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Mechanical characterization of polymers and composites with a servohydraulic high-speed tensile tester

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Résumé. — Cette étude aborde la mesure des propriétés mécaniques des polymères et composites soumis à des vitesses de déformation moyennes. Elle illustre au travers de deux exemples l'utilisation d'une machine d'essais servo-hydraulique à grande vitesse. Elle présente également un capteur optique d'extensométrie de conception nouvelle. Dans la première partie de l'étude, la nature vibratoire des essais de traction à vitesse élevée réalisés sur une machine servo-hydraulique est discutée, et une technique d'amortissement est proposée. Dans une deuxième partie, cette technique est appliquée à l'étude des propriétés en traction du PEEK non recuit et recuit dans le domaine des vitesses de déformation de 3×10^{-2} à $2,4 \times 10^{2}\ s^{-1}$ Les résultats montrent un accroissement systématique de la contrainte au seuil plastique ainsi que de la contrainte conventionnelle de rupture. Ces essais confirment que le traitement thermique de recuit accroît les propriétés mécaniques. La troisième partie de l'étude porte sur des essais de délaminage en mode I d'un composite unidirectionnel IM6/PEEK à des vitesses de déformations nominales comprises entre 1×10^{-6} et $8 \times 10^{-1}\ s^{-1}$. Aux vitesses les plus élevées on a recours au filtrage par la FFT pour l'analyse de la mesure de la force. Les résultats mettent en évidence une légère diminution des propriétés interlaminaires lorsque les vitesses de déformation augmentent.

Abstract. — This study is concerned with the measurement of the mechanical behaviour of polymers and composites at intermediate strain rates. It illustrates with two examples, the application of a high speed servohydraulic testing machine and a newly designed optical extensometer. In the first part of the study, the dynamic nature of high speed tensile testing with servohydraulic apparatus is discussed, and a damping technique is proposed. In the second part, this technique has been applied to the measurement of the tensile properties of annealed and un-annealed neat PEEK at strain rates between 3×10^{-2} and $2.4 \times 10^{2}\ s^{-1}$. Both materials show increased yield stress and drawing stress with increasing strain rate. However, annealed specimens have been shown to exhibit superior mechanical properties. In the third part, mode I delamination tests have been performed on unidirectional IM6/PEEK composites at nominal strain rates between 1×10^{-6} and $8 \times 10^{-1}\ s^{-1}$. At the higher velocities the analysis is performed by means of FFT filtering. A moderate reduction in interlaminar fracture toughness was found with increasing loading rate.

1. Introduction.

There is currently increasing interest in the study of the behaviour of polymers and composites at high strain-rate and under impact conditions, with manufacturers often looking to modify the basic organic material to improve their impact properties.

In order to understand the deformation mechanisms of polymers, it is desirable to be able to measure their mechanical behaviour over several decades of strain rate.

The use of several test methods to cover a wide range of strain rate for material characterization often fails to provide consistent data. Moreover, even results from different impact tests do not correlate [1].

The aim of the present work is to evaluate the possibility of developing new testing methods to measure the strain-rate dependency of polymer based materials. The tests investigated are semi-routine tests with the aim of avoiding long and expensive instrumentation of specimens.

Manufacturers of testing equipment produce servohydraulic high speed testers able to provide a large controlled displacement for velocities up to 20 m/s. At velocities in excess of about 1 m/s, the tests become dynamic in nature. This involves metrological problems, which as yet, have remained mostly unsolved, either by the manufacturers or by the users.

In this study we will try to clarify some specific problems encountered in the regime in which inertial forces become non-negligible.

In the first example, the inertial forces creating harmonic vibrations are induced by the pickup device of the testing equipment, as is common to all high speed servohydraulic testers. It involves the uniaxial tensile testing of neat PEEK from strain rates of 3×10^{-2} to $2.4 \times 10^{2}\ s^{-1}$

Harmonic vibrations induced by the tested structure itself are treated in the second example. This is a study of mode I delamination of a unidirectional composite APC2 at opening speeds of 1 mm/min to 1 m/s.

2. Medium velocity servohydraulic equipment, and specific instrumentation.

These tests are based on the servohydraulic tensile equipment Schenck shown in figure 1. The piston rod is driven by a 2 stage servo valve for velocities up to 1.3 m/s, and by a 3 stage valve with proportional opening up to 11 m/s. An LVDT transducer located in the actuator measures the position of the piston. This measurement is used to control the servo-loop of the machine. Essential parts of the set-up have been adapted or developed in this laboratory as will be mentioned where appropriate in the text.

