

# The Effect of Maleic Anhydride Modified Polypropylene on the Mechanical Properties of Feather Fiber, Kraft Pulp, Polypropylene Composites

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**ABSTRACT:** Composites made of feather fiber (Ff), kraft pulp fiber (Pf), polypropylene (PP), and maleic anhydride modified polypropylene (MaPP) were tested in tension and three-point bend tests. Composite panels were compression molded from multiple plies of nonwoven, fabric-like prepreg manufactured with wetlay papermaking equipment. Composites containing all four materials were manufactured with MaPP substitutions for PP of 0–10 wt % at 2 wt % increments. The best mechanical properties were observed with a substitution of 8 wt % MaPP. Composites containing either Pf or Ff were manufactured with MaPP

substitutions for PP of 0, 4, and 8 wt %; mechanical properties improved with increases in MaPP content. The improvements in mechanical properties were attributed to fiber/matrix interface improvement by the addition of MaPP. Scanning electron microscopy revealed evidence of improved interfacial bonding on the tensile fracture surfaces. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 92: 3771–3783, 2004

**Key words:** feathers; cellulose; composites; polypropylene; maleic anhydride modified polypropylene; biofibers; interfaces; mechanical properties; renewable resources

## INTRODUCTION

In recent years, there has been substantial growth in the research, development, and application of biocomposites. A composite containing at least one constituent (e.g., matrix or reinforcement) that is derived from readily renewable resources may be considered a biocomposite. In the year 2000, over 180 million kilograms of natural fiber were used in North America. Over 95% of this \$150 million market consisted of wood flour or fiber used as filler in thermoplastics.<sup>1</sup> However, cellulose fibers from kenaf, hemp, ramie, flax, sisal, coir, and jute are also being used as reinforcement. Some of these fibers are already in the waste stream and therefore are readily available and inexpensive. Utilizing fibers derived from waste not only reduces waste, but reduces the energy consumed to manufacture new materials. Other biofibers are cultivated specifically for use as reinforcement in biocomposites. Although they are not derived from the waste stream, they come from a readily renewable resource, which means their production is less dependent on petroleum supplies, requires one-fourth the energy for

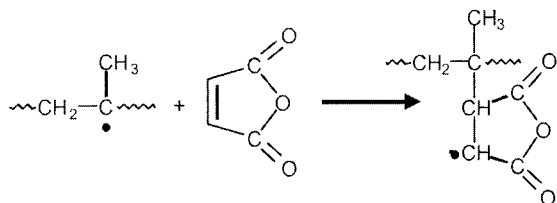
production, and absorbs carbon dioxide while generating oxygen.<sup>2</sup> Biocomposites offer additional advantages such as low density, high strength-to-weight ratio, nonbrittle fracture, and good acoustical and thermal insulation properties.

This study focused on a cellulose biofiber (kraft pulp fibers) and a noncellulose biofiber (poultry feather fibers). Poultry farms in the United States produce about 1–2 billion kilograms of chicken feathers every year.<sup>3</sup> Waste feather is often turned into a low-quality protein supplement for animal feed, by breaking down most of the proteins in the feather, which sells for about \$0.50 per kilogram, making it a marginally profitable venture with supply exceeding demand.<sup>4</sup> In addition to being an abundant byproduct of the poultry industry, feather fibers have a relatively low density of 0.8 g/cm<sup>3</sup> and have good thermal and acoustical insulating properties.<sup>5,6</sup> These properties result from the air pockets within the keratin filaments of the feathers.<sup>7</sup> These characteristics qualify feather fibers as excellent candidates for bulk quantity, inexpensive, lightweight, low-load bearing composite applications such as interior paneling and furniture in automobiles, trucks, homes, offices, and factories.

In 1998, the Agricultural Research Service was awarded a patent for a process of cleaning, chopping, and separating feather fibers from the quill. This patent, authored by Gassner et al., discusses the use of this fiber in polymer composites, filters, absorbent products, or insulation.<sup>8</sup> The quill (about half of the

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**Figure 1** Schematic of the modification of PP with maleic anhydride group.

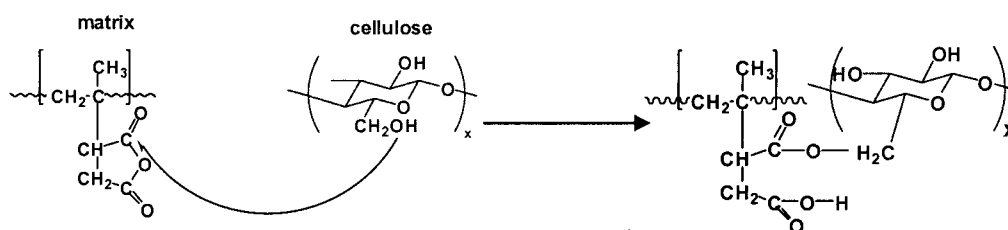
yield) could still be sold as an animal feed. The unique shape of these feather fibers, a center fiber with many branching fibers, makes feathers ideal for random orientation processes such as injection molding, dry mat formation, or wetlay. In composites with thermoset polyesters, feathers were reported to increase strength by 20% and decrease weight by 50%.<sup>1</sup> Dweib et al. used vacuum-assisted resin transfer molding to infuse feather mats along with sheets of recycled paper with soybean oil based resin.<sup>5</sup> These materials were combined with structural foam to construct sandwich beams. The beam of recycled paper and chicken feathers had a global modulus of 950 MPa and a failure load of 24.2 kN compared to the woven E-glass beam, which had a global modulus of 1580 MPa and a failure load of 39.3 kN when tested in four-point bend. The flexural rigidity and strength of the feather/recycled paper beam were comparable to values for cedar wood.

Previous work has demonstrated the capability to manufacture polypropylene (PP) composites containing feather fiber (Ff) (Featherfiber Corp., Nixa, MO) via the wetlay papermaking process.<sup>9,10</sup> Randomly oriented, nonwoven prepreg was manufactured with varying compositions of Ff, glass fiber, reclaimed kraft pulp fiber (Pf) (CreaFill Fibers Corp., Chestertown, MD), kenaf bast fiber, and PP fiber (FiberVisions, Inc., Covington, GA) utilizing a wetlay papermaking process modeled after that described in U.S. patents by Weeks and Geary.<sup>11,12</sup> The facility and processes for wetlay manufacture of composites are detailed in previous publications.<sup>9,10</sup> Processed Ff was found to be thermally stable to approximately 195°C. At temperatures greater than 195°C, degradation was demonstrated by a color change and noticeable odor. These

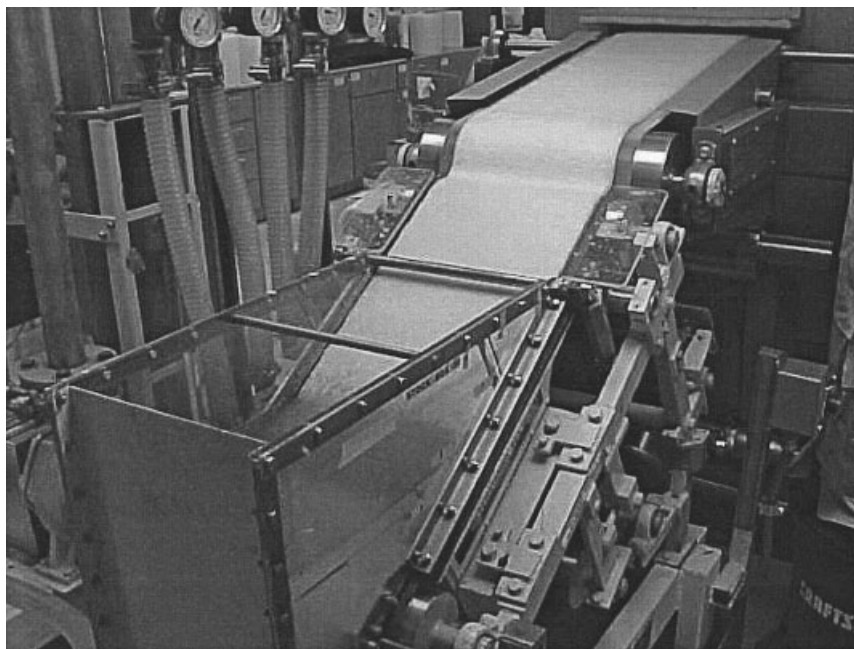
observations agree with Schuster's observation of surface oxidation at 200°C.<sup>13</sup> Thermal stability to 195°C allows for processing with commodity thermoplastics such as polyethylene and PP. However, melt blending and injection molding of filled PP often are performed at temperatures of 200°C or greater, thus exposing feather fiber to degradative conditions for several minutes. The stamping of moldable, filled PP sheets results in a much shorter exposure time to processing temperatures than melt blending processes. Therefore, wetlay papermaking equipment was utilized to manufacture prepreg mats for compression molding.

