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FINAL REPORT

NASA CONTRACT NUMBER NAS1-10347

DEVELOPMENT AND PRODUCTION

OF

HIGH-ALTITUDE DECELERATOR, LIGHTWEIGHT LOW PERMEABILITY BRACE MATERIAL

Submitted To:

National Aeronautics and Space Administration
Langley Research Center, Mail Stop 126
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ABSTRACT-SUMMARY

New light-weight low permeability structures were developed and the following samples were produced for NASA evaluation:

PZ-5508.35 - .00025" Mylar*/Metallizing/Saran Coating/Adhesive/.00015" Mylar

PZ-5508.36 - .00025" Mylar/Metallizing/Saran Coating/Adhesive/Metallizing/ .00015" Mylar

PZ-5508.37 - .00015" Mylar/Saran Coating/Adhesive/Metallizing/.00015" Mylar

PZ-5508.38 - .00025" Mylar/Saran Coating/Heat Resistant Coating

Experimental work included selection of adhesive and coating formulations and development of appropriate manufacturing sequence and manufacturing conditions. The .00025" Mylar can be handled with reasonable care while the .00015" Mylar was difficult to process. A minimum number of process steps requiring handling of the .00015" Mylar are desirable as defects are readily introduced and these tend to be magnified in subsequent processing operations. For this reason, PZ-5508.37 proved difficult to make and is the least desirable from a manufacturing standpoint.

The preferred manufacturing sequence involved metallizing, then Saran coating the metallized film, then adhesive laminating or coating. In laminating, the adhesive should be applied to the heavier web. Control of tension and balance of tensions between webs during lamination are very important. Temperature and air movement used to dry coatings must also be carefully controlled.

All four films exhibited very low permeability. Permeability data is presented in the report along with physical property data, formulas, and process conditions.

*Mylar is a registered trademark of the duPont Company.

FINAL REPORT - NASA CONTRACT NUMBER - NASI-10347

PROJECT TITLE: LIGHT WEIGHT BRACE MATERIAL FOR HIGH ALTITUDE DECELERATOR

OBJECTIVE

Develop a process for making a laminate comprised of thin gauge Mylar, Saran coating, vacuum deposited aluminum, and protective coating. The protective coating may be a second layer of Mylar. Three specific laminates were targeted at the start of the program:

.00025" Mylar/Saran-Aluminum/.00015" Mylar
.00025" Mylar/Saran-Aluminum/Protective Coating
.00015" Mylar/Saran-Aluminum/.00015" Mylar

The program was designed to be conducted in two stages. In Stage I the experimental work was to be conducted and in Stage II 1000 sq.yd. samples were to be made. If any or all of the products could not be made in Stage I they would be eliminated from Stage II. Stage II consisted of preparation of quantities of materials for test by NASA.

STAGE I - EXPERIMENTAL

Introduction

Initial work involved selection of materials and formulations, evaluation of various preparation sequences and development of process conditions suitable for the steps involved. Some of the specific considerations included:

1. Selection of a protective coating formulation. The coating must provide abrasion protection for the aluminum layer and be sufficiently heat resistant to withstand conditions used to fabricate the brace. The coating formulation must not attack the Saran coating or Mylar and must dry or cure under conditions that the thin gauge Mylar will tolerate. Finally, the coating must adhere securely to the metallized web and not provide a weak boundary layer when adhesive bonded in the final fabrication.

2. Saran resin formulation. A formulation already existed for coating Mylar to achieve moderate to good barrier properties. Some solvent adjustment was anticipated due to the limited drying conditions permitted with the thin gauge web.

STAGE I - EXPERIMENTAL (CONTINUED)

3. Preparation sequence. Several sequence variations were possible -- particularly with the laminates containing two plies of Mylar. The following sequences are examples:

-- Metallize Mylar, Saran coat metallized surface, apply adhesive to Saran, mount to second web.

-- Saran coat Mylar, then metallize, apply adhesive to metallized layer and mount to second web.

-- Metallize one web, Saran coat one web, apply adhesive to metallized web and mount to Saran coated web.

-- Metallize one web, Saran coat one web, apply adhesive to Saran coated web and mount to metallized web.

-- In the structure using a single web, the protective coating would replace the adhesive in the above examples and the mounting step would be eliminated.

-- To obtain optimum barrier properties, give maximum protection to the metal layer and minimize bonding problems in later fabricating we much preferred to have the metal layer buried in the structure as in the above examples. We were permitted, if necessary, to make a clear laminate and metallize the outside surface. This pertains primarily to the .00015" Mylar since we were not sure initially that our metallizer could handle a single ply of this thin web.

The choice of sequence was potentially affected by several considerations. It was anticipated that the metal coating would be more uniform and adhesion better if the metal was applied to Saran. Application of aluminum vapor to Saran, however, was known to cause a reaction that could lead to heat buildup and possibly spontaneous combustion in large rolls. If the reverse sequence was used, great care would be needed to prevent scratching of the metallized surface while coating.

4. An appropriate adhesive formula had to be selected. Again, existing adhesive polymers were expected to be adequate, but formulation adjustments and solvent changes were anticipated.

STAGE I - EXPERIMENTAL (CONTINUED)

5. Behavior of the thin gauge Mylar in the metallizer had to be studied to determine what special procedures or equipment might be required. Web breaks with the thin gauge films were a possibility. Such breaks are particularly troublesome in vacuum metallizing because a great deal of time is lost breaking the vacuum, re-webbing and pumping the chamber down to re-establish the vacuum.

6. Appropriate conditions had to be established for the various operations performed with the solvent coater-laminator. It is desirable to use as much heat and air velocity as possible to get good drying of the coatings and adhesives. The web, however, tends to stretch when heated under tension. The stretching causes loss of width and can result in stresses being built into the film that can cause problems later. It is, therefore, desirable to use low heat and/or tension. Since drying is a function of heat and air movement, a relatively high air velocity would seem desirable. High air velocity can cause web flutter which may cause coating irregularities, wrinkles or other problems. With heavier webs, the flutter would be less of a problem and could be countered by increasing web tension. With the light weight webs used in this project, the heat, air velocity and tension had to be adjusted to get the best compromise of conditions.

   In addition to the above conditions, an appropriate balance of tensions between the two webs had to be maintained to insure stability of the laminated product.

