JOURNAL OF MATERIALS SCIENCE 37 (2002) 2693–2698

Determination of moisture effects on impact properties of composite materials

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Many applications of structural materials involving composites include impact or dynamic loading in a humid environment. Composite materials are known to degrade when subjected to humid conditions, and therefore the humidity confounds the difficulty of determining the high strain rate behavior of composites. Several researchers have found that water absorption by composites causes degradation of matrix dominated quasi-static properties. However, very little is known of the effect of absorbed moisture on the high strain rate properties of polymer matrix composites, that are useful in the automotive, aerospace, and naval applications of composite structures. A Split-Hopkinson Pressure Bar facility is used herein to study the effect of absorbed moisture in high strain rate tests (200–1200/s) of a unidirectional IM7/8551-7 graphite/epoxy composite. The study includes dry, medium, and saturated moisture conditions. The tests show significant variation of high strain rate properties from static properties, and the reasons are identified. In addition, a better understanding of the effect of the matrix and fiber/matrix interface on the high strain rate properties of composites is achieved.

1. Introduction

Polymer matrix composites are presently used in many fields due to their excellent mechanical properties such as high specific strength and stiffness. The fact that mechanical properties vary with strain rate makes it necessary to know the properties of the composites at the strain rates present in dynamic applications in order to achieve a more accurate design or analysis. The Split-Hopkinson Pressure Bar (SHPB) has been accepted as the test method for high strain rates since Kolsky [1] first presented the test methodology for compression in 1949. The method was developed based on wave propagation theory in elastic bars and the interaction between a stress wave and a short specimen. After Kolsky's introduction, the method was expanded to tensile testing by Nicholas [2] and shear testing by Duffy *et al.* [3]. Zukas [4] has shown that for many metals, the mechanical properties vary significantly with strain rate. This new data obtained in this investigation will add to what is now available for other material systems obtained using the same high strain rate facility that include glass/epoxy, glass/polyester, graphite/epoxy, carbon/metal matrix, and carbon/ceramic matrix, and the most recent papers regarding these materials are given in the References [5–7]. There are other several researchers that have published high-strain-rate experimental data; however, direct comparison of results would be misleading due to lack of standardized test specimen geometry and dimensions.

The effect of moisture on the static properties of composites has been studied for a while and there are numerous reports have been published [8–11]. No study has been found in the literature correlating the effect of moisture with the high strain rate properties of polymer matrix composite materials. This study is undertaken to investigate the effect of moisture on the dynamic properties of polymer based composites.

2. Specimens and experimental methods

2.1. Specimen preparation

The IM7/8551-7 carbon/epoxy specimens were fabricated from continuous fiber unidirectional preimpregnated tape. The tape was 16 in. wide and was cut into 48 in. long strips. A 75 ply unidirectional laminate was fabricated by hand lay-up to achieve the desired thickness after processing. The laminate was processed in a computer-controlled autoclave. The finished laminate was then sectioned using a water-cooled masonry saw equipped with a diamond grit blade, which does not produce delamination and rough surfaces. Cylindrical specimens were fabricated with an aspect ratio (length to diameter ratio) of 1.5 from the sectioned pieces. The diameter and the length of the samples were approximately 0.635 cm (0.25 in) and 0.953 cm (0.375 in)

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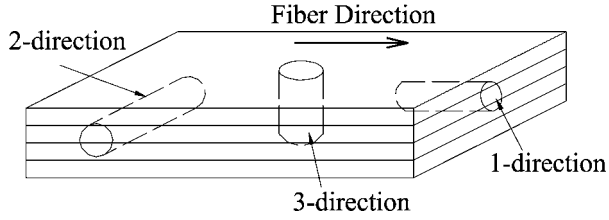

Figure 1 Orientations of fabricated specimen.

respectively. Fig. 1 shows the three possible orientations of the test specimens.

