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Brittle–tough transition of rapid crack propagation in polyethylene

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The brittle–tough transition of rapid crack propagation in pressurised polyethylene pipes is examined. The transition is correlated with the presence of shear lips. Two new tests are described and then used to examine the mechanism of shear lip formation. The mechanism is found to be largely governed by the post-yield drawing behaviour of the polyethylene and is sensitive to both rate and temperature. The degree of drawing before failure is discussed from a micro-structural viewpoint and is shown to depend, at least in part, on lamella size and hence processing conditions. © 1998 Elsevier Science Ltd. All rights reserved.

(Keywords: brittle; tough; shear)

INTRODUCTION

It is well known that many polymers can behave in either a brittle or a tough manner. Usually a polymer will show a transition between the two behaviours at a critical temperature: the brittle–tough transition temperature (T_{BT}). The T_{BT} is dependant on many parameters, the most important of which are flaw size, deformation rate and geometry. The transition behaviour is classically described as a competition between fracture and yield stress[1]. Whichever stress is reached first dictates the type of failure. However, little progress has been made in quantifying this behaviour to allow predictive methods to be developed for general geometries and loading conditions.

Investigation of rapid crack propagation (RCP) in pressurised polyethylene pipes (a catastrophic failure mode) using a small-scale, steady-state (S4) pipe test[2] has highlighted one area where this transition is of particular importance. Plastic pipes display a critical pressure below which it is impossible to sustain rapid crack propagation. Above a material and geometry dependant T_{BT} this critical pressure rises sharply from a lower plateau (*Figure 1*). The work here concentrates on the nature of this form of brittle–tough transition, but has the ultimate aim of providing a more general explanation for all forms.

Figure 1 shows that the T_{BT} of modified 'third-generation', pipe-grade high density polyethylene (HDPE) pipe is also dependant on the method of cooling during extrusion. Normally the pipe is 'single-cooled' by spraying only the external surface with water. An alternative method cools both internal and external surfaces ('dual cooled'). Although initially more expensive to set up, dual cooling has two major advantages. Firstly, the residual stress profile through the pipe wall is symmetrical. This allows mitred joints to be made quickly and reliably, without first squaring up the pipe ends as in the case of single-cooled pipe. Secondly, the extrusion line length can be reduced by a factor of four for large-diameter pipe production. These economic advantages may be offset by an increased value of T_{BT} and hence an increased risk of RCP. Consequently, dual cooled, third generation HDPE pipe is produced solely for research and development purposes.

The ideal pipe material (in terms of RCP) is one which can be relied upon always to operate above its T_{BT}. The requirement of the pipe industry is a small-scale, easily-performed test which quantifies material properties relevant to the T_{BT}. Ideally, the material properties measured by this test could then be used to predict the T_{BT} in any pipe geometry. The test geometry should allow for specimens to be taken directly from a pipe (for quality control and investigation of processing conditions). The Inverted Charpy test introduced here is aimed at meeting these requirements.

SHEAR LIPS

Rapid crack propagation in pressurised polyethylene pipes is characterised by a brittle fracture surface (smooth, with no sign of ductility), and an axial crack velocity exceeding 100 m s^{-1}. However, at temperatures just below T_{BT}, shear lips less than 0.5 mm wide appear along the bore edge of the pipe fracture surface (*Figure 2*). Above T_{BT} shear lips grow rapidly prior to crack arrest. Scoring a 1 mm deep razor blade slit along the pipe bore eliminates both the shear lips and the transition[3].

The appearance of shear lips thus correlates with the T_{BT}, but what role do the shear lips play? The T_{BT} could result from a reduced crack velocity at higher temperatures, due to a lower material stiffness. If the dynamic fracture resistance rises rapidly below some critical crack velocity, then the crack will not propagate above the temperature associated with this critical velocity. If the shear lip formation process is suppressed at high rates, then it can be seen as a passive effect of the process controlling T_{BT} rather than the cause of it.

