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Alternative means for evaluating fibre–matrix adhesion in composites

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This paper presents an alternative means, for evaluating fibre–matrix adhesion in composites, which uses simple unidirectional composites in a test geometry that ensures the predominant composite fracture mechanism is that associated with interfacial failure, thus providing direct information regarding the characteristics of the fibre–matrix interface. We have investigated these interfacial failure events by means of acoustic emission (AE) during tensile testing of simple composites whose fibres are oriented perpendicular to the tensile direction. The strain and stress range over which these AE events occur determine the strength of the fibre–matrix interface, while the relative total number of recorded events give indication as to the interfacial failure mode. By varying the treatment of the fibre surface, this changes the nature of the interfacial bonding and is clearly reflected in the AE and mechanical responses. In general, the results obtained and presented in this paper demonstrate that the method shows good sensitivity to changes in the level of fibre–matrix adhesion in composites, providing information on the nature of fibre–matrix adhesion, the strength of the bonds and the failure mode, all in one experiment.

1. Introduction

With the current knowledge that the interface between fibres and resin-matrix in composites contributes substantially to the performance of these materials, particularly where applications may have stresses applied perpendicular to the fibre direction, it has become increasingly necessary [1, 2] to be able to characterize and understand the fibre–matrix interface with a view to tailoring it to meet performance requirements. This has not been an easy task, notably when using real composites, due to interferences from the various failure mechanisms (fracture of the fibres, fracture of the matrix, debonding and/or pull-out of fibres from the matrix, etc.) occurring during testing of such materials.

Nonetheless, a variety of methods, based on single fibre composite models have been proposed as a means of measuring fibre–matrix adhesion in composites. Herrera and Drzal [3], reviewed these methods and concluded that none of them offers a complete and unambiguous means of evaluating the level of fibre–matrix adhesion in composites not to mention the interfacial failure mode. This was due to the scatter of data from the various methods, while using the same fibre and resin–matrix materials, and which may be attributed to both the sample geometries and to the fact that the associated mathematical formalisms of the methods use an oversimplified representation of the state of stress occurring at the interface. Additionally, in considering the measured interfacial property, the roles played by the resin–matrix shrinkage about the fibre, by interfacial bonding (physical or molecular) and by interfacial friction, are not clear.

In this paper, we present an alternative means for evaluating fibre–matrix adhesion in composites, which employs simple specimens in a test geometry, such that the predominant composite failure mechanism is that associated with interfacial failure. Peters [4], has addressed issues connected with transverse cracking and fibre–matrix bond strength using composite laminates, instead of the special test samples used in this work and which has the advantage that interfacial failure does not immediately lead to composite failure. This has been attributed to the low fibre volume fraction in our composite test pieces.

The predominant fracture mechanism occurring during tensile testing of simple fibre bundle composites with the fibre axis perpendicular to the tensile direction is interfacial failure events. The associated stress waves or acoustic emission (AE) are detected by a piezoelectric transducer placed in contact with the test specimen, thus one is able to access the strain and stress range over which failure ensues at the fibre matrix interface region. The relative total number of recorded events gives an indication as to the interfacial failure mode (interfacial ≡ when fracture occurs at the fibre surface or interphasial ≡ when fracture occurs in the matrix close to the fibre surface).

We have applied this method of adhesion to composite systems such as: E-glass/polyester; Kevlar-49/polyester; E-glass/epoxy; and Kevlar-49/epoxy, with

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various treatments of the fibre surface prior to composite manufacture. Such fibre surface treatments affect the nature of the interfacial bonding and are reflected unambiguously in the AE record, yielding the characteristics of the interface. Results from this method are compared to those previously obtained [5] using the single fibre composite (SFC) multifragmentation method of adhesion, and the indications are that the new fibre-bundle composite method would give a clearer picture of the characteristics of the fibre–matrix interface.

2. Experimental procedure

2.1. Materials preparation

The fibre systems employed in this study included E-glass (Fibre-Glass (UK) Ltd; Equerove, Silane-sized, EC13, 600Tex) and Kevlar-49(Dupont (UK) Ltd; Den 2160, Dtex 2400, finish free). Matrix materials used were Crystic polyester 272 resin(Scott-Bader(UK) Ltd.) and Epoxy LY5025(Ciba-Geigy Polymers(UK)). The resin preparations followed the recommended procedure for producing laminates; 100 parts of Crystic polyester 272 resin was used to 2 parts of Crystic catalyst(methylethylketoneperoxide) and 1 part of cobalt accelerator E in styrene, and after degassing were poured into dogbone shaped moulds of silicon rubber, containing the fibre system. This was left to cure at room temperature for 7 days. For the epoxy, 100 parts of the resin LY5025 was used to 38 parts of the hardener HY5025, and the preparation cured for 8 h at 80 °C.