Recently, a high strain rate study was conducted using the same type of graphite/epoxy composite that examined various aspect ratios ranging from 0.5 to 2 with increments of 0.25 proved to be essential for this study [7]. The aspect ratio study included 84 specimens. The diameter of the specimens remained fixed at 0.663 cm (0.261 in), set by the diameter of the core drill, while the length varied from 0.323 cm (0.127 in) to 1.3 cm (0.512 in). The aspect ratio study was conducted on the 1 and 2 direction specimens, the specimen orientations are as shown in Fig. 1. Tests were not performed in the thickness direction (3-direction), as the length of the specimen could not be varied over the desired range. However, another study of the same IM7/8551-7 graphite epoxy composite shows that the 2- and 3-directions for an aspect ration of 1.5 have similar properties, and therefore 2-direction testing would give adequate insight into the material properties in the 3-direction in a dry condition [11]. Within the range of variables examined, statistically no significant differences could be found in the material properties and this provided the basis for making cylindrical samples with the specific dimensions used in this study.

The cylindrical samples for this moisture effect investigation were immersed in distilled water at room temperature and 65°C after fabrication. The weight of five specimens in 1, 2, and 3 directions, Fig. 1, was measured periodically to determine the amount of moisture gained. A scale (a Mettler H80), which can accurately measure up to 0.0001 grams, was used to measure the weight of the samples.

2.2. Split-Hopkinson pressure bar

The Split-Hopkinson Pressure Bar facility used in this study is shown in Fig. 2. To begin a test, a specimen is placed between two long, 1.9 cm (3/4 in) diameter Inconel bars, the 'incident bar' and the 'transmitter bar', which are supported by Teflon® bearings. Each end of the specimen is lubricated. Impact is initiated by releasing nitrogen gas from a pressurized chamber. The gas propels a striker bar (supported by and riding on Teflon® rings) through a guiding barrel, at the end of which it strikes the incident bar. The velocity of the striker bar just prior to impact is measured using two infrared beams at the end of the barrel. At impact, the incident bar receives an elastic compressive stress wave with specific wave velocity and a wave shape that is a function of time. When the wave reaches the incident bar/specimen interface, a portion of the incident wave is reflected back into the incident bar as a tensile wave. The remaining portion of the wave is transmitted into the specimen as a compressive wave. The wave transmitted into the specimen travels through the specimen and reaches the specimen/transmitter bar interface, where a portion of the wave is reflected back into the specimen as a tensile wave. The rest is transmitted into the transmitter bar as again a compressive wave. The transmitter bar is also displaced along its length axis, and comes to rest when it reaches a dashpot.

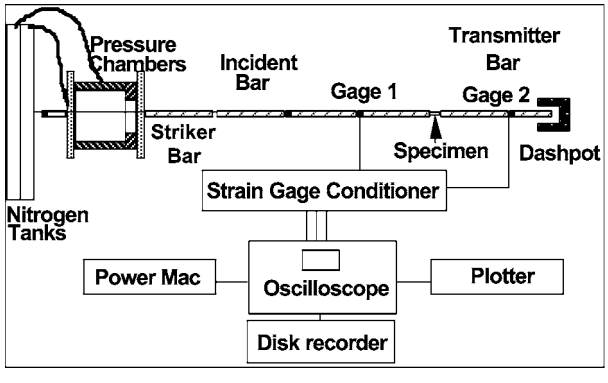

Figure 2 The Split Hopkinson pressure bar apparatus.

The initial stress wave in the specimen undergoes numerous internal reflections during the test because the specimen length is short, and therefore, the wave reflections in the specimen are not shown in the Lagrangian diagram of the SHPB. The stress distribution in the specimen is assumed to be uniform due to the numerous reflections. In addition it is assumed that the stress waves undergo minimal dispersion, that the bars remain elastic, and that the ends of both the incident and transmitter bars in contact with the specimen remain flat. The Lagrangian diagram shows that there is a characteristic time window corresponding to the duration of the stress wave [11]. The wave pulse time window of the SHPB used is approximately 290 microseconds. This means that the specimen must fail within 290 microseconds after the initial portion of the wave reaches the specimen for failure to be accurately characterized. This has not been a problem with the relatively brittle composites tested to date.

