JOURNAL OF MATERIALS SCIENCE 20 (1985) 4527–4531

An evaluation of acoustic emission from fibre-reinforced composites

Part 2 *The application of acoustic emission techniques to aramid fibre-reinforced model composites*

I. NARISAWA, T. OBA
Department of Polymer Materials Engineering, Faculty of Engineering,
Yamagata University, Yonezawa 922, Japan

An analysis of acoustic emission (AE) from model composites consisting of a single aramid fibre and different epoxy matrix systems has been carried out to identify the source of acoustic emission. The AE activity was observed in a narrow range of strain when fibre fracture occurred, whereas in a relatively wide range of strain, debonding occurred at the fibre–matrix interface. Ion-etched fibres showed a good adhesion of the fibres to the matrix so as to produce fibre fracture in place of interfacial debonding. The total number of AE events has one to one correspondence with the number of broken fibres. The effect of surface treatment and matrix systems on the shear fracture strength between the fibre and matrix were described based on the critical length of the broken fibres using AE results.

1. Introduction

It is well known that the performance of fibre-reinforced composites is greatly dependent on the fibre–matrix interfacial properties. Therefore, several test methods have been developed to evaluate the strength of the interfacial bond between the fibre and matrix [1–3]. They are all based on the pull-out test which was originally suggested by Kelly and Tyson [4]. According to this model, the interfacial shear strength τ is given by the following expression:

$$\tau = r\sigma_f/L_c \tag{1}$$

where r is the radius of the fibre, σ_f is the fracture stress of the fibre and L_c is the critical length. When a continuous fibre which is sufficiently longer than the critical length is embedded in a matrix, the fibre subsequently fails during tensile loading of a composite until the critical length is reached. Therefore, if we measure the critical length of the broken fibres on a model composite containing a continuous single fibre with the aid of an optical microscope [5], we can evaluate the interfacial shear strength between the fibre and matrix using Equation 1. However, this technique is seldom used in practice since it is a tedious and time-consuming way of obtaining the critical length, involving the measurement of lengths of a large number of broken fibres.

In a previous paper [6], we have shown that acoustic emission (AE) techniques for monitoring fracture processes in composite materials can be successfully used to evaluate the critical length and, as a result, the interfacial shear strength at the carbon–epoxy and glass–epoxy boundaries has been obtained using AE monitoring data, since it was clearly shown that the AE events have one to one correspondence with the number of broken fibres. The present investigation was undertaken to extend the AE technique to aramid fibre-reinforced composites and to elucidate the effects of surface treatment of the fibres and of mechanical properties of the matrix systems on the interfacial shear strength.

2. Experimental details

The equipment and transducer for monitoring AE events have been described in detail in a

0022–2461/85 $03.00 + .12

previous paper [6] and may be summarized as follows. The AE signals which passed through a band filter of 100 kHz to 1 MHz were totally amplified by 80 dB to give envelope outputs. The output signal, which exceeded a threshold level of 0.5 V, gave one pulse per one event. A wide band transducer made of a poly(vinylidiene fluoride) film was used throughout this experiment.

The matrix was a diglycidyl ether of bisphenol A type epoxy resin, Epikote 828 (Shell). Methylnagic acid anhydride (MNA) and tridimethyl aminomethyl phenol were used as hardener and curing agent, respectively. The four matrix systems with hardener ratios of 40, 60, 80 and 100 per one hundred resin by weight (phr) were studied. The content of curing agent was 1 phr for all systems. The aramid fibres used in this experiment are high modulus fibres of a new type which has been developed by Teijin under the name HM-50. Their chemical structure essentially consists of rigid *para*-oriented benzene rings, a similar structure to Kevlar fibres of du Pont, but the rings are linked by not only amide groups but also by ether groups. The tensile modulus of HM-50 fibres was 76.4 GPa. The breaking strength and strain were 3.2 GPa and 4.5%, respectively. When compared to mechanical properties of Kevlar fibres, these new fibres have an intermediate modulus between Kevlar fibre types 29 and 49, and higher strength and strain at break than those of both types. HM-50 fibres are intended for uses needing high toughness. The fibres of 9 and 28 μm diameter were used with and without surface treatment. The surface treatment was carried out using an ion etching technique. Etching conditions were 1400 V d.c. in air at a pressure of 0.05 torr for 30 min.

Unidirectional model composites with a single fibre were prepared by casting into the mould consisting of a paper gasket of 0.3 mm thick, and glass plates. Curing and post-curing were carried out at 363 K for 4 h and 423 K for 15 h, respectively. Rectangular specimens of 30 mm length and 4.0 mm width were loaded in the fibre direction at a crosshead speed of 0.25 mm min^{-1}. All tests were made in air at 296 K and 50% r.h.