To demonstrate the sensitivity of our method (of measuring adhesion in composites) to changes in the level of fibre–matrix adhesion, two types of fibre surface treatment were considered: in one case to minimize adhesion, both fibre systems were coated with a silicone-oil prior to composite manufacture. In the other case to promote adhesion, the Kevlar fibre system was subjected to a chemical treatment (30 min in a dilute solution of H_2SO_4 + HNO_3) that raises the concentration of the oxygen bearing functional group, C–O, at the surface of the fibres. This was performed since it has been reported [6] that raising the surface concentration of oxygen bearing functional groups in Kevlar fibres, improves their adhesion to epoxy. That the treatment does enhance the surface concentration of oxygen in the Kevlar fibres, was deduced from comparing the elemental peaks of carbon, oxygen and nitrogen revealed in the XPS(X-ray photoelectron spectroscopy) spectra of as-received and chemically treated Kevlar-49 fibres, which showed that the surface of the chemically treated fibres is richer in oxygen and poorer in nitrogen than the surface of the as-received fibres. This chemical treatment also etches the fibres (and is not recommended for long durations since this has been observed to drastically weaken the fibres), and we believe that resin anchoring into the fibres contributes also to the apparent improvement in adhesion.

The moulds were formed from 2 flat sheets of silicon rubber with a number of parallel dogbone shapes punched out of them. A bottom sheet is placed on a flat glass slab prior to mounting of a fibre bundle across the shapes. The bundle ends are taped onto the glass slab before the top rubber sheet is carefully aligned and mounted. After pouring in the resin, the mould is closed with a top glass slab. The entire process ensures that the fibre bundle is accurately aligned. The composite of interest to us is the case where the fibre system is centrally located but oriented transverse to the longitudinal axis of the dogbone shapes and which we term transverse bundle of fibre composites (TBFC). However, to demonstrate the difference in fracture mechanisms occurring during deformation of composites with various fibre orientations, composites were also prepared with longitudinal orientation (LBFC) of the fibre bundle. The fibre volume fraction in LBFCs is about 1.32% for Kevlar-49 and 1.87% for E-glass, while in TBFCs, within the specimen domain containing the fibre bundle, these values are 2.7% for Kevlar-49 and 4.2% for E-glass. All composite gauge dimensions were 40 × 5 × 2.5 mm.

2.2 Materials testing

Tensile tests on the composites were undertaken using a Lloyd-6000R (research grade) testing machine at a constant crosshead movement rate of 0.5% per min. During the tests, the AE technique was employed as a means of monitoring the time of occurrence and also discriminating the fracture mechanisms in operation during deformation of the composites [7]. For this purpose, a commercial AE transducer, AC375L, with a resonance frequency of 375 kHz, manufactured by Acoustic Emission Technology Corporation, was utilized. Silicone grease was used as an acoustic couplant in bringing the transducer into intimate contact with the composite test specimens mounted in the grips of the tensile machine. AE signals detected by the transducer were preamplified by 60 dB, using a preamplifier (AECL 2100/PA) with narrow band filtering (208–530 kHz bandpass) around the transducer resonance and further processed using the AECL 2100 M acoustic emission system. This processing, depending on the system and instrumental settings, enables one to access the event size (single or multiple pulse events) and some of the possible AE signal parameters (initial peak voltage, event rise time, event duration and the ringdown counts per event or group of events, i.e., the number of positive threshold crossings of the signal) that can be related to the nature of the events. The ringdown count was used in this study as it is related to the relative acoustic energy [8] released by the fracture events. The event size and ringdown count are passed via an integral analogue-to-digital computer interface board to a computer which stores these values and the corresponding strain and load values. Note, however, that the values obtained for the ringdown counts are considered relative since they depend on the threshold voltage applied. Another computer with a specially adapted h/software package acquires and stores the AE event signal waveforms for further detailed spectral analysis. AE instrument settings were: dead time of 0.2 ms and signal threshold of 0.1 volts. We observed that such a threshold voltage

was adequate to minimize the detection of electronic background noise, noise from the grips and the tensile machine, thus received signals could be associated with fractures occurring in the composite being tested.

3. Results and analysis

Typical AE signal waveforms from fracture events occurring during tensile deformation of longitudinal (LBFCs) and transverse bundle of fibres composites (TBFCs) of Kevlar-49/polyester are presented in Fig. 1 (a and b respectively). It can be seen that the relative acoustic energy content of any one signal (i.e., the area enclosed by the chosen signal) in Fig. 1a is much greater than that in Fig. 1b. When this parameter (or the associated number of ringdown counts per event-signal) is used to discriminate the event type, the deduction would be that the fracture events responsible for the signals in Fig. 1a are definitely of different origin from those emanating the signals in Fig. 1b. This is further substantiated by the different lineshape or envelope of a typical AE signal waveform from either composite, shown correspondingly in Fig. 2(a and b). While the signal-lineshape of a fracture event from LBFCs gives the impression of a single pulse event, that from TBFCs gives the impression of a multiple fracture process such as a tearing. Additionally, the events, in the case of LBFCs, and as shown in Fig. 3a, were observed to occur close to composite failure strain, possessing ringdown counts per event (Ne) that exhibited an increasing trend with applied stress. We have previously [9] reported this trend in tests involving fibre bundles in air. In the case of TBFCs, as shown in Fig. 3b, the fracture events were observed to occur at low strains with a relatively smaller number of ringdown counts per event. From the above mentioned observations and known mechanical effects, the events recorded during tensile

