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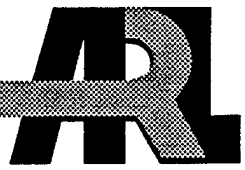

Thermal Degradation Effects on Consolidation and Bonding in the Thermoplastic Fiber-Placement Process

by Bruce K. Fink, John W. Gillespie, Jr., and Nuri B. Ersoy

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Abstract

Effects of elevated temperature exposure during thermoplastic fiber placement on bonding and consolidation are investigated experimentally for AS4/polyetherketoneketone (PEKK) composite. Coupons of 24 layers are consolidated on the University of Delaware Center for Composite Materials (UD-CCM) fiber-placement robot at deposition rates of 20 and 40 mm/s over a range of process temperatures (700–900 °C, with 50 °C increments). The main torch and preheater distances and the compaction force are held constant for all coupons. Two competing mechanisms governing strength buildup are considered: (1) polymer bonding and (2) degradation. Coupons are sectioned, and one-half of each coupon is reconsolidated in a hot press at conventional processing conditions (i.e., 30 min at 370 °C and 0.70-MPa pressure) in order to remove any effect of poor consolidation on strength. Void content of the robot-consolidated panels is measured. Short-beam shear (SBS) tests are performed on the specimens cut from each coupon. Strength and void-content measurements for robot-consolidated panels are presented to illustrate the effect of processing parameters on product quality. Results of the SBS strength tests performed on reconsolidated coupons indicate that there is significant decrease in the strength of coupons consolidated at 20-mm/s deposition rate and high torch temperatures, possibly due to polymer degradation, whereas approximately the same value of reconsolidated strength is measured for the 40-mm/s deposition rate, suggesting that polymer degradation is insignificant at that rate. Effects of void content on SBS tests are discussed.

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1. Introduction

The recent advances in automated manufacturing technology of composite materials resulted in a highly promising process called fiber placement. In the fiber-placement process, as opposed to filament winding, (1) the fibers are not restricted to near-geodesic paths, (2) paths can start and end at any point, (3) the fibers can be placed along concave mandrel sections, and (4) the amount of fiber and the width of the fiber band can be changed along the width of the delivery path. This makes it possible to fabricate very general nonaxisymmetric shapes with varying thickness.

The advantages of fiber-placement processes can be combined with the high throughputs that can be achieved using thermoplastic prepreg tape that can be shaped and consolidated on line. This is due to the chemical structure of the resin that enables melting under heat and consolidation before cooling. On-line consolidation technology thus eliminates the use of an autoclave by applying heat directly at the nip-point, welding the tape on the preceding layer under compaction pressure, and consolidating shortly behind the nip-point where the temperature is lower.

Mechanisms governing strength buildup and consolidation during thermoplastic fiber-placement processes should be well understood in order to control the processing parameters to obtain the optimum product quality.

2. Experimental Work

2.1 Fiber-Placement Equipment. At the University of Delaware Center for Composite Materials (UD-CCM), the first experiences with thermoplastic filament winding and tape placement were gained by Wells and Steiner [1]. Considerable improvements to the entire robotic fiber-placement workcell concerning heating sources, mandrel manipulator, placement head capabilities, and process control have been integrated as a result of several investigations [2, 3].

The centerpiece of the robotic tape-placement workcell used in this investigation was a Model 762 AEG-Westinghouse PUMA industrial robot. It has a repeatability of 0.02 mm, six axes, a maximum reach of 1,244 mm, and a payload of 200 N. The PUMA control system operates on the VAL II high-level language. It can be linked to the environment through 32 input/output ports and control signals.

The prototype tape-placement head is mounted to the robot arm by a pair of linear bearings. As shown schematically in Figure 1, thermoplastic prepreg tape is fed from a supply spool and guided through several eyelets, the rollers of the tape start lever, and the cutting unit, which allows cutting and refeeding of the tape. The magnetic brake attached to the supply spool exerts a constant tape tension. After passing the cutter, the incoming tape, as well as the formerly placed substrate, are preheated by the first hot gas torch. The tape is then guided through the lay-down roller that establishes contact between the substrate and the preheated tape. The second torch heats through the thickness to melt the entire tape and tape-substrate interface. Passing the air-cooled compaction roller, the tape consolidates onto the substrate under pressure and cools down, while the voids within and between the tape and substrate are reduced and bonding develops. In this way, the tape can be placed on a flat mandrel by laying one strip next to another.

The hot gas torches are fed with nitrogen. The flow rate and the temperature of the outcoming gas for two torches are controlled separately by a hot gas torch control system manufactured by Automated Dynamics Corporation. National Instruments LabVIEW-based process control software is linked to the robot controller and the torch controller, which allows for on-line setting of the process parameters such as gas flow rate, torch temperatures, and compaction force.

2.2 Materials. The material used for experiments was AS4 graphite/polyetherketoneketone (PEKK) with 58% fiber-volume fraction. The void content of the incoming tow is measured to be 3.8 ± 0.8%. PEKK was developed by DuPont as a high-performance thermoplastic matrix system for advanced composites. The carbon-fiber-reinforced composite laminates with

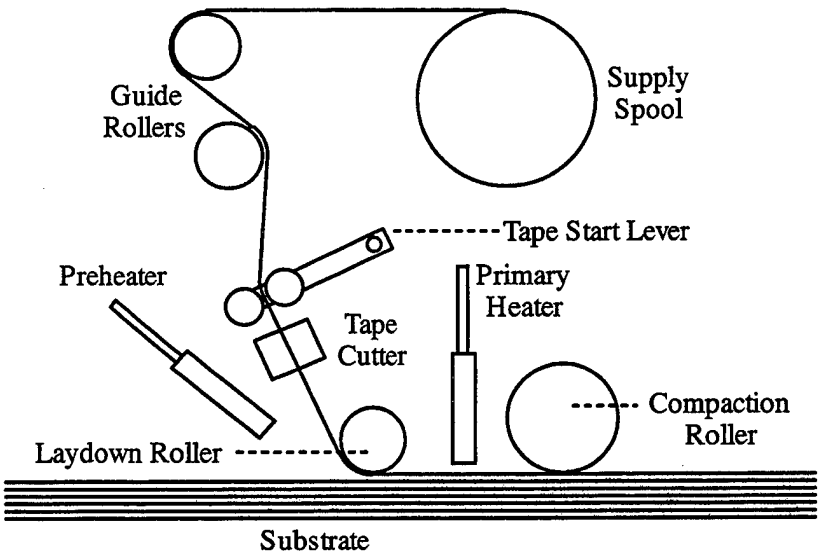

Figure 1. Schematic of Fiber-Replacement Process Showing Components.

PEKK matrix, prepared from the propriety melt impregnated tows, show high flexural, shear, and compressive strengths and good environmental durability and hot-wet stability.

2.3 Preparation of Test Coupons. Test coupons were consolidated on a graphite plate with deposition rates of 20 and 40 mm/s. Both torch temperatures were varied from 700 to 900 °C with 50 °C increments. The main torch and preheater distances and the compaction force were held constant for all test coupons. Figure 2 shows the process parameters, and Table 1 shows the settings used for consolidating the test coupons. For the case of 900 °C torch temperatures and 20-mm/s deposition rate, a test coupon could not be consolidated because excessive polymer degradation prevents deposition of fresh tows. After consolidating on the robot, test coupons were sectioned, and one-half of each coupon was reconsolidated in the hot press at conventional processing conditions (i.e., 30 min at 370 °C under 0.70-MPa pressure) in order to remove any effect of poor consolidation and bonding on strength. Later, short-beam shear (SBS) specimens were cut from both the robot-consolidated and reconsolidated coupons.

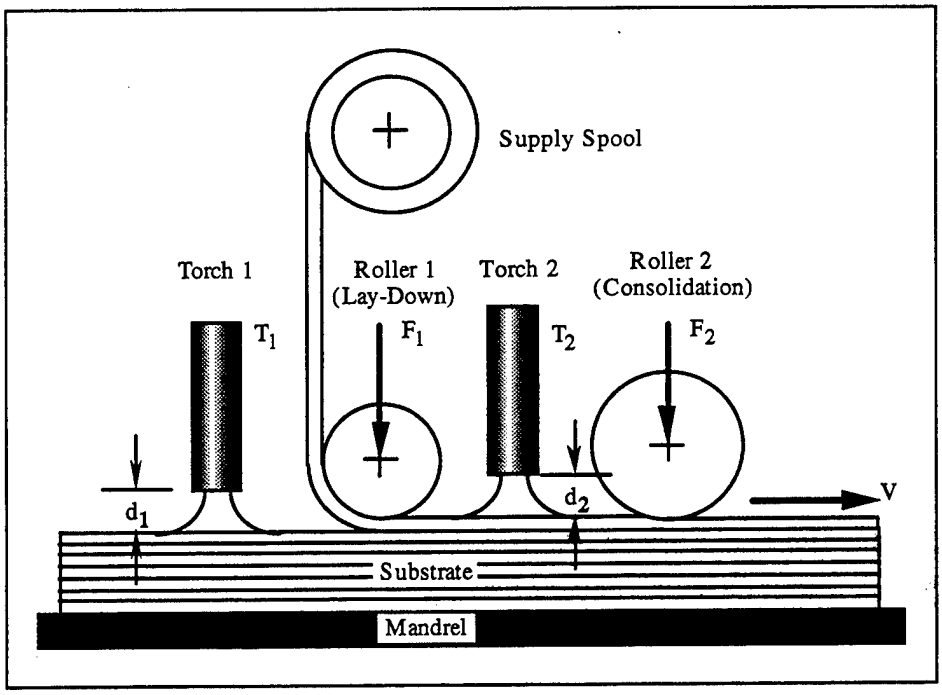

Figure 2. Schematic of Fiber-Placement Process Showing Variables.

Table 1. Process Variables and Settings

Process Variable	Abbreviation	Settings
Deposition Rate	V	20 and 40 mm/s
Compaction Force[a]	F_2	320 N
Preheater Temperature	T_1	700–900 °C, with 50 °C increments
Preheater Distance[a]	D_1	20 mm
Main Torch Temperature	T_2	700–900 °C, with 50 °C increments
Main Torch Distance[a]	D_2	4 mm

[a] Real-time computer-controlled process variables.

2.4 SBS Tests. The SBS strength of test coupons was measured according to the American Society for Testing of Materials (ASTM) D 2344-84 [3, 4]. Accordingly, this method is useful for quality control and specification purposes and is also applicable for research and development purposes concerned with interply strength. Although the apparent shear strength obtained in this test cannot be used as a design criterion, it can be utilized for comparative testing of composite materials, if all failures are in horizontal shear.

