

Fracture Toughness and Creep Fracture Studies of Polyethylenes

by

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Abstract

The fracture toughness of two grades of polyethylene were characterised using the Linear Elastic Fracture Mechanics (LEFM) and the Elastic-Plastic Fracture Mechanics (EPFM) approaches. LEFM was successful in measuring the fracture toughness (K_{IC} or G_{IC}) of one of the grades over a range of temperatures, but the extreme toughness of the other grade invalidated the application of this method. K_{IC} and G_{IC} have been shown to be compatible if the requirements for plane strain conditions and the linearity criterion for the load-displacement curve are satisfied. Given the limitation on specimen thickness, EPFM was adopted to study the fracture behaviour of the materials. An attempt was made to determine J_{IC} values using the single specimen method which has rarely been applied to polymers. The results agree well with those obtained from the multiple specimen method, which is often used for polymers and has proved to be successful. Other polymers were also tested using the single specimen method in order to verify the validity of the technique. The effects of specimen size, loading rate and side grooving have been studied for one of the polyethylenes. In general a constant J_{IC} value is achieved. The advantages and drawbacks of the old and new ASTM E813 standard method are discussed and the blunting line suggested in the standard is found to be inappropriate for describing the blunting mechanisms of polymers.

Although fracture toughness studies provide valuable information about materials, like most conventional material testing, it generates data which are not strictly relevant for use in predicting service life-time of plastics. LEFM is a useful tool in predicting the time of fracture initiation and the slow crack propagation behaviour under creep loading. Crack length was measured indirectly from the change of compliance of the notched sample and the results are found to be more satisfactory than monitoring the crack length by a travelling microscope. It is noted that the initiation time is controlled not only by the applied K level, but also depends on the

net section stress in the ligament. The J -integral is shown to be more appropriate in correlating the initiation time with the loading conditions. The slow crack propagation phenomenon is generally represented in the form of $K-\dot{a}$ curve and it is found that the shape of the curve depends on the proportion of the size of the shear lips to the specimen thickness. It is demonstrated that the parameter C^* provides a better means of characterising slow crack growth behaviour in polyethylenes, since the $C^*-\dot{a}$ curves for the polyethylenes are independent of specimen size, side-grooving and initial loading conditions.

To my parents

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Nomenclature

Most of the symbols used in the text is listed below. Some of the symbols may bear more than one meaning in order to keep their common recognition and the Sections where these symbols appear are given in the list. The definition and usage will also be given in the text.

A	Function of a/W [2.7.3] / Area under the load displacement curve[3.4.2] / Pore area in the craze[4.2.2].
a	Crack length.
$\dot{a}$	Crack propagation speed.
B	Specimen thickness.
b	Uncracked ligament length.
B_e	Effective thickness of side grooved specimen.
B_N	Net thickness of side grooved specimen.
C	Compliance.
c	Length of the line zone.
C^*	Dissipative work rate per unit crack growth area.
C_{cm}	Crack mouth opening compliance.
C_{cr}	Load line compliance due to the presence of the crack.
C_i	Initial compliance.
C_o	Load line compliance of uncracked beam.
D	Constants.
δA	Crack growth area.
Δa	Length of crack extension.
Δa_b	Crack growth due to blunting.
E	Elastic modulus.
E'	Effective modulus.
E^*	Effective time dependent modulus.
E_o	Value of modulus at unit time.
F	External work done.
f	Deflection of the beam.
g	Depth of side-groove.
G	Energy release rate per unit crack area.
G_{IC}	Value of G at mode I fracture under plane strain condition.
h	Surface heat transfer coefficient.
J	J -integral
J_e	Elastic component of J .

J_{IC}	Value of J at mode I fracture under plane strain condition.
J_p	Plastic component of J .
K	Stress intensity factor.
k	Constant[4.2.2] / Thermal conductivity[4.5].
K_{app}	Apparent K_c value.
K_c	Subcritical fracture toughness.
K_{c1}	Plane strain component of subcritical fracture toughness.
K_{c2}	Plane stress component of subcritical fracture toughness.
K_i	Initial applied K_c .
K_{IC}	Value of K at mode I fracture under plane strain condition.
K_{max}	K_Q calculated from maximum load.
K_Q	Measured K_{IC} value.
K_s	Actual K_c value.
$\tilde{K}_s$	Average actual K_c value.
L	Span of supports.
l	Characteristic length[4.2.2] / Half of the specimen thickness[4.5].
M	Plastic constraint factor.
m	Power law index.
n	Power law index / Outward bound vector[3.3].
Nu	Nusselt number.
P	Load, pressure.
$P_{5\%}$	5% offset load.
P_a	Atmospheric pressure.
P_{max}	Maximum load.
Q	Constant.
R	Fracture resistance.
r	Distance from the crack tip.
r_p	Plastic zone size.
r_s	Size of the shear lips.
S	Loading span.
s	Arc length along the line integral path.
T	Traction vector.
t	Time.
T_c	Final cooling temperature.
t_{dev}	Craze development time.
t_f	Failure time.

t_i	Initiation time.
T_m	Softening temperature of polymers.
t_p	Propagation time.
U	Internal stored energy.
u	Displacement vector.
$\dot{U}_{dis}$	Rate of energy dissipated.
U_c	Work done for loading a cracked body.
U_{co}	Complimentary energy.
U_{dis}	Dissipated energy.
U_o	Energy for deforming the uncracked beam.
U_p	Potential energy.
U_r	Released strain energy.
U_s	Potential energy of the surface of the crack.
U_T	Total energy input.
v	Load line displacement.
W	Specimen width.
x	Distance.
$\dot{X}$	Cross head speed.
Y	Geometry factor.
μ	Viscosity.

List of Greek Symbols

α	Constants[3.3, 3.4.1] / Crack length to width ratio[2.7.3, 4.7.2].
δ	Crack opening displacement.
δ_c	Value of δ at fracture.
δ_{cm}	Transfer function.
δ_{cr}	Transfer function.
$\dot{\epsilon}_n$	Nominal strain rate.
$\dot{\epsilon}$	Strain rate.
ϵ_{nom}	Nominal strain.
ϵ_{ij}	Strain tensors.
ϕ	Calibration factor.
Φ	Curvature of the beam.
Γ	Contour round the crack tip.
γ_e	True surface energy.

γ_p	Plastic strain work.
η	Geometry dependent constant.
θ	Angle measured from the crack plane.
σ	Stress.
σ_a	Applied stress.
σ_a	Residual stress.
σ_c	Stress at the line zone.
σ_f	Failure stress.
σ_{ij}	Stress tensors.
σ_N	Net section stress.
σ_o	Time dependent yield stress at unit time.
σ_p	Plastic collapse load.
σ_s	Actual applied stress.
σ_y	Yield stress.
ν	Possion's ratio.
Ω	Strain energy.
μ	Viscosity.

Abbreviations

ASTM	American Society for Testing and Materials.
EGF	European Group on Fracture.
EPFM	Elastic plastic fracture mechanics.
ESC	Environmental stress cracking.
HDPE	High density polyethylene.
HRR	Hutchinson, Rice and Rosengren.
LEFM	Linear elastic fracture mechanics.
LVDT	Linear variable displacement transducer.
MDPE	Medium density polyethylene.
MFI	Melt flow index.
PMMA	Poly(methyl methacrylate).
PS	Polystyrene.
PVC	Poly(vinyl chloride).
PVDF	Polyvinylidene difluoride.
SEN	Single edge notched.

Introduction and Overview

A. A. Griffith[1920] was the first to study the quantitative connection between strength and cracks in materials. He proposed that all materials contained flaws and the strength could be characterised as the energy per unit area required to produce new surfaces. In the next twenty years, problems like Allied fighters falling from the sky without being shot by the Germans, and the explosion of liquidified natural gas storage tanks showed that something had been overlooked during the design process. Not until after the Second World War was the subject of fracture mechanics raised again by Irwin[1948]. His work on linear elastic fracture mechanics laid the foundation of the subject and he proposed the fracture parameters G , the *energy release rate*, and K , the *stress intensity factor*. Further advances were made when Wells[1961] suggested the crack opening displacement approach and Rice[1968] presented his J -integral. These approaches are now regarded as elastic-plastic fracture mechanics. In fact, all the approaches share the same philosophy: fracture will occur when the characterising parameter reaches a critical value which is often referred to as the fracture toughness of the material. The only differences are the scale of plasticity and nonlinearity being considered in the analysis. However, all of them will converge and can be equated to each other in the linear elastic regime. Fracture mechanics is now accepted as the best tool for studying fracture problems.