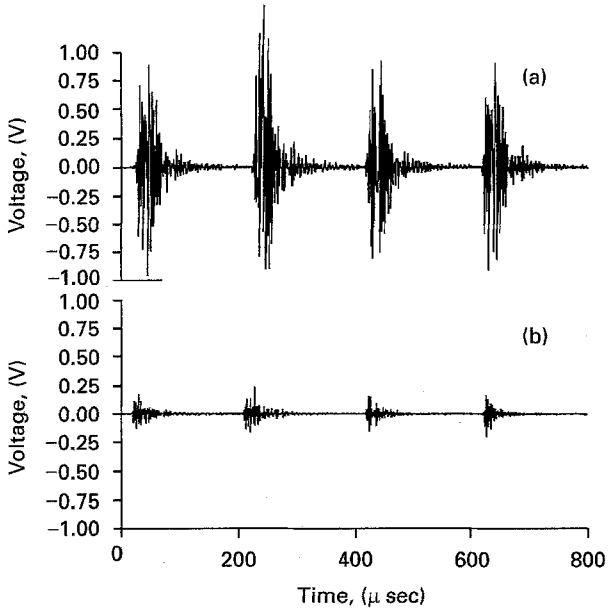

Figure 1 Comparison of the relative acoustic energy content of typical AE signals from fracture events occurring during tensile deformation of undirectional bundle of fibres composites. (a) Longitudinally (LBFC) and, (b) transversely (TBFC) to the fibre axis.

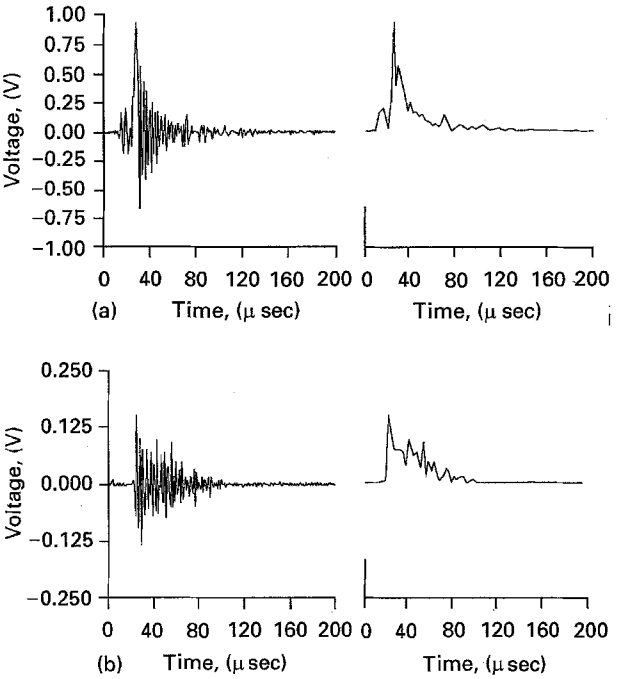

Figure 2 Comparison of the typical lineshape or envelope of the signals shown in Fig. 1. The signals from (a) LBFCs and, (b) TBFCs, have respectively been associated with fibre fracture in the composite and fracture occurring in the region of the fibre–matrix interface. The envelopes shown enclose only the positive part of the signal.