SBS specimens were cut from test coupons by a diamond saw to nominal dimensions of 6.35 mm width and six times the specimen thickness. Although 24 layers were deposited for each test coupon, the thickness of the coupons varied with processing parameters; hence, the thickness of the specimens was an uncontrolled parameter. Ten specimens were used for each case in order to obtain a satisfactory average. The thickness and width of each specimen were measured to the nearest 0.025 mm at midpoint. The specimens were placed in the test fixture, as shown in Figure 3. The side supports of the fixture were pushed to the span of four times the specimen thickness. The specimens were aligned so that the midpoint was centered and the long axis was perpendicular to the cylindrical axis (under the loading nose). The tests were performed using an Instron 1125 screw-driven test frame at room temperature using 454-kg (1,000 lb) load cell at full scale and 1.3-mm/min (0.05 in/min) crosshead speed. The failure load was recorded as the first maximum load in the load-displacement diagram. The shear strength was calculated as

$$S_H = 0.75\frac{P_B}{bd}, \tag{1}$$

where S_H is the shear strength, P_B is the breaking load, b is the width, and d is the thickness of specimen. Arithmetic mean and standard deviation of values obtained for each test coupon were calculated.

2.5 Microscopic Examinations. Samples for microscopic examination were prepared by cutting the robot-consolidated test coupons by a diamond saw perpendicular to the fiber direction. Samples were embedded in epoxy resin and ground using an automated fixture on 180-, 360-, and 600-grid silicon carbide paper and then finished on 12.5-, 9.5-, 5-, 3-, and 1-μm polishing cloths, applying the specified alumina powder/water suspension. An ultrasonic water bath was used to clean the samples between successive steps. The samples were examined under reflected light with 100× magnification. Ten snapshots of capturing frames at the surface and middle of each test coupon were recorded to videotape and imported to a Macintosh Quadra 650 workstation equipped with a video-capture board. Void-content measurements were performed with NIH Image 1.47 image analysis software.

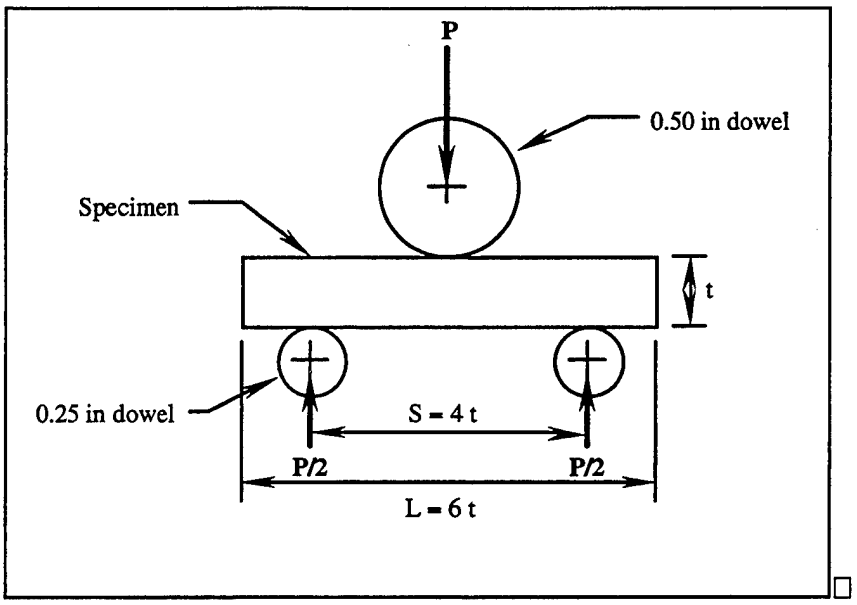

Figure 3. Schematic of SBS Test.

3. Results and Discussion

The strength and void-content measurement results from the fiber-placement process are presented with a two-fold objective: (1) to illustrate the influence of the physical processing parameters on the bond-strength development and void-content reduction and (2) to delineate the relative effects of two competing mechanisms governing strength buildup (polymer bonding and degradation).

3.1 SBS Tests. Arithmetic mean and standard deviation of values obtained from 10 SBS samples for each test coupon were calculated and are tabulated in Tables 2 and 3 for 20- and 40-mm/s deposition rates, respectively. From Table 3, for 40-mm/s deposition rate, approximately the same value strength is obtained upon reconsolidation. Hence, the average of the reconsolidated strength values for that deposition rate, which comes out to be 82.6 ± 2.9 MPa (11.98 ± 0.42 Ksi), is used as a baseline.

In Figures 4 and 5, SBS strength values of the robot-consolidated and reconsolidated coupons are plotted vs. torch temperature for 20- and 40-mm/s deposition rates, respectively. As can be

Table 2. SBS Data for 20-mm/s Deposition Velocity

Torch Temperature (degrees Celsius)	Robot-Consolidated SBS Strength (MPa [Ksi])	Reconsolidated SBS Strength (MPa [Ksi])
700	44.9 ± 6.1 [6.51 ± 0.89]	80.2 ± 2.2 [11.63 ± 0.32]
750	56.5 ± 2.8 [8.19 ± 0.41]	80.6 ± 3.2 [11.69 ± 0.47]
800	65.1 ± 2.5 [9.44 ± 0.36]	73.4 ± 2.3 [10.64 ± 0.34]
850	67.2 ± 1.3 [9.75 ± 0.19]	59.0 ± 2.6 [8.55 ± 0.38]

Table 3. SBS Data for 40-mm/s Deposition Velocity

Torch Temperature (degrees Celsius)	Robot-Consolidated SBS Strength (MPa [Ksi])	Reconsolidated SBS Strength (MPa [Ksi])
700	43.4 ± 5.3 [6.30 ± 0.77]	79.4 ± 1.5 [11.52 ± 0.22]
750	49.9 ± 6.0 [7.23 ± 0.87]	81.4 ± 3.3 [11.80 ± 0.48]
800	55.8 ± 4.9 [8.09 ± 0.71]	82.9 ± 2.8 [12.02 ± 0.41]
850	64.1 ± 2.2 [9.30 ± 0.32]	83.6 ± 4.7 [12.12 ± 0.68]
900	62.5 ± 4.3 [9.07 ± 0.63]	82.8 ± 2.9 [12.00 ± 0.42]

seen from Figure 4, there is a considerable drop in reconsolidated strength for torch temperatures of 800 and 850 °C, indicating that full strength represented by the baseline cannot be recoverable upon reconsolidation. At the deposition rate of 40 mm/s, the dwell time of the polymer at high temperatures under preheat and main torches is shorter, and polymer thermal degradation is expected to be insignificant and to not affect further strength buildup upon reconsolidation.

The origin of polymer degradation is uncertain. It may be due to (1) decomposition of the polymer matrix at the surface of the tow due to exposure of high-temperature stream of nitrogen gas, reducing the thickness of the resin rich layer and hence unveiling the carbon fibers, or (2) cross-linking or cyclization of the polymer chains, reducing the chain mobility and hence, ability to diffuse and bond. Fourier-transform infrared (FTIR) and thermal gravimetric analysis (TGA) characterization studies of neat PEKK films and PEKK-based composites are presented in the Appendices. While degradation resulting in weight loss can be monitored with these techniques, the temperatures at which weight-loss degradation occurs is presumed to be

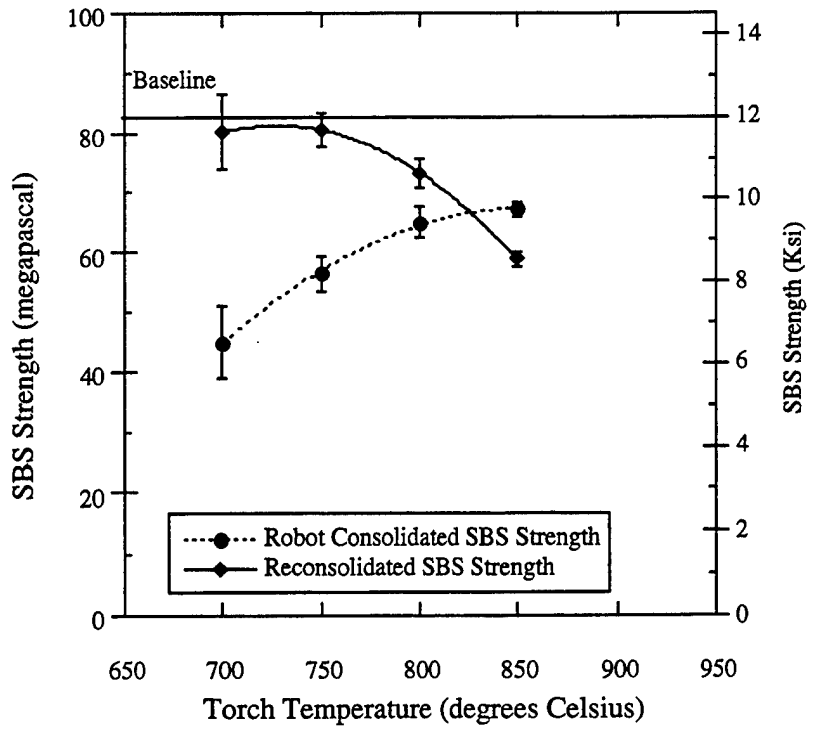

Figure 4. Effect of Torch Temperature on SBS Strength for 20-mm/s Deposition Rate.

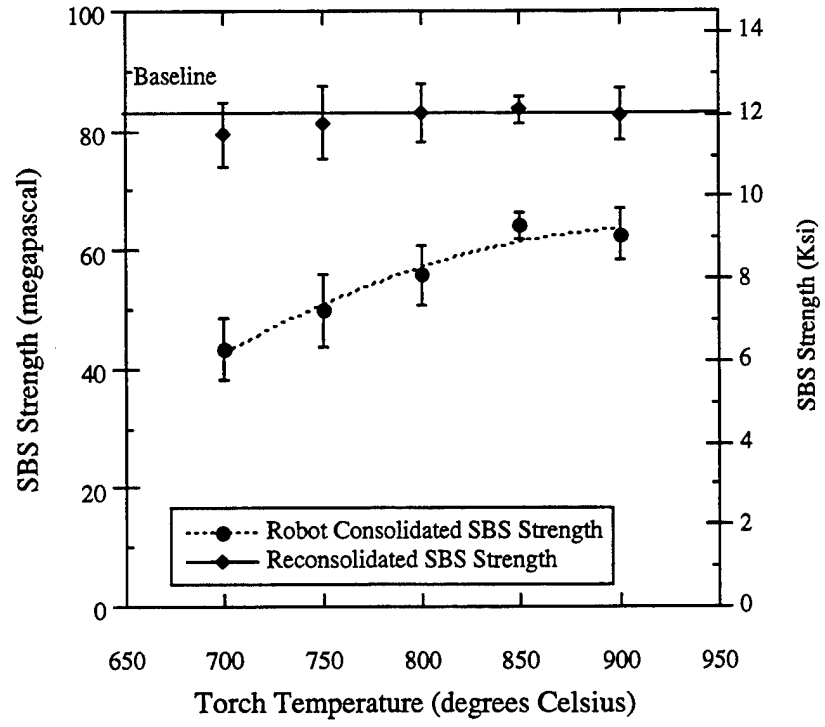

Figure 5. Effect of Torch Temperature on SBS Strength for 40-mm/s Deposition Rate.

purposefully avoided in the manufacturing process. The origin of nonweight-loss polymer degradation and its effect on bonding needs further investigation.