Strain gages, marked Gage 1 and Gage 2 in Fig. 2, are mounted on both bars equidistant from the specimen interfaces and measure the strains in the bars due to the stress wave propagation. The gages are connected to a Fluke PM3394A recording oscilloscope. The strain gage mounted on the incident bar also acts as trigger for the oscilloscope. The oscilloscope records the strain gage's output as a voltage versus time graph. Using this data, along with the speed of the striker bar and the physical dimensions of the bars and specimens, stress versus strain curves can be generated for different strain rates.

3. Results and discussions

3.1. Moisture absorption

Fig. 3 shows the moisture absorption of the IM7/8551-7 graphite/epoxy composite samples. The moisture absorption is monitored by periodically removing the specimens from the bath tanks and keeping them outside on clean surfaces for about 1 minute where excess water on the specimen's surface evaporates.

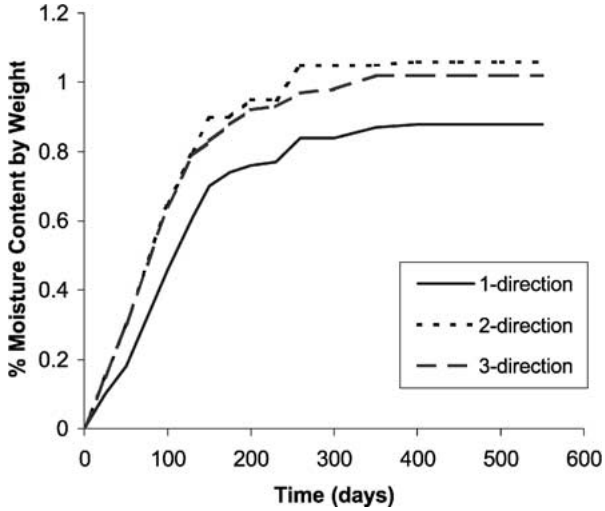

Figure 3 Moisture absorption of cylindrical IM7/8851-7 carbon/epoxy composite specimens immersed in a distilled water bath at RT.

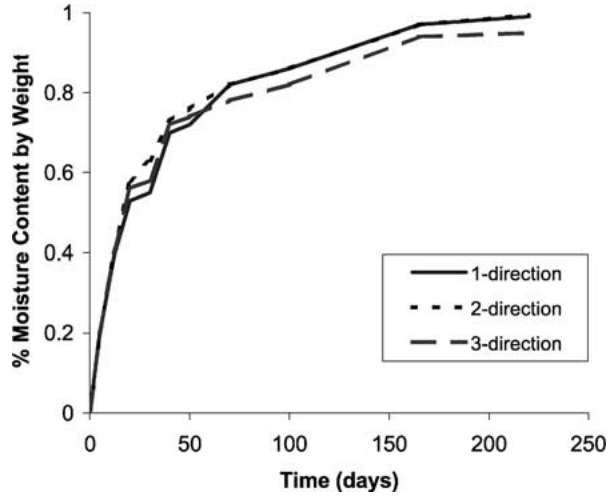

Figure 4 Moisture absorption of cylindrical IM7/8851-7 carbon/epoxy composite specimens immersed in a distilled water bath at 65°C.

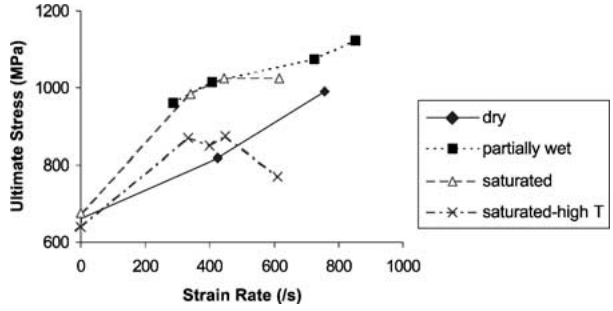

Figure 5 Ultimate stress vs. strain rate for different moisture conditions in the fiber direction.