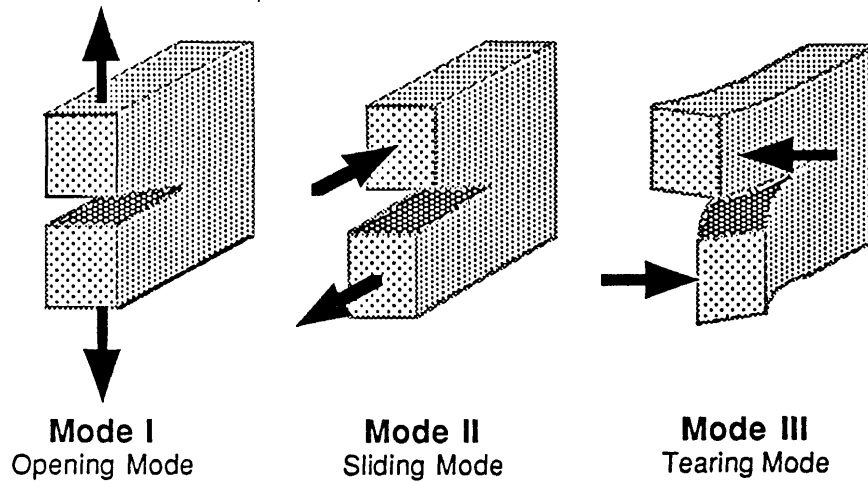


Fig.1.1 The three modes of loading

It is also a major consideration in designing large structures and for integrity assessment.

Three basic modes of loading can be considered, they are crack opening, sliding and tearing modes as illustrated in Fig.1.1. Mode I loading draws most of the concern since this mode is found in many practical situations. Fracture toughness measured under plane strain conditions is considered to be the most important. The fracture toughness at a particular temperature depends on the degree of constraint at the crack tip, and the constraint is maximum under plane strain conditions. The fracture toughness value in plane strain will be minimum and is called the plane strain fracture toughness. It can be considered as a material property for characterising the fracture resistance. International and national standard test methods on the determination of plane strain fracture toughness have been established by different working parties. These tests are designed to provide information on the performance of the material in practice. Although most of the early work was done on metals, the application of fracture mechanics has been extended to non-metallic materials in recent years.

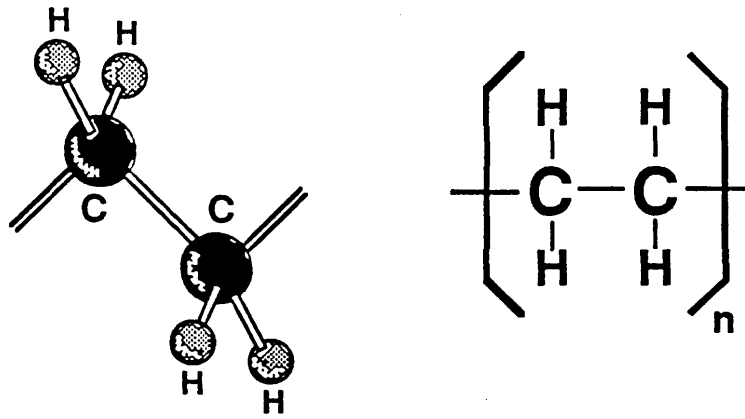


Fig.1.2 The structure of the polyethylene molecule
(n =number of repeating units)

Thermoplastics have been finding increased use as structural materials in the past two decades. The ease of manufacturing, superb resistance to corrosion and high strength to weight ratio make plastics excellent substitutes for traditional materials. One of the most widely used thermoplastics is polyethylene. Polyethylene was first produced by ICI in the 1940's and its generic structure is shown in Fig.1.2. Polyethylene is a semi-crystalline polymer and consists of crystalline and amorphous regions. The molecules are arranged in three dimensional order in the crystalline regions and are randomly distributed in the amorphous regions. The crystals take the form of thin platelets, or lamallae, with layers of amorphous material between them. The lamallae grow in all directions from different nuclei and develop through sheaf-like entities into spherulites. The microstructure of polyethylene is described in detail by Young[1983]. The mechanical properties of the amorphous regions are largely dependent on the degree of entanglement of the molecules. The material is sometimes viewed as a two-phase material and the properties can be estimated using the *rule of mixtures* which is commonly used to evaluate the properties of fibre-composite materials. However, the actual structure and deformation of semi-crystalline polymers are more complex and are not yet fully understood.

Polyethylene is a very versatile material, the properties can be changed by varying the density, degree of crystallinity, molecular weight or introducing side groups. It exhibits high ductility at failure in most cases. The applications of the material are extensive, including packaging film, electrical insulation, bottles, water and gas pipings. High density polyethylene pipes are widely used in gas distribution systems nowadays. The bursting of a polyethylene bag full of supermarket shopping in a high street may be embarrassing, but the bursting of a gas pipe in the same place can be lethal. Since inspection and maintenance of the pipes will be difficult after burial, it is important to estimate the service life of the pipes so that replacements can be installed before any catastrophic accidents can happen.

The standard fracture tests have been demonstrated to be successful in characterising the fracture toughness of polyethylenes (Chan[1982], Hashemi et al.[1984, 1986 i, ii, iii]). Ductile failure is always encountered in these tests. However, gas pipes always fail in a brittle manner at an operating stress well below the failure stress measured from the short term tests. This is due to the viscoelastic nature of polymers, and that the material properties are time dependent. The time scales involved in the short term tests are different from those in long term failure. The service life time is conventionally estimated from hydrostatic stress rupture tests. The failure times of pipes under different constant pressures are recorded. Results acquired from these tests provide little information about the initiation and crack growth mechanisms. Application of fracture mechanics to the time dependent fracture of polymers was first postulated by Williams et al.[1975]. This approach was later shown to be adequate by many workers. The prediction of initiation time, failure time and crack growth kinetics can all be embodied in the analysis. The fracture mechanics test method has the potential to be a quick method of quality control to characterise the long term performance of polyethylene, and the material costs for testing are also considerably reduced. Certain environments have

detrimental effects on the crack resistance of polyethylenes, such as detergent which is widely known as one of the strong environmental stress cracking agents. The study of environmental stress cracking becomes important since detergents are often used in the field for soap-bubble testing for checking leakage of joints and as lubricants for cable laying.

Although the results from the standard fracture tests are not directly applicable to long term failure prediction, they provide a means of ranking materials in term of their plane strain fracture toughness. They can also be used to estimate the critical flaw size of materials. The initiatives of this research project are to study the fracture resistance of two polyethylenes under short term and long term loading conditions and to evaluate and modify some of the existing testing techniques for such tests. In Chapter 2, the mechanical properties of the polyethylenes over a range of temperatures and strain rates are reported. The plane strain fracture toughness of the polyethylenes under these loading conditions have been characterised using the linear elastic fracture mechanics approach. Chapter 3 describes the application of the J -integral in measuring the plane strain fracture toughness and fracture resistance of the materials. The feasibility of applying the unloading compliance method to polymers has been discussed and the results are presented in parallel with those obtained from the the multiple specimen method. The effects of specimen size and side-grooving on the determination of the fracture resistance have been included and the merits of utilizing the standard test methods for testing polymers are discussed.

Chapter 4 deals with creep fracture of the materials. Most of the studies on creep fracture of polyethylenes in the past focused on crack propagation, and limited research was done on investigating crack incubation. The significance of the incubation time will be discussed here. It includes the use of fracture mechanics concepts in evaluating the initiation time, failure time and describing crack growth

kinetics. The results obtained from specimens of various sizes and side-grooved specimens are compared. Some results of the environmental effects on the crack growth resistance of one of the polyethylenes are also presented. In addition, fractographic studies on the fracture surfaces are given in this chapter.

