8.0108	26 F	0	14.3	40	2521.53	0	7.7%	200	7.7%									
0.0111	33 F	9	14.8	48	2187.20	9	7.5%	200	7.7%									
0.0106	32 F	8	15.2	44	2317.87	8	7.9%	200	7.7%									
8,8112	27 F	0	14.2	42	2395.74	8	8.5%	288	7.7%									
0.0106	29 F	Ø	i3.8	42	2233.65	0	8.3%	200	7.7%									
8.0106	39 F	1	9.8	79	449.37	1	5.8%	208	4.8%		1	280	4.8%	0.0107	10.2	86	487.60	ERR
9.9181	48 F	1	9.4	81	440.52	1	4.4%	200	4.8%									
0.0118	<b>4</b> 2 F	1	9.5	80	454.07	1	4.2%	200	4.8%									
0.0114	43 F	1	10.4	81	402.58	1	4.8%	280	4.8%									
8.8114	46 F	1	10.1	82	585.86	1		299	4.8%									
9.8112	38 F	1	11.3	78	498.50	1	5.2%	280	4.87									
0.0103	41 F	1	10.2	81	468.27	1	4.8%	288	4.8%									
0.0103	44 F	1	10.5	82	552.01	1	5.07	290	4.9%									
0.0102	36 F	ſ	11.5	82	516.57	1	4,47	200	4.87									
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	0	-	/10	00	00010,		U	5	v		v	in	U	0.6677	7.5	C0	236.31	
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2.0103	44 F	2	13.2	40	2119.38	â	5.65	( 20 <b>9</b>	4.82		A	299	4.9	7 ឆិ ឆិ ឆេ	17 Q	47	000T 80	200
0.0193	41 F	8	13.4	44	2189.25	8	4.82	200	4.8%			200			10.0	72	2000.90	ENA
0.0112	37 F	Ø	13.8	45	1814.39	2	5.23	. 200	4.87									
0.018i	40 F	Ø	13.4	41	2215.61	ē	4.42	289	4.82									
0.0114	46 F	8	14.2	42	2532.61	3		200	4.82									
8.0106	39 F	8	14.6	45	1964.56		5.82	200	4.8%									
9.9114	43 F	ā	13.2	45	1654.65	ดี	4.81	200	4.97									
8.0112	38 F	0	13.3	41	2027.35	ā	5.2%	200	4.82									
0.0118	42 F	9	13.7	34	2233.67		4.22	200	4.82									
8.8182	36 E	ß	14.8	45	2083.44	8	4.4%	200	4.87									
9.0164	79 F	1	9.8	73	774.23	1	10.57	150	19.87		1	159	ស្រី នុវ	( 91.6110	9 Q	75 Ø	949 C	EDO
ā. 9114	72 F	1	8.6	63	1010.12	1	11.17	150	18.87		•	100	10.04	. 0.0110	1.1	/3.0	600.2	ENA
9.0112	69 F	i	19.4	78	753.06	i	11.12	150	10.92									
8.6198	68 F	1	10.4	82	853.69	1	9.57	150	10.82									
0.0106	67 F	1	10.0	76	1016.77	1	11.5%	150	18.82									
8.0116	71 F	1	18.0	78	753.49	î	10.92	150	10.81									
6.0104	70 F	Ø	14.6	42	2359.91	Я	10.52	150	10.8%		3	150	19. Rž		14 5	74 5	3175 <b>3</b>	500
0.0114	72 F	8	16.4	31	3436.86	4	11.17	158	10.87		v	100	10.04	0.0110	10.0	2010	01/2.0	ENK
9.8112	69 E	ē.	16.8	37	3752.92	8	11.17	150	10.8%									
<b>A</b> .0116	71 F	й	16.2	39	2696.77	Ģ	10.9%	150	19.87									
8.0108	68 F	3	18.5	35	3823.82	ñ	9.52	159	10.82									
0.0104	67 F	ñ	16.6	36	3464.99	2	11.5%	150	18.82									
Ø. 9108	73 F	-	1010	00	0.0.0	ā	11.1%	150	10.87									
0.2108	73 F					1	11.12	159	18.82									
<b>A.0113</b>	57 E	1	19.6	72	726.20	i	8.2%	150	7.71		1	150	7 77	0 0i12	12.6	75 Q	A17 0	E89
0.0110	60 F	1	9.5	7Ñ	575.45	î	8.47	150	7.77		•	100		0.0111	11.0	1017	01/11	Lun
0.0111	61 F	1	9.9	78	559.27	1	8.97	159	7.7%									
0.311A	59 F	1	19.9	76	643.39	1	6.8%	156	7.71									
0.0101	67 E	í	9.9	77	615.76	1	7.6%	150	7.72									
0.0107	58 E	1	11.6	76	710.26	1	7.9%	150	7.7%									
9.9121	56 F	1	26.8	82	499.49	1	8.3%	158	7.7%									
0.0111	61 F	2	15.0	38	2625.59	Â	8.02	150	7.72		9	150	7.74	0 0112	15 A	79 5	519 <u>8</u> 5	EDD
0.0119	59 F	A	14.5	37	2589.25	Ģ	6.82	150	7.71		2			010112	1011	0110	47791L	Lun
A. A121	56 E	3	17.2	40	2458.96	Ř	8.32	150	7.71									
0.0101	62 F	Ð	16.9	48	3166.97	5	7.6%	150	7.7%									
9.0107	58 F	- A	14.2	42	2393.89	8	7.7%	150	7.7%									
0.0119	60 F	- g	15.2	40	2141.88	ē	8.4%	150	7.7%									
8.0113	57 F	8	14.9	39	2152.76	ē	8.2%	150	7.7%									
0.9105	55 F	i	9.8	82	483.91	1	4.1%	150	4.5%		\$	159	4.57	A. A189	10.1	897. i	499 8	FRR
9.8094	47 F	1	9.3	77	551.08	1	5.2%	159	4.5%		-					0011	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	Entry.
6.0100	48 F	1	9.5	80	476.15	1		150	4.5%									
8.0117	54 F	1	10.9	82	531.87	i	4.07	150	4.5%									
0.0111	50 F	1	9.4	76	473.43	1	4.27	150	4.5%									
0.0123	52 F	1	11.1	84	454.95	1	4.9%	150	4.5%									
0.0115	51 F	1	10.6	80	464.52	1	4.6%	150	4.5%									
0,0123	52 F	9	17.8	37	2545.94	8	4.92	150	4.5%		0	150	4.5%	0.0109	14.7	40.7	2190.5	ERA
0.0105	55 F	9	15.2	38	2552.03	9	4.17	158	4.52		-				/	• •		
0.0117	54 F	9	13.6	43	1940.91	8	4. <del>2</del> 1%	150	4.57									

											FULI	LER IF	3237	F SERIES	GROUP	AVERAG	ES	
									GROUP									
AVERAGE			LOAD	STRAIN	KODULAS		ACTUAL		AVERAGE	PILLING			PERCENT	AVERAGE	LOAD	STRAIN	MODULAS	PILLING
THICKNESS	5 SAMPLE CO	DI	R AVG	AVG	AVG	DIR	PICKUP	SPEED	PICKUP	TEST	DIR	SPEED	PICKUP	THICKNESS	AV6	AVG	AVG	AVG
0.0099	UNTREATED	Ø	11.7	52	1114.77		9	0	8		1	NA	6	0.0099	11.7	52	1114.77	
0.0099	UNTREATED	1	9.5	86	338.39		9	6	0		0	NA	8	8.0897	9.5	86	338.39	
0.9094	47 F	9	13.7	43	2006.40	មិ	5.2%	150	4.5%									
8.9190	48 F	0	14.2	42	2261.93	0		156	4.5%									
9.0111	5 <b>0</b> F	8	13.7	38	1984.97	6	4.2%	150	4.5%									
0.0115	51 F	Ð	15.8	45	2041.13	8	4.6%	150	4.5%									
8.8113	53 F					0	4.6%	158	4.5%									
0.0113	53 F					1	4.6%	150	4.5%									

