

Evaluations of an Aramid Fiber in Nonwoven Processes for Honeycomb Applications

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ABSTRACT: Aromatic polyamide paper has been used as the primary honeycomb core material in aircraft interior applications because of its flame-resistant properties. This study investigated the relationships between material requirements in making sheets for honeycomb core applications by characterizing and processing a model aramid fiber into nonwoven samples, which were further characterized

and whose adaptability in honeycomb application was evaluated. © 2002 Wiley Periodicals, Inc. *J Appl Polym Sci* 86: 1149–1156, 2002

Key words: aramid; nonwoven; honeycomb; structure–property relations

INTRODUCTION

Honeycomb has been used as a lightweight core in a sandwich structure for structural applications because of its high strength-to-weight ratio. A honeycomb sandwich structure is especially desirable in the aerospace industry where weight is a sensitive and crucial parameter.^{1,2} In airplane interior applications, honeycomb made from resin-impregnated Nomex® paper is utilized. Nomex is an aramid material and is selected over other honeycomb materials mainly because of its excellent flammability characteristics.^{3–5} It is desirable to develop an alternative material, however, because the material cost of Nomex honeycomb is higher than that of other honeycomb materials.

Previous research efforts in the honeycomb area have focused studies mainly on the performance, process, and property of the overall sandwich structure.^{1,6–11} However, attempts to understand honeycomb core's performance with respect to core material and processing requirements are limited. It is well known that raw material properties influence process conditions, structures, and properties of the final product. Moreover, existing manufacturing conditions and new product requirements could dictate the choice of raw materials.

The objective of this study was to investigate the relationships between material requirements in making a paper honeycomb core and its end use.

This knowledge is important to develop process parameters, which could enable a better understanding of the honeycomb manufacturing process as well as an improved quality control. A model aramid fiber was characterized and processed in this study using two nonwoven processes. This study focused on the characterization of fiber materials, understanding of sheet formation processes using aramid fibers, and the limitations of processing new materials with existing equipment. Fiber characterizations were first assessed, followed by sheet characterizations using thermal analysis, microscopy, and mechanical analysis.

EXPERIMENTAL

Materials

Commercially available Nomex® 410 paper (DuPont Advanced Fiber Systems) was selected as a comparison to the model material. Figure 1 depicts the chemical structure of Nomex, poly(*m*-phenylene isophthalamide). Note that Nomex 410 paper is used in electrical applications, whereas Nomex 412 paper is used for honeycomb applications. These different papers are processed differently but both are manufactured from the aramid material, which is Nomex. Detail process descriptions of Nomex paper can be found in the literature.¹²

The model aramid material, with the chemical structure shown in Figure 2, is a polyamide-imide polymer. The model material was supplied in the forms of overdrawn (OD) and nonoverdrawn (non-OD) fibers in tow. Overdrawn fibers are formed by further stretching the non-OD fibers to provide more strength

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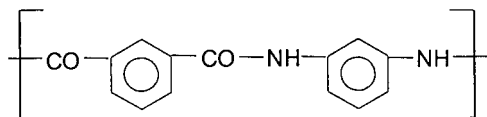


Figure 1 Chemical structure of Nomex material.

to the fiber in the longitudinal direction. The draw ratio (i.e., the ratio of the final length to the initial length) was slightly over 3.

Web processing

Web samples were manufactured from the model fibers by two process methods: handsheet formation and a dry laying process. Table I lists the model fibers and model samples that were tested for the study.

Samples P10–P13 were handsheets that were processed in the laboratory as follows. First, non-OD model fibers were cut into short segments approximately 0.3 to 1.0 cm. Many fines were generated in the cutting process, given that the fiber was relatively weak without further drawing. A mixture of chopped fiber with water (10 wt % fiber) was refined to 11,000 revolutions in a PFI mill according to the TAPPI standard 248.¹³ Handsheets were then made according to the TAPPI standard 205¹⁴ using the refined model fiber, cellulose fiber, and starch. Following the standard method, the areal weight of the handsheets was approximately 64 g/m². These handsheets were further treated by pressing them in a hydraulic press (Tetrahedron) at 211°C (412°F) and 50 kN (11 klb_f) for 1 min. Samples in the paper or web forms were pressed for better consolidation by thermal bonding. By thermal pressing, the surface smoothness of the samples was also improved.