Leevers[4] has developed a 'thermal decohesion' model which gives accurate, quantitative predictions for the

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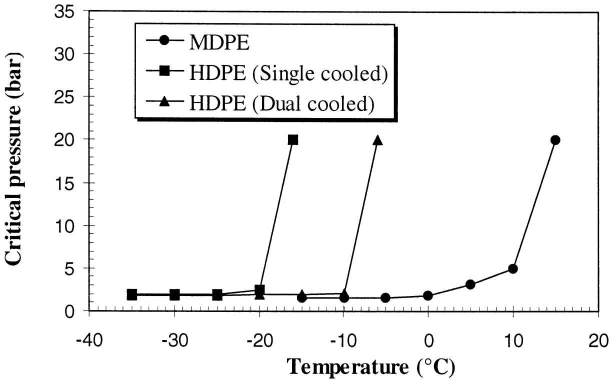

Figure 1 Critical pressure measured using a small scale, steady-state (S4) pipe test. (250 mm diameter, 22.7 mm wall thickness)

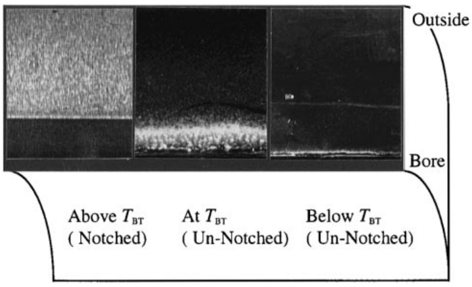

Figure 2 HDPE pipe fracture surfaces

dynamic fracture resistance (G_D) of semi-crystalline polymers. The model does not explicitly account for the formation of shear lips but does show a transition from high to low toughness as crack speed increases. For both MDPE and HDPE the transition speed is in the region of 25 m s^{-1}. However, the hypothesis that shear lips play a passive role is contradicted by the notching effect. The notch suppresses shear lip formation and eliminates the transition but does not alter the stiffness of the material.

Further investigation into the role of shear lips was made by Clutton *et al.*[5] and Morgan[6]. By applying an energy partitioning analysis to the instrumented Charpy test, they demonstrated that the post-initiation energy, calculated from the force-displacement history, increased with temperature. Clutton showed that this energy was related to the formation of shear lips and disappeared if the specimen was side-grooved. Defining a nil-ductility T_{BT}, at which the post-initiation energy extrapolated to zero, gave a qualitative ranking of pipe-grade materials which agreed with air-pressurised pipe test results. Quantitatively, however, the T_{BT} measured by the Charpy test is approximately 15°C above that seen in the S4 pipe test (90 mm diameter, 8.2 mm wall thickness).

THE HIGH SPEED DOUBLE TORSION TEST

The high speed double torsion (HSDT) test, developed by Leevers[7] and later Wheel[8,9], can be used to measure the dynamic fracture resistance (G_D) of a material, as a function of crack velocity. Recent developments have also allowed this test to be used to investigate the T_{BT} of polyethylene.

The 'V' grooved high speed double torsion test

Current high speed double torsion (HSDT) test methodology has been reported in detail by Ritchie[10]. The test specimen is a 100 by 200 mm plate, 10 mm thick. A 'V' groove on the under-side of the specimen along the axial centreline defines the crack path. The test drives a rapid crack along the specimen at a controlled speed which can be varied between 175 and 350 m s^{-1}.

The value of G_D for HDPE (3.6 kJ m^{-2}) is fairly constant with crack speed and agrees well with predictions from the thermal decohesion model. The results and predictions for MDPE are only slightly lower. Unfortunately, due to dynamic effects[10], it is not possible to obtain reliable results from the HSDT test at crack speeds where the thermal decohesion model predicts a transition to tough behaviour. The calculated values of G_D show little dependence on test temperature.

In summary, 'V' grooved HSDT specimens can be used to measure crack speed effects on G_D which agree with Leevers' thermal decohesion model, but do not show shear lips, or a T_{BT}.

The plane high speed double torsion test

Subtle changes to the HSDT method allow a straight crack to propagate *without* a 'V' groove. As expected, a shear lip appears on the lower side of a polyethylene fracture surface, decreasing in size with decreasing temperature and increasing crack velocity. In HDPE a crack could not be propagated at temperatures above −33°C, even at the maximum available loading rate. However, 2°C below this temperature crack propagation occurred, even at low loading rates. The results for G_D at 2°C below the T_{BT} are only slightly (1 kJ m^{-2}) higher than predicted thermal decohesion values. However, at 1°C below the transition temperature the composite G_D (brittle fracture plus shear lip energy) shows a sharp increase and the crack can only be propagated at approximately 150 m s^{-1}. Only three tests have been performed on an MDPE, but have located the T_{BT} at -2.5 ± 2°C. Scanning electron microscopy of sections through HDPE fracture surfaces revealed that the shear lips had separated at one of two $\pm 45°$ lines of high void density. The thermal decohesion model predicts that G_D decreases with increasing temperature. This prediction may seem counter-intuitive but is borne out by the HSDT results.