SELECTION OF MATERIALS

With the extensive experience at this location on the use of adhesives and coatings, we were able to select materials for this purpose mainly by review of data from prior applications. Some of the factors considered are discussed below:

Saran Coating

Saran is available commercially in latex form and as soluble resin. The soluble resin grades range from essentially amorphous to highly crystalline. The highly crystalline varieties are soluble only in strong solvents such as tetrahydrofurane. A resin with a low degree of crystallinity, Dow Chemical's QX-2364.2 was chosen because it was soluble in solvent blends that adapted to our equipment and was known to provide good adhesion to polyester film. Proof press trials indicated that QX-2364.2 could also be applied on the metallized surface of polyester film without damage to the metallizing.

SELECTION OF MATERIALS (CONTINUED)

Adhesive

The laminating process normally used for polyester and other similar flexible films involves application of a relatively high molecular weight adhesive polymer to one web from a solvent solution. Adhesives may be thermoplastic or they may contain reactive ingredients that will cure the adhesive to a thermoset form. Application can be done by any of a number of coating techniques. For the coating weights required in this application, we normally use a gravure applicator. Once applied, the adhesive coated web is passed through an oven where the solvent is removed by a flow of hot air. After drying, the adhesive coated film is combined with a secondary web and the combination is passed between heated nip rolls to melt the adhesive slightly and provide intimate contact with the second web. In the case of the thermoset adhesives, some cure is also initiated by the heated nip. The remaining cure normally takes place on standing for several days at room temperature. Other processes such as extrusion or hot melt lamination can be used but were not considered here because low coating weights are not readily attained with these processes.

Any number of adhesives are available that would be expected to work in this application. A cure type was selected for permanence and heat resistance. Cure type adhesives suitable for bonding Mylar are available based on polyester and polyurethane polymers and various modifications of these -- all cured with some type of isocyanate containing co-reactant. A cure type urethane adhesive designated Unoflex by Polymer Industries was chosen because it is available in a convenient one component form and is known to develop bond quickly after lamination. Proof trials indicated that the Unoflex adhesive could be applied to Saran coated or metallized Mylar without difficulty. It should be noted that the proof press samples referred to are air dried, whereas heat and air velocity are used on the coater-laminator. Some formulation adjustments, particularly in regard to solvent blend, may be required in going from the proof press to the coater-laminator.

Protective Coating

As with adhesives, a number of different polymer compositions hold potential as a coating for polyester film. As heat resistance is built in to many compositions, however, they tend to become brittle and provide poor adhesion to the base film. A nitrocellulose polymer, RS Cotton, supplied by Hercules Corporation was known to provide a good combination of heat resistance, toughness and adhesion. Proof press

SELECTION OF MATERIALS (CONTINUED)

Protective Coating

trials indicated this coating could be applied to Saran coated or metallized film. It was noted, however, that this coating did not provide protection from solvent attack. The adhesive used to fabricate the brace could probably not be applied to this protective coating. A trial was made in which adhesive, a polyester adhesive from duPont, was applied to the uncoated side of the Mylar. After the solvent evaporated, the adhesive was bonded to the nitrocellulose coated surface using a laboratory model Sentinel heat sealer to provide heat and pressure. Good adhesion to the nitrocellulose coating was obtained although the bond to the uncoated polyester was marginal. Treatment of the polyester by chemical or electrical discharge or selection of a different adhesive should provide better bond at that surface. This aspect was not pursued as we have no details on the fabrication methods used.

PROCESS SEQUENCE

Initially it was decided to attempt metallizing after Saran coating. This would involve running a relatively narrow web through the metallizer. Before this was attempted, a preliminary run (Order No. 35118) was made using .00025" x 30" wide Mylar from our plant stock. No metal was applied during this run as web handling was the only concern. No difficulty was noted in this run and it appeared that .00025" film was sufficiently strong that the narrower web would run satisfactorily.

A roll of .00025" x 20" Mylar was then coated with Saran resin. A coating weight of 3.1 lb./ream was applied using minimum web tension and minimum (30 ft./min.) line speed. (See Order No. 35110 - Run 1). The coating appeared dry and had good adhesion. Web width of the coated web was 19-5/8". The quality of the coated roll with regard to wrinkles, gauge bands, etc. was good but not as uniform as desired for use in the metallizer. Rewinding was considered but rejected to minimize the handling of this light gauge web. The coated roll was successfully metallized (Order No. 35110 - Run 2) but roll quality of the metallized material was poor as defects in the coated roll were magnified. Following metallizing, the .00025" web was coated with 2.2 lbs./ream of adhesive. Adhesive was applied from methyl ethyl ketone solution, dried and the adhesive coated web was mated with .00015" Mylar and passed through a nip roll (See Order No. 35110 - Run 3). The result was a promising first sample but the following defects were noted:

PROCESS SEQUENCE (CONTINUED)

1. A whitening of the Saran coating was noted, indicating solvent attack on the Saran coating by the adhesive.

2. Fine ridges in the machine direction were noted in the Saran coating indicating that the coating had not flowed out to a uniform film. The whitening made these ridges visible.

3. Some very fine wrinkles were noted in the laminate.

A small additional run (Order No. 35110 - Run 4) was made laminating plain .00025" Mylar to plain .00015" Mylar to refine laminating conditions. Adhesive was again applied to the .00025" web which was then mated to the .00015" web from the secondary unwind.

An additional quantity of the .00025" Saran coated and metallized Mylar was coated with a protective nitrocellulose coating. (See Order No. 35111). The coating applied well and adhesion was good. The whitening of the Saran noted in the adhesive laminated samples was not evident with the coated material.

Samples from Orders 35110-3, 35110-4 and 35111 were tested for barrier properties. The results were considerably better than required.

From the above experiments we concluded:

1. Better quality rolls would be required for use in the metallizer. Since the metallizer is a sealed chamber during operation there is no opportunity to correct defects that start on the rewind roll. A wrinkle at the start of the roll tends to be quickly magnified as the roll builds up.

2. It may be necessary to metallize before Saran coating to get adequate quality rolls for metallizing.

3. A more uniform Saran coating application was needed.

4. Adjustment was needed in the adhesive formula to eliminate solvent attack on the Saran coating.

The following steps were planned:

1. Coat additional .00025" Mylar using a slower solvent blend to permit better flow out. Evaluate the use of a smoothing bar to provide a more uniform coating.

PROCESS SEQUENCE (CONTINUED)

2. Prepare Saran coated Mylar for the metallizer with .001" leader stock to insure a smooth start-up on the rewind roll in the metallizer.