Unlike handsheet samples, dry laying samples were not made in the laboratory. Instead, they were supplied and specially manufactured by an outside source. The dry laying samples contained the model OD fibers and also poly(phenylene sulfide) (PPS) fibers as an additional binder in the fiber bed. Long fibers (50 to 60 mm) were carded to form the fiber web and a thermobonding (calendering) step was then applied to the fiber web. The PPS binder was consolidated by hot melt to fill holes in the fiber bed and therefore would resist resin permeation through the web as well. The dry laying samples were compared through four variables: differences in areal weight,

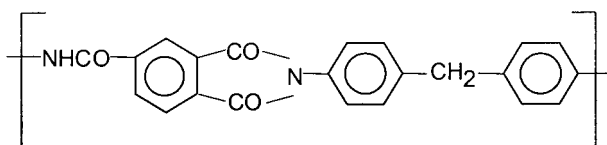


Figure 2 Chemical structure of the model material.

TABLE I
Samples Made from the Model Polymer Fiber Material

Sample	Description
Fibers	
F1	Non-OD model fiber in tow
F2	OD model fiber in tow
Handsheets	
P10	75% non-OD model fiber; 25% cellulose pulp; 0.6 g starch
P11	P10 pressed
P12	75% non-OD model fiber; 25% cellulose pulp; 0.3 g starch
P13	P12 pressed
Dry laying	
W2	70% OD model fiber; 30% PPS; 25 g/m ²
W3	W2 plus a 19 g/m ² of model material as a coating (single coating) on both sides; 44 g/m ²
W4	W2 plus two layers of model material coatings (double coating); 63 g/m ²
W5	70% OD model fiber; 30% PPS; 50 g/m ²
W6	W5 plus a 15 g/m ² of model material as a coating (single coating) on both sides; 65 g/m ²
W7	Double-calendered W5; 50 g/m ²
W9	50% OD model fiber; 50% PPS; 70 g/m ²
W10	50% OD model fiber; 50% PPS; 50 g/m ²

coating effects, double-calendering effects, and fiber/PPS ratios. For example, as shown in Table I, samples W2 and W5 were made by the same process but one is heavier than the other. Sample W3 is a coated W2, which was made by taking sample W2 through a bath that contained the model material.

Analyses

Samples were analyzed by various techniques to evaluate their physical and mechanical properties. Thermal analysis was utilized as the first step to determine the physical properties of the material, such as T_g , T_m , degradation temperature, and moisture content. Specifically, thermal analysis was used as the base comparison between the commercial and model aramid materials. Differential scanning calorimetry (DSC 910; TA Instruments, New Castle, DE), thermal gravimetric analysis (TGA; TA Instruments TGA 2950), dynamic mechanical analysis (TA Instruments DMA 983), and thermal mechanical analysis (TA Instruments TMA 2940) were utilized.^{15, 16} TGA analysis determined the upper limit of the material usage, which was the degradation temperature of the material. DSC analysis was used mainly to detect the T_g of the polymer, moisture content of the material, and other curing reactions or melting peaks that may have been occurring in the samples.

TMA analysis was conducted mostly on samples to detect the T_g of the material. In general, all thermal analyses were conducted under a nitrogen gas envi-

ronment with a heating rate of 10 or 20°C/min. The film samples used in the TMA were approximately 2.54 cm long and 0.5 cm wide and were under a constant normal stress of 0.001 newton.

