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Intrinsic Characterization of Continuous Fibre Reinforced Thermoplastic Composites—I

TOUGHNESS CHARACTERIZATION OF CARBON FIBRE/POLYETHER ETHER KETONE (CF/PEEK) LAMINATES

(Technical Report)

Prepared for publication by

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Intrinsic characterization of continuous fibre reinforced thermoplastic composites—1: Toughness characterization of carbon fibre/ polyether ether ketone (CF/PEEK) laminates

ABSTRACT

After reviewing some of the early studies of toughness measurement of continuous carbon fibre reinforced Poly Ether Ether Ketone (PEEK), this work reports on some experimental studies of toughness measurements conducted by eight independent laboratories. The measuremnts were of two types; either plate type specimens of angle ply specimens studied by instrumented falling weight impact or by the adoption of linear elastic fracture mechanics on unidirectional laminates.
The comparative measurements are supplemented by additional observations that enable an objective interpretation to emerge from the data from the eight laboratories. The fracture mechanics parameters for assessing toughness provides a reasonable agreement on the measurement of toughness and indicates that the isotropic geometry functions that are necessary in the analysis of the data work well for unidirectional laminates.
The agreement is far from good for the instrumented falling weight impact data and some explanation for this is discussed.
The work was part of an IUPAC Working Party (4.2.1) study.

1. INTRODUCTION

IUPAC working party 4.2.1 (Structure and Properties of Commercial Plastics) has been involved in a series of studies of the mechanical and morphological behaviour of continuous fibre reinforced thermoplastic composites. This is the first of a number of reports relating to this study.

This particular report relates to a study of toughness in which eight laboratories have participated:-

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LABORATORY 2 - ICI PLC, Materials Centre, Wilton, UK.
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LABORATORY 5 - Cranfield, UK.
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LABORATORY 6 - Rheometrics, West Germany.
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The subject of the study has been a composite based on 61% by volume of carbon fibres impregnated with a poly (ether ether ketone) matrix. The aims of the work have been several fold.

(a) To provide a background knowledge and review of a new technology based on continuous fibre reinforced thermoplastic composites (conducted by laboratory 5).

(b) To identify some of the early documented results on toughness (laboratory 5).

(c) To study toughness experimentally in its many manifestations for CF/PEEK and in particular to provide an understanding of how toughness measurements may be interpreted.

(d) To examine the likely problems that occur when a single material undergoes toughness measurements by many laboratories, who believe that they may be measuring similar properties but possibly are not!

Sample preparation (laboratory 8) resulted in the production of both unidirectional ($[0]_{20}$ and $[0]_{40}$) and angle-ply laminates ($[\pm 45]_{2S}$). Pre-pregs of CF/PEEK were consolidated into laminates using an autoclave process. The pre-pregs stacks were bagged at room temperature in order to create a vacuum. Heating was conducted at a rate of 5°C/min until a temperature of about 380-400°C was reached. The stack was held at this condition for a time calculated on the basis of approximately 1 minute per ply then cooled to room temperature at 10°C/min after which pressure and vacuum were released. All laminates were ultrasonically C-scanned in order to confirm that the consolidation process had been successful.

2. BACKGROUND TECHNOLOGY

2.1 Thermoplastic versus thermoset matrix material

A major driving force towards interest in long fibre reinforced composites has been the demand from the transport industry, particularly the aircraft industry, for weight reduction. Indeed, continuous fibre reinforced composites possess good specific properties, ie. high stiffness and strength to density ratio, providing the opportunity of weight saving and hence energy saving.

So far, thermoset based composites have been used in aerospace applications but their drawbacks in terms of:-

- Limited shelf life of prepreg
- Lengthy and cumbersome curing cycles
- No scrap recovery
- Poor impact resistance, low toughness
- Poor performances when hot and wet
- Limited solvent resistance

have led to a new generation of long fibre composites based on thermoplastic matrices. This new class of material is unlikely to replace conventional thermoset based composites completely, but will lead to the extension of composite properties especially in tougher applications and hostile environments. The advantages of thermoplastic based composites are mainly:-

- Higher mechanical profile, ie higher ductility and toughness

- Better 'environmental' resistance for certain matrix systems, ie. better water and solvent resistance

- Ease of processing, leading to an increase of production rates

2.2 Composites based on PEEK

Continuous carbon fibre reinforced thermoplastic composites have been under development and evaluation since 1975. J T Hoggatt in 1975 (1) investigated the processing parameters, the environmental and mechanical properties of composites based on polysulfone and polyethersulphone matrices. The same kind of work was performed in 1979 by G E Husman and J T Hartness (2) on polyphenylsulfone matrix composites. One of the major drawbacks of amorphous matrices like polysulfone and polyether- sulphone however is their lack of solvent resistance, which will limit their applications in aircraft components.

The first production methods for this new class of materials, which are currently being used are based on three distinct processes:-

- The film stacking method which consists of interlaying layers of fibres (in the form of either unidirectional layers or fabric) with films of thermoplastic, and compression moulding to give a sheet stock.
  This method was devised and developed by D J Murphy and L N Phillips from RAE Farnborough (3). However for good wetting of individual fibres, this batch process requires application of high pressures (for up to 4 hours), which makes it relatively expensive.

- The second process is known as solvent coating. The resin selected is dissolved in solvent, after which a specially surface-treated fibre is impregnated with the solution by means of a coating head. The solvent is subsequently removed from the fibre by the process of evaporation.

  This processing method cannot be employed with PEEK because of its outstanding solvent resistance as is pointed out by R B Rigby (4).

- As the process of solvent evaporation possesses some unfavourable environmental connotations, a third process known as hot-melt-coating is often preferred. High temperature (370-420°C) replaces the solvent in order to reduce the plastic material to a suitable viscosity for thorough fibre impregnation, which is accomplished by means of a specially adapted head. This process is usually used for matrices limited in thermal stability like Nylon and Poly(ethylene terephthalate) as is mentioned by P E McMahon and M Maximovitch (5).

In these last two processes, the coated fibres are then taken up on a heated drum and fused together in parallel to make tapes of various width, or used to make woven fabrics. The actual processing technology used to produce the aromatic polymer composite, based on the PEEK matrix, has not been quoted in any papers related to the present topic. Nevertheless, ICI has developed a technique for continuously impregnating carbon fibres using both high temperature and pressure. The product, typically, is made as a uniaxial tape about 0.1mm thick, which can be processed into sheet and moulded items.

As is mentioned by G R Belbin (6), this aromatic polymer composite (APC), possesses an important advantage in that it can be processed using adapted techniques borrowed from both the metal working industry and the thermoset composites industry. I Brewster and J B Catanach (7) gave a detailed account of current knowledge on the processing methods used to produce components out of PEEK based long-fibre composites. A new technology can be used because compared with thermoset composites, APC materials possess the inherent advantages of thermoformability and reprocessability. One can distinguish two broad manufacturing methods:-

- The first one is based on a preconsolidated sheet, and the steps in the process are as follows:

  Production of blanks, ie laying down the different layers (either unidirectional prepreg plies or woven plies).