3.2 Microscopic Examinations. Arithmetic mean and standard deviation of void content values in the bulk of each test coupon are calculated and tabulated in Tables 4 and 5 for 20- and 40-mm/s deposition rates, respectively. In Figure 6, void contents of the test coupons are plotted against torch temperature. For high temperatures, the difference between the surface and bulk void contents is higher. At these temperatures, the region at the melt state is expected to be deeper, and void reduction and further consolidation are expected to take place upon subsequent passes when fresh tows are placed on already consolidated substrates.

Table 4. Void Content Measurements for 20-mm/s Deposition Velocity

Torch Temperature (degrees Celsius)	Void Content (bulk) (percent)	Void Content (surface) (percent)
700	1.60 ± 0.48	4.00 ± 0.80
750	1.27 ± 0.25	2.48 ± 0.38
800	1.13 ± 0.23	2.37 ± 0.30
850	1.00 ± 0.19	3.06 ± 0.67

Table 5. Void Content Measurements for 40-mm/s Deposition Velocity

Torch Temperature (degrees Celsius)	Void Content (bulk) (percent)	Void Content (surface) (percent)
700	2.58 ± 0.95	3.27 ± 0.87
750	1.55 ± 0.37	2.16 ± 0.41
800	0.84 ± 0.24	1.07 ± 0.19
850	0.37 ± 0.09	1.27 ± 0.40
900	0.33 ± 0.09	1.66 ± 0.55

Figures 7–10 show typical micrographs at the surface and in the bulk of the cross sections perpendicular to the fiber direction for four extreme cases. As can be seen qualitatively from these micrographs, most of the voids are located at the intralaminar region, and interlaminar

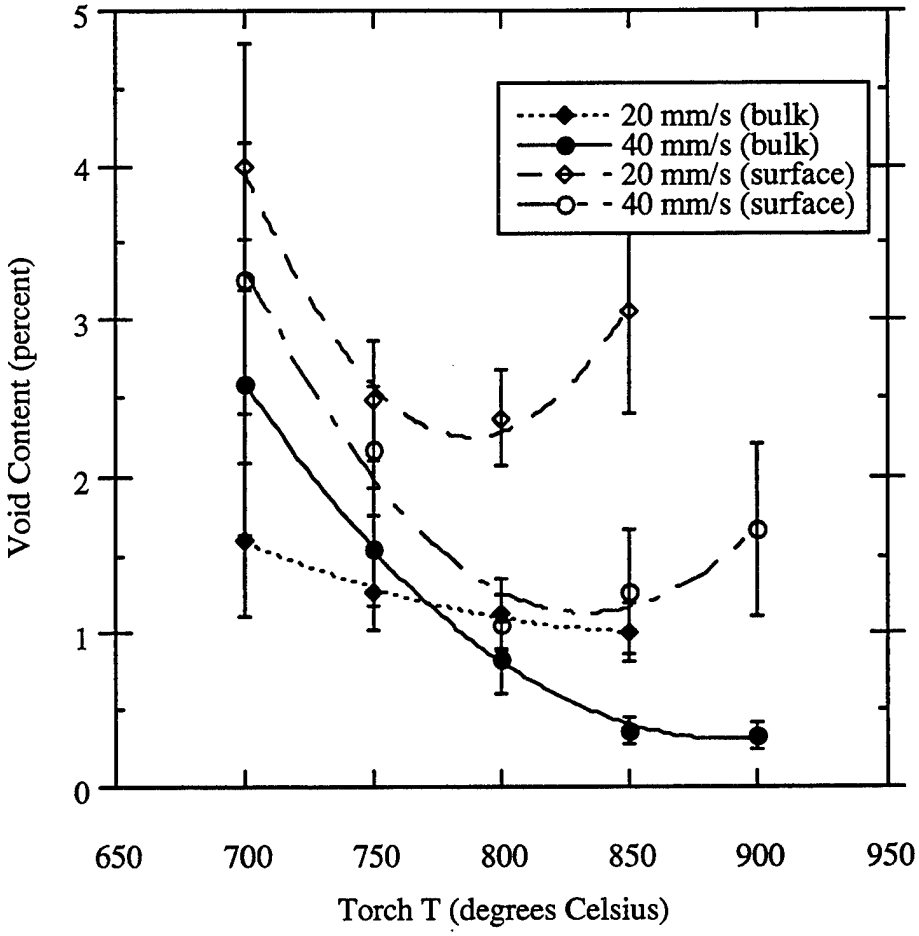

Figure 6. Effect of Processing Conditions on Void Content.

voids and regions of poor bonding can be seen for low torch temperatures at both deposition rates (Figures 7 and 9). Bigger voids in the topmost layer are evidence of void growth promoted by high torch temperatures and long dwell times.

3.3 Effect of Voids on SBS Strength. As indicated in the previous section, most of the voids observed were located in the intralaminar region. These voids may generate stress concentrations and may promote intralaminar fracture during short-beam shear tests, complicating the task of obtaining true interlaminar bond strength. Hence, a study was conducted to evaluate the effect of voids on the measured interlaminar shear strength.

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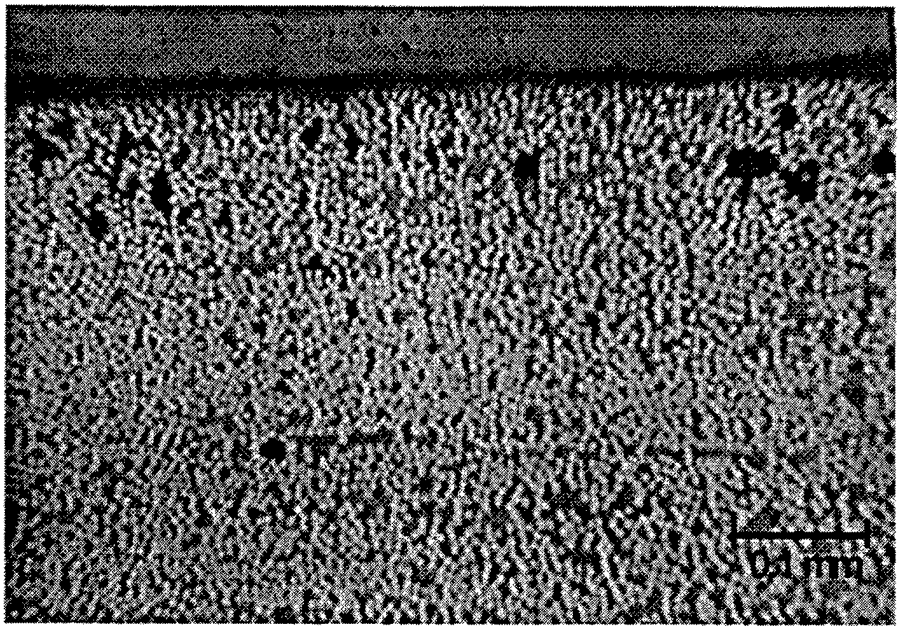


(b)

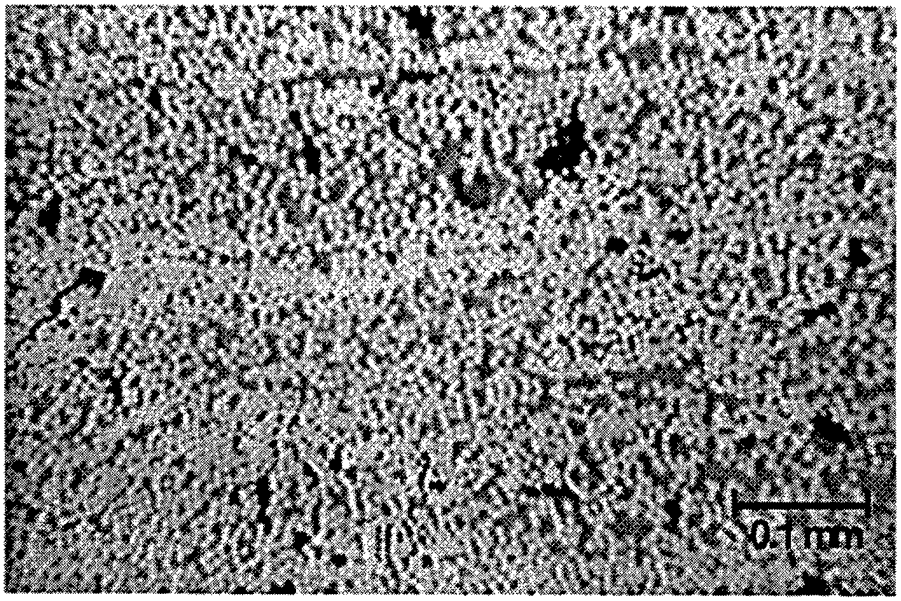
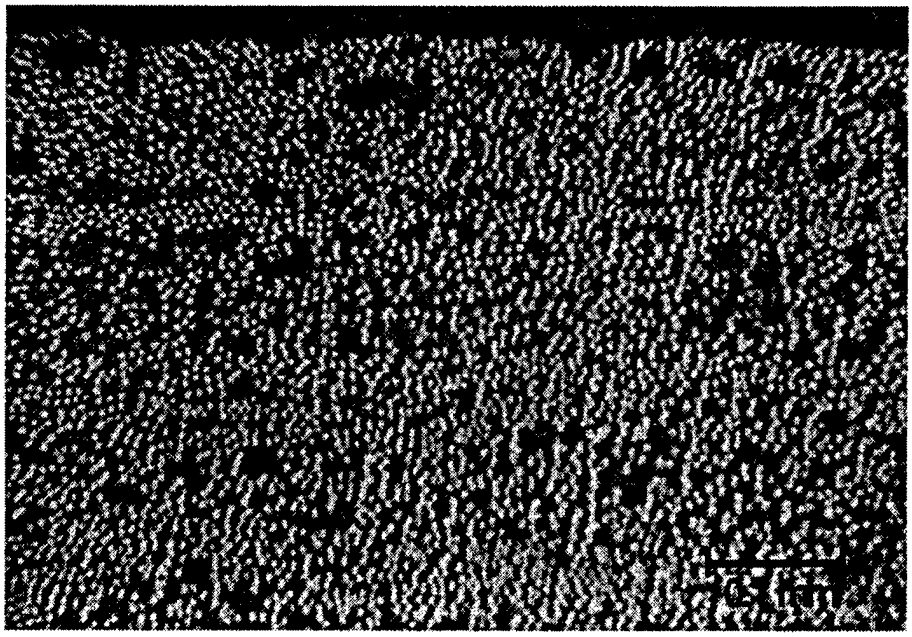


Figure 7. Typical Micrographs of the (a) Surface and (b) Bulk of the Test Coupon Consolidated at 700 °C Torch Temperatures and 20-mm/s Deposition Rate.

