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Fracto-emission from single fibres of Kevlar

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Fracto-emission (FE) is the emission of particles (e.g. electrons, ions and photons) during and following fracture. In this paper, we present data on electron emission (EE) and positive ion emission (PIE) from the tensile fracture of Kevlar* single fibres. The fibres were initially fractured in pure tension, where a stranded form of fracture was observed, often with multiple peaks spread over several hundred microseconds. The loading condition was then changed by stretching and breaking the fibres over a dull metal edge. With this change in the loading, different forms of fracture were observed, each with distinctive forms of emission curves. When fracture was accompanied by extensive fibril formation, total emission was high and both EE and PIE decay times were long relative to fractures in which little fibril formation occurred. The results of this study suggest that FE has some applicability as a tool for the detection of fracture mechanisms of single fibres.

1. Introduction

The fracture behaviour of fibre-reinforced composites is strongly dependent on the properties of the fibres, the matrix, and the interface. The role of debonding, fibre fracture, and fibre pull-out can be critical in determining the toughness of fibre-reinforced composites. Recently we have been examining various characteristics of a phenomenon called fracto-emission, exploring its relationships to failure mechanisms in composites including fibre- and particulate-reinforced epoxies [1, 2] as well as particulate-filled elastomers [3–5]. By fracto-emission (FE) we mean the emission of particles (e.g. electrons, positive ions, neutral species and photons) during and following the formation of a crack in a stressed material ([4, 6] and references found therein, contain our recent work on a wide variety of materials). As part of our FE studies, we have examined the FE from the fracture of single fibres of materials such as E-glass, S-glass, graphite, and Kevlar. For example, we were able to measure the mass of the PIE accompanying the fracture of single-fibre Kevlar [7]. The fracture properties of single fibres under tensile stress are of considerable interest because of their contribution to strength in elongation. Studies of the strength and fracture morphology of Kevlar fibers under a variety of loading conditions have been reported by Bunsell [8], Hearle and Wong [9, 10] and Chiao *et al.* [11].

In this paper we present results of recent measurements of electron emission (EE) and positive ion emission (PIE) from the fracture of single fibres of Kevlar-49. In this work we focus on the time dependence and total intensities of the emission produced by such fracture. These measurements give clear indications of the time required for the fibres to undergo failure as well as the "damage" being produced during fracture. For fibres of only a few microns diameter, the duration of the FE signals indicate that the duration of the failure of the fibre can be surprisingly long. We propose that such FE measurements may prove useful for examining the manner in which fibres break.

2. Experimental procedure

The Kevlar-49 Aramid fibres used for this research

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*Trade name of E. I. Du Pont de Nemours and Co.

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are characterized by high crystallinity, high tensile strength and modulus, and low density. The samples used here were single fibres adhesively bonded to tabs made of aluminium sheet shaped to fit into clamps in our vacuum system. The fibre length and diameter were 18 mm and 13 μm, respectively. The fibres were strained at a rate of 1% $\sec^{-1}$ until failure occurred. Almost all fibres fractured under pure tension broke in various stranded forms. For comparison, some of the fibres were stretched across a dull aluminium edge with a radius of curvature of approximately 50 μm during straining; this frequently produced "smoother" fractures with less splitting and shredding due to the more localized stress concentration and perhaps some pre-failure abrasion.

EE and PIE were measured using a Galileo Electro-Optics Model 4821 channel electron multiplier (CEM) positioned within 3 cm of the sample. The front of the CEM was biased at + 300 V for electrons and − 2500 V for positive ions. The pulse output (10 nsec pulse width) from the CEM was amplified and fed into a 100 MHz discriminator and then into a multichannel analyser, allowing counts as a function of time to be recorded at 1 μsec per channel. The emission curves varied in duration and are displayed here on various time scales, emphasizing the observed bursts of emission. Because of the tiny cross-sectional area of the fibres the bursts were frequently small and the corresponding decay curves rather weak. Nevertheless, per original sample cross-sectional area, the Kevlar fibres were the most intense electron and ion emitters we have observed. Total particle counts are provided with each emission curve and in the tables.