Plane Strain Fracture Toughness Study : Linear Elastic Fracture Mechanics Approach

2.1 Introduction

In this chapter, the principles of Linear Elastic Fracture Mechanics, LEFM, and the application of this approach to characterise the fracture toughness of polyethylenes will be discussed. It is not intended to present a complete review on the subject here. Texts and reviews devoted to this subject can be found in the literature (Broek[1987], Ewalds et al.[1985], Kannien et al.[1985] and Williams[1978, 1984]). LEFM is the most well developed fracture theory. It has been developed within the bounds of the theory of elasticity and is applied to materials which obey Hooke's law. However, the theory can be applied to materials which exhibit plastic deformations provided that the energy absorption is localised around the crack tip and the body as a whole behaves elastically. In the LEFM regime, there are two independent but closely related approaches, the *Energy Balance Approach* and the *Stress Intensity Factor Approach*. The former uses the parameter G , known as the *Elastic Energy Release Rate*, which is a measure of energy required to propagate a crack over a unit area. The latter uses the *Stress Intensity Factor* K , which can uniquely define the stress field around a crack tip in a linear elastic body. Both parameters are material independent but fracture will occur when these parameters exceed certain critical values, K_{IC} and G_{IC} , accordingly,

where the subscript I denotes mode I loading. K_{IC} and G_{IC} are true material properties independent of loading conditions and are often regarded as the fracture toughness of the material. LEFM provides a useful tool in describing various types of fracture phenomena, but all the work presented in this chapter will be confined to the determination of the plane strain fracture toughness. The application of LEFM to polymers may seem dubious since polymers are neither linear nor elastic, the modification and use of the theory to polymers will be discussed in this chapter.

2.2 The Energy Balance Approach

2.2.1 The Griffith Approach

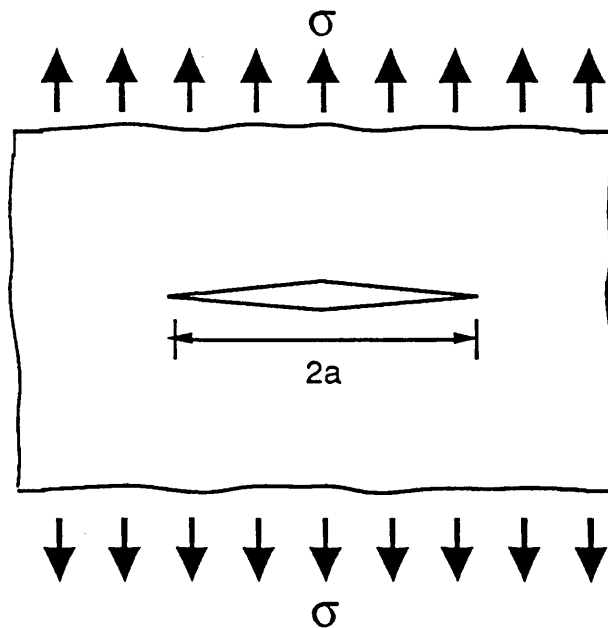


Fig.2.1 An infinite cracked plate of unit thickness

The energy balance approach is based on the hypothesis proposed by Griffith[1920]. He suggested that every body contains flaws. For a crack of length $2a$ in an infinite body with unit thickness subjected to a remote tensile loading, as illustrated in Fig.2.1, he stated that fracture would occur when the energy required for crack

growth was the same as the energy release upon crack propagation. This can be expressed as:

$$\frac{d}{da} (U_r - U_s) = 0 \quad (2.1)$$

where U_r is the strain energy released due to crack extension and U_s is the potential energy of the surface of the crack. By using the stress analysis developed by Inglis[1913], he further developed a relationship between the failure stress σ_f , the crack size a and the true surface energy γ_e :

$$\sigma_f = \sqrt{\frac{2E\gamma_e}{\pi a}} \quad (2.2)$$

where E is the elastic modulus. Modifications to the theory were made by Irwin[1948] to include the plastic strain work γ_p . By rearranging Equation(2.2), he defined that the energy release rate at fracture initiation, G_c , was given as:

$$G_c = \frac{\pi \sigma_f^2 a}{E} = 2(\gamma_e + \gamma_p) = R \quad (2.3)$$

He proposed that fracture would only occur when the energy release rate G was at least equal to the fracture resistance R . However, the analysis was still restricted to materials which showed limited amount of plasticity and have an ideally sharp crack.

2.2.2 The Derivation of Energy Release Rate G

The energy change in a system due to crack growth of an area δA can be considered as the changes in external work F and in the internal stored energy U . Thus G can be defined as:

$$G = \frac{d}{dA} (F - U) \quad (2.4)$$

Consider a cracked linear elastic body under a load P with the corresponding load-line displacement v as depicted in Fig.2.2. When the crack extends by da , G can be written as:

$$G = \frac{d}{Bda} (Pv - U) \quad (2.5)$$

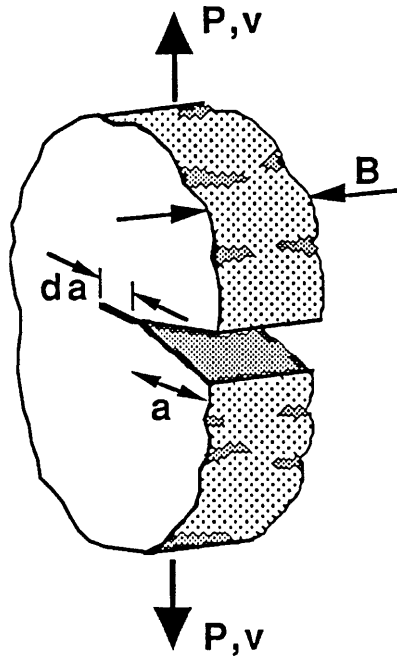


Fig.2.2 Loading a general cracked body

For an elastic body,

$$v = CP \quad (2.6)$$

where C denotes the compliance. The stored elastic energy is given as:

$$U = \frac{1}{2} Pv = \frac{1}{2} CP^2 \quad (2.7)$$

Substituting from Equation (2.7), Equation (2.5) can be expressed in the form of crack growth under constant load or constant displacement conditions,

$$G = \frac{-1}{B} \left[\frac{\partial U}{\partial a} \right]_v = \frac{1}{B} \left[\frac{\partial U}{\partial a} \right]_P \quad (2.8)$$

Combining Equations(2.5), (2.6) and (2.7),

$$G = \frac{1}{B} \left(P^2 \frac{dC}{da} + CP \frac{dP}{da} - \frac{1}{2} P^2 \frac{dC}{da} - CP \frac{dP}{da} \right) = \frac{P^2}{2B} \frac{dC}{da} \quad (2.9)$$

It can be seen from Equations(2.8) and (2.9) that G is independent of the loading conditions for linear elastic materials. This can also be illustrated graphically. The load deflection diagram for the cracked body shown in Fig.2.2 is illustrated in Fig.2.3.

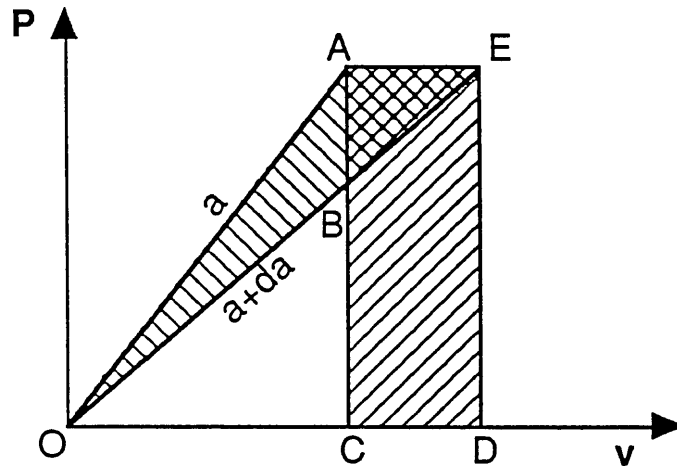


Fig.2.3 Load displacement diagram for a cracked body under loading

For crack extension under constant displacement, the energy release is represented by the area OAB. If crack extension occurs at constant load, the increase in the internal energy of the system is given by the area OAE. Since the area ABE is small and can be neglected for infinitesimal crack growth, this again indicates that the energy available for crack extension is the same for both types of loading. Therefore G_c of any system can be evaluated using equation (2.9) if C as a function of a is known.