(a)



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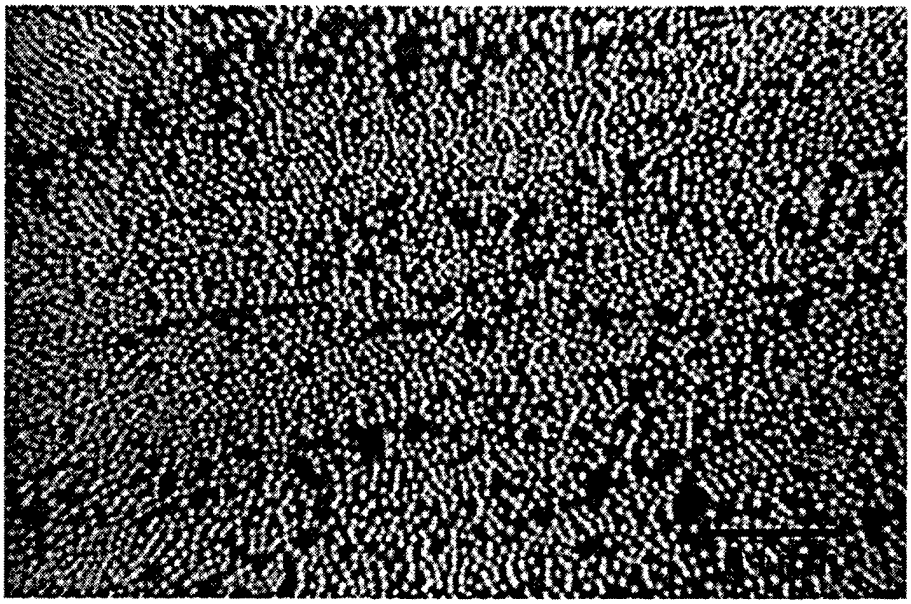
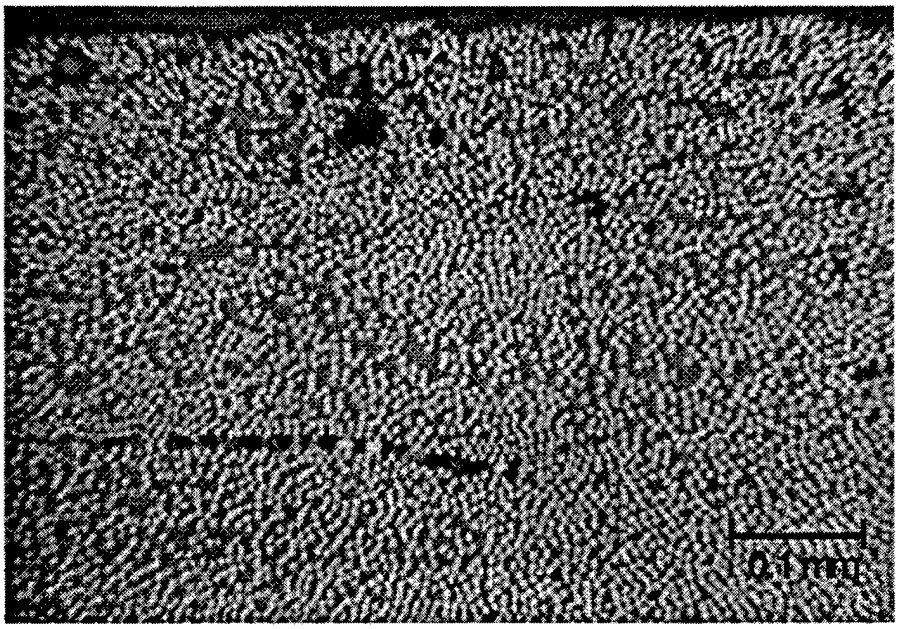


Figure 8. Typical Micrographs of the (a) Surface and (b) Bulk of the Test Coupon Consolidated at 850 °C Torch Temperatures and 20-mm/s Deposition Rate.

(a)



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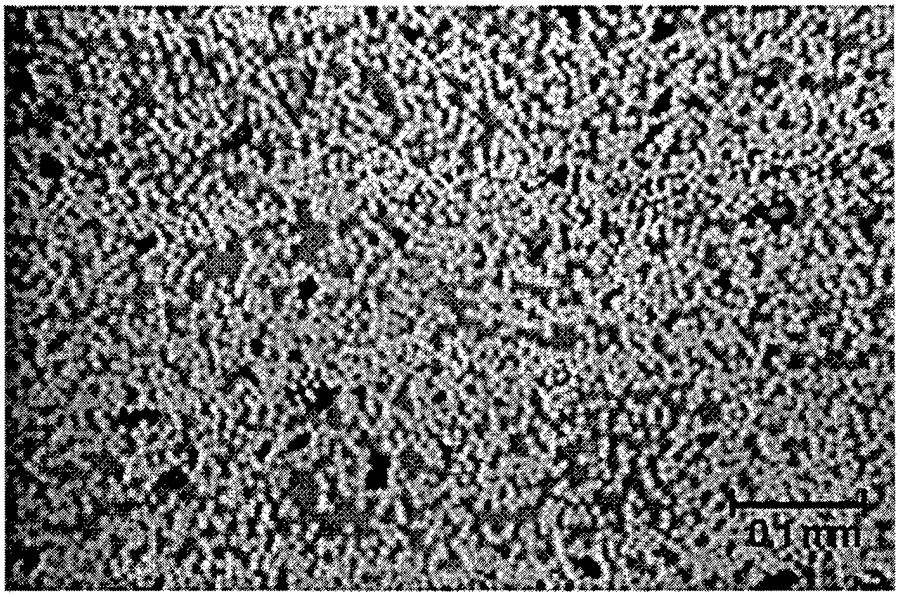
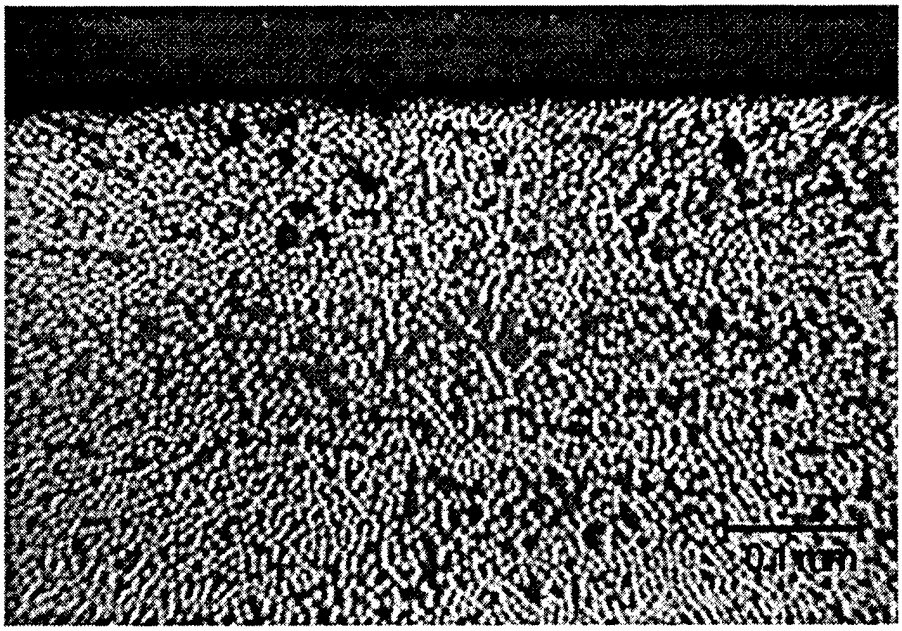


Figure 9. Typical Micrographs of the (a) Surface and (b) Bulk of the Test Coupon Consolidated at 700 °C Torch Temperatures and 40-mm/s Deposition Rate.

(a)



(b)

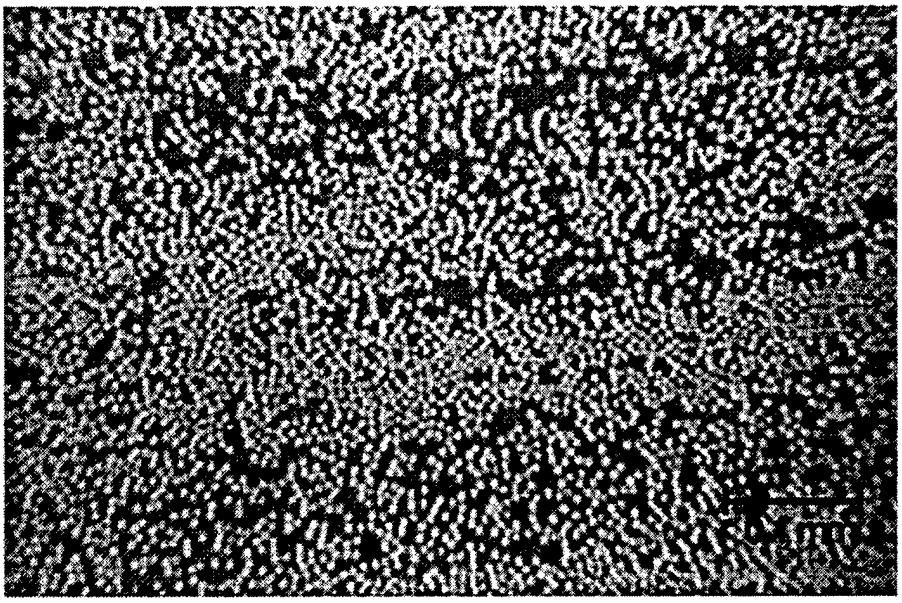


Figure 10. Typical Micrographs of the (a) Surface and (b) Bulk of the Test Coupon Consolidated at 850 °C Torch Temperatures and 40-mm/s Deposition Rate.