One of the challenges for creating composites with biofibers and PP is finding ways to improve the bonding between the nonpolar PP matrix and the polar cellulose and keratin fibers. One system that was studied extensively in bonding between PP and cellulose is maleic anhydride modified polypropylene (MaPP). MaPP was shown to increase the strength of wood flour/PP, wood fiber/PP, jute fiber/PP, sisal fiber/PP, and kenaf bast fiber/PP composites.<sup>14–19</sup> MaPP is formed by reacting maleic anhydride (MA) with PP in the presence of an initiator to produce PP chains with pendant MA groups (Fig. 1). The PP portion of MaPP can entangle and cocrystallize with the unmodified PP, while the maleic anhydride groups can bond to the hydroxyl (—OH) groups on the fibers. When mixed with cellulose, the hydroxyl group of the cellulose breaks one of the C—O bonds in the MA group and forms a new bond between one of the carbons from the MaPP group and the oxygen from the cellulose (Fig. 2). The resulting chemical bond between the oxygen of the cellulose and the carbon of the MA group enhances the bond between fiber and matrix.

The strength increases reported for cellulose-reinforced PP suggest that Ff/PP composites might have improved properties with the addition of MaPP.<sup>14–19</sup> The major amino acids of keratin protein, in order of abundance, in feather barbs were determined with nuclear magnetic resonance: serine (16%), proline (12%), glycine (11%), valine (9%), and cysteine (7%).<sup>20</sup> The MA group should be able to bond to the hydroxyl group in serine, the mercapto (—SH) group in cysteine and the amino (—NH<sub>2</sub>) and hydroxyl groups on the ends of the protein chains. Schuster's work with MaPP



**Figure 2** Schematic of the MA group attaching to a hydroxyl group on cellulose.



**Figure 3** Formation of nonwoven mat on the inclined forming wire as it conveys out of the head box.

and feathers suggests a reaction between MaPP and keratin takes place.<sup>13</sup> Schuster added 2 wt % feather to MaPP (2 wt % MA). The MaPP was melt blended with Ff and hot-pressed at 5 MPa and 180°C. With this small amount of feather, the tensile strength improved by 50% and the impact energy improved by 20% over the strength and impact energy of neat MaPP.<sup>13</sup> However, these increases were for MaPP, which is much weaker than PP containing a small addition of MaPP. Therefore, one of the questions addressed in this article is the influence of small substitutions of MaPP for PP containing significant (60 wt %) quantities of feather. For example, a 10 wt % substitution of MaPP (1.0 wt % MA) is equivalent to 0.1 wt % substitution of MA. Furthermore, in the research discussed above,

MaPP was melt-blended with the PP and fibers. Therefore, another question to answer is whether or not MaPP will have an effect at all when placed into a mat (Fig. 3) of Ff, Pf, and PP via the wetlay papermaking process and then compression molded, or, is melt-blending of the MaPP, PP, and reinforcement required to achieve the necessary entanglement and bonding to improve composite strength? Also, the MaPP (Crompton Corp., Middlebury, CT) will be added to the wetlay slurry in chip, not fiber, form. Will these chips disperse well in wetlay and are they small enough to adequately disperse the MaPP throughout the composite?

**EXPERIMENTAL**

**Experimental plan**

The first stage of the experiment analyzed a composite of Pf, Ff, and PP that had previously been successfully manufactured without MaPP by using the wetlay process.<sup>10</sup> The Pf and Ff content were held constant at 30 wt % each. MaPP was substituted for PP such that the sum total of PP and MaPP was held constant at 40 wt %. Six different compositions were prepared with MaPP (1.0 wt % MA) substitutions of 0, 2, 4, 6, 8, and 10 wt % (Table I). The objective of this stage of the experiment was to determine whether MaPP could be successfully transferred to the wetlay mat in chip form, whether it would improve the mechanical properties of the composites manufactured via wetlay, and if so, the quantity of MaPP that yielded the best im-

**TABLE I**  
Experimental Compositions for Ff, Pf, PP, MaPP Composites

Identifier	Ff (wt %)	Pf (wt %)	PP (wt %)	MaPP (wt %)
FPMP0	30	30	40	0
FPMP2	30	30	38	2
FPMP4	30	30	36	4
FPMP6	30	30	34	6
FPMP8	30	30	32	8
FPMP10	30	30	30	10
FMP0	60	0	40	0
FMP4	60	0	36	4
FMP8	60	0	32	8
PMP0	0	60	40	0
PMP4	0	60	36	4
PMP8	0	60	32	8

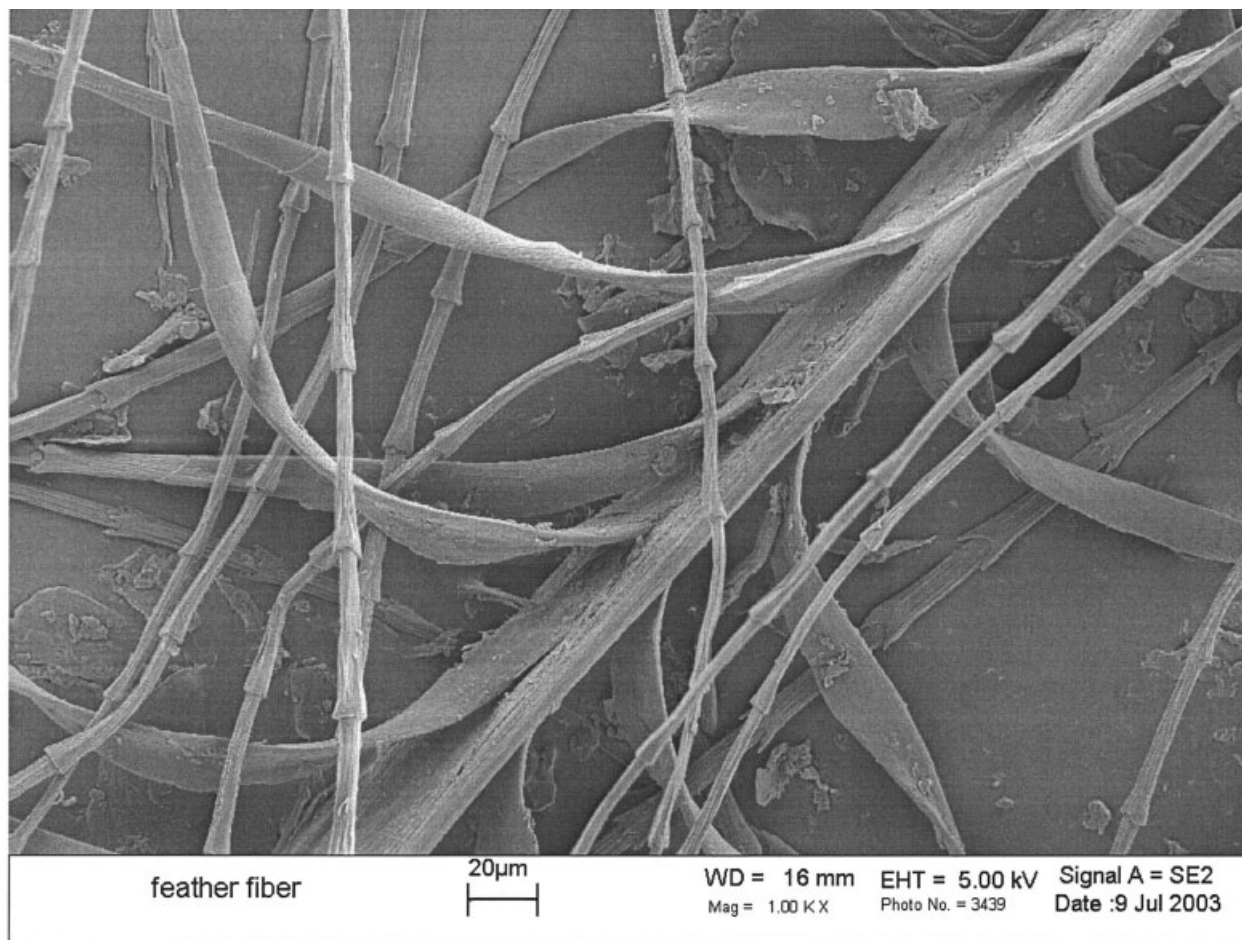


Figure 4 Scanning electron microscope image of feather fiber.