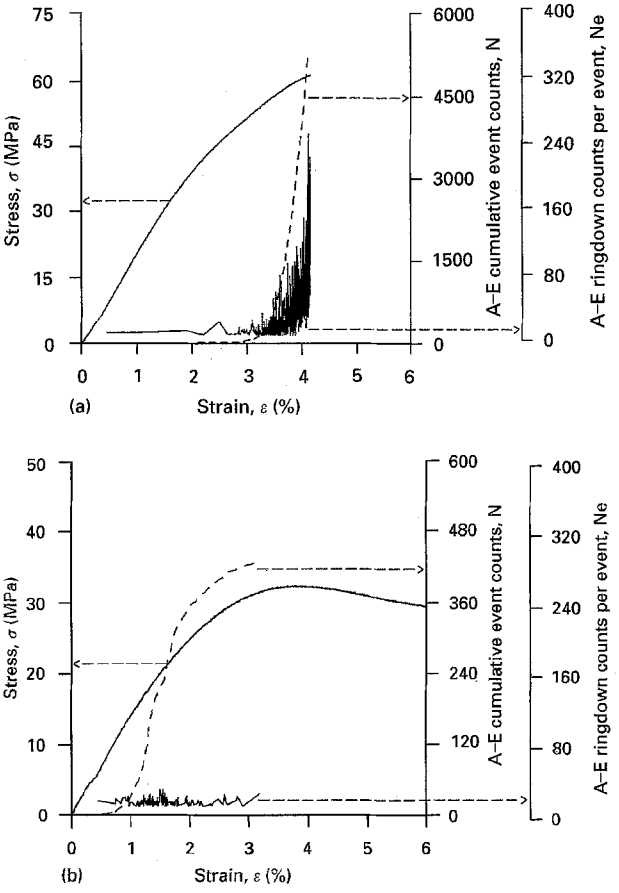

Figure 3 Stress-strain responses (σ–ε) of, (a) longitudinal (LBFC) and, (b) transverse (TBFC) bundle of fibres composites of Kevlar-49/polyester matrix, including the AE events-strain responses (N–ε) and the associated number of ringdown counts per event (Ne).

deformation of the TBFCs were associated with fracture in the region of the fibre–matrix interface, while for the LBFCs, the events were mainly associated with fibre fractures, since in the composite stress range over which these events occurred, the main load bearing constituent of the composite are the fibres, and their fracture leads to the failure of the composite as a whole. These results demonstrate the possibility, using the AE technique, of monitoring failure in the region of the fibre–matrix interface during tensile deformation of TBFCs and hence obtaining information regarding the characteristics of this region in composite materials. The method has been applied to compare the level of fibre–matrix adhesion in a variety of composite systems, including under varying fibre surface conditions.

Test data for TBFCs of Kevlar-49/polyester with varying fibre surface treatments (fibres were (a), coated with silicone oil; (b), as-received and; (c), subjected to a chemical treatment, as described earlier in the text) are presented in Fig. 4, showing the composites tensile stress–strain response and corresponding AE cumulative events counts N. The composites exhibited identical stress-strain responses and this is attributed to the low fibre volume fraction, such that the role played by the nature of the fibre–matrix interface region is not immediately apparent. However, in all our investigations with different TBFCs presented in this paper, specimen failure occurred within the region containing the fibre bundle, clearly indicating that failure in the vicinity of the fibre–matrix interface initiated the specimen failure.

Since the recorded AE events are associated with fracture in the region of the fibre–matrix interface, then with the AE events–strain response as an indicator, one can say that the interface characteristics of the three composites are different. In comparison to the strain range over which these events occurred in the composite with the as-received fibres, one notes

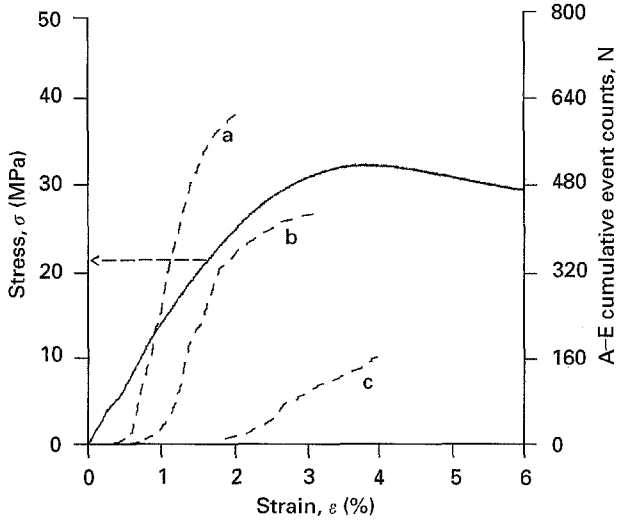

Figure 4 Test data of transverse (TBFCs) bundle of fibres composites of Kevlar-49/polyester, comparing the tensile stress-strain (σ–ε) and AE events-strain (N–ε) responses, when the fibres are (a) pretreated with silicone oil, (b) as-received and, (c) subjected to a chemical treatment (see text for details), prior to composite manufacture.

that they occurred at lower strains in the composite whose fibres are coated with silicone oil and at higher strains in the composite whose fibres were chemically treated. This clearly demonstrates that with this method of measuring adhesion in composites, one can easily access the strength of the fibre–matrix interfacial bonding, and also compare this parameter in different situations. One remarks also that there are more recorded AE events when the fibres are coated with silicone oil than with the as-received fibres, and fewer events when the fibres are subjected to the chemical treatment. In the latter case, the very few fibres exposed after specimen failure were covered with the matrix material, indicating that failure, here, occurred predominantly in the matrix close to the fibre surface. As this region of the matrix is common to many fibres, it is therefore not surprising that there are fewer recorded events during interfacial failure in this composite. For the composite whose fibres were coated with silicone oil, there were more exposed fibres and these were entirely free of the matrix material, consistent with failure occurring at the fibre–matrix interface (i.e. at the fibre surface). In the composite with the as-received fibres, some of the exposed fibres were partially covered with the matrix material. The reason for this is not very clear, since Kevlar is not known to show any affinity towards polyester, unless it was subjected to an unspecified surface treatment by the manufacturers or that the partial adhesion is purely physical. Nonetheless, it is clear from this method of measuring adhesion in composites, that the relative recorded total number of fracture events gives indication as to the interfacial failure mode. Clearly, these results demonstrate the potential of this means of measuring adhesion in composites. It has shown unambiguously in Kevlar-49/polyester composite systems that in comparison to the composite made using the as-received fibres, silicone oil treatment of the fibres prior to composite manufacture minimizes fibre–matrix adhesion whereas a given chemical treatment, which raises the concentration of the oxygen bearing functional group C–O, at the fibres surface, improves adhesion.