The experiments were carried out in a vacuum chamber pumped by a diffusion pump with a liquid nitrogen cold trap; the background pressure in the chamber was 1×10^{-5} Pa.

The nature of the broken fibre was qualitatively characterized in terms of the degree of damage to the fibre by means of optical microscopy. For clarity, only one side of the broken fibre was photographed; to the eye the extent of damage on both sides was approximately the same. The fibres were photographed at a magnification of × 200 using a Zeiss Photomicroscope III. The "damage" was then qualitatively correlated with the intensity and time distributions of the EE and PIE from each fibre.

3. Results and discussion

Typical results of EE measurements from the purely tensile fracture of Kevlar fibres are presented in Figs. 1a and b. Photographs of the corresponding fracture surfaces are shown in Figs. 1c and 1d, respectively. As can be seen from the photographs, the fibres generally fractured into individual fibrils, a well known tendency of Kevlar fibres [7–12]. The extent of splitting is seen to vary qualitatively between Figs. 1c and 1d. In Fig. 1c, the fibre fracture involves limited splitting, and the corresponding EE is of very short duration and in the form of two short bursts with a total of 308 counts. On the other hand, the fibre in Fig. 1d is extensively split after fracture. The EE curve in Fig. 1b shows more intense, multiple bursts, characteristic of a more complicated fracture process. The emission occurred in four distinct bursts each longer in duration than the bursts in Fig. 1a. From examination of Fig. 1d, one can see that substantially more splitting and fibril fracture has occurred.

Similar results were obtained from the PIE measurements. The PIE, on the time scale shown in Fig. 2a, is characterized by multiple, long lasting bursts. Note the PIE build-up and decay of the second burst in Fig. 2a (about 100 μsec). This relatively slow build-up of the emission is most likely an indication of the time required for the splitting or separation process for the formation of the individual fibrils, while the decay time or after emission is due to thermally stimulated relaxation of the fracture surfaces [3]. The extensive fibril formation corresponding to these bursts of charge can be seen in Fig. 2b. It is obvious from Figs. 1 and 2 that both EE and PIE are more intense when fibres fracture into separate fibrils.

These emission curves and photographs (Figs. 1 and 2) show the complicated forms of fracture that occur from the tensile deformation of Kevlar single fibres. In Table I we have summarized the total emission counts for a 1 msec interval during and after fracture, the number of *distinguishable* EE or PIE bursts (peaks), and the time duration between the occurrence of the first burst and the last burst. This time duration is an indication of the time from initial fibre damage to final failure for the slow strain rate used in this experiment. Under these loading conditions, it shows that substantial damage events are occurring over a time scale *much*