2.3 The Stress Intensity Factor Approach

2.3.1 Stresses Around a Crack Tip

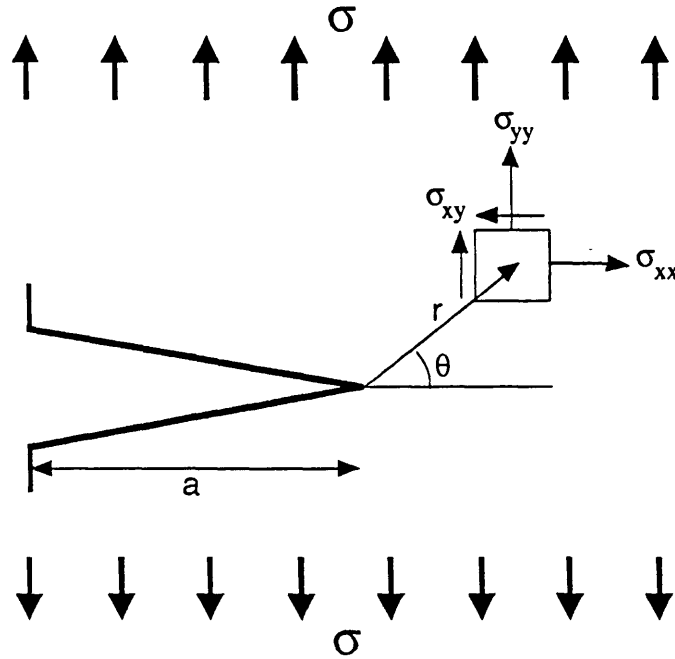


Fig.2.4 Stresses in the vicinity of a crack tip

The single parameter G_c as a fracture criteria provides a means to characterise the fracture toughness of materials but the determination of C is not always achievable. An alternative approach was proposed by Irwin[1957] and is known as the stress intensity factor approach. By using the stress function solutions developed by Westagaard[1939], Irwin considered the stresses in the vicinity of a crack tip in an elastic body subjected to mode I loading, as shown in Fig.2.4. The elastic stress field near the crack tip can be expressed as:

$$\sigma_{ij} = \frac{K_I}{\sqrt{2\pi r}} f_{ij}(\theta) + \text{non singular terms in } r \quad (2.10)$$

where σ_{ij} are the stress tensors acting on an element at angle θ from the crack plane and at a distance r from the crack tip. $f_{ij}(\theta)$ is a function of θ and K_I is defined as the mode I stress intensity factor. For different loadings, similar expressions to Equation (2.10) can be obtained. It can be seen from Equation (2.10) that the stress becomes singular near the crack tip, therefore for the region $r \ll a$, the singular term will be dominant and the non singular terms can be omitted. The magnitude of the stress field is then characterised by K_I . K_I of an infinite cracked plate illustrated in Fig.2.1 is given as:

$$K_I = \sigma \sqrt{\pi a} \quad (2.11)$$

Two cracked bodies made of the same material, but with different geometries, should have the same value of K_I at the onset of fracture, and this value is called the critical stress intensity factor K_{IC} , which is a material property. Irwin further considered the work done to extend the crack by a small amount and expressed the stress and displacement in terms of K_I . He demonstrated that the relationship between K and G was:

$$G_{IC} = \frac{K_{IC}^2}{E''} \quad (2.12)$$

where $E'' = E$ for plane stress and $E'' = E/(1-\nu^2)$ for plane strain conditions. ν is the Poisson's ratio. The same results can be obtained by combining Equations (2.3) and (2.11). Thus the plane strain fracture toughness characterised by G_{IC} can be equally characterised by K_{IC} .

2.3.2 Crack Tip Plasticity

From Equation (2.10), a stress singularity at the crack tip is unrealistic for real materials with finite yield stresses, therefore a small region of plastically deformed

material, termed a plastic zone, ahead of the crack tip is expected. Equation (2.10) can be used as a first approximation for determining the distance r_p along the crack plane over which the material is plastically deformed:

$$r_p = \frac{1}{2\pi} \left(\frac{K}{M\sigma_y} \right)^2 \quad (2.13)$$

where σ_y is the yield stress and M is the plastic constraint factor which accounts for the degree of plastic constraint at the crack tip due to triaxial stresses. The value of M varies from 1 to 3 and M is equal to 1 for the plane stress case. Irwin suggested that the crack would behave as if it were longer than its actual size. The notional crack length is $(a+r_p)$ with a yield region r_p ahead of the notional crack, and the K stress field dominates beyond this point as illustrated in Fig.2.5.

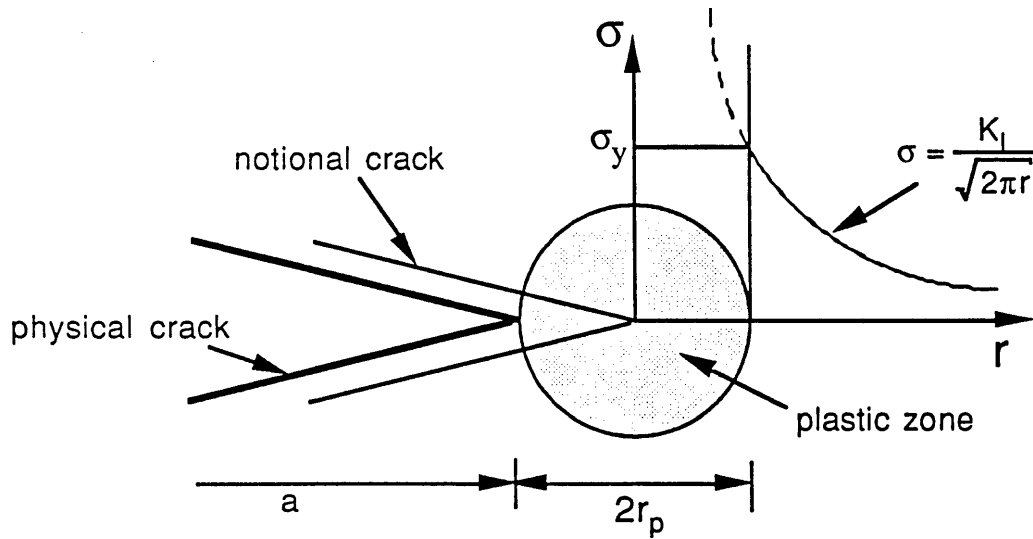


Fig.2.5 Plastic zone at a crack tip

K_{IC} as a fracture criteria will still be valid provided that r_p is sufficiently small compared to the K -dominated stress field. This condition is commonly referred to as the *small scale yielding* condition. A simple correction on the K_{IC} calculation can be made by replacing a with $(a+r_p)$ due to the presence of the plastic zone.

2.4 Evaluation of K_{IC} and G_{IC}

The solutions for K are rigourously true for an infinite plate. For a body with finite dimensions, the effect of geometry of the specimen can be corrected by introducing a geometry factor Y , so that:

$$K=Y\sigma \sqrt{a} \quad (2.14)$$

where Y is a function of (a/W) and W is the specimen width. Expressions of Y for different geometries are normally obtained by numerical analysis and can be found in the literature (Brown & Srawley[1966], Rooke & Cartwright[1976]).

G_{IC} can be determined by evaluating the stored strain energy U before fracture. By using Equation (2.7), Equation (2.9) can be written as:

$$G_{IC} = \frac{U}{BW} \left(\frac{1}{C} \frac{dC}{d(a/W)} \right) \quad (2.15)$$

It can be simplified to

$$G_{IC} = \frac{U}{BW\phi} \quad (2.16)$$

where

$$\phi = \frac{C}{dC/d(a/W)} \quad (2.17)$$

ϕ is the calibration factor and may be deduced from Y via Equations (2.9), (2.12) and (2.14). K_{IC} and G_{IC} of a particular material can be determined experimentally using specimen geometries for which $Y(a/W)$ and ϕ are known.