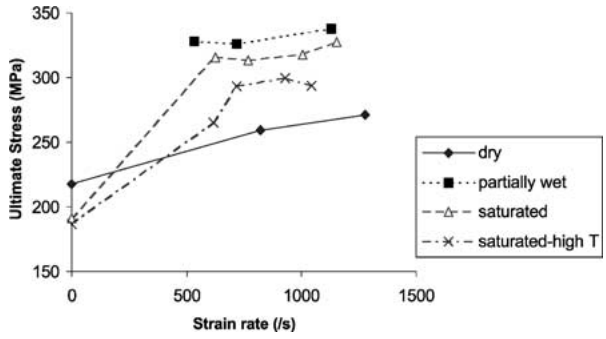

Figure 6 Ultimate stress vs. strain rate for different moisture conditions in the transverse direction.

Dynamic tests were performed using the SHPB after 64 days of immersion at room temperature in distilled water, which was equivalent to 0.25, 0.38 and 0.38% moisture in the 1, 2, and 3 direction samples respectively, and after 559 days which was equivalent to 0.89, 1.06, and 1.02% moisture intake in the 1, 2, and 3 direction samples respectively. In Fig. 3, the moisture absorption rate for the 1-direction samples is a little slower than the rate for the other directions. This might be caused by the presence of the fibers around the surface of the cylinder and limiting the diffusion of water into the specimens. In addition, the moisture intake at full saturation for the 1-direction samples is clearly lower than the others. Theoretically, all the samples should have the same absorbed moisture percentage at full saturation, however, the differences of final moisture intake are considered to be insignificant for this investigation.

Dynamic tests were also performed on the SHPB after more than seven months immersion in 65°C bath. A higher temperature (65°C) bath was used to shorten the time the samples would take to reach full saturation, because it took a long time for the samples to attain full saturation at room temperature. Fig. 4 shows the moisture absorption of the IM7/8551-7 graphite/epoxy composite samples in a 65°C distilled water bath. In the case of high temperature immersion, the saturation moisture level seems to be similar for all samples in the three different directions. In all weight measurements of the samples, there was about a maximum of ±0.02% error due to the accuracy of the machine.

3.2. High strain rate testing

The results of high strain rate tests of dry specimens obtained by the authors from a previous experimental study [7] are used for comparison with the moisture containing specimens tested in this study. The first set of wet-high strain rate experiments was performed after 64 days of immersion on the samples that were kept in water at room temperature. Three replicates were tested, i.e., three samples were used to get the average dynamic property for specific strain rates and directions. Further tests were performed after full saturation (almost 19 months) on the wet samples kept at room temperature. Because, it took a long time for the samples to attain full saturation at room temperature, as a third case, a higher temperature (65°C) bath was used to shorten the time the samples would take to reach full saturation. Higher temperature bath specimens took nearly 7 months to attain full saturation and were tested.

The SHPB test results for the moisture-impact study are shown in Figs 5–7. The strain rates vary between 200–1200/s, corresponding to chamber pressure of 30–100 psi. The average values with standard deviation and coefficient of variation are given in Tables I–III. Moisture is usually taken to have a deteriorating effect on composite material properties as mentioned previously. At high strain rates, however, the strengths of