provement. The next part of the experiment was to create composites with 60 wt % reinforcement of only Ff or only Pf. For both the Ff and the Pf trials, the sum of PP and MaPP again was held constant at 40 wt % with MaPP (1.0 wt % MA) substitutions of 0, 4, and 8 wt % (Table I). The purpose of this stage of the experiment was to determine whether the MaPP would bond to the Ff or if the mechanical property improvements seen in the first stage of the experiment were solely due to MaPP/Pf interactions.

### Materials

The feather fibers were supplied by Featherfiber Corp. (Nixa, MO). The supplier reported the fibers to have

lengths of 3–13 mm and diameters of 6–8  $\mu\text{m}$ . The unique fiber architectures are evident in the scanning electron microscope image shown in Figure 4. The reclaimed kraft wood pulp fibers were supplied by CreaFill Fibers Corp. (Chestertown, MD). The Pfs are flat with a thickness of 1–2  $\mu\text{m}$  and a width of about 30  $\mu\text{m}$ ; the fiber lengths vary from about 500 to 5000  $\mu\text{m}$ . The ribbon-like structure of these fibers can be seen in Figure 5. The uncrimped, 15-denier polypropylene fiber was supplied by FiberVisions, Inc. (Covington, GA) with a length of 5 mm and a diameter of 48.3  $\mu\text{m}$ . This PP has a melt temperature of 165°C and a melt flow rate (230/2.16) of approximately 40 g/10 min. Tensile and flexural properties for this PP are listed in Table II. The MaPP (containing 1.0 wt % maleic anhydride) was provided by Crompton Corp. (Middlebury, CT). The MaPP was supplied in the form of beads that were too large to suspend in the wetlay slurry. Therefore, the beads were chipped in a blender to produce chips that would disperse in the wetlay slurry and better distribute the MaPP throughout the wetlay mat.

TABLE II  
Tensile and Flexural Properties of PP

Test	Modulus (GPa)	Strength (MPa)
Tensile	1.20	27.0
Flexural	1.32	48.6

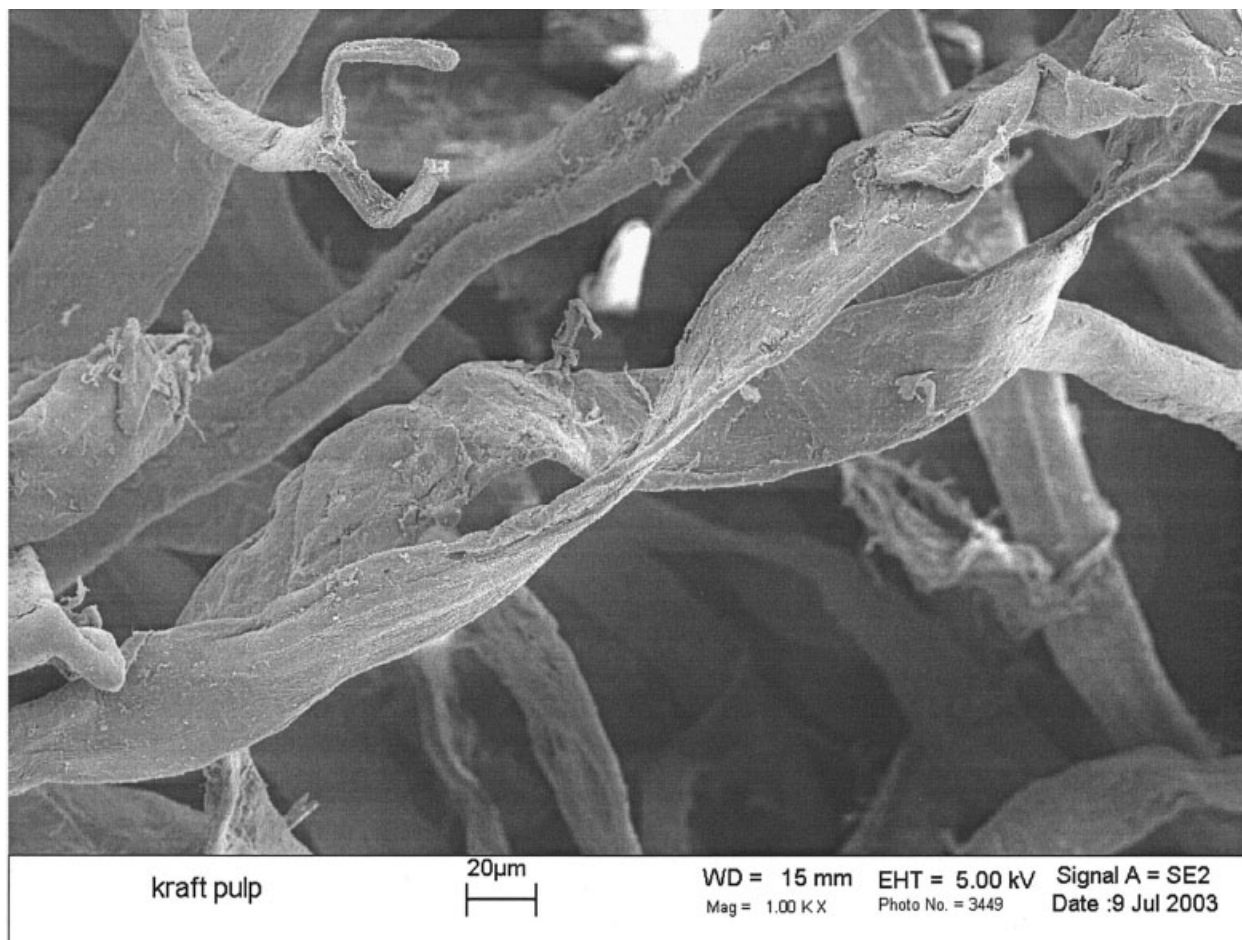


Figure 5 Scanning electron microscope image of kraft pulp fiber.

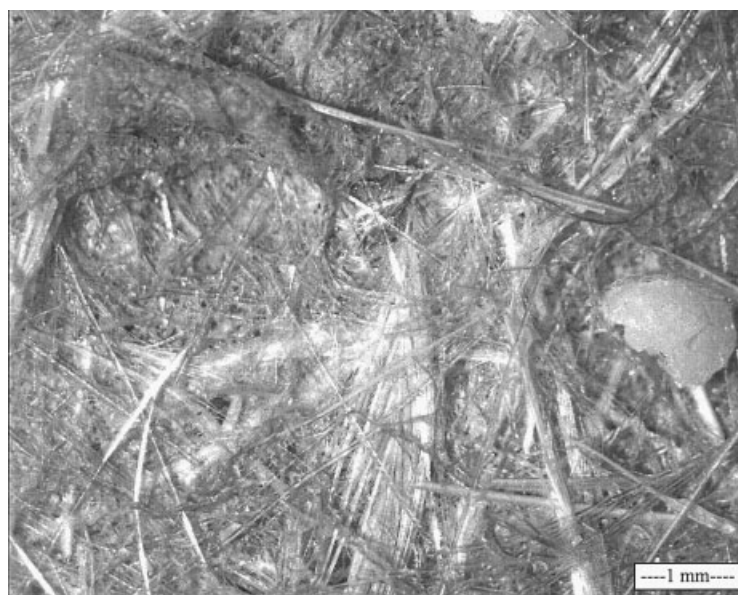


Figure 6 Stereomicroscope image of wetlay mat after dewatering and prior to heating in convection oven.

