

Nanoscratch Testing to Assess the Fiber Adhesion of Short-Carbon-Fiber Composites

Julia A. King,¹ Ibrahim Miskioglu,² Debra D. Wright-Charlesworth,³ Charles D. Van Karsen²

¹Department of Chemical Engineering, Room 203 Chemical Science and Engineering Building, 1400 Townsend Drive, Michigan Technological University, Houghton, Michigan 49931-1295

²Department of Mechanical Engineering–Engineering Mechanics, Mechanical Engineering and Engineering Mechanics Building, 1400 Townsend Drive, Michigan Technological University, Houghton, Michigan 49931-1295

³Department of Biomedical Engineering, Michigan Technological University, Houghton, Michigan 49931-1295

Received 6 August 2005; accepted 27 December 2005

DOI 10.1002/app.23959

Published online in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: In a composite material, the degree of adhesion between the fiber and the matrix plays an important role in the overall performance of the material. Because the load between the fiber and the matrix is realized throughout the interphase region material, a lot of effort has gone into characterizing the strength of the interphase. In this study, nanoscratch tests on the composite samples were used to provide a relative measure of adhesion in different composite materials. Carbon-filled nylon 6,6 and polycarbonate resins were evaluated with this method. The carbon fillers we used were polyacrylonitrile-based carbon fibers sized and

surface-treated for the respective matrix and pitch-based carbon fibers without any sizing or surface treatment. Tensile and X-ray photoelectron spectroscopy data for the composites we considered are also presented to compare to the nanoscratch results. It is shown that nanoscratch testing on the composites, with the proposed data analysis, can be an effective tool for determining the relative degree of adhesion between different composites. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 103: 328–335, 2007

Key words: fibers; nylon; polycarbonates; strength

INTRODUCTION

During the manufacture of a composite material, an interphase between the reinforcement and the matrix forms as the result of bonding and chemical reactions. This interphase can have significantly different material properties than those of the matrix and the reinforcement. Because the interphase facilitates the load transfer between the matrix and the reinforcement, it plays an important role in the overall performance of the composite.^{1–4} An ongoing effort in this area is the tailoring of the interphase by the surface treatment of the reinforcement to improve the interphase properties. However, the challenge still remains to find efficient analytical and experimental tools for characterizing the interphase at the submicrometer level.

The properties of the interphase are quantified in terms of *interfacial strength*, or degree of adhesion between the fiber and the matrix. Among the most popular experimental techniques used to investigate the mechanical properties of the interphase are fiber pull-out, push-out, and push-in techniques⁵ and, more recently, microindentation and nanoindentation

techniques.^{6–9} It has been speculated that the simplifying assumptions, such as uniform stress field along the fiber and perfect bonding between the fiber and the matrix might not be realistic.¹⁰

In this article, an approach is presented that takes advantage of new instrumentation to run nanoscratch tests. These tests were performed to determine whether the data we obtained could provide a measure of the filler–matrix adhesion. The material systems we considered were carbon-filled nylon 6,6 and polycarbonate resins. Carbon fillers were polyacrylonitrile-based carbon fibers sized and surface-treated for the respective matrix and pitch-based carbon fibers without any sizing or surface treatment. We prepared the specimens mixing the carbon fiber and the matrix, pelletizing the mixture, and injection molding it into test specimens.

EXPERIMENTAL

Materials

Two matrix materials were used in this project. The first matrix was DuPont Zytel 101 NC010 (Wilmington, DE), an unmodified semicrystalline nylon 6,6 polymer. The second matrix used was Lexan HF 1110-111N, which is an amorphous engineering thermoplastic produced by GE Plastics (Pittsfield, MA). The properties of these polymers were discussed in detail elsewhere.^{11–13} Table I shows selected mechanical properties of these polymers.

Correspondence to: J. A. King (jaking@mtu.edu).

Contract grant sponsor: National Science Foundation; contract grant numbers: DMI-9973278 and CMS-MRI-9977462.

TABLE I
Properties of Zytel 101 NC010 and Lexan HF 1110-111N¹¹⁻¹³

Property	Zytel 101 NC010	Lexan HF 1110-111N
Tensile modulus (GPa)	3.1	2.4
Flexural modulus (GPa)	2.8	2.3
Tensile strength (MPa)	83	62
Elongation at fracture (%)	60	120

Four different Akzo Nobel Fortafil polyacrylonitrile-based carbon fibers (Rockwood, TN) were used in this project. All these fibers were from the same parent fiber, which was surface treated to add heteroatoms to the carbon fiber surface. Two 3.2-mm chopped fibers, Fortafil 243 and Fortafil 201, were used. Fortafil 243 contains a proprietary polymer (sizing, also called coating) that promotes adhesion to nylon. Fortafil 201 contains a proprietary polymer (sizing) that promotes adhesion to polycarbonate. Two 200- μm milled fibers, Fortafil 482 and Fortafil 402, were used. Fortafil 482 contains a proprietary polymer (sizing) that promotes adhesion to nylon. Fortafil 402 contains a proprietary polymer (sizing) that promotes adhesion to polycarbonate.¹⁴

One pitch-based 200- μm milled carbon fiber, BP/Amoco/Cytec ThermalGraph DKD X (Alpharetta, GA), was used. This fiber is highly graphitized, and was not surface-treated or sized to promote fiber adhesion to a matrix. The properties of these five fibers were discussed in detail elsewhere.^{11,14-16} Table II shows selected mechanical properties of these fibers.

