

CHARACTERISING FRACTURE IN POLYMERS AND COMPOSITES

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INTRODUCTION

Technical Committee 4 of ESIS has been working for about ten years on the problems associated with characterising the toughness of polymers and composites. The primary goal has been to develop protocols for standard test procedures which can be adopted as standards. Standard tests are crucial to the use of any fracture mechanics design methodology, since they rely on using data obtained under well defined conditions.

There are special problems associated with polymers, but there is a large simplification which can be made since for many cases of practical significance LEFM conditions pertain. Thus the problems associated with ductility which dominate many metals tests are absent but are often balanced by other difficulties. In some cases, of course, the materials show considerable ductility, particularly in films and then ductile fracture mechanics must be pursued.

$K_c - G_c$ TESTING OF POLYMERS

The first major effort of the committee was to establish a standard to determine K_c and G_c for polymers. The procedure was based on the ASTM standard for metals, E399, and used SENB and CT specimens. Notching is a special problem for polymers and the standard defines methods for generating natural cracks which are then initiated in the test. Fatigue loading, as used in metals, is not very useful in polymers because of difficulties in growing stable cracks and with hysteretic heating if high frequencies are used. For glassy polymers a tapped new razor blade is used, while for tougher materials, such as polyethylene, a sliding blade is employed. The definition of initiation is as problematic here as in metals and the $P_{5\%}$ load is used. This defines the load for 5% decrease in stiffness and, while not a rigorous definition, can be determined with reasonable repeatability.

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Validity of the results is determined via the degree of linearity via $P_{max}/P_{5\%} < 1.1$ which ensures that the calculation method is correct. The size criteria used are the same as for metals;

$$B, W - a, a > 2.5 \left(\frac{K_c}{\sigma_y} \right)^2$$

which ensures plane strain at the crack tip. When fixing these criteria the data available is always limited, but such data that exist would suggest that this is a sensible limit. The timescale of the tests must always be defined because of the visco-elastic effects and the same one must be used to measure σ_y .

The procedure has been found to be reasonably successful and it has been adopted as an ASTM standard and is currently under consideration by ISO. From a fundamental point of view, however, it should be noted that the K_c value is for some prescribed amount of crack growth which is size dependent and there is no rigorous way of determining the stress state. If a resistance or 'R' curve exists for the material, then this method will result in size dependent values. In many cases the 'R' curves are apparently rather flat so there is little variation. It should also be noted that notching is crucial and that any blunting, or local damage can lead to high initiation values usually signified by unstable fractures and rough fracture surfaces. Initiation from a true natural crack usually results in stable growth and a smooth fracture surface in these tests.

G_c is often preferred to K_c for polymers because it is widely used in composites and, it can be argued, is a better indication of toughness. The simple expedient of using K_c^2/E is not available for polymers since E is not usually known at the test rate so the standard uses the energy route to find G_c directly;

$$G_c = \frac{U}{BW\phi}$$

where U is the energy to initiation. Care must be taken to correct for indentation and a useful cross check is provided by computing E from K_c and G_c and from the compliance. The method appears to be satisfactory, but the limitations in defining initiation remain.

SHORT FIBRE COMPOSITES

A modification of the basic method has been developed for short fibre reinforced polymer composites. There are special problems here since the samples are almost always injection moulded and have a distinct skin-core structure. They are also anisotropic so the crack direction must be defined. Any notion of a critical thickness is invalid and the values determined are for a particular thickness, though it is sometimes possible to partition these into skin and core values by testing different thicknesses. The fracture initiation condition is also rather untidy in these systems, since the crack will often start growing, but then arrest, with a significant load rise thereafter. This is much akin to the "pop-in" condition in metals and care must be exercised in reporting the initiation condition. Even with these limitations this remains one of the best methods available for characterising these rather complex materials.

IMPACT TESTING

A further extension has been to use the same methodology for tests up to 1m/s testing speed. Many polymers show significant toughness decreases at these higher speeds so that impact testing is important. The speed is limited to 1m/s to enable the load trace to be used and limits are applied to the load oscillations allowed. To achieve this, damping of the load point is used and it is a balance between the rate induced by the speed and the slowing down from the damping. Interestingly the damper induces initial non-linearity, so that the rate of loading at impact is still high, but the total time to failure is increased. It is not clear whether it is this current rate, \dot{K}_c , which is important or the total failure time. Damping rather complicates the energy analysis since the damper absorbs a large fraction of the energy applied. An apparently satisfactory protocol is now available for the test.

For testing speeds greater than 1m/s the load route must be abandoned and a method based on measuring failure time has been developed. Here a modulus must be assumed to compute the load, or energy, but the method seems to work well since only a time is measured. Initiation is determined from a gauge on the specimen which greatly complicates this experiment. Speeds of up to 25m/s have been used and there is some evidence of a minimum loading time occurring.