  Consolidation of the blanks into flat sheets using high temperatures (360-400°C) and low pressure (12atm).

  Heating the blanks prior to thermoforming by means of infra-red sources.

  Forming of the sheet; this last operation uses processes practised for many years in the metal fabrication industry such as rubber forming (ie. Hydroforming, Low pressure diaphragm forming, Rubber press forming), or Cold roll forming to produce structural sections.

- The second processing method is based on prepregs as the starting point, and covers both filament winding and tape laying. These two processes use existing equipment with small modifications concerning the heating of the filament or tape.

Of course, as the chemistry has been removed from the production stage, the cycles are much shorter because the material needs only to be heated for as long as it takes to form, or join - typically a few seconds.

One parameter, however, which is of considerable importance with the APC system, is the cooling stage, since PEEK is a semicrystalline material. This means that the morphology of the polymer will be affected not only by the cooling rate, but also by

the pressure and stresses in the component to be produced. As is pointed out by A Lustiger (8), crystallinity which does not occur in conventional thermoset based composites, needs to be considered in detail if optimum properties of the product are to be achieved.

It seems that optimum crystallisation, leading to the balance of mechanical properties, is obtained when the component is cooled from 380-200°C in less than 5 minutes. R F Dickson et al. (9) report on the development of a new version of APC, namely APC2, which possesses a much broader processing window.

The APC system and other thermoplastic based long fibre composites present other fabrication advantages. Namely they are much easier to repair, as only local heating needs to be applied, and to bond (heating and pressure replace the use of adhesives). F N Cogswell and D C Leach (10) mention a repair kit based around a soldering iron. Moreover the scrap material can be recovered to produce high performance short fibre thermoplastic composites for use in injection moulding.

2.3 The characteristics of the APC system

As was already mentioned, the APC system is based on the PEEK matrix, ie poly(ether ether ketone). This polymer is semi-crystalline and is then characterised by both a glass transition temperature Tg (=143°C) and a melting temperature Tm (=340°C). In other words, useful properties can be obtained above Tg, and R B Rigby (4) reports useful properties up to 300°C for applications involving intermittent heating. A study of the microstructure of the APC system was carried out by F N Cogswell (11), in which he showed that the fibre distribution was of the uniform random kind so that the thickness of the resin phase around the fibres was comparable with the thickness of the fibre. The dimension of the crystalline entities, ie spherulites, was shown to be of the order of 3μm for a material rapidly cooled from the melt to a temperature range of 20-200°C.

This crystalline character confers upon PEEK exceptional environmental resistance. F N Cogswell and M Hopprich (12) studied the environmental resistance of carbon fibre reinforced PEEK. The material was found to be very resistant to common solvents (Acetone, Cyclohexane ...), aircraft fluids (hydraulic fluid, kerosene) and paint stripper. The APC system absorbs less moisture than a "state of the art" epoxy based composite, and the matrix does not suffer from plasticization by water leading to a decrease in the glass transition temperature.

It can be shown that the higher the degree of crystallinity, the better the solvent resistance but the lower the toughness. Therefore by tailoring the morphological variables, through for example cooling and subsequent annealing, this material can be adapted to the unique requirements of particular components. The carbon fibres act as nucleating agents, and columnar crystals grow perpendicular to the reinforcing units. This transcrystallinity affects the properties of the composite and a balance has often to be found:-

- A good interface between matrix and fibre produces a high level of transcrystallinity. F N Cogswell (11) reports that fracture surfaces of tensile specimens do not show the clean fibre surfaces resulting from extensive debonding and the consequent fibre pull-out that are responsible for energy absorption during fracture in commercial thermoset based composites. For broken fibres in APC are always coated with resin, indicating the very good bonding at the surface.

- Lustiger comments on relatively poor long-term strength brought about by areas of weakness created by spherulitic boundaries. A high volume fraction of fibres may lead to too high a degree of transcrystallinity which in turn may lead to impingement of the transcrystalline regions, creating long spherulite boundaries and thus lines of weakness.

2.4 Historic toughness measurements on CF/PEEK composites

The first quoted value of the interlaminar fracture toughness G_{IC} for PEEK matrix composite seems to have been reported by J T Hartness in 1982 (13). The author used the double cantilever beam also known as mode I peel test. A value of G_{IC} of 1990 J/m^2 was found for the PEEK/graphite cloth system produced by the film stacking sequence. The equivalent epoxy based composite was much less tough with a value of G_{IC} of only 235 J/m^2.

The mode I interlaminar fracture toughness on an early development grade of CF/PEEK pre-preg was first determined by D R Carlile and D C Leach in 1983 (14).

Parallel sided specimens were made out of unidirectional material in the form of double cantilever beams. G_{IC} was determined using the area method devised by J M Whitney et

al.(15). Two distinct modes of failure were observed: a cleavage mode corresponding to fast crack propagation, for which G_{IC} was found to be 1870 J/m^2, and a ductile mode corresponding to slow crack propagation, with a much higher value of G_{IC}, namely 3200 J/m^2.

The same test was performed on a state of the art epoxy based composite (AS4 carbon fibres/Epoxy 3501-6) which was found to be nearly 10 times less tough, with a G_{IC} of 210 J/m^2.

Using the same test (DCB and area method to reduce the data), J T Hartness (16) found a G_{IC} value of 1400 J/m^2 for the cleavage mode and a higher value of 1800 J/m^2 for the ductile mode of failure, when evaluating the mechanical properties of a similar early development grade of CF/PEEK. These values of G_{IC} are less than those reported by Carlile and Leach. J T Hartness also used an edge delamination specimen to determine another value of the interlaminar fracture toughness G_{IC}. Specimens of lay-up configuration $[+30/-30_2+30/90_2]_S$, leading to the generation of high interlaminar stresses, were used for the test. Total failure of the laminates occurred before any delamination. Therefore using the strain to failure, G_{IC} was found to be 1400 J/m^2.

G F Dickson et al (9) were the first ones to determine a value of critical stress intensity factor in mode I, K_{IC}, for a laminate based on APC material. For a laminate of lay-up configuration $[0,90]_{3S}$, using centre notched coupons, they found a value of K_{IC} of 69 MPa$\sqrt{m}$ for the APC system and of only 50 MPa$\sqrt{m}$ for the same composite in terms of lay-up configuration but based on an epoxy matrix.

Another value of G_{IC} for an early development grade of CF/PEEK was determined by C Y Barlow and A H Windle (17) using a novel razor blade test. This test is based on the propagation of a crack ahead of a thin wedge. The initial theory was developed to determine the splitting energy of mica, and is based on simple beam theory for isotropic materials; it does not take into account elastic end effects, material anisotropy or the existence of a plastic zone at the crack tip.

However work on a wide range of long carbon fibre reinforced composites proves the basic theory is valid. For their CF/PEEK composite, the authors reported a value of G_{IC} of 2290 $\pm$ 600 J/m^2.