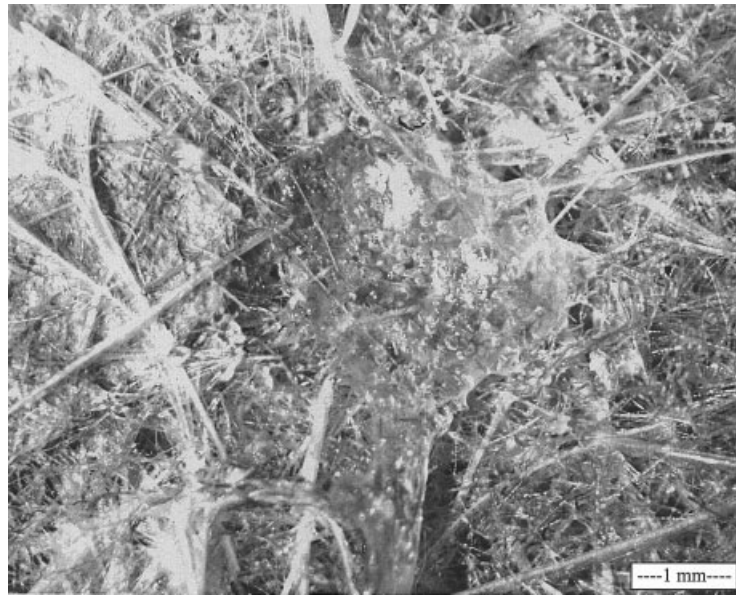


Figure 7 Stereomicroscope image of wetlay mat after heating in convection oven.

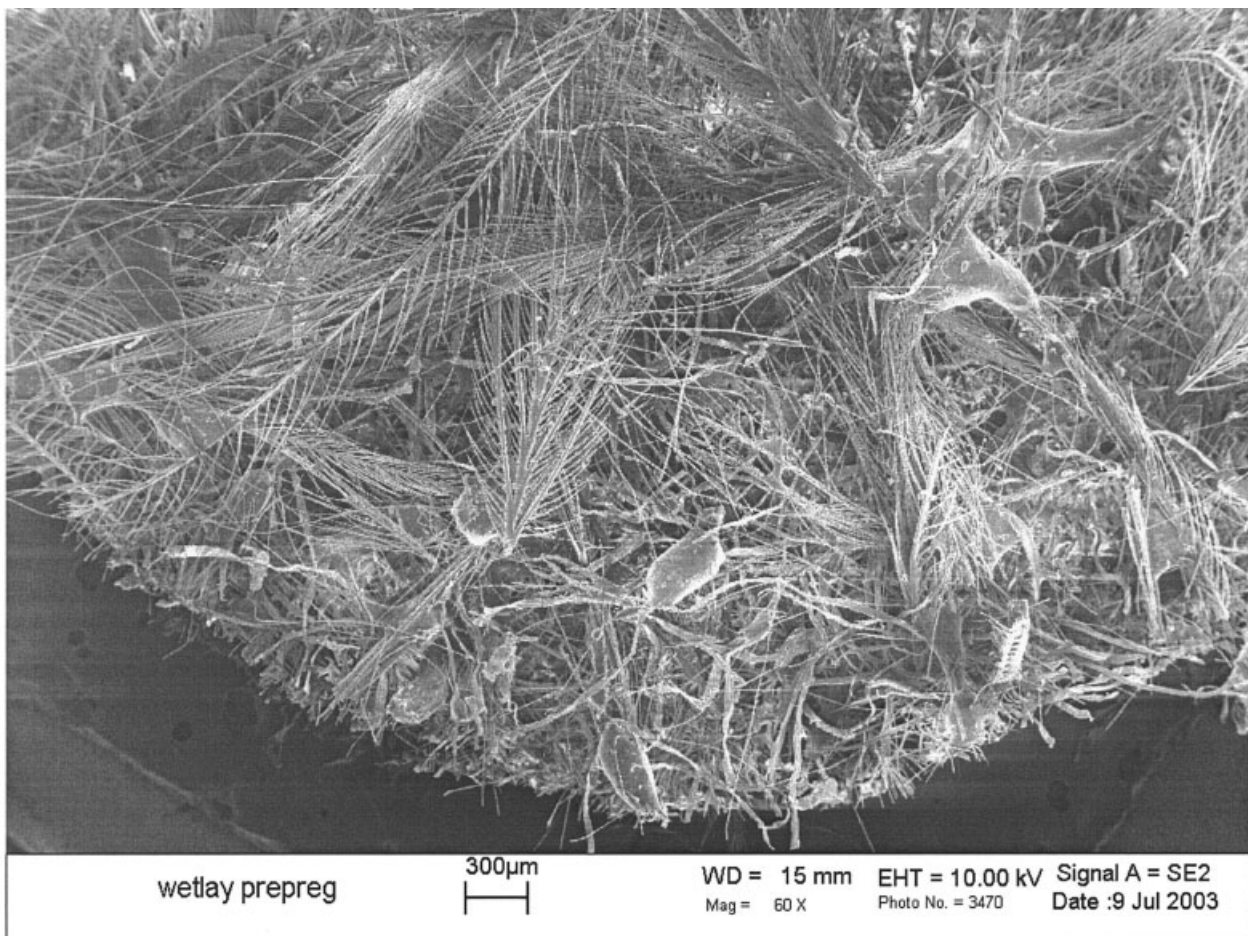


Figure 8 Scanning electron microscope image of wetlay mat after heating in convection oven.