3. Results and discussion

3.1. AE characteristics

Figs. 1 and 2 show typical examples of AE characteristics together with a stress–strain curve of the specimen composed of a single filament of 9 and 28 μm diameter, respectively. Reinforcement with a single fibre has little effect on the gross stress–strain relations of the epoxy matrix, but it can be seen that the AE activity became quite different from the epoxy matrix system, which is shown in Fig. 4 of a previous paper [6], i.e. there are two regions in which the AE emission occurs actively. As the emission produced by matrix cracking begins to occur near the yield point, the emission at a lower strain prior to yield strain is presumed to be generated by fracture of the fibre or interfacial

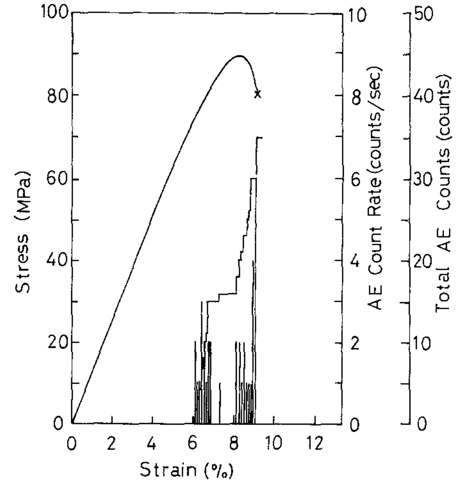

Figure 1 A typical example of acoustic emission characteristics and stress as a function of strain for a composite reinforced with a single aramid fibre of 9 μm diameter.

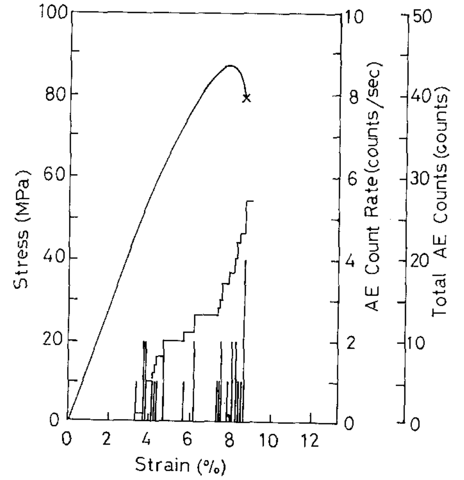

Figure 2 A typical example of acoustic emission characteristics and stress as a function of strain for a composite reinforced with a single aramid fibre of 28 μm diameter.

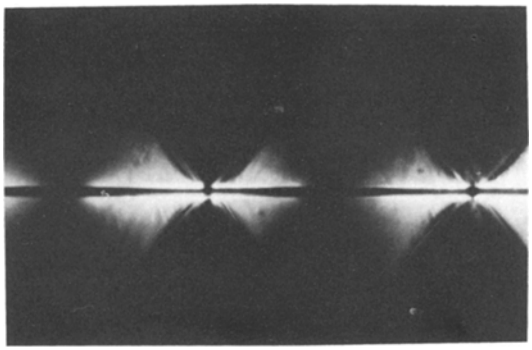

Figure 3 A polarized microphotograph of fractured aramid fibres of 9 μm diameter.

debonding between the fibre and matrix. In Fig. 1 the sharp activity between 6 and 7% strain can be seen, whereas emission occurs within the comparatively broad range between 3 and 6% strain in Fig. 2. Figs. 3 and 4 show polarized microphotographs of the fibre which correspond to Figs. 1 and 2, respectively, when the AE activity occurred in a lower region of strain. In Fig. 3, the bright zones of triangular shape within the matrix along the fibre are related to interfacial shear fracture due to shrinking and sliding which originates from broken ends of the fibre. In Fig. 4, a different type of bright zone along the fibre–matrix interface is observed without fibre fracture. This clearly reveals that the AE bursts counted for a specimen with a fibre of 28 μm diameter are generated by local debonding at the fibre–matrix interface, depending on distribution of the shear strength along the fibre. Fig. 5 shows the relationship between the total number of AE counts and the number of broken fibres obtained from microscopic counting for four different matrix systems with a single fibre of 9 μm diameter. As shown, the AE events have a nearly one to one correspondence with the fibre fracture. However, the AE counts on ductile matrix specimens of 40 phr hardener content have a tendency to overestimate the number of broken fibres because the AE activity due to matrix cracking at lower strains is relatively higher in the ductile matrix specimens than in the brittle ones.