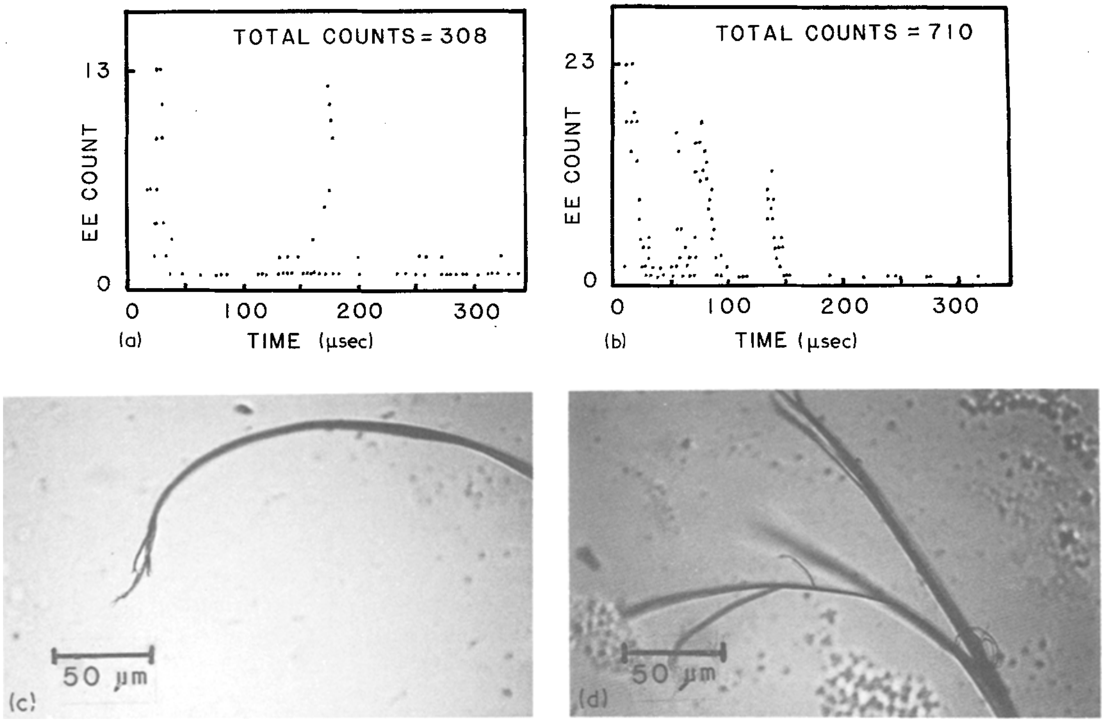

Figure 1 Electron emission and photographs of fracture surfaces of Kevlar single fibres fractured in pure tension: (a), (c) limited splitting, and (b), (d) extensive splitting of fibrils.

longer than simple dynamic crack propagation through the cross-section of the fibre. In general, from the parameters in Table I, we see that when the total emission for either EE or PIE is high, the splitting and formation of fibrils is significant, and there is evidence of extensive plastic deformation (as observed with the optical microscope). Also, a high total emission count was generally accompanied by bursts of EE or PIE with long decay. We tentatively suggest that long after-emission may be related to a higher degree of fibril formation and/or the accompanying plastic deformation, which in itself involves extensive damage.

This form of fracture, namely the splitting and formation of fibrils, was previously seen by Bunsell [8], Konopasek and Hearle [10] and Chiao *et al.* [11] while testing Kevlar/epoxy strands in tension. Chiao *et al.* also detected particles or defects within the fibrils which did not appear to be artifacts, but were an inherent characteristic of the fibre. Owing to the presence of these defects, some fibrils fracture at a lower stress level than others, thus producing an initial

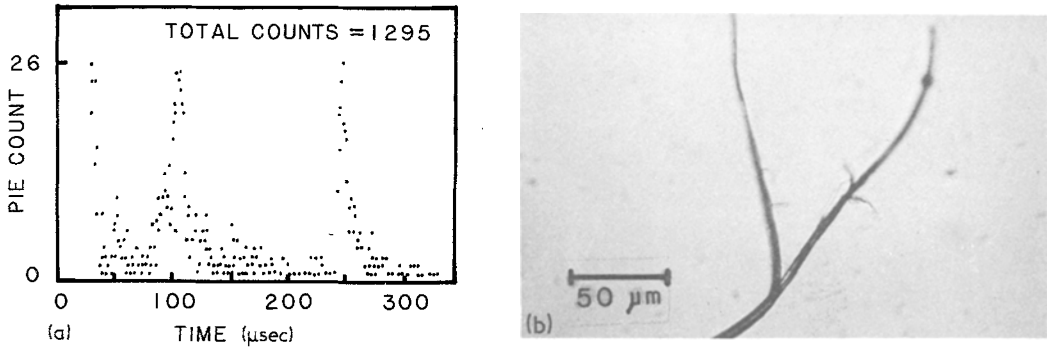

Figure 2 Positive ion emission and photographs of the corresponding fracture surfaces of a Kevlar single fibre fractured in pure tension.