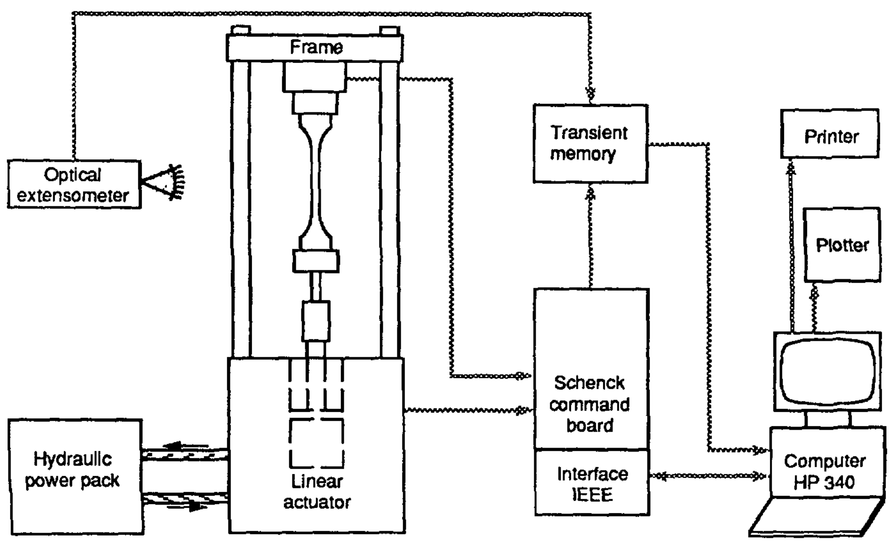

Fig. 1. — Schematic diagram of test apparatus for obtaining intermediate rates of strain.

2.1 DYNAMIC EFFECTS. — On account of the high mass of the piston rod in hydraulic testers, it is accelerated progressively to the desired velocity. The stabilized velocity is reached after a maximum acceleration displacement of 100 mm. For this reason, the piston is accelerated without contact with the specimen. The device used to transmit the motion to the specimen is shown in figure 2. It will be called the « pickup » unit. The motion is transmitted to the specimen once the end of the track rod reaches the stop cup.

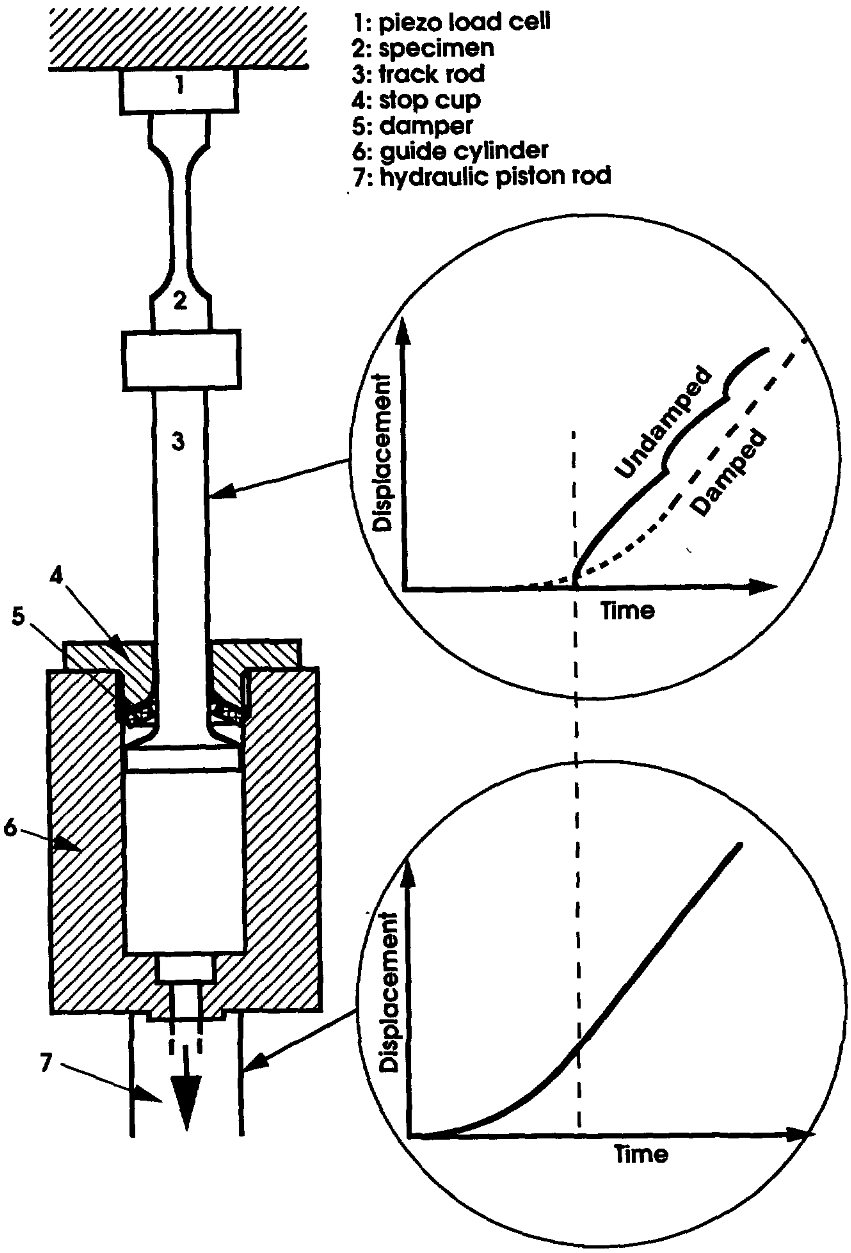

Fig. 2. — Mechanical design of the pickup unit.

The dimensions and consequentially the masses of these components vary from one manufacturer to another, but the principle remains the same.

In the first stage of this work we have studied the influence of the contact established in the pickup unit on the nature of the test.

Contact of the titanium track rod against the steel stop cup will produce an elastic interaction. Owing to the momentum balance, the motion transmitted to the specimen is a damped harmonic linear motion. The initial velocity of the track rod is higher than that of the piston rod, and stabilizes after a few oscillations. The testing velocity at a given piston speed depends on the stiffness of the materials at the surfaces of contact, the compliance of the specimen, and the masses of the mechanical fixtures. The test is harmonic in nature, being an impact test.