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											FULI	ER IF	3237	F SERIES	GROUP	AVERA68	ES	
AVERAGE			LAD	STRAIN	ΧΩΒΙΗ ΔΩ				GROUP				DEDCENT	AUEDAGE	1 ñAD	CTDAIN	номи че	STEETHE
THICKNES	S SAMPLE CO	BIR	AVG	AVG	ΔUG	ករទ	PTCVIP	GPEEN	DICKNOL	TECTIO	nış.	OPECN	PICVIE	TUINVUECO	LUNU	AUE	Alle	AUC
A. 8099	INTREATED	A	11.7	57	1114.77	DIN	9 TUNUI 0	9 21 10	I ICKUS	1601	1	NA	1107.01	0 0000	117	57	1114 77	HVO
8.8077	UNTREATED	1	9.5	84	779 79		a	0	2		a	NH VA	0 3	0.0077 9.0000	05	JZ D1	1114.//	
0.00//	ORTHERIES	1	/10	00	330.01		D	Ð	e		10	RH.	Ð	0.0077	7.J	00	336.37	
6.0108	15 F	1	9.9	65	1321.31	1	18.3%	200	18.7%		1	200	10.77	6.6114	18.2	74	744.53	ERR
0.0122	14 F	1	12.3	79	559.89	1	10.5%	208	10.7%	ı.								
0.0121	12 F	1	18.7	75	6 <b>00.</b> 91	ł	11.5%	200	10.7%									
0.0125	13 F	i	10.9	71	592.22	1	11.17	208	18.7%									
8.0188	17 F	1	10.3	83	534.54	1	11.5%	200	18.7%									
8.0112	18 F	1	10.9	74	728.05	1	16.1%	200	16.7%									
0.0108	10 8	1	9.7	81	564.72	1	11.1%	289	18.7%									
8.8119	16 F	1	9.9	62	1336.55	1	11.1%	298	10.7%									
0.0113	11 -	1	10.6	88	565.70	1	10.3%	200	10.7%									
8.8181	21 F	1	8.1	59	701.38	1	9.3%	200	18.7%									
8.8122	14 F	8	14.5	45	1657.44	8	18.5%	200	10.7%		Ø	200	18.7%	6.0114	14.5	41	2181.48	ERR
0.0112	18 F	6	14.5	33	2551.89	6	10.1%	200	10.7%									
0.8125	13 -	5	13.6	45	1598.18	8	11.1%	200	10.7%									
8.0108	15 F	8	15.6	41	2348.56	6	10.3%	200	16.7%									
6.0113	11 F	8	14.1	42	2155.18	Ø	18.3%	288	10.7%									
0.0121	12 F	8	1/.6	48	2659.36	8	11.5%	200	10.7%									
0.0119	16 F	6	16.0	46	1829.28	8	11.17	298	16.7%									
0.0108	1/ F	8	14.6	43 77	2379.26	۲ ۲	11.5%	288	10.77									
8.0101	21 F	9	11.8	59	2448.17	10 A	9.37	208	10.77									
0.0108		6	15.5	40	2196.25	10	11.1%	280	10.7%									
6.8115	30 F	1	16./	/4	511.08	1	8.7%	200	1.1%		1	288	7.7%	8.0111	18.3	88	501.72	ERR
8.0112	31 F	1	9.7	/3	557.23	I	6.9%	200	7.72									
8.6198	26 F	1	18.5	82	686.//	I	1.1%	200	1.12									
6.0118 a.a.a.	54 F	1	10.0	78	310.93	1	5.3%	200	1.14									
5.0105 a.a.sa	32 F 75 F	1	10.5	ଅଟ ମନ୍ଦ	374.3/	1	7.9%	200	1.14 אר ר									
0.0120 0.0120	30 F	1	10.2	00 00	300.07 E9E 00	1	7.3%	200 555	1.16									
0.0100	29 F 77 F	1	7.7 0 i	50 01	JZJ.07 455 70	1	8.34	200	1.14									
0.0111 a ataz	33 F 35 F	1 f	7.0 (( 7	15 דד	432.17	1	7.34	200	1.14									
0.0104	20 F 37 F	1	11.0 (0.7	77 00	J10./7	4	1.04	200 000	7.74									
0.0112	2/ F 7+ F	1	10.0	02 17 -	J/0.JJ	i	8.JA / 04	∠00 50-0	1.1%		a	-10-0	7 74	a 8111		10.0	017 70	
8.0112 0.0450	. JIF 75 F	ю л	11.0	10. 19.	2170.1/ 2013 DE	10 (4	0.7% 7 7%	200 000	1.14		Ð	790 790	1.15	9.0111	14.8	4Z Z	247.38	EKK
0.0120 a aiito	ол г 34 б	o A	1417	972 - 1 1 1	2011.0J 7005 17	ю Д	1.3%	200 Элд	1.14									
	30 E	0 0	13.7	. דד גר	1044 07	Ci	0.JA 8 74	200	/•/* 7.7%									
0.011J 3 3194	30 F 25 E	е 0	15.7	42 17 1	1799177 9527 10	8	0.1% 7 74	200 739	ייין אר ד									
0.0104 0.0100	20 F 26 E	a a	13.7	40 °	2000.17	0 0	7.5%	200 700	7.7%									
0.0100 0.111	20 र रर ह	a	14.0	10. Aù	2321.33	a.	7 59	200 799	7.7K 777									
0.0111 0.0111	30 F	5	15 9	44 -	2107120	e	7 97	200 288	אין									
9 9110	52 1 27 F	อ	14.7	42	2317107	ý.	9.57 9.57	722	7.7%									
0.0112	27 T	a	17.8	47	2070.74	ē	8 37	200	7.7%									
B A194	27 T 39 F	1	9 g	79	449 77	ĩ	5 397	700 700	4.97		5	200	4 97	a atae	191 7	94	107 Lū	500
9 9 9 91	49 F	1	9 A	81	440 57	1	1 47	288	4.0%		I	200	4.05	0.0107	10.2	00	40/.00	בתת
0.0101	40 F	1	9.5	80	454 07		A 97	200	4.0%									
9.9114	17. F	1	10.4	81	497.58	1	4.87	290 799	4 97									
A. Aila	14 F	1	10.1	82	585.94	t	1.04	280	4.97									
8.8117	38 F	•	11.3	79	498.58	1	5.27	289	4.97									
A. 0103	41 F	1	10.2	81	468.27	1	4, 97	200	4.97									
9,9103	44 F	t t	19.5	82	552.01	1	5.07	298	4.97									
8.8187	36 F	f	11.5	82	516.57	1	4.47	202	4.82									
		-				-												

FULLER IF 3237 F SERIES GROUP AVERAGES

									COGIO						011001	111 6411141		
AVERAGE			LOAD	STRAIN	MODULAS		ACTUAL		AVERAGE	PTILING			PERCENT	AVERAGE	ם מה ו	STRATE	NOTHLAS	PTIITNG
THICKNES	S SAMPLE CO	DIR	AVG	AVG	AVG	DIR	PICKUP	SPEED	PICKUP	TEST	DIR	SPEED	PICKUP	THICKNESS	AVG	AVE	AVE	AVG
0.0099	UNTREATED	8	11.7	52	1114.77		13	6	8		1	NA		8.0099	11.7	52	1114.77	
8.8899	UNTREATED	1	9.5	86	330.39		8	ទី	8		Ø	NA	9	0.0099	9.5	86	339.39	
0.0112	37 F	1	9.3	78	508.28	1	5.22	200	4.8%									
0.0103	44 F	G	13.2	40	2119.38	9	5.62	209	4.8%		6	209	4.82	8.0199	13.8	42	2683.48	ERR
0.0193	41 F	0	13.4	44	2189.25	8	4,8%	298	4.8%									
8.0112	37 F	9	13.8	45	1814.30	9	5.22	288	4.8%									
8.8181	40 F	8	13.4	41	2215.61	9	4.47	208	4.8%									
6.0114	46 F	8	14.2	42	2532.61	B		206	4.8%									
8.8196	57 F	8	14.6	45	1964.56	8	5.8%	200	4.8%									
9.9114	43 F	9	13.2	45	1654.65	9	4.8%	200	4.87									
0.0112	-38 F	č	13.3	41	2027.35	ų	5.22	298	4.8%									
0.0119 a atao	42 F	9 0	15.7	54 45	2233.67	8	4.2%	. 298 	4.82									
0.0102 a ataz	36 F 70 F	łi ł	19.8	40	2083.44	10	4.4%	200	4,87				(3. ON	3 64/3		75 8		
8.8184	/10 F 70 F	1	9.8 0 i	13	1/4.23	1	10.04	108	10.84		1	150	10.87	0.0110	9.9	/5.8	868.2	EKR
0.0114	12 F 10 F	1	0.0	0.3 70	1010.12	1	11.14	100	10.84									
0.0112 2 2190	67 F 19 C	1	10.7	/a 05	733.00	1	0 57	1.00	10.04									
a a (a)	60 ! 47 E	1	10.4	02 71	19:4 77	1	7.34	150	10.07									
B 6114	71 F	1	19.0	73	753 49	1	10.0%	150	10.04									
6.0104	79. F	Â	14.6	42	2359.91	Â	18.57	150	10.0%		ß	150	(3.97		16 5	74 5	3179 3	600
0.0114	72 F	9	16.4	31	3436.86	8	11.12	158	10.8%		v		10.0%	010110	10.0	5015	517215	Chit
0.0112	69 F	e	16.8	37	3252.92	8	11.17	159	19.87									
8.8116	71 F	8	16.2	39	2696.27	9	16.9%	150	10.8%									
0.0108	68 F	9	18.5	35	3823.02	Ø	9.5%	150	10.8%									
0.8106	67 F	8	16.6	36	3464.99	3	11.5%	150	18,8%									
6.8108	73 F					8	11.17	150	19.8%									
8.8168	73 F					1	11.17	158	18.87									
0.0113	57 F	1	10.6	72	726.20	i	8.2%	150	7.7%		1	150	7.7%	0.0112	12.6	75.9	617.2	ERR
8.8119	60 F	1	9.5	7 <del>0</del>	575.45	1	8.4%	150	7.7%									
0.01ti	61 F	1	9.9	78	55 <b>0.</b> 27	i	8.07	150	7.7%									
9.3110	59 F	1	19.8	76	643.39	i	6.8%	150	7.7%									
0.0191	· 62 F	i	9.9	77	615.76	1	7.6%	150	7.7%				,					
0.0107	58 F	1	11.6	76	710.26	1	7.2%	150	7.7%									
0.8121	56 F	1	26.8	82	499,48	1	8.3%	150	7.7%									
9.9111	61 F	0	15.6	38	2625.59	9	8.8%	150	7.7%		8	150	7.7%	0.0112	15.4	39.5	2498.2	ERR
6.0118	54 F	ال م	14.5	3/	2589.25	8	6.8%	158.	/./%									
0.01/1 0.01/1		Ю л	1/.2	40 40	2406.96	Ŭ	5.34	100	1.14									
0.0101 0.0407	67 F 50 E	U A	10.7	410	0100.7. 1787 CO	S a	1.0%	128	7.7%									
0.0107	38 F 10 E	10 01	14.2	42 40	2233.07	U Gi	7.2A 0.47	150	ייי / ייע אר ד									
0.0117	00 F 57 C	o a	13.2	40 70	2141.00	o e	0.44	150 158	7.7%									
0.0113 0.9105	55 F	1	9 Q	57 87	197 91	1	4 17	150	1 57		í	159	A 57	9 9120	(0) 1	0.00 +	409 0	COD
P 2094	47 F	1	9.3	77	551.98	i	5 2%	158	4.57		1	100	7.05	0.0107	10.1	00.1	7/0.0	Criti
6.0120	48 F	1	9.5	80	476.15	1	0124	150	4.5%									
9.0117	54 F	1	19.9	82	531.87	1	4.07	158	4.5%									
0.0111	50 F	1	9.4	76	473.43	-	4.27	150	4.5%									
6.0123	52 F	i	11.1	84	454.95	1	4.9%	150	4.5%									
0.0115	51 F	1	18.6	80	464.52	1	4.62	150	4.5%									
0.0123	52 F	9	17.0	37	2545.94	0	4.9%	158	4.5%		8	150	4.5%	8.8169	14.7	40.7	2190.5	ERR
0.0195	55 F	8	15.2	38	2552.03	8	4.17	158	4.5%									
0.0117	54 F	8	13.6	43	1949.91	8	4.8%	150	4.5%									