The last paper reported here, dealing with fracture mechanics parameters of the PEEK based composite, was written by S L Donaldson in April 1985. (18). Using the notched off-axis tensile test, Donaldson determined the critical values of G_{IC} and K_{IC} (for θ = 90°C, θ being the angle between the tensile axis and the fibre axis), and mixed mode parameters K_I, G_I, K_{II}, G_{II}, for different orientations, ie 75°, 60°, 45°, 30°, 15°, 10°, 7°, 5° and -15°.

The 8-ply unidirectional composite results based on an early development grade of CF/PEEK were compared with those obtained from 8-ply unidirectional composite based on an epoxy matrix. K_{IC} was found to be 3.7 MPa$\sqrt{m}$ corresponding to a G_{IC} of 1120 J/m^2 for the CF/PEEK system. The epoxy based material, however, was much less tough with a K_{IC} of 0.96 MPa$\sqrt{m}$ and a corresponding G_{IC} of only 78 J/m^2.

Critical values of K_{IIC} and G_{IIC} were also determined using the modified three rail shear test. The same trend was observed for the mode II. K_{IIC} was found to be 14.4 MPa$\sqrt{m}$ with a corresponding value of G_{IIC} of 4930 J/m^2 for the PEEK matrix composite, whereas values of 4.8 MPa$\sqrt{m}$ and 506 J/m^2 were determined for K_{IIC} and G_{IIC} respectively for the thermoset system.

An evaluation of the anisotropy of fracture toughness has been reported by Leach and Moore (19), together with evaluation of toughness of CF/PEEK in some practical industrial tests.

3. FRACTURE MECHANICS TOUGHNESS

3.1 Preamble

Linear elastic fracture mechanics theory (20) provides two important contributions to the characterisation of toughness. First, a claim to provide geometry independent material properties. Second, two material properties which can be considered as fracture toughness (G_{IC}) and fracture strength (K_{IC}). More formally, these two properties can be defined as:-

$$G_{IC} = \frac{\Delta U}{\Delta A} \qquad (1)$$

where G_{IC} is the critical value of strain energy release rate, in a crack opening mode (I)

ΔU is the energy required to propagate a crack of new area ΔA.

$$K_{IC} = \sigma_F Y a^{\frac{1}{2}} \quad (2)$$

where K_{IC} is the critical value of stress intensity factor, in a crack opening mode (I)
σ_F is a fracture stress remote from a crack of length 'a'

Y is a geometry function which is well defined in the literature (21)

Equations 1 and 2 are general to all geometries, but G_{IC} can only be useful in this form when crack growth can be easily monitored. In all our fracture mechanics experiments, unstable fractures have to be accommodated. Therefore, it is useful to use an alternative expression for G_{IC}, namely:-

$$G_{IC} = \frac{U_F}{A\phi} \quad (3)$$

where U_F is the total energy absorbed at fracture of a ligament of area A.

ϕ is another geometry term that ensures that the energy term U_F is suitably divided between elastic strain energy and energy to propagate a crack (20,22).

It is implicit that the two geometry functions (Y and ϕ) are material independent. They have been calculated for isotropic materials only, therefore their validity in use with anisotropic materials (such as unidirectional continuous fibre composites) must be either assumed or verified. The two fracture properties are themselves related.

$$K_{Ic}^2 = E\, G_{Ic} \quad (4)$$

E is a material modulus (measured at the same strain rate as that for the fracture parameters).

For the case of anisotropic materials exhibiting fibre symmetry (such as unidirectional continuous carbon fibre composites), an orthotropic modulus E* can replace the more common isotropic term (23).

$$\frac{1}{E^*} = \pi \left(\frac{a_{11}\, a_{22}}{2}\right)^{\frac{1}{2}} \left[\left(\frac{a_{22}}{a_{11}}\right)^{\frac{1}{2}} + \frac{2a_{12} + a_{66}}{2a_{11}}\right]^{\frac{1}{2}} \quad (5)$$

where a_{ij} are the elastic compliances associated with the principal material directions. These will be discussed in a second IUPAC paper relating to this project on CF/PEEK but literature values (10) for these terms provide the following for unidirectional laminate:-

$a_{11} = 0.109 \quad GPa^{-1}$
$a_{22} = 0.008 \quad GPa^{-1}$
$a_{12} = 0.0003 \quad GPa^{-1}$
$a_{66} = 0.244 \quad GPa^{-1}$

In turn, this yields a value of E* of 12.9 GPa.

3.2 Fracture mechanics geometry functions for anisotropic materials

Most experimental measurements of G_{IC} and K_{IC} on CF/PEEK will relate to unidirectional laminates. These materials exhibit more anisotropy than any other form of laminate. Consequently, it becomes crucial to establish that the necessary geometry functions (Y and ϕ) of equations 2 and 3 respectively which are known for isotropic materials, can be adequately applied to an anisotropic case.

In the case of three point flexure of a notched beam (see figure 1) the compliance is known to be a function of notch depth (a) and specimen depth (W):- (22)

$$C_o\,(a/W) = \phi\,(a/W)\; Y^2(a/W)\; \frac{9S^2}{2BW^2\, E}\; \frac{a}{W} \quad (6)$$

S - is the specimen span

B and W - are thickness and depth (see figure 1)

E - is an appropriate modulus of the beam specimen in this configuration.

C_o - is the theoretical compliance as described above where the two geometry functions $\phi\,(a/W)$ and $Y\,(a/W)$ are the isotropic values

The compliance of each specimen can also be determined by experiment (C_e). A comparison of compliance by experiment and calculation then reveals the suitability of ϕ and Y to an anisotropic case.

This procedure was adopted for a range of beam specimens machined from a $[0]_{40}$ laminate. With reference to figure 2, specimens with crack directions 1 and 2 were used, although this will be discussed more fully in the next section.

A rebound technique (24) was used to determine an appropriate modulus (E) for both types of specimen, unnotched. After introducing a sharp machined notch, this rebound technique was also used to measure C_e.