Since the characteristics of the T_{BT} seen in the plane HSDT test are very similar to those in the S4 pipe test, the test can be used to examine the fracture energies associated with the T_{BT} in a quantitative manner.

NEW TEST METHODS FOR TOUGH AND BRITTLE BEHAVIOUR

So far, direct correlations in T_{BT} between the plane HSDT and S4 pipe tests are encouraging. There are important similarities in the RCP mode between these two tests: Firstly, computational analysis of the S4 test[11] shows that the crack driving force in air-pressurised pipe is applied via a bending moment, as it is in the HSDT test, putting the pipe bore under additional tension near the crack front. In both tests the shear lip occurs along the edge associated with the surface under greatest tension. Secondly, both tests approach a steady-state condition such that below the T_{BT} a running crack leaves behind a smooth fracture surface, with a small shear lip of constant width.

In the S4 pipe, HSDT, and Charpy tests, the brittle–tough transition is seen as a balance between two mechanisms of failure: brittle fracture (as described by the thermal decohesion model) and shear lip formation. As the fracture

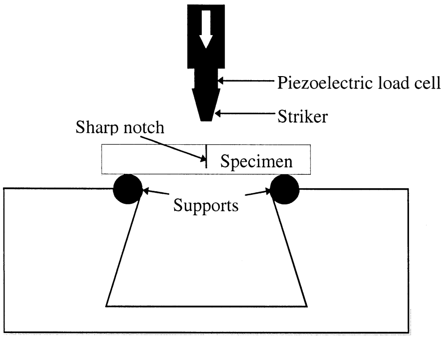

Figure 3 Inverted Charpy test rig

surface becomes dominated by shear lips the combined process enters the tough regime. From the results presented earlier at least two hypotheses could be formulated:

(1) As T_{BT} is approached from below, the shear lips absorb an increasing amount of crack driving force, eventually causing the crack to arrest. The large increase in energy seen at the T_{BT} is due to the shear lips alone.
(2) A combined process is occurring where the shear lips actively decelerate the crack into a falling portion of the G_D/crack-velocity characteristic so that G_D increases further; a dynamically unstable process leading to crack arrest[12]. If the shear lips are rate sensitive, the reduction in crack velocity would cause them to consume more of the crack driving force, further slowing down the crack. This positive feedback mechanism then promptly arrests the crack.

Since we can now quantitatively predict the brittle mode of failure using the thermal decohesion model, our next aim is to investigate the process of shear lip formation. The Inverted Charpy test of Ritchie[13] allows this process to be studied in isolation.

The instrumented Inverted Charpy test

To put this test into context it is worth noting the disadvantages of the standard Charpy test with respect to the investigation of shear lips.

(1) The process is not steady-state: the shear lip width increases to a maximum near the mid-specimen, and then decreases again.
(2) The rate and thickness at which the shear lips are formed is, in part, controlled by the growth of the internal brittle crack initiating from the notch.
(3) The test results cannot be used to predict quantitatively how the pipe T_{BT} will change with pipe wall thickness[14].

Because in the Charpy, pipe and HSDT tests the crack front trails at the side edges, the shear lip effectively develops between a free surface and a sharp notch. The Inverted Charpy test aims to reproduce this environment.

The loading geometry is similar to the standard Charpy test but the specimen is inverted so that the notch is on the 'wrong' side (*Figure 3*). A sharp notch is cut through 85%