In addition to thermal analysis, mechanical properties, particularly tensile strength and surface strength, of most samples were evaluated. The tensile strength of samples was evaluated according to ASTM standard D828.¹⁷ An EJA tensile tester (THWING ALBERT Intellect-TI-STD Dual Screw Material testing instrument) with a 1000-lb. load cell was used. A strip of paper was clamped vertically between the two instrument grips. The length of paper between the two grips was 10 cm and the width of paper was 1.5 cm. The grips moved at a constant rate of 2.54 cm/min, pulling the paper apart. Given the anisotropic nature of the sample, tests were conducted both in the machine direction and the cross direction. A total of five tests were performed on each sample and the average tensile modulus was reported.

DMA 2980 (TA Instruments) was also used in the TMA mode to obtain comparative tensile information of handsheets that were not large enough to be tested by the ASTM D828 method. A sample strip approximately 17 × 4 mm was elongated in a controlled force speed of 0.5 N/min at a constant temperature of 30°C. In contrast to the ASTM D828 method, the controlled force rate was employed instead of controlled pulling speed.

The surface strength of samples was determined by the method ASTM D2482 (equivalent to -TAPPI T459), which is also called the wax pick test. The method is designed to measure the resistance of the paper from picking with waxes numbered from 2 to 26. Waxes numbered from 9 to 23 were available during this study, however. A higher numbered wax indicates its better adhesion to the paper. The test was done on both the wire (bottom) side and top side of the paper. The wax pick test gives the "critical wax strength number" (CWSN), which is the average highest numerical designation of the wax that does not disturb the surface of the paper. Several samples were tested: regular Xerox paper, Nomex 410, W5, W6, and W7. Note that the reproducibility of this test is low because the results depend highly on the personnel and/or equipment. Therefore, all tests in this study were conducted in a short period of time by one person with one set of equipment.

Finally, morphologies of the sample cross section and surfaces were studied by either optical microscopy or scanning electron microscopy (SEM).

RESULTS AND DISCUSSION

Thermal analysis of aramid materials

Material characteristics evaluated by thermal analysis are summarized in Table II. From TGA, both the com-

TABLE II
Physical Characterizations of Samples as Determined by Thermal Analysis

Sample	Thermal analysis summary
Nomex 410	Degradation temperature at 400°C by TGA $T_g = 280^\circ\text{C}$; endotherm at 110°C by DSC
F1	Degradation temperature at 400°C by TGA $T_g = 320^\circ\text{C}$ by TMA
F2	Degradation temperature at 400°C by TGA $T_g = 330^\circ\text{C}$ by TMA
W9	$T_g = 320^\circ\text{C}$ by TMA; $T_g = 330^\circ\text{C}$ by DSC Endotherm at 110 and 280°C by DSC

mercial paper and model fibers (samples F1 and F2) were found to lose weight significantly after 400°C, indicating degradation. A weight loss of 4–5% was seen in both materials before 100°C, suggesting a volatile, possibly water, being removed from the materials. DSC analysis also showed an endothermic signal at 100°C, indicating moisture desorption from the material. The glass-transition temperature (T_g) was determined to be 280°C for Nomex material and 320–330°C for model materials. The measured glass transition of Nomex corresponded with previous literature value.^{3,18–20} The OD model fibers showed a slight increase in the T_g value (330°C) compared to that of the non-OD fibers (320°C), perhaps because of further alignment in molecular structures of the OD fibers.

Both DSC and TMA could detect the T_g of samples, although TMA was preferred in this study. Difficulties in interpreting DSC and TMA data for aramid fibers were discussed previously in the literature.¹⁸ However, the onset and the transition temperatures in the TMA analysis were not precise because of the extremely small fiber diameters. Any defect in the fiber or variations in fiber diameters were found to cause inconsistencies in the transitions observed. In addition, difficulties in obtaining exact sample length in TMA analysis affected the transition temperature as well. On the contrary, few variations were observed from the DSC analysis attributed to sample or fiber differences. However, although some samples showed transitions comparable to T_g transitions, for other samples no transition was seen. It may be that, because the aramid materials are highly oriented, the crystalline portion in the sample masked the glass transition from the amorphous portion of the sample.¹⁶ Although fibers were highly oriented, there were no melting points' (T_m) detected in the DSC analysis for either Nomex or model fibers. This observation may be attributed to the fact that aramid materials are liquid crystalline polymers.