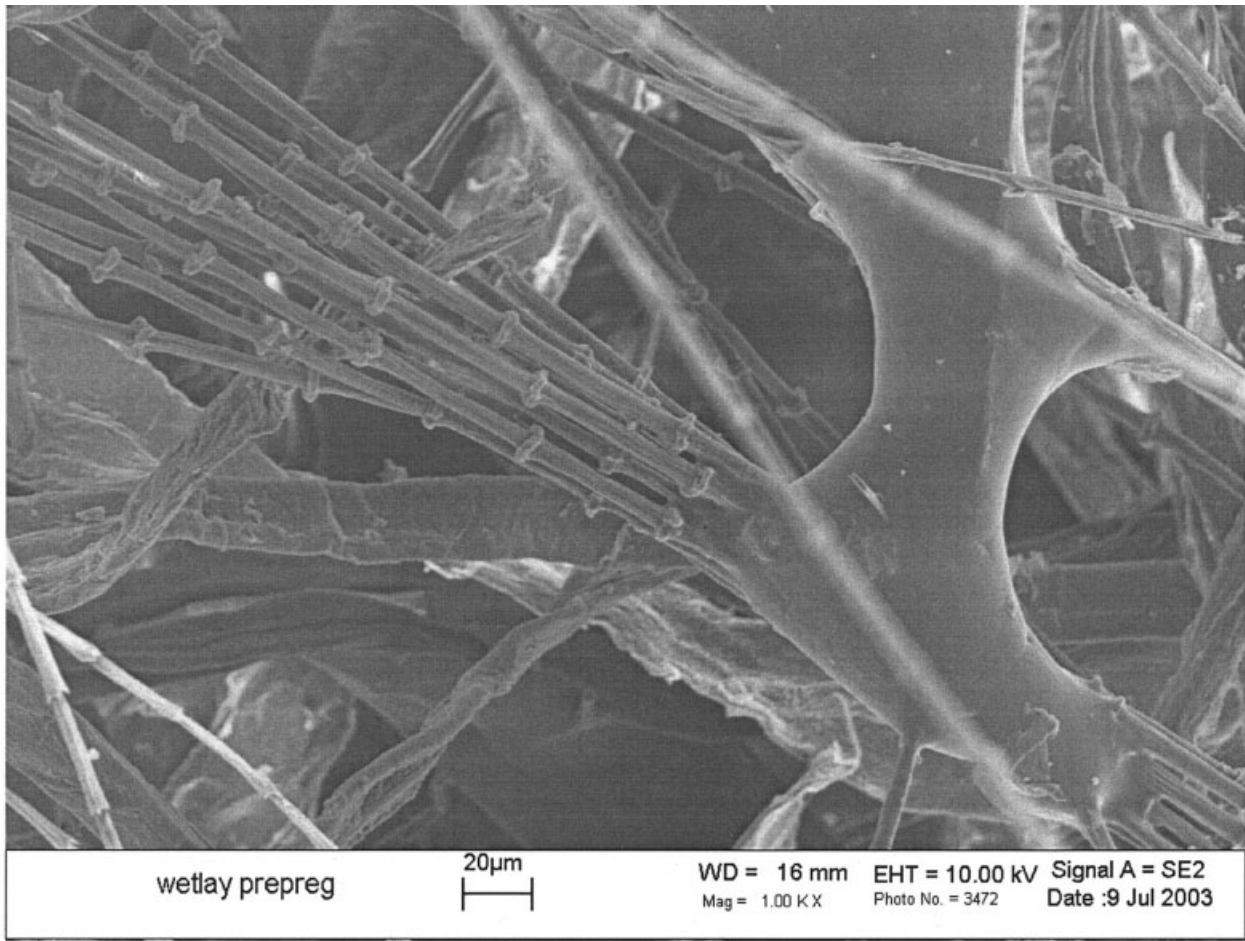


Figure 9 Scanning electron microscope image of wetlay mat after heating in convection oven.

**Production of wetlay prepeg**

The wetlay equipment and process are described in previous publications<sup>9-12</sup>; however, Figure 3 is included to show the formation of the wet, nonwoven

mat from the slurry. The fibers were mixed in a ratio of 500 g of fiber per 400 L of treated water with 250 ppm of Nalco 2388 viscosity modifier. The viscosity modifier thickens the water, thus aiding in maintain-

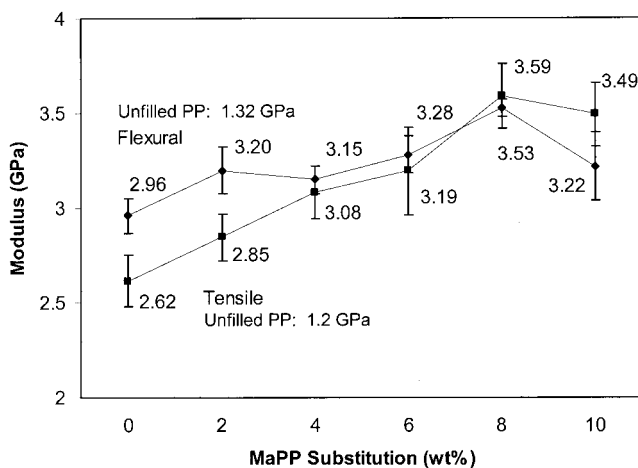


Figure 10 Influence of MaPP substitution for PP on the modulus of Ff/Pf/PP composites.

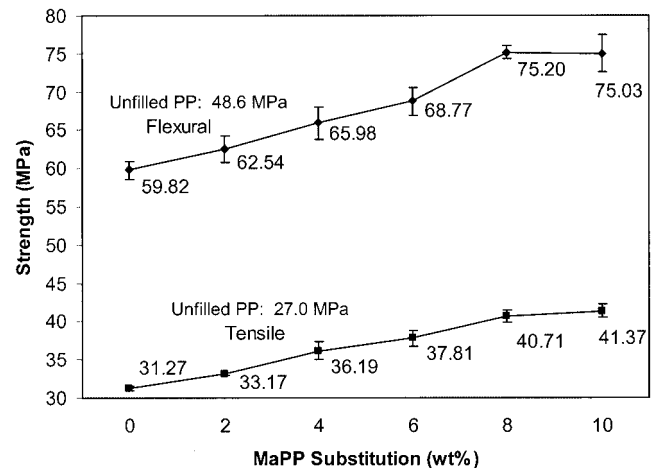


Figure 11 Influence of MaPP substitution for PP on the strength of Ff/Pf/PP composites.

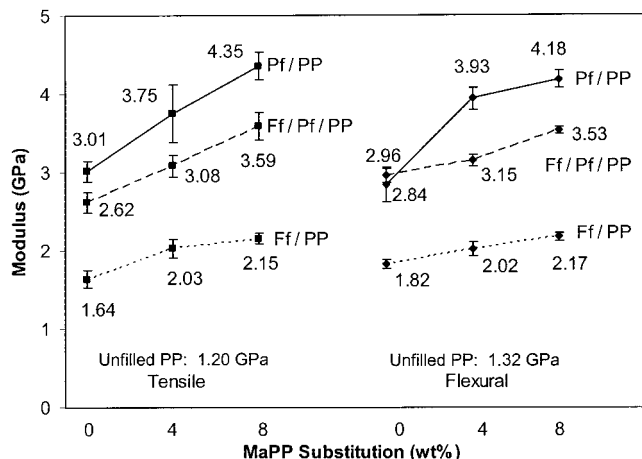


Figure 12 Influence of MaPP substitution on modulus of Ff/PP and Pf/PP composites.

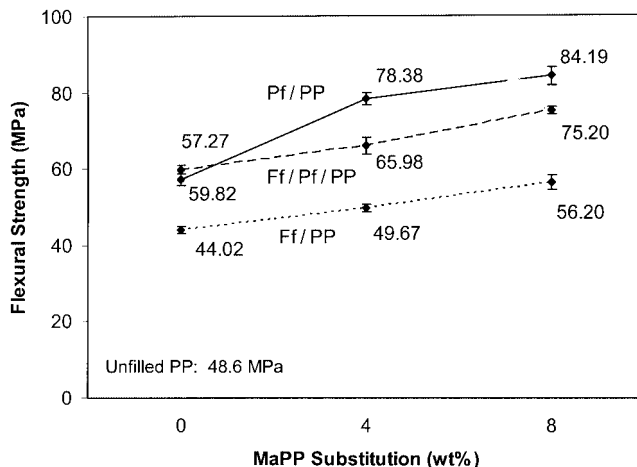


Figure 14 Influence of MaPP substitution on flexural strength of Ff/PP and Pf/PP composites.

ing the suspension of the fibers in the slurry. Following dispersion of the PP fiber, MaPP chips, Ff, and Pf, the slurry was pumped to a stock tank. From the stock tank, the slurry was pumped to the head box of the wetlay line at 37.9 L/min, where it was diluted with treated water entering at 246 L/min. A nonwoven mat was cast on the forming wire, which carried the mat at 1.07 m/min out of the head box. The mat was transferred to the oven wire, which conveyed the mat through a 182°C convection oven at 1.07 m/min. This oven temperature was sufficient to melt the PP and MaPP without degrading the feathers.

An image of the dewatered, nonwoven mat prior to heating in the convection oven taken through a stereomicroscope is shown in Figure 6. The Pf appears as the fuzzy, gray material with white and gray Ff mixed in. The PP fiber appears as translucent rods with a single chip of MaPP visible in the right side of the

image. Figure 7 shows wetlay prepreg after exiting the convection oven. This image was also taken through a stereomicroscope at the same magnification. The large globule of polymer in the center of the image is thought to be MaPP versus the smaller, hazy globules of polymer dispersed around the image that are thought to be PP. The larger blob is thought to be MaPP because the MaPP chips were much larger than individual PP fibers. SEM images of the wetlay prepreg are shown in Figures 8 and 9. Different Ff architectures and material distribution are visible in these images. It is notable that the Ff appear to lie relatively flat in the plane of the prepreg, and the individual branches of the Ff are distinct from one another. There was concern that wetting of the Ff would cause the individual branches to clump together. In general, these images show that the constituents of the prepreg are well-dispersed.