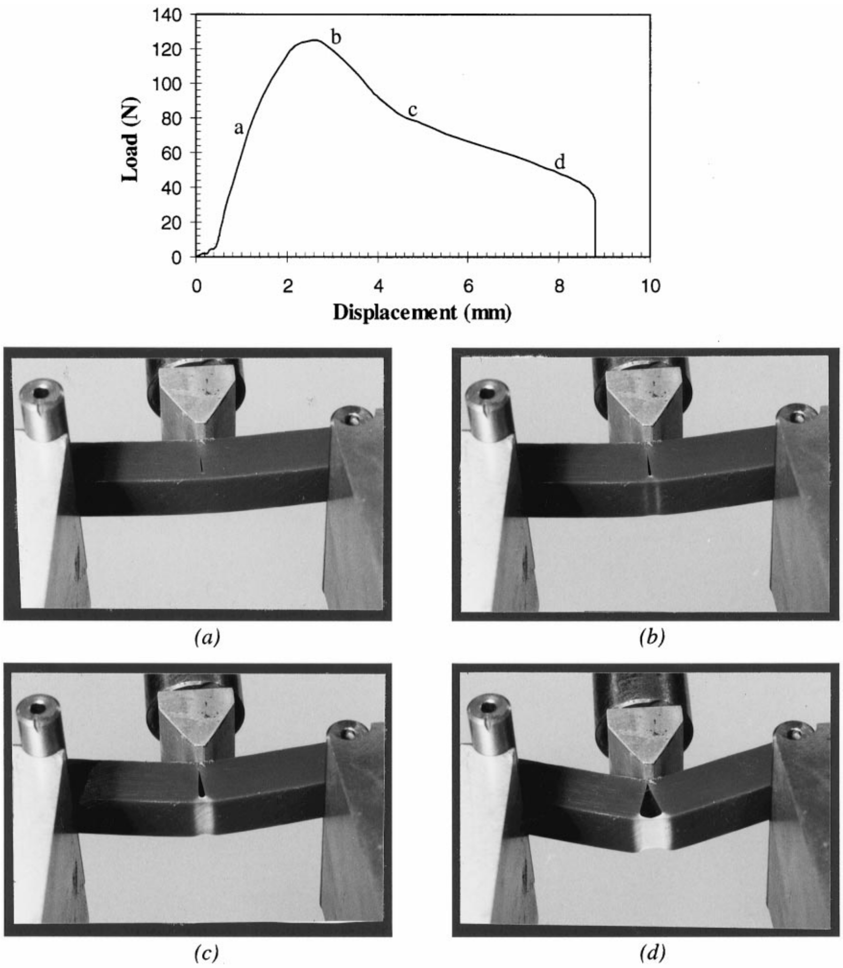

Figure 4 Load trace for a HDPE sample loaded at 5 mm/min, 23°C and pictures of the associated deformation taken from below

of the specimen width using a 0.25 mm-thick razor blade. This simulates a section where a brittle crack front has already passed. The shear lip width is controlled by the notch depth and its extension rate is governed by the impact speed.

Room-temperature tests were performed on an HDPE at slow rates to study the general deformation during the test. *Figure 4* shows the deformation in sequence, together with the recorded load trace. As the specimen is loaded, localised plastic deformation starts at the notch tip and develops by the growth of two symmetrical 45° shear bands through to the free surface. After reaching the free surface (peak load point) the shear bands propagate towards each other as draw fronts. The material between the two shear bands then necks down until failure.

The results can be simply analysed as follows. Firstly the nominal tensile stress on the ligament at peak load is calculated assuming:

(1) At peak load the presence of shear bands from notch tip to free surface implies a uniform tensile stress on the ligament, equal to the uni-axial yield stress (σ_y).
(2) The neutral axis of bending lies along the centre of the specimen.
(3) There is no horizontal component to the reaction force at either the load or support points.
(4) Inertial forces are negligible.

Taking moments about the support point for static equilibrium of the half specimen gives:

$$\sigma_y = \frac{PL}{4Bs(W-s)}$$

where P is the load, L the span, B and W the specimen thickness and width, respectively, and s the remaining ligament width after notching. Although the above assumptions are obviously invalid prior to shear band growth, this equation is used to normalise the full load displacement curve.

The post-yield energy per unit ligament area was calculated as:

$$\frac{1}{sB}\int_{v_p}^{v_0} P\mathrm{d}v$$

where v_p is the load-point displacement at peak load and v_0 is that when the load first returns to zero after v_p.

Inverted Charpy tests were performed on MDPE and HDPE samples machined from compression moulded sheet ($B = W = 10$ mm and $L = 55$ mm). The surface cooling rate was slow but unknown, so the specimens were notched such that the ligament extended through the sheet thickness. The tests were performed over a range of temperatures using a high-rate Instron tensile testing machine. Low test temperatures were achieved using an environment chamber cooled by liquid nitrogen.