In this study, 20 wt % of the carbon fibers discussed previously were added to either nylon or polycarbonate. Tensile and nanoscratch properties were measured on these composites. Also, X-ray photoelectron spectroscopy (XPS) was used to determine the surface composition of the carbon fibers.

Test specimen fabrication

For this entire project, the fibers were used as received. Zytel 101 NC010 and Lexan HF 1110-111N were dried in an indirectly heated dehumidifying drying oven and then stored in moisture barrier bags.

The extruder used was an American Leistritz Extruder Corp. (Somerville, NJ) model ZSE 27 instrument. This extruder had a 27-mm corotating intermeshing twin screw with 10 zones and a length/diameter ratio of 40. The screw design was chosen to obtain the maximum possible tensile strength and was described in detail elsewhere.¹⁷ Hence, we desired a minimum amount of filler degradation, while the fillers were still dispersed well in the polymer. The same screw design was used for this entire project. The polymer pellets were introduced in Zone 1. A side stuffer was located at zone 7 and was used to introduce the carbon fiber into the polymer melt. Two Schenck AccuRate gravimetric feeders (Whitewater, WI) were used to accurately control the amount of each material added to the extruder.

After passing through the extruder, the polymer strands (3 mm in diameter) entered a water bath and then a pelletizer that produced pellets nominally 3 mm long. After compounding, the pelletized composite resin was dried again and then stored in moisture barrier bags before injection molding.

A Niigata injection-molding machine (model NE85-UA₄) (Tokyo, Japan) was used to produce test specimens. This machine had a 40 mm diameter single screw with a length/diameter ratio of 18. The lengths of the feed, compression, and metering sections of the single screw were 396, 180, and 144 mm, respectively.

A four-cavity mold was used to produce 3.2 mm thick ASTM Type I tensile bars (end gated). The tensile properties of all formulations were determined. The nylon 6,6 based samples were tested dry as molded. The polycarbonate-based samples were conditioned at 50% relative humidity for 24 h at 23°C before testing.

Fiber length, aspect ratio (AR), and orientation test method

To determine the length of the carbon fiber in the tensile test specimens, solvent digestion was used. A 0.2-g sample cut from the center gauge section of a tensile test specimen was dissolved at 23°C with formic acid to remove the nylon 6,6 and methylene chloride to remove the polycarbonate. The fillers were then dispersed onto a glass slide and viewed with an Olympus

TABLE II
Properties of Fortafil and ThermalGraph DKD X Carbon Fibers¹⁴⁻¹⁶

Fiber	As-received length (μm)	Diameter (μm)	Tensile modulus (GPa)	Tensile strength (MPa)
Fortafil 201	3200	7.3	227	3800
Fortafil 243	3200	7.3	227	3800
Fortafil 402	200	7.3	227	3800
Fortafil 482	200	7.3	227	3800
ThermalGraph DKD X	200	10	827	>1900

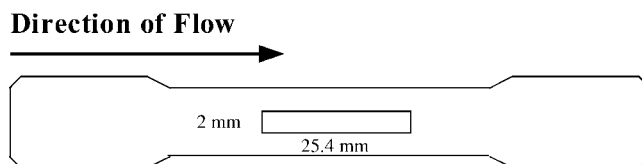


Figure 1 Portion of tensile bar from which nanoscratch specimens were cut.

SZH10 optical microscope (Melville, NY) with an Optronics Engineering LX-750 video camera (Goleta, CA). The images (at $60\times$ magnification) were collected with Scion Image (version 1.62) software. The images were then processed with Adobe Photoshop 5.0 (San Jose, CA), and the Image Processing Tool Kit (version 3.0) (Ashville, NC). The length and AR (length/diameter) of each fiber were measured. For each formulation, at least 500 fibers were measured.

To determine the orientation of the carbon fibers, a polished composite sample was viewed with an optical microscope. One $25\text{ mm} \times 2\text{ mm}$ rectangle was cut from the center of a tensile specimen, as shown in Figure 1. This sample was cast in a two-part epoxy plug, as shown in Figure 2. Then, the sample was polished and viewed with an Olympus BX60 reflected-light microscope at a magnification of $200\times$. The images were then collected and processed as described in the previous paragraph. For each formulation, the orientation was determined by the viewing of at least 700 fibers.