												FULI	ER IF	3237	F SERIES (	GROUP	AVERAG	ES	
										GROUP									
VERAGE				LOAD	STRAIN	MODULAS		ACTUAL		AVERAGE	PILLING			PERCENT	AVERAGE	LOAD	STRAIN	MODULAS	FILLING
HICKNESS	SAMPLE	CO	DIR	AV6	AV6	AV6	DIR	PICKUP	SPEED	PICKUP	TEST	DIR	SPEED	PICKUP	THICKNESS	AV6	AV6	AVG	AVG
0.0099	UNTREATED		9	11.7	52	1114.77		8	9	9		1	NA	0	0.0077	11.7	52	1114.77	
8.0099	UNTREATED		i	9.5	86	338.39		8	8	0		8	NA	0	0.0899	9.5	86	338.39	
<b>e.9</b> 874	47	F	8	13.7	43	2006.40	Ø	5.2%	150	4.5%									
0.2130	48	F	3	14.2	42	2261.93	Ø		150	4.5%									
<b>3.</b> 0111	50	F	ō	13.7	38	1984.97	ß	4.27	150	4.5%									
0.0115	51	F	Ø	15.8	45	2041.13	8	4.6%	150	4.5%	•								
<b>8.81</b> 13	53	F					0	4.6%	158	4.5%									
0.0113	22	F					i	4.6%	158	4.5%									

#### APPENDIX 4. MULLEN BURST AND FILLING TEST RESULTS FOR ALL GENERATED FABRICS

			AVERAGE	AVERAGE	AVERAGE	AVERAGE		AVERAGE	SPEED
			LEFT	CENTER	RIGHT	SHEET	PILLIN6	PICKUP	
F	10 :			45.4	39,5	42.4	4.8	18.7	200.0
F	11		43.3	39.8		41.6	4.0		
F	12			43.1	56.3	49.7	5.0		
F	13 :			42.4	43.1	42.7	4,0		
F	14		56.4	41.2		48,8	4,0		
F	15			40.4	49,4	44,9	3.0		
F	16		43.6	46.4		45.0	4.0		
F	17 1			39,8	49,9	44.8	5.0		
F	18 1		47.3	37.6		42.5	5,0		
F	21			36.9	43,4	48.1	3.8		
		AVERAGE	47.7	41.3	46.9	44.3	4.1		
F	25		45.7	37.5		41.6	4.8	7.7	200.0
F	26 3			43.2	46.2	44,7	2.0		
F	27 1		51.7	44,4		48,0	2.0		
F	29			47.9	42.1	45.0	2.0		
F	30			41,5		20.8	3.0		
F	31 1			39,5	42.7	41,1	5.0		
F	32		44.6	41.3		43,0	5.6		
F	33 1			51.7	51,9	51.8	4.0		
F	34		52.3	44.9		48,6	5,0		
F	35 1			42.3	45.3	43,8	4.0		
		AVERAGE	48.6	43.4	45.6	42,8	3.6		
F	36 ;			43.8	43.7	43.8	3.8	4,8	200.0
F	37		47.7	39.0		43.4	4.0		
F	38			44,4	45.5	45,0	4.0		
F	39 1		50.8	37.8		44.3	4.0		
F	40			38.2	43.1	40.7	3.0		
F	41		48.1	38.1		43.1	5.0		
F	42 3			44.2	47,0	45,6	4.0		
F	43		46.7	38.i		42.4	2.0		
F	44	ţ		41.8	39.5	40.6	3.0		
F	45	1	50,4	45.2		47.8	4,8		
		AVERAGE	48.7	41.1	43.8	43.7	3.6		
F	46	1		41.5	45.0	43.3	4.8	4.5	150.0
F	47		40,3	37.4	_	38.8	4.0		
F	48	i I		40.0	39.2	39.6	4,8		
F	49	5	50.7	49,3		50,0	4,0		
F	50	1		46.9	54,1	50.5	5.0		
F	51	i i	47.9	40.1		44.0	4.0		
F	52	í I		44,8	42.4	43.6	4,8		
F	53	ł	50.5	40.5		45.5	4.0		
F	54	1		38.8	47.1	43.0	4.0		
F	55	<b>B</b> F	43,8	41.6		42.7	3.0		
		AVERAGE	46.6	42.1	45.6	44.1	4.8		

			AVERAGE	AVERAGE	AVERAGE	AVERAGE		AVERAGE	SPEED
			LEFT	CENTER	RIGHT	SHEET	FILLING	PICKUP	
F	56 (			49.9	46.3	48.1	5.0	7.7	150.0
F	57 1		51.8	41.6		46.7	4,0		
F	58			43.8	44.6	44.2	5.0		
F	59		38.2	43.7		41.0	4.0		
F	691 I			50.0	49.7	49.6	5.0		
F	61		48.5	36.0		42.3	4.4		
ŗ	42 1		1010	10 7	â <b>ā</b> 7	42.5			
. Г С			AE 7	78.1	,,,,	7213 A1 2	7.U A G		
г. т	181		4 <b>4</b> .0	37.7 A7 E	11 7	41.D 83 4	7.0		
F F	· 64 i		47.6	- 410-D	44,1	44.1 	4.Vi 4.3		
ŕ	60 i		4/.0	41.8		44,4	4.0		
		AVERAGE	46.2	42.8	45.9	44.4	4.3		
F	67		49.8	51.6		50.7	4.0	10.8	150.0
F	68 (			37.5	54.4	46.0	4.0		
F	69		52.1	39.5		45,8	4.0		
F	70			40.3	40.7	40.5	3.8		
F	71		41.7	47.7		44.7	4.0		
F	72 1			42.8	58.4	46.6	5.0		
E.	77 1		49 A	79 S		44 7	A. 0		
Ē	74			40 5	47 A	A1 5	4 0		
r C	77 1		A.A. A	-10,0 At G	72.0	47.5 AZ 1	4 Q		
r r	131		77,7	71.7	3 A A	. 70.1	T - D A D		
F	/ō i			07.0	44.9	42,1	4.0		
	A	VERAGE	47.5	42,1	46.5	44.6	4,0		
FB	1			35.7	36.i	35.9	5.8	4.9	200.0
FB	24			36.7	43.9	40.3	5,0		
FR	3;			46.0	40.8	43.0	5.0		
FB	. 4 1			45.0	44.5	44.8	5.0		
FB	5 i			40.9	41.9	41.4	5.0		
FB	6 1			39.3	40.8	40.6	5.0		
FB	7			47.5	43.3	45,4	5.0		
FR	8 1			47.3	41.1	44,2	5.0		
FR	9 1			39.1	42.7	40.9	5.0		
FB	10			41.4	42.0	41.7	5.0		
•		AVERAGE		41,9	41.6	41.8	5.0		
77 D	4.4 1			2 G A	74 ik	ר סד	E D	7 1	<b>788 8</b>
1 B	11 i 10 i		47 7	48.4 AD C	00.U	30.2	יים 13יר	1.0	70 <b>0.</b> 0
F 8	12		40.0	40.3 7/ 4	76 7	41.7 7/ 0	3.0 c 7		
1 H	15		/	35,4	32./	35.6	5.10 		
۲â	14 1		39.1	42.9		41.0	5.K		
FB	15 5		_	44,1	48.8	45,5	5.0		
FB	16 1		42.7	36.7		39.7	5.0		
FB	17 1			41.3	34,4	37.8	5.0		
FB	18		38.0	38.5		38.3	5.0		
FB	19			39.6	39,5	39.5	5.0		