3. Metallize a quantity of .00025" uncoated Mylar to evaluate the process of metallizing first and then coating.

4. Evaluate an adhesive formula containing a less aggressive solvent.

In addition to the above, our ability to Saran coat and/or metallize .00015" Mylar had to be determined.

Additional quantities of .00025" Mylar were coated (Order No. 35204 - Run 1) using a Saran formulation in methyl ethyl ketone in place of the methyl ethyl ketone/acetone blend used earlier. Various smoothing bar arrangements were tried and an arrangement was adapted using a smoothing bar held in a fixed position with light contact to the web. The resulting roll appeared to be of good quality and a .001" thick leader was spliced onto the end of the roll for metallizing.

Attempts were then made to Saran coat the .00015" Mylar (See Order No. 35112 - Run 1). The .00015" film is not intended for laminating application and roll quality as it applies to laminating requirements is considerably inferior to that of the .00025" film. As expected, it was much more difficult to balance heat, tension and air velocity with the .00015" web than with the .00025" film. The light film had more tendency to lift near the oven exhaust and it was necessary to add a baffle to the oven to prevent flutter of the web near the exhaust. The .00015" film had been purchased at the maximum width (20") that the laminator used could handle to allow for the anticipated loss of width through the coating and laminating operations. Coated web coming out of the drying oven had one or both edges folded over and stuck down by the coating. The folding may have been caused by air lift at the web edge as the air passed through the minimal clearance or the web may have touched guides at the edge of the web path or both. A number of attempts were made to avoid the edge fold without success. The balance of the .00015" film was then saved for metallizing and an additional quantity of .00015" x 19.5" film was ordered for further coating trials.

Due to scheduling difficulties related to a temporary plant shut down, several weeks elapsed between the Saran coating of .00025" film in Order No. 35204 - Run 1 and the metallizing trial with this coated film. Blocking was discovered in the roll and the film could not be unwound satisfactorily in the metallizer (Order No. 35204 - Run 2). At this point, attempts to metallize Saran coated film were abandoned.

STAGE II - SAMPLE PREPARATION

At this point we committed to metallizing before Saran coating. Preparation of samples for NASA commenced although it will be apparent to the reader that many of the process variables for each step will need to be worked out at the start of that step.

Approximately 20,000 ft. of uncoated .00025" x 30" film from stock was metallized on Order No. 35237 - Run 1.

Initial attempts to metallize .00015" Mylar met with numerous web breaks. These are very time consuming since the vacuum has to be broken to re-web the equipment, then the chamber must be pumped down again. Finally the tension control mechanism was disconnected from the unwind and a lightly weighted belt was constructed to act as a drag on the unwind roll. No further web breaks were encountered and 10,000 ft. of film was run on Order No. 35205.

Saran coating trials on the .00015" x 19.5" web did not exhibit the same degree of edge fold-over problem noted with the 20" wide web, however, operator manipulation was required to run successfully. Since the .00015" film was not perfectly flat, occasional build-up of coating weight was noted at the edge of the web. Overall quality of the coated film was judged to be satisfactory and 3000 ft. was run on Order No. 35238 - Run 1.

Saran coated .00015" film from Order No. 35238 - Run 1 was adhesive coated and laminated to .00015" metallized film on Order No. 35238 - Run 2. Material ran fair with a considerable number of wrinkles developed in the laminate. Edge waviness of the .00015" film resulted in some excess adhesive being built-up at the film edge. The excess adhesive tended to dry poorly and squeeze out when laminated causing edge blocking in the roll. A sample quantity of approximately 2000 ft. was slit to 17-5/8" for evaluation by NASA. Considerable loss was noted at the slitter from the edge blocking. A very slight leather grained appearance was noted in the finished laminate. The cause was not investigated because the laminate appeared to be of adequate quality for initial testing by NASA. The pattern could be caused by differential stresses induced in the two webs during laminating. The web being coated with adhesive goes through an oven and is readily stretched while hot. If stretched, the web will tend to shrink back after cooling. Since the secondary web is cool, it is purposely subjected to more tension to attain a balance between the two webs. Considerable experimentation would be required to get an exact match between the webs. In addition to the differential stress, trace quantities of adhesive solvent may be trapped in the web which could accentuate the effect of the differential tension.

STAGE II - SAMPLE PREPARATION (CONTINUED)

Metallized .00025" Mylar was Saran coated on Order No. 35239 - Run 1 using slightly more heat than on Order No. 35204 - Run 1. The coated metallized film was laminated to .00015" film on Order No. 35239 - Run 2. Following the procedure used on Order No. 35238, the adhesive was applied to the .00015" film. This procedure gives each film web one pass through the oven -- one for Saran coating, the other for adhesive application. As before, some excess adhesive build-up was noted on one edge. Otherwise the material ran well and approximately 2500 ft. of good laminate was prepared.

After standing overnight, many tunnels (approximately 1/8" wide x 6" or longer) appeared between layers of the laminated film. It was concluded that the procedure used created too great a stress differential between the two films. Laminated material from Order No. 35239 - Run 2 was scrapped and another run was planned in which the adhesive would be applied to the Saran coated metallized .00025" film and the .00015" film would be fed from the secondary unwind. With the .00025" film being heated and the .00015" film being fed in at ambient temperature, the built in stress would be more evenly matched between the two films. This run was designated Order No. 35239 - Run 3. Only enough plain .00015" Mylar remained to make approximately 900 ft. of good laminate. An additional 1100 ft. of laminate was prepared using metallized .00015" Mylar as the secondary web on Order No. 35239 - Run 4. Rolls of laminated film were pulled tight and securely taped to restrain the film from movement while the adhesive cured to maximum strength. These rolls were of good quality after standing. Samples were slit to 17-5/8" for evaluation by NASA.

Metallized, .00025" film was Saran coated on Order No. 35240 - Run 1 and an additional 1 lb./ream protective coating was applied over the Saran on Order No. 35240 - Run 2. The protective coating provides heat resistance needed for fabricating and also aids in avoiding abrasion of the Saran barrier layer. The coating operation ran well and the finished roll looked good. Approximately 2000 ft. was slit to 17-/58" for evaluation by NASA. When removed from the roll, this structure had a tendency to curl and may be difficult to work with.

Samples from the finished rolls prepared for NASA were tested for barrier properties. The results are shown in Table II.

A series of physical property tests were requested by NASA. These tests and the results are shown in Table III. Test conditions are described in Appendix A. Actual and theoretical weight per area values are shown in Table I.