Tensile test method

The tensile properties (at ambient conditions, 16.5 cm long, 3.2 mm thick, ASTM Type I sample geometry) from all formulations were determined with ASTM D 638 at a crosshead rate of 5 mm/min for reinforced plastics.¹⁸ An Instru-Met Sintech screw-driven me-

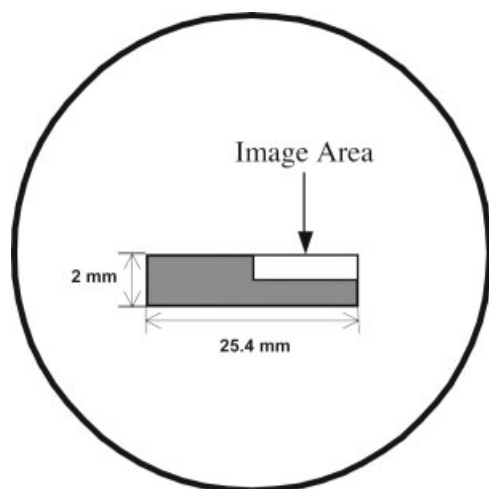


Figure 2 Sample arrangement for filler orientation analysis.

chanical testing machine (Union, NJ) was used. For each formulation, at least five samples were tested.

Nanoscratch testing

Nanoscratch tests were performed on samples cut from the center of tensile specimens, as shown in Figure 1. Then, the 3.2 mm thick \times 2 mm wide face was mounted in epoxy and tested with a MTS Nano Indenter XP (Oak Ridge, TN). The typical test was run under a constant load of 40 mN. The scratch length was $500\text{ }\mu\text{m}$, the scratch speed was $10\text{ }\mu\text{m/s}$, and data were sampled at 5 Hz. For each test sample, five scratches were made, and two test samples were used for each formulation. A Berkovich indenter (Oak Ridge, TN) was used for the tests with scratches made in the edge-forward direction, as shown in Figure 3. Data collected included force on sample, penetration of the indenter relative to the surface of the sample, force along the scratch direction (friction force), and force normal to the scratch direction (lateral force). All the data were recorded with respect to distance along the scratch. The friction and lateral forces are also depicted in Figure 3.

A scratch test is performed in three stages, the original profile, the scratch segment, and the residual profile. The original profile is obtained under a very small load ($20\text{ }\mu\text{N}$) and is used to determine the original morphology of the surface along the scratch path. This information is then used to correct the depth measurements during the scratch segment for roughness and initial slope of the sample. Same corrections are applied in the determination of the residual profile. In this study, the information on the residual profile was not used in the analysis. The friction and penetration during the scratch

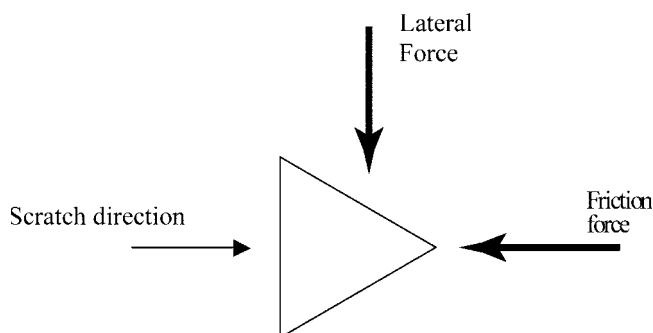


Figure 3 Scratch direction used in the tests with the Berkovich indenter.

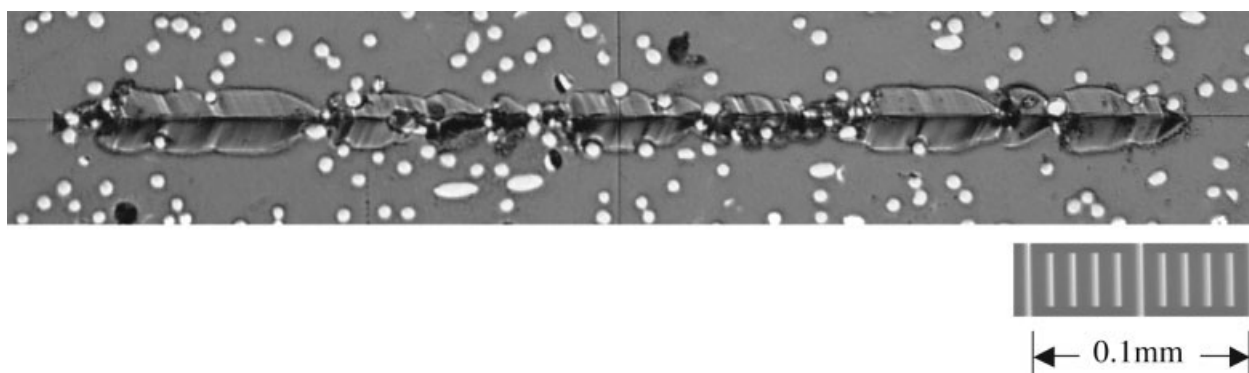


Figure 4 Micrograph showing the relative size of the scratch grooves with fiber diameters under a constant load of 40 mN for sample AP20

segment were analyzed with the crest factor to discern differences between fibers and matrices.

The friction force during scratch and the penetration during scratch were characterized by the crest factor,¹⁹ a parameter used as a measure of spikiness in the data. This measure is mainly used in digital signal analysis of waveforms and is defined as the peak amplitude divided by the root mean square:

$$\text{Crest factor} = \frac{|y_{\max} - y_{\min}|}{\sqrt{\frac{1}{N} \sum_{j=0}^{N-1} y_j^2}} \quad (1)$$

where y_{\max} is the friction force or maximum penetration along the scratch length, y_{\min} is the minimum friction force or minimum penetration along the scratch length, y is either the friction force (mN) or the penetration (nm) along the scratch length and N is the number of data points considered for a given scratch. During a typical scratch test, data is collected over 500 μm length of scratch distance. To avoid any end effects at the beginning and end of the scratch length, data over the first and last 10 μm of the scratch were omitted in the calculations of the crest factor.