The model material had a higher T_g than that of the Nomex material. This higher T_g may provide broader applications for the model material because it could be utilized at a higher temperature than the Nomex ma-

TABLE III
Tensile Properties of Model Material Handsheets and Nomex Paper

Sample	Tensile strength ^a (N/m)	Sample thickness (mm)	Tensile modulus (MPa)
P10	580	0.16	1.7
P11	660	0.10	3.6
P12	410	0.14	0.4
P13	410	0.08	3.0
Nomex 410			
Cross direction	2160	0.04	26.0
Machine direction	2160	0.04	41.7

^aCalculated from the DMA 2980 analysis using the tension clamp.

terial. On the other hand, a high T_g may require higher costs in manufacturing, given that high-temperature equipment may be needed. For example, the model material may have the disadvantage of requiring a high calendering temperature, in that calendering requires a temperature just below material's T_g to be effective.

Handsheet samples

In forming handsheets, fibers were refined. For this study, the necessity of refining synthetic fibers before handsheet formation was apparent from the experimental trials. It was realized that unrefined fibers could not form a handsheet because there were no forces binding fibers together and the fiber-water mixture (pulp) was analogous to short human hair strands placed in water. Refined model fibers showed improvements over the unrefined model fibers in the ability to retain more water and to disperse better in water, which may have contributed to rough fiber surfaces. However, handsheet made from pure 100% model fibers still was not possible, despite the 2improvement after fiber refining, because patches of fiber web could not separate completely from the wire mesh of the handsheet machine.

The 100% refined model fibers could not be formed into a handsheet; thus another pulp (cellulose pulp) and a wet strength additive (starch) were added to the handsheet process. A handsheet was formed by adding a minimum of 25% wood pulp and 0.3 g of starch to the refined model fibers in the handsheet-formation process. Handsheets were then pressed to compare the differences to nonpressed handsheets. From experimental observations, pressed handsheets had smooth surfaces like a regular paper and nonpressed handsheets had rough surfaces similar to the surface of a paper towel.

Because of the limited sample size, the tensile strength and modulus of the handsheet were not evaluated by the standard ASTM D828 method. Instead, the tensile strength of the handsheet samples was measured using the DMA tension clamp of the equip-

ment DMA 2980 (TA Instruments), which enabled us to determine the tensile properties of the samples using a small sample size. The tensile strength was then determined from maximum tensile force and the tensile modulus was calculated from the initial slopes over the sample thickness.

The calculated data are shown in Table III. The data show that by pressing the handsheet, sample thickness decreased and its tensile properties increased. By adding more wet strength additives (starch) in the handsheet-formation process, the tensile strength increased. As Table III indicates, doubling the amount of starch added, from 0.3 to 0.6 g, increased the tensile strength of the sample by 40%. A higher starch content in the handsheet process enhanced the interfiber bonding and therefore increased the tensile properties. However, the exact compositions of starch and cellulose pulp in the handsheet could not be determined because both starch and cellulose pulp degraded at the same temperature range. Figure 3 shows the TGA analysis of the two samples containing 75% model fiber with 0.3 g (sample P12) and 75% model fiber 0.6 g of starch (sample P10). Both samples showed two distinct degradation steps, one with onset at 300°C and the other with onset at 400°C. The first degradation was attributed to cellulose pulp and starch, whereas the second degradation was attributed to the model aramid fibers. Although a different amount of starch was added into the process, the TGA analysis could not distinguish one sample from another. One possible explanation may be that the actual starch amount retained on the handsheet was too small for the TGA to detect.