As an example, figure 3a shows the stress, and the moving grip position *versus* time for a 4 m/s tensile test on annealed PEEK, with an undamped contact in the pickup unit.

As well as the nature of the contact, the amplitude and the frequency of the load oscillations depend on the mass and the rigidity of the load cell and the grips (mass spring behaviour).

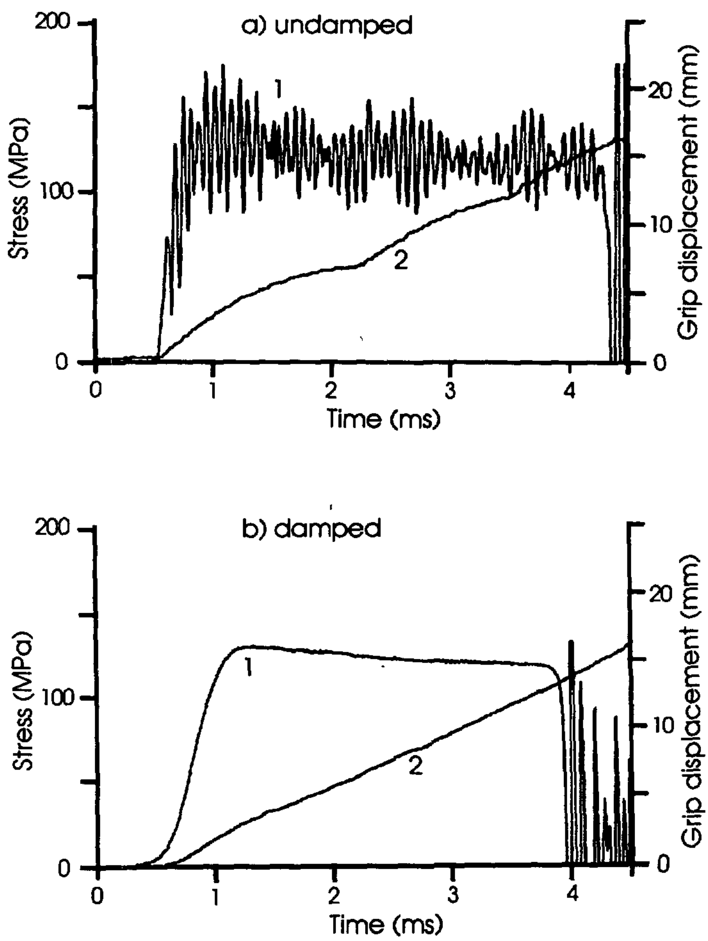

Fig. 3. — Typical stress (1) and grip displacement (2) *versus* time measurement at 4 m/s, a) undamped contact, b) damped contact.

With the insertion of a viscoelastic material between the contact surfaces, the impact is damped and thus the track rod is smoothly accelerated. A viscoelastic contact in the pick up unit will increase the rise time of the velocity of the moving grip. The speed of the track rod will progressively tend to that of the piston rod, without bouncing.

Figure 3b, shows the same test a 4 m/s with a damped contact. The contact-material used in this study is a filled polyurethane elastomer, with a low crosslinking density (Viscolas®, Chattanogga Corporation), designed for orthopaedic application. It has been selected for its excellent damping properties [2].

The examples of figure 3 show that this technique changes a damped harmonic strain rate into a nearly constant strain rate, in equilibrium throughout the specimen and the mechanical parts of the equipment.

It is important to realize that when a test produces transient stress waves in the specimen, stress and strain variables have to be measured at the same location on the specimen. The reason for this is that sensors placed at different locations will record a phase shift between stress and strain, owing to the wave velocity, which is not negligible at intermediate strain rates.

It is open to discussion whether more can be learned by switching from a dynamic test of the impact type, to a rapid but quasi-static test, which is in many aspects similar to a low velocity test. In this study we wish to separate the dynamic effects, which are highly dependent on the shape and the size of the specimen, from intrinsic material properties at high rates of strain.