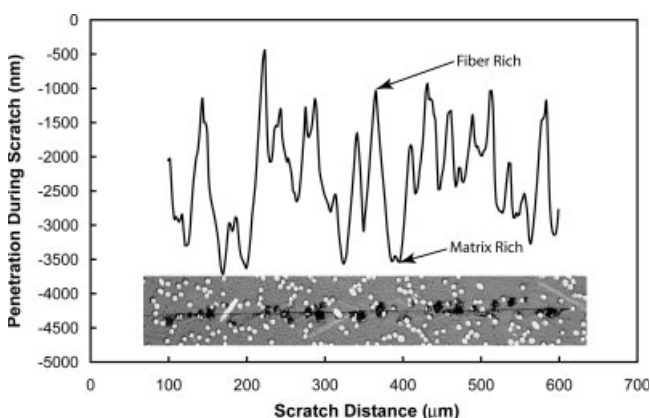


Figure 5 Displacement normal to the surface under a constant force of 40 mN for sample AN20.

Scratch tests performed on a heterogeneous material under a constant normal load give the local compliance of the material, so it is possible to detect the fiber-rich and matrix-rich areas along the scratch path. A shallow scratch depth indicates a high-stiffness (fiber-rich) material. A larger scratch depth indicates a lower stiffness (matrix-rich) material. Because the width of the groove generated by the scratch tip (ca. 30 μm) is large compared to the diameter (7.3 or 10 μm) of the carbon fibers (see Fig. 4), it is not possible to record the load-displacement response as the tip travels from the matrix onto a single fiber. Hence, the load-displacement response obtained from a scratch test is an averaged response of the local material that is in contact with the indenter tip, which may be fiber rich or matrix rich. Figure 5 shows the indenter displacement as a function of the scratch distance for the sample AN20, which consisted of 20 wt % Fortafil 243 in nylon 6,6. Figure 6 depicts the similar load-displacement response for AP20, which consisted of 20 wt % Fortafil 201 in polycarbonate. In both figures, it is evident that the presence of the fibers affected the penetration of the indenter. The transition region between the matrix-rich and fiber-

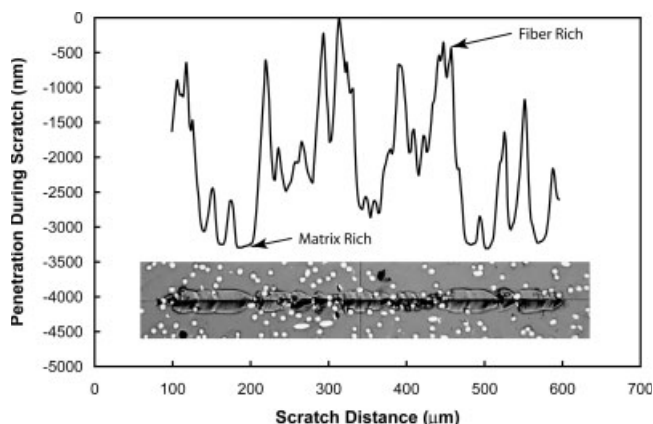


Figure 6 Displacement normal to the surface under a constant force of 40 mN for sample AP20.

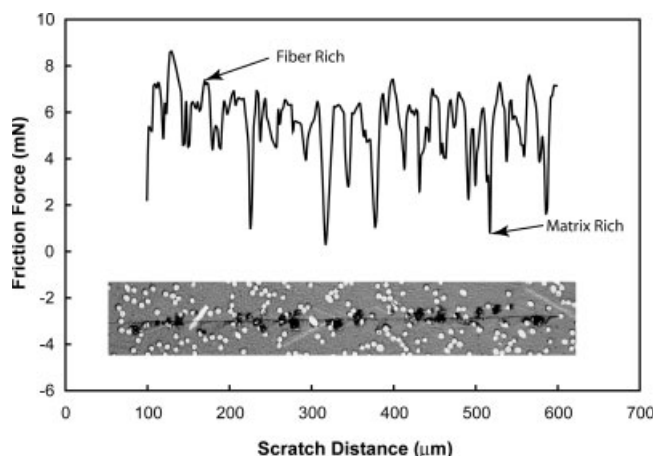


Figure 7 Friction force along the scratch direction for sample AN20.

rich areas was influenced by the degree of adhesion between the fiber and the matrix. In all tests, a 40-mN normal load was applied when the scratch distance reached 100 μm , and the sample was unloaded when the scratch distance was 600 μm .

The friction force measured during a scratch test for samples AN20 and AP20 are shown in Figure 7 and 8, respectively. A similar observation was made from these plots of friction force versus scratch distance,²⁰ that is, friction force increased as the indenter tip approached the fiber. The change in the friction force along the scratch is a measure of the resistance to the motion of the scratch tip through the material, and as fiber-rich areas are approached, this force increases indicating higher strength and stiffness in the material in the vicinity of the indenter tip. By comparing the scratch test results, one can assess the level of adhesion between the matrix and the fiber for a given matrix. The data presented in Figures 5 through 8 are typical of all the scratch tests performed on the composites.