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			AVERAGE	AVERAGE	AVERAGE	AVERAGE		AVERAGE	SPEED
			LEFT	CENTER	RIGHT	SHEET	PILLING	PICKUP	
FB	20		36.8	38.5		37,6	5.0		
		AVERAGE	40.0	39,9	38,9	39.7	5.0		
FB	21			35.9	39.5	37.7	5.0	18.0	200.0
F₿	22		38.4	44.6		41.5	5.0		
FB	23 (			40.3	38.2	39.3	5.0		
FB	24 ;		42.9	37,4		40.1	5.0		
FB	25 1			35.9	35.9	35.9	5.0		
FB	26		36.7	35,5		36.1	5.0		
FB	27			39,9	37.1	38,5	5.0		
F8	28		40.3	37.6		39.0	5.0		
FB	29			39.9	37.8	38.6	5.0		
FB	30 1		43.6	38.5		41.0	5,0		
		AVERAGE	40.4	38.5	37.7	38.8	5.0		
	<b></b> .			75 7	<b>7</b> 5 b	7.4			
F B	51			32.J	35.9	4,1	3.0 	10.1	150,0
₽B	32 5		42,8	45.1		44.0	5,6		
FB	33 1			40.0	46.0	46.0	5.8		
F8	34 3		42.2	42.8		42.5	2.10		
FB	35 1			44.4	57.9	41.1	5.6		
FB	36		37.0	41.1		39.0	5.0		
FB	37			33.9	37.3	35.6	5.0		
FB	38 1		39,7	38.i		38,9	4.0		
FB	39 1			42.3	39.7	41,0	5.0		
FB	40		36.5	35,0		35.8	5.8		
		AVERAGE	39.6	39.5	38.2	39.2	4.9		
FR	41 1			38.7	39.5	39,1	5.0	7.8	150.0
FB	42 1		45.4	36.2		40.8	4,0		
FR	43 (			48.5	34.7	37.6	5.0		
FR	44 1		43.7	34.8		39.3	5.0		
FR	45 (			40.4	36.4	38.4	5.0		
FR	46.1		35.4	37.8		36.6	5.0		
FR	47 !			37.2	36.9	37.1	5.0		
FR	48 1		43.8	41.8		42.8	5.0		
FR	49 1			47.9	37.8	42.8	5.0		
FB	50 ;		43.8	38.5		41.1	5.6		
		AVERAGE	42,4	39,4	37.1	39.6	4.9		
FB	51 1			51.3	39.2	45.3	5,8	5.1	150.8
FR	57 1		39.7	53.3		46.5	5.6		
FR	53			58.6	41.3	50.0	5.0		
FR	54 !		47.5	48.3		45.4	5.0		
FR	55 1		( <b>-</b> , <u>-</u> , <u>-</u> ,	34.4	36.0	35.2	5.8		
6 B	54 1		<b>4</b> 1 (	47.8	0010	47.2	5.9		
5 D E D	50 1		72.02	AA A	39.4	43. A	5.0		
E B B	59 1		41 7	53.2	Sec. 1 M	47.2	5.0		
ΥĽ	30 )		71.1	2001	<i>I</i>	-3	ere		
					4	-0			

				AVERAGE	AVERAGE	AVERAGE	AVERAGE		AVERAGE	SPEED
				LEFT	CENTER	RIGHT	SHEE 7	PILLING	PICKUP	
FB	59	1			48.2	45.0	46.6	4.0		
FB	6 <b>0</b>	ļ		39,9	47.4		43.6	5.0		
			AVERAGE	40.9	48,4	40.2	44.5	4,9		
F8-5	61	ł			50.4	39.6	45.0	5.8	5.3	200.0
FB-S	62	į		41.5	53.3		47.4	5.0		
FB-S	63	ţ			42.6	48.4	45.5	5,8		
FB-S	64	ł		47.8	51.5		49.6	5.0		
FB-S	65	Ì				47.8	23.5	5.0		
FB-S	66	ţ		36.7	48.2		42.5	4.0		
FB-S	67	ļ,			42.8	39.3	41.0	4.0		
F8-5	68	i		43.6	49.3		46.5	5.0		
FB-S	69	÷				43.2	21.6	5.8		
F8-5	70	;		46.1	49.2		47.7	4.0		
			AVERAGE	43.1	48.4	43.5	41.0	4,7		

FY	1 1		36.6	39.2	37.9	4.0	7.0	200.0
FY	2 (	48.5	48.2		48.4	3.0		
FŸ	3 1		38.7	42.9	40.8	4.0		
FY	4 i	45.3	33,4		39.3	4.0		
FΥ	5 ;		39.8	45.6	42.7	4.0		
FΥ	6	38.2	45.6		41.9	5,6		
F¥	7 1		31.8	46.6	39.2	4.0		
FY	8 🕴	35.4	44.5		40.0	5.0		
F۲	9		42.8	48.4	45.6	3.0		
FY	10 ;	25.7	31.i		28.4	4,0		
	AVERAGE	38.6	39.3	44,5	40.4	4.0		
FY	11		37.5	42.2	39.9	3,8	5.5	200.0
FY	12 /	44.8	35.1		40.0	3.0		
FY	13 1		38,4	31.2	34.8	3.0		
FΥ	14	41.2	41.3		41.3	4.0		
FY	15 1		35.6	37.7	36.7	5.0		
FY	16 ;				6.8	4.0		
F¥	17				6.6	4.6		
FY	18 ;				0.0	4,8		
FY	19				0.0	4.0		
FΥ	20				0.0	4.0		
	AVERAGE	43.0	37,6	37 <b>.0</b>	19.3	3.8		
FY	21				0.0	5.0	19.1	200.0
F¥	22				0.0	4.0		
FY	23 1				0.0	5.0		
FY	24				6.6	5.0		
					4-4			

		AVERAGE	AVERAGE	AVERAGE	AVERAGE		AVERAGE	SPEED
		LEFT	CENTER	RIGHT	SHEET	PILLING	PICKUP	
EV	75 1				a a	7 0		
EV	22 1				0,0 A A	0.0 6 0		
Ē¥	20 1				0.0 0 0			
FV	28 1				a a	4 D		
FV	29 !				6.0 A.0	4.0		
FY	30		41.1	48.5	44.8	4.0		
	AVERAGI	E	41.1	48.5	4.5	4.3		
<b></b>	71 1				а а	4.0	7.4	
F 1 E 2	31 i 73 i				V.0 . 0	4.0 E G	/.4	196.6
τ¥ τν	3Z i 77 i				0.0	J.0 7 0		
F 1 F 2	33 ) 74 (		2 j J	44 G	10.10 AG 1	0.10 A G		
FY FV	34 i 75 i		01.4 55.0	44.C 53 /	40,1 Et 0	4.0 / D		
FY FV	30 : 7/ I		57.0 57.0	31.0	01.0 10 /	4.0 E A		
F1 FV	00 I 77 I		07.0 A7 A	44.J 83.4	43.0 40.0	3.10 A D		
E ! EV	3/ 1 70 i	4D 4	40.4	42,1	42.0	7.D		
₹Υ EV	26 i 70 i	47:4	3177 2077	A/ D	10.J 17 7	0.0 A D		
רז דע	37 i 60 i		40,0 51 5	40.0 A7 G	4/.J E. J	4.0 1 a		
r î rv	4942) 1 A 1 1	70.0	びきょい フォート	4/.0	30.0 77 E	4.0 7.0		
71	+ 1 +	22.7	04+1		00.0	0,0		
	AVERAGE	41.2	48.6	46. <b>0</b>	33.9	3.9		
FY	42		44.i	37.3	40.7	3.0	4.9	150.6
FΥ	43 1	48.3	38.3		43.3	4.0		
FΥ	44		47.2	41.3	44.2	4.8		
FΥ	45 1	39.9	42.6		41.3	5.0		
FΥ	46		44.3	48.4	42.3	4.6		
FY	47	36.5	38.8		37.7	5.0		
FΥ	48 ;		41.7	33.2	37.4	3.6		
F¥	49	41.5	46.6		44.1	4.0		
FY	50		41.4	36.5	39.0	4.0		
FY	51 (	31.8	44.8		37.9	4.0		
	AVERAGE	39.6	42.9	37.7	40.8	4.0		
FY	52 (		29.6	31.8	30.7	5.0	9.8	150.0
Fγ	53 (	35.7	37.6		36.6	4.0		
F۲	54 (		39,7	38.8	39.3	5.0		
FY	55 1	40.0	39.2		39.6	5.0		
FY	56 1		37.1	40.3	38.7	4.0		
FΥ	57	32.3	34.6		33.4	5.0		
FY	58 1		31.8	30.7	31.3	4.0		
FΥ	59 1	36.4	36.7		36.6	4.0		
FY	60		35.7	37.1	36.4	4.0		
FY	61 3	34.5	34.0		34.2	4.0		
			·		7	2 *		
	AVERAGE	: 35.8	35.6	ა5.7	35.7	4,4		

44.1 22.1 4.0 10.2 200.0

			AVERAGE	AVERAGE	AVERAGE	AVERAGE		AVERAGE	SPEED
			LEFT	CENTER	RIGHT	SHEET	PILLING	PICKUP	
F	2 1			46.0	43.3	44.6	4.0		
F	3 (				43.0	21.5	4.8		
F	4			47.Ø	57.4	49.7	5.0		
Ę	5 3			41.2	46.1	43.7	3.0 7.0		
Ę	6			44.2	45.6	44.9	4.17		
F	7			41.0	47.9	44.5	3.0		
F	8			46.6	46.5	46.5	4.0		
F	9 !		57.0	47.9	1010	52.0	4.2		
E	11		0.10	50.6	46.3	48.5	4,0		
		AVERAGE	57.0	45.5	46.1	41.8	3.9		
Ē	12 3			53. <b>0</b>	49.0	51.0	4.0	7.7	200.0
E	13			54.0	55,6	54.8	5.0		
Ē	14			44.6	43.6	44.1	5.0		
Ē	15			39.2	43.6	41,4	4,0		
F	16					0.0	4.0		
Ē	17			45.4	45.3	45.3	4,8		
F	18			43.0	41.7	42.4	4.8		
F	19			39.6	46.0	42.8	4.0		
Ē	20			48.8	47.5	48.1	5.0		
Ē	21			42.6	45.9	44.3	5.0		
Ε	22			42.0	45.6	43.8	4.0		
		AVERAGE		45.2	46.4	41.6	4,4		
E	23			44.0	39.8	41.9	3.0	4.7	208.0
Ε	24			48.8	45.0	46.9	3.0		
Ē	25 8			43,4	40.6	42.0	4.0		
Ε	26			46.2	48.0	47.1	3.0		
E	27			43.8	40.7	42.3	3,0		
Ε	28			64.6	48.7	56.6	4.0		
Ē	29			43.4	45.8	44.6	4.0		
£	30 (			41,4	38.4	39.9	4.0		
Ε	3i	•		42.2	49.2	45.7	4.0		
Ε	32			48.2	48.8	44.5	4.0		
		AVERAGE		46.6	43.7	45.1	3.6		
Ē	33			40.8	47.6	44.2	4.8	4.7	150.8
Ε	34	1		48.2	46.7	47.5	5.0		
E	35			44.8	45.2	45.0	5,0		
Ē	36	é 3		45.2	43.7	44.5	5.0		
E	37	}		48.2	45.2	46.7	4.8		
Ē	38	<b>i</b>		54.4	43.3	48.8	4.0		
E	39			46.0	44.6	45.3	4,0		
Ε	40	i		41.4	38.2	39.8	3.0		
E	41			45.4	38.3	41.8	4.8		
Ε	42	1		42.0	45,7	43.9	3.0		
		AVERAGE		45.6	43. <i>4</i>	44.7	4.1		