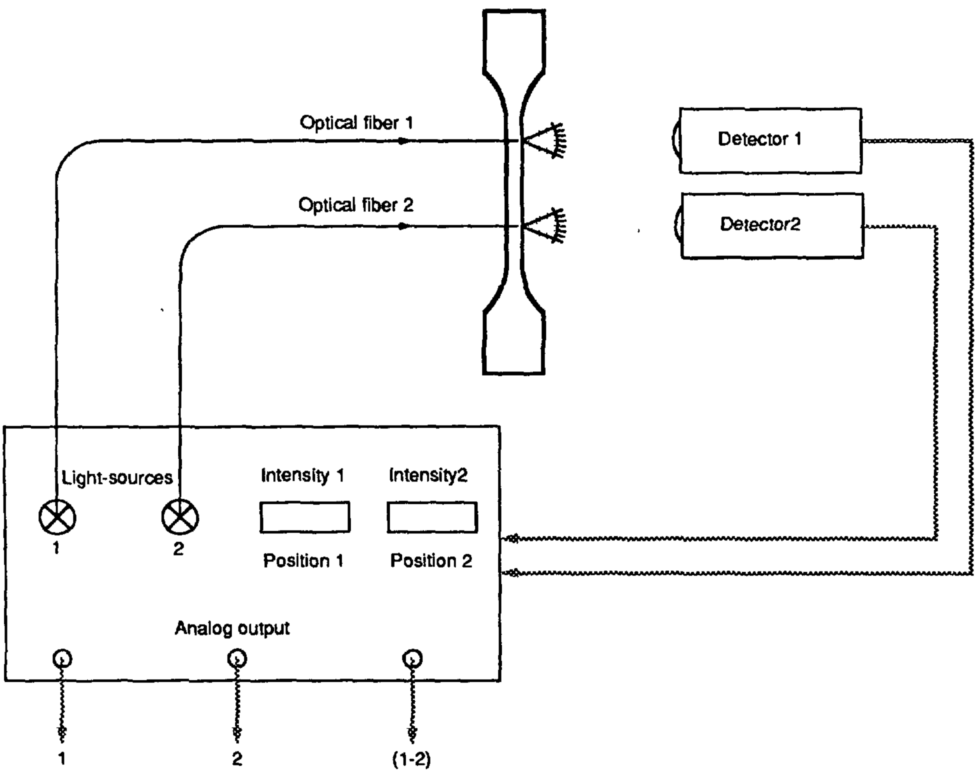

Fig. 4. — Optical extensometer, schematic diagram.

2.2 DISPLACEMENT AND TRUE STRAIN MEASUREMENT. — The position of the piston rod is easy to measure by use of the LVDT of the machine. In fact rapid change in the piston rod velocity are not possible because of its relatively large mass.

Owing to the use of the pickup unit, neither the position of the track rod, nor of the moving grip, nor the strain in the specimen can be deduced from the position of the piston. Any classical mechanical device for measuring strain would be slipping away or even be damaged by the high acceleration and deceleration during the test.

For this reason we have developed a new optical extensometer. The design of this device is shown schematically in figure 4.

The system is based on the continuous measurement of the position of one or two light points placed on the moving parts. The light is emitted by lasers, and injected into optical fibres. The free ends of the optical fibres are then attached to the moving or strained part, acting as light emitters. Because of the low mass per unit length (~ 70 mg/m) of these thin fibres (250 μm in diameter), they are virtually unaffected by the acceleration, and hence no stress is induced in the specimen.

One or two optical detectors monitor the position of the light points. An electronic device supplies one or two analog signals proportional to the absolute or relative position of the points.

The lens of the detector can be changed according to the measuring range needed. The system is independent of ambient light.

2.3 LOAD MEASUREMENT. — The load is measured by means of a piezoelectric crystal ring mounted in the fixture assembly and placed on the stationary side of the specimen.

As a rule, it is necessary to decrease as much as possible the inertial effect of the fixture assembly. We have developed rings and fixations with reduced masses, mostly titanium parts. The two assemblies built for the present work have the following properties (without sample) :

Test type	Mass (kg)	Resonant frequency (kHz)
Tensile	0.4	~ 14
DCB	0.0065	~ 60

We show here two examples of application of this testing equipment to thermoplastic based materials. More recently it has also been used by Barbezat (3) to characterize the mechanical properties of epoxy based materials.

3. Uniaxial tensile test on an unreinforced tough thermoplastic.

The above described technique has been applied to uniaxial tensile testing at room temperature.

3.1 MATERIAL. — Dog-bone samples of PEEK (Poly-Ether-Ether-Ketone) 450 G have been moulded by injection at 400 °C. The mould temperature was 120 °C instead of 200 °C as recommended by the supplier. As shown by Jar *et al.* (4) in their study on these specimens, a low mould temperature results in an outer skin which is nearly amorphous over a depth of about 100 μm. The bulk has a semi-cristalline structure with a degree of crystallinity of 28 %. This external amorphous structure changes to a homogeneous structure with 31 % crystallinity after annealing the samples for 24 hours at 250 °C in an oven.

3.2 TENSILE TESTS. — Annealed and unannealed samples have been tested at increasing testing velocity.

The sample cross-section was 6 mm by 3 mm, and the length of the reduced cross-section was 40 mm. The initial length between light points was 30 mm. For velocities of the piston rod higher than 0.1 m/s ($\dot{\varepsilon} > 3\ s^{-1}$), the contact in the pickup unit has been damped as described in section 2.

The velocities used were 0.001, 0.01, 0.1, 1, 4 and 8 m/s, and correspond to measured strain rates $\dot{\varepsilon}$ of 0.03, 0.3, 3, 30, 140 and 240 s^{-1}

Figure 5 shows typical stress-strain curves for a) unannealed and b) for annealed samples.