XPS

XPS was used to determine the surface composition of the various carbon fillers. Because each element had a unique set of binding energies, XPS was used to determine the elements present in the top 50–100 angstroms of the sample surface. A PerkinElmer PHI 1600 XPS system (Wellesley, MA) was used in an ultrahigh vacuum chamber. The carbon fibers were mounted on the sample holder with double-sided copper tape.²¹

Statistical analysis

The data were analyzed with a two-way analysis of variance (ANOVA) with the Statistica software package (StatSoft, Inc., version 5.5, Tulsa OK). The independent variables were the matrix type (Lexan or Zytel) and the type of filler (Fortafil 2xx, Fortafil 4xx,

or ThermalGraph DKD X). As stated previously, the Fortafil series of fibers have coatings (sizings) that are different for each of the matrix materials, but the parent fibers are the same. Fortafil 243 and 201 (3.2 mm long) were considered as one fiber type in the two-way ANOVA. Fortafil 482 and 402 (200 μm long) were considered as another fiber type in the ANOVA. Comparisons were not considered between matrices due to the different coating on the fibers. The dependent variables were the mechanical properties of the materials (ultimate tensile strength, tensile modulus, penetration, and friction). The effect of the filler on the mechanical properties of each matrix was determined with a *post hoc* Newman–Keuls test.²² A *p* of less than or equal to 0.05 was considered statistically significant (95% confidence level).

RESULTS AND DISCUSSION

Fiber length, AR, and orientation results

Table III shows the mean fiber and AR results for the composites used in this project.^{16,17,23} As expected, the fiber length in the composite sample was the highest for those samples containing Fortafil 243 and 201 (the as-received fiber was initially 3.2 mm long).

For all the composites in this study, the fibers were primarily orientated in the longitudinal tensile test direction, which was also the direction of the polymer flow into the end-gated tensile test specimen. Photomicrographs depicting this orientation were shown elsewhere.¹⁶ This fiber orientation is also evident in the nanoscratch photomicrographs in Figures 4–8, which display the typical end view of a carbon fiber.

Tensile results

Table III shows the tensile results (mean, standard deviation, and number of samples tested) for each formula-

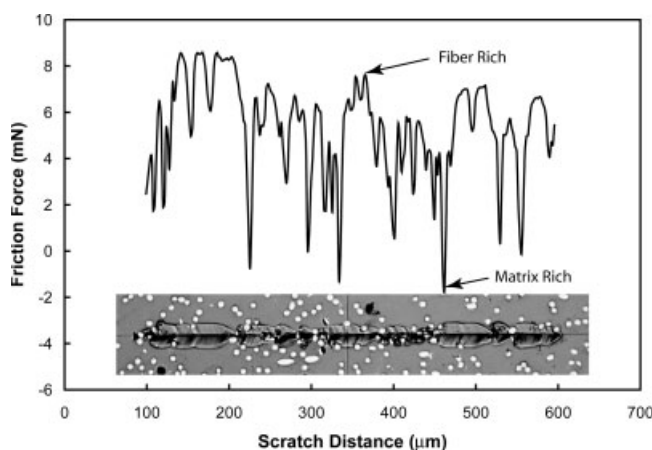


Figure 8 Friction force along the scratch direction for sample AP20.

TABLE III
Properties of Composites Containing 20 wt % Carbon Fiber^{16,17,23–25}

Sample	Matrix	Fiber	Tensile modulus (GPa)	Ultimate tensile strength (MPa)	Mean fiber length (μm)	Mean fiber AR
AN20	Zytel	Fortafil 243	14.68 \pm 0.54 <i>n</i> = 5	194.48 \pm 3.93 <i>n</i> = 5	117	16.0
BN20	Zytel	Fortafil 482	10.02 \pm 0.27 <i>n</i> = 6	126.50 \pm 0.62 <i>n</i> = 6	80	11.0
NCN20	Zytel	ThermalGraph	11.96 \pm 0.79 <i>n</i> = 6	99.92 \pm 0.75 <i>n</i> = 6	95	9.5
AP20	Lexan	Fortafil 201	11.24 \pm 0.70 <i>n</i> = 8	123.75 \pm 1.03 <i>n</i> = 8	117	16.0
BP20	Lexan	Fortafil 402	7.45 \pm 0.41 <i>n</i> = 5	86.11 \pm 0.48 <i>n</i> = 5	75	10.3
NCP20	Lexan	ThermalGraph	9.62 \pm 0.45 <i>n</i> = 5	67.49 \pm 1.24 <i>n</i> = 5	84	8.4

n, number of samples tested.

tion.^{24,25} For the nylon-based (Zytel) composites, the ultimate tensile strength was statistically significantly different for the three different fibers. The composites containing the 3.2-mm chopped Fortafil 243 had the highest tensile ultimate strength (194.5 MPa), followed by the 200- μm milled Fortafil 482 (126.5 MPa). The composites containing ThermalGraph DKD X (the 200- μm milled pitch-based carbon fiber that was not surface-treated or sized to promote adhesion to a matrix) had the lowest ultimate tensile strength (99.9 MPa).

