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Critical length measurements in carbon fibers during single fiber fragmentation tests using acoustic emission

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The single fiber fragmentation test (SFFT) is a well-known experimental test, which allows the study of fiber failure in its simplest form [1, 2]. The principle is that a single fiber embedded in a matrix repeatedly breaks until all fragment lengths are within a critical distance. Once the fiber has broken for the first time, successive breakage happens in regions removed from the initial failure, often referred to as *polled areas* [3]. Acoustic emission (AE) has been extensively used for the detection and localization of fiber breakage during SFFT tests, in carbon [4, 5], Kevlar [6, 7] and glass fiber composites [8, 9]. In all cases, in spite of the small diameter of the fibers, nearly all fiber breakages appeared to have been detected and associated with a single acoustic emission event, an occurrence that has been defined as *one-to-one correspondence* [10]. Moreover, the number of acoustic emission events detected corresponded with considerable accuracy with the fiber breakages observed by other methods e.g., optical microscopy [11]. This allowed the determination of Weibull statistical strength parameters for the fibers based on the number of acoustic emission hits detected [12]. From the above parameters and the average length of fiber fragment, the interfacial strength can be determined, assuming that the fibers are constant in diameter, which is acceptable for glass and carbon fibers, much less so for brittle e.g., ceramic, monofilaments [13].

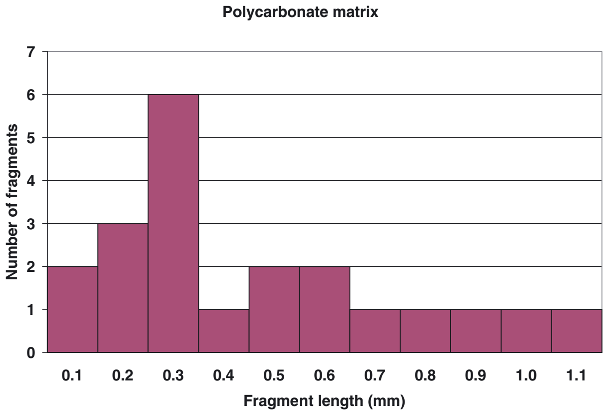
When dealing with localization of acoustic emission events, a considerable number of issues arise. Ideally, in an environment with sufficient signal to noise ratio, two sensors can be calibrated for localization, using at every location the correct value for wave velocity, which may vary up to 10% with the applied stress [14]. If this is the case, differences in wave arrival time at the sensors of less than a microsecond can be measured, so that AE events can be localized with accuracy in the order of 0.1 mm. This can be achieved by most recent acoustic emission systems by placing the sensors at a mutual separation distance not exceeding 30–40 mm, therefore compatible with the most common gauge lengths applied in SFFT tests.

In this work, a single T300 high strength carbon fiber filament (7 μm diameter) was embedded in three different polymeric matrices (polycarbonate, polyurethane and epoxy). A total number of fifteen dog-bone specimens, five per matrix, were prepared, each with total length 45 mm. Tests were performed in displacement control mode with a crosshead speed equal to 0.13 mm/min. During these tests, acoustic emission was continuously monitored using pairs of piezoelectric microsensors (5 mm diameter), resonant at 300 kHz, placed at a mutual distance center-to-center of 30 mm, each 15 mm from the middle of the sample. AE data were treated using a PAC Mistras 2000 system. Only single burst signals have been considered, triggering the two channels i.e., yielding an AE event in an interval of less than 11.5 μs, corresponding to the time at which the 30 mm distance between the sensors is covered at the wave speed, measured along the carbon fiber, of 2600 m/s.

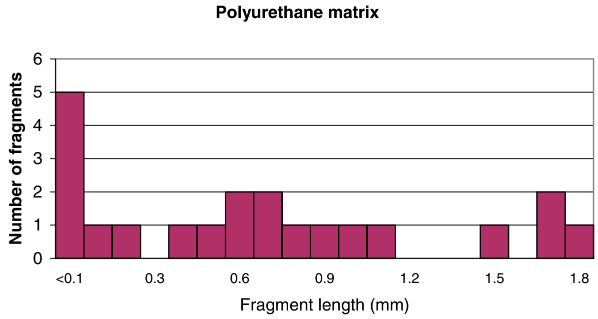
The strain measured on the fibers during SFFT tests was 1.02 ($\pm$0.20)%. A number of conditions need to be verified in order for the test to be valid for critical fiber length and Weibull statistical measurements. These include fiber and matrix both behaving linearly and elastically during fragmentation, fiber-matrix adhesion being sufficiently strong, so that no pullout occurs [15], and the interface being of the same thickness across the composite [16]. It has been recently observed that the performance of SFFT tests at higher strain rates, in place of giving shorter fiber fragments as the major effect, induces a larger occurrence of creep, so that the test is no longer valid for fracture mechanics purposes [17].