TABLE I Summary of results of partially wet specimens

Type	Statistical propoerty	Strain rate (m/m)	Stress (MPa)
	Average	286.6	139.23E+03
	Standard deviation	23.40	17.4E+03
	Variance	8.16%	12.51%
	Average	407.3	147.21E+03
	Standard deviation	66.44	9.29E+03
pw-1	Variance	16.31%	6.31%
direction	Average	723.9	155.92E+03
	Standard deviation	32.23	3.89E+03
	Variance	4.45%	2.49%
	Average	851.4	162.80E+03
	Standard deviation	21.39	4.98E+03
	Variance	2.51%	3.06%
	Average	533.5	47.6E+03
	Standard deviation	43.78	.72E+03
	Variance	8.21%	1.51%
	Average	718.2	47.3E+03
pw-2	Standard deviation	32.60	4.09E+03
direction	Variance	4.54%	8.66%
	Average	1128.5	49.0E+03
	Standard deviation	51.59	1.06E+03
	Variance	4.58%	2.15%
	Average	495.0	47.3E+03
	Standard deviation	56.15	3.86E+03
	Variance	11.34%	8.17%
	Average	586.6	49.3E+03
	Standard deviation	8.94	2.54E+03
pw-3	Variance	1.52%	5.16%
direction	Average	976.8	49.5E+03
	Standard deviation	10.52	3.31E+03
	Variance	1.08%	6.68%
	Average	1181.0	47.0E+03
	Standard deviation	40.95	2.91E+03
	Variance	3.47%	6.19%

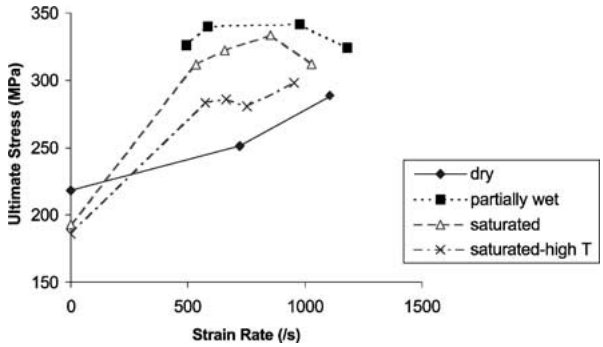

Figure 7 Ultimate stress vs. strain rate for different moisture conditions in the thickness direction.

the IM7/8551-7 graphite/epoxy composite kept in water have in general increased due to the moisture intake when compared to quasi-static properties. Detailed discussion of the change in properties is warranted because of the unexpected nature of the results.

Quasi-static and dynamic tests were performed to understand the effect of moisture on the IM7/8551-7 graphite/epoxy composite. The specimens were of the same size and taken from the same batch of samples. The high strain rate results are definitely the reverse of the quasi-static results in most cases as shown in Figs 5–7. Fig. 5 shows the dynamic ultimate stress vs. strain rate characteristics for different moisture conditions for specimens tested in the 1-direction. In this figure, the quasi-static values of the dry specimens are

TABLE II Summary of results of fully wet specimens (room temperature bath)

Type	Statistical propoerty	Strain rate (m/m)	Stress (MPa)
	Average	339.7	142688.0
	Standard deviation	34.53	12286.0
	Variance	10.17%	8.61%
rtw-1	Average	444.7	148617.0
direction	Standard deviation	24.58	5498.3
	Variance	5.53%	3.70%
	Average	615.7	148617.0
	Standard deviation	56.58	1273.1
	Variance	9.19%	0.86%
	Average	625.7	45766.0
	Standard deviation	20.21	3201.5
	Variance	3.23%	7.00%
	Average	768.3	45423.0
	Standard deviation	13.05	2983.8
rtw-2	Variance	1.70%	6.57%
direction	Average	1005.0	46109.0
	Standard deviation	12.53	1442.8
	Variance	1.25%	3.13%
	Average	1153.3	47530.0
	Standard deviation	22.30	1267.4
	Variance	1.93%	2.67%
	Average	535.0	45202.5
	Standard deviation	12.73	1351.3
	Variance	2.38%	2.99%
	Average	657.3	46746.0
	Standard deviation	4.04	509.2
rtw-3	Variance	0.61%	1.09%
direction	Average	855.0	48363.0
	Standard deviation	4.36	1018.4
	Variance	0.51%	2.11%
	Average	1029.0	45276.0
	Standard deviation	10.44	640.8
	Variance	1.01%	1.42%

approximately the same as the saturated specimens that were immersed in room temperature bath, and slightly lower for the saturated samples that were immersed in 65°C bath. The high strain rate values demonstrate that the wet samples exhibit higher strength values, 10–25% higher compared to the dry specimens. The saturated samples immersed in 65°C bath displayed an overall increase, but exhibit slightly lower strength than the dry specimens at a strain rate value of 611/s.