The morphologies of the samples are shown in Figures 4 and 5. In these two micrographs, the handsheet showed distinguishable fibers and small particles. The small particles may be starch added in the handsheet process and the fibers were model aramid fibers or cellulose fibers. The differences between man-made fibers and cellulose fibers are easily distinguished in these micrographs. In comparison, the wood fibers are smaller and shown as a ribbon shape instead of a rod as in Figure 5. Because cellulose fibers are hollow, the

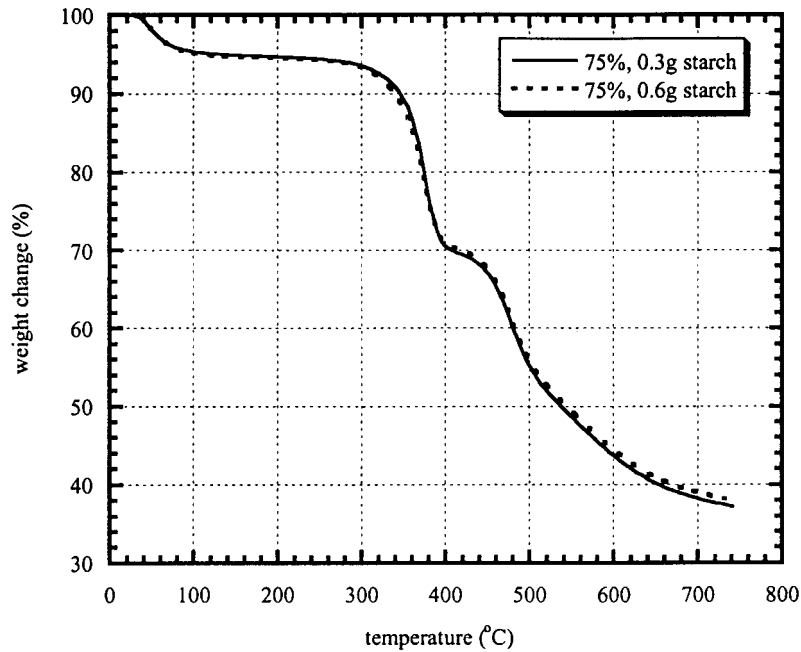


Figure 3 TGA analysis of model-fiber handsheets.

refining process puts pressure on the fibers, forcing the circular diameter fibers to collapse into a ribbon shape, so that each fiber can provide a larger contacting area while improving the overall ability to retain water.²¹ In contrast, model aramid fibers, which are not hollow, appeared rodlike, whereas some were split into two ends, as shown in Figure 4. This finding indicated that the refining process on the synthetic fibers forced fibers to split rather than to be deformed.

Although the addition of wood pulp and starch increased interfiber bonding of the handsheets, they caused model handsheets to degrade at an earlier temperature of 310°C instead of 400°C with pure model material. Furthermore, a simple Bunsen burner test indicated that the flame-retardant feature of the

handsheet was reduced. Once removed from the flame source, the handsheet samples continued to burn, whereas the pure model material only charred. Because fire-retardant properties are very important for aerospace usage, it is suggested that nonflammable additives be used in the future studies.

Dry laying samples

Dry laying samples showed an endothermic peak at 280°C in DSC analysis, which corresponded to the melting point of PPS. Table II shows a summary of results from thermal analysis for the sample W9. For these dry laying samples, 280°C would be the absolute upper temperature limit in applications because of PPS addition.

