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February 1987

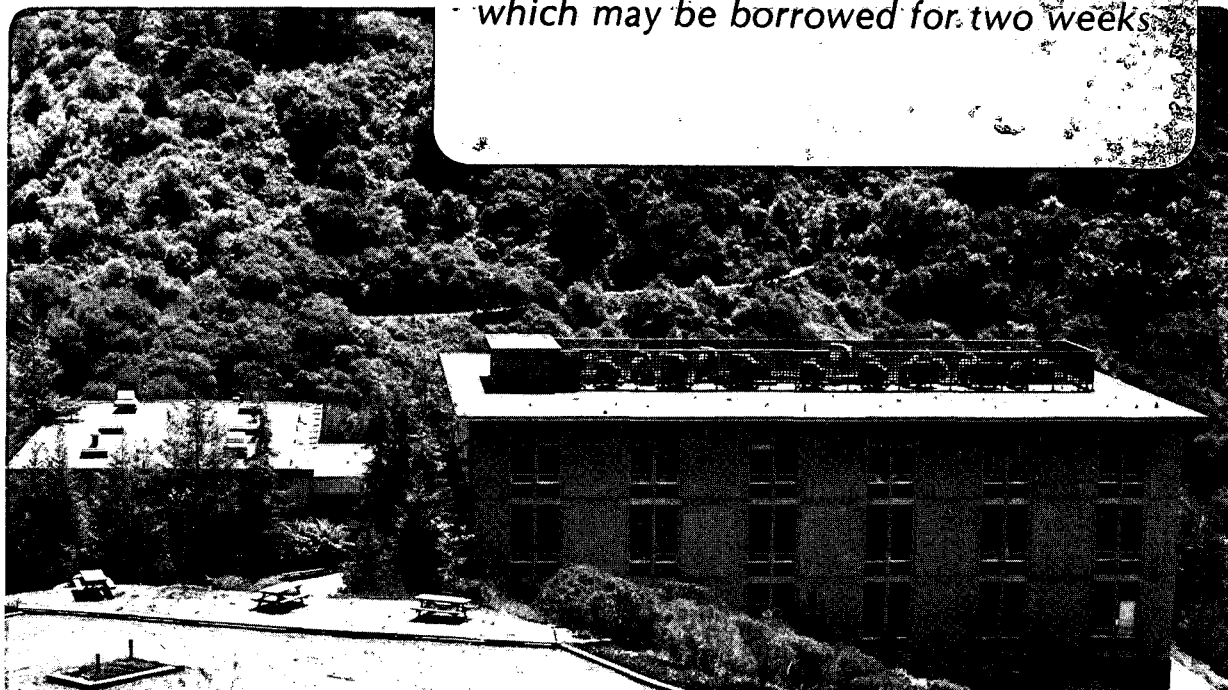
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DELAMINATION FRACTURE AND ACOUSTIC EMISSION IN CARBON, ARAMID AND GLASS-EPOXY COMPOSITES

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ABSTRACT

The results of an investigation into the opening mode (Mode I) delamination fracture behavior of carbon, aramid and glass-epoxy composites are described. The effect of loading rate and reinforcement geometry (unidirectional vs woven) on fracture toughness was determined, and observation of the fracture surface was used to derive possible microfailure modes. The results show that the crack energy release rate for a woven composite was greater than that of unidirectionally reinforced composites. Acoustic emission was employed to detect crack initiation in an attempt to obtain correlation with the delamination fracture toughness. The earliest signal appeared to correlate well with the delamination fracture toughness, indicating that the processes involved in fracture initiation determines the magnitude of the steady state fracture toughness. The results of this investigation show that the three different composite materials tested behave in different ways (i.e., have different failure modes) during delamination, suggesting that a single theory cannot be expected to explain delamination in all composite materials.

INTRODUCTION

Delamination fracture is an important failure mode in laminated composite materials. It is associated with failure of the weakest components of the composite material - the matrix and the fiber-matrix interface. Delamination is often the limiting factor in the use of composites for structural applications, particularly for long term use, where thermal cycling, mechanical fatigue and environmental effects such as moisture are present. It can occur at stress concentrations such as holes, material discontinuities such as ply drop-offs, or at joints. The study of delamination fracture, therefore, has drawn increasing attention. Recently, an entire conference was devoted to the subject (Johnson, 1985).