Much more significant differences between quasi-static and high strain rate properties are clearly depicted in the other directions, as shown in Figs 6 and 7. In the 2-direction and 3-direction tested specimen, the quasi-static values clearly demonstrate the deteriorating effect of moisture on composites. There are up to 12% and 14% decrease in the quasi-static strength values in the 2 or 3-direction samples immersed in room temperature and 65°C baths, respectively. The dynamic strength however shows increase of up to 30% and 40% for partially wet samples immersed at room temperature bath in the 2 and 3 directions, respectively. For fully saturated samples, the increases are up to 25% and 30% in the 2 and 3 directions, respectively. For fully saturated samples immersed at 65°C baths, the 2 and 3-direction strength properties exhibit an increase of up to 15 and 10% compared to the dry samples at high strain rates, respectively.

The overall increase in the material property for the samples kept at room temperature bath can be attributed

TABLE III Summary of results of fully wet specimens (high temperature bath)

Type	Statistical propoerty	Strain rate (m/m)	Stress (MPa)
	Average	333.6	126214.2
	Standard deviation	16.46	10083.5
	Variance	4.93%	7.99%
	Average	399.4	123303.6
	Standard deviation	32.71	5570.9
htw-1	Variance	8.19%	4.52%
direction	Average	448.0	126714.0
	Standard deviation	26.27	3887.9
	Variance	5.86%	3.07%
	Average	611.2	111631.8
	Standard deviation	72.43	5373.6
	Variance	11.85%	4.81%
	Average	617.2	38484.6
	Standard deviation	31.76	2436.4
	Variance	5.15%	6.33%
	Average	718.4	42541.8
	Standard deviation	5.50	1540.3
htw-2	Variance	0.77%	3.62%
direction	Average	929.3	43414.0
	Standard deviation	21.36	1472.4
	Variance	2.30%	3.39%
	Average	1043.0	42556.5
	Standard deviation	83.44	1767.1
	Variance	8.00%	4.15%
	Average	574.7	41111.0
	Standard deviation	16.07	978.8
	Variance	2.80%	2.38%
	Average	663.3	41454.0
	Standard deviation	13.65	388.9
htw-3	Variance	2.06%	0.94%
direction	Average	753.3	40719.0
	Standard deviation	147.89	2058.0
	Variance	19.63%	5.05%
	Average	954.5	43218.0
	Standard deviation	64.35	0.0
	Variance	6.74%	0.00%

to the plasticization of the matrix and it can be concluded that the matrix plays a major role in the high strain rate properties of the composite. Clearly if the matrix is not a major factor, the moisture tests should not have resulted in higher values than the dry ones. There have been arguments that the fibers are more strain rate sensitive than the matrix [12]. However, the researchers have used off-axis studies and came to such a conclusion because of the decrease in the strain rate sensitivity of the composite as the off-axis angle increases. This would be erroneous because the researchers neglected the importance of the change in the mode of failure at different off-axis angle [13].

In general, fibers do not absorb moisture and therefore in this investigation any change in material property due to moisture would only occur in the matrix. Since, the properties in the longitudinal direction are dominated by the fibers, the variation in properties between wet and dry samples, and between quasi-static and dynamic are not significant compared to the properties in the transverse and thickness directions. The matrix is known to determine the transverse and thickness properties. Since the matrix absorbs all the moisture in our experiment, this would explain the significant differences between quasi-static and high strain rate properties in the 2-direction and 3-direction.