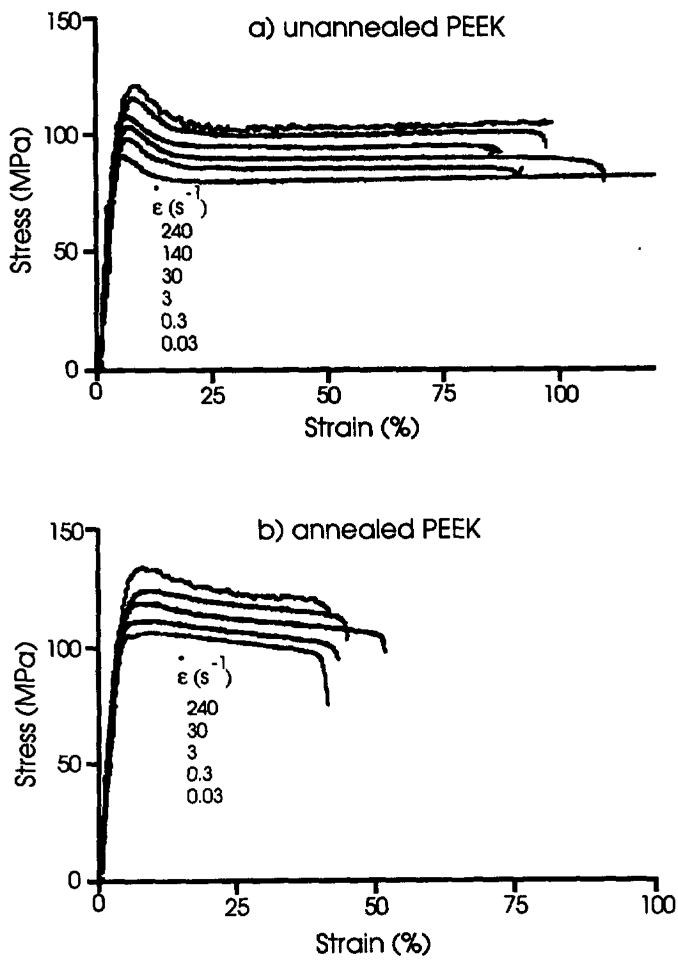

Fig. 5. — Tensile behaviour at different strain rates a) unannealed b) annealed PEEK.

3.3 RESULTS AND DISCUSSION. — The yield stress and the engineering post yield stress of the annealed PEEK were higher than those of the unannealed PEEK over the whole range of strain rate. The yield stress is plotted *versus* the log of the strain rate in figure 6. The increase of the yield stress over 4 decades of strain rates amounts to about 30 %. Similar results have been obtained for the engineering rupture stress (Fig. 7).

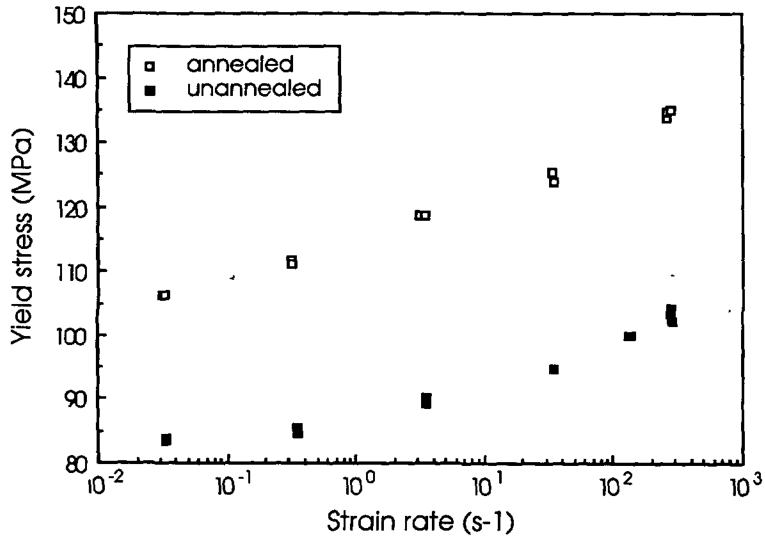

Fig. 6. — Yield stress σ_y of PEEK *versus* strain rate $\dot{\varepsilon}$.

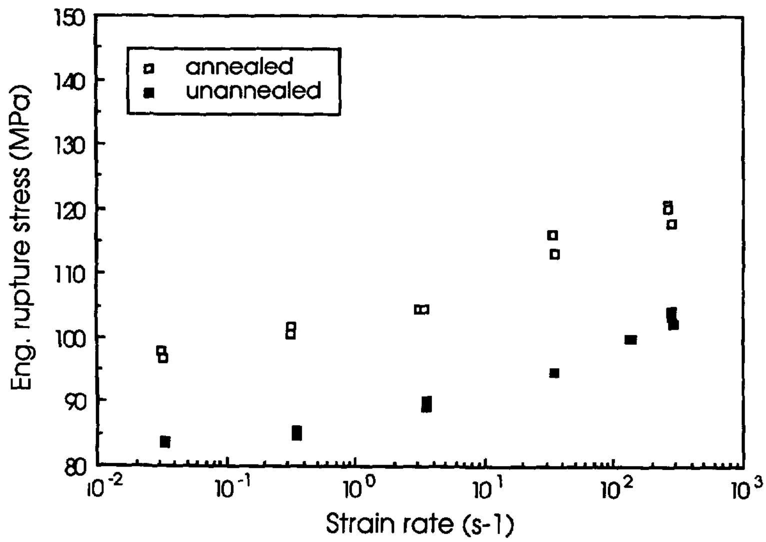

Fig. 7. — Engineering rupture stress σ_r of PEEK *versus* strain rate $\dot{\varepsilon}$.

Differences have been noted concerning the post yield plastic deformation process. For the unannealed material, the necking is initially localized and tends to propagate along the gage section. Apart from at the lowest velocity, rupture occurs before the completion of necking of the gage section. The plastic deformation of the annealed samples is simultaneously distributed over a larger volume of the gage section than for the unannealed material, and the necking is less pronounced.

Defects growing during the plastic deformation process of annealed PEEK are the major reason for its relatively low elongation at break as compared with the unannealed case.