In this paper, the results of an investigation into the

opening mode (Mode I) delamination fracture behavior of carbon and aramid-epoxy composites are described. The effect of loading rate and reinforcement geometry (unidirectional vs woven) on fracture toughness was determined, and observation of the fracture surface was used to derive possible microfailure modes. A micromechanical delamination model, proposed in an earlier investigation is used to estimate the relative contributions of the matrix and the fiber-matrix interface to the overall delamination fracture toughness (Saghizadeh and Dharan, 1986). Finally, acoustic emission was employed to detect crack initiation in an attempt to obtain correlation with the delamination fracture toughness.

EXPERIMENTAL PROCEDURE AND ANALYSIS

The double cantilever beam (DCB) specimen was used to characterize Mode I behavior. The Mode I specimen shown in Fig. 1 is a DCB specimen with bonded hinged metal tabs attached at one end for the application of load perpendicular to the interlaminar layer. Initiation of delamination along the interlaminar layer was accomplished by the insertion of a piece of polytetrafluoroethylene (PTFE) tape at the desired interface at the time of fabrication. In all specimens, the PTFE tape was located at the mid-thickness of the laminate also shown in Fig. 1.

Laminates were constructed from woven and unidirectional Union Carbide's Thornel 300/Fiberite 934 carbon pre-preg, from woven Dupont's Kevlar/Fiberite 934 pre-preg and woven glass/Fiberite 934 pre-preg. The weave for the carbon, glass and aramid materials was a square weave with 16.5 tows/cm in each direction (42x42 tows/square inch). The laminates were prepared as 300 mm square plates, 2.2 mm to 2.33 mm thick. The unidirectional material was layed up with all plies at 0 degrees and the woven material was layed up in the 0/90 direction. In addition, laminates were prepared with two epoxy films at the mid-plane, with the PTFE tape between the films. This was done to measure the in-situ delamination fracture toughness of the matrix.

All the laminates were cured in an autoclave at 170 C for 4 hours at 0.7 MPa pressure. The resulting fiber volume was found to be 65 ± 1 per cent. After cure, specimens were cut from the plate using an abrasive cutter. The fiber volume fraction was determined by the acid digestion method for the carbon composites and by the solvent technique for the aramid composites and was found to be 65 ± 1 per cent. Hinged metal end tabs were adhesively bonded at the ends of the specimen as shown in Fig. 1. The lateral edges of the specimens were marked at 0.5 cm increments prior to testing as an aid in the crack length measurements.

The specimens were loaded at constant cross-head velocity in an Instron machine. Initially, before crack length measurements were made, each specimen was loaded to grow the embedded crack (initiated from the PTFE tape) to an initial crack approximately 30 mm long. The specimen was loaded at constant crosshead

velocity until the crack just began to propagate. The specimen was then unloaded and the process repeated until the crack grew to about 75 percent of the specimen length. The crack length was measured during the re-loading cycle just before crack propagation began.

The acoustic emission (AE) response during fracture was monitored using an Acoustic Emission Technology Model AET-5000 system, in which the AE transducer was placed at the specimen end opposite to the load application end. Fig. 2 shows the arrangement of the instrumentation. The signals were recorded on videotape and also viewed in real time on an oscilloscope. Scanning electron microscopy (SEM) was used to observe the fracture surfaces and identify the failure modes.

For Mode I crack propagation, linear elastic fracture mechanics (LEFM) gives the crack energy release rate

$$G_{IC} = (P_C^2 / 2w) (dC/da) \quad (1)$$

where P_C is the critical load at which crack propagation begins, w is the width of the specimen, C is the specimen compliance, and a is the crack length. Using a compliance calibration of the specimen, G_{IC} can be determined from Eq. (1) (Saghizadeh and Dharan, 1986).

In the foregoing, there are the implicit assumptions that shear deformations are small, and that viscoelastic effects are restricted to a small zone at the crack tip. Whitney et. al. (1982) have derived an expression for the DCB specimen for the crack energy release rate in which shear deformation is included. However, the shear deformation effect can be neglected for crack lengths that are large relative to the thickness of the specimen, which is the case in the current investigation. Viscoelastic effects in delamination have been studied by Devitt et. al. (1980) for Mode I delamination fracture and were found to be negligible if viscoelasticity were to be restricted to a small region near the crack tip.