Inverted Charpy results

Typical load traces are shown in *Figure 5*, and processed results in *Figures 6 and 7*. The peak nominal stress is only slightly higher than the 10% flow stress[15] and differs little between the two materials, but large differences in the post-peak energy are evident.

Examination of the specimen failure surfaces reveals the mechanism involved. *Figure 8* schematically shows the deformation just prior to failure of an HDPE sample at −30°C. After the shear bands have grown through to the free surface, drawing becomes localised at one band, probably because adiabatic heating causes softening[16]. At the same time tearing proceeds downward from the base of the notch, leaving characteristic upward-pointing chevron markings on the surface. The remaining ligament region continues to neck and at some critical point the specimen fails along a contour within the large voided area. The male half of the specimen, with the protruding shear lip (M) shows two distinct areas: one of smooth, whitened appearance (D) corresponding to the torn and drawn material around the notch, and a voided area (V) corresponding to failure of the necked ligament. The area D decreases with decreasing temperature until it almost disappears. The voided area shows a decrease in coarseness until, at the lowest temperatures, the failure surface appears brittle with no evidence of whitening or ductility. The female half of the specimen (F) shows similar

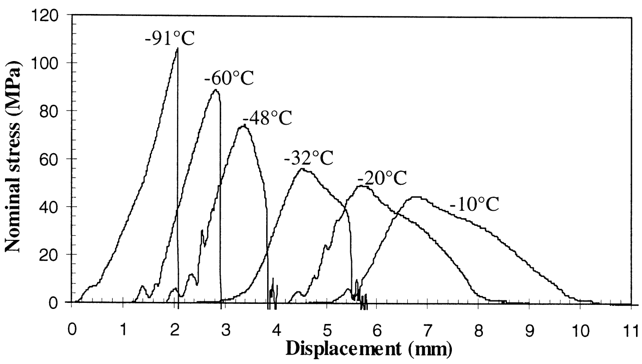

Figure 5 Inverted Charpy load traces for HDPE (displacement rate = 1.7 m s^{-1}).

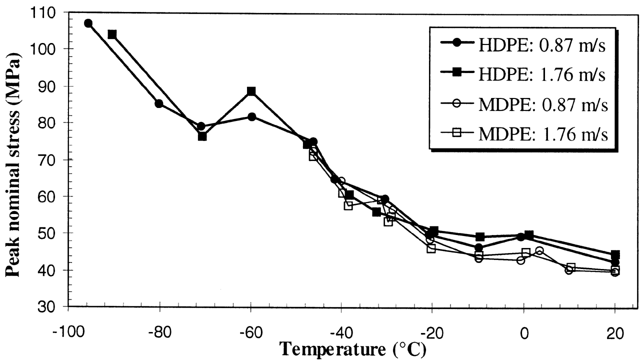

Figure 6 Peak nominal stress from the Inverted Charpy test

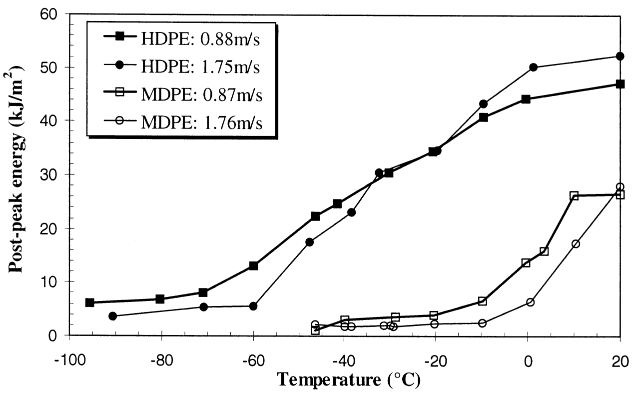

Figure 7 Post-peak energy from the Inverted Charpy test

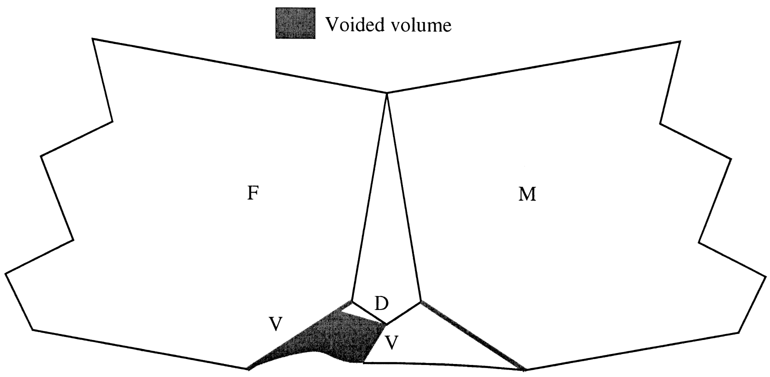

Figure 8 Schematic of the Inverted Charpy deformation mechanism in HDPE. (In reality the voided volume is less then 0.2 mm wide.)

matching features with a thin lip of material protruding from along the base of the notch. This disappears at the lower temperatures.