STAGE II - SAMPLE PREPARATION (CONTINUED)

Coating and adhesive formulas are shown in Appendix B.

Equipment used is described in Appendix C.

Details of the process conditions developed and used to prepare the samples submitted to NASA are shown in Appendix D.

CONCLUSIONS

1. In producing a metallized and coated web, the preferred procedure is to metallize first and then coat. The best possible roll quality is required for metallizing and if the film is coated first, any defects introduced in the coating step will decrease the chances of getting a good quality roll after metallizing.

2. Metallizing .00025" Mylar proceded without difficulty. Special tension adjustments were required to handle the .00015" film after which metallizing proceded without difficulty.

3. Coating or laminating .00025" Mylar can be accomplished without unusual problems if tensions are carefully controlled and kept at a minimum. Handling the .00015" film is much more difficult. Original quality of the .00015" film is not that of the .00025" film. It is desirable to run the .00015" film through as few operations as possible because defects are easily introduced. These tend to be magnified in subsequent operations.

4. The formulations chosen for Saran coating, adhesive bonding and heat resistant coating appear to meet the requirements of the end use when applied and handled according to the conditions developed.

5. Control of tension during the lamination step is very important. If more stress is induced in one web than in the other, distortion, wrinkling or spot delamination may occur as the films try to return to their original dimension. Tensions were easier to match when adhesive was applied to the heavier .00025" web and this was mated to the .00015" web from the secondary unwind.

6. The product made by applying a heat resistant coating to metallized and Saran coated .00025" film ran well but a tendency to curl was noted in the finished product.

CONCLUSIONS (CONTINUED)

7. With the above factors considered, the most desirable of the laminates from a production standpoint was:

   PZ-5508.35 - .00025" Mylar/Metallizing/Saran Coating/Adhesive/ .00015" Mylar

   This structure allows the .00015" Mylar to be fed directly from the secondary unwind without additional handling.

   The second choice of the laminates was:

   PZ-5508.36 - .00025" Mylar/Metallizing/Saran Coating/Adhesive/ Metallizing/.00015" Mylar

   This structure involved metallizing of .00015" Mylar but it also ran well. An occasional web break in the metallizer can be anticipated using the .00015" film.

   The least desirable of the laminates was:

   PZ-5508.37 - .00015" Mylar/Saran Coating/Adhesive/Metallizing/ .00015" Mylar

   This structure requires one machine pass of one .00015" web and two machine passes of the other .00015" web. It will be much more difficult to get consistent quality with this structure than with PZ-5508.35 or PZ-5508.36.

   The single ply structure ran without difficulty. This structure was:

   PZ-5508.38 - .00025" Mylar/Metallizing/Saran Coating/Heat Resistant Coating

8. All of the above combinations met the barrier requirements specified.

TABLE I

WEIGHT PER UNIT AREA

lbs/sq.ft.

SAMPLE	THEORETICAL	ACTUAL
PZ-5508.35	.00441	.00475
PZ-5508.36	.00441	.00472
PZ-5508.37	.00372	.00370
PZ-5508.38	.00297	.00316

Theoretical values were calculated from nominal film yields and coating or adhesive weights.

Actual values were obtained by weighing 4" x 4" specimens.

The difference between theoretical and actual is due mainly to the .00025" film being supplied slightly thicker than the nominal value.

TABLE II

BARRIER DATA - FINAL SAMPLES

SAMPLE	GAS TRANSMISSION cc(STP)/(100 sq.in.)(24 hrs.)(atm) OXYGEN	NITROGEN	CARBON DIOXIDE	THICKNESS (mils)
PZ-5508.35	0.028	0.011	0.115	0.75
	0.041	0.011	0.096	0.75
	0.041	0.011	0.096	0.75
	0.037	0.011	0.102	0.75
PZ-5508.36	(0.28)	0.011	0.058	0.75
	0.041	0.011	0.134	0.75
	0.055	0.011	0.058	0.75
	0.048	0.011	0.083	0.75
PZ-5508.37	0.055	0.011	0.21	0.55
	0.055	0.011	0.19	0.55
	0.055	0.011	0.23	0.55
	0.055	0.011	0.21	0.55
PZ-5508.38	0.006	0.011	0.019	0.45
	0.028	0.011	0.115	0.45
	0.028	0.011	(Leaker)	0.45
	0.021	0.011	0.067	0.45

The above data was determined by our Chemical Physics Research Laboratory using the Mass Spectrometer Method at 23°C. Results were confirmed by our Research Services Laboratory by measuring CO_2 permeability with the Dow cell.

TABLE III

PHYSICAL PROPERTY DATA

SAMPLE	GAUGE (mils)	TENSILE AT YIELD (psi x 10^3)	TENSILE AT BREAK (psi x 10^3)	% ELONGATION AT BREAK (%)	ENERGY OF RUPTURE (in-lbs)/in.	MODULUS OF ELASTICITY (psi x 10^3)
PZ-5508.35						
MD	0.71	9.5	14.0	97	8.65	369
45°	0.71	9.1	14.9	79	7.27	
CMD	0.71	9.0	17.8	53	5.85	461
PZ-5508.36						
MD	0.71	9.5	13.2	82	7.03	377
45°	0.73	9.1	14.8	62	6.00	
CMD	0.73	8.7	17.1	51	5.55	460
PZ-5508.37						
MD	0.6	9.2	12.4	93	6.45	388
45°	0.6	8.6	11.9	82	5.68	
CMD	0.58	9.0	17.1	51	4.32	475
PZ-5508.38						
MD	0.4	12.5	15.6	66	3.90	504
45°	0.4	12.1	19.6	54	3.69	
CMD	0.4	12.2	21.2	48	3.66	640
PZ-5525.05*						
MD	0.9	13.1	19.5	89	14.1	530
45°	0.9	12.8	19.2	70	11.3	
CMD	0.9	12.5	23.8	58	10.6	585

* PZ-5525.05 is .00050" Mylar/Saran Coating/Adhesive/.00025" Polyester

Prepared earlier for NASA

SUPPLEMENT TO TABLE III

PHYSICAL PROPERTY DATA

MODULUS OF ELASTICITY DETAILS

SAMPLE	GAUGE (mils)	MODULUS OF ELASTICITY psi x 10^3	AVERAGE YIELD (lbs)
PZ-5508.35			
MD	0.7	369	6.0
CMD	0.7	461	5.8
PZ-5508.36			
MD	0.7	377	6.2
CMD	0.7	460	5.9
PZ-5508.37			
MD	0.6	388	5.1
CMD	0.6	475	4.8
PZ-5508.38			
MD	0.4	504	4.3
CMD	0.4	640	4.2
PZ-5525.05			
MD	0.9	530	11.3
CMD	0.9	585	10.4

APPENDIX A

PHYSICAL TEST CONDITIONS

1. Gauge was measured by dead weight micrometer with precision to ± 0.00005 inches.

2. Tensile Properties; ASTM D-882-67

   a. Instron Conditions:

Initial Grip Separation	4 inches
Rate of Grip Separation	2 inches/minute
Chart Speed	1 inch/minute
Extensometer	10% blip
Integrator	On
Break Detector	On
Specimen Width	1 inch

   b. Procedure

Ten specimens were taken from each sample in the film machine direction and in the film cross-machine direction. Four to six specimens were then taken in one diagonal direction, the remaining specimens (to provide a total of ten) were taken in the other diagonal direction.