RESULTS AND DISCUSSION

Table 1 shows crack energy release rates for the materials tested. Also shown are data for unidirectional glass-reinforced epoxy reported by Devitt et. al. (1980), and for unidirectional carbon-reinforced epoxy (Thornel 300/Fiberite 934) reported by Garg and Ishai (1984). It is evident that increasing the loading rate by a factor of ten results in an increase in the crack energy release rate by about 15 per cent in the three types of laminates tested and the epoxy layer. Thus, there is a rate dependent effect in delamination fracture.

There is also an increase in the crack energy release rate for a woven composite compared to a unidirectionally reinforced composite for carbon composites (the only material tested in both woven and unwoven configurations). This can be explained by observations of the fracture surface.

TABLE 1 - Mode I Crack Energy Release Rates (J/m^2).

	<u>Loading Rate (mm/sec)</u>	0.083	0.83
1. Unidirectional carbon-epoxy (Thornel 300/934)	90	---	---
	103 *	---	---
2. Woven carbon-epoxy (Thornel 300/934)	232		266
3. Woven aramid-epoxy (Kevlar)/934	330		382
4. In-situ epoxy layer	164		191
5. Unidirectional glass-epoxy	525 - 1020 **	---	---

* Data from Garg and Ishai (1984)

** Data from Devitt et. al. (1980)

The Fiberite 934 resin system is relatively brittle at room temperature. In the Mode I delamination tests conducted in this investigation, there was no fiber participation in the fracture process. The delamination crack propagated in the matrix between the fibers and along the fiber-matrix interface. The fracture surface for the Thornel 300/934 composites is characterized by clean fiber surfaces exposed by the crack, indicating low interfacial energies. Similar fracture morphology was reported by Richards-Frandsen and Naerheim (1983) for delamination of carbon-epoxy composites. The fracture surface showed the characteristic exposed clean fiber surface with traces of removed fibers separated by the fractured epoxy matrix surface which was characterized by hackles.

In Mode I crack propagation in unidirectionally reinforced composites, locally, at the crack tip, the crack has to work around the embedded fibers as it propagates. Locally, therefore, the fracture process involves both Mode I and Mode III components for unidirectional laminates in which the "macroscopic" opening mode crack front propagates parallel to the fiber direction. This is discussed in more detail in the model proposed in the next section.

For woven laminates, due to the waviness of the weave, the crack front is more convoluted than in the unidirectionally reinforced material, and, locally, all three fracture modes are present. This observation, and the fact that the fracture surface is larger on a local (microscopic) level than the projected area that is used to calculate the fracture toughness, could account for the increase in the fracture toughness of woven composites over that of unidirectional composites. For carbon-epoxy composites, the woven geometry increases the crack energy release rate by a factor of about 2.5 over the unidirectional material.

The woven Kevlar/934 laminate showed a higher fracture toughness than the woven carbon laminates. Scanning electron microscopy showed that, like the carbon composites, there is very little evidence of matrix adhesion to the fiber surfaces exposed by the delamination crack. However, one also observes the presence of several broken fibrils (Saghizadeh and Dharan, 1986). The well known tendency of aramid fibers to fibrillate, ie. split longitudinally into fibrils, may account for the higher delamination fracture energy observed in aramid

composites. A possible mechanism is that, as the opening mode delamination crack propagates, some fibrils are separated and bridge the crack. As the crack propagates, these fibrils have to fracture, thus introducing a contribution to the delamination fracture energy from the fibers, in addition to the normal matrix and interfacial failures.

Finally, the crack energy release rates for the epoxy alone, measured in-situ by using molded-in thin resin films, show that, for the carbon composites, the addition of fibers results in lowering of the overall opening mode fracture toughness. For the glass composites, however, the addition of fibers results in an increase in the composite fracture toughness. This suggests that the interfacial strengths of the carbon fiber is significantly less than that for the glass composites. This result and the volume fraction dependence of the opening mode fracture toughness was derived using a micromechanics model in an earlier investigation and will not be repeated here (Saghizadeh and Dharan, 1986).