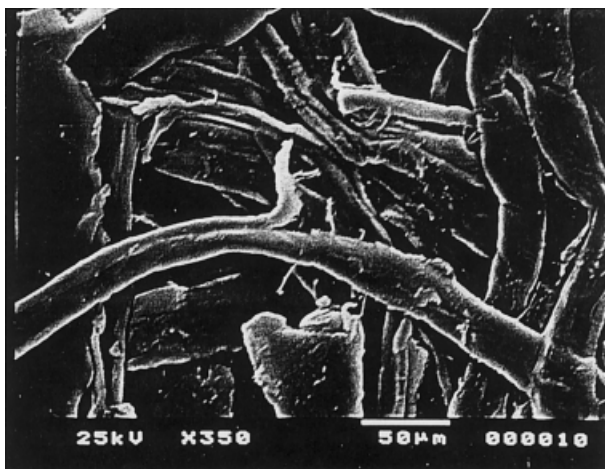


Figure 4 Surface microscopy of handsheet structure made from model-fiber material (×350).

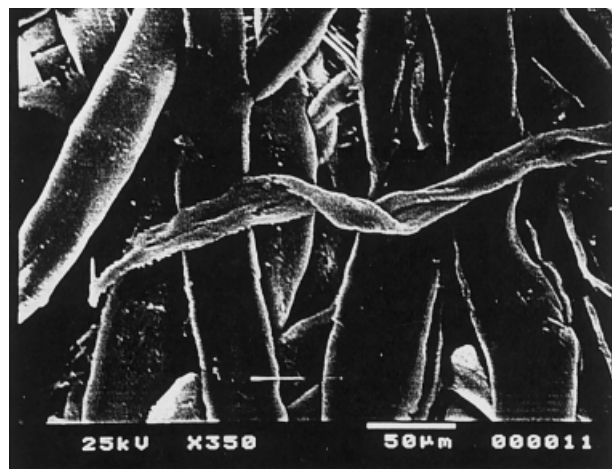


Figure 5 Another surface microscopy of handsheet structure made from model-fiber material (×350).

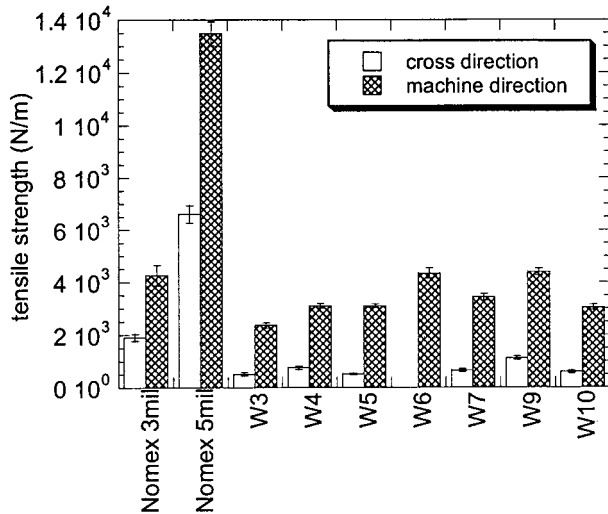


Figure 6 Tensile strength of samples, using ASTM D828 method.

The tensile strength of most as-received web samples was tested with the ASTM D828 method, as shown in Figure 6. Note that the results obtained from the ASTM D828 method were not compared directly to the results obtained from the DMA tension clamp for the handsheet samples because of sample size differences and different testing techniques. The DMA used a controlled force rate, whereas the ASTM D828 method required a controlled pulling speed.

The tensile strength of Nomex 410 3-mil and 5-mil papers was tested in the laboratory, the results of which are listed in Figure 6. Comparing experimental data to values in the DuPont technical brochure, the experimentally measured values were lower than what were indicated in the technical brochure specifically for Nomex 3-mil papers, which was 35% lower than indicated values, even though the same standard test method was followed.