Similar trends were observed when the matrix material is an epoxy, and the test data are shown in Fig. 5. While the stress–strain responses of the composites, for all fibre surface treatments, appear identical, the AE events–strain responses again show, when compared to the case with as-received fibres, that the silicone oil treatment reduces adhesion, whereas the chemical treatment improves it. With silicone oil treatment, interfacial failure events start earlier in strain and at correspondingly lower stress values. A significant increase in the number of recorded AE events is seen to occur, which is consistent with failure occurring predominantly at the fibre matrix interface. In the case with chemically treated fibres, the failure events, which are relatively few in number, occur at higher strains and at correspondingly higher stresses. However, by closer inspection of Figs. 4 and 5, one would not fail to notice that there are differences in the interface characteristics of Kevlar-49/epoxy and Kevlar-49/polyester composite systems. In comparing

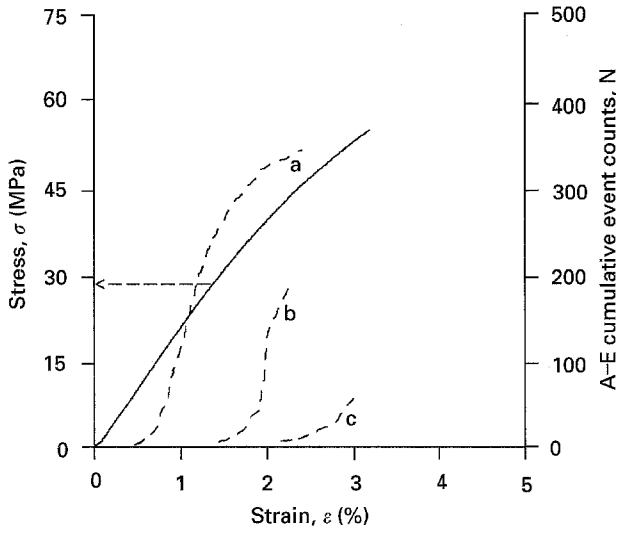

Figure 5 Similar comparison as in Fig. 4, but with an epoxy matrix.

the AE events–strain/stress relationships for both composite systems made using the as-received fibres (case b in both figures), one can only conclude that interfacial bonding is stronger in a Kevlar/epoxy composite than in a Kevlar/polyester composite. This is probably a result of interactions between Kevlar, the amine hardener and the epoxy resin. One remarks also that the application of silicone oil to the fibre surface prior to composite manufacture (case a in both figures), appeared to have a more adverse effect on adhesion in a Kevlar/epoxy composite than in a Kevlar/polyester composite. In the Kevlar/epoxy composite system, the difference in the interfacial failure strain-range between the cases involving the as-received fibres and the fibres coated with silicone oil is large in comparison to the difference between similar cases in the Kevlar/polyester composite system. While this may be attributed to the silicone oil hindering interactions between Kevlar, the amine hardener and the epoxy resin, it also demonstrates that the Kevlar used in this study shows poor interactions with Crystic polyester. One thus deduces that adhesion between Kevlar and polyester may be largely a physical effect due to resin shrinkage onto the fibres. Resin shrinkage may also explain the fact that a zero strength was not observed in all the cases involving fibres coated with silicone oil prior to composite manufacture, since such an action would displace some of the silicone oil and consequently minimize its effect. The indications, however, are that the polyester resin displaces more of the silicone oil than the epoxy, which is consistent with polyester exhibiting larger shrinkage volume than epoxy.