It can also be argued that the same increase in properties should always be true with the samples that were kept at high temperature bath, but the experiments demonstrate both increase and decrease of material properties. Plasticization of the matrix still occurs with moisture intake, however, the rate of moisture absorption is much faster and the deterioration of the fiber/matrix interface is more severe for the high temperature saturated samples. In all the tests, plasticization leads to an effective increase in the dynamic strength of the composite material at all saturation level regardless of the temperature of the water bath. However, the effect of the fiber/matrix interfacial strength needs to be investigated. In order to have a clear understanding, scanning electron microscopy of the fracture surface of the dry, partially saturated, fully saturated, and fully saturated at high temperature bath specimens was conducted after the high strain rate tests. Some of these micrographs are shown in Figs 8–11. The pictures clearly identify the deterioration of the fiber/matrix interface and this deterioration diminishes the high strain rate properties of the high temperature saturated samples compared to the dry specimens. In addition, the micrographs demonstrate increasing damage of the interface corresponding to the weakening of the high strain rate properties. The dry specimens displayed resin adhering to the fibers indicating substantial fiber/matrix adhesion, Fig. 8. This happens to be true in both quasi-static and high strain rates tested specimens. The partially wet specimens, which were tested only in high strain rate, exhibited only slightly less resin adhering to the fibers than the dry case, Fig. 9. However, the fully saturated wet specimens show significant reduction in the adhering resin, Fig. 10. The case for fully high temperature bath saturated samples is much worse, with almost all fibers exhibiting clean surfaces being observed under SEM investigation, Fig. 11. It is understood that the fiber-matrix interface is a major component of a composite material system. This importance came about because load is transferred from the fibers to the matrix or vice versa through the interface. Therefore, Figs 8–11 demonstrate to what extent the interface can be crucial in the transfer of load without sustaining significant damage. A conclusion that can be reached from these observations is that the fiber/matrix interface is a major factor in high strain rate testing of composite materials.

Figure 8 Scanning electron micrograph of a dry specimen.

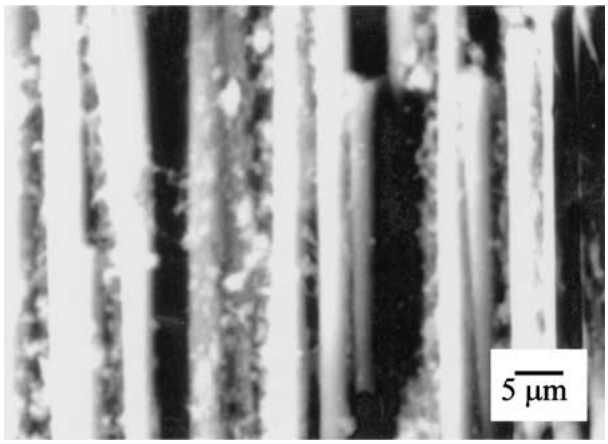

Figure 9 Scanning electron micrograph of a partially wet specimen.

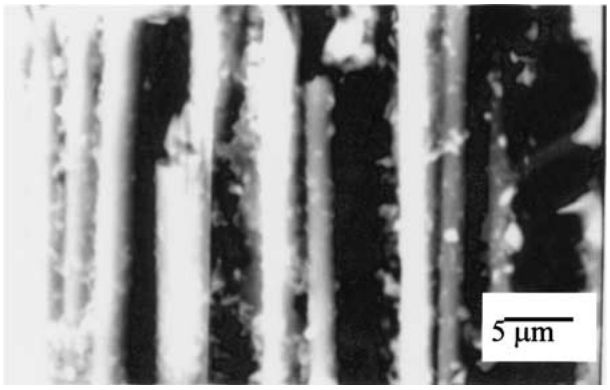

Figure 10 Scanning electron micrograph of a saturated specimen.

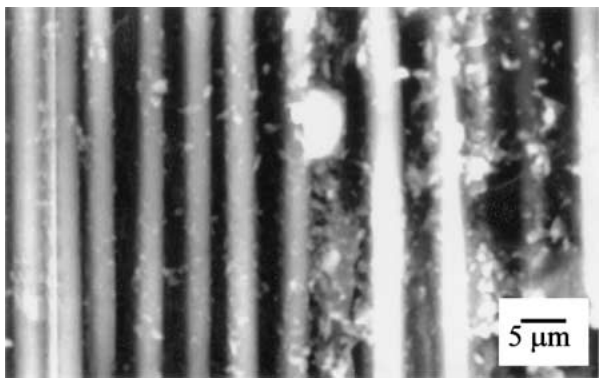

Figure 11 Scanning electron micrograph of a saturated (high temperature) specimen.