For the polycarbonate-based (Lexan) composites, the ultimate tensile strength was significantly different for the three different fibers. The composites containing the 3.2-mm chopped Fortafil 201 had the highest ultimate tensile strength (123.8 MPa), followed by the 200- μm milled Fortafil 402 (86.1 MPa). The composites containing ThermalGraph DKD X (the 200- μm milled pitch-based carbon fiber that was not surface treated or sized to promote adhesion to a matrix) had the lowest ultimate tensile strength (67.5 MPa).

These composite tensile strength results follow the same trend as the fiber AR, with the composites with a higher AR yielding higher tensile strengths. In addition, fiber–matrix adhesion could affect composite tensile strength. As stated previously, often carbon fibers are surface-treated to improve fiber–matrix adhesion, which increases the composite tensile strength.²⁶ These ultimate tensile strength results also follow this trend.

For the tensile modulus for the nylon-based (Zytel) materials, all the values were statistically significantly different from each other and were ranked in the following order from highest to lowest: Fortafil 243 > ThermalGraph DKD X > Fortafil 482 > neat Zytel (no filler). For the tensile modulus for the polycarbonate-based (Lexan) materials, all the values were statistically significantly different from each other and were ranked in the following order from highest to lowest: Fortafil 201 > ThermalGraph DKD X > Forta-

fil 402 > neat Lexan (no filler). The composite tensile modulus is often unaffected by improved fiber–matrix adhesion.²⁶

XPS results

The results from the XPS analysis for the fibers used are shown in Table IV.^{21,27} ThermalGraph DKD X had carbon (96.3%) and oxygen (3.7%) present on the carbon fiber surface. The Fortafil carbon fiber had carbon (86.6%), oxygen (8.5%), nitrogen (1.5%), and sodium (3.4%) present. The increased oxygen and nitrogen content on the Fortafil fiber was expected because this fiber was surface-treated to add oxygen and nitrogen. Often, carbon fibers are surface-treated to improve fiber–matrix adhesion, which increases the composite tensile strength and often does not affect the composite tensile modulus.²⁶ The tensile results in Table III agree with these trends. For both matrix materials, the composites containing Fortafil carbon fibers had higher tensile strength than the composites containing ThermalGraph DKD X. In addition, the ARs were similar for sample BN20 (AR = 11.0) and NCN20 (AR = 9.5), yet the composite tensile strength was higher for sample BN20 (tensile strength = 126.5 MPa) than for sample NCN20 (tensile strength = 99.9 MPa). A similar observation was made when we compared samples BP20 and NCP20. This suggested that improved fiber–matrix adhesion yielded a higher composite tensile strength.

TABLE IV
XPS Results^{21,27}

Material	Oxygen on filler surface (atom %)
Zytel 101 NC010	—
Lexan HF 1110- 111N	—
ThermalGraph DKD X	3.7
Fortafil carbon fiber	8.5

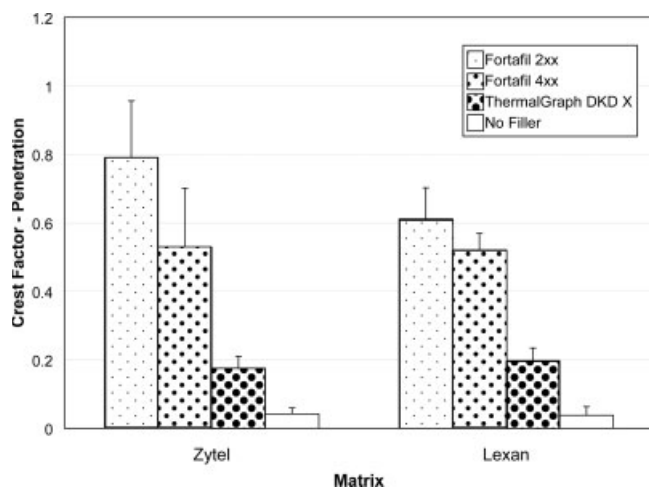


Figure 9 Crest factors for penetration during the scratch test. The error bars show one standard deviation.

Nanoscratch results

The results for the crest factor are depicted in Figures 9 and 10 for friction force and penetration, respectively. The results for the composites reflect the combined effect of the fibers and the interphase around the fiber. Because the properties of the interphase affected the global response of the composite, a stronger bond between the fiber and the matrix should have resulted in relatively better mechanical properties. The crest factor, by virtue of with the maximum range in the data measured, could be considered a measure of the resistance of the material to scratching. A material system that has better adhesion between the fiber and the matrix should have higher resistance to penetration and scratching, which results in smaller penetration depths and higher friction forces. As shown in these figures, the crest factor for the matrix materials Lexan and Zytel was almost zero