2.5 The Thickness Effect

The K and G analysis were originally limited to plane strain or general plane stress conditions. It can be seen from Equation (2.13) that the plastic zone size for plane strain conditions is smaller than that for the plane stress state. It is expected that the fracture toughness K_{IC} or G_{IC} will be a minimum under full plane strain conditions. Consider a cracked plate with finite thickness B . Plane stress conditions will prevail near the surfaces while the central region attains plane strain conditions. Thus the size of the plastic zone may be expected to vary along the thickness direction as illustrated in Fig.2.6.

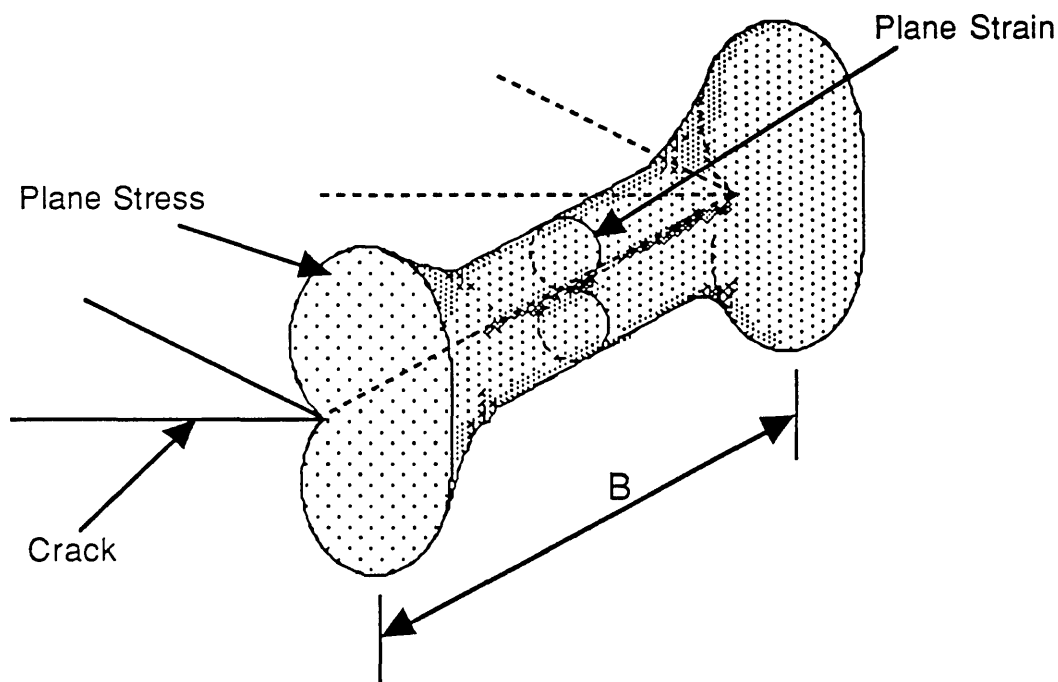


Fig.2.6 Variation of plastic zone size across the specimen thickness B

This will give rise to a distribution of K_{IC} values across the thickness. In reality, any measured K_{IC} value is an average of this distribution. Assuming the σ_y does not vary along the thickness, an approximation of the measured K_{IC} value, K'_{IC} , has been given by Williams[1984] :

$$K'_{IC} = K_{IC1} + \frac{K_{IC2}^2}{\pi B \sigma_y} (K_{IC2} - K_{IC1}) \quad (2.18)$$

where K_{IC1} and K_{IC2} are the K values over the central region and the surfaces of the specimen, respectively.

In order to maintain a plane strain condition, the specimen thickness must be large compared to the plastic zone size. Brown and Srawley[1966] showed that $(K_{IC} / \sigma_y)^2$ was a reasonable assumption of the characteristic dimension of the plastic zone. They found that constant K_{IC} values could be obtained for various types of steel if:

$$B, a, (W-a) \geq 2.5 \left(\frac{K_{IC}}{\sigma_y} \right)^2 \quad (2.19)$$

This criteria was later included in the standard testing procedure, ASTM E399, established by the ASTM for acquiring plane strain fracture toughness of metals.

2.6 Application of LEFM to Polymers

It has been mentioned in Section 2.1 that at first sight the viscoelastic nature of polymers makes the successful application of LEFM to polymers look doubtful. Williams[1987] argued that most polymers are essentially linear and are only slightly viscoelastic, therefore the conventional theory of LEFM can be utilized to characterise the fracture toughness of polymers without much modification. LEFM can be used to explain and predict various fracture phenomena in polymers, and have been discussed in detail by Williams[1978, 1984]. Since the mechanical properties of polymers are time and temperature dependent, it is expected that G_{IC} and K_{IC} also depend on these two parameters. K and G for polymers are often described as

functions of crack speed $\dot{a}$. This is particularly useful in predicting the long term performance of the materials and will be discussed in more detail in Chapter 4. Marshall et al.[1973] showed that for glassy polymers like PS and PMMA, instability occurred at high crack speed, and K became independent of $\dot{a}$. This K value corresponded to the K_{IC} of the materials. Thus, if the time dependence is not excessive and the loading period is short, it is not unreasonable to use LEFM for plane strain fracture toughness characterisation. In order to achieve brittle fracture for toughened polymers, it may be necessary to reduce the test temperature or increase the strain rate so that the yield stress is increased.

Chan and Williams[1981] demonstrated that the plane strain fracture toughness of several polyethylenes at various temperatures could be characterised successfully using the K_{IC} approach. They suggested that a valid K_{IC} could be achieved if the specimen size was larger than a certain size requirement, and they found that the ASTM size criterion, see Equation (2.19), was sufficient to ensure the plane strain condition. They also showed that K_{IC} was independent of initial crack length a . The same conclusion was reached by Mandell et al.[1983] on testing different types of medium density polyethylene. He pointed out that valid K_{IC} could only be obtained at low temperatures but the specimen thickness required was slightly larger than that calculated from the ASTM criterion. Hashemi et al.[1984] further investigated the applicability of LEFM to other polymers. He found that the ASTM size criteria were adequate for single edge notched (SEN) bend samples but a larger specimen would be necessary for SEN tension specimens. Therefore it was suggested that high constraint type specimens should be used for K or G tests in order to reduce the minimum specimen size required. The notches were normally prepared by machining with a flycutter and sometimes the machined notches were further sharpened by tapping or pushing a razor blade into the notches. Mandell et al.[1983] showed that notches introduced using this method were appropriate, and that the measured K_{IC}

value would not be affected if the tip radius was smaller than 0.1 mm. Cayard et al.[1989] also investigated the effect of precracking techniques on the measured K_{IC} of polycarbonate. Fatigue pre-cracking, razor notch guillotine and razor sawing methods were used and they found that each of these methods would provide adequate crack tip acuity, and similar K_{IC} values were obtained.

LEFM has been widely used for characterising the fracture toughness of polymers. A testing protocol for determining K_{IC} and G_{IC} for plastics has been drafted by Williams[1989] based on the efforts of the European Group on Fracture(EGF) task group on polymers and composites. The EGF protocol is basically the same as the ASTM E399 standard K_{IC} test method for metals but it has been modified slightly in order to deal with the unique properties of polymers. The procedures for the determination of G_{IC} are also included in this protocol.

2.7 Experimental

In this study, the fracture toughness of two grades of high density polyethylene (HDPE) have been characterised using both parameters G_{IC} and K_{IC} . The EGF test protocol was adopted. The SEN bend type specimen was chosen because it offers a higher constraint at the crack tip than the SEN tension geometry. As fracture toughness is time and temperature dependent, experiments have been conducted at various temperatures and at different strain rates at room temperature. The effect of initial crack size on the fracture toughness has also been investigated. In order to understand the behaviour of the materials, it was necessary to measure the yield stress and modulus at various test conditions. This also provided information for determining the required specimen size for valid plane strain fracture toughness.

2.7.1 Materials and Specimen Preparation

Two grades of HDPE supplied by DSM Kunststoffen BV in the form of slow cooled compression moulded sheets were tested. The properties of the materials are listed in Table 2.1.

