ESIS TC4 ACTIVITY ON HIGH RATE TESTING OF PLASTICS

A. Pavan*

This is an account of a part of the activity that ESIS Technical Committee 4 applied itself to in the area of the high rate testing of plastics. The technical issue it focused on was the development of a standard procedure to determine K_{IC} and G_{IC} at 1 m/s testing speed.

This was pursued through a series of round-robins covering a range of material samples, specimen geometries, test instruments and operational conditions. This activity involved about thirty European laboratories in eight years and enabled the group to gain sufficient experience to establish and validate a satisfactory testing protocol which has now been submitted to ISO for adoption as a standard.

The main problems faced in developing the protocol are here recollected and the solutions devised and taken in the protocol are briefly illustrated.

INTRODUCTION

Technical Committee 4 of ESIS has been working since its establishment in 1985 on the problems associated with characterizing the toughness of polymers and composites. The primary goal has been to develop protocols for testing procedures which can be adopted as standards.

The first major effort of the committee was to establish a standard to determine K_{IC} and G_{IC} for polymers. A protocol validated by a series of round robins was finalized in 1990 (Williams (1)) and it has been adopted as a standard by ASTM (2) and is currently under consideration by ISO (3).

An extension of that procedure to test polymeric materials at high rates of loading (testing speeds of the order of 1 m/s) was deemed desirable. Many polymers show significant toughness decreases at these higher speeds so that characterization of fracture resistance under these conditions is important. Developments in instrumentation in recent years, on the other hand, offer nowadays the possibility of visualizing the high speed loading processes precisely and suggest the possibility of applying the same basic fracture mechanics methodology also to impact testing. The problems arising from the "dynamic effects" which are encountered at high loading rates, however, are to be recognized and taken into proper consideration.

* Dipt. di Chimica Industriale e Ingegneria Chimica, Politecnico di Milano, Italy

DYNAMIC EFFECTS

The dynamic phenomena observed when a testpiece is rapidly loaded have two possible origins. One is the finite, though great, speed of the stress wave propagation in the material under test, which prevents stresses from attaining equilibrium during the short lapse of the impact event, and is inherent in fast loading; the second is mainly instrumental, depending largely on the characteristics of the test apparatus used. The relative importance of the two varies with the rate of loading.

At very high loading rates the time-scale of the fracture event is comparable with the time taken by the stress waves to travel across the test piece, and stress wave reflections and interference with the crack may show up some effect.

At moderately high loading rates (load-point displacement rates of the order of 1 m/s, loading times of the order of 1 ms) it is the dynamic effects arising from the specimen motion which predominate: the inertial forces caused by the acceleration set on the specimen produce vibrations in the test system, oscillations in the recorded signal, forces on the test specimen different from the forces sensed by the test fixture, and possibly loss (and regaining) of contact between the specimen and the tup of the moving arm of the testing machine and also between the specimen and the shoulders of the mounting vice.

At still lower loading rates these effects become negligible and the fracture mechanics methods normally used for quasi-static test conditions (e.g. (1)) can be applied as they stand.

It is evident from the examples in Fig.1 that at high rates the amplitude of the oscillations may represent a high proportion of the total load, and the interpretation of the test record becomes difficult.

Considerable work has been published, dealing with the assessment, analysis, modelling and control of the dynamic effects manifested by fluctuations of the measured force signal such as shown in Fig.1 (e.g. Böhme and Kalthoff (4), Williams (5) and Williams and Adams (6), Zanichelli et al. (7)). As a result we are in the position now to answer the question of if and how the characteristic responses of the test specimen and the force measuring system can be separated. In fact, since a part of these effects is instrumental in origin, there is room for controlling these effects by improving machine design and adopting suitable test expedients. One such expedient is to damp the load point and this is illustrated below. For details, the reader should refer to the ESIS document (8).