We present in Fig. 6(a and b) the test data of TBFCs of E-glass (fibres were (i), coated with silicone oil and; (ii), as-received with silane sizing) in polyester and epoxy matrices respectively. Here, again this method of measuring adhesion has demonstrated its sensitivity to changes in the level of fibre–matrix adhesion. In addition, when one compares the AE event–strain data in Fig. 6a obtained from the as-received E-glass fibres/polyester composite to that in Fig. 4 obtained from the as-received Kevlar-49 fibres/polyester com-

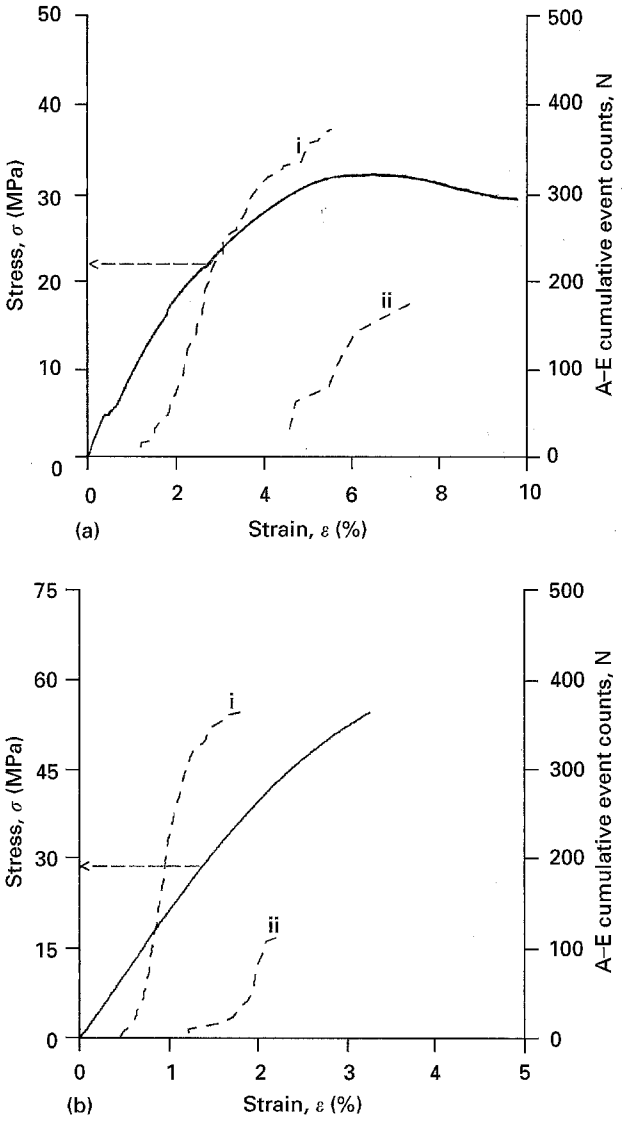

Figure 6 Test data of transverse (TBFCs) bundle of fibres composites of, (a) E-glass/polyester and, (b) E-glass/epoxy, showing the tensile stress-strain (σ–ε) and AE events-strain (N–ε) responses, when the fibres are (i) pretreated with silicone oil and, (ii) as-received with silane sizing.

posite, it becomes evident that this method of measuring adhesion in composites clearly indicates that the E-glass/polyester interface is much stronger than that of Kevlar-49/polyester. In the latter composite, the AE recorded events occurred at low strains and within the elastic limit of the matrix material, while in the former composite, the events occurred well beyond the yield point of the matrix material and were fewer in number (which is consistent with failure occurring predominantly in matrix close to the fibre surface). This information unambiguously shows that the E-glass used in this study, unlike the Kevlar-49, adheres well to Crystic polyester. This distinction between the strength and nature of the fibre–matrix interface in the two composites did not manifest itself clearly in adhesion tests involving the single fibre composite (SFC) multifragmentation method [5], from which close interfacial shear strength values were deduced for both composites, and may be regarded as suggesting similar levels of adhesion. The enhanced fibre–matrix adhesion in the E-glass/polyester composite has been

attributed to the interactions between the silane coupling agent and polyester[10]. The silane coupling agent is also known to interact with epoxy, which explains the AE recorded events obtained during tensile deformation of TBFCs of as-received E-glass/epoxy occurring at relatively high strains and at correspondingly high stresses (see Fig. 6b). In fact, indications are that the E-glass used in this study shows similar levels of adhesion with both the polyester and epoxy resin-matrices employed in the preparation of the composites.

Since for the tests geometry of the TBFCs, one may assume equal load sharing between the fibres and the matrix (although unrealistic [11]), then the macroscopic sample stress range over which the fracture events occurring in the region of the fibre–matrix interface are recorded by means of acoustic emission can be regarded as the range of the fibre–matrix bond strength. That the fracture events occur over a range of stress may be attributed to non-uniform loading in the region of the fibre–matrix interface, due probably to some degree of misalignment of the fibres in the bundle, edge effects, etc. Nonetheless, with the apparent close relationship between the commencement of the fracture events and the anticipated effect, on interfacial properties, of the state of the surface of the fibres used in manufacturing the composites, we propose, since the local stress distribution in the vicinity of the fibre–matrix interface is most likely to be very complex, that the corresponding macroscopic sample stress at the occurrence of the first few events be taken as a measure related to the local fibre–matrix bond strength. Using this approach, we present in Table 1 the relative fibre–matrix bond strength of the composites considered in this study. The values in the table are based on the strength obtained for the chemically treated-Kevlar fibres/epoxy composite.