Designation	MFI (g/10 min)	Density(kg/m ³)
8621	0.16	958
5731	0.03	945

Table 2.1. Properties of the polyethylenes

Density distributions across the sheets were measured according to the ASTM D792 method A1. Non-uniform density distribution for both materials can be observed in Fig.2.7. It indicates that scatter in the experimental results will be expected due to this anomalous density distribution even if the specimens were cut from the same sheet. It was also found that the fresh sheets gave a lower E than the

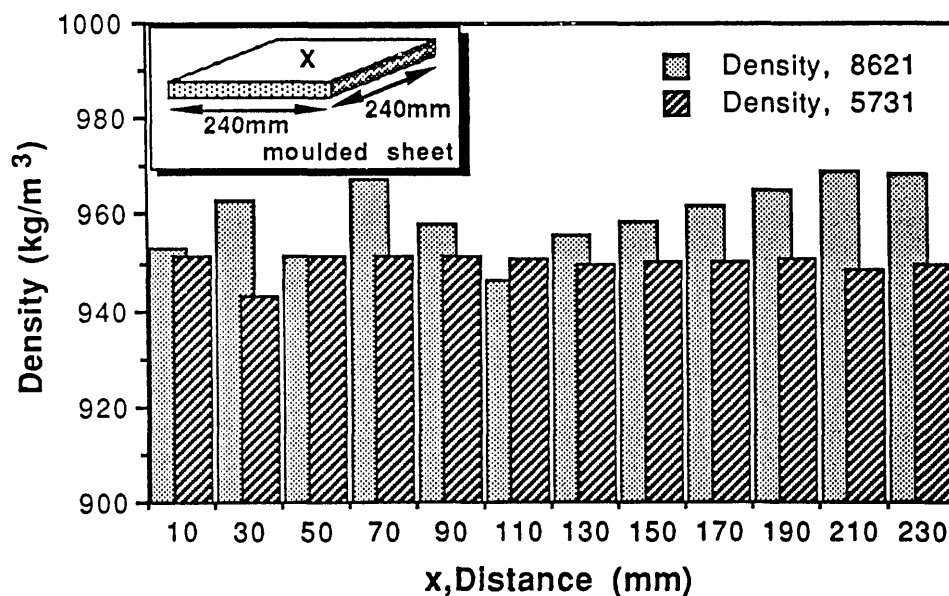


Fig.2.7 Density distribution across the compression moulded sheets

aged ones. Therefore all sheets were stored at room temperature for at least one month before any specimens were fabricated from them.

SEN specimens were fabricated from the 12 mm thick plaques in the way shown in Fig.2.8. A slot was first made with a slot cutter and the final 2 mm of the notch was further sharpened with a single point fly cutter or by slicing a new razor blade into the notch. The tip radii of the fly cutter and the razor blade were measured using an optical shadowgraph and were found to be approximately $16\text{ }\mu\text{m}$. According to the results from Mandell et al.[1983], notches prepared by either of these methods should provide sufficient sharpness at the crack tip. Specimens were notched to various crack to width ratios and stored for at least two days before testing took place. This helped to relax the residual stresses at the crack tip induced by the notching process.

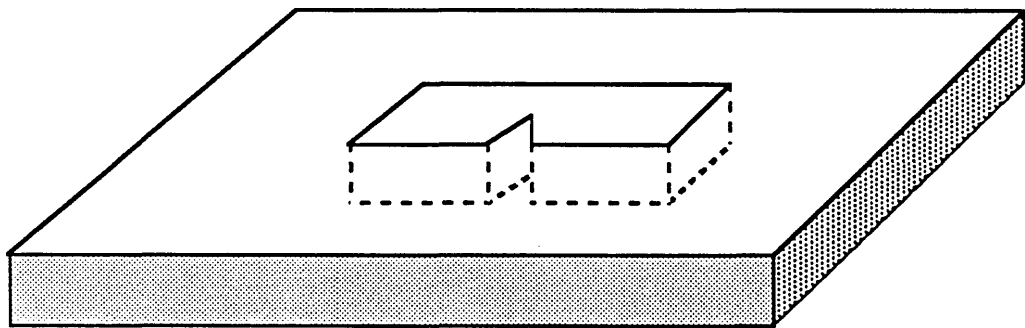


Fig.2.8 Fabrication of Specimen

Dumb-bell shaped 4 mm thick specimens with dimensions shown in Fig.2.9 were used to measure the yield stress and elastic modulus of the materials. Rectangular blanks were cut from the sheets using a circular saw and machined to the required shape and size.

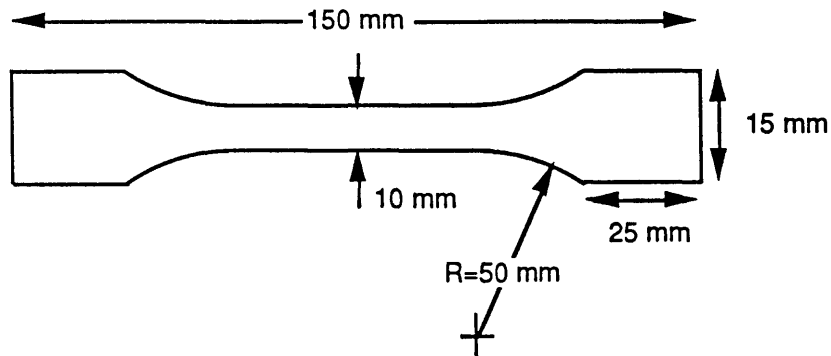


Fig.2.9 Tensile specimen configurations

2.7.2 Testing Procedures

2.7.2.1 Fracture Testing

Three point bend tests were performed on an Instron testing machine using a three point bend rig with adjustable free roller supports shown diagrammatically in

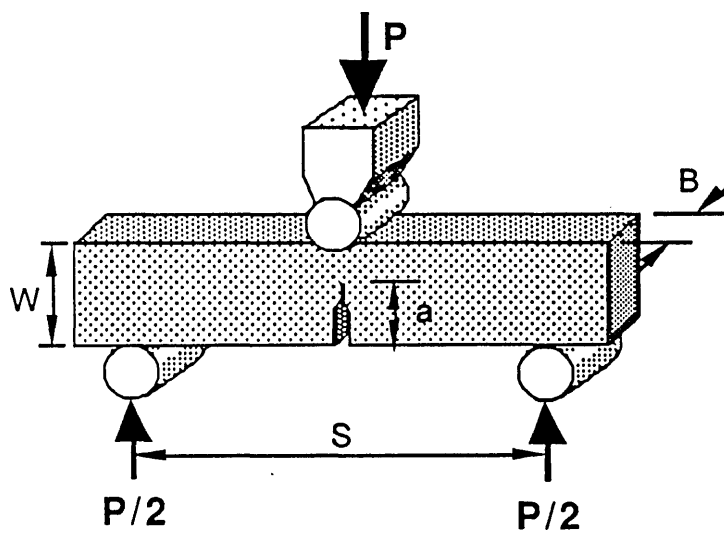


Fig.2.10 Set-up for three point bend tests

Fig.2.10. The load and load-line displacement were recorded by a computer and on a chart recorder simultaneously. All specimens were loaded beyond the maximum load.

Low temperature tests were carried out in an insulated box cooled by vapourised liquid nitrogen. The temperature inside the box was monitored by a thermocouple located close to the crack tip and was controlled by a "Eurotherm" unit capable of maintaining the desired temperature to an accuracy of $\pm 1^\circ\text{C}$. All the specimens were 12 mm thick and 30 mm wide with a/W of 0.6. They were loaded with a span to width ratio of 4 at a cross head speed of 2 mm/min over a temperature range of $+20^\circ\text{C}$ to -120°C . The specimens were kept inside the box at the test temperature prior to testing until thermal equilibrium was reached.

Specimens with various widths were tested. For each width, the specimens were notched to different a/W ratios ranging from 0.1 to 0.5. The nominal strain rate $\dot{\epsilon}_n$ of three point bend samples depends on the specimen size and the cross head speed $\dot{X}$ of the testing machine, it is given as:

$$\dot{\epsilon}_n = 6 \dot{X} \left[\frac{W}{S^2} \right] \quad (2.20)$$

In order to maintain a constant strain rate, the cross head speed was adjusted to the closest value to the one calculated using Equation (2.20) for specimens of different size. Tests were performed at -120°C to ensure that brittle fracture would be achieved.