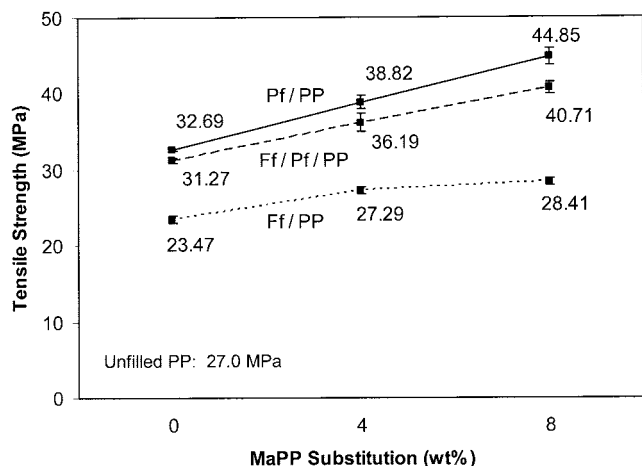


Figure 13 Influence of MaPP substitution on tensile strength of Ff/PP and Pf/PP composites.

Composite panel fabrication

Composite panels were consolidated from 24 to 27 plies of wetlay prepreg; the number of plies was selected to provide the mass of material needed to achieve a consolidated thickness of approximately 3.2 mm. The 152 × 152 mm plies were cut from the center of the 300-mm-wide wetlay prepreg. The plies were

TABLE III  
Percent Increase in Properties with the Substitution of 8 wt % MaPP

Reinforcement	Tensile modulus	Flexural modulus	Tensile strength	Flexural strength
Ff	31.1%	19.2%	21.0%	27.7%
Ff/Pf	37.0%	19.3%	30.2%	25.7%
Pf	44.5%	47.2%	37.2%	47.0%



**TABLE IV**  
**t-Test Comparison of MaPP Substitution Levels**

Property	0-4 wt% MaPP			4-8 wt% MaPP		
	Ff/PP	Pf/PP	Ff/Pf/	Ff/PP	Pf/PP	Ff/Pf/
			PP			PP
Tensile modulus	99.9	99	99	N	98	99
Flexural modulus	99	99.9	99	99	98	99.9
Tensile strength	99.9	99.9	99.9	99	99.9	99.9
Flexural strength	99.9	99.9	99.9	99.9	99	99.9

stacked in the mold with alternating machine and transverse directions to eliminate directionality effects on mechanical properties. A pressure of 4.40 MPa was applied as the 152 × 152 mm mold reached 180°C following a 3°C/min ramp from ambient temperature. The consolidation pressure was held as the mold was then cooled to ambient temperature at ~ 15°C/min.

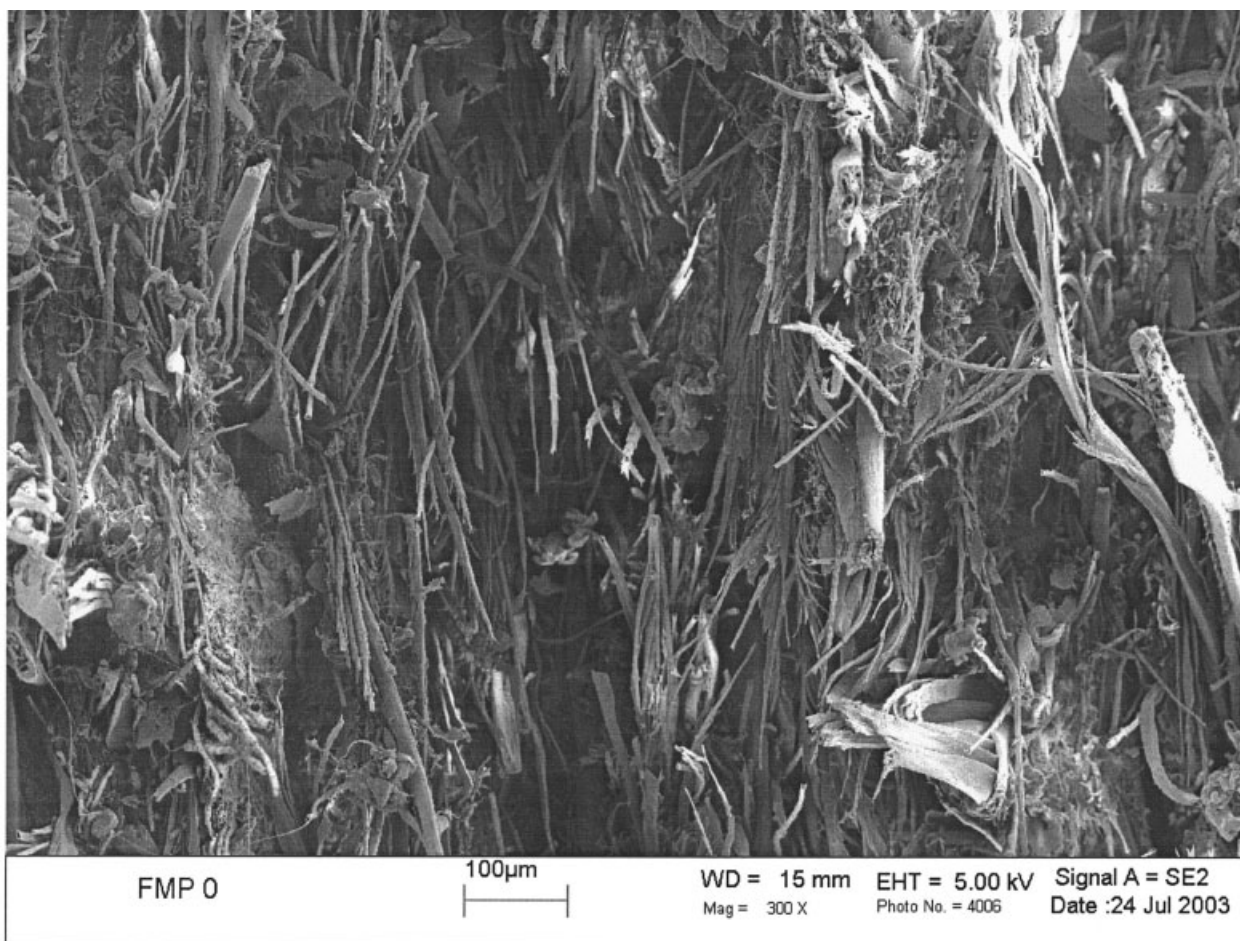
**Mechanical testing**

All of the specimens were conditioned according to the ASTM Standard Practice for Conditioning Plastics