The acoustic emission results for carbon, aramid and glass-epoxy composites during opening mode fracture are shown in Figs. 3 and 4. Fig. 3 shows the number of events increasing as one goes from carbon to aramid to glass composites. This is true for other measures of acoustic emission such as the cumulative number of events for all three materials, and reflects the general correlation with the measured increasing opening mode fracture toughness in the three composites. The foregoing data were obtained during steady-state crack propagation. However, careful observation revealed that, long before steady-state propagation begins, there is evidence of a small burst of acoustic emission energy. This is shown in Fig. 4 for the three materials, and were recorded during the loading phase of the test.

Fig. 5 shows schematically the observation of AE activity at a load (A) corresponding to the point on the load deflection curve that is well below the peak value (B) when steady-state crack propagation begins. This observation suggests that microfailures are initiated at load levels well below the load required for crack propagation to begin. When the fracture toughnesses corresponding to these loads (corresponding to failure initiation) are plotted against the number of AE counts per unit crack area (measured during steady state crack propagation), a correlation appears to exist as shown in Fig. 6. Here a straight line on a log-log plot fits the data for these three very different composites. This results suggests an empirical relationship between the opening mode fracture toughness $G_{Ic}(\text{AE initiation})$ corresponding to failure initiation and the AE counts per unit crack area, N :

$$N = C [G_{Ic}(\text{AE initiation})]^m$$

where $m = 0.91$.

CONCLUSIONS

1. The results show that the Mode I delamination fracture toughness of carbon composites is small relative to glass composites. Measured values of 90 J/m^2 were obtained for unidirectional T-300/934 compared with $525 - 1020 \text{ J/m}^2$ for glass-epoxy obtained by other investigators.

2. The effect of loading rate was to increase the crack energy release rate by about 15 per cent for a ten-fold increase in the loading rate. This relative increase was obtained for all the materials tested.

3. The fracture surface of the carbon-epoxy laminates was characterized by clean exposed fiber surfaces indicating poor interfacial fracture toughness. The aramid composites also showed relatively clean fiber surfaces; however, some fibrillation was observed. The fibrillation could account for the higher fracture toughness of the aramid composite over the carbon-epoxy material because it introduces some fiber participation in the delamination process.

4. The acoustic emission data show that the number of events and the AE energy during steady state opening mode crack propagation increase from carbon to aramid to glass composites reflecting the ranking observed during the measurement of the delamination fracture toughness.

5. Acoustic emission activity corresponding to microfailure initiation was observed in all three materials at load levels significantly lower than that required for steady state crack propagation.

6. A correlation was obtained between the fracture toughness corresponding to failure initiation and the number of AE counts per unit crack area during steady state crack propagation for carbon, aramid and glass-epoxy composites suggesting that initiation processes during loading appear to determine the steady state fracture response during delamination fracture of carbon, aramid and glass-epoxy composites.

ACKNOWLEDGEMENT

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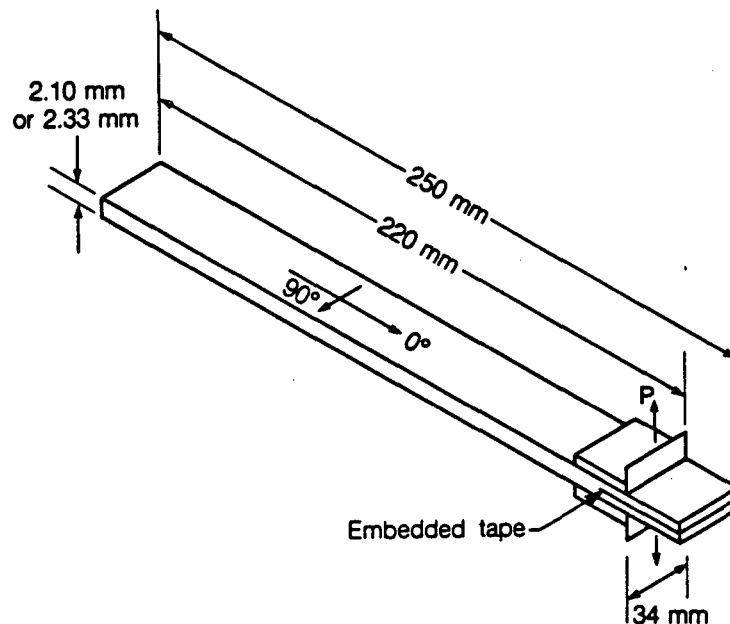


Fig. 1. Specimen geometry and dimensions.