CONTROL OF DYNAMIC EFFECTS

Once the fracture test has been performed and the load/time or load/displacement curve has been obtained, one of the problems arising is the identification of the point of fracture initiation. Several techniques are possible but most of them require sophisticated instrumentation and complex calibration procedures, especially so in the case of high-rate testing. Yet, major concern in developing scientifically based testing protocols is to maintain their practicability within the reach of laboratories having medium-level expertise and standard equipment. For this, it is desirable that the point of fracture initiation can be deduced from the load record. Two approaches are possible: either to reduce the oscillations of the recorded signal *a posteriori* by electronic filtering or to control the dynamic effects by some expedient. The latter is to be preferred as electronic filtering may wipe out real effects in the specimen response and it would not help when the period of the oscillations is comparable with the duration of the test.

Previous studies have shown that the force oscillations recorded by force transducers mounted in the tup are considerably greater than the ones actually experienced by the specimen at its crack tip (Venzi et al. (9) and Kalthoff et al. (10)) and depend largely on the 'contact stiffness' of the tup-specimen interface (Williams (6)). Some reduction of these effects by proper control of the 'contact stiffness' can thus be envisaged as possible. With impact testers the impact may be cushioned by means of a soft pad, placed where the tup strikes the specimen. With servo-hydraulic testing machines, initial acceleration of the specimen can be controlled by means of a damper applied in the motion transmission unit.

Ample evidence of the effectiveness of this expedient has been gathered within ESIS TC4. As the example in Fig. 2 shows, signal oscillations can be practically suppressed and thus the point of fracture initiation can be easily identified and the critical load easily read.

If damping is contained no adverse effects are observed. The value of the load at fracture is not affected and the load-point displacement rate (in a displacement control mode of testing) can be kept constant during the test, provided the testing machine is of sufficient capacity. Time to fracture is somewhat increased due to damping, so the testing speed needs to be adjusted to maintain the load-point displacement rate or the time-to-fracture fixed. (Both alternatives are considered in the protocol since it is still debated whether it is the current rate of loading, dK/dt, which is important (see e.g. Béguelin and Kausch (11)) or the total failure time (Frassine et al. (12))).

Overdamping may induce some initial non-linearity in the load trace. That effect must be balanced against the effect on load oscillations: to this end the protocol requires that damping is contained to a minimum sufficient to confine load oscillations within allowed limits (Fig. 3).

DETERMINATION OF K_{IC}

As in the low rate case (1) a limited degree of ductility is allowed here too and the same method is used to determine fracture initiation: the 5% offset load is taken and validity of the results is determined via the degree of linearity according to the $P_{max}/P_{5\%} < 1.1$ criterion.

In the presence of some, even if minor, oscillations in the load signal, however, application of the 5% offset construction is less straightforward. The protocol suggests to "smooth" first the recorded load trace by curve fitting it to the following equation

$$\bar{P}(t) = a(t - t_o) - b(t - t_o)^n \qquad \text{.......................(1)}$$

This procedure was tested on both linear (brittle) and moderately non-linear (semiductile) fractures, such as occurring with polyvynilchloride and rubber-toughened polymethylmethacrylate, respectively, and it was found valid.

DETERMINATION OF G_{IC}

Besides the characterization of toughness in terms of the critical stress intensity factor,

K_{IC}, based on the measurement of load, there is also some interest for plastics in determining the energy per unit area of crack, G_{IC}. In principle this could be obtained from K_{IC} via the modulus, E, but for polymers E is rather rate- and temperature-sensitive and so the low-rate testing protocol (1) has been extended to include the determination of G_{IC} directly from energy. Both load and displacement are measured in order to derive energy from integrating the load versus load-point displacement diagram; the method must include careful measurements and corrections for machine compliance and specimen indentation (unless an external displacement measuring device is used, e.g. optical).

At impact speeds and with a mechanical damping device in place, the area under the measured load/displacement curve, U_Q, contains additional spurious contributions which it needs to be corrected for before G_{IC} can be calculated.

As in the low-rate case, a portion of the correction can be estimated from a separate test, to be performed on an unnotched specimen (8). More problematic is the evaluation of the kinetic energy of the moving test specimen and of the energy associated with the inertial loads.