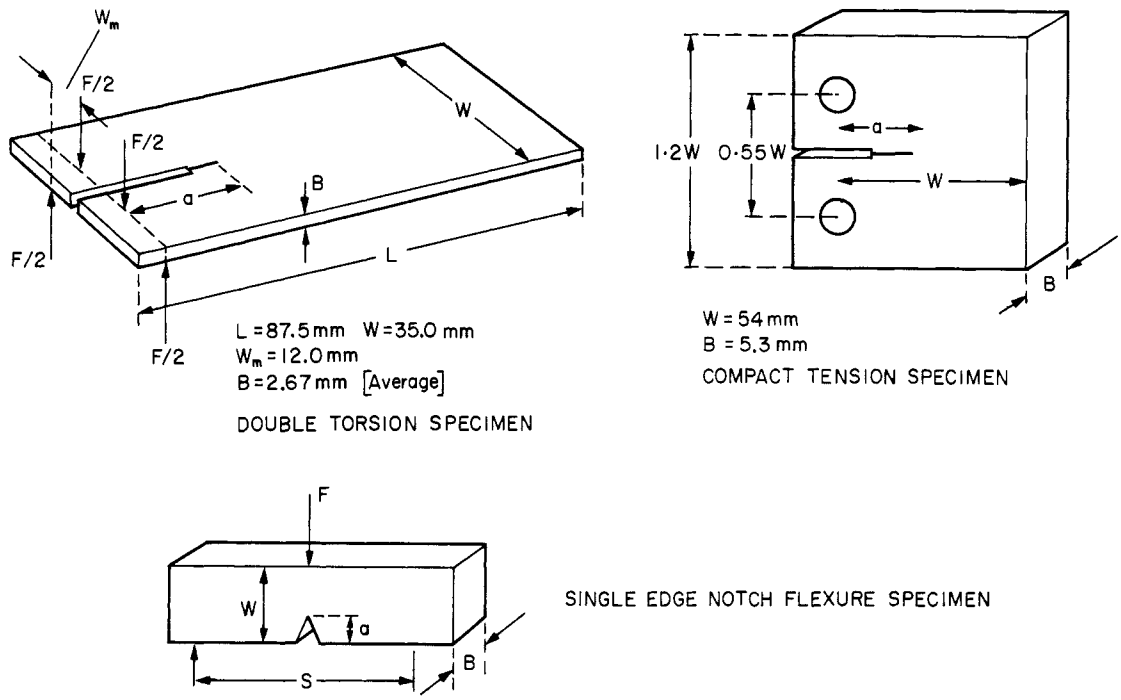


Fig. 1. Fracture mechanics specimens

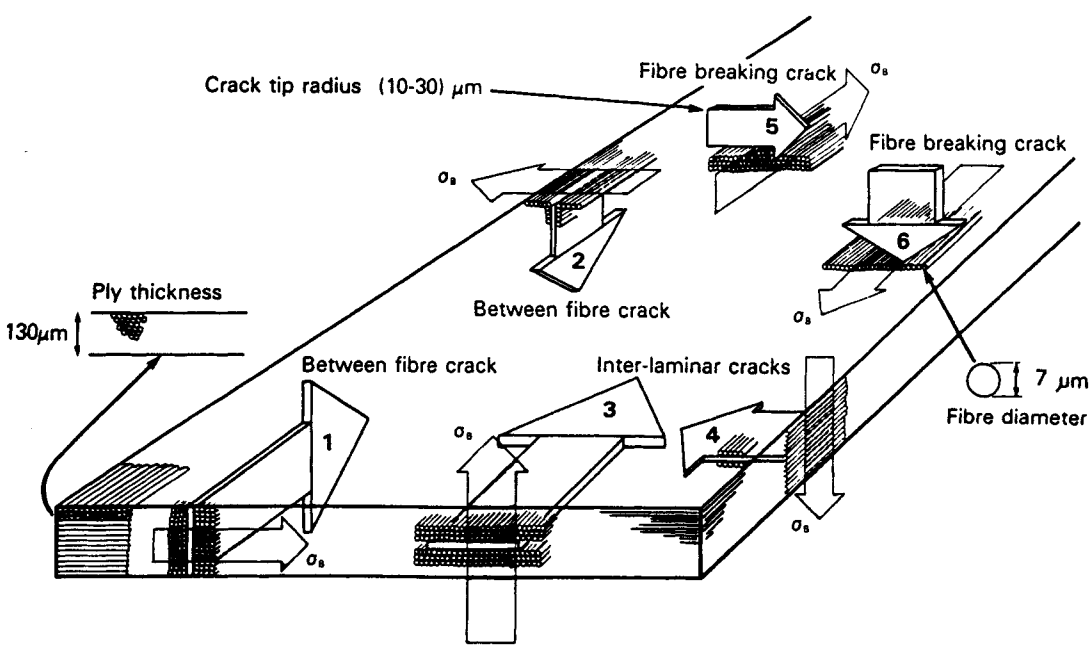


Fig. 2. Crack propagation in a unidirectional composite

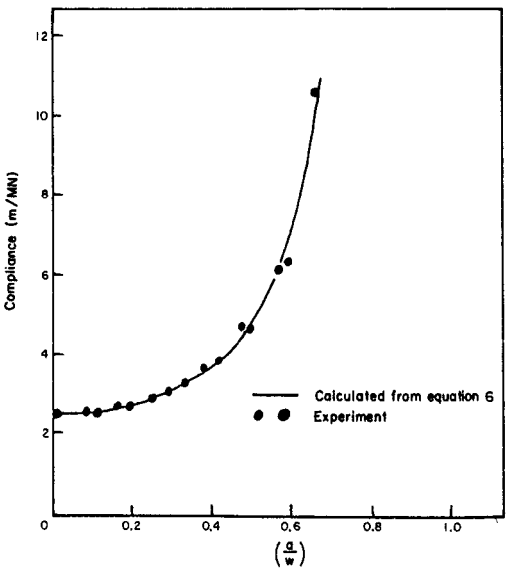


Fig. 3. Comparison between measured and calculated compliance for three point bend specimens with notch $[0]_{40}$ CF/PEEK

A comparison of calculated and measured compliance as a function of ($^{a}/_{W}$) is shown in figure 3. The good agreement between calculation and experiment implies that the isotropic geometry functions employed in the calculations must be suitable for the anisotropy exhibited by these laminates.

3.3 Presentation and discussion of the fracture mechanics results for K_{IC} and G_{IC}

The majority of fracture mechanics experiments for the determination of G_{IC} and K_{IC} were conducted on specimens machined from unidirectional laminates, $[0]_n$ where n = 20 and 40. Three different loading geometries were employed, namely, three point flexure, double torsion and compact tension. (figure 1) All experiments were conducted with a view to investigating the influence of either test temperature or test speed on fracture strength and fracture toughness. Consequently, results can be presented for temperatures in the range -60°C to +23°C and for test speeds in the range 8 x 10^{-3}mm/s to 2.5 x 10^{3}mm/s (more than five decades of test speed). It was also planned to explore different crack directions relative to fibre direction and ply orientation. This is more fully illustrated in figure 2 where six directions of crack propagation can be seen for unidirectional laminate, σ being the direction of applied stress. In figure 2 the various crack directions are labelled. Our results will relate to three of these directions, namely directions 1 and 2 (both intra-laminar fracture) and direction 5 (trans-laminar fracture). It was felt that the large literature on inter-laminar fracture (directions 3 and 4) could not be usefully added to by this work, and that it was better to concentrate on toughness characterisation where less information is documented.

A summary of the K_{IC} and G_{IC} results at 23°C is given in table 1. Included with the averaged values of K_{IC} and G_{IC} is a comment on the method used in the data analysis. It can be seen that several different approaches have been used. The values of K_{IC} and G_{IC} are averaged from a few to sometimes a dozen specimens. The scatter associated with each value is variable in a range of 5% to 20% for the coefficients of variation.

Three test geometries were employed for test direction 1 at a test speed of (8 x 10^{-3}) mm/s. Values for K_{IC} are in the range 3.8 to 6.5 MPa$\sqrt{m}$.