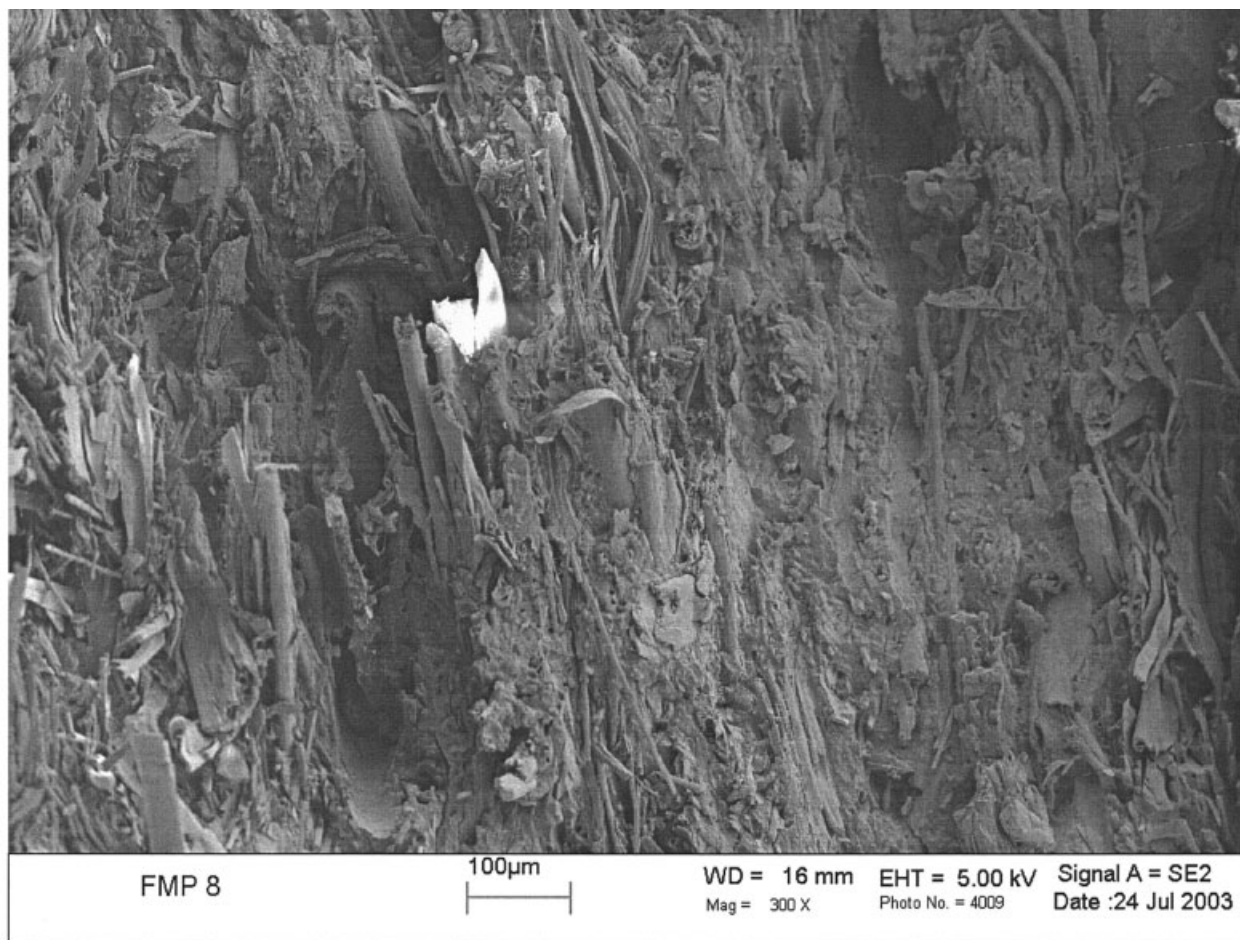
for Testing (D618-00) at 50% relative humidity and 23°C for 40 h after they were cut for testing. All specimens were measured at three prescribed points for thickness and width and the averages of these measurements were used in property calculations. Tensile specimens were ~ 152 mm long, 3.2 mm thick, and 12.7 mm wide. Flexural specimens were ~ 76 mm long, 3.2 mm thick, and 12.7 mm wide.

Tensile tests were conducted in accordance with the ASTM Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials (D3039-00). All six specimens were extended to fracture with a constant crosshead speed of 2 mm/min. Each grip held 25.4 mm of material and the gauge length was 25.4 mm. The modulus was calculated over the linear region of 0.001 strain to 0.003 strain. The strain during testing was measured utilizing a 25.4 mm MTS632.11B-20 extensometer attached at the center of the specimen. The fracture surfaces of tensile specimens were inspected via SEM.

Three-point bend flexural tests were conducted according to the ASTM Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials (D790-00) with



**Figure 15** Scanning electron microscope image of tensile fracture surface of Ff/PP composites with 0 wt % MaPP.



**Figure 16** Scanning electron microscope image of tensile fracture surface of Ff/PP composites with 8 wt % MaPP.

a support span of 50.8 mm. Because previous specimens did not yield during flexural testing at 1.35 mm/min (0.01 strain rate in the outer surface) before a strain of 0.05 was reached, a higher crosshead speed of 13.5 mm/min (0.10 strain rate in the outer surface) was used. The reported modulus was calculated over the linear region of 0.002 to 0.006 strain.

## RESULTS AND DISCUSSION

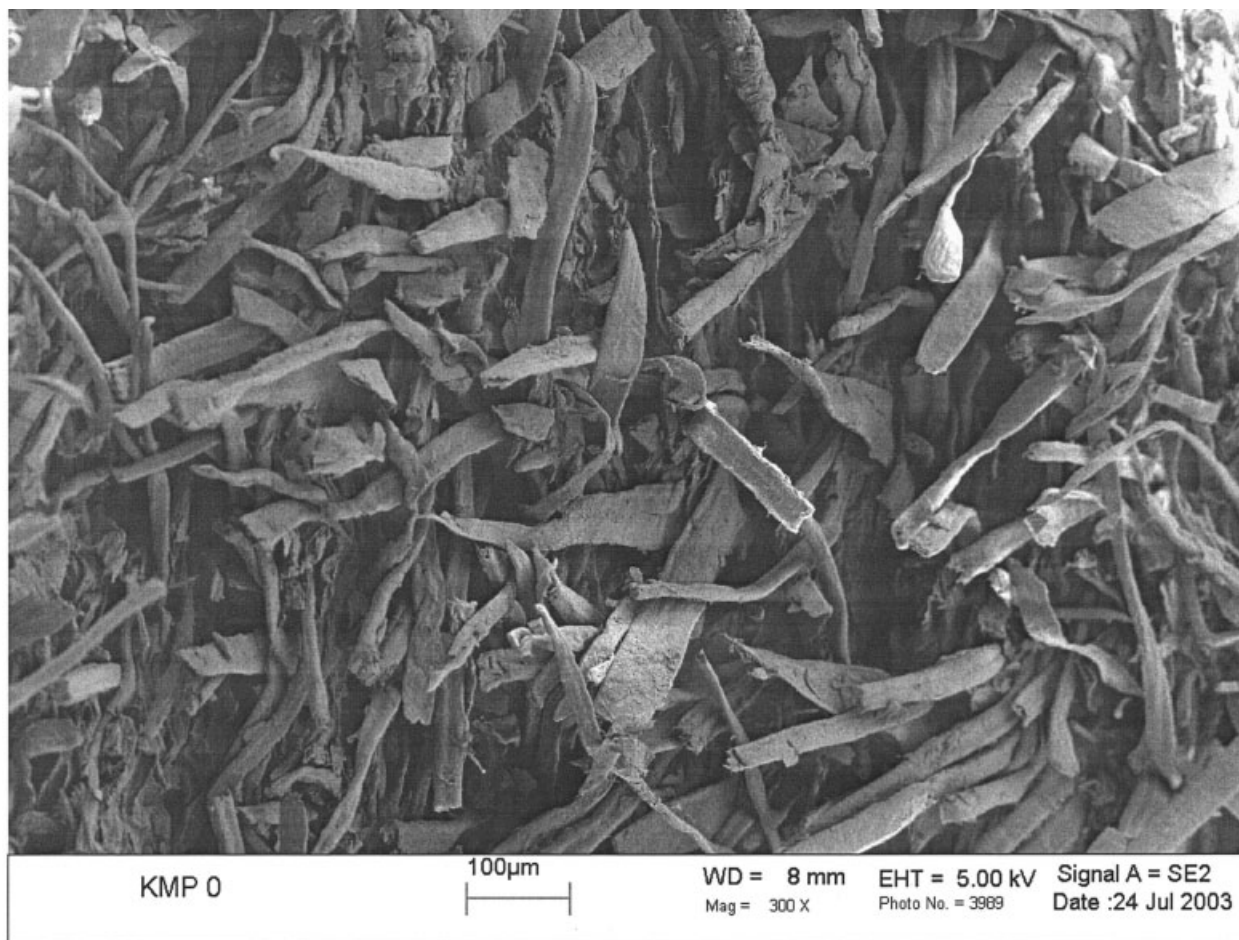
### Addition of MaPP to the Ff/Pf/PP composite

The flexural and tensile moduli improved with the substitution of up to 8 wt % MaPP (Fig. 10). The error bars are for a 95% confidence interval based on the standard deviation of each panel and the number of test specimens. The flexural and tensile strengths also improved with the substitution of up to 8 wt % MaPP (Fig. 11). Although the strength does not decline between 8 and 10 wt % as the modulus does, it does level off between 8 and 10 wt % MaPP. It is clear that substitution of MaPP is improving the strength and moduli of the composites. The values are statistically different between 0 and 4 wt % MaPP and between 4

and 8 wt % MaPP. The next stage of the experiment only investigated substitutions of 4 and 8 wt % MaPP.