The effect of loading rate on K_{IC} was investigated by loading the specimens with the same dimensions as those used in the low temperature tests, at different cross head

speeds, at room temperature. The nominal strain rates were evaluated using Equation (2.20). At least three replicates were tested for each test condition.

A displacement correction was made to account for elastic and plastic indentations of the specimen by the loading head and the support rollers. This could be done by using the actual test fixture and the specimen used in the test as shown in Fig.2.11. The support rollers were butted together to provide a short span. A broken and undeformed specimen was placed on the butted rollers with the same orientation as that during the test. The specimen was loaded to the actual test load at half the fracture test loading speed such that the loading time was about the same. The load-displacement correction curve was then subtracted from that in the fracture test to obtain the true displacement. In the G_{IC} tests, the energy was corrected by subtracting the area under the correction curve from that of the fracture test load displacement curve.

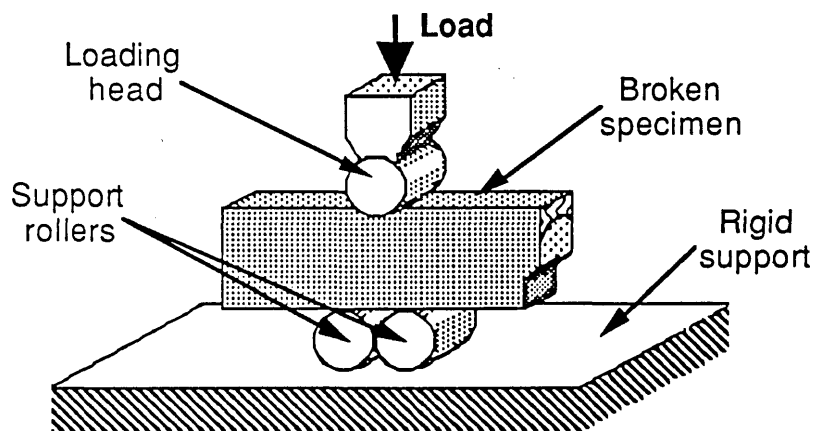


Fig.2.11 The testing fixture for indentation correction

2.7.2.2 Yield Stress Tests

Yield stress tests were performed at various cross head speeds at room temperature. The maximum load and the original cross sectional area were used to

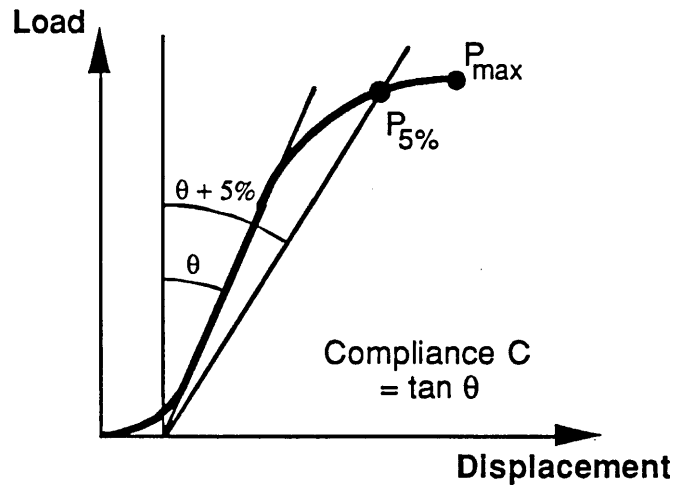
calculate the yield stress. Yield stresses at low temperature were measured at a cross head speed of 5 mm/min and experiments were carried out in an insulated box as described in previous section. Elastic modulus measurement was made by mounting an extensometer on the specimen to monitor the gauge length extension. The load and displacement were registered by a computer and the modulus was determined from the initial part of the stress-strain curve where the material exhibited elastic response.

2.7.3 Determination of K_{IC} and G_{IC}

The ideal load displacement curve should be linear up to the maximum load and then drop abruptly to zero load. The experimentally measured K_{IC} value, K_Q , can be calculated from the maximum load in this case. However, it is well known that the fracture process in most cases is not comprised of a single sudden explosive-type change from initial crack length to total failure and non-linearity in the load displacement diagram is often observed. It may be caused by plastic deformation at the crack tip, non-linear elasticity or general viscoelasticity which will violate the LEFM assumptions. It may also indicate that slow crack growth takes place prior to instability and the maximum load does not truly define the initiation point in this case. The EGF protocol suggests an arbitrary rule in which the maximum load P_{max} and the 5% load $P_{5\%}$ determined from the 5% increase of initial compliance were measured from the load-displacement curve, see Fig.2.12. The test is considered to be valid if:

$$\frac{P_{max}}{P_{5\%}} < 1.1 \quad (2.21)$$

This gives an allowance for 10% on non-linearity. The smaller of the P_{max} and $P_{5\%}$, termed as P_Q , is used for evaluation of K_Q and G_{IC} . The initial crack length was measured from the fracture surface after testing using a travelling microscope.

Fig.2.12 Determination of $P_{5\%}$ and C (Williams[1989])

K_Q can be calculated from the equation:

$$K_Q = f \frac{P_Q}{BW^{1/2}} \quad (2.22)$$

and for the SEN bend specimen with $S/W=4$, ($0 < \alpha < 1$)

$$f = 6\sqrt{\alpha} \frac{[1.99 - \alpha(1-\alpha)(2.15 - 3.93\alpha + 2.7\alpha^2)]}{(1+2\alpha)(1-\alpha)^{3/2}} \quad (2.23)$$

where $\alpha = a/W$. If K_Q satisfies the size criteria shown in Equation (2.19), the measured K_Q will be the K_{IC} of the material.

G_{IC} can be evaluated from the corrected energy U via Equation (2.16), and the calibration factor ϕ for SENB specimens with $S/W=4$ is given as:

$$\phi = \frac{A+18.64}{dA/d\alpha} \quad (2.24a)$$

$$A = \frac{16\alpha^2}{(1-\alpha)^2} [8.9 - 33.717\alpha + 79.616\alpha^2 - 112.952\alpha^3 + 84.815\alpha^4 - 25.672\alpha^5] \quad (2.24b)$$

The elastic modulus of the material can be evaluated alternatively from the load displacement curve of the fracture tests. By measuring the initial compliance C_i ($\tan \theta$ in Fig.2.12), the modulus can be calculated from the expression given by Bakker[1990]:

$$E = \frac{A+18.64}{BC_i} \quad (2.25)$$

where A is the expression given in Equation (2.24b) and B is the specimen thickness.

2.8 Results and Discussion

2.8.1 Tensile Yield Stress and Modulus

2.8.1.1 Temperature Effect

The variation of yield stress σ_y with temperature for the two grades of HDPE is shown in Fig.2.13. All specimens showed necking at maximum load, and elongation of the gauge length at the maximum load decreased with decreasing temperature. The 5731 HDPE was more ductile and had a lower yield stress than the 8621 material over the whole range of temperatures. Yield stress increases with decreasing temperature for both materials.