Direction of crack in the unidirectional laminates has a profound influence on toughness. There are subtle differences between the two intra-laminar fractures (test directions 1 and 2). These might be more than experimental scatter since the fibre alignment in test direction 1 could well be more than that for test direction 2. Hence higher toughness is associated with test direction 2. These observations have also been reported elsewhere (19). The difference between intra-laminar fracture toughness and trans-laminar fracture toughness is huge. For example, a typical K_{IC} for crack direction 1 (intra-laminar) is (3-4) MPa$\sqrt{m}$, whilst a value for crack direction 5 (trans-laminar) is an order of magnitude larger at 37 MPa$\sqrt{m}$.

Five laboratories were involved with the determination of these fracture toughness data. A laboratory number is included with the summarised data in Table 1. When the data relating to the test conditions three point geometry (A) and crack direction 1 are examined, then values for K_{IC} lie in the range 2.0 MPa$\sqrt{m}$ to 5.7 MPa$\sqrt{m}$. All values in this range satisfy the geometry criteria for obtaining valid fracture mechanics data (25).

These data were determined at a range of test speeds, from 2.5 x 10^3 mm/s to 8 x 10^{-3}mm/s. In addition, two methods of data analysis were used in deriving K_{Ic}. The spread in K_{Ic} for this data group as 2.0 MPa$\sqrt{m}$ to 5.7 MPa$\sqrt{m}$. There is a suggestion of a trend with test speed, where the lower values relate to higher test speeds. Curiously, the range of values for G_{Ic} does not reflect this scatter, since the range for test geometry A at test direction 1 is 1.0 kJ/m^2 to 1.9 kJ/m^2. Further understanding of such scatter is limited. One laboratory (number 3) reported that K_{Ic} depends on notch depth but it remains difficult to fully explain the range of toughness values. On the other hand, it is apparent that a study involving common test conditions in an inter- laboratory investigation might shed more light on the problem.

A possible cause of scatter in these fracture mechanics parameters might lie in the quality and characteristics of the notch in the test specimens. One of the laboratories (Laboratory 5) investigated this possibility. Using a three point bend specimen machined from $[0]_{40}$ laminates, a crack was initiated in direction 2 where three different methods were used for introducing the cracks, namely:-

(a) Unsharpened notches machined at 23°C
(b) Sharpened notches (sharpened at 23°C)
(c) Sharpened notches (sharpened at liquid nitrogen temperatures, - 196°C)

Values of K_{Ic} were obtained from testing nine specimens at each notch condition. K_{Ic} values of 4.3, 4.2 and 3.9 MPa$\sqrt{m}$ were obtained, respectively. The notch sharpness is not influential in the magnitude of measured toughness and is therefore unlikely to be a source of inter-laboratory data scatter, at least for these continuous fibre composites.

One of the laboratories (No 1) examined the influence of temperature on the fracture toughness of three point bend specimens tested at 2.5 x 10^2 mm/sec on a pendulum impact machine. Figure 4 plots G_{IC} versus temperature where the scatter bands relate to the 95% confidence limits determined in fitting best lines to the experimental data. Despite the scatter, it can be concluded that intra-laminar fracture toughness is temperature independent in the temperature range -60°C to 23°C.

TABLE 1 Summary of K_{IC} and G_{IC} Results on Unidirectional Laminates at 23°C

Laboratory	Test Direction	Test Geometry	Lay-up	Test Speed (mm/s)	K_{IC} (MPa m)	Comment	G_{IC} (kJ/m²)	Comment
1	1	A	$[0]_{40}$	$2.5x10^3$	4.3	Calculated via E*	1.45	Plati & Williams (22) Perdulum Test
2	1	A	$[0]_{40}$	$1.0x10^3$	2.0	Brown & Srawley (21) IFWI	1.0	Plati & Williams (22)
3	1	A	$[0]_{40}$	$1.0x10^3$	3.5	Brown & Srawley (21)	1.1	Plati & Williams (22)
1	1	A	$[0]_{40}$	$8x10^{-2}$	3.7	Brown & Srawley (21)	1.9	Plati & Williams (22)
4	1	A	$[0]_{40}$	$8x10^{-2}$	4.9	Brown & Srawley (21)	--	Experiment
5	1	A	$[0]_{40}$	$8x10^{-3}$	5.7	Brown & Srawley (21)	1.35	Compliance by Experiment
5	1	B	$[0]_{40}$	$8x10^{-3}$	6.5	Brown & Srawley (21)	--	
5	½	C	$[0]_{20}$	$8x10^{-3}$	3.8	Calculated via E*	1.12	Crack Growth Analysis
3	2	A	$[0]_{40}$	$1.1x10^{-3}$	4.5	Brown & Srawley (21)	1.9	Plati & Williams (22)
4	5	A	$[0]_{40}$	$8x10^{-2}$	37	Calculated via E*	26	Compliance by

A = Three-point bend geometry, B = Compact tension geometry, C = Double-torsion geometry, Test direction 1, 2 & 5 are clarified in figure 2

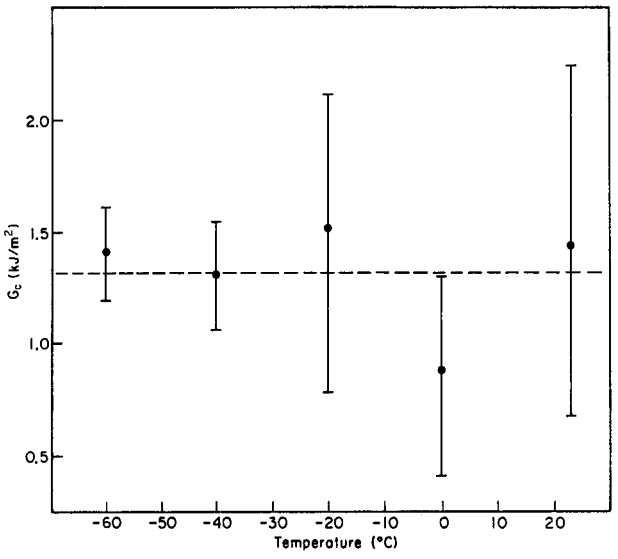


Fig. 4. Fracture toughness from three point flexure on $[O]_{40}$ laminates of CF/PEEK versus temperature. Test direction 1(8x10^{2}mm/sec)

4. IMPACT TOUGHNESS

4.1 Preamble

Toughness can be measured in other ways compared with fracture mechanics parameters. A popular alternative is to measure the energy absorbed by a specimen during impact. Such a measure relates toughness to this absorbed energy, but since it may apply to many different geometric arrangements for specimen, support and impactor, it is inevitable that this toughness is a combination of material property and geometry. This does not of necessity matter because it allows impact trends to be followed; but it does place great store on adequate interpretation.

Many recent approaches to this measurement of impact toughness involve instrumented falling weight techniques (IFWI). The force-time signal is collected during the course of impact, stored on a micro-processor, analysed and manipulated with some appropriate software and data are presented in a number of ways. Several of these methods are well reported in the literature (26, 27, 28). A key feature of the data collection and analysis is the degree of electronic filtering of the signal. Typical resonance frequencies for the apparatus are of the order of 9kHz. Some analogue low pass filters operate down to 2kHz, whilst many approaches involve effectively no filter. The detail on the force-time impact plot is drastically influenced by the degree of filtering (28).