The effect of void fraction on interlaminar shear strength has been analyzed by Greszczuk [5], Bowles and Frimpong [6], and by Olson et al. [7]. Bowles and Frimpong theoretically derived the following equations for two possible configurations for voids in composites—cylindrical and spherical:

cylindrical: $$\text{ILLS}_r = 1 - [4V_v / 3.14(1 - V_{fv})]^{1/2}, \quad (2)$$

spherical: $$\text{ILSS}_r = 1 - 0.785[6V_v / 3.14(1 - V_{fv})]^{2/3}, \quad (3)$$

where V_{fc} is the fiber-volume fraction of the composite with voids, V_v is the void-volume fraction, and ILSS_r is the interlaminar shear strength of the composite with voids relative to that of the void-free composite.

In Tables 6 and 7, the ILSS values calculated using equations (2) and (3) for both cylindrical and spherical voids, together with normalized SBS strength data, are tabulated for 20- and 40-mm/s deposition rates, respectively. A baseline strength value of 82.6 MPa is used to normalize the strength data. In Figures 11 and 12, these values are plotted against the torch temperature. ILSS_r calculated for cylindrical voids yields a more conservative estimate, and most of the voids observed in robot-consolidated panels were of cylindrical geometry.

Table 6. Normalized Strength Data for 20-mm/s Deposition Velocity

Torch Temperature (degrees Celsius)	Robot-Consolidated SBS Strength	Reconsolidated SBS Strength	ILSS_r (Sph. Voids)	ILSS_r (Cyl. Voids)
700	0.55 ± 0.07	0.98 ± 0.03	0.78	0.86
750	0.69 ± 0.03	0.98 ± 0.04	0.80	0.88
800	0.79 ± 0.03	0.89 ± 0.03	0.81	0.89
850	0.82 ± 0.02	0.72 ± 0.03	0.83	0.90

Table 7. Normalized Strength Data for 40-mm/s Deposition Velocity

Torch Temperature (degrees Celsius)	Robot-Consolidated SBS Strength	Reconsolidated SBS Strength	$ILSS_r$ (Sph. Voids)	$ILSS_r$ (Cyl. Voids)
700	0.55 ± 0.07	0.98 ± 0.03	0.78	0.86
750	0.69 ± 0.03	0.98 ± 0.04	0.80	0.88
800	0.79 ± 0.03	0.89 ± 0.03	0.81	0.89
850	0.82 ± 0.02	0.72 ± 0.03	0.83	0.90

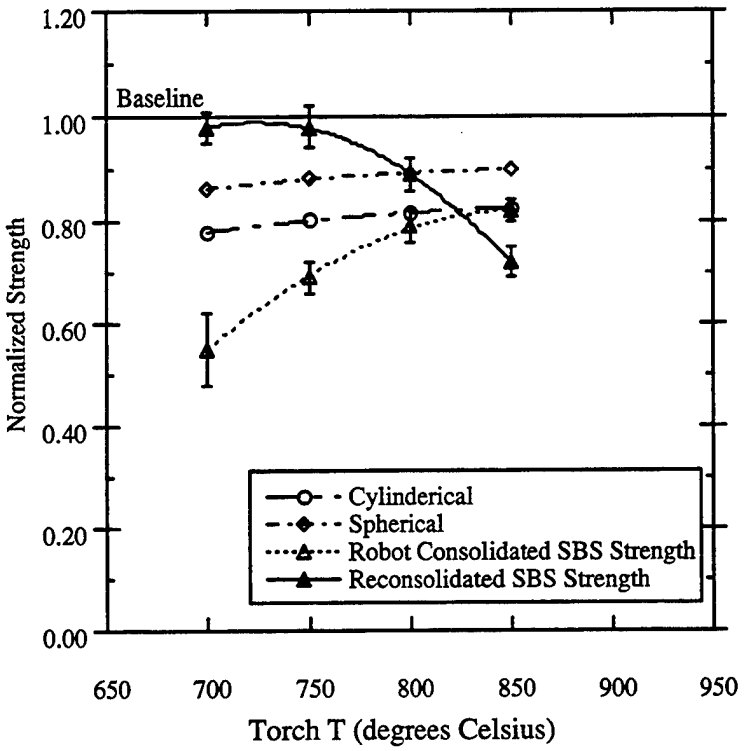


Figure 11. Effect of Void Content on SBS Strength for 20-mm/s Deposition Rate.

As can be seen from Figures 11 and 12, this analysis overestimates the ILSS, except for the torch temperatures of 800 and 850 °C for 20-mm/s deposition rate, indicating that the main mechanism contributing to the strength is interlaminar bonding. In fact, Bowles-type analyses assumes that the composite is a homogeneous material with voids and without any weak interface. For torch temperatures of 800 and 850 °C, polymer degradation occurring at the

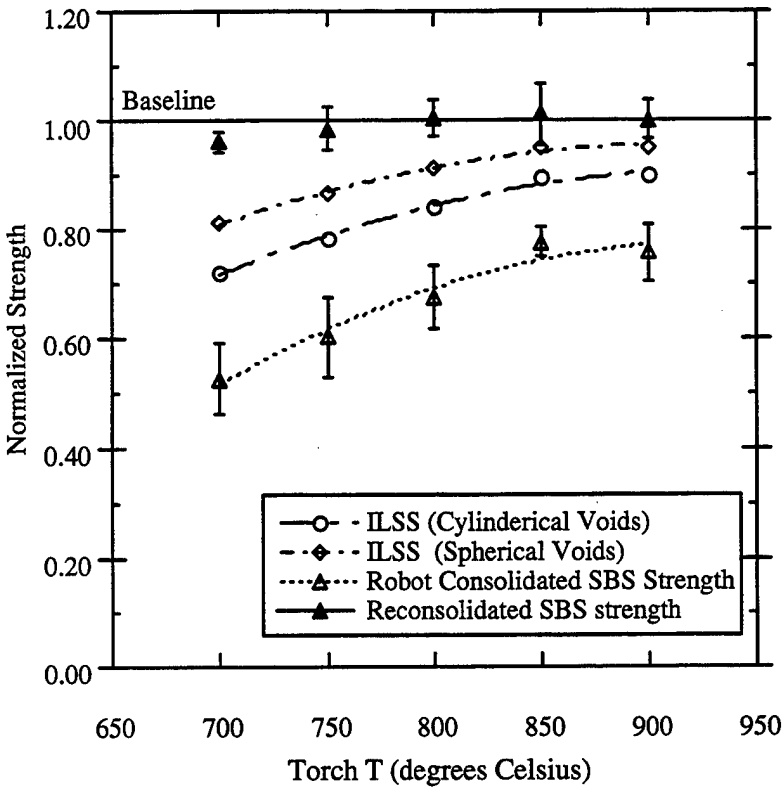


Figure 12. Effect of Void Content on SBS Strength for 40-mm/s Deposition Rate.

interface is believed to be the limiting factor for full-strength development, resulting in a strength loss that is not recoverable upon reconsolidation. Figures 13 and 14 show a fractured SBS specimen manufactured with settings of 700 °C torch temperatures and 20-mm/s deposition rate and 850 °C torch temperature and 20-mm/s deposition rate, respectively. For the former case, a single crack jumping from one interface to another is observed. For the latter case, the crack density was higher, but the cracks were still located at the interface and were separated from each other with a distance of one tow thickness.

4. Conclusions

Effect of processing variables on the quality of the coupons consolidated by thermoplastic fiber-placement method is investigated experimentally, in terms of strength and void content. As a general rule, at higher torch temperatures and slower deposition velocities, higher SBS

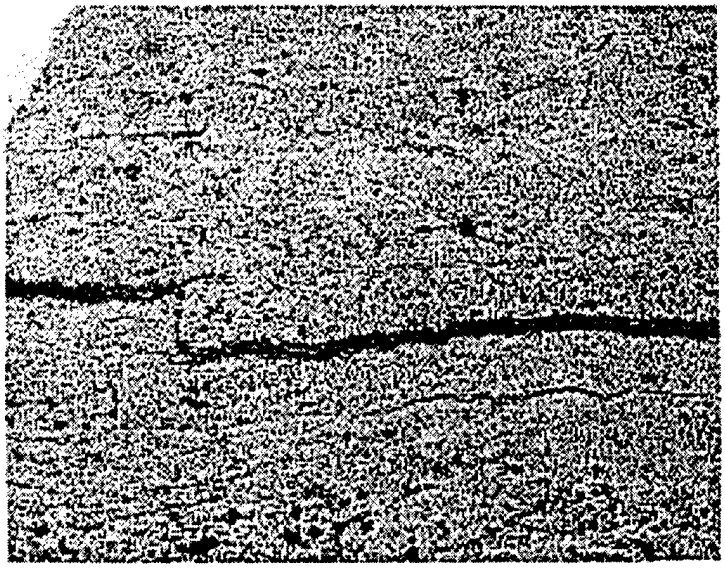


Figure 13. Micrograph of a Typical SBS Specimen Consolidated at 700 °C Torch Temperatures and 20-mm/s Deposition Rate.

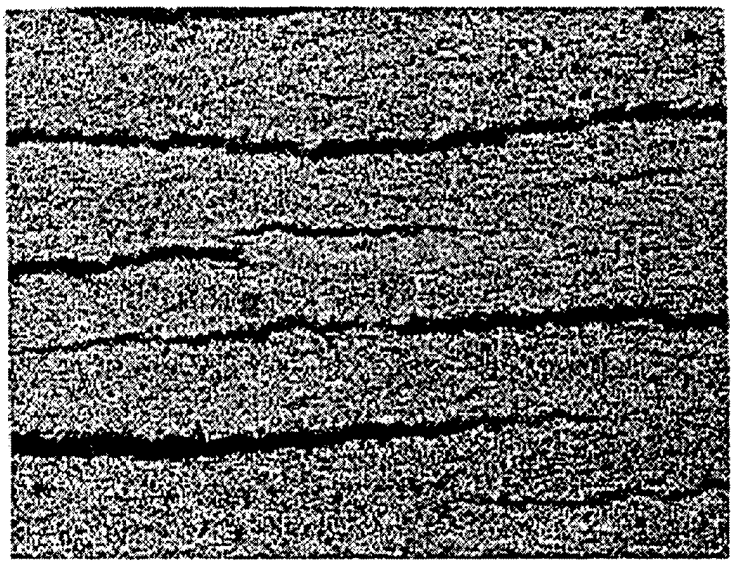


Figure 14. Micrograph of a Typical SBS Specimen Consolidated at 850 °C Torch Temperatures and 20-mm/s Deposition Rate.

strengths were obtained. Void content decreases with increasing torch temperature. However, at high torch temperatures and long dwell times, the "tradeoff" of polymer degradation prevents full-strength development during the process.