Each specimen was placed in the Instron grip. The Extensometer was attached to the specimen and the integrator was zeroed. The specimen was pulled apart and the load-extension curve charted.

Load at yield and at break was determined directly from the load-extension chart. The tensile at yield and at break was determined by dividing the load by the minimum gauge of the specimen.

A 10% blip film extensometer provides a blip on the load strain curve each time 10% elongation occurs. In addition, a minor blip occurs 5% after each 10% blip. Elongation at break was determined to the nearest percent ± 1%.

Energy of rupture was originally determined in ft-lbs. per specimen. This was calculated as follows:

APPENDIX A

Using the 20 lb. load scale, Integrator calibration gives
5,000 Intg. Units = 20 lbs. x 1/6 ft. = 3.33 ft.-lbs.

and

$$\text{Energy of Rupture} = \frac{\text{Intg. Units Under Curve}}{\text{5,000 Intg. Units}} \times 3.33 \text{ ft.-lbs.}$$

Using the 50 lb. load scale, Integrator calibration gives:

5,000 Intg. Units = 50 lbs. x 1/6 ft. = 8.34 ft.-lbs.

$$\text{Energy of Rupture} = \frac{\text{Intg. Units Under Curve}}{\text{5,000 Intg. Units}} \times 8.34 \text{ ft.-lbs.}$$

The units ft.-lbs./specimen were converted to inch-lbs./inch as follows:

$$\text{ft.-lbs./specimen} \times \frac{\text{12 inches/ft.}}{\text{4 inches length/specimen}}$$

$$= \frac{\text{inch-lbs.}}{\text{inch specimen length}}$$

The four inches is taken from the initial gauge separation. It should be noted that neck-in has not been considered in this calculation. The error caused by neck-in is a variable which depends on many factors. The greatest factor in this case is the maximum load. The effect of neck-in is to lengthen the effective specimen length. The initial gauge separation is four inches. As the film yields, part of the film is pulled from between the grip faces thus adding to the effective specimen length. In this work, the effective specimen length is estimated to be between 4.2 and 4.6 inches based on the comparison of chart computed % elongation and extensometer computed % elongation.

3. Modulus of Elasticity:

a. Instron Conditions:

Initial Grip Separation	5 inches
Rate of Grip Separation	0.5 inches/min.
Chart Speed	20 inches/min.
Strain/Chart Ratio	0.5% per inch
Specimen Width	1 inch

APPENDIX A

b. Procedure

Three specimens were cut and pulled in the machine direction and in the cross machine direction. The specimens were pulled to the yield point.*

The modulus of elasticity was determined by drawing a straight line along the initial linear portion of the load strain curve. The slope of this line was determined and converted into modulus by the following equation.

$$\text{Modulus of Elasticity} = \frac{(\text{Load})}{(\%\ \text{Strain})(\text{Gauge})(\text{Width})}$$

$$= \frac{\text{lbs.} \times 10^5}{(\%)(\text{Mils})(\text{Inches})} = \frac{\text{lbs.}}{\text{sq.in.}}$$

The yield point has been reported along with the modulus, since this value may have significance in the end use of these products. The yield is significantly lower at the lower strain rate conditions.

The limit of linearity is only about half of the yield value.

*ASTM D-882 requires a minimum of five specimens per test per direction. Since modulus of elasticity does not exhibit the same degree of variability as other tensile properties, three specimens per direction should provide a reasonably precise value.

APPENDIX B

FORMULATIONS

The formulas listed below were used for coating or laminating the final samples submitted to NASA for evaluation:

SARAN COATING

Ingredients	Parts by Weight
QX-2364.2 Dow Saran Resin	20
Methyl Ethyl Ketone	80

Resin is dissolved by thorough mixing and the solution viscosity is adjusted with MEK to 35 seconds tested using a number four Ford cup.

LAMINATING ADHESIVE - CURING TYPE

Unoflex - Polymer Industries Adhesive	53
Toluene	47

Adhesive solution is diluted with toluene and mixed well.

NOTE: Water will affect cure of this adhesive and care must be taken to avoid moisture contamination either in the solvents used or through unnecessary exposure of the product in open containers.

Thin with toluene to a number four Ford cup viscosity of 20 seconds.

HEAT RESISTANT COATING - NON-CURING TYPE

½ Second R. S. Cotton	25
Ethyl Acetate	75

Mix well and adjust with ethyl acetate to a number four Ford cup viscosity of 35 seconds.

APPENDIX C

EQUIPMENT DESCRIPTION

ATF Gravure Proof Press

The ATF Gravure Proof Press is supplied by the American Type Foundry, Klingrose Gravure Division, Mount Vernon, N. Y. It has a 48 inch wide impression drum, 58½ inch circumference, capable of producing proofs up to 40 inches wide. The drum has a ratchet type gripping and tension device to hold and tension the substrate to the impression drum. This equipment will provide a proof sample approximately 48 inches long.

The equipment will handle the knurls and gravure cylinders normally used in production. The doctor blade holder is designed with a weighted arm to provide variable tension against the knurl or cylinder. The impression roll advance/retract system is motor driven. The final impression pressure is established by manual adjustment. A hand crank wheel is used to turn the impression cylinder for printing.

Faustel Laboratory Model Coater-Laminator

The Faustel coater-laminator, Serial No. 66076, is manufactured by Faustel, Inc., Butler, Wisconsin. This equipment is basically a laboratory or research and development machine. It is designed to do many types of coatings and laminations. It will handle material as light as 10 lb. tissue and .00025" foil. The limiting factor in the heavy gauge substrates is the wrap on the small idler and transfer rolls. These rolls are approximately three inches in diameter and material that will not wrap these rolls will crack or splinter.