		AVERAGE	AVERAGE	AVERAGE	AVERAGE		AVERA6E	SPEED
		LEFT	CENTER	RIGHT	SHEET	PILLING	PICKUP	
F	43 1		48.4	45.0	46.7	3.0	7.7	150.0
F	44 (		49,4	47,7	48.5	4.0		
Ē	45		44,4	44.6	44.5	4.0		
Ē	46 (			40.6	20.3	5.8		
E	47 }		47.2	46.7	47.0	4,0		
Ē	48		51.7	44.5	48.1	4.0		
E	49		47.2	46.6	46.9	4.0		
Ē	50		46.8	45.6	46.2	5.0		
Ē	51 (		55.6	48.0	51.8	5.ð		
E	52 (		52.2	44.5	48.4	4.0		
	AVERAGE		49.2	45.4	44.8	4.2		
E	53 (			46.9	23.4	5.8	18.0	158.8
Ē	54 1		46.8	48.1	47.5	5.0		
Ē	55		57.2	44.3	50.8	4.0		
E	56		63.9	48.3	56.1	3.0		
Ē	57 ;		58.6	44.4	51.5	4.0		
Ε	58		44.0	48.3	46.1	4.0		
Ē	59		51.7	51.4	51.5	3.0		
E	60 (		51.2	51.0	51.1	4.0		
Ē	61 1			43.3	21.6	4.0		
Ē	62 }		48.2	46.4	47.3	4.0		
	AVERAGE		52.7	47.2	44.7	4,0		

ΕZ	1 1		54.2	52.9	53,5	5.0	7.1	200.0
F7	2 1		45.3	54,9	50.1	5.0		
F7	3		46.4	48.0	47.2	5.0		
F7	4 :	54.6	43,5		49.8	5.0		
F7	53		42.6	58.2	50.4	4.0		
F7	6.1	53.6	46.1		49.9	5.0		
F7	7 1		47.1		23.6	5.0		
F7	8	50.1	46.2		48.2	5.0		
F7	9	_	45.8	50.2	48.8	5.0		
EZ	10	56.8	42.2		49.5	5.0		
	AVERAGE	53,8	45.9	52.8	46.9	4.9		
F7	AVERAGE	53,0	45.9 45.9	52.8 53.8	46.9 49.8	4.9 5.0	5.4	200.8
EZ F7	AVERAGE	53.8 48.4	45.9 45.9 41.9	52.8 53.8	46.9 49.8 45.1	4.9 5.0 5.0	5.4	200.0
EZ EZ F7	AVERAGE	53.8 48.4	45.9 45.9 41.9 48.0	52.8 53.8 47.6	46.9 49.8 45.1 47.8	4.9 5.0 5.0 5.0	5.4	200.0
EZ EZ EZ	AVERAGE	53.8 48.4 58.4	45.9 45.9 41.9 48.0 47.3	52.8 53.8 47.6	46.9 49.8 45.1 47.8 52.8	4.9 5.0 5.0 5.0 5.0	5.4	200.0
EZ EZ EZ E7	AVERAGE	53.8 48.4 58.4	45.9 45.9 41.9 48.0 47.3 45.0	52.8 53.8 47.6 46.0	46.9 49.8 45.1 47.8 52.8 45.5	4.9 5.0 5.0 5.0 5.0 5.0 5.0	5.4	200.0
EZ EZ EZ EZ EZ	AVERAGE	53.8 48.4 58.4 48.6	45.9 45.9 41.9 48.0 47.3 45.0 40.8	52.8 53.8 47.6 46.0	46.9 49.8 45.1 47.8 52.8 43.5 43.5	4.9 5.0 5.0 5.0 5.0 5.0 5.0 5.0	5.4	200.0

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		AVERAGE	AVERAGE	AVERAGE	AVERAGE	AVE	RAGE	SPEED
		LEFT	CENTER	RIGHT	SHEET PIL	LING PIC	KUP	
EZ	18 ;	60.6	40.1		50,4	5.0		
ΕZ	17		42.5	53.2	47.9	5.0		
EZ	20	54.2	47,8		51.0	5.0		
	AVERAGE	54,8	43.9	58.7	48.1	5.8		
EZ	21 )	48.8	48.8		48.8	4.0	7.9	150.0
ΕZ	22		49.6	45.4	47.5	5.0		
E2	23 1	51.6	53.2		52.4	5.8		
EZ	24 1		58.8	51.8	55.3	5.0		
ΕZ	25 )	54.6	54.6		54.6	5.0		
ΕZ	26 (		51.2	46.4	48.8	5.0		
ΕZ	27	45.2	47.4		46.3	5.0		
ΕZ	28		53.6	51.0	52.3	4.0		
ΕZ	29 (	56.4	58.0		57.2	5,8		
	AVE <b>RA</b> GE	51.3	52.8	48.6	51,5	4.8		

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#### APPENDIX 4. MULLEN BURST AND FILLING TEST RESULTS FOR ALL GENERATED FABRICS

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		AVERAGE	AVERAGE	AVERAGE	AVERAGE		AVERABE	SPEED
		LEFT	CENTER	RIGHT	SHEET	PILLIN6	PICKUP	
F	10 1		45.4	39.5	42.4	4.8	18.7	200.0
F	11 1	43.3	39.8		41.6	4,8		
F	12 1		43.1	56.3	49.7	5.0		
F	13		42.4	43.1	42.7	4.8		
F	14 !	56.4	41.2		48.8	4.8		
F	15 1		40.4	49.4	44,9	3.8		
۲ ۲	16 1	43.6	46.4		45.0	4.0		
r r	17 !		39.8	49,9	44.8	5.8		
r F	19 1	47.3	37.6		42.5	5.0		
F	21 1		36.9	43.4	40.1	3. <b>8</b>		
	AVERAG	47.7	41.3	46.9	44.3	4.1		
r	95 I	45.7	37.5		41.6	4.8	7.7	200.0
r E	23 i 02 i	1211	43.2	46.7	44,7	2.0		
r r	20 1	51 7	44.4	1011	48.8	2.8		
r r	2/ 1 50 i		47 9	47. i	45.8	2.0		
t r	27 i 70 i		At 5		20.8	3.6		
۲ ۲	348 i 74 i		705	42.7	At 1	5.9		
۲ ۲	31 1	8.8 <i>i</i>	37.3 Ai 3	72.7	AT 0	5.6		
} -	32 :	44.0	91.3 Et 7	51 Q	5t Q	4 Q		
ŀ	33 ; 74 )	E1 7	Ji./	21.7	48.6	5.0		
r F	34 :	32.3	42.3	*5.7 75.3	43.8	4.0		
	AVERAGE	E 48.6	43.4	45.6	42.8	3.6		
c	72 1		43.8	43.7	43.8	3.6	4.8	268.8
r r	77 1	47 7	39.0		43.4	4.0		
r r	37 1		44.4	45.5	45.8	4.0		
r r	30 (	50 8	37.8		44.3	4.8		
र स	57 i	0010	38.2	43.1	40.7	3.0		
r	780 I A 1 I	48 i	38-1		43.1	5.6		
r r	11 i 40 i		44.7	47.R	45.6	4.8		
r r	92 · 47 i	AL 7	גם <b>ו</b>		47.4	2.0		
r r	43 i	70.7	A1 9	76 5	40.4	τ.A		
F	44 ; 45 :	58.4	45.2	0110	47.8	4.8		
	AVERAGE	48.7	41.1	43.8	43.7	3.6		
F	46		41.5	45.0	43.3	4.8	4.5	159.9
F	47 1	40.3	37.4		38.8	4.8		
F	48 !		40.0	39.2	39.6	4.0		
F	49	50.7	49.3		58.6	4.0		
, F	58 !		46.9	54.1	50.5	5.8		
F	51 !	47.9	48.1		44.2	4.8		
۶ ۲	57 !	1/1/	44.8	42.4	43.E	4.8		
r E	57 I	50.5	4 <b>A</b> .5		45.5	4.6		
r F	54 !	5015	38.8	47.1	43.4	4.0		
F	55	43.8	41.6		42.7	3.0		
	AVERA	GE <sup>46.6</sup>	42.1	45.6	14.1	4.0		