Using the relative fibre–matrix bond strengths given in Table 1 as an indicator of the level of adhesion it can be said that as-received E-glass fibres exhibit similar levels of adhesion in both polyester and epoxy resin-matrices, while as-received Kevlar fibres show much better adhesion to epoxy than to polyester. The already mentioned chemical treatment of the Kevlar fibres improves their adhesion to both polyester and epoxy resins. In all cases, silicone-oil treatment of the fibres prior to composite manufacture minimized adhesion and the effect is more pronounced in systems (silane treated E-glass/polyester, silane treated E-glass/epoxy, and Kevlar/epoxy) where, in the absence of silicone-oil at the surface of the fibres, there would have been strong molecular interactions between the fibres and the resin-matrix. One notes with interest that the fibre–matrix bond strength in an as-received E-glass-fibres/polyester composite is about twice that of an as-recieved Kevlar-fibres/polyester composite, which may be interpreted as E-glass exhibiting much better adhesion than Kevlar to polyester. Fibre–matrix interfacial shear strength values deduced from single fibre composite (SFC) multifragmentation adhesion tests have often been quoted [3] as a measure of the level of adhesion in composites, and in a recent study by the authors [5], using this method of adhesion with the same fibres and resins employed in the current study, we deduced interfacial shear strength values of 36.8 MPa and 43.4 MPa for Kevlar-49/polyester and E-glass/polyester composites respectively. Similar values were obtained, in each case, when the single fibres were pre-treated with silicone oil prior to composite manufacture. Obviously, these do not clearly manifest the lack of adhesion between Kevlar and polyester and the minimization of adhesion, in both composites when silicone oil is present at the fibre surface. The possible origins of these shortcomings were explained. Rather, in using the TBFC method of adhesion, the clear agreement observed between experiment and expectation suggests that this method shows better sensitivity to changes in the level of adhesion between a resin-matrix and different fibre systems and *vice versa*, including under varying fibre surface conditions.

While employing the TBFC method of adhesion, we observed also that the relative acoustic energy released by fracture events occurring in the region of the fibre–matrix interface varied with the state of cure of the composite. This is illustrated in Figs 7(a and b), which compare tensile and AE test data obtained during deformation of TBFCs of as-received Kevlar-49 fibres/polyester composites cured at 20 °C for 7 days (Fig. 7a), and further post-cured at 120 °C for 1 h (Fig. 7b). In comparing both figures, it can be seen that postcuring stiffens the composite and also renders it less ductile. This is a known and well documented effect [12]. For both composites, the recorded AE fracture events occurred in the same strain range, but at different macroscopic sample stresses. However, a significant increase in the number of recorded AE events is seen to occur with post-curing. This has been attributed to brittle matrix cracking events, occurring in the sample domain containing the fibre bundle, and may either have been initiated by interfacial failure events[13] or as a consequence of the high stresses that may have built up in this region of the specimen after the post-cure, due to the presence of the fibres. Whatever, in the post-cured composite, one notes that

TABLE I Relative fibre–matrix bond strength (RBS), deduced from the macroscopic sample stress giving initial AE events, for as-received and for silicone-oil treated E-glass and Kevlar fibres, including also chemically treated Kevlar fibres, embedded in polyester and epoxy resins. N is the number of specimens tested

Resin system	Fibre system	Fibre treatment	RBS (MPa)	N
Polyester	Kevlar-49	Silicone-oil	0.21	3
	Kevlar-49	As-received	0.35	4
	Kevlar-49	Chem-treatment	0.63	3
	E-glass	Silicone-oil	0.33	5
	E-glass	As-received	0.74	5
Epoxy	Kevlar-49	Silicone-oil	0.28	3
	Kevlar-49	As-received	0.83	2
	Kevlar-49	Chem-treatment	1.0	3
	E-glass	Silicone-oil	0.32	3
	E-glass	As-received	0.79	2

Chem-treatment ≡ chemical treatment (30 min in a dilute solution of $H_2SO_4 + HNO_3$).