The impact toughness of CF/PEEK has been obtained on angle-ply laminates $[\pm 45]_{2S}$ ie. 8 ply material. In the two sections that follow, it will be possible to present a range of data from a number of different experimental techniques. It will be shown, however, that meaningful data emerge only when a full understanding of the force-time signal is possible.

4.2 Impact toughness of $[\pm 45]_2S$ laminates

Three laboratories conducted a range of impact experiments on $[\pm 45]_{2S}$ laminates with the following themes:-

(a) Impact of plate specimens at 23°C for various combinations of circular support diameter and diameter for hemispherical nose impactor. (Laboratory 3)

(b) Impact of plate specimens at a fixed geometric arrangement at various temperatures (-40°C to +200°C) and various test speeds (penetration speeds 8mm/s to 8,000mm/s). (Laboratory 6)

(c) Tensile impact tests at 23°C at 5,000mm/s. (Laboratory 7)

Before contemplating experimental results and analysis, it is useful to compare qualitatively the force-time traces obtained on typical specimens at approximately common test speed (in the range 5,000 to 8,000 mm/s). This is achieved in figure 5. It must be emphasised that these plots are not to any consistent scale, for either force or time. The plate specimens for laboratory 3 and laboratory 6 show generally similar features, except that no filter of the electrical signal has been used by laboratory 3.

The force-time signal from laboratory 7 is filtered but will be different anyway because it was obtained by a tensile test. The degree of filtering associated with each of these force-time signals appears to be different. The degree of detail on the curves is influenced by often undefined specification of the equipment. As a consequence, if an analysis were to be based on the energy absorbed by a specimen at the first peak of these signals, there could be no guarantee that a similar failure process in impact was being articulated. It would be preferable to obtain IFWI signals without filtering in order to maximise the opportunity of consistent comparisons of toughness.

The results from themes (a) and (b) above reveal a picture in their own right. For example, geometry effects can be examined from the work of laboratory 3, where Table 2 summarises geometry configurations and results. Laboratory 3 provides evidence that first damage of the specimen occurs at peak A (see figure 5) and also indicates that peak B is relevant to the failure processes. (ie the maximum force peak). Consequently, energies absorbed by the specimens corresponding with these peaks are included in Table 2. Evidence as to why peak A is first damage is discussed in due course.

The results in Table 2 indicate a strong geometry dependence in impact toughness and support the claims discussed in the preamble. Such results pose more questions about interpretation, than answers to evaluation of toughness. For example, how much is geometry contributing to the measurements? Also, if peak A is first damage, then what interpretation can be associated with total energy absorbed by the specimen? How much of this energy is due to initiation and propagation of cracks and how much is contributed by 'geometric' factors?

Theme (c) explored by laboratory 6 reveals a temperature dependence of impact toughness. Figure 6 illustrates energy up to the maximum force (shaded area of figure 5) versus temperature. Although some expected trends are revealed in figure 6, namely, a reduction of impact toughness with decreasing temperature, it remains difficult to apply an interpretation to the results. This is the case because it is not known what the energy to maximum force means in terms of failure mechanisms and geometric contribution.

4.3 Experiments to interpret the force-time signals

4.3.1 Introduction

Two sets of experiments have been used in order to understand the force-time impact signals from plate flexure impact experiments. First, photography of the tension surface during impact in order to relate failure events with absorbed energy. This has been conducted by laboratory 2 at three different test temperatures. Second, low energy instrumented falling weight impact by laboratories 2 and 3, in order to help resolve where first damage to a specimen occurs.

4.3.2 Photographed impact

Instrumented falling weight impact tests were conducted on specimens (60 x 60) mm cut from $[\pm 45]_{2S}$ laminates. A support diameter of 50mm and impact diameter of 12.5mm was used at an impact speed of 5m/s (impactor mass 8.2kg, drop height 1.3m). Tests were conducted at test temperature of -20°C, +23°C and +60°C.

During the impact tests photographs were taken of the tension surface (full details are in reference 29). The black specimens would have made resolution of the surface damage difficult and therefore the tension surface was sprayed with a white primer.

Photographs could be obtained at any time during the measurement of the impact event. Precise timing was a fundamental requirement and this was achieved by using a twin channel transient recorder which stored the force-time signal on the first channel whilst simultaneously storing the time when the photograph was taken by means of a light intensity versus time signal in the second channel. The source of the light was a photographic flash unit whose emission of light was detected by a photodiode connected to the transient recorder. During the flash a photograph of the tension surface was recorded. An electronic timing device was used to delay the flash by a pre-set interval.

With this arrangement the time when the flash reached its maximum intensity could be determined with an accuracy comparable with the duration of the flash (50 μs). By progressively increasing the pre-set time delay for successive impact tests a series of photographs was obtained covering each stage of the fracture process. Consequently, a comprehensive interpretation of the impact force-time signal was possible.

TABLE 2 Impact Toughness for Different Support and Impactor Geometries (23°C)

Test	Geometry	Energy (Mean) At Peak A	Energy (Mean) At Peak B
Support Diameter (mm)	Impactor Diameter (mm)	(First Damage) (Joules)	(Maximum Force) (Joules)
40	10	0.99 (0.13)	2.05 (0.4)
50	12.5	1.60 (0.2)	3.15 (0.52)
40	20	1.7 (0.6)	3.63 (0.7)

- Impact speed 5m/s, mass of impactor 8kg, Input energy = 100J.
- Specimens are freely supported on a ring.
- (standard deviations are quoted in brackets)

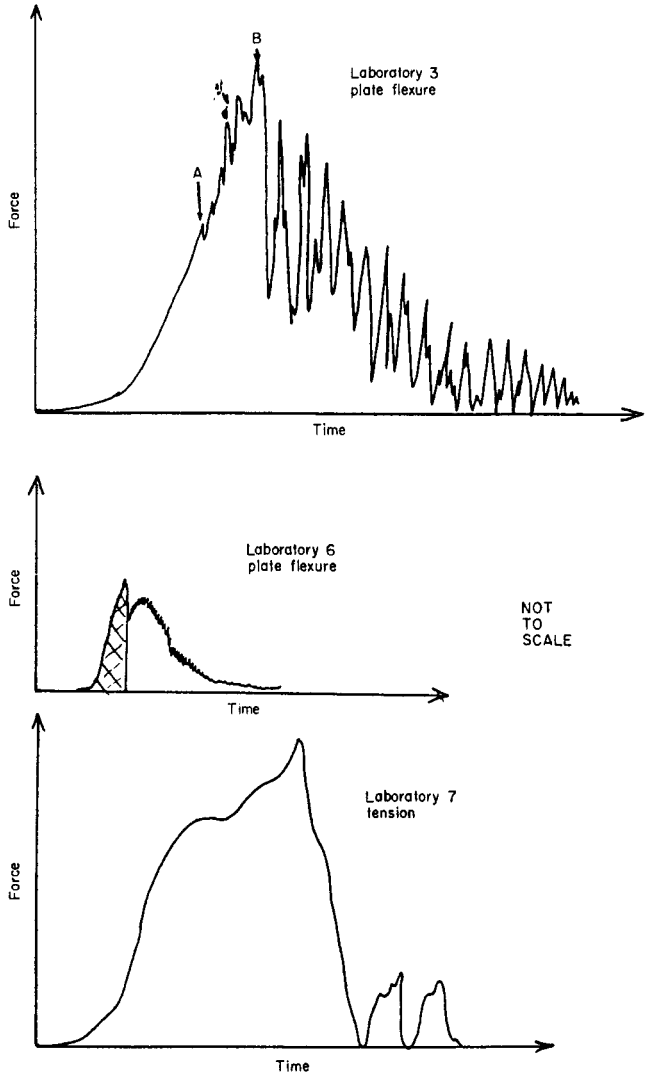


Fig. 5. Force-time signals from instrumented impact tests on specimens from $[\pm 45]_{2s}$ laminates of CF/PEEK at 23°C