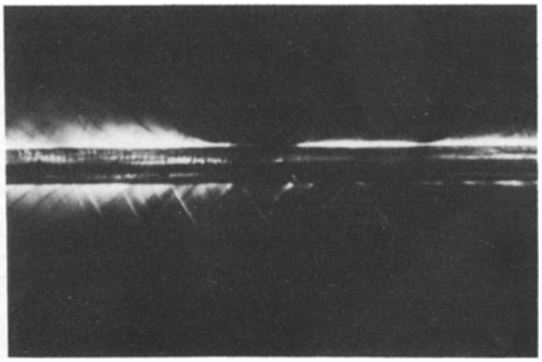

Figure 4 A polarized microphotograph of aramid fibres of 28 μm diameter in which debonding occurred at the fibre–matrix interface.

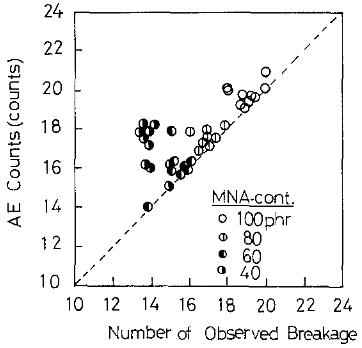

Figure 5 Relation between total counts of acoustic emission and the number of broken fibres along the fibre direction for a composite containing a single aramid fibre of 9 μm.

3.2. Effect of surface treatment by ion-etching on AE characteristics

Usually, a good adhesion of aramid fibres to the epoxy matrix can be obtained because there are many amide groups in the chemical structure of the fibres, their surface treatment other than coating with appropriate soft materials [7] has not received much attention from the practical point of view. However, a better adhesion of the fibres to the matrix can be expected by removing the weak regions of the fibre surface and mechanically roughening it, so as to allow strong physical interlocking of the fibre with the matrix. Fig. 6 shows a scanning electron micrograph of the ion-etched fibre in which the surface is quite roughened by removing the flat layer of skin structure. Fig. 7 shows the AE events against strain curves for the specimen composed of an ion-etched single fibre of 28 μm diameter. A sharp generation of AE bursts occurs at a strain level of 6%, similar to the behaviour in Fig. 1 in which the AE characteristics due to fibre fracture are shown. The microscopic observations also revealed for the ion-etched fibres that

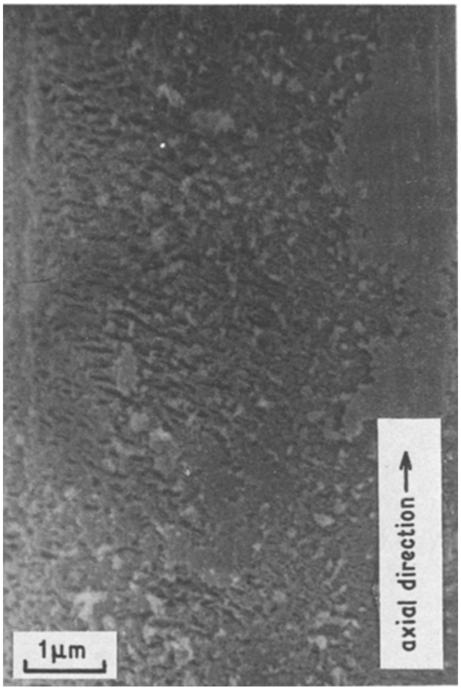

Figure 6 A SEM photograph of the ion-etched fibre surface.

fracture of the fibres occurred without debonding at the fibre–matrix interface. Fig. 8 shows the relationship between the total AE counts and the number of broken fibres. It can be seen again that there is a one to one correspondence between the AE signals and fibre fracture.

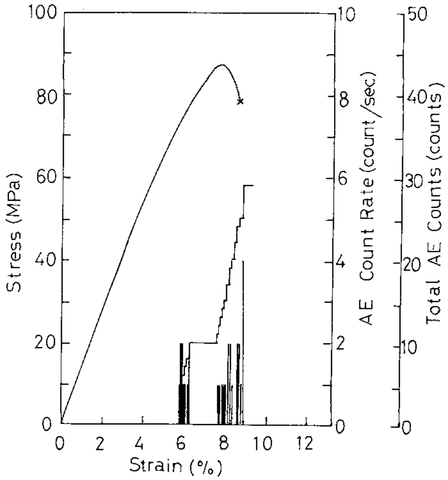

Figure 7 A typical example of acoustic emission characteristics and stress as a function of strain for a composite containing an ion-etched single fibre of 28 μm diameter.