In Figure 6 there were no data for sample W6 in the cross direction because samples were not long enough in the cross direction to perform the testing. Sample W2 was tested; however, no values were recorded because the tester did not catch failure, even when the sample was totally separated. Coated samples improved the tensile strength properties over those of the noncoated samples, as indicated in Figure 6 for

samples W2–W5 and W5 versus W6. Sample W5, which was made using the same process as W2 but twice as heavy (areal weight of 50 versus 25 g/m²), was a stronger material than W2. It was noted that a heavier nonwoven web increased the tensile strength more efficiently than coating a lighter nonwoven web when both were the same areal weight. For example, sample W3 showed lower tensile strength values than W5, although both samples had similar areal weight. Also, sample W4 had lower tensile strength values compared to those of sample W6, even though both had similar areal weight. It was also noted that most dry laying samples with similar areal weights exhibited similar tensile strength values in both the fiber direction and machine direction. For example, samples W6 and W9, and samples W5 and W7 that had similar areal weights had similar values in tensile strength.

Comparing samples W5 and W7, one found that double-calendering dry laying samples increased the tensile strength of one-pass calendered samples only slightly. Also, comparing samples W10 and W5, which had the same areal weight and the same processing steps but had different model material/PPS ratio, one found that changing the fiber/binder ratio had only a slight effect on tensile strength of the sample. It was then concluded that calendering and fiber/binder ratio were not significant parameters in improving the tensile strength of dry laying samples.

Of these model samples, W6 and W9 had similar areal weight and tensile strength in the machine direction to the Nomex 3-mil tested. However, the strength in the cross direction was 33% lower than that of the Nomex 3-mil papers. Nomex papers showed an approximately a 2-to-1 ratio between the tensile strength in both fiber and cross directions, whereas model fiber samples showed more than 3-to-1 ratios. The lower tensile strength in the cross direction may be the result of different processing conditions in the commercial paper and in model samples.

The surface strength of dry laying samples W5–W7 was tested and the results, critical wax strength number (CWSN), are summarized in Table IV. The CWSN is the average highest numerical designation of the wax that does not disturb the surface of the paper. The results of these samples were compared to commercial

TABLE IV
Surface Strength of Samples by the Wax Pick Test

Sample	CWSN	Notes
Xerox paper	16	Surface ruptured completely at wax number 18.
Nomex 410, 3-mil	>23	There were no surface disturbances observed at wax number 23.
W5	9–12	Few fiber strands broke.
W6	<9	Surface coating lifted with wax number lower than 9. Surface coating lifted and few fibers broke at wax number 14.
W7	18–23	Few fiber strands broke.

papers such as regular Xerox paper and Nomex 410, which are also listed in Table IV. The results in Table IV show that regular Xerox paper had a CWSN of 17 and Nomex paper was above 23. The surface strength, however, of dry laying samples varied, depending on the process parameters. For example, double-calendered web samples showed a greater increase in CWSN than that of their one-pass calendered web samples (comparing samples W5 and W7). This finding may imply that double-calendered web samples had desirable surface properties. Coated samples had a smooth surface, yet they exhibited separations from the coating and fiber web interlayer region during the wax pick test and gave no improvement in the surface strength compared to that of the noncoated samples, as seen in samples W5 and W6. The purpose of a coating material was to alter the surface properties such as surface smoothness and resin permeation rate, while simultaneously maintaining the sample's integrity by acting as a load transfer media between fibers. Results indicated that the coated layer adhered easily with the wax stick and readily delaminated from the fiber web. Coated samples that were further processed with the calendering step did not show much improvement on surface strength compared to that of noncalendered, coated samples. This finding may imply that, although the calendering process improved the surface smoothness, it did not enhance the bonding between fiber web and the coating layer. Of all the model fiber samples tested, sample W7 had the highest CWSN value. Sample W7 was a double-calendered fiber web containing 70% model fibers with 30% PPS fibers.

The tensile and surface properties of the samples were examined together to compare the various processing conditions. Samples W5, W6, and W7 had the same web formation and thus were compared. Comparing samples W5, W6, and W7 from Figure 6 and Table IV, the coated sample (W6) showed improvement on tensile strength, although the surface strength worsened. The double-calendered sample (W7) showed a slight improvement in tensile strength and surface strength compared to those of the one-pass calendered sample (W5). Overall, it could be said that samples without a separate coating layer are preferred and a calendering step after web formation is favorable for the honeycomb application. However, although including the calendering step in the process improved the strength slightly whereas coating the samples improved the strength considerably, these model samples still required higher strength in the cross direction and better surface integrity.