COMPOSITE LAMINATES

Polymer matrix laminate which use carbon or glass fibres are widely used so there is a demand for a measure of inter-laminar toughness. This has produced a large world wide effect on mode I testing using the DCB test and a standard has been submitted to ISO which has been agreed between ESIS, ASTM and JIS. Again the system is LEFM since the uniaxial laminates used are dominated by the fibres and linear deformation results. The specimens are calibrated via compliance measurements during the tests in which load, displacement and crack length are measured simultaneously during the stable crack growth. Direct area methods can be used, but are inaccurate and beam theory corrected for shear, crack tip rotation and large displacements is used. The form using all three measurements is preferred since it avoids the use of E in order to find G , but E may be determined as an extra check on the method.

The test gives an 'R' curve and steeply rising curves are usually associated with fibre bridging. Initiation is again a problem and hence the P5% is used as well as values at onset of non-linearity and of visual detection. These latter are rather subjective, but can give lower values and are sometimes favoured.

Laminates are one of the rare cases in which mixed mode fracture can occur and particularly in impact Mode II is believed to be important. There has thus been much effort expended on establishing tests in pure mode II and in various mixed mode ratios. These tests have proved to be very difficult to develop to the degree of consistency achieved in the mode I test. This is largely due to problems with calibration, since in all of these the zero crack length compliance is substantial, while for the DCB it is zero. The methods all require dC/da and it is simply very difficult to achieve sufficient accuracy. In principle this could be overcome by numerical calibration as in K_c specimens, but the variability of the samples inject sufficient inaccuracy to thwart this approach. The matter remains unresolved at this time.

An interesting factor in laminates is the pre-notching of the specimens. Much early work in the field did not use pre-cracking because of the occurrence of fibre bridging, ie a steep 'R' curve was assumed and thus initiation value could only be obtained from a non-bridged crack. This led to the use of moulded in flaws, but these are often blunt, giving high initiation values and subsequent crack jumping. Whilst current practice allows this approach, pre-cracking which avoids bridging is preferred, eg by pre-cracking in mode II.

The methods used in laminates result in 'R' curves of G versus crack growth with various initiation conditions. These are directly comparable with the G values obtained in the polymer tests which is why the G_c approach is more popular in polymers.

DUCTILE FRACTURE

For many polymers the tougher materials have low yield stresses which promotes ductility, but leads to large thickness requirements for LEFM tests. Some of these materials cannot be made in sufficiently large sizes and it is unreasonable to try and achieve plane strain conditions. However it is often desirable to compare toughnesses of such materials and schemes based on the J method used for metals have been explored.

A protocol for determining the 'R' curve via the multiple specimen method has been developed which uses the SENB specimen with a deep notch. For fully plastic deformation this has been shown to be close to plane strain conditions and early versions of the metals protocol defined initiation as deviation from a blunting line and stated that it would be equivalent to the LEFM G_{IC} value. In more recent versions this simple, and rather logical, position has been abandoned and initiation is defined at a crack growth of 0.2mm. The distinction between blunting and crack growth has also been removed since it is believed that the difference between the two cannot be determined. The protocol for polymers has followed this trend and the current version measures an 'R' curve and characterises it as,

$$J = C \Delta a^N$$

and the nature of the material is defined via C and N . For flat 'R' curve of course, N is small and J tends to a constant value. Typically for tough polymers such as nylon and polyethylene N is about 0.6. Initiation is defined at $\Delta a = 0.2mm$ and is arbitrary. There is no evidence to support size limitations and, indeed, these tests do not claim geometry independence. The 'R' curves so obtained are useful for comparing materials, but their fundamental basis is precarious.

It is possible to use the J scheme to determine 'R' curves for thin sheet, or film which gives the toughness under plane stress conditions. Such data is useful for comparing materials which are only ever used in thin form, but the procedure is rather complicated. A variant of the method which has become popular, is that of determining the Essential Work of fracture w_e . The method

assumes an energy per unit area of crack growth, the Essential Work, and a volumetric term. The former is assumed to scale with the ligament length while the latter will scale with the ligament length squared. A series of tests with different ligament lengths is performed and the total energy to fracture is determined and a plot of energy per unit ligament length versus ligament length should be linear extrapolating to w_e at zero ligament length. The method seems to work quite well and materials, particularly films, can be usefully compared on the basis of w_e .

The whole argument, of course, is based on the notion that the fracture energy can be partitioned and a close examination of the data shows that this is only an approximation. Whether it is a better approximation than defining an initiation value is open to question. A comparison of w_e and J_c values based on blunting lines has shown equality in some case, but the basis is not secure. There is much to be done in this field to develop sound methods which are practically useful.