TABLE I Selected parameters for electron and positive ion emission from the fracture of Kevlar single fibres

Emission type	Total emission counts per 1 msec	Number of bursts	Time duration* (μsec)
EE	308	5	681
	615	4	125
	640	3	209
	860	4	116
	576	3	132
	264	2	147
	429	3	111
PIE	1265	3	219
	195	1	–
	882	2	19
	744	4	59

*Time between the occurrence of the first and last burst.

fracture in one part of the fibre, followed by failure of the stronger fibrils at later times. Antel [12] has found evidence that fibrils, in fact, extrude or pull out of sockets much like "fingers out of gloves". Either form of fracture might thus yield successive bursts of emission over measurable time periods. At this point, we can not distinguish clearly the order of events producing the sequence of EE bursts that we observe.

Fig. 3 shows typical EE measurements and corresponding photographs of Kevlar single fibres stretched and broken over the dull metal edge. Similar results of PIE measurements and photographs of their respective fracture surfaces are presented in Fig. 4. Although each graph or photograph in Figs. 3 or 4 is for a particular fibre, they are representative of approximately fifty samples we studied.

For samples broken across the metal edge, three distinctive forms of fracture can be qualitatively distinguished by the examination of the emission curves and the photographs. The first type was characterized by low total emission followed by a rapid decay; this corresponds to a "clean" fracture where the fibre cleaved relatively cleanly across its cross-section (see Figs. 3a and b, 4a and b). Second, a "splitting" type of fracture was observed which was accompanied by greater counts of both EE and PIE, with longer decay times (see Figs. 3c and d, 4c and d, and Table II). This form of fracture was the type that dominated when fibres were fractured in pure tension. In the third form of fracture, the fibre broke mainly across its cross-section, but the resulting fracture surfaces are frayed. Such "frayed" fracture was characterized by several bursts of electrons and positive ions, with some bursts lasting up to 200 μsec (see Figs. 3e and f, 4e and f).

Table II presents average values of the parameters: total counts, number of bursts, and time duration to characterize typical behaviour for each type of fracture.

In highly strained Kevlar fibres the extended polymer chains along the fibril axes are covalently bonded, whereas the fibrils are probably held to their surroundings by hydrogen bonds [13]. When the fibrils separate or pull out of their surroundings a large number of the weak hydrogen bonds are broken. When the fibre fracture is "clean", a limited number of covalent bonds and very few hydrogen bonds would be broken. In addition, the newly created fracture surface area would be small. Therefore, the total emission count is low with a rapid decay (see Figs. 3a and 4a). In cases where fibrils are formed extensively the total number of failed covalent bonds may be no higher than that found in the "clean" fracture, but a very large number of hydrogen bonds are broken as well as considerably more surface area formed. The more intense emission may thus be due to both the larger fracture surface created as well as to the possibility that fibril formation and/or pull-out may produce

TABLE II Averages of selected parameters of EE and PIE for various forms of Kevlar-49 single-fibre fracture (fibres stressed over metal edge)

Fracture microstructure	Emission type	Total emission counts per 1 msec	Number of bursts	Time duration* (μsec)
Clean	EE	334	3	15
	PIE	42	1	–
Stranded	EE	470	2	25
	PIE	641	2	6
Frayed	EE	1168	6	5390
	PIE	768	4	470

*Time between the occurrence of the first and last burst.