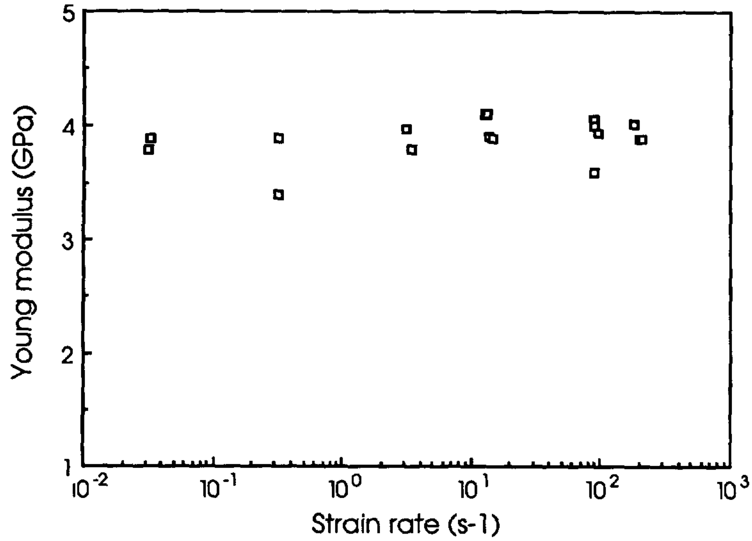

Fig. 8. — Young's modulus E of annealed PEEK *versus* strain rate $\dot{\varepsilon}$.

Young's modulus *versus* the log of the strain-rate for the annealed PEEK is plotted in figure 8. The diagram shows no significant change in the modulus.

Our tests demonstrate the relatively low strain rate dependence of the mechanical behaviour in tension of PEEK. The nearly amorphous skin due to the injection moulding conditions increases substantially the elongation at break by changing the macroscopic deformation process.

More morphology-based studies of the influence of the structure of PEEK for lower strain rates have shown similar enhancement of the mechanical properties with annealing [4-6].

4. Fracture mechanics on unidirectional composites.

A composite material has been tested in mode I delamination over a wide range of opening rates.

4.1 MATERIAL. — The material is the APC2 (Aromatic Polymer Composite), manufactured by ICI. The PEEK matrix is unidirectionally reinforced with IM6 carbon fibres, 5 μm in diameter.

The volume fraction of fibres is 62 %. Panels of 3 mm thickness have been made by assembly of 24 plies of prepreg under pressure at elevated temperature (380 °C) in a heating press. The controlled cooling at a rate of 50 K/min provides a crystallinity of the matrix of ~ 25 %.

4.2 DELAMINATION TESTS. — The panels were cut into bars of 20 mm width and 180 to 200 mm in length. Double cantilever beam specimens (DCB) where prepared by gluing hinges for load introduction. According to Davies *et al.* [7], the thickness of the specimen (3 mm) tends to decrease the risk of fibre bridging during the propagation. Aluminium starter films (12 μm thick) where inserted at the mid-thickness. Davies *et al.* [8] tested the influence of the thickness of such films, and concluded that a 12 μm aluminium foil is a suitable starter.

The specimen shape is shown in figure 9. Note the lightened hinges (~ 3 g) in order to minimize additional masses.

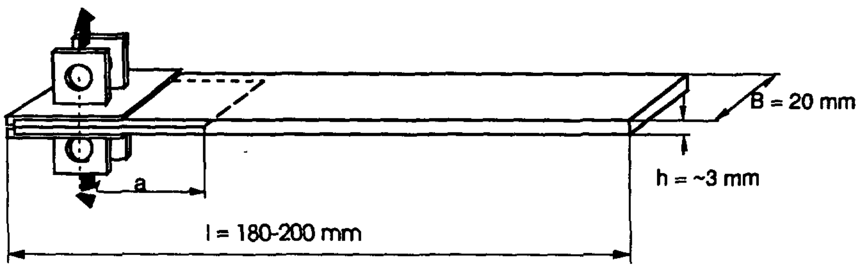

Fig. 9. — DCB test specimen with lightened hinges.

Tests in the range of 1 to 100 mm/min opening rate have been performed on an Instron 1122 machine. From 1 m/min to 1.1 m/s the high speed servohydraulic Schenck was used.

The damping in the pickup unit has not been utilized for these tests. In view of the relatively low testing velocities and the large compliance of the specimens, the dynamic effects induced in the pickup unit are likely to have been dissipated when first crack initiation occurs.

4.3 DATA REDUCTION. — The interlaminar fracture toughness is characterized by the critical strain energy release rate G_{Ic}. As it was not possible to perform either real time optical measurements of the crack position or post-fracture measurements, the values of G_{Ic} were calculated using Berry's experimental compliance [9, 10]:

$$C = ka^n$$

Where a is the crack length, and k and n are empirical parameters. Note that these parameters have been measured over the whole range of testing speeds.

G_{Ic} is expressed as:

$$G_{Ic} = \frac{nP_c\,\delta}{2\,Ba}$$

n Berry's coefficient
P_c . the critical load
δ the opening of the specimen at the load P_c
a the crack length.

For a pair of linear elastic cantilever beams, joined at their fixed ends, the maximum strain deflection is:

$$\varepsilon = \frac{3}{4}\frac{h\delta}{a^2}$$

h the depth of each side of the DCB specimen
δ the opening displacement.

The crack tip strain is related to this nominal strain by the strain intensifying effect of the crack.