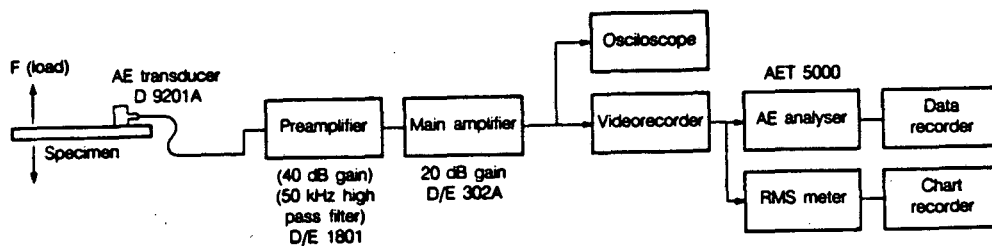


Fig. 2. Instrumentation for acoustic emission measurement during opening mode crack propagation.

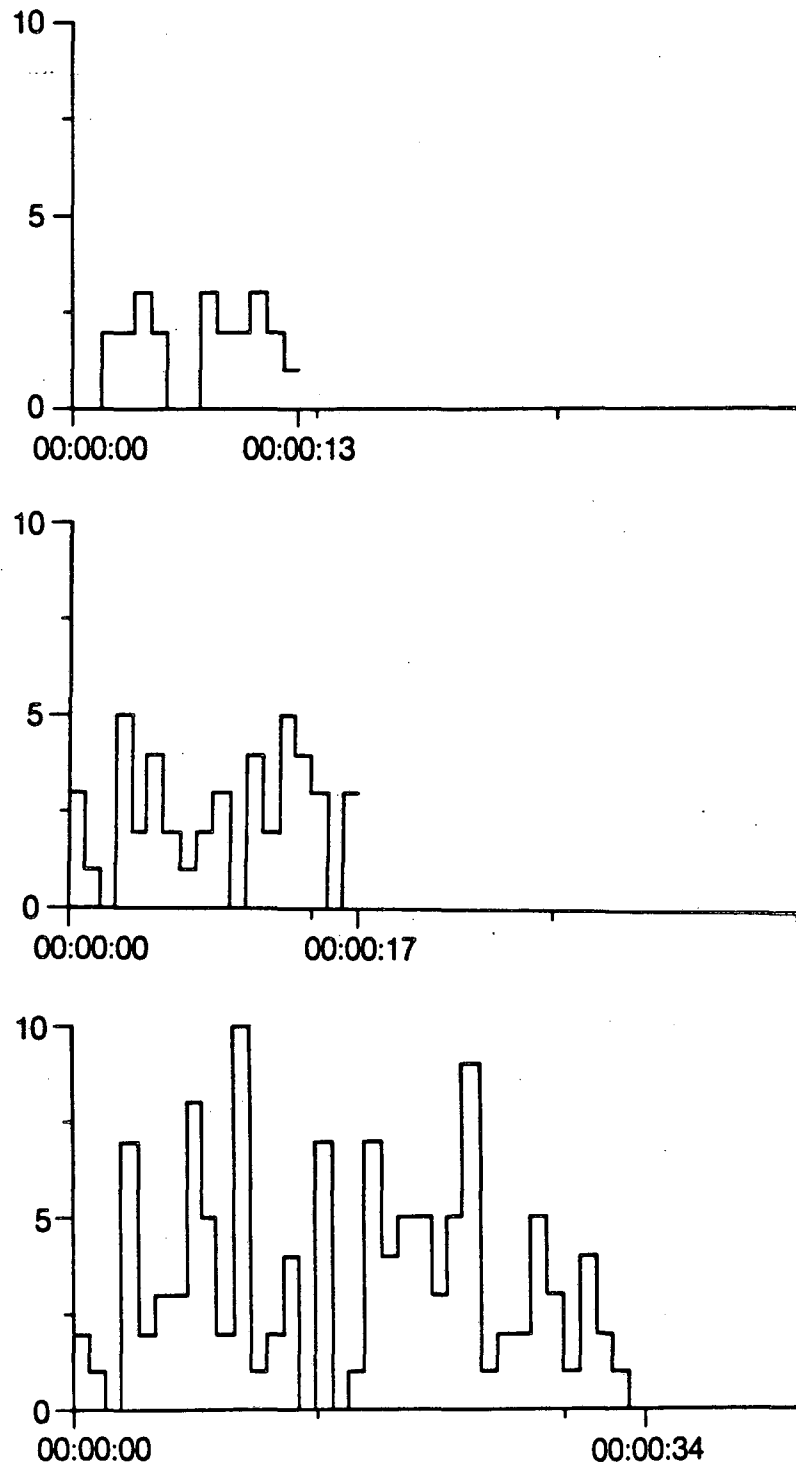


Fig. 3. Acoustic emission events (during steady-state opening mode delamination) vs time for graphite, aramid and glass