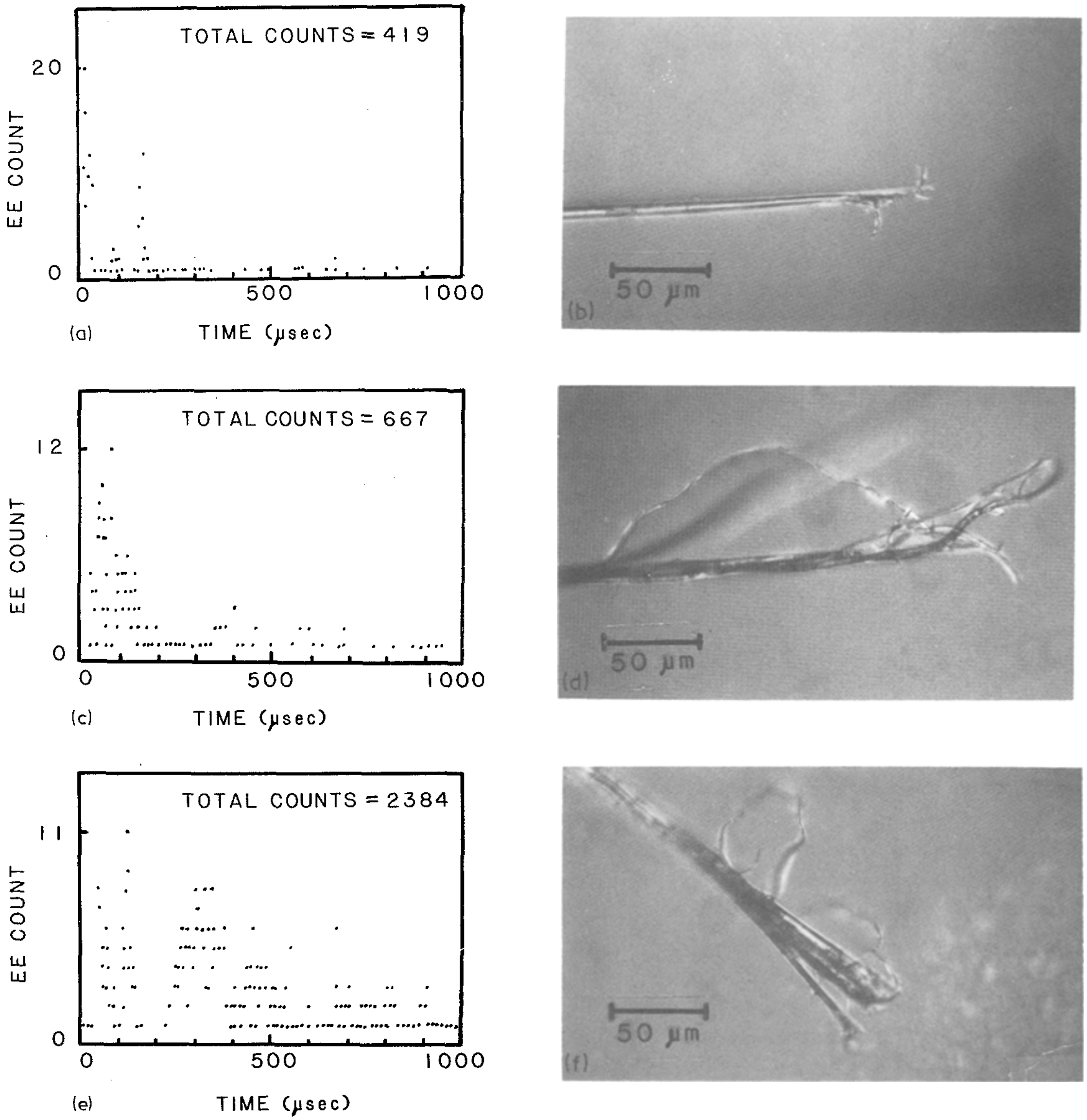

Figure 3 Electron emission and photographs of the corresponding fracture surfaces of Kevlar single fibres fractured across a dull metal edge: (a), (b) flat; (c), (d) stranded; and (e), (f) frayed fracture.

charge separation. We have shown that charge separation during fracture plays a key role in the emission mechanism; in general, when charge separation is intense so is EE and PIE, e.g. in cases of delamination or interfacial failure, which produces intense emission [1–5, 14, 15]. Thus the FE may be providing signals indicative of the manner in which the fibres are failing.

4. Conclusions

The emission of electrons and positive ions from the fracture of single fibres of Kevlar has been examined on the microsecond time scale. Evidence of the time required from initial damage to ultimate failure of the fibre has been provided, showing times ranging from a few microseconds to several hundred microseconds. Secondly, the total emission from the entire fracture event tends to correlate with the extent of "damage" to the fibre produced by fracture. Finally, by examination of the shape and intensity of the EE/PIE bursts, it may be possible to differentiate between fibril formation and fibril pull-out. A measurement of other EE/PIE characteristics such as kinetic energy or use of other FE components (e.g. neutral emission or photon emission)