It has been demonstrated in (7) that inertial loads are essentially independent of crack length and the same can be held true for the kinetic energy term. Hence it is suggested in (8) to follow an alternative, multispecimen procedure which circumvents the need of evaluating those two terms at all. G_{IC} can be determined from the slope of a plot of fracture energy, U, versus the crack-length dependent calibration factor, ϕ (1), to be obtained by testing a series of specimens with equal dimensions but varying crack length. Since the two parasitic energy terms mentioned above are essentially independent of crack length, the slope will be unaffected and no correction is necessary.

The method is sketched in Fig.4.

CONCLUSIONS

Application of fracture mechanics to characterize impact toughness deserves special attention because of dynamic effects inherent in the test. With polymers, at speeds around 1 m/s these effects can be contained or circumvented, and K_{IC} and G_{IC} can be determined with sufficient accuracy. The protocol developed within ESIS TC4 (8) has thus now been submitted to ISO for adoption as a standard (13). It is believed that it can oust outdated impact tests such as the conventional Charpy and Izod tests.

For testing speeds greater than 1 m/s, control of the dynamic effects by means of mechanical damping may not be successful: the load route must be abandoned and other approaches must be sought. Developments in this direction are underway within ESIS TC4. A method based on measuring failure time is being tested and a protocol is being shaped.

ACKNOWLEDGEMENTS

The following organizations took part in several of the eight round-robins carried out to establish the 1 m/s testing protocol: Delft University of Technology (NL); EPFL, Lausanne (CH); Univ. de Oviedo, Gijón (E); IWM, Freiburg (D); BP Chemicals, Grangemouth (UK); Du Pont, Wilmington, DE (USA); Ciba-Geigy, Marly (CH);

Solvay, Brussels (BE); Martin-Luther Univ., Merseburg (D); The Welding Inst., Abington (UK); Univ. of Kaiserlautern (D); Montan Univ., Leoben (A); Pipeline Developments, Manchester (UK); ICI, Wilton (UK); Eastman Kodak, Kingsport, Tenn. (USA); Rhône-Poulenc, Aubervilliers (F); LMPL-ENSMA, Futuroscope (F); Cranfield Univ., Bedford (UK); Technical Univ., Budapest (HU); BASF, Ludwigshafen (D); Univ. of Twente (NL); INSA Lyon, Villeurbanne (F); EAHP, Strasbourg (F); VTT, Espoo (FI); DSM, Geleen (NL); Elf-Atochem, Serquigny (F); Imperial College, London (UK); Politecnico di Milano (I).

REFERENCES

(1) "A Linear Elastic Fracture Mechanics (LEFM) Standard for Determining K_C and G_C for Plastics", Testing Protocol prepared for ESIS TC4 by J.G.Williams, 1990.

(2) ASTM D 5045-93 "Standard Test Methods for Plane-Strain Fracture Toughness and Strain Energy Release Rate of Plastic Materials".

(3) ISO/DIS 13586 "Plastics. Determination of fracture toughness (G_C and K_C) - Linear elastic fracture mechanics (LEFM) approach".

(4) Böhme, W., and Kalthoff, J. F., J.de Physique, Coll.C5, No.8, Tome 46, 1985, pp. 213-218.

(5) Williams, J. G., Int.J.Fracture, Vol. 33, 1987, pp. 47-59.

(6) Williams, J. G., and Adams, G. C., Int.J.Fracture, Vol. 33, 1987, pp. 209-222.

(7) Zanichelli, C., Rink, M., Riccò, T., and Pavan, A., Polym.Eng.Sci., Vol. 30, 1990, pp. 1117-1124.

(8) "A Linear Elastic Fracture Mechanics (LEFM) Standard for Determining K_{IC} and G_{IC} for Plastics at High Loading Rates", Testing Protocol prepared for ESIS TC4 by A.Pavan (final version: Sept. 1997).