Both MDPE and HDPE were tested at two rates (*Figures 6 and 7*). As expected, the yield stress hardly changes since the rates differ only by a factor of two. However, there is a definite increase in post-peak energy for the lower rate at low temperatures. The effect of rate on HDPE at −40°C was examined more closely (*Figure 9*). At a displacement rate of $3.8\ \mathrm{m\,s^{-1}}$ the strain rate (50–$75\ \mathrm{s^{-1}}$) is approximately equivalent to that in the high speed double torsion test. At this rate the post-peak energy has virtually dropped to the lowershelf value of the lower rate, lower temperature tests.

Effect of processing conditions on modified high density polyethylene

To investigate the effect of cooling rate on shear lip formation (*Figure 1*), Inverted Charpy tests were performed using HDPE plaques manufactured with two different thermal histories: slowly cooled ($0.5^{\circ}\mathrm{C\,min^{-1}}$), and rapidly cooled ($2^{\circ}\mathrm{C\,s^{-1}}$).

Figure 10 shows the variation in yield stress with temperature, as calculated from the Inverted Charpy results. As expected, the yield stress increases with decreasing temperature but surprisingly, the change appears to be almost the same for both cooling rates. However, *Figure 11* shows clear differences in the post-yield energies, slowly cooled samples requiring more energy to break. This reflects the fracture behaviour of pressurised HDPE pipe: T_{BT} is decreased by single-surface (as opposed to dual-surface) cooling.

The role of tensile drawing in shear lip formation

Over a range of temperatures, normalised load–elongation curves from the Inverted Charpy test are similar to those obtained from un-notched tensile tests. Both show a similar yield stress and post-peak energy absorption, but the nominal failure strain in the Inverted Charpy test is restricted by the deep notch.

This difference was investigated further using simple tensile tests on the same thermal-history controlled plaques. Specimens were machined to the same thickness as the ligament in the Inverted Charpy test, and from the same location, ensuring the same thermal history. A high-rate Instron tensile testing machine was used to apply the same nominal strain rate as that at the underside of the Inverted Charpy specimens (approximately $35\ \mathrm{s^{-1}}$).

Figure 10 shows the variation in yield stress with temperature for these tensile draw tests. As in the Inverted

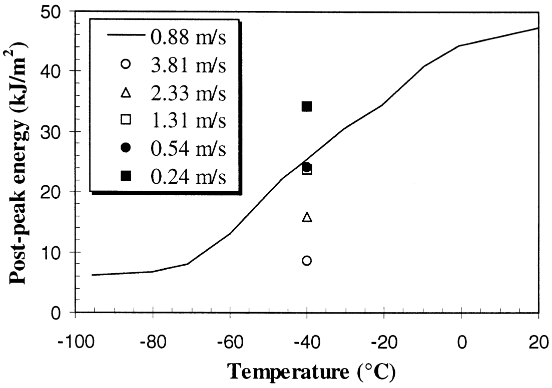

Figure 9 Inverted Charpy rate effect for HDPE

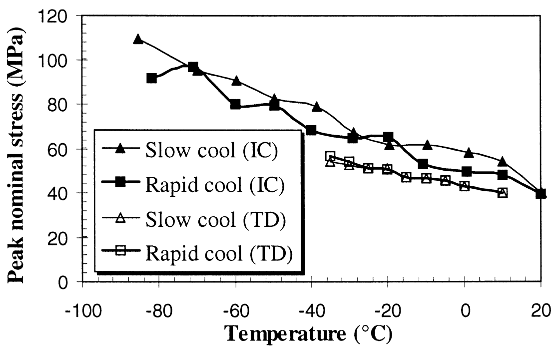

Figure 10 Inverted Charpy (IC) and tension draw (TD) peak nominal stresses in HDPE processed at different cooling rates. (Approximate nominal strain rate $= 35\ \mathrm{s^{-1}}$)