composites, respectively (top to bottom). Interval size = 1 sec, number of intervals = 60).

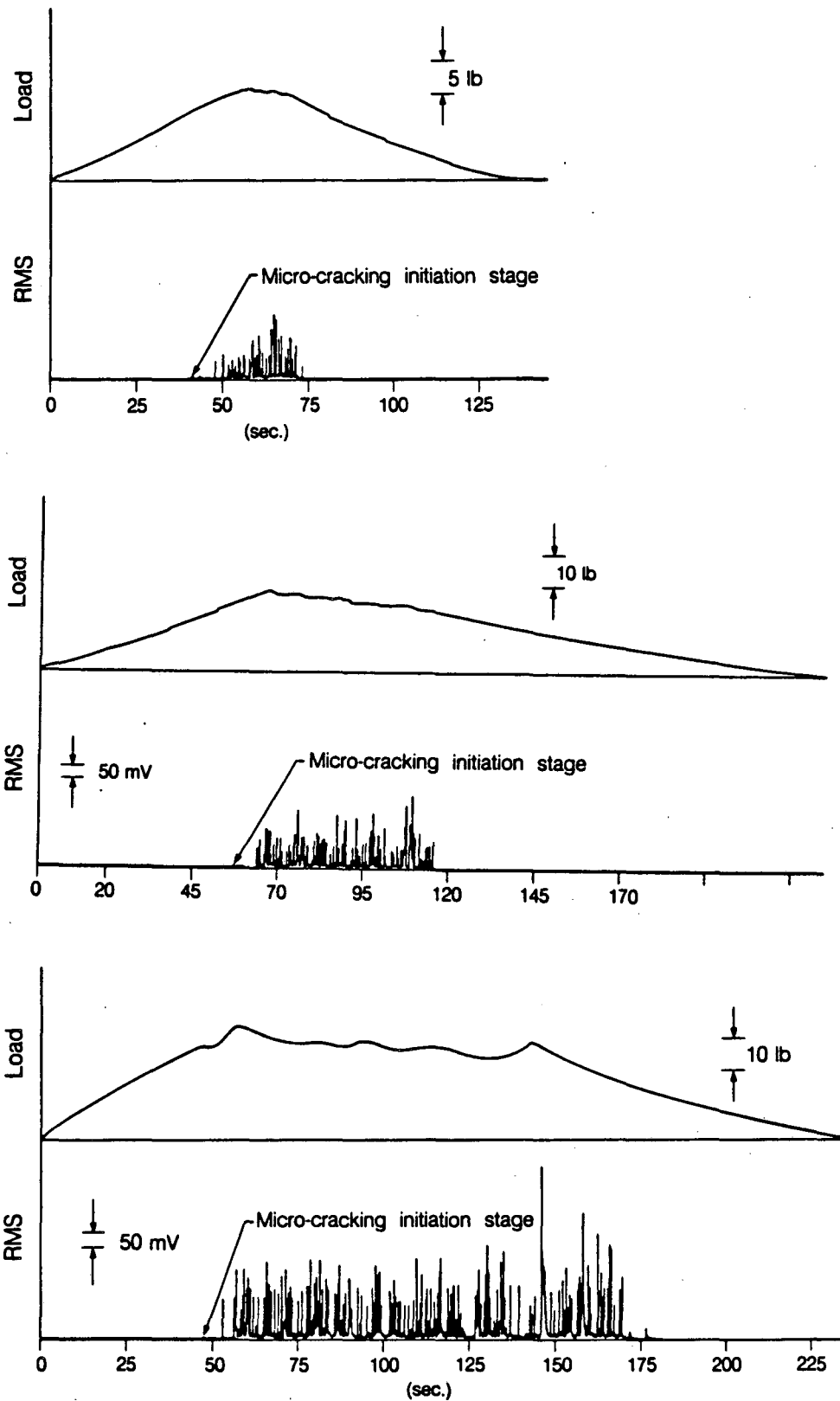


Fig. 4. Load and AE (rms) vs time for carbon, aramid and glass-epoxy composites, respectively (top to bottom) showing AE activity corresponding to micro-cracking initiation.