(9) Venzi, S., Priest, H. A., May, M. J., "Influence of Inertial Load in Instrumented Impact Tests", in ASTM STP 466, Am.Soc.Test.Mat., Philadelphia, Penn., USA, 1970, pp. 165-180.

(10) Kalthoff, J. F., Böhme, W., Winkler, S., and Klemm, W., in Proc. CSNI Specialists Meeting on Instrumented Precracked Charpy Testing, Palo Alto, Cal., USA, 1980.

(11) Béguelin, Ph., and Kausch, H.H., J.Mater.Sci., Vol.29, 1994, pp. 91-98

(12) Frassine, R., Rink, M., and Pavan, A., "On the viscoelastic time-dependence of fracture toughness at high loading rates in polymers", in "Impact and Dynamic Fracture of Polymers and Composites", ESIS 19. Edited by J.G.Williams and A.Pavan, MEP, London, England, 1995, pp.103-111.

(13) ISO/NWIP 694 "Plastics. Determination of fracture toughness (K_{IC} and G_{IC}) at high loading rates".

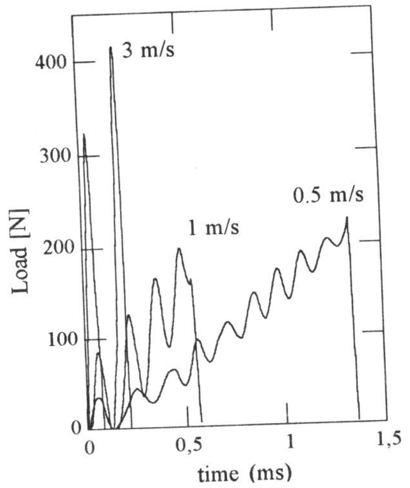

Figure 1 - Typical force/time curves recorded from SE(B) impact tests at different speeds. Material: nylon-6. Tester: CEAST Fractovis falling weight.

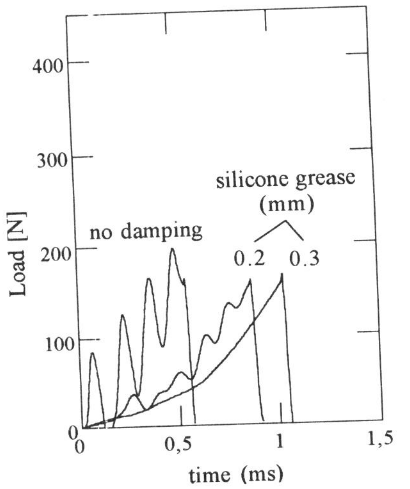

Figure 2 - Effect of placing a layer of silicone grease of varying thickness on a specimen of nylon-6 struck in three point bending at 1 m/s.

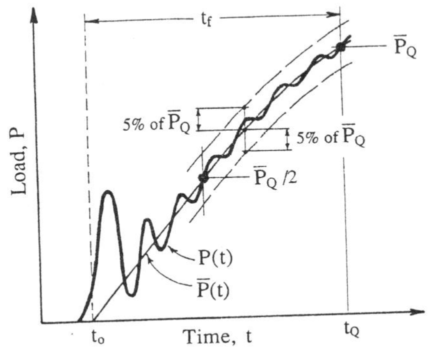

Figure 3 - Limits of allowable force fluctuations for the determination of fracture initiation load $\bar{P}_Q$ (see ref.(8) for symbols and details).

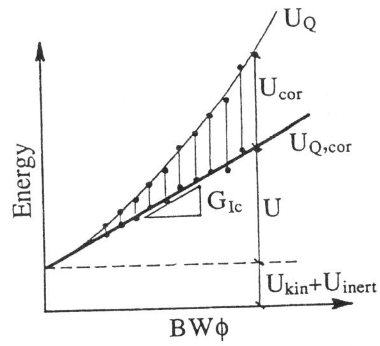

Figure 4 - Determination of G_{IC} from corrected energy, $U_{Q,COR} = (U_Q - U_{COR})$, with BW=const (see ref.(8) for symbols and details).