The machine is equipped with single unwind and rewind stands. The unwind stands are equipped with manual friction brakes and all of the shafts are interchangable. The core plugs for these shafts are stepped so that they can accommodate a three or six inch core. The unwinds will accommodate a 30 inch O. D. roll. Web width is limited to 19½" due to the oven construction. On light weight webs, it is necessary to reduce this width to 18½ - 19 inches because air velocity in the oven will cause the edges to fold over or wrinkle.

There is one rewind unit on the machine. The rewind shaft is driven through cars by a disc type Horton air clutch manually controlled and with a manual valve for disengaging. The rewind shaft will wind rolls to a maximum of 24 inch diameter with a maximum 2 lb./inch tension.

APPENDIX C

Faustel Laboratory Model Coater-Laminator (Continued)

The rewind unit is fed by a draw roll unit. This unit consists of a steel impression roll and a spring set rubber pressure roll. This unit is driven by a chain drive through a PIV unit connected to the main machine drive.

The primary unwind is followed by a series of herringbone idler rolls which are staggered to provide web support and guidance. These are followed by the gravure coating station. This station consists of a vertical roll stack consisting of a spring set rubber impression roll above a chrome coated steel knurl. The knurl dips into an adhesive pan mounted beneath the assembly. The excess adhesive is wiped off the knurl by a doctor blade. The doctor blade holder can be oscillated by means of an air piston. The gravure station also consists of an air motor driven polishing roll and a stationary mayer rod holder.

The oven unit is a 20 foot, two zone, medium impingement type dryer. It is an arched unit equipped with herringbone, tendency driven rollers for web support. The circulating air is heated by two gas fired burners, one for each zone. Individual temperature controllers allow different temperature settings for each oven zone.

The secondary unwind is followed by another series of transfer rolls which feed the secondary web over a steel, oil heated, pre-heat roll. This roll is mounted above, and to one side, of the hot combining station. The combining station consists of a steel, oil heated, roll and a rubber impression roll. The rubber roll is manually advanced against the impression roll through a cam drive to provide laminating pressure. The temperature of the impression roll can be regulated to accommodate the substrates being laminated. The primary web, from the oven, and the secondary web are laminated at this point.

After leaving the combining nip the laminated web passes in an S wrap configuration over two chrome plated steel chill rolls. The temperature of these rolls can be controlled to vary processing conditions. From the chill rolls the web passes through the draw roll unit to the wind-up.

Metallizer

The metallizer used for this project is production unit that is part of our manufacturing facility. It is a custom unit built by the N.R.C. Division of the former Norton Company. Diffusion pumps provide an operating pressure of 10^{-4} TORR. Induction heating is used to melt the aluminum metal. The unit of coating one or two sides of webs up to 48" in width.

APPENDIX D

RUN CONDITIONS

The attached sheets give details on the conditions used in coating and laminating samples for evaluation by NASA. The reader will recognize that the conditions shown are specific for the equipment used. Experienced laminators will be able to use this information with the equipment description in Appendix C to estimate conditions required on other equipment. Some experimentation will be required to establish exact run conditions for other laminator-coaters. Equipment used for processing these webs must have the ability to operate smoothly at minimum tension and provide an air flow pattern that will cause minimum left and flutter of the web even at the low tension.

LAMINATOR DATA SHEET — CPRL PILOT PLANT

DOW NO.	ORDER NO.	CHARGE NO.	DATE
	35238-1	825-1202 0601000	1-7 & 8 - 71

FOR	COMBINATION	CHARTED BY
NASA	.00015" MYLAR/SARAN COATING	Campbell

APPLICATION METHODS (Check Method Used)

- [X] 1. DIRECT GRAVURE
- [ ] 2. REVERSE ROLL
- [ ] 3. HOT MELT
- [ ] 4. DIRECT TRANSFER + MAYER ROD
- [ ] 5. TRANSFER ROLL APPLICATION + SMOOTHING BAR
- OR 6. ______________________

XXXXXX COATING

WT.-LBS/RM	COLOR	TYPE	NO.	VISCOSITY	SOLVENT NO.	SOLVENT TYPE
3.0	Cl	Saran	QX-2364.2	35	---	MEK

REVERSE ROLL COATING

GEAR RATIO	ROLL SPEED	DOCTOR ROLL GAP

WET LAMINATION

CYLINDER ETCH	SMOOTHING BAR	IMP. ROLL TEMP.	AIR KNIFE PRESSURE	AIR IMPINGEMENT ANG.	MAYER ROD NUMBER
55 Q	Yes - Fixed				

HOT MELT COATING

POLISHING ROLL TEMP.	FOUNTAIN TEMP.	MAYER ROD TEMP.	MAYER ROD NUMBER

DRY LAMINATION

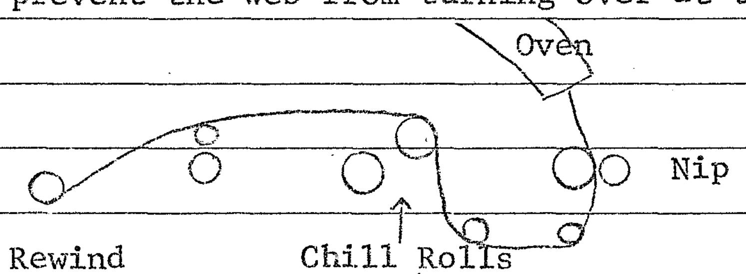
HOT ROLL TEMP.	WRAP	COOL ROLL TEMP.	#1	#2

LAMINATOR LINE SPEED	SCRAP	WEIGHT OFF
100 FPM		

		PRIMARY	SECONDARY	
OVEN	TEMPERATURE	150°F.	235°F.	
	DAMPERS	Full Open	Full Open	
	VENT OPENINGS	1/8	1/2	
STOCK	NUMBER			
	TYPE	Mylar Polyester		
	SIZE	19½"		
	GAUGE	.00015"		
	QUANTITY	3000 ft.		
SHRINKAGE	WET END			
	COMB. END			
	REWIND	½" shrink	CMD to 19"	

COMMENTS

A light impression on was required at the nip to maintain tension and prevent the web from turning over at the edge.