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Appendix A:

Thermal Gravimetric Analysis of Degradation of Polyetherketoneketone (PEKK)

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Thermal gravimetric analysis (TGA) was used to determine the temperature range in which the weight-loss mechanism is dominant. Figure A-1 shows the TGA diagrams of polyetherketoneketone (PEKK) films, as-received and processed at 900 °C torch temperature and 20-mm/s deposition rate. TGA tests were run both at air and nitrogen atmosphere. The weight-loss mechanism is not effective until a temperature of 550 °C, and only 0.4% of the weight is converted at a temperature of 450 °C. In order to delineate the effect of carbon fibers on degradation behavior, AS4/PEKK composite is also subjected to TGA tests, both in air and nitrogen atmosphere. TGA diagrams of AS4/PEKK composite are shown in Figure A-2. Comparing Figures A-1 and A-2, it can be asserted that the presence of carbon fibers does not affect the general behavior of degradation and the temperature at weight-loss starts.

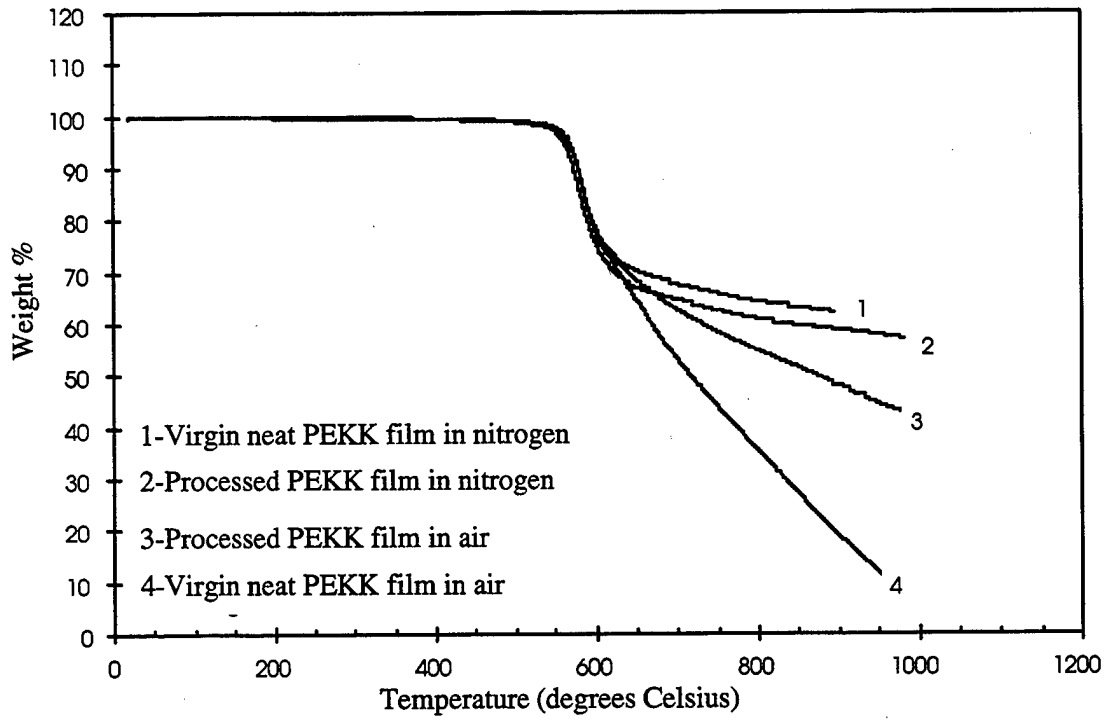

Figure A-1. TGA Diagram of PEKK Neat Films.

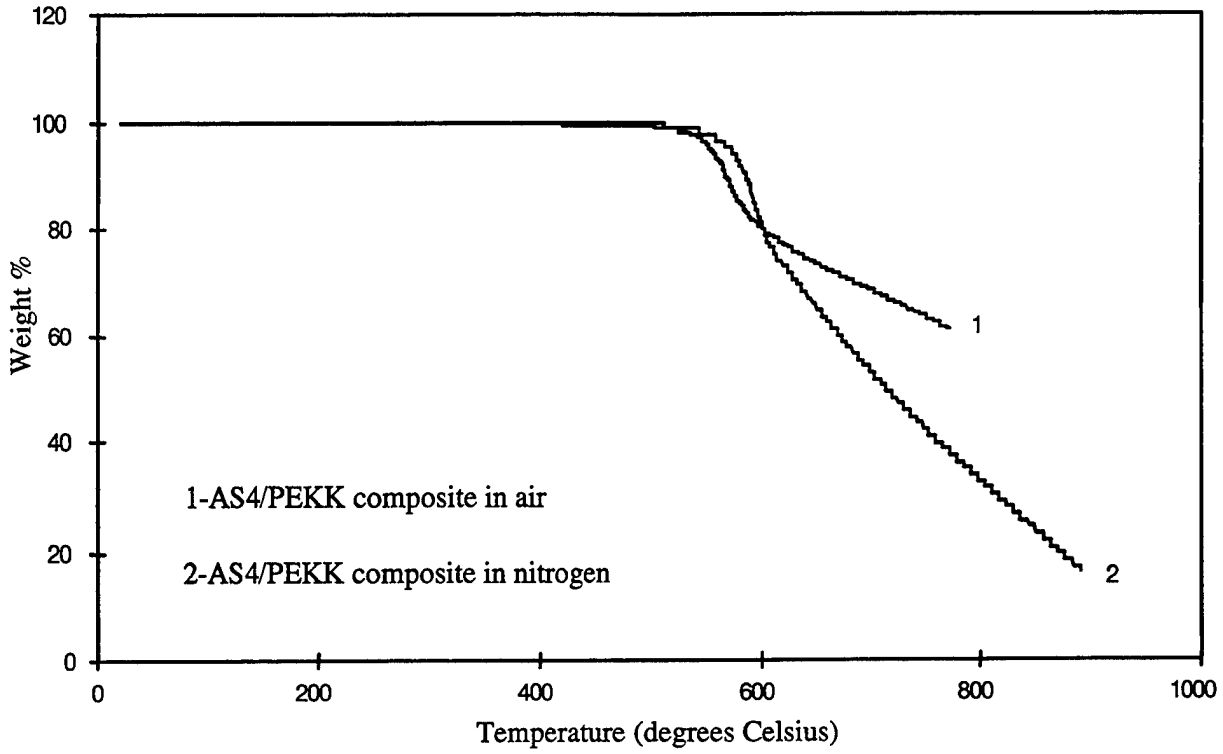

Figure A-2. TGA Diagram of AS4/PEKK Composite.

Figure A-3 shows the actual temperature profiles of the tow surface at 20-mm/s deposition rate for various torch temperature settings, recorded by an AGEMA Thermovision 900 thermal camera. Figure A-4 shows the model-predicted temperature profiles for various torch temperatures. Model-predicted and actual maximum temperatures are tabulated in Table A-1 and plotted vs. torch temperature settings in Figure A-5. Comparing model-predicted and actual maximum surface temperatures, it can be seen that our process simulation overestimates the maximum temperature. The maximum recorded temperature was 454 °C, which is far below the temperature at which weight loss is a dominant degradation mechanism.

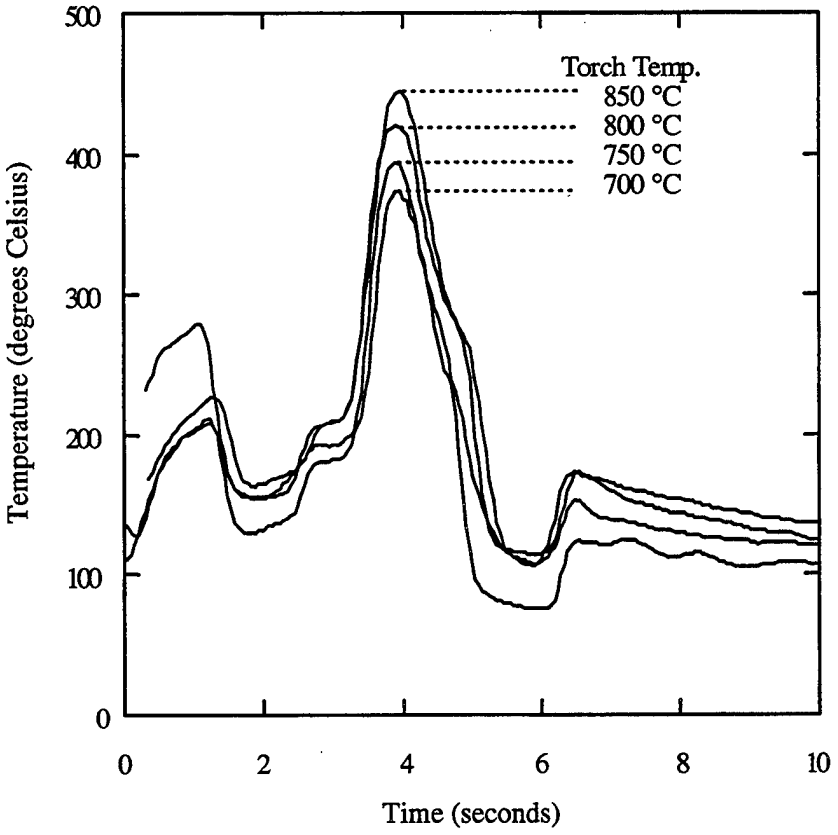

Figure A-3. Infrared Camera Readings of Surface Temperature at 20-mm/s Deposition Rate.