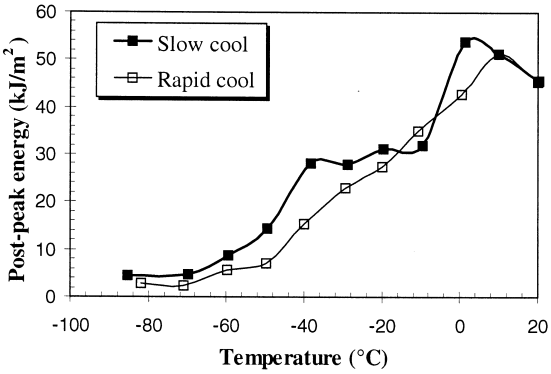

Figure 11 Inverted Charpy post-peak energy results for HDPE processed at different cooling rates. (Displacement rate $= 1.7\ \mathrm{m\,s^{-1}}$)

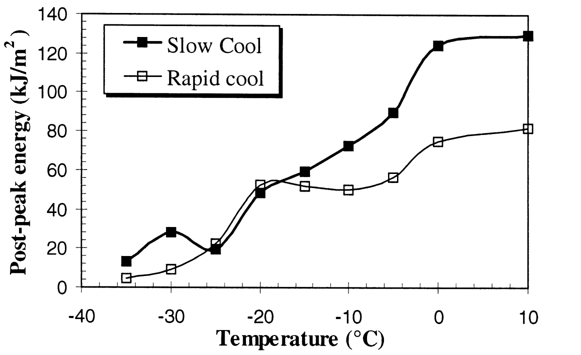

Figure 12 Comparative load traces between the Inverted Charpy and tensile tests (HDPE, 0°C)

Charpy test, there is no significant effect of cooling rate, and the values from both methods are in good agreement. The post-yield energy (*Figure 12*) follows the same trend with temperature as in the Inverted Charpy results, slower cooling again giving a distinctly higher energy absorption.

DISCUSSION

Test methods for tough and brittle behaviour

The Inverted Charpy test successfully simulates the process of shear lip formation in the pipe without the additional complications of a brittle fast running crack. The present analysis is simplistic but its results clearly show the rate and temperature dependence of shear lip formation. At any given temperature, MDPE and HDPE show differences in necking and drawing behaviour which introduce a different T_{BT} for rapid crack propagation. Both materials show similar lower bound values of post-yield energy. For HDPE at least, increasing the rate shifts T_{BT} upwards. The sharply defined transition seen in polyethylene pipe and the plane HSDT test is not seen in the process of shear lip formation. The positive feedback process of the second hypothesis, for the mechanism by which shear lips play a part in the transition, therefore seems reasonable.

However, it is possible that at rates appropriate to rapid crack propagation in the HSDT and S4 pipe tests, the transition between the lower and upper bound post-peak Inverted Charpy energies becomes sharper. This is hinted at by *Figure 7,* but comparisons are not straightforward since the notch tip tearing mode seen in these tests is absent on pipe and HSDT fracture surfaces. A high rate Inverted Charpy transition as sharp as that seen in the pipe test would weaken the argument for any amplifying mechanism.

The Inverted Charpy method generates homogenous drawing between a sharp notch and a free surface. The measured maximum stress remains approximately equal to the yield stress even when the failure surface has a brittle appearance and a shear lip is not observed. The thermal decohesion model attributes brittle fracture to a colinear-craze formation and separation process propagated from the notch tip. Presumably this process loses its viability with the loss of constraint as a free surface is approached.

Comparison of Inverted Charpy and tensile draw test results is encouraging for two reasons. Firstly, they corroborate evidence of a necking and cold-drawing mechanism occurring in the Inverted Charpy test. With this established, we have a foundation to begin modelling the deformation behaviour. Secondly, both of these tests show a sensitivity to cooling rate similar to that of extruded HDPE pipe.

MICROSTRUCTURE AND THE BRITTLE–TOUGH TRANSITION

The effect of microstructure on the un-notched tensile behaviour of polyethylene is well documented for a range of molecular weights[17]. The ductile–brittle transition temperature is reduced by quenching and has been discussed in terms of the higher density of tie molecules linking individual lamellae in the crystalline fraction.