### Addition of MaPP to Ff/PP and Pf/PP composites

The second portion of this experiment was intended to isolate the effect of MaPP on Ff and Pf. The composites still contained 60 wt % fiber, but only Ff or Pf, not both. The results plotted in Figures 12-14 demonstrate that the substitution of MaPP increased the modulus and strength of the composites for both types of fibers. The percent increase, above that of 0 wt % MaPP, in each property for the 8 wt % MaPP substitution is listed in Table III. The MaPP substitution had the most influence on the properties of the Pf/PP composites. Such increases in Pf/PP strengths and moduli were expected because of previously cited literature results for cellulose-reinforced PP matrix composites. However, the increases seen in Ff/PP strengths and moduli are the first reported, that the authors are aware of, for a Ff-reinforced PP matrix composite in which the matrix is mostly PP and not solely MaPP. The substitution of 8 wt % MaPP generated strength and modulus increases of about 20 to 30%.



**Figure 17** Scanning electron microscope image of tensile fracture surface of Pf/PP composites with 0 wt % MaPP.

### Statistical analysis

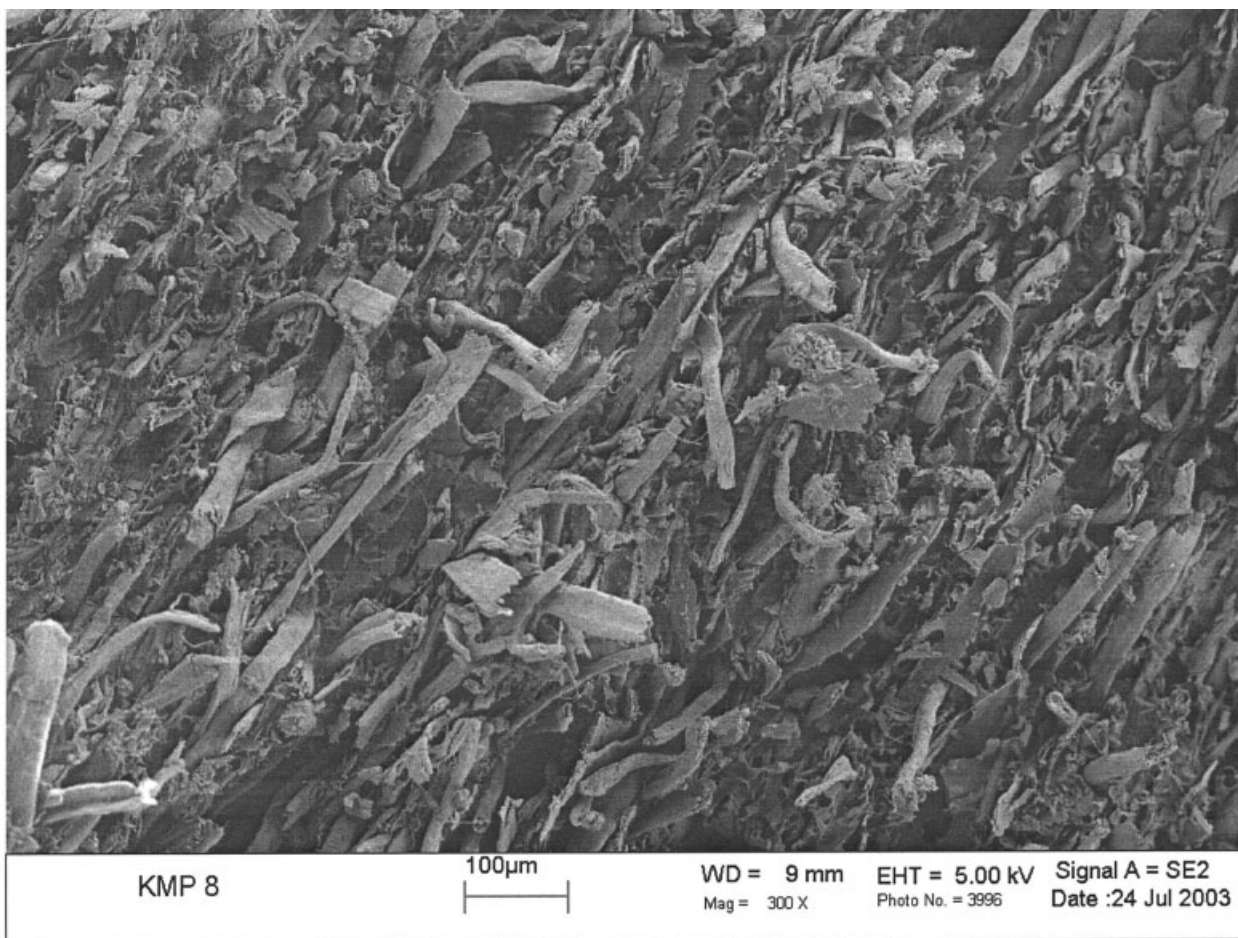
Statistical analysis was performed for both experiments to determine whether or not the mean values were actually different between 0 and 4 wt % MaPP and between 4 and 8 wt % MaPP for each of the compositions (Ff/Pf/PP, Ff/PP, and Pf/PP). Only one panel was fabricated for each trial, so it was impossible to determine the variance in mechanical properties between panels made of the same materials. The random variance that occurs in the wetlay process and the panel manufacture are therefore not taken into account. The variance observed is between specimens cut from the same panel and can be due to differences in the material strength throughout a panel, random testing differences, random calculation or measurement discrepancies, and random distribution flaws.

Analysis of variance tests for the strengths and moduli of the Ff/PP and Pf/PP trials all have  $P$  values at or below  $10^{-5}$ , indicating that the addition of MaPP most certainly has an effect on the strength and modulus. Table IV shows the probability (99.9, 99, 98, 95) that the mean values are not different by chance alone between 0 and 4 wt % MaPP and between 4 and 8 wt

% MaPP for each of the compositions (Ff/PP, Pf/PP, and Ff/Pf/PP) according to a two-tailed, two sample  $t$ -test assuming equal variances. The  $N$  indicates that the two means are not statistically significantly different at an  $\alpha$  level of 5%.

### Fracture surface analysis

Qualitative analysis of tensile fracture surfaces by using SEM revealed a significant decrease in the amount of fiber pullout with the addition of 8 wt % MaPP. Figures 15 and 16 offer a comparison of the tensile fracture surfaces of Ff/PP composites containing 0 and 8 wt % MaPP. There are many more fibers apparent on the fracture surface of the 0 wt % MaPP composite than on the fracture surface of the 8 wt % MaPP composite. In addition, the fibers visible on the MaPP-containing composite fracture surface are much shorter. It appears that the substitution of MaPP for PP has improved the bonding between the Ff and PP, thus resulting in less fiber pullout and more fiber breakage. These observations complement the increases seen in strength and modulus. The tensile



**Figure 18** Scanning electron microscope image of tensile fracture surface of Pf/PP composites with 8 wt % MaPP.

fracture surfaces of the Pf/PP composites containing 0 and 8 wt % MaPP are similar to the Ff/PP fracture surfaces in that substitution of 8 wt % MaPP appeared to reduce the number and length of fibers present on the fracture surface (Figs. 17 and 18).

### CONCLUSION

There were several questions regarding the addition of MaPP to Ff/Pf/PP composites addressed by this project. First, it was found to be possible to add MaPP to the Ff/Pf/PP wetlay prepreg in chip form. The MaPP added to the wetlay prepreg increased the strength and modulus of the composite panels compression molded from this prepreg, demonstrating that the fiber, PP, and MaPP do not need to be melt blended to achieve improvements in strength in modulus. Generation of the wetlay slurry provides a method to uniformly disperse the composite constituents without melt blending. Avoiding the need for melt blending for uniform dispersion and formation of the bond between MaPP and the reinforcement can be important in cases where processing temperatures

threaten the stability of the reinforcement. For example, extrusion of filled PP usually takes place at temperatures of 200°C or greater. Temperatures this high will degrade Ff. In contrast, stamping or compression molding of Ff/PP prepreg can be done at temperatures less than 190°C, thus not threatening the thermal stability of the Ff. Finally, it was demonstrated that addition of small quantities (less than 1% of the total composite mass) of maleic anhydride via MaPP to Ff-reinforced, PP-matrix composites can improve the strength and modulus of the composites by 20–30%, suggesting MaPP reacts with certain amino acid groups within keratin protein.

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