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		AVERAGE	AVERAGE	AVERAGE	AVERAGE		AVERAGE	SPEED
		LEFT	CENTER	RIGHT	SHEET	PILLING	PICKUP	
F	56 ;		49,9	46.3	48.1	5.0	7.7	150.0
F	57 (	51.8	41.6		46.7	4.8		
F	58		43.8	44,6	44,2	5,8		
F	59 1	38.2	43.7		41.8	4.0		
F	68 1		50.0	49.2	49.6	5.0		
F	61 1	48.5	36.0		42.3	4,6		
F	62		46.2	44.7	42.5	4.0		
F	63 1	45.3	37,9		41.6	4.0		
F	64		43.5	44.7	44.1	4.0		
F	65 (	47.0	41.8		44.4	4.8		
	AVE	RAGE 46.2	42.8	45.9	44.4	4.3		
F	67	49.8	51.6		50.7	4,8	10.8	150.0
F	68 (		37.5	54.4	46.8	4,8		
F	69 (	52.1	39.5		45.8	4.0		
F	70		40.3	40.7	40.5	3.0		
F	71 1	41.7	47.7		44.7	4.6		
F	72 :		42.8	50.4	46.6	5.8		
F	73 1	49.6	39.8		44.7	4.0		
F	74		40.5	42.6	41.5	4.0		
F	75 1	44.4	41.9		43.1	4.0		
F	76 1		39.8	44.4	42.1	4.6		
	AVER	AGE 47.5	42.1	46.5	44,6	4.0		
FB	1 1		35.7	36.1	35.9	5.0	4.9	280.0
FB	2		36.7	43.9	40.3	5.8		
FR	2		46.8	40.8	43.0	5.0		
FB	4 1		45.0	44.5	44.8	5.8		
FB	5 1		48.9	41,9	41.4	5.0		
FB	6		39.3	40.8	46.6	5.0		
FB	7 1		47.5	45.5	43.4	5.0		
FB	8 1		47.3	41.1	44.2	5.8		
FB	9 i		39.1	42.7	40.9	5.0		
FB	10		41.4	42.B	41.7	5.0		
	AVE	RAGE	41.9	41.6	41.8	5.8		
FB	11		40.4	36.0	38.2	5.0	7.6	200.0
FB	12 1	43.3	48.5		41.9	5.0		
FB	13 1		36.4	35.7	36.8	5.0		
FB	14 ;	39.1	42.9		41.0	5.0		
FB	15 (		44.1	48.8	46,5	5.0		
FB	16 :	42.7	36.7		39.7	5,0		
F8	17 1		41.3	34.4	37.8	5.0		
FB	18	38.0	38.5		38.3	5.0		
FB	19 1		39.6	39.5	39.5	5.0		

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			AVERAGE	AVERAGE	AVERAGE	AVERAGE		average	SPEED
			LEFT	CENTER	RIGHT	SHEET	PILLING	PICKUP	
FB	20	•	36.8	38.5		37.6	5.8		
		AVERAGE	40.0	39,9	38.9	39,7	5,8		
FB	21			35.9	39.5	37.7	5.0	16.6	200.0
FB	22 ¦		38.4	44.6		41.5	5.0		
FB	23			40.3	38.2	39.3	5.0		
FB	24 1		42.9	37.4		40.1	5.0		
FB	25 I			35.9	35.9	35.9	5.0		
FB	26 ;		36.7	35.5		36.1	5.0		
FB	27			39.9	37.1	38.5	5.6		
FB	28		40.3	37.6		39.8	5.6		
FB	29 :			39.9	37.8	38,8	5.0		
FB	30 i		43.6	38.5		41.8	5.8		
		AVERAGE	40.4	38.5	37.7	38.8	5.0		
FR	31 !			32.3	35.9	34.1	5.6	10.1	15 <b>0</b> .A
FR	32 1		47_8	45.1		44.0	5.4		
F 10	77 !		,2,0	10,1 10,1	40.0	49. A	5.8		
FR	74 1		47 2	47.8		42.5	5.0		
FR	75 1		7414	44.4	37.9	41.1	5.4		
FD	76 1		<b>7</b> 7 0	<u>41</u> 1	Dit i	39.0	5.6		
E D	30 1		0/ <b>.</b> U	37.9	37-3	37.6	5.2		
г.р. С D	י <i>ו</i> כ זי מז		<b>70</b> 7	33,, 39 i		79 Q	ΔQ		
, D E D	100 1070		Q7.7	10.1 17 7	<b>TQ_7</b>	21 B			
FD FD	، (ن نا <u>رن</u>		36.5	35.8	0,11	75. â	5.0		
1.0	-71U i		0010			00,0	210		
		AVERAGE	39.6	39.5	38.2	39.2	4.9		
FA	41 1			38.7	39.5	39.1	5.0	7.8	158.0
FR	47		45.4	36.2		40.8	4.B		
FR	43			48.5	34.7	37.6	5.8		
FR	44 !		43.7	34.8		39.3	5.0		
FR	45 !			48.4	36.4	38.4	5,8		
FB	46 1		35.4	37.8		36.6	5.8		
FA	47 1			37.2	36.9	37.1	5.0		
FR	48 :		43.8	41.B		42.8	5.8		
FB	49 1			47.9	37.8	42.8	5.0		
FB	50 ;		<b>43.</b> B	38.5		41.1	5.8		
		AVERAGE	42.4	39.4	37.1	39.6	4.9		
F D	51 1			51 3	39.2	45.3	5.8	5.1	150-8
гр Ср	י גר ו ליק		70 7	57.7	071L	46.5	5.0		
го Со	JZ i 57 i		37.1	50.5 50 L	<b>4</b> 1 र	-0,0 -50,0	5.0		
F Ø F Ø	JJ i Ed i		17 S	10.0 10.7	4114	4 7 <u>8</u>	5.0 5.0		
rø CD	J# 1 55 1		74.5	70.0	0 A7	4.5r ر عز	2.10 5.02		
F Ø E D	JJ i EL I		A1 7	17 B	30,0	10 C	5.0 5.0		
F Ø C D	30 5		41.0	7320 AL A	70 L	72.2 A7 0	5.2		
г Ø Г D	J/i SQI		A1 0	70.7 57 7	2 F # M	13.0 A7 7	5.D 5.D		
ГÐ	10 i		71.2	JJ.Z	A 11	7/.2	0,10		
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			AVERAGE	AVERAGE	AVERAGE	AVERAGE		AVERAGE	SPEED
			LEFT	CENTER	RIGHT	SHEET	PILLING	PICKUP	
FB	59 1	•		48.2	45.0	46.6	4.0		
FB	60 ;		39.9	47.4		43.6	5.0		
		AVERAGE	46.9	48.4	48.2	44.5	4,9		
FB-S	61 1			50.4	39.6	45.0	5.8	5.3	200.0
FB-S	62		41.5	53.3		47.4	5.8		
FB-S	63 1			42.6	48.4	45.5	5.6		
F8-S	64 1		47.8	51.5		49.6	5.0		
F8-S	65 i				47.0	23.5	5.8		
F8-S	66 ;		36.7	48.2		42.5	4.8		
FB-S	67			42.8	39.3	41.8	4.0		
FB-S	68 1		43.6	49.3		46.5	5.0		
F8-5	69 ;				43.2	21.6	5.6		
F8-S	70 ¦		46.1	49.2		47.7	4.8		
				40 4	A7 5	A 1 0	47		

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FY	1 :		36.6	39.2	37.9	4.0	7.8	200.0
FΥ	2 :	48.5	48.2		48.4	3.0		
FY	3 (		38.7	42.9	40,8	4,0		
FΥ	4 1	45,3	33.4		39.3	4.6		
Fγ	5 :		39.8	45.6	42.7	4.0		
FY	6 1	38.2	45.6		41.9	5.8		
FY	7		31.8	46.6	39.2	4.0		
FY	8 ;	35.4	44.5		40.0	5.6		
FY	9;		42.8	48.4	45.6	3,0		
FY	10 ;	25.7	31.1		28.4	4.0		
	AVERAGE	38.6	39.3	44.5	48.4	4.0		
FY	11 :		37.5	42.2	39,9	3.0	5.5	200.0
FY	i2 :	44.8	35.i		40.0	3.0		
FY	13 1		38.4	31.2	34.8	3.0		
FY	14 :	41.2	41.3		41.3	4.0		
FY	15		35.6	37.7	36.7	5.0		
FY	16 :				6.0	4.0		
FY	17				8.0	4.8		
FY	18 1				8.0	4.8		
FY	19				8,8	4.8		
FY	20				0.0	4.0		
	AVERAGE	43.0	37.6	37.0	19.3	3,8		
FY	21				8.6	5,8	10.1	200.0
F¥	22 1				8.8	4.0		
FY	23 1				6.8	5.6		
FY	24 ;				0.0	5.0		
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		AVERAGE	AVERAGE	AVERAGE	AVERAGE		AVERAGE	SPEED
		LEFT	CENTER	RIGHT	SHEET	PILLING	PICKUP	
FV	75 ! ·				a a	र. <b>9</b>		
FΥ	76 5				0.0 0.0	4.0		
FΥ					0.0 0.0	5.8		
FV	28 :				R. A	4.0		
FV	29 1				R. R	4.0		
FY	30 :		41.1	48.5	44.8	4,8		
	AVERAGE		41.1	48.5	4,5	4.3		
۳V	71 i				a 0	A D.	7.4	150 0
rt rv	01 ) 73 1				6.0	9.0 5.0	/.4	130.0
FT FV	32 i 77 i				0.C	3.0 7 8		
F 1 P 0	८८ । 74 ।		5i /	44 G	0.6 40 1	3.10 A G		
FT EV	04 i 75 i		51.4 53.0	44.0 51 2	49,1 E4 0	4.0		
71 CV	33 : 7/ 1		32.0 57 0	0,1L 5 66	01.0 40 1	4.6 5 a		
FT FV	30 i 77 i		JZ.0 47 A	44.J 47 1	40.0 40.0	3.10 A Di		
f f rv	3/1 701	10 A	7017	42.1	42.0 53 5	4.0 7 0		
ΓΪ ΓV	38 i 70 i	47:4	JI./ 10 i	AL 0	JB.J 17 7	3.18 A D		
F1 mv	37 i 80 i		40.0 51 5	40.0	47.3 COLO	4.0 1 3		
FT EV	910 i 4 i i	70 0	ሀቀምር የሐሳ	4/.0	अधः छ रर म्	7.0 7.0		
Γ1	41 ·	32.7	0711		00.0	010		
	AVERAGE	41.2	48.6	46.10	33.9	3.9		
FY	42 1		44.1	37.3	48.7	3.0	4.9	150.0
FY	43	48.3	38.3		43.3	4.8		
FY	44 :		47.2	41.3	44.2	4.8		
FY	45 :	39.9	42.6		41.3	5.0		
FY	46		44.3	46.4	42.3	4.8		
FY	47 1	36.5	38.8		37.7	5,0		
FΥ	48 1		41.7	33.2	37.4	3.8		
F۲	49 1	41.5	46.6		44.1	4.0		
FY	50 1		41.4	36.5	39.0	4.8		
FY	51 :	31.8	44.8		37.9	4.8		
	AVERAGE	۶.6	42.9	37.7	48.8	4.8		
FY	52 (		29.6	31.8	30.7	5.8	9.8	150.0
FY	53 1	35.7	37.6		36.6	4.0		
FY	54 :		39.7	38.8	39.3	5.0		
FΥ	55	40.0	39.2		39.6	5.0		
FY	56 1		37.1	40.3	38.7	4.0		
FΥ	57	32.3	34.6		33.4	5.0		
FY	58 1		31.8	30.7	31.3	4.8		
FY	59	36.4	36.7		36.6	4.0		
FY	60		35.7	37.1	36.4	4.0		
FY	61	34.5	34.0		34.2	4.0		
	AVERAGE	35.8	35.6	35.7	35.7	4.4		