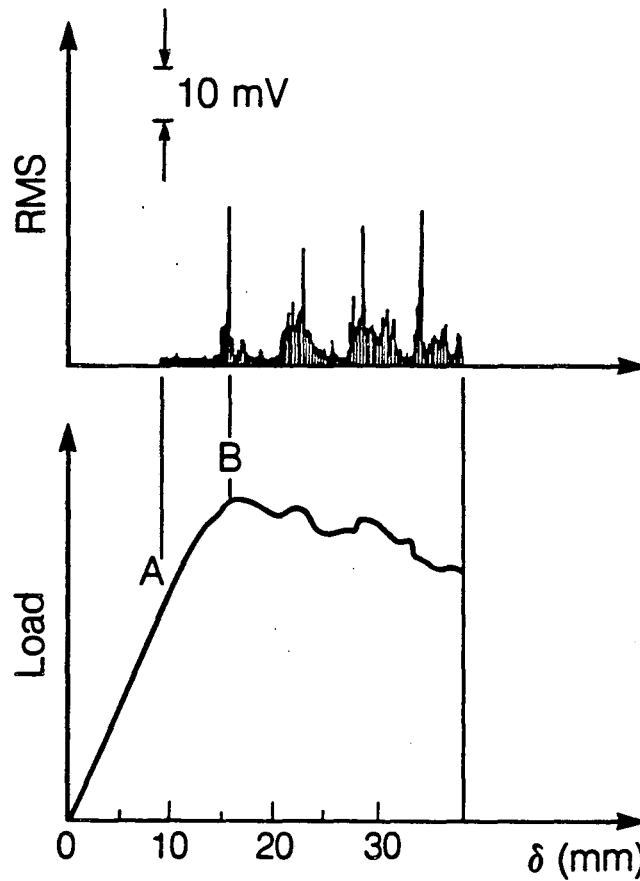


Fig. 5. Schematic of the load A and the load B corresponding to micro-cracking initiation and steady-state delamination, respectively.

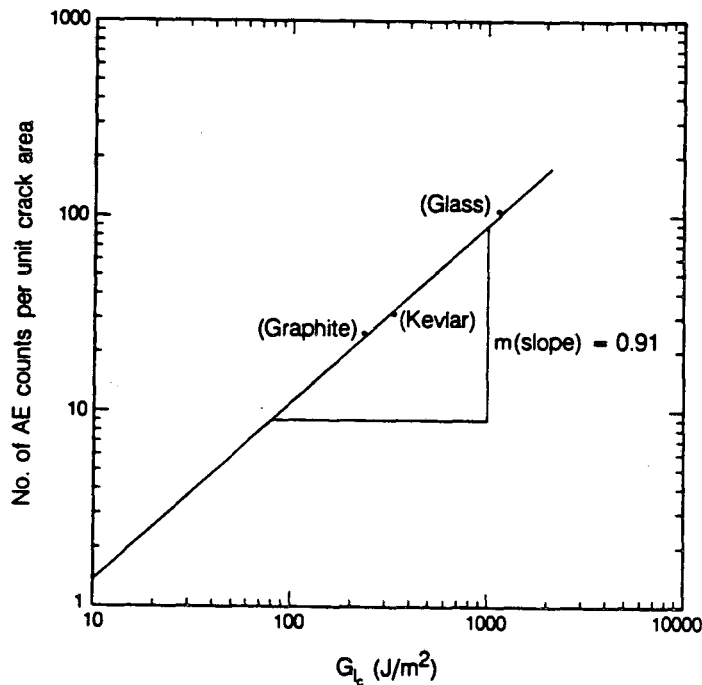


Fig. 6. Empirical relationship between the number of AE counts per unit crack area (during steady-state delamination) and the opening mode crack energy release rate corresponding to micro-crack initiation for carbon, aramid, and glass-epoxy composites.

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