According to Mall *et al.* [11], the nominal strain rate near the crack tip $\dot{\varepsilon}$ is then expressed through the opening velocity $\dot{\delta}$ as:

$$\dot{\varepsilon} = \frac{3}{4}\frac{h\dot{\delta}}{a^2}$$

4.4 RESULTS AND DISCUSSION. — At crosshead speeds above 1 mm/min, which approximately correspond to nominal strain rates larger than $10^{-5}\ s^{-1}$, the crack propagation was unstable, with stick-slip behaviour. This allows to measure several crack initiation values on the same sample. Figure 10 shows typical measurements at low opening speed.

For higher velocities, the free oscillations of the arms of the sample, occurring immediately after crack propagation tend to mask the reloading of the specimen.

Despite the high resonant frequency of the stationary load cell, it becomes more difficult to define the crack initiation points on the load plot at a velocity higher than 0.1 m/s. For these cases, we use a filtering technique based on the Fast Fourier Transform (FFT).

The time-spectrum data from the load cell are converted into a time-frequency spectrum by the FFT. The resonances occurring during the test become evident in the frequency spectrum (Fig. 11). Those frequencies are filtered from the spectrum by total removal of the signal intensity within the window where high amplitude resonance occurs. Then the inverse FFT is applied.

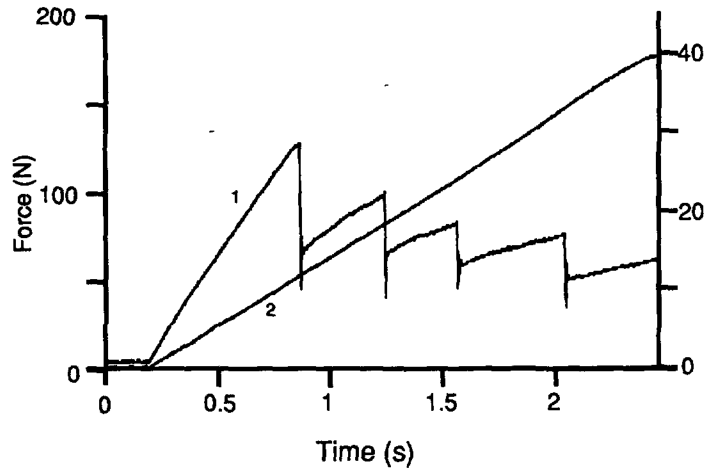

Fig. 10. — Typical measurement at low velocity (1 m/min). 1) Force. 2) Displacement.

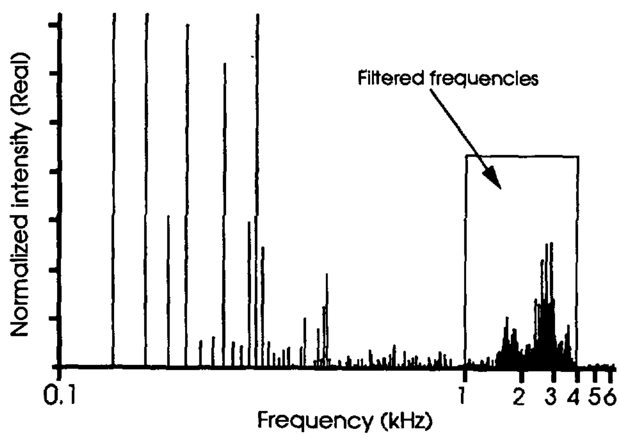

Fig. 11. — Force curve of figure 12 (1), real intensity of the frequency spectrum.

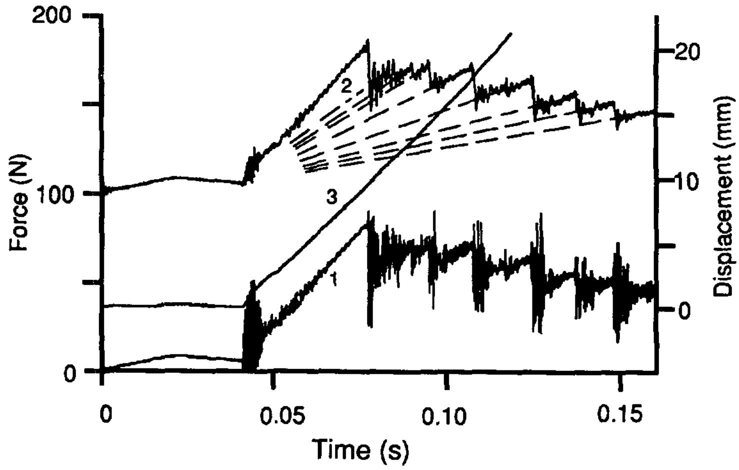

Fig. 12. — Typical measurement for an opening velocity of 0.5 m/s. 1) Original force trace. 2) Same curve (shifted 100 N) after removal of the 1 to 4 kHz spectrum. 3) Sample opening at the tabs.

Figure 12 shows clearly the interest of this technique. On these data, the propagation points and the subsequent reloading appear more clearly in the filtered data.

Calculations and measurements have shown that the frequencies removed in this example are exactly in the range of the resonant frequency of a single cantilever beam of the shape, size and material used here.

This demonstrates that for this composite it is necessary to modify the sample geometry in order to study the delamination at higher rate.

Tests performed at 2.5 m/s have not been considered here in view of the difficulty of data interpretation.