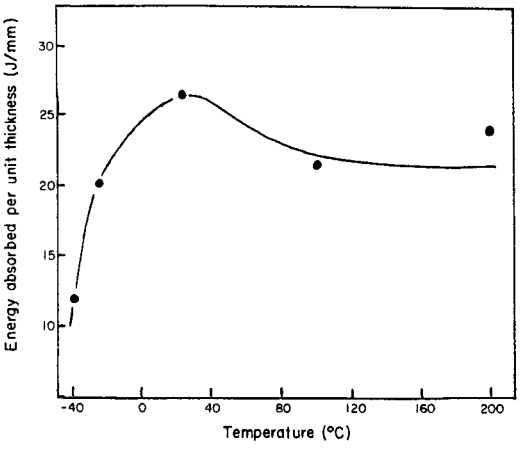


Fig. 6. Absorbed energy (shaded area of Fig. 5) versus temperature for plate flexure of $[\pm 45]_{2s}$ specimens of CF/PEEK

The photographs of the damaged tension surfaces enabled a quantitative analysis of the crack for various absorbed impact energies. Total crack lengths were measured using a Hewlett Packard 9874A digitiser connected to a Hewlett Packard 9845 computer, after taking account of the magnification factor of the photograph. If it is assumed that the crack penetrates the full thickness of the specimen, then crack area can be determined by multiplying the total crack length by specimen thickness.

Figure 7 illustrates a sequence of photographs for various stages of impact at 23°C. It must be emphasised that each photograph relates to a specific specimen with a specific force-time curve. Consequently, the force-time curve included in figure 7 is typical and included for purposes of illustration. The energies absorbed by each specimen which are quoted with the photographs were measured from the appropriate force-time curves.

It is clear from figure 7 that first damage on the tension surface occurs before the maximum force in the force-time signal. This is also the case at -20°C and +60°C. The force-time signal in figure 7 relates to a filtered signal, and it is known that the point of first damage for an unfiltered signal coincides with a peak (see figure 5 for laboratory 3).

It is also possible to provide an interpretation of the energy to the maximum force peak by using this photographic study. The crack has propagated to the supports by the time the maximum force peak has been reached. Therefore the associated energy is approximately the sum of energy to initiate a crack and propagate it to the supports. It is not certain that the crack has penetrated the full thickness of the specimen although the photograph at 4 Joules hints at this being so. If this is correct then by implication, the remainder of the energy absorbed by the specimen is frictional and geometric in nature. Consequently, this residual energy is unrelated to the material toughness.

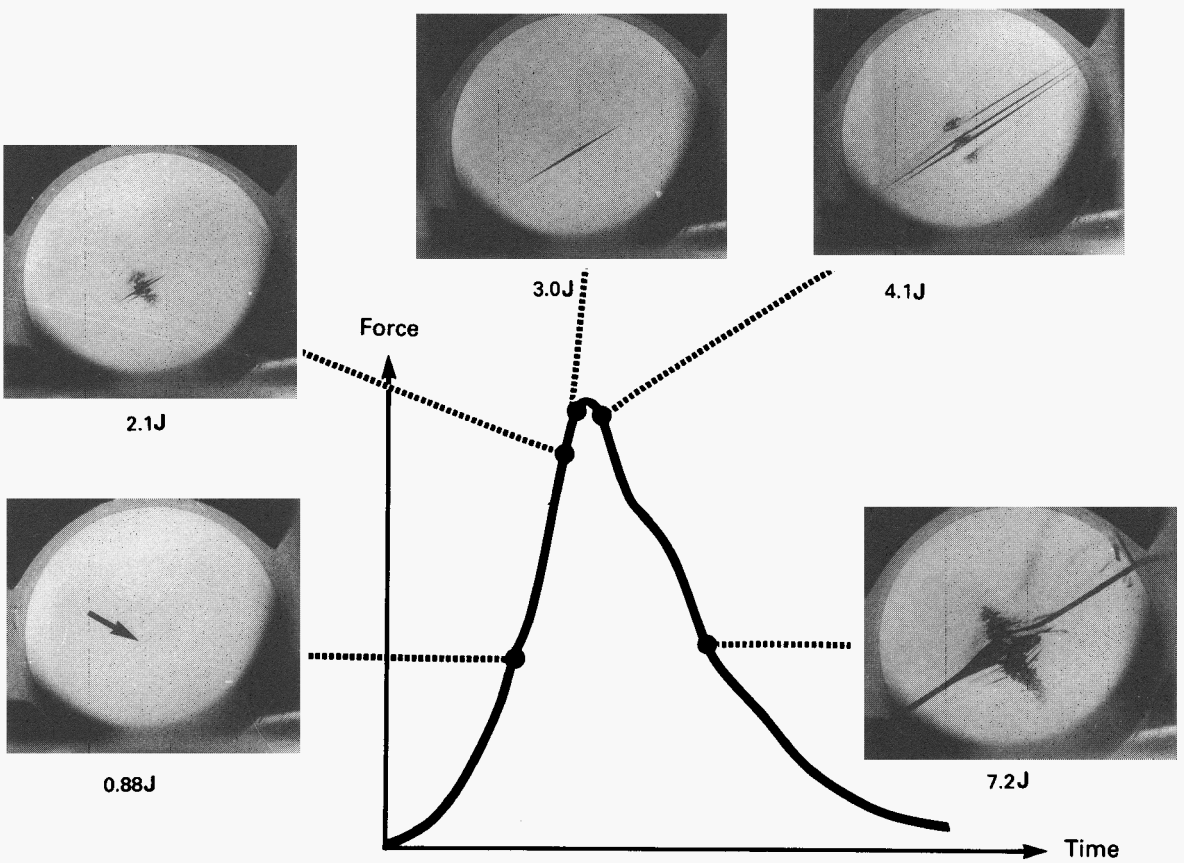


Fig. 7. Photographed impact at 23°C — CF/PEEK $[\pm 45]_{2s}$ laminates (typical force-time plot at 23°C shown — filtered 2.2kHz)