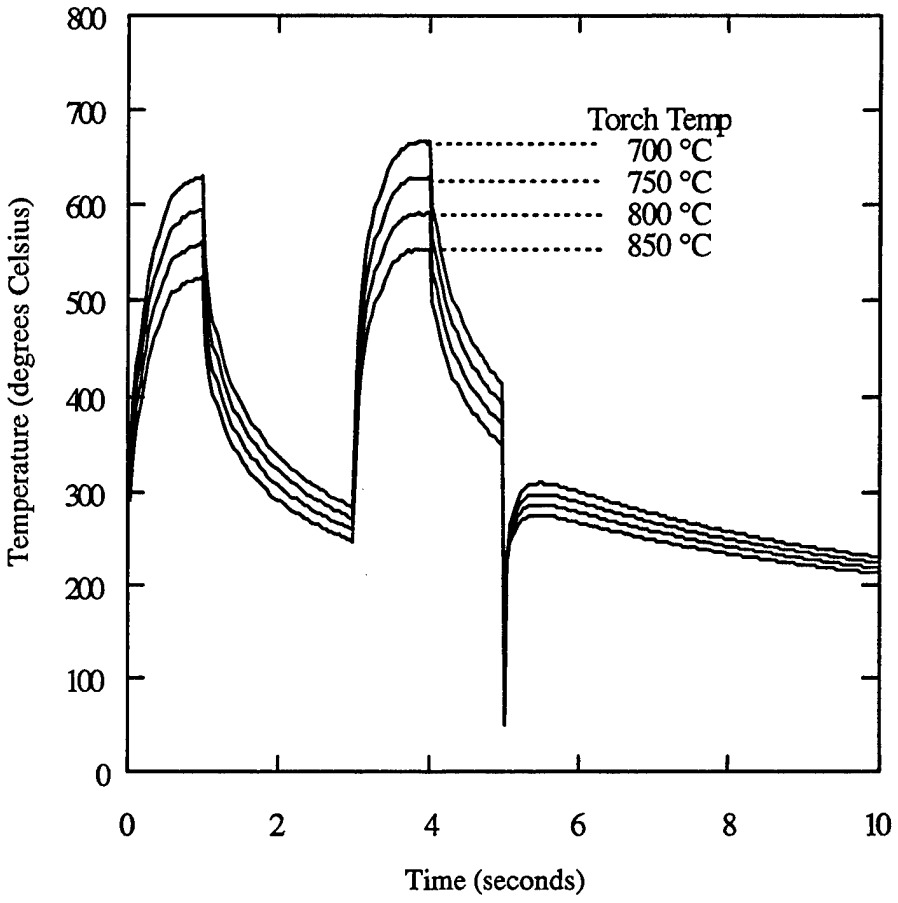

Figure A-4. Model Predictions for Surface Temperature at 20-mm/s Deposition Rate.

Table A-1. Maximum Surface Temperatures for Various Torch Temperature Settings at 20-mm/s Deposition Rate

Torch Temperature Setting (degrees Celsius)	Infrared Camera Reading (degrees Celsius)	Model Prediction (degrees Celsius)
700.00	373.3	553.4
750.00	394.2	591.5
800.00	419.8	629.6
850.00	445.4	667.7

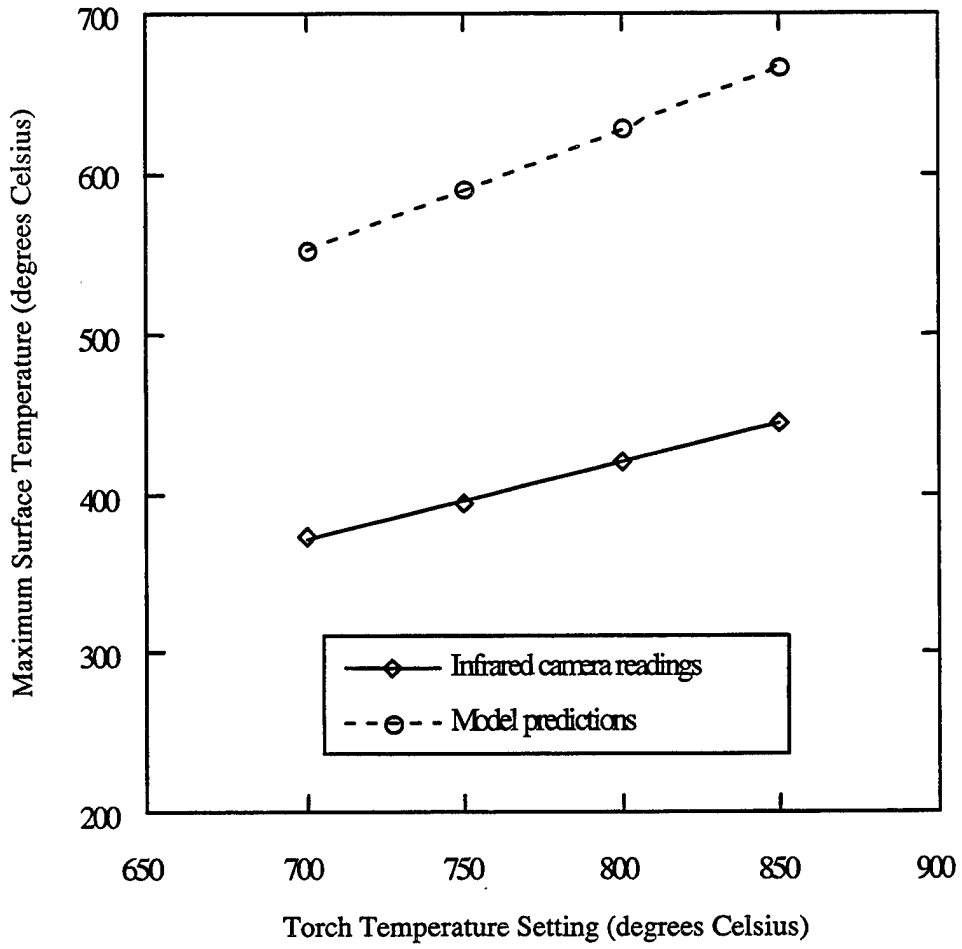

Figure A-5. Maximum Surface Temperature for Various Torch Temperature Settings at 20-mm/s Deposition Rate.

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Appendix B:

Fourier Transform Infrared Spectroscopic Study of Thermal Degradation of AS4/Polyetherketoneketone (PEKK) Composite

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Diffused reflectance Fourier-transform infrared spectroscopy (DRFTIR) has been used by Cole and Casella[1] to study the thermal degradation in polyetheretherketone (PEEK) carbon composites. Here, the same method is adopted to study the thermal degradation of AS4/polyetherketoneketone (PEKK) composite during the tow placement process. Although diffused reflection spectra provide more limited information than a.t.r. spectra, they are, nevertheless, useful for following changes that occur upon thermal degradation at the surface of the composite.

Samples were prepared by processing tows of AS4/PEKK composite on a fiber-placement robot. However, for a single pass, no change in the diffuse reflectance spectra was observed and, therefore, the tow was forced to degrade by multiple passes of the fiber-placement head. Deposition velocity was held constant at 20 mm/s, and the torch temperature was varied to be 700, 800, and 900 °C. DRFTIR spectra were measured on a Nicolet 170SX instrument equipped with a "diffuse reflectance" fixture. The sample was mounted with its surface in a horizontal plane and oriented with the fiber direction parallel to the beam direction of the instrument. For each spectrum, 512 scans were accumulated at a resolution of 4 cm^{-1}. For qualitative treatment, the spectra were converted to Kubelka-Munk units. They were then baseline-corrected to remove the underlying carbon absorption. A baseline was drawn through the 1,750, 1,520, 1,460, 1,360, 1,040, 820, and 560 cm^{-1} valleys. As the polymer degradation increased upon multiple passes, the overall intensity of the spectra decreased; hence, for qualitative comparisons, each spectra was integrated and scaled with respect to the reference spectrum of the undegraded tow.

Diffuse reflectance spectra for 700, 800, and 900 °C torch temperatures corrected for baseline and overall intensity are shown in Figures B-1, B-2, and B-3. The spectra are shifted vertically for better visualization. As can be seen from the figures, a new species in carbonyl

[1] Cole, K. C., and I. G. Casella. "Fourier Transform Infra-Red Spectroscopic Study of Thermal Degradation in Poly(Ether-Ether Ketone)-Carbon Composites." *Polymer*, vol. 34, no. 4, pp. 740–745, 1993.

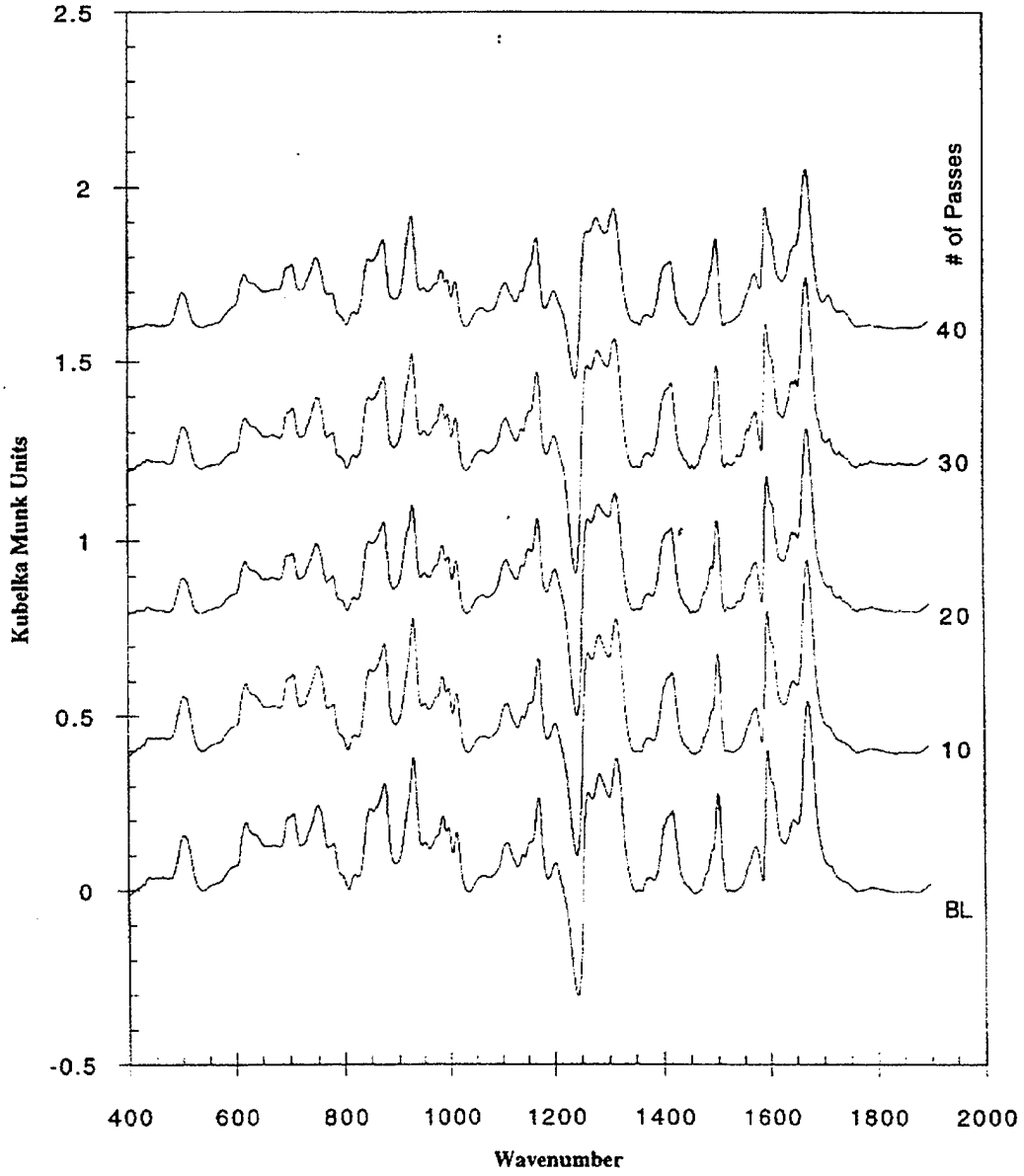

Figure B-1. Diffuse Reflectance Spectra for 700 °C Torch Temperature.

region (1,716 cm^{-1}) grows with increasing number of passes, and the rate of growth of this peak is higher for higher torch temperatures. Another change occurring in the spectra is the reduction in the height of 500 cm^{-1} peak. The origins of these species are uncertain. Cole and Casella[1]

[1]Cole, K. C., and I. G. Casella. "Fourier Transform Infra-Red Spectroscopic Study of Thermal Degradation in Poly(Ether-Ether Ketone)-Carbon Composites." *Polymer*, vol. 34, no. 4, pp. 740–745, 1993.