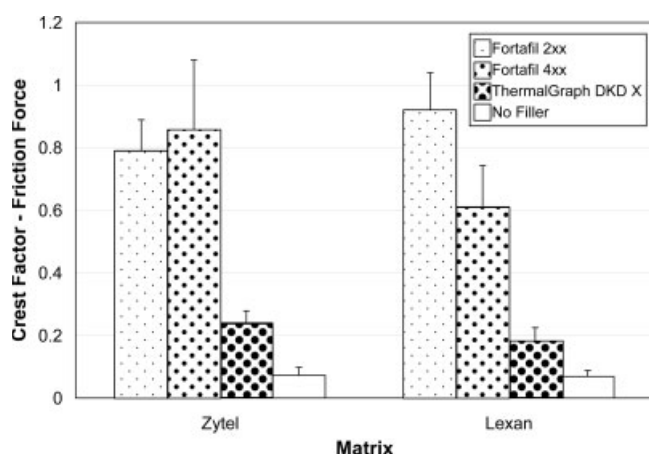


Figure 10 Crest factors for friction force during the scratch test. The error bars show one standard deviation.

because there was no perturbation in the displacement due to lack of fibers.

Statistical analysis of the crest results for the friction data yielded the following statistical differences. For the Zytel matrix, the composites containing Fortafil 243 and Fortafil 482 were not significantly different from each other but were statistically higher than both the Zytel/ThermalGraph DKD X and neat matrix. Composites containing ThermalGraph DKD X had a statistically higher crest factor than the neat matrix. For the Lexan matrix, the composites containing Fortafil 201 were statistically the highest, followed by the composites containing Fortafil 402. Lexan/ThermalGraph DKD X and Lexan with no filler were statistically the lowest, but there was no difference between them.

When looking at the crest factor for the penetration data, we saw a similar result. For the Zytel matrix, all of the crest factors were significantly different than each other and were ranked in the following order from highest to the lowest: Fortafil 243 > Fortafil 482 > ThermalGraph DKD X > Zytel matrix. For the Lexan matrix, composites containing Fortafil 201 and Fortafil 402 had the highest crest factor but were not significantly different from each other. These were followed by Lexan/ThermalGraph DKD X and then neat Lexan (lowest), which were statistically different from each other and from the composites containing Fortafil 201 and Fortafil 402.

A comparison of the figures for Lexan/Fortafil 2xx (Figs. 6 and 8) and Lexan/ThermalGraph DKD X (Figs. 11 and 12) revealed that the measured penetration depth or the friction force range between the matrix-rich and fiber-rich areas was larger for the Lexan/Fortafil than for the Lexan/ThermalGraph DKD X composite. Although the graphs for Zytel/Fortafil and Zytel/ThermalGraph are not presented for sake of brevity, the same observations held for these composites; that is, composites with surface-

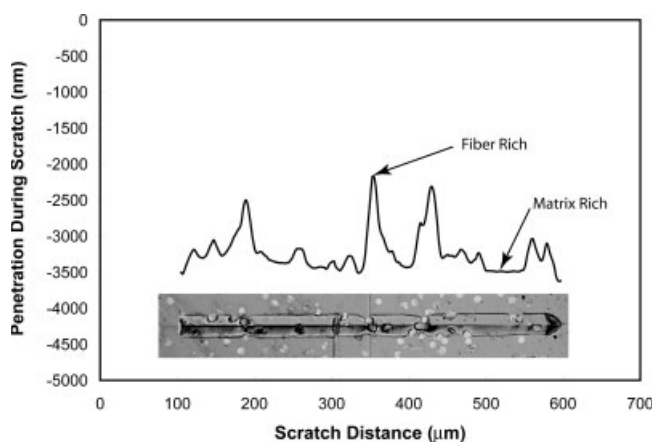


Figure 11 Displacement normal to the surface under a constant force of 40 mN for sample NCP20.

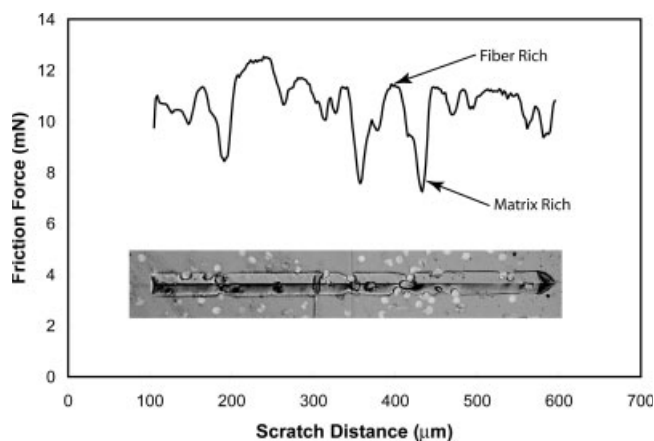


Figure 12 Friction force along the scratch direction for sample NCP 20.

treated fibers resulted in a larger range in friction force and penetration. On the sole basis of the higher tensile modulus of the ThermalGraph DKD X fiber (827 GPa in Table II) as compared to that of the Fortafil fibers (227 GPa in Table II), one would have expected a larger difference in the fiber-rich versus matrix-rich regions for penetration during scratch and friction force. Instead, the opposite was observed. Hence, it appears that the friction force and penetration depth provide an indication of fiber–matrix adhesion over that of the constituent fiber properties. Thus, the crest factor as defined in eq. (1) can be used as a measure to compare the relative degree of adhesion between different composites having the same matrix but different types of fibers. The results in Figures 9 and 10, coupled with the statistical analysis and the tensile strength results shown in Table III, indicate that higher crest factors are evidence of better adhesion between the fiber and the matrix.