The differences in dry laying sample structures that resulted in different mechanical properties could be seen from optical analysis. Sheets made from model materials showed different internal structures based on different process methods. Figure 7 is a SEM image

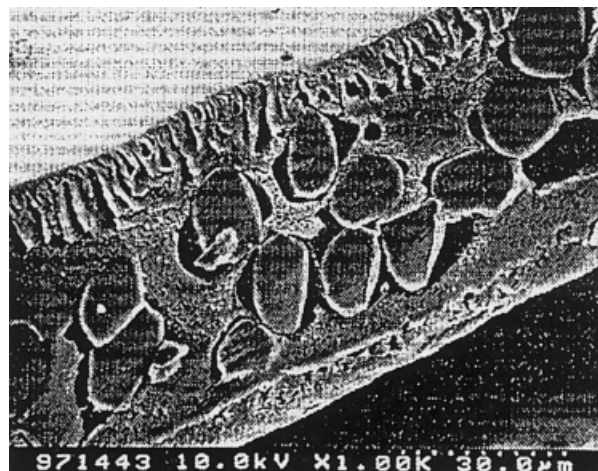


Figure 7 Cross section of sample W3 by SEM ($\times 100$).

of the cross section of sample W3, which is a coated dry laying sample. The picture shows that sample W3 contained fibers of circular diameter with coating material above and below the fiber web. Sheets formed with circular diameter fibers instead of ribbonlike fibers tend to be weaker because of fewer surfaces interlocking between fibers.²¹ Furthermore, Figure 7 shows that the sample contained numerous voids at the model fiber-PPS binder interface and in the coating layers near surfaces. As mentioned previously, the coating material was intended as a load-transfer medium between fibers so that the surface properties could be improved from the noncoated fiber web. However, the process method that was used to produce sample W3 resulted in many voids in the sample structure, which created a weak fiber-coating interface and a weak coating layer. This weak fiber-coating interface could explain the observed results from the surface test for all coated samples, where the coating layer adhered to the wax stick and was peeled off from the fiber web.

CONCLUSIONS

An aramid model material was investigated as an alternative honeycomb paper source. This model material was processed from fibers into webs by two processes: handsheet and dry laying. The samples were analyzed through thermal, mechanical, and optical characterizations. From this study, it was found that the model fiber had a glass transition around 320–330°C and degraded at 400°C. Handsheets were made from refined nonoverdrawn model fibers mixed with cellulose fibers and starch particles. The presence of cellulose fibers ensured the formation of the handsheet, whereas the addition of starch further improved the tensile strength. However, the presence of cellulose fibers and starch resulted in a flammable handsheet that was not desirable.

Dry laying samples were made to compare four variables, which were areal weight, fiber/binder ratio, coating layers, and double-calendering, in the processing. Samples of similar areal weight were found to show similar results in tensile strength. Generally, tensile strength increased as sample areal weight was increased. However, changes in the fiber/binder ratio did not affect the results in tensile strength. Through mechanical tests, results were improved for dry laying samples that were coated compared to samples not coated, although surface strength was decreased significantly with coated samples. Double-calendering samples improved the surface strength significantly but improved the tensile strength of samples only slightly.

Two samples, W6 and W9, showed similar areal weight and tensile strength in fiber direction as those of the Nomex 3-mil papers, yet the tensile strength in the cross direction and surface strength were significantly lower than those in Nomex papers. The weaknesses in tensile strength in the cross direction and in surface strength of the samples were indicated from optical analysis where voids were found in the samples. Further explorations in fiber structure modification by pretreatment and in using additives in improving properties need to be considered in future studies.

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