Therefore, two competing factors, the plasticization of the matrix and the degradation of the fiber/matrix interface, control the high strain rate behavior of the specimens. The above observations therefore can be explained by understanding whether the effect of plasticization of the matrix or the degradation of the fiber/matrix interface is more pronounced. In this study the effect of plasticization is much more pronounced than the effect of fiber/matrix interfacial degradation, leading to an increased strength of the composite. It can be concluded that in initial stages of moisture absorption, the effect of interfacial strength degradation is neglected. However, with increase in moisture absorption this effect increases, lowering the gain obtained from plasticization of the matrix. Degradation of the interface becomes even more prominent at increased temperature.

4. Conclusions

This study is unique because it is the first investigation of the effect of moisture on high strain rate properties of composites and significant results were achieved. The conclusions from this investigation can be summarized as follows:

1. The plasticization of the matrix clearly is the dominant factor in high strain rate properties when materials are partially or fully wet—immersion at room temperature bath.
2. The matrix is more strain rate sensitive and therefore plays a more important role than the fiber in determination of the high strain rate material behavior of polymer matrix composites.
3. Degradation of the fiber/matrix interface leads to the decrease of the enhancing effect of plasticization of the matrix.
4. An overall increase in material properties occurs due to absorption of moisture, except in high temperature bath caused due to the extreme degradation of the interface.
5. There is an amount of absorbed moisture that gives optimum material properties that corresponds to the highest plasticization of the matrix with minimal degradation of the fiber/matrix interface.

Acknowledgements

This research is sponsored by ONR Grant N000 14-93-1-1014, with Dr. Y. D. S. Rajapakse as Project officer.

References

1. H. KOLSKY, *Proc. Phys. Soc.* B **62** (1949) 676.
2. T. NICHOLAS, *Exp. Mech.* **21** (1971) 177.
3. J. DUFFY, J. D. CAMPBELL and R. H. HAWLEY, *J. Appl. Mech.* **38** (1971) 83.
4. J. ZUKAS (ed.), "Impact Dynamics" (John Wiley and Sons, New York, 1982).
5. B. M. POWERS, J. R. VINSON, I. W. HALL and R. F. HUBBARD, *AIAA Journal* **35** (1997) 553.
6. E. PREISSNER, E. WOLDESENBET and J. R. VINSON, in Proceedings of 38th AIAA/ASME/ASCE/AHS/ASC Structures, Structural Dynamics and Materials Conference, 1997, Kissimee, FL, p. 935.
7. E. WOLDESENBET and J. R. VINSON, *AIAA J.* **37** (1999) 1102.
8. A. J. BARKER and V. BALASUNDARAM, *Composites* **18** (1987) 217.
9. C. H. SHEN and G. S. SPRINGER, in "Environmental Effects on Composite Materials" (Technomic Pub. Co., Lancaster, PA, 1981) p. 79.
10. E. M. SILVERMAN, C. R. WIACEK and R. A. GRIESE, in "Composite Materials: Testing and Design, ASTM STP 1120" (American Society for Testing and Materials, West Conshohocken, PA, 1992) Vol. 10, p. 118.
11. E. WOLDESENBET, "High Strain Rate Properties of Composites", Ph.D. Dissertation (Department of Mechanical Engineering, University of Delaware, 1998) p. 111.
12. G. H. STAAB and A. GILAT, *J. Composite Mater.* **29** (1995) 1308.
13. E. WOLDESENBET and J. R. VINSON, *ibid.* **35**(6) (2001) 509.

Received 30 July 2001
and accepted 14 March 2002