CONCLUSIONS

Nanoscratch tests performed on carbon-filled nylon 6,6 and polycarbonate resins provided data that could be used to measure the relative strength of the adhesion. The scratch data was characterized by a parameter called the crest factor, mainly used in the study of dynamic signal processing. It was shown that this parameter could effectively characterize the relative level of adhesion between the fiber and the matrix. The sample preparation for scratch tests was simple, and the tests could be run relatively quickly. Data analysis was quick, and hence, crest factor results could be obtained for quick comparison of the adhesion in different composite samples. Although both penetration and friction force data was used in this study, the results indicate that just penetration or friction force data would be sufficient for the comparisons. However, because a typical nanoscratch instrumentation

would be capable of recording these data, we recommend that both friction force and penetration data be used to check the consistency of the results.

The nanoscratch findings from this study were consistent with the composite tensile strength and the amount of oxygen and nitrogen present on the fiber surface.

The authors thank BP/Amoco/Cytec and DuPont for providing polymer and carbon fibers. The authors also thank S. Li for his help with the scratch testing.

References

- Ishida, H. *Interfaces in Polymer, Ceramic, and Metal Matrix Composites*; Elsevier: New York, 1998.
- Ishida, H. *Controlled Interfaces in Composite Materials*; Elsevier: New York, 1998.
- Hughes, J. D. *Compos Sci Technol* 1991, 40, 13.
- Mackin, T. J.; Haverson, T. L.; Sottos, N. R. *Polym Compos* 2005, 26, 241.
- Marshall, D. B.; Oliver, W. C. *J Am Ceram Soc* 1987, 70, 542.
- Haerberle, D. C.; Lesko, J. J.; Case, S. W. *Proceedings of the American Society for Composites, 16th Technical Conference, 2001*; Hyer, M. W.; Loos, A. C., Eds.; Paper 211; CRC Press: Boca Raton, FL.
- Ash, J. T.; Cross, W. M.; Griswold, C.; Henderson, B.; Kellar, J. J.; Kjerengtroen, L. In *Proceedings of the 2002 SEM Annual Conference and Exposition on Experimental and Applied Mechanics*; The Printing House, Inc.: Stroughton, WI, 2002; p 65.
- Oliver, W. C. *Mater Res Soc* 1986, 11(5), 15.
- Oliver, W. C.; Parr, G. M. *J Mater Res* 1992, 7, 1564.
- Frantziskonis, G. N.; Matikas, T. E.; Karpur, P.; Krishnamurthy, S. In *Proceedings of the First International Conference on Composites Engineering*; Hui, D., Ed.; Technomic: Lancaster, PA, 1994; p 147.
- Clingerman, M. L.; Weber, E. H.; King, J. A.; Schulz, K. H. *Polym Compos* 2002, 23, 911.
- DuPont Zytel Nylon Resin Product and Properties, Version 95.9; DuPont Engineering Polymers; Wilmington, DE; 1995.
- GE Engineering Thermoplastics Product Guide: Lexan PC Resin, CDC-6621 (2/98) CA; GE Plastics: Pittsfield, MA.
- Akzo Nobel Fortafil Carbon Fibers Technical Data Sheet 931B; Rockwood, TN, 1993.
- Amoco Performance Products: High Thermal Conductivity Pitch Based Graphite Fibers; Amoco Polymers: Alpharetta, GA, 1996.
- Heiser, J. A.; King, J. A.; Konell, J. P.; Miskioglu, I.; Sutter, L. L. *J Appl Polym Sci* 2004, 91, 2881.
- Krueger, Q. J.; King, J. A. *Adv Polym Technol* 2003, 22, 96.
- Tensile Properties of Plastics*; ASTM Standard D 638; American Society for Testing and Materials: Philadelphia, 1998.
- Broch, J. T. *Mechanical Vibrations and Shock Measurements*; Bruel & Kjaer: Naerum, Denmark, 1972.
- Kim, J. K.; Sham, M. L.; Wu, J. S. *Compos A* 2001, 32, 607.
- Clingerman, M. L. Ph.D. Dissertation, Michigan Technological University, 2001.
- Glantz, S. A. *Primer of Bio-Statistics*, 3rd ed.; McGraw-Hill: New York, 1992.
- Clingerman, M. L.; King, J. A.; Schulz, K. H.; Meyers, J. D. *J Appl Polym Sci* 2002, 83, 1341.
- Konell, J. P.; King, J. A.; Miskioglu, I. *Polym Compos* 2004, 25, 172.
- Clingerman, M. L.; Konell, J. P.; Weber, E. H.; King, J. A.; Schulz, K. H. In *Proceedings of the Society of Plastics Engineering Annual Technical Conference, Society of Plastics Engineers, Brookfield, CT, 2001*; p 1364.
- Kardos, J. L. *CHEMTECH* 1984, 88, 431.
- Clingerman, M. L.; Weber, E. H.; King, J. A.; Schulz, K. H. *J Appl Polym Sci* 2003, 88, 2280.