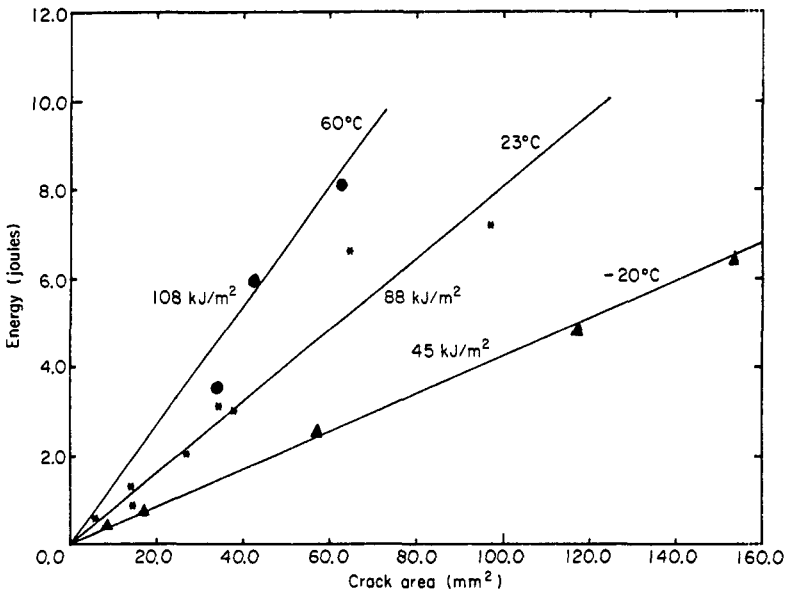


Fig. 8. Energy-crack area plots at three temperatures from photographed impact of $[\pm 45]_{2S}$ laminates of CF/PEEK

TABLE 3 Low Energy Impact Results for CF/PEEK $[\pm 45]_{2S}$ Laminates at 23 °C, Test Speed Range (1-2) m/s

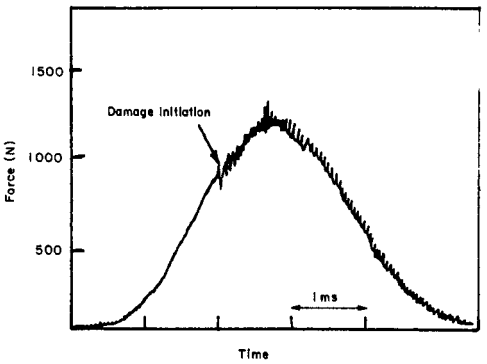
Test Support Diameter (mm)	Impactor Diameter (mm)	Mean Energy to Initial Damage (Joules)	
40	20	1.38 (0.4)	LABORATORY 3
50	12.5	0.87 (0.2)	LABORATORY 2

An overview of the temperature dependence of toughness emerges from the results presented in figure 8. Energy absorbed by the specimen is plotted against notional crack area (total crack length x specimen thickness). Surprisingly these data appear to be straight line plots for which energy per unit area can be associated with a notional fracture toughness.

4.3.3 Low energy instrumented falling weight impact

Two laboratories have conducted some low energy impact tests (30, 31) in order to further resolve and confirm the onset of initial damage in terms of interpretation of the force-time signal.

Several failure mechanisms are known to be possible for impact of $[\pm 45]_{2S}$ laminates of CF/PEEK. These include intra-laminar fracture, delamination fracture (inter-laminar fracture) or fibre cracking (trans-laminar fracture). It is probable that several of these mechanisms can be active at the same time. One particular issue that can be addressed from low energy IFWI is whether the energy to create surface splits (intralaminar fracture) can occur more readily than delamination fracture. Consequently, impacted specimens that received only small amounts of impact energy were ultrasonically C-scanned subsequent to impact testing, in order to obtain a measure of delamination damage.



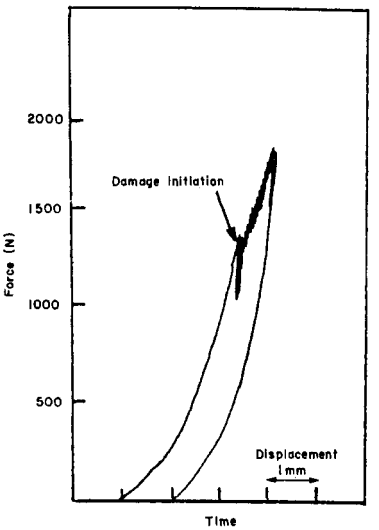
LABORATORY 2

[±45] 2m/s

$U_{INPUT} = 2J$

50mm SUPPORT DIAMETER

12.7mm IMPACTOR DIAMETER



LABORATORY 3

[±45] 1.4m/s

$U_{INPUT} = 2J$

40mm SUPPORT DIAMETER

20mm IMPACTOR DIAMETER

Fig. 9. Force-time curves from low energy IFWI at 23°C on CF/PEEK

Typical force-time curves from the two laboratories for low energy impact tests are shown in figure 9. Damage initiation is associated with larger force oscillations in the force-time signal. (In both laboratories, no filtering of these signals occurred). Consequently, this feature enabled the energy to initiate damage to be determined. The geometries used in the two laboratories were made deliberately different and results are summarised in Table 3.

The mean energy to initiate damage in impact from the results for laboratory 2 and Table 3 is similar to the energy absorbed by a specimen in creating a tension surface crack in the photographed impact study. Also, the results from laboratory 3 agree generally well with previous data for energy to create first damage (see Table 2). Photographs of their low energy impact specimens indicate the presence of surface cracks when the input energy exceeded 1.4 Joules. It is therefore likely that the first damage is tension surface splitting and this can be detected on the force-time plot.

Ultrasonic C-scans of the low energy impact specimens revealed delamination damage. An approximate analysis of input energy versus measured area of C-scan damage enabled the threshold energy for delamination to be approximately determined (30). A threshold energy of about 1.3 Joules for the geometry employed for laboratory 2 was obtained. Although this value of delamination energy is tentative, it is larger than that energy associated with tension surface splitting (0.87 Joules) implying that the first failure mechanism must be tension surface splitting.

Overall, these observations clarify the various features that occur on the force-time signals collected in the impact studies.

5. CONCLUDING COMMENTS

This study of toughness of CF/PEEK composites has provided an early history of developments of thermoplastic based composites, together with an identification of some of the early toughness studies. These were mainly based on delamination toughness (inter-laminar fracture). The results reported here expand on the early approaches. In particular, attention is given to intra-laminar fracture toughness.

The dilemma of the influence of anisotropy in the derivation of the compliance functions, and in particular some of the geometry terms, has been resolved. To a good approximation, it would appear that the isotropic geometric functions can be used for unidirectional composites.

Impact toughness obtained from instrumented impact machines presents problems in the interpretation of the force-time signals. The characteristics of these signals are strongly influenced by the degree of electrical filtering in the monitoring circuitry. Reliable data can be obtained from instrumented tests only if an unfiltered signal is analysed. Photographed impact and low energy impact help resolve some of these issues and a clear understanding of the failure events follows.

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