550

LAMINATOR DATA SHEET CPRL PILOT PLANT

DATE
1/8/71

DOW NO.	ORDER NO.	CHARGE NO.	CHARTED BY
	35238-2	825-1202 0601000	Campbell

FOR	COMBINATION
NASA	MYLAR-SARAN COATED/METALLIZED MYLAR

APPLICATION METHODS (Check Method Used)

- [X] 1. DIRECT GRAVURE
- [ ] 2. REVERSE ROLL
- [ ] 3. HOT MELT
- [ ] 4. DIRECT TRANSFER + MAYER ROD
- [ ] 5. TRANSFER ROLL APPLICATION + SMOOTHING BAR
- OR 6. ____________________

ADHESIVE							REVERSE ROLL COATING		
WT.-LBS/RM	COLOR	TYPE	NO.	VISCOSITY	SOLVENT NO.	SOLVENT TYPE	GEAR RATIO	ROLL SPEED	DOCTOR ROLL GAP
2.2	Cl	Ure-thane	Uno-flex	18	---	Tol			

WET LAMINATION					
CYLINDER ETCH	SMOOTHING BAR	IMP. ROLL TEMP.	AIR KNIFE PRESSURE	AIR IMPINGEMENT ANG.	MAYER ROD NUMBER
110 Q	No				

HOT MELT COATING				DRY LAMINATION				
POLISHING ROLL TEMP.	FOUNTAIN TEMP.	MAYER ROD TEMP.	MAYER ROD NUMBER	HOT ROLL TEMP.	WRAP	COOL ROLL TEMP.	#1	#2

LAMINATOR LINE SPEED	SCRAP	WEIGHT OFF

		PRIMARY	SECONDARY	
OVEN	TEMPERATURE	125°F.	150°F.	
	DAMPERS	Full Open	Full Open	
	VENT OPENINGS	1/8	1/2	
STOCK	NUMBER			
	TYPE	Saran Ctd. Mylar	Metallized Mylar	
	SIZE	19"	20"	
	GAUGE	.00015"	.00015"	
	QUANTITY			
SHRINKAGE	WET END			
	COMB. END			
	REWIND			

COMMENTS

Some build-up of heavy adhesive coating at edge of film particularly on drive side of laminator. Hand slit approximately 3/4" from bad portions of run.

550

LAMINATOR DATA SHEET — CPRL PILOT PLANT

DATE
1/12/71

DOW NO.	ORDER NO.	CHARGE NO.		CHARTED BY
	35239-1	825-1202	0601000	Campbell

FOR	COMBINATION
NASA	SARAN COAT .00025" METALLIZED MYLAR

APPLICATION METHODS (Check Method Used)

- [X] 1. DIRECT GRAVURE
- [ ] 2. REVERSE ROLL
- [ ] 3. HOT MELT
- [ ] 4. DIRECT TRANSFER + MAYER ROD
- [ ] 5. TRANSFER ROLL APPLICATION + SMOOTHING BAR
- OR 6. ____________

~~XXXXXX~~E COATING

WT.-LBS/RM	COLOR	TYPE	NO.	VISCOSITY	SOLVENT NO.	SOLVENT TYPE
3.0	C1	Saran	QX-2364.2	35	---	MEK

REVERSE ROLL COATING

GEAR RATIO	ROLL SPEED	DOCTOR ROLL GAP

WET LAMINATION

CYLINDER ETCH	SMOOTHING BAR	IMP. ROLL TEMP.	AIR KNIFE PRESSURE	AIR IMPINGEMENT ANG.	MAYER ROD NUMBER
55 Q	Yes - Fixed				

HOT MELT COATING

POLISHING ROLL TEMP.	FOUNTAIN TEMP.	MAYER ROD TEMP.	MAYER ROD NUMBER

DRY LAMINATION

HOT ROLL TEMP.	WRAP	COOL ROLL TEMP.	#1	#2

LAMINATOR LINE SPEED	SCRAP	WEIGHT OFF
100 FPM		

		PRIMARY	SECONDARY	
OVEN	TEMPERATURE	200°F.	235°F.	
	DAMPERS	Full Open	Full Open	
	VENT OPENINGS	¼	½	
STOCK	NUMBER	Metallized		
	TYPE	Mylar Polyester		
	SIZE	19½"		
	GAUGE	.00025"		
	QUANTITY			
SHRINKAGE	WET END			
	COMB. END			
	REWIND			

COMMENTS

550

LAMINATOR DATA SHEET   CPRL PILOT PLANT

DATE
1/14/71

DOW NO.	ORDER NO.	CHARGE NO.	CHARTED BY
	35239-3	825-1202 0601000	Campbell

FOR	COMBINATION
NASA	Mylar-Saran Coated/Metallized Mylar

APPLICATION METHODS (Check Method Used)

- [X] 1. DIRECT GRAVURE
- [ ] 2. REVERSE ROLL
- [ ] 3. HOT MELT
- [ ] 4. DIRECT TRANSFER + MAYER ROD
- [ ] 5. TRANSFER ROLL APPLICATION + SMOOTHING BAR
- OR 6. ______

ADHESIVE							REVERSE ROLL COATING		
WT.-LBS/RM	COLOR	TYPE	NO.	VISCOSITY	SOLVENT NO.	SOLVENT TYPE	GEAR RATIO	ROLL SPEED	DOCTOR ROLL GAP
2.0	C1	Ure-thane	Uno-flex	15	--	Tol			

WET LAMINATION					
CYLINDER ETCH	SMOOTHING BAR	IMP. ROLL TEMP.	AIR KNIFE PRESSURE	AIR IMPINGEMENT ANG.	MAYER ROD NUMBER
110 Q	No				

HOT MELT COATING				DRY LAMINATION				
POLISHING ROLL TEMP.	FOUNTAIN TEMP.	MAYER ROD TEMP.	MAYER ROD NUMBER	HOT ROLL TEMP.	WRAP	COOL ROLL TEMP.	#1	#2

LAMINATOR LINE SPEED	SCRAP	WEIGHT OFF

		PRIMARY	SECONDARY	
OVEN	TEMPERATURE	125°F.	150°F.	
	DAMPERS	Full Open	Full Open	
	VENT OPENINGS	¼	½	
STOCK	NUMBER	Metallized & Saran		
	TYPE	Coated Mylar	Mylar Polyester	
	SIZE	19-1/8	19-1/2	
	GAUGE	.00025"	.00015"	
	QUANTITY		1000 ft.	
SHRINKAGE	WET END			
	COMB. END			
	REWIND			

COMMENTS

Combination ran well and looked good.