3.3. Evaluation of interfacial shear strength between fibre and matrix

As it has been made clear that the total number of emissions indicates the number of broken fibres, the average critical length can be calculated using the total number of AE counts. Table I shows the shear strength at the fibre–matrix interface obtained from the AE results. The interfacial shear strength increases with increasing matrix modulus which is brought about by increasing hardener content, but the strength rise is not as marked as in the case of the carbon–epoxy interface in which nearly a 100% increase was observed for the same matrix system. Clearly, the adhesion of fibres of 28 μm diameter to the polymer has been greatly improved by ion etching, but there is a slight increase in shear strength for fibres of 9 μm diameter. It is the general observation that the tensile fracture of aramid fibres is the result of axial splitting which usually originages from surface flaws or weak points. The longitudinal shear stresses within the fibre split the fibrils because of their weak interfibrillar bonding [8]. Physical interlocking certainly can be improved by surface roughening by ion etching. However, when once a good adhesion of fibres to the matrix has been reached, the interfacial shear stress between the fibres and matrix enhances the longitudinal shear splitting of the fibrils close to the surface within the fibres and, as a result, the shear strength at the fibre–matrix boundary is

TABLE I Shear strength at the fibre–epoxy matrix interface for aramid and carbon fibres

Fibre		Shear strength (MPa) for hardener content (phr)			
		40	60	80	100
Aramid fibre (9 μm)	not treated	20.2	23.0	24.5	27.4
	ion-etched	21.6	25.9	28.8	–
Aramid fibre (28 μm)	not treated		debonding		
	ion-etched	debonding	17.9	22.4	26.9
Carbon fibre*	not treated	20.0	27.7	38.7	41.7

*From previous paper [6].

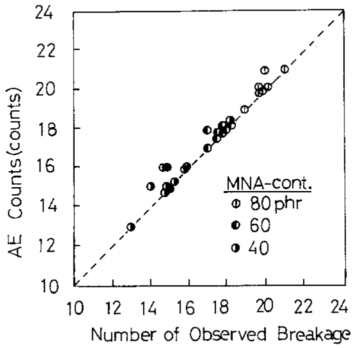

Figure 8 Relation between total counts of acoustic emission and the number of broken fibres along the fibre direction for a composite containing an ion-etched fibre of 28 μm diameter.

determined by the interfibrillar bond strength of the fibres. This failure mechanism can explain that the interfacial shear strength between aramid fibres and matrix is less sensitive to an increasing modulus of matrix systems than that of carbon–epoxy matrix interface.

4. Conclusions

AE monitoring in a uniaxial model composite composed of a single aramid fibre was found to give useful information on a quantitative evaluation of the interfacial shear strength between the fibre and matrix. Surface treatment of aramid fibres by an ion etching technique improved the adhesion of the fibres to epoxy matrix so as to produce fibre fracture in place of debonding at the fibre–matrix interface. However, the interfacial shear strength, which was relatively weak and insensitive to a modulus of the matrix systems when compared to carbon fibre composites, is the result of the shear fracture caused by the fibril splitting close to the fibre–matrix boundary.

Acknowledgements

This work was supported in part by a Ministry of Education Grant-Aid for Scientific Research. The authors acknowledge the help of Teijin Co Ltd in supplying yarn samples and Yuka-Shell Co Ltd in supplying epoxy samples.

References

1. C. C. CHAMIS, "Composite Materials", Vol. 6 (Academic Press, New York, 1974) p. 75.
2. L. J. BROUTMAN, "Interfaces in Composites", ASTM STP 452 (American Society for Testing and Materials, Philadelphia, 1969) p. 27.
3. P. JARRLA, K. W. LATINEN, J. PUROLA and P. TORMALA, *Int. J. Adhesion Adhesives* **3** (1983) 141.
4. A. KELLY and W. R. TYSON, *J. Mech. Phys. Solids* **13** (1965) 329.
5. M. ISHIKAWA, I. NARISAWA and H. OGAWA, "Mechanical Behavior of Materials", Vol. I, Proceedings of the 1974 Symposium on Mechanical Behavior of Materials (The Society of Materials Science, Kyoto, 1974) p. 579.
6. I. NARISAWA and H. OBA, *J. Mater. Sci.* **19** (1984) 1777.
7. Y. W. MAI and F. CASTINO, *ibid.* **19** (1984) 1638.
8. L. KONOPASEK and J. W. S. HEARLE, *J. Appl. Polym. Sci.* **21** (1977) 2791.

Received 28 December 1984
and accepted 31 January 1985