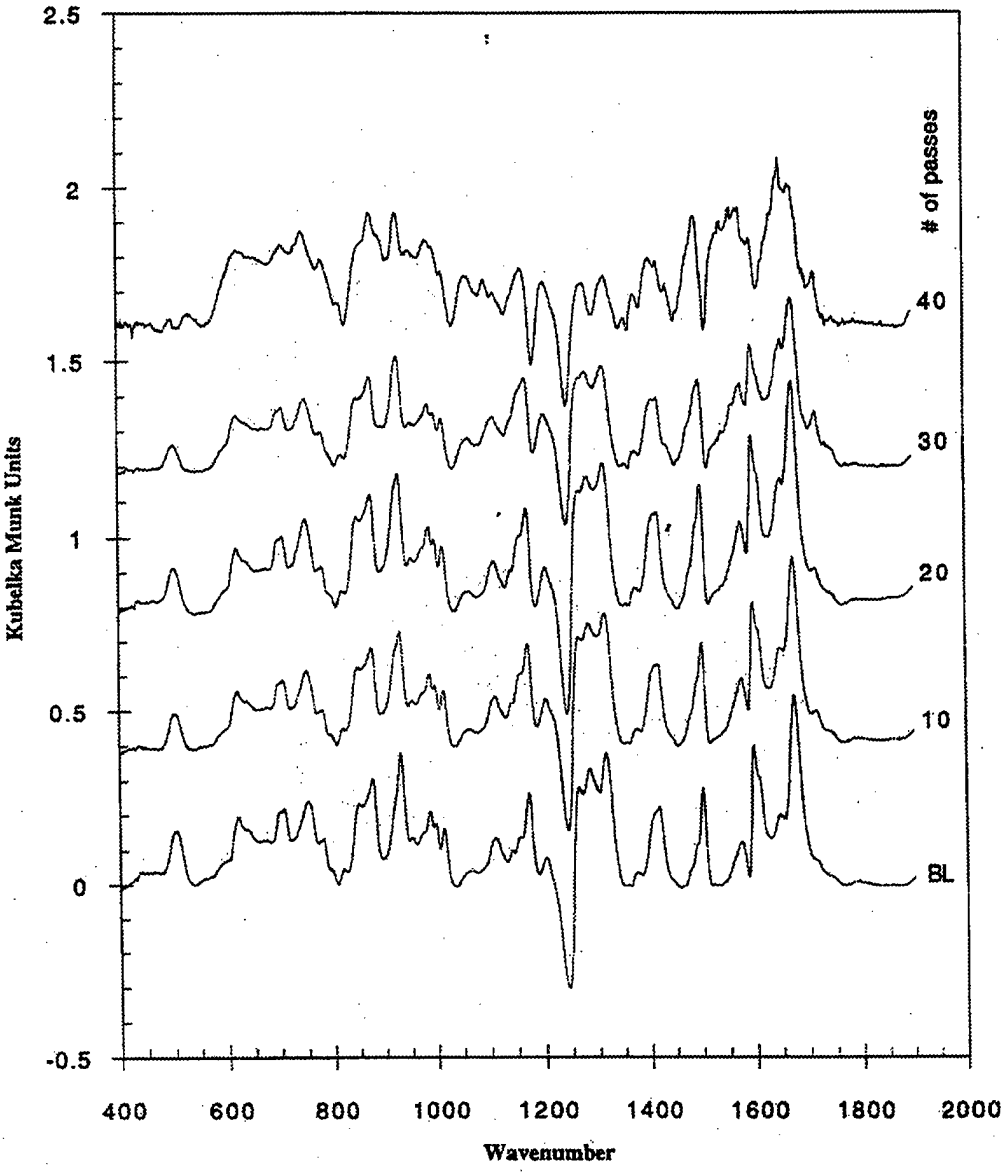

Figure B-2. Diffuse Reflectance Spectra for 800 °C Torch Temperature.

found a similar growing peak at 1,711 cm^{-1} for degradation of PEEK and proposed that this peak may result from cyclization of a diradical. Cyclization may reduce chain mobility and, hence, the ability to bond the polymer.

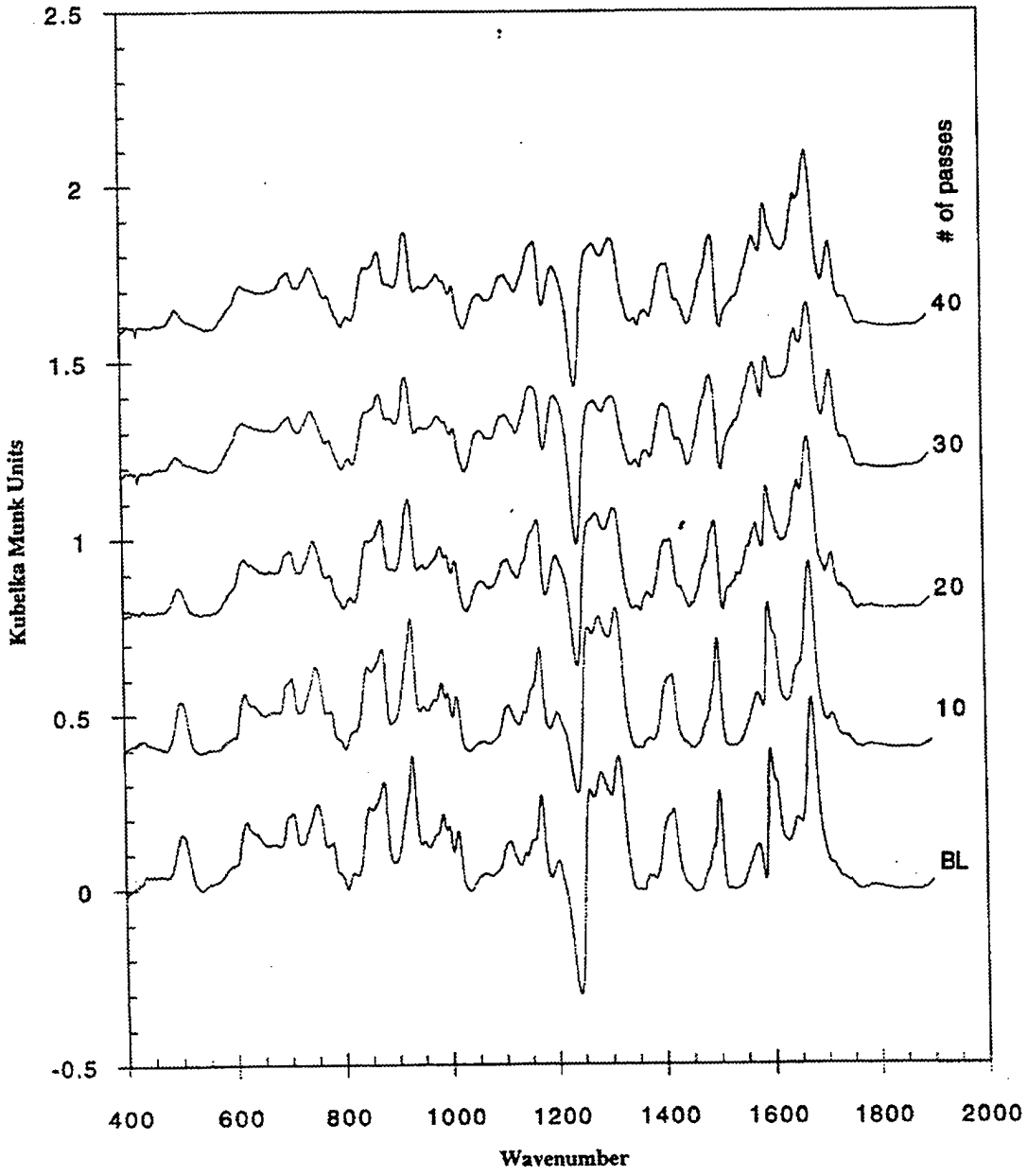

Figure B-3. Diffuse Reflectance Spectra for 900 °C Torch Temperature.

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Effects of elevated temperature exposure during thermoplastic fiber placement on bonding and consolidation are investigated experimentally for AS4/polyetherketoneketone (PEKK) composite. Coupons of 24 layers are consolidated on the University of Delaware Center for Composite Materials (UD-CCM) fiber-placement robot at deposition rates of 20 and 40 mm/s over a range of process temperatures (700–900 °C, with 50 °C increments). The main torch and preheater distances and the compaction force are held constant for all coupons. Two competing mechanisms governing strength buildup are considered: (1) polymer bonding and (2) degradation. Coupons are sectioned, and one-half of each coupon is reconsolidated in a hot press at conventional processing conditions (i.e., 30 min at 370 °C and 0.70-MPa pressure) in order to remove any effect of poor consolidation on strength. Void content of the robot-consolidated panels is measured. Short-beam shear (SBS) tests are performed on the specimens cut from each coupon. Strength and void-content measurements for robot-consolidated panels are presented to illustrate the effect of processing parameters on product quality. Results of the SBS strength tests performed on reconsolidated coupons indicate that there is significant decrease in the strength of coupons consolidated at 20-mm/s deposition rate and high torch temperatures, possibly due to polymer degradation, whereas approximately the same value of reconsolidated strength is measured for the 40-mm/s deposition rate, suggesting that polymer degradation is insignificant at that rate. Effects of void content on SBS tests are discussed.

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