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22.1 4.8 10.2 200.0

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		AVERAGE	AVERAGE	AVERAGE	AVERAGE		AVERAGE	SPEED
		LEFT	CENTER	RIGHT	SHEET	PILLING	PICKUP	
F	9 ! ·		46.9	43.3	44.6	4 Q		
F	3			43.0	21.5	4.9		
Ē	4 !		47.9	52.4	49.7	5.0		
Ē	5 ;		41.2	46.1	43.7	3.0		
Ε	6 1		44.2	45.6	44.9	4.B		
Ε	7 ;		41.8	47.9	44.5	3.0		
Ε	8 !		45.6	46.5	46.5	4.0		
Е	91	57.0	47.8		52.0	4.8		
E	11		50.6	46.3	48.5	4.0		
	AVERAGE	57 <b>.0</b>	45.5	46.i	41.8	3.9		
Е	12 ;		53.0	49.0	51.8	4.0	7.7	200.0
Ē	13		54.0	55.6	54.8	5.0		
Ε	14		44.6	43.6	44.1	5.0		
Ε	i5 (		39.2	43.6	41.4	4.0		
Ε	16				0.0	4.8		
Ε	17 ;		45.4	45.3	45.3	4.8		
Ε	18 :		43.0	41.7	42.4	4.0		
Ε	19 :		39.6	46.0	42.8	4,8		
Е	28		48.8	47.5	48.1	5.8		
Ε	21 1		42.6	45.9	44.3	5.0		
E	22		42.0	45.6	43,8	4.8		
	AVERAGE		45.2	46.4	41.6	4.4		
Е	23 (		44.8	39.8	41.9	3.6	4.7	208.0
E	24 1		48.8	45.0	46.9	3.0		
Е	25		43.4	40.6	42.0	4.0		
Ε	26		46.2	48.0	47.1	3.8		
Ε	27 1		43.8	40.7	42.3	3.0		
Е	28 1		64.6	48.7	56.6	4.8		
E	29		43.4	45.8	44.6	4.0		
E	30 1		41.4	38.4	39.9	4.8		
E	31 ;		42.2	49.2	45.7	4.8		
Ε	32		48.2	40.8	44.5	4.8		
	AVERAGE		46.6	43.7	45.1	3.6		
E	33 ;		48.8	47.6	44.2	4.8	4.7	150.8
Е	34 1		48.2	46.7	47.5	5.0		
E	35 1		44.8	45.2	45.0	5.0		
E	36 1		45.2	43.7	44.5	5.0		
Е	37 (		48.2	45.2	46.7	4.0		
E	38 (		54,4	43.3	48.8	4,8		
Е	39 1		46.0	44.6	45.3	4.0		
Е	40 :		41.4	38.2	39.8	3.8		
Ε	41 1		45.4	38.3	41.8	4,8		
£	42		42.0	45.7	43.9	3.8		
	AVERAGE		45.6	43.9	44.7	4.1		

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		AVERAGE	AVERAGE	AVERAGE	AVERAGE		AVERAGE	SPEED
		LEFT	CENTER	RIGHT	SHEET PI	LLING	PICKUP	
F	- 43 :		48.4	45.0	46.7	3.0	7.7	150.8
ç	44 :		49.4	47.7	48.5	4.0		
F	45 1		44.4	44.6	44.5	4.0		
F	46 :			48.6	28.3	5,6		
Ē	47 :		47.2	46.7	47.0	4.0		
Ē	48		51.7	44.5	48.1	4.0		
Ē	49		47.2	46.6	46.9	4.0		
E	58		46.8	45.6	46.2	5.0		
E	51		55.6	48.6	51.8	5.0		
E	52		52.2	44.5	48.4	4.8		
	AVERAGE		49.2	45,4	44.8	4.2		
Ē	53 :			46.9	23.4	5.0	18.0	150.0
Ε	54		46.8	48,1	47.5	5.0		
Ε	55 1		57.2	44.3	50.8	4.0		
Ε	56 1		63.9	48.3	56.1	3.8		
Ε	57 1		58.6	44.4	51.5	4.0		
Ε	58 :		44.0	48.3	46.1	4.2		
Ē	59		51.7	51.4	51.5	3.0		
Ē	68 1		51.2	51.8	51.1	4.8		
Ε	61 :			43.3	21.6	4.8		
Ε	62		48.2	46.4	47.3	4.0		
	AVERAGE		52.7	47.2	44.7	4.8		

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ΕZ	1		54.2	52.9	53,5	5.0	7.1	200.0
ΕZ	2 ;		45.3	54.9	56.1	5.0		
٤Z	3 1		46.4	48.0	47.2	5.0		
ΕZ	4 1	54.6	43.5		49.0	5.0		
ΕZ	5 1		42.6	58.2	50.4	4.0		
ΕZ	6 1	53.6	46.1		49.9	5.0		
ΕZ	7		47.1		23.6	5.0		
ΕZ	8 ;	50.1	46.2		48.2	5.0		
ΕZ	9		45.8	50.2	48.0	5.0		
ΕZ	10 :	56.8	42.2		49.5	5.0		
	AVERAGE	53.0	45.9	52.8	46.9	4.9		
<b>F</b> 7								
Ε <i>L</i>	11 ;		45.9	53.8	49.8	5.8	5.4	200.0
EZ EZ	11   12	48.4	45.9 41.9	53.8	49.8 45.1	5.8 5.8	5.4	200.0
EZ EZ EZ	11   12   13	48.4	45.9 41.9 48.0	53.8 47.6	49.8 45.1 47.8	5.8 5.8 5.8	5.4	200.0
EZ EZ EZ EZ	11   12   13   14	48.4 58.4	45.9 41.9 48.0 47.3	53.8 47.6	49.8 45.1 47.8 52.8	5.0 5.0 5.0 5.0	5.4	200.8
EZ EZ EZ EZ EZ	11   12   13   14   15	48.4 58.4	45.9 41.9 48.0 47.3 45.0	53.8 47.6 46.0	49.8 45.1 47.8 52.8 45.5	5.0 5.0 5.0 5.0 5.0	5.4	200.0
EZ EZ EZ EZ EZ EZ	11   12   13   14   15   16	48.4 58.4 48.6	45.9 41.9 48.0 47.3 45.0 40.8	53.8 47.6 46.0	49.8 45.1 47.8 52.8 45.5 44.7	5.8 5.8 5.8 5.8 5.8 5.8	5.4	200.8
EZ EZ EZ EZ EZ EZ	11   12   13   14   15   16   17	48.4 58.4 48.6	45.9 41.9 48.0 47.3 45.0 40.8 39.4	53.8 47.6 46.0 53.6	49.8 45.1 47.8 52.8 45.5 44.7 46.2	5.8 5.8 5.8 5.8 5.8 5.8 5.8	5.4	280.8

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		AVERAGE	AVERAGE	AVERAGE	AVERAGE		AVERAGE	SPEED
		LEFT	CENTER	RIGHT	SHEET PI	LLING	PICKUP	
ΕZ	18 ;	60.6	40.1		50.4	5.0		
ΕZ	19		42.5	53.2	47.9	5.0		
E7	1778 - H 499 - J	54.2	47.8		5t.0	5.0		
	AVERAGE	54.0	43.9	58.7	48.1	5.0		
E2	21 1	48.8	48,8		48.8	4.8	7.9	150.0
E2	22		49.6	45.4	47.5	5.0		
ΕZ	23 1	51.6	53.2		52.4	5.0		
ΕZ	24 1		58.8	51.8	55,3	5.0		
ΕZ	25 1	54.6	54.6		54.6	5.0		
ΕZ	26 ;		51.2	46.4	48.8	5.0		
EZ	27	45.2	47.4		46.3	5.0		
€Z	28 :		53.6	51.0	52.3	4.0		
EZ	29 ;	56.4	58.0		57.2	5.0		
	AVE <b>RA</b> GE	51.3	52.8	48.6	51.5	4.8		

APPENDIX 5.