The low strain rate adopted resulted in a linear elastic behavior of fibers and matrix, although in some cases fiber-matrix adhesion was not as strong as expected, and this gave rise to early pullout of the fibers, even if no significant torsion of the samples was observed. As a consequence, results from the specimens showing early pullout were not considered for analysis. Weibull parameters measurements can still be fairly accurate, since their values were shown, as reported below, not to change very much with the polymer used as matrix in the SFFT samples. However, fragment length distribution may differ considerably, being less or more scattered, as can be observed by comparing Fig. 1a, for a polycarbonate specimen, with Fig. 1b, for a polyurethane specimen.

Acoustic emission counts of the signals associated with fiber breakages (Fig. 2a) appear to be only slightly affected by the position of the fiber: to work out a possible trend, further testing would be needed. Even more dubious appears the relation of AE energy, expressed in units representing the envelope area under the signal, with the level of strain on the fiber (Fig. 2b). This indicates that the measured acoustic energy does not exactly represent the stored elastic energy in the segment



(a)



(b)

Figure 1 (a) Length distribution of T300 fiber fragments in a polycarbonate matrix. (b) Length distribution of T300 fiber fragments in a polyurethane matrix.

at failure, because in the latter case it should be proportional to the stress on the fiber [18].

Analyzing according to a two-parameter Weibull distribution, a number of approaches have been developed to predict the critical fiber length from measurements of fiber fragment length obtained using acoustic emission. The theory developed in [19] was adopted, that, neglecting the effect of fiber ends shielding possible flaw sites from fracture, as is possible in a low stress region, gives for the number of fiber breaks N over a gauge length L_o this simple power law:

$$N\,(L_o) = \left(\frac{\sigma_f}{\alpha_o}\right)^{\beta} \tag{1}$$

where σ_f is fiber stress and α_o and β are size and shape parameters. Since in this work, strain ε_a is measured in place of stress, and from the former, stress σ_f is obtained as $\sigma_f = \varepsilon_a E_f$.

In this way Equation 1 can be written as $\ln N = \beta \ln \varepsilon_a E_f - \beta \ln \alpha_o$, so that β can be determined from the slope of the ln N vs. ε_a, and α_o from its intercept at ln $N = 0$, as suggested in [11]. Typical curves obtained for the three matrices are represented in Fig. 3. The difference in slope observed between the initial and the following part of the curve has to be ascribed to the combination of two effects, the initial irregularities in loading behavior and the occurrence of creep after the fiber underwent the first few failures [11]. The presence of both effects would rather suggest considering a linear regression of the whole curve than excluding a part of it. The inaccuracy of this measurement can be reduced with the alternative method described below.

In place of considering $N(L_o)$, α_o and β can be determined using the mean fiber fragment length $l(\sigma)$, obtained from acoustic emission localization:

$$l(\sigma) = \left(\frac{\sigma}{\alpha_o}\right)^{\beta} \tag{2}$$

Hence, adopting the suggestion from [20] over the Kelly-Tyson [15] expression for maximum critical fiber length l_c, that the actual fragment lengths vary from 0.5 l_c to l_c, so that, assuming a Gaussian distribution, the mean fragment length l is 0.75 l_c, the interfacial yield stress can be obtained from:

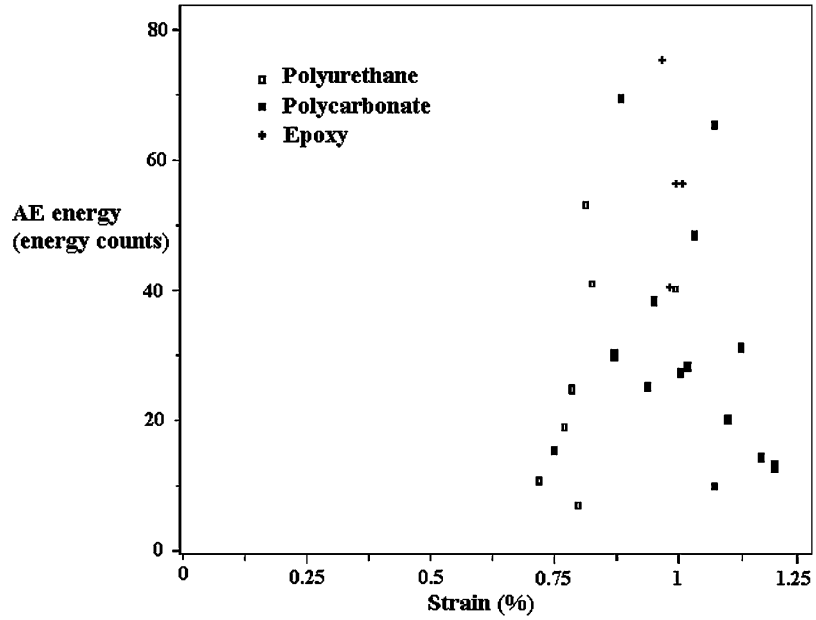
$$\tau = \frac{d\sigma_f}{2\ell} 0.75 \tag{3}$$