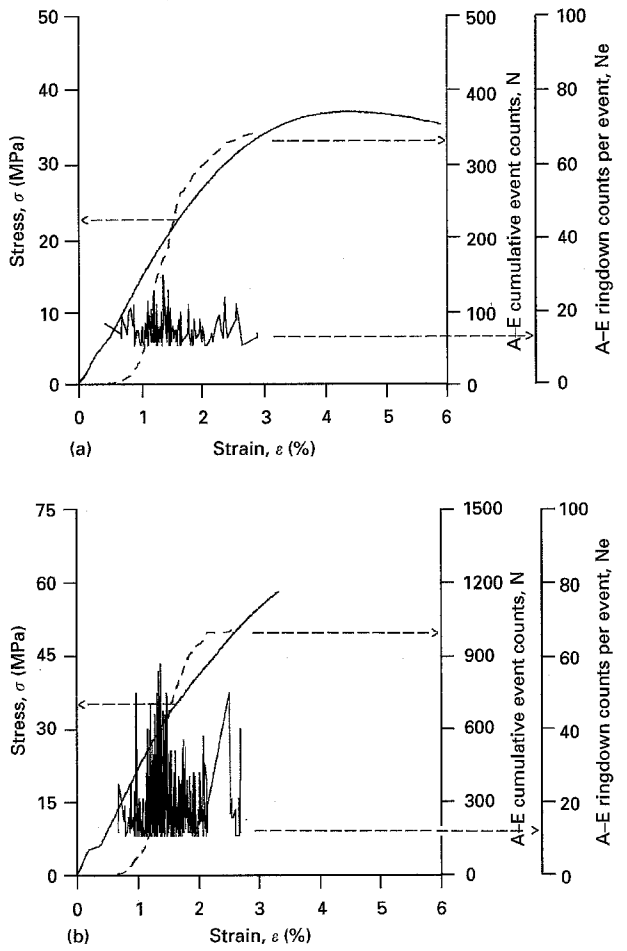

Figure 7 Comparison of the tensile stress-strain (σ–ε), AE events-strain (N–ε) responses and the associated number of ringdown counts per event (relatable to the relative energy content of a fracture event) in transverse (TBFCs) bundle of fibres composites of Kevlar-49/polyester cured at, (a) 20 °C for 7 days and, (b) further post-cured at 120 °C for 1 h.

the AE ringdown counts per fracture event Ne, which is a measure of the acoustic energy content of a fracture event, is about twice that from the sample cured only at 20 °C for 7 days. This may be partially attributed to brittle matrix cracking events occurring in the region of the fibre–matrix interface as has been suggested by Sato and Kurauchi [13]. While this shows that the energy released during fracture processes occurring in the region of the fibre–matrix interface varies with the state of cure of the composite, and may even be large and comparable to the energy released by fibre fractures, it may also be hinting that the method of adhesion developed and presented in this paper, could be used also, given that the recorded fracture events are occurring in the region of the fibre–matrix interface, to deduce the degree of cure in the matrix closest to the fibres. The state of cure of the matrix in the closest vicinity to the fibre surface has been reported [14, 15] to differ from that in the matrix farther from the fibre surface, and to date, dynamic mechanical analysis has been the only means of studying this. We think that the method presented in this paper might provide much needed complementary information for a better understanding of the state of cure in the region of the fibre–matrix interface. This, however, requires further investigation.

4. Conclusion

In this study, based on experimental evidence, the following conclusions can be drawn;

The characteristics of the fibre–matrix interface in fibre reinforced plastics can be deduced using simple multifibre composites in a mechanical test geometry such that failure in the region of the fibre–matrix interface becomes the predominant fracture process occurring. With suitably adjusted acoustic emission (AE) instrumentation, the associated stress-wave emissions from the fracture events can be detected and analysed to deduce the strength of the fibre–matrix bond, the dominant failure mode (i.e., either at the fibre surface or away from it), and consequently the nature of the fibre–matrix bond (physical or molecular).

In applying this approach to investigate fibre–matrix adhesion in a variety of composite systems, we have shown that silane-sized E-glass fibres exhibit similar levels of adhesion to both polyester and epoxy resins, which is consistent with silane exhibiting strong affinity for both plastics. Untreated Kevlar-49 fibres show much better adhesion to epoxy than to polyester, and this was attributed to possible interactions between the fibres, the amine hardener and the epoxy resin. Kevlar-49 is not known to show strong affinity for polyester, which explains one of our observations, using this method of adhesion, that the interface strength in silane-sized E-glass fibres/polyester composite is about twice that in untreated Kevlar-49 fibres/polyester composite. However, when Kevlar-49 fibres are subjected to a chemical treatment that raises the surface concentration of the oxygen bearing functional group C–O, this substantially improved the adhesion of these fibres to both polyester and epoxy. In all cases, silicone-oil treatment of the fibres prior to composite manufacture minimized adhesion. The approach did provide evidence also that when there are strong interactions between the fibres and the matrix, failure occurred predominantly in the matrix close to the fibre surface, while in the absence of such interactions, failure occurred mainly at the fibre–matrix interface (i.e., at the fibre surface). In the latter case, the relative total number of recorded AE fracture events is large, while it is smaller in the former case.

The agreement observed between experiment and expectations, when using AE to evaluate adhesion, clearly demonstrates that the method of adhesion described in this paper, shows good sensitivity to changes in the level of fibre–matrix adhesion in composites. Moreover, the simiplicity of the method suggests that it can be used both for routine evaluation of adhesion in composite systems and evaluation of interfacial adhesive performance in a variety of conditions. There are indications also that the method developed here could be used to evaluate the state of cure of the matrix in the closest vicinity to the fibre surface.

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