The effect of thermal history seen here appears to contradict this description. However, modified ‘third-generation’ HDPE is different to previous polyethylene grades in many aspects, of which few are non-proprietary. During the polymerisation process most of the comonomer content is assigned to the high molecular weight chains. Therefore, when the polymer is cooled from the melt, short (relatively branch-free) chains form the crystalline fraction, and longer (highly branched chains) are segregated to the amorphous phase. The length of these chains implies a high density of tie molecules whatever the cooling path taken from the melt temperature. Without testing at extremely low temperatures[17], the effect of cooling rate cannot be discussed in the same context. Also, the T_{BT} apparent in the S4, Inverted Charpy, and tensile tests is controlled by post-yield behaviour, making detailed comparisons with previous results difficult.

Both the tensile and Inverted Charpy tests were performed at temperatures above the γ relaxation range, so the yield stress should normally reflect crystallinity[17]. However, taking a value of $290\,J\,g^{-1}$ for a perfect polyethylene crystal, differential scanning calorimetry (using a Perkin–Elmer Pyris machine) gave values of 69% crystallinity for the slowly cooled sample, and 61% for the rapidly cooled. Therefore, a difference of 8% in the overall sample crystallinity does not appear to change the yield stress of this material (*Figures 10, and 12*).

The HDPE morphology was assessed using scanning electron microscopy. A well-documented surface treatment procedure was used to remove the amorphous fraction[18,19]. Although precise measurements of lamellae dimensions could not be made from these pictures, a qualitative difference in crystalline texture was evident. Slowly cooled material appears to be constructed from a smaller number of large crystalline blocks.

The extent of stable drawing in a tensile test is determined principally by the strain at which strain hardening produces a second Considére tangent on the true-stress/strain curve[20]. If no second tangent exists, failure will occur at the initial neck. In polyethylene, strain hardening occurs by the transformation of the original lamellae into a fibrillar structure[21], which increases the stiffness of the necked element in tension. The process involves the partial fragmentation of original lamellae and their re-arrangement into a new, aligned structure, held together by the original tie molecules. Since the majority of specimen elongation occurs during this process, a difference in crystalline texture would be expected to influence the extent of post yield deformation. A larger initial lamella size (with respect to tie molecule density), would require more lamella fragmentation events for sufficient strain hardening to occur. However, translating this behaviour to a higher post yield energy absorption for a slow cooled morphology is speculative. The behaviour of HDPE samples with different thermal histories seems to be reflected in the propagation of a stable neck: the 'better' materials cold-draw to higher strains.

During cold-drawing plastic work is dissipated as heat. The high strain rates in the current tests will lead to a localised temperature rise in the material just ahead of a stable neck[22] and reduce the yield stress of undrawn material. This effect of adiabatic thermal softening superimposed on a morphology-controlled strain hardening process is currently under study. If softening were sufficient only to delay hardening, it could increase the draw ratio without significantly reducing the draw stress. A greater degree of softening, however, could precipitate early collapse at a low draw ratio. In order to decouple adiabatic thermal effects from isothermal true-stress/true-strain curves, work is proceeding on the basis of a 'semi-inverse' simulation method.

CONCLUSIONS

Rapid crack propagation in pressurised polyethylene pipes displays a sharp brittle–tough transition temperature (T_{BT}) above which it is impossible to propagate a crack, even at the highest available test pressure. This T_{BT} is controlled by shear lips. The most important property of a polyethylene, with respect to these shear lips, is its post-yield energy absorption, not its yield stress. The post-yield energy decreases with rate and increases with temperature, but only within a relatively narrow window of either variable. Increasing the cooling rate during processing inhibits the drawing capability of modified HDPE, and hence increases T_{BT}. Preliminary work indicates this to be correlated with a decrease in lamella size. Understanding the mechanisms which lead to a higher post-yield energy absorption will hopefully allow the T_{BT} of extruded pipe to be better controlled in the future.

The plane high speed double torsion test shows a T_{BT} which has very similar characteristics to that seen in pressurised pipe. The test can be used in a simple, non-instrumented, go/no-go form to locate the T_{BT}. However, this test is not suitable for evaluating the geometry dependence of the T_{BT} or investigating the microstructural features involved in the process of shear lip formation.

The Inverted Charpy test is a suitable tool for examining the shear lip formation process and may well prove to be a suitable test for predicting T_{BT} for pressurised polyethylene pipe.

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