The values obtained from acoustic emission localization for α_o, β, $l(\sigma)$ and τ using Equations 1–3 are reported in Table I.

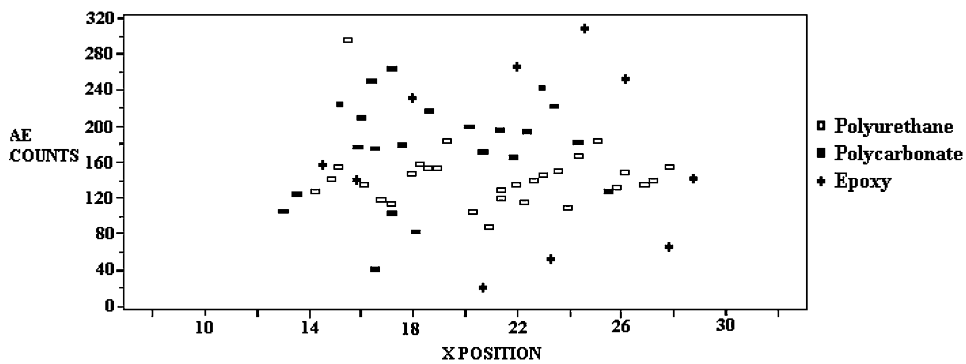
Comments on the results obtained should refer first to the fact that, as previously observed [14], the matrix has very little effect on the values of Weibull parameters: however, in these tests they appear to be substantially lower than those measured for T300 fibers in [10]. This should rather be ascribed to the experimental set-up adopted for tensile tests, which was not able to completely rule out a slight torsion of the samples. Less important appeared to be the limitation of obtaining fiber stress from an indirect measurement of strain, because the effect of matrix cracks or pullout on stress-strain linearity has been shown to be negligible for high strength carbon fibers [21]. In addition, the accuracy of acoustic emission prediction of Weibull values was observed to grow with the number of breakages in each fiber [22]. In these tests, although a visual one-to-one correspondence between acoustic emission events and fiber breaks was observed, the number of fiber breakages was probably not high enough to offer a good level of accuracy. In spite of this, the evolution of acoustic emission technique leading to more accurate systems, involving a measurement of real fracture energy during the tests, may allow integration of Weibull parameter results with other information on the single fiber breakage. This would ultimately contribute to a fuller understanding of the process of fiber fragmentation and to the local measurement of stresses at fiber-matrix interface.

TABLE I Values obtained for Weibull parameters, average fragment length, interface stress and strain at failure

Sample	α	β	L_{av} (mm)	ε_{max} (%)	τ (MPa)
PC 1	13 590	6.87	0.44	0.986	13.9
PC 2	15 800	5.58	1.54	0.883	3.4
PU 1	13 100	11.23	0.61	0.720	7.1
PU 2	14 580	6.89	0.48	0.845	10.5
EP 1	15 190	12.25	0.47	0.872	11.1
EP 2	16 300	11.66	1.37	0.883	3.9



(a)



(b)

Figure 2 (a) Energies of acoustic emission events in three SFFT samples, one per matrix. (b) Counts of acoustic emission events in three SFFT samples, one per matrix.

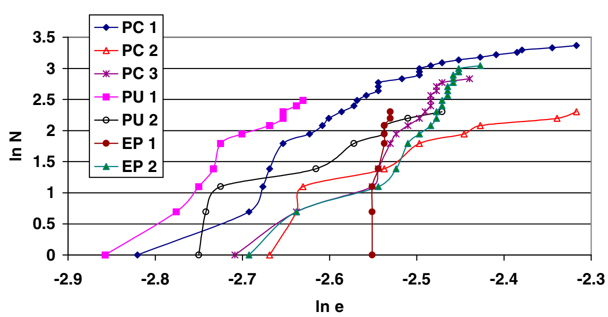


Figure 3 Typical curves obtained from acoustic emission study of fiber fragmentation (N = number of fiber breakages).

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