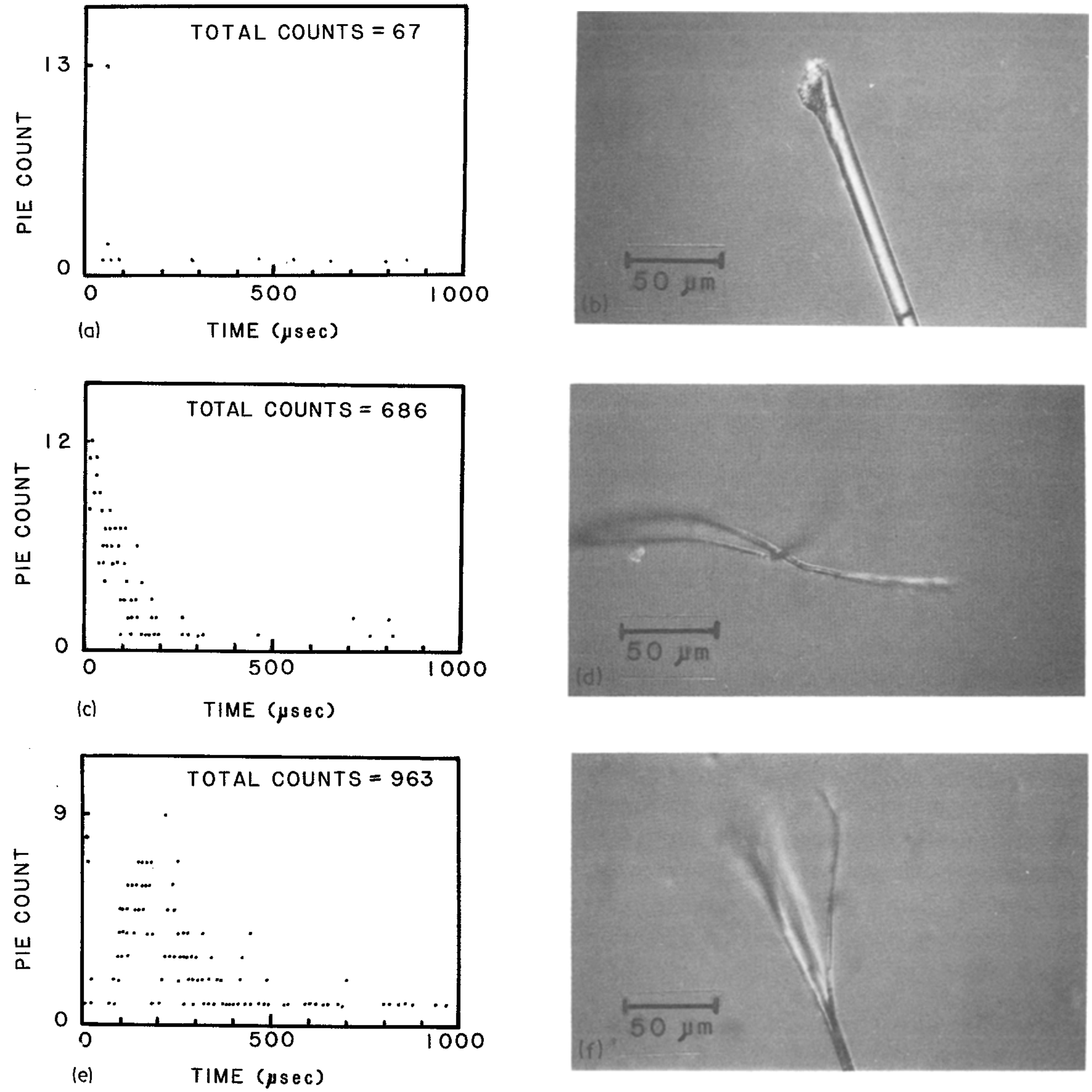

Figure 4 Positive ion emission and photographs of the corresponding fracture surfaces of Kevlar single fibres fractured across a dull metal edge: (a), (b) flat; (c), (d) stranded, and (e), (f) frayed fracture.

might provide more deformation information on the fracture processes occurring where such fibres are stressed to failure.

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