550

LAMINATOR DATA SHEET CPRL PILOT PLANT

DOW NO.	ORDER NO.	CHARGE NO.		DATE / CHARTED BY
	35239-4	825-1202	0601000	DATE 1/14/71; CHARTED BY Campbell

FOR	COMBINATION
NASA	

APPLICATION METHODS (Check Method Used)

- [X] 1. DIRECT GRAVURE
- [ ] 2. REVERSE ROLL
- [ ] 3. HOT MELT
- [ ] 4. DIRECT TRANSFER + MAYER ROD
- [ ] 5. TRANSFER ROLL APPLICATION + SMOOTHING BAR
- OR 6. ______________________.

ADHESIVE							REVERSE ROLL COATING		
WT.-LBS/RM	COLOR	TYPE	NO.	VISCOSITY	SOLVENT NO.	SOLVENT TYPE	GEAR RATIO	ROLL SPEED	DOCTOR ROLL GAP
2.0	Cl	Ure-thane	Uno-flex	15	--	Tol			

WET LAMINATION

CYLINDER ETCH	SMOOTHING BAR	IMP. ROLL TEMP.	AIR KNIFE PRESSURE	AIR IMPINGEMENT ANG.	MAYER ROD NUMBER
110 Q	No				

HOT MELT COATING				DRY LAMINATION				
POLISHING ROLL TEMP.	FOUNTAIN TEMP.	MAYER ROD TEMP.	MAYER ROD NUMBER	HOT ROLL TEMP.	WRAP	COOL ROLL TEMP.	#1	#2

LAMINATOR LINE SPEED	SCRAP	WEIGHT OFF

		PRIMARY	SECONDARY	
OVEN	TEMPERATURE	125°F.	150°F.	
	DAMPERS	Full Open	Full Open	
	VENT OPENINGS	1/4	1/2	
STOCK	NUMBER	Metallized & Saran	Metallized	
	TYPE	Coated Mylar	Mylar Polyester	
	SIZE	19-1/8"	20"	
	GAUGE	.00025"	.00015"	
	QUANTITY		1100 ft.	
SHRINKAGE	WET END			
	COMB. END			
	REWIND			

COMMENTS

Combination ran well and looked good in finished roll.

550

LAMINATOR DATA SHEET   CPRL PILOT PLANT

DATE	1/21/71

DOW NO.	ORDER NO.	CHARGE NO.		CHARTED BY
	35240-1	825-1202	0601000	

FOR	COMBINATION
NASA	MYLAR METALLIZED/SARAN COATING

APPLICATION METHODS (Check Method Used)

- [X] 1. DIRECT GRAVURE
- [ ] 2. REVERSE ROLL
- [ ] 3. HOT MELT
- [ ] 4. DIRECT TRANSFER + MAYER ROD
- [ ] 5. TRANSFER ROLL APPLICATION + SMOOTHING BAR
- OR 6. ____________

ADHESIVE							REVERSE ROLL COATING		
WT.-LBS/RM	COLOR	TYPE	NO.	VISCOSITY	SOLVENT NO.	SOLVENT TYPE	GEAR RATIO	ROLL SPEED	DOCTOR ROLL GAP
3	C1	Saran	OX-2364.2	35	---	MEK			

WET LAMINATION

CYLINDER ETCH	SMOOTHING BAR	IMP. ROLL TEMP.	AIR KNIFE PRESSURE	AIR IMPINGEMENT ANG.	MAYER ROD NUMBER
55 Q	Yes - Fixed				

HOT MELT COATING				DRY LAMINATION				
POLISHING ROLL TEMP.	FOUNTAIN TEMP.	MAYER ROD TEMP.	MAYER ROD NUMBER	HOT ROLL TEMP.	WRAP	COOL ROLL TEMP.	#1	#2

LAMINATOR LINE SPEED	SCRAP	WEIGHT OFF

		PRIMARY	SECONDARY	
OVEN	TEMPERATURE	200°F.	235°F.	
	DAMPERS	Full Open	Full Open	
	VENT OPENINGS	¼	½	
STOCK	NUMBER	Metallized		
	TYPE	Mylar Polyester		
	SIZE	19½"		
	GAUGE	.00025"		
	QUANTITY			
SHRINKAGE	WET END			
	COMB. END			
	REWIND			

COMMENTS

550

LAMINATOR DATA SHEET   CPRL PILOT PLANT

DOW NO.	ORDER NO.	CHARGE NO.	DATE / CHARTED BY
	35240-2	825-1202 0601000	DATE: 1/22/71 CHARTED BY: Campbell

FOR: NASA

COMBINATION: SARAN CTD. METALLIZED MYLAR/HEAT RESISTANT COATING

APPLICATION METHODS (Check Method Used)

- [X] 1. DIRECT GRAVURE
- [ ] 2. REVERSE ROLL
- [ ] 3. HOT MELT
- [ ] 4. DIRECT TRANSFER + MAYER ROD
- [ ] 5. TRANSFER ROLL APPLICATION + SMOOTHING BAR
- OR 6. ______________________

ADHESIVE							REVERSE ROLL COATING		
WT.-LBS/RM	COLOR	TYPE	NO.	VISCOSITY	SOLVENT NO.	SOLVENT TYPE	GEAR RATIO	ROLL SPEED	DOCTOR ROLL GAP
1	C1	N/C							

WET LAMINATION					
CYLINDER ETCH	SMOOTHING BAR	IMP. ROLL TEMP.	AIR KNIFE PRESSURE	AIR IMPINGEMENT ANG.	MAYER ROD NUMBER
150	No				

HOT MELT COATING				DRY LAMINATION				
POLISHING ROLL TEMP.	FOUNTAIN TEMP.	MAYER ROD TEMP.	MAYER ROD NUMBER	HOT ROLL TEMP.	WRAP	COOL ROLL TEMP.	#1	#2

LAMINATOR LINE SPEED	SCRAP	WEIGHT OFF

		PRIMARY	SECONDARY	
OVEN	TEMPERATURE	125°F.	150°F.	
	DAMPERS	Full Open	Full Open	
	VENT OPENINGS	¼"	½"	
STOCK	NUMBER	Metallized & Saran		
	TYPE	Coated Mylar		
	SIZE	19-1/8"		
	GAUGE	.00025"		
	QUANTITY	Approx. 2500 ft.		
SHRINKAGE	WET END			
	COMB. END			
	REWIND			

COMMENTS

SOME BAG AT EDGE OF METALLIZED, SARAN COATED FILM.

550