For some specimens, the force-displacement diagram was slightly non-linear just prior to the drop in force due to unstable crack propagation. This deviation from linearity is believed to be due to the first appearance of damage around the crack tip, such as multiple microcracking, crack shape adjustment or crack blunting [12]. In this study, calculation of G values has been based exclusively on the peak-loads prior to crack propagation.

The results of these tests are plotted in figures 13 and 14.

In figure 13, only the initiation values of the first crack propagation in each specimen have been plotted. These crack propagations are initiated in matrix-rich regions of the samples [8].

Further re-initiation of the cracks (Fig. 14) is in principle more strictly related to interlaminar regions, but after a crack propagation of 5 to 10 mm, fibre bridging occurs, tending to increase the G values. This, along with the experimental compliance calibration, is the main source of scatter in the data.

These results do not show the catastrophic decrease of the interlaminar properties of APC2 with increasing strain rate, found by Smiley [13] for similar materials. The results show only a moderate reduction of G with increasing strain rate.

5. Conclusion.

High rate testing of materials often produces dynamic phenomena. In the present study these have been treated differently depending on the source of harmonic vibrations.

In the first example, it was shown that the motion imparted on the specimen by the tensile tester is responsible for the dynamic nature of the tensile tests. The use of a viscoelastic material in the pickup unit reduces the acceleration of the specimen. This practice results in

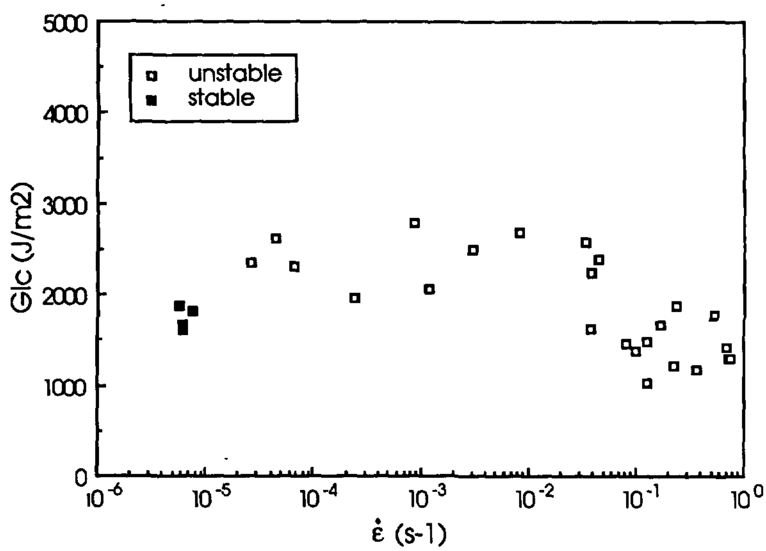

Fig. 13. — Interlaminar fracture toughness (G_{Ic}) in mode I for APC2, *versus* nominal strain rate $\dot{\varepsilon}$. Initiation from the aluminium film pre-crack only.

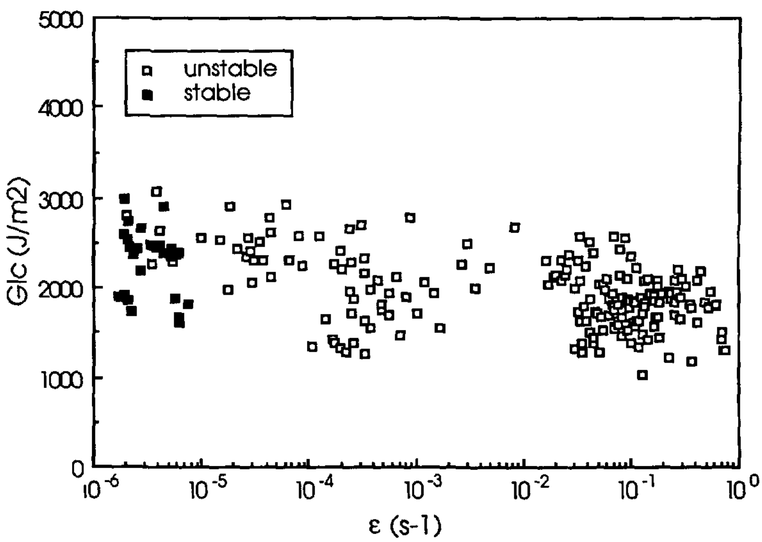

Fig. 14. — Interlaminar fracture toughness (G_{Ic}) in mode I for APC2, *versus* nominal strain rate $\dot{\varepsilon}$. All propagation data presented.

the test being quasi-static rather than dynamic in nature. It may be seen as an extension of the usual low rate testing of materials, as it uses the same variables and analysis.

This approach does not provide impact data but permits the measurement of intrinsic material parameters at intermediate rates of loading, which we believe to be very useful for numerical simulation of impacted structures.

To apply this technique, it is essential to be able to measure the true motion of the moving parts with an appropriate device such as the optical extensometer used here.

In the second example the harmonic oscillations appearing on the load recordings of delamination tests result from the fracture process itself. In this case the data are filtered by the use of the Fast Fourier Transform.

Although this technique is employed more and more often in the interpretation of impact data [14], it should be used with caution.

It is applicable in our particular case because the decay of the oscillations emitted by crack propagation is nearly completed before any further crack initiation occurs.

It has to be kept in mind that filtering should be used only as an aid to data interpretation. Thus, it should be applied only to a copy of the original data, measured with cells having the highest possible resonant frequency.

Acknowledgments.

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