FURTHER STRESS/STRAIN PLOT COMPARISONS FOR RUNS CONDUCTED IN THE "E" SERIES (FA-252 RESIN, MEDIUM FINENESS)

## E 200YPM 4.7/7.7/10.1%

### MACHINE DIRECTION



## E 200YPM 4.7/7.7/10.1%

CROSS DIRECTION



E, 150/200YPM, 4.7%

MACHINE DIRECTION



## E, 150/200YPM, 4.7%

### CROSS DIRECTION



# E, 150/200YPM, 7.7%

### MACHINE DIRECTION





### CROSS DIRECTION



## E 150/200 YPM, 10%

### MACHINE DIRECTION


# E 150/200 YPM, 10%

## CROSS DIRECTION



#### APPENDIX 6.

FURTHER STRESS/STRAIN PLOT COMPARISONS FOR RUNS CONDUCTED IN THE "FY" SERIES (IF-1977 RESIN, MEDIUM FINENESS)

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FY 150, 4.9% VS. 200, 5.5%

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MACHINE DIRECTION



FY 150, 4.9% VS. 200, 5.5%

CROSS DIRECTION



#### APPENDIX 7.

#### FURTHER STRESS/STRAIN PLOT COMPARISONS FOR RUNS CONDUCTED IN THE "E" (FA-252 R) AND "FY" (IF-1977 RESIN) SERIES (BOTH MEDIUM FINENESS GRADE)

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# E VS. FY 200YPM, 7.7%

## MACHINE DIRECTION



## E VS. FY 200YPM, 7.7%

## CROSS DIRECTION



# E 10.2%/FY 10.1% 200 YPM

## MACHINE DIRECTION



# E 10.2%/FY 10.1% 200 YPM

CROSS DIRECTION



#### APPENDIX 8.

#### STANDARD 3M KIT SOLUTIONS AND METHOD FOR DETERMINING FP SURFACE OIL REPELLENCY

#### 3M KIT TEST FOR SURFACE OIL REPELLENCY

KIT NUMBER	VOLUME CASTOR OIL* m1.	VOLUME TOLUENE* m1.	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

#### PROCEDURE FOR DROP TESTS

THE SAMPLES WERE TESTED FOR SURFACE OIL REPELLENCY (3M KIT TEST, TAPPI UM 557) AS FOLLOWS:

FIVE DROPS OF EACH KIT # SOLUTION WERE PLACED ON THE CURED TEST SPECIMENS AND ALLOWED TO STAND FOR 15 SEC. THE DROPS WERE THAN SWABBED, AND THE # OF DROPS THAT REMAINED ON THE FABRIC SURFACE WERE RECORDED.

APPENDIX 9.

PROCEDURE FOR ISOPROPYL ALCOHOL DROP TESTS

ONE DROP EACH OF 70% AND 90% ISOPROPYL ALCOHOL WAS PLACED ON THE SAMPLES AND ALLOWED TO REMAIN FOR 5 MIN. THE ALCOHOL WAS THEN SWABBED OFF AND THE REMAINING SPOT (IF ANY) RATED AS TO DEGREE OF SURFACE PENETRATION. APPENDIX 10.

FORMATION, CURING AND TESTING PROCEDURES FOR UV-CURABLE FILMS

#### Film formation

- (1) The knife was placed on one end of a glass plate (cleaned with acetone and oven dried).
- (2) A small quantity of resin was poured along the width of the knife.
- (3) The knife was then drawn along the plate at one rapid, constant motion.
- (4) Knife
  - (a) Setting 10 mils
  - (b) Actual = 3.5 mils



# Curing (1) The plate and resin were passed through the oven as many times (1-4 times) a necessary to remove the "tackiness" of the film. (2) Curing unit (a) Speed - .17 - .18 (b) Dose - = 2450 mJ/cm<sup>2</sup>

Film Removal (1) Soak in warm water then dry the film

Testing

- 1. Cure Check for tackiness ٥. 2. Thickness Federal Micrometer ٥. Tensile Testing з. Instron 1125 ٥. ь. Cycling test (1) Elongation - : (2) Number of cycles Elongation 599% с. Modulus 100 - Hycar 26120 **d** . Tensile strength 1023 psi е. f. Method Sample 1" x 3.5" x 4 mil (+- 1 mil) (1) Gauge length 1.5" (2) (3) Load Cell 100 lb. (4) Jaw Speed
  - (5) Jaw Size
  - (6) Paper Speed
  - (7) Full Scale Range

#### APPENDIX 11.

## PUMP OPERATION, START-UP AND SHUT-DOWN PROCEDURE FOR THE NORDSON LIQUID SPRAYGUN SYSTEM



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SINGLE PISTON PUMP OPERATION

#### REGULAR STARTING PROCEDURE



Read and understand the safety pages 1-0-1 thru 4 before operating this pump. Failure to observe recommended safety procedures may result in personal injury.

- 1. Remove siphon rod (1) from solvent. Drain-off rod (2) stays in container.
- 2. Close circulation value (3).
- 3. Open pump drain-off value (4), slowly.
- 4. With air pressure regulator (6) backed off, open air supply value (7).
- 5. Slowly pressurize the system by adjusting the air pressure regulator (6) until pump begins to stroke, approximately 8-10 strokes per minute.
- 6. Allow pump to run until all solvent is discharged through drain-off value and air bubbles come through drain-off rod (2).

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REGULAR STARTING PROCEDURE, (Continued)

7. Reduce pressure to zero at the regulator (6).

- Slowly open filter drain (8) at bottom of paint filter to release fluid pressure. (As experience indicates disassemble, thoroughly clean and reassemble the filter (9).) Close filter drain (8).
- 9. Place siphon rod (1) in coating material to be applied.
- 10. Slowly pressurize the system by adjusting the air pressure regulator (6) until the pump begins to stroke, approximately 8-10 strokes per minute.
- 11. Allow at least 1 pint (1/2 liter) of material to flow out of the pump drain-off value (4).
- 12. Close drain-off valve (4).
- 13. Adjust air pressure regulator (6) to desired operating pressure.
- 14. Open circulation value (3) slightly to allow pump to stroke, approximately 8-10 strokes per minute.
- 15. Turn heater (5) on, if heated system.
- 16. Check temperature of heater (5) and when thermometer (10) reads within 5° of desired temperature, adjust pump stroke for proper circulation.
  - NOTE: The airless pump in a heated circulating system should always be operating and circulating when heaters are turned "ON". Failure to observe this can result in heater plugging and heat limiter failure.

Pumps used for handling materials which settle quickly should be kept in operation with the circulation valve properly adjusted or system should be flushed and refilled with solvent.

#### CHANGING OF MATERIALS

Reduce pressure to zero, then perform steps of regular start-up (Steps 1, 2, 3 and 6 thru 16). If materials are non-compatible (such as oil base paint and lacquer), two complete flushes with solvent are necessary. First flush with a solvent compatible with the material removed, then with the solvent compatible with the new material to be introduced into the system.

#### DAILY SHUT-DOWN PROCEDURE

- 1. Turn off heater (5) 10 to 15 minutes before pump shut-down to prevent paint build-up in heater.
- 2. Reduce air pressure at air pressure regulator (6) to zero. If solids in material do not tend to separate from liquids, coating material may be left in system overnight. If solids separate quickly or system won't be used for 24 hours or more, follow steps 3 thru 16, below.
- 3. Remove siphon rod (1) from coating material.
- 4. Close circulation valve (3).
- 5. Open drain-off valve (4).

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	SUPERSEDES 5/81 ISSUED 11/81		
DA	ILY SHUT-DOWN PROCEDURE, (Continued)		
6.	Slowly pressurize system by adjusting air pressure regulator $(6)$ until pump begins to stroke. Allow pump to discharge all material through drain-off rod $(2)$ into coating material container.		
7.	Open filter drain (8)		
8.	Place siphon rod 1 in solvent.		
9.	Allow 1 quart (1 liter) of material to discharge through filter drain valve (8) . Close valve.		
10.	Allow several quarts of material to discharge through drain-off rod $(2)$ .		
11.	Close drain-off valve (4) on pump.		
12.	Open circulation valve $\textcircled{3}$ until pump strokes slowly, circulate for about one minute.		
13.	Trigger gun ten (10) seconds allowing solvent to flow through gun.		
14.	Shut off air supply valve $(7)$ .		
15.	Open drain-off valve (4).		
16.	Remove and clean nozzle $(1)$ and turbulence plate or restrictor $(12)$ .		
17.	Interior of system should not be exposed to air. Leave solvent in system when not in use.		
	DAY-TO-DAY MAINTENANCE		
The regu	Nordson system is easy to maintain. However, certain steps should be taken Marly to assure its correct operation.		
1.	The high pressure filter (9) must be cleaned daily or per shift, unless experience indicates less frequent cleaning is adequate. Place clean, spare screen in filter and soak dirty screen in solvent. Inspect screen for rupture or distortion prior to installation.		
2.	Inspect solvent chamber (13) daily. When solvent level rises to a point half-way between filler line and overflow hole on solvent filler cup, drain and replace with fresh solvent using Nordson solvent. See page 19-5-1.		
3.	Occasionally lubricate snapping mechanism on air valve with a few drops of lubricant.		
4.	Check air line oiler level $(14)$ and rate of delivery. Use Nordson vitalizer oil or approved substitute only. See page 19-6-1.		
5.	System components may be wiped clean with a solvent soaked cloth to remove paint splashes. Do not soak any component in solvent. Hose covering and seals may be affected by some solvents. See Section 7 for nozzle care and cleaning instructions.		



6.

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Check all system components grounding device to insure that components are bonded to earth ground to prevent static electrical sparking and possible fire or explosion.

#### APPENDIX 12.

#### DESIGN DETAILS OF CONSTRUCTED MOTOR-DRIVEN BELT SYSTEM FOR FABRIC TRANSPORT

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1/2 bose K- 3%-> holes for assembly Bolts for assembly 4 4(6 × 7 × 1/8 plate) ) <u>1</u>. 4(43×18 L) 4(7×18 L) kixi<del>k</del> 43" オボ . 12-1 1/8" plate contered centered 1/2ley,1x # leftend + right and Plate adjustable to Y4" above levelof top of rolls and to Y4" below level of top of rolls Side View Fine A Front & Back Sile As. figure C

4(6×7x1/8 plate) 4(16x1/8 L)



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End View Figure B

left & Right End

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12-3



12-4