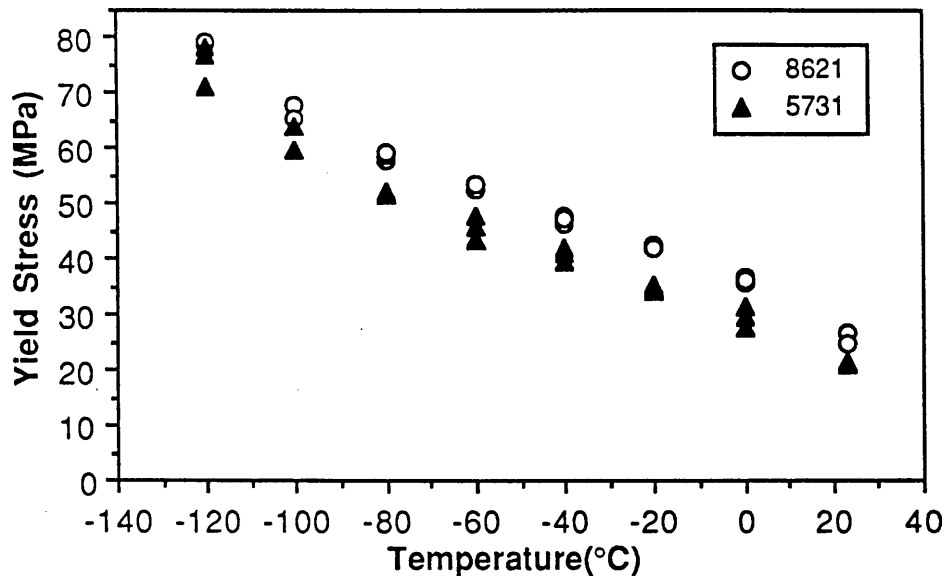


Fig.2.13 Variation of yield stress with temperature

The modulus at different temperatures is shown in Fig.2.14. It can be seen that the results from the tensile tests show a high degree of scatter some of which may be due to the calibration of the extensometer. The extensometer was calibrated at room temperature, and it was mounted on the specimen before loading in an attempt to minimize the error caused by the temperature difference. However, the temperature change caused thermal contraction of the extensometer which induced errors in displacement measurement. Moduli measured from the fracture tests, using Equation (2.25), are also included in Fig.2.14. Less scatter is observed and both sets of data follow the same trend as temperature changes.

It is considered that the modulus calculated from the initial compliance is more appropriate here since the results are more consistent and the values have the same order of magnitude as those measured from tensile tests. The values of the moduli measured from the initial compliance are shown in Fig.2.15. For both polyethylenes, the modulus increases with decreasing temperature.

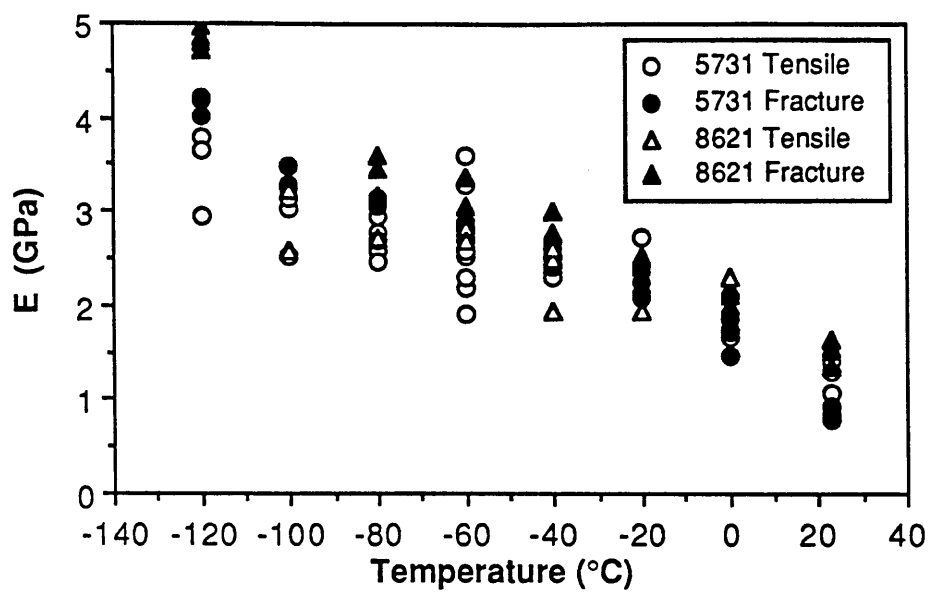


Fig.2.14 Variation of modulus with temperature

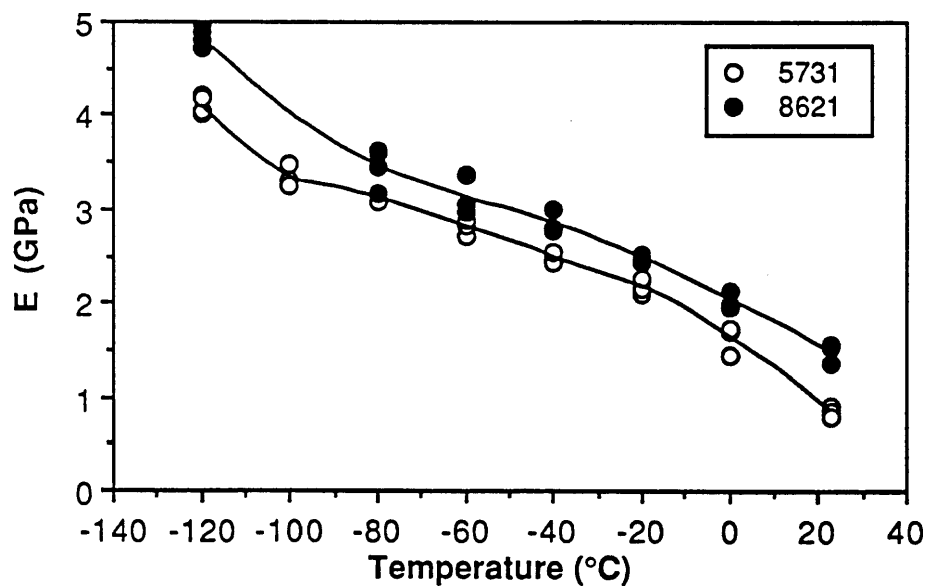


Fig.2.15 Temperature dependence of modulus

2.8.1.2 Strain Rate Effect

The effect of strain rate on the yield stress for both HDPE materials is shown in Fig.2.16. It can be seen that the relationship between σ_y and the strain rate $\dot{\epsilon}$ can be represented in the form of the power law:

$$\sigma_y \propto \dot{\epsilon}^m \quad (2.26)$$

where m is the rate dependence measured from the slope of the line in Fig.2.16. m was found to be 0.1 and 0.09 for the 8621 HDPE and 5731 HDPE respectively for strain rates below 10^{-2}min^{-1} . It can be noted that m decreases at high $\dot{\epsilon}$. Fig.2.17 shows the variation of modulus with strain rate. Similarly, the strain and the modulus can also be related by a power law with a rate dependence n as:

$$E \propto \dot{\epsilon}^n \quad (2.27)$$

The modulus determined from the fracture tests for the 8621 HDPE are shown to be

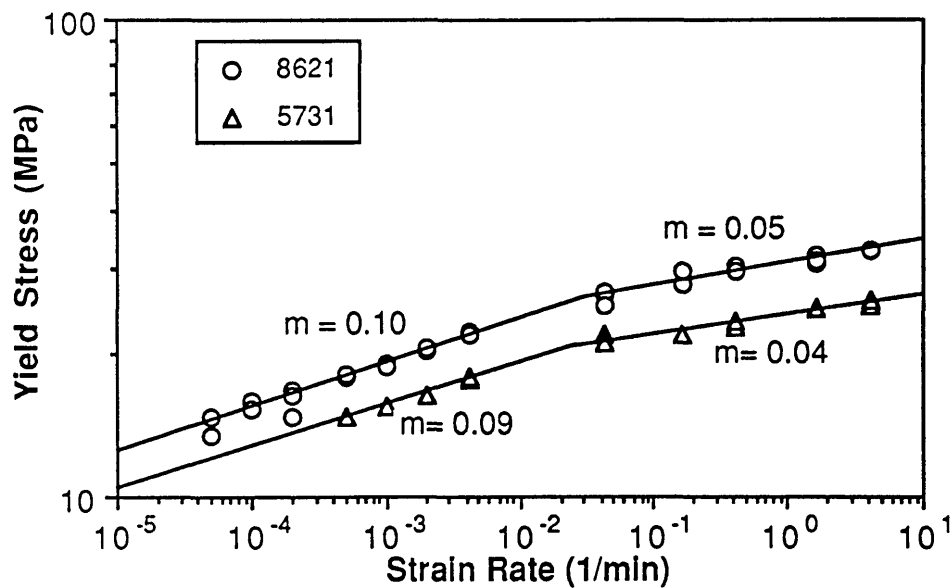


Fig.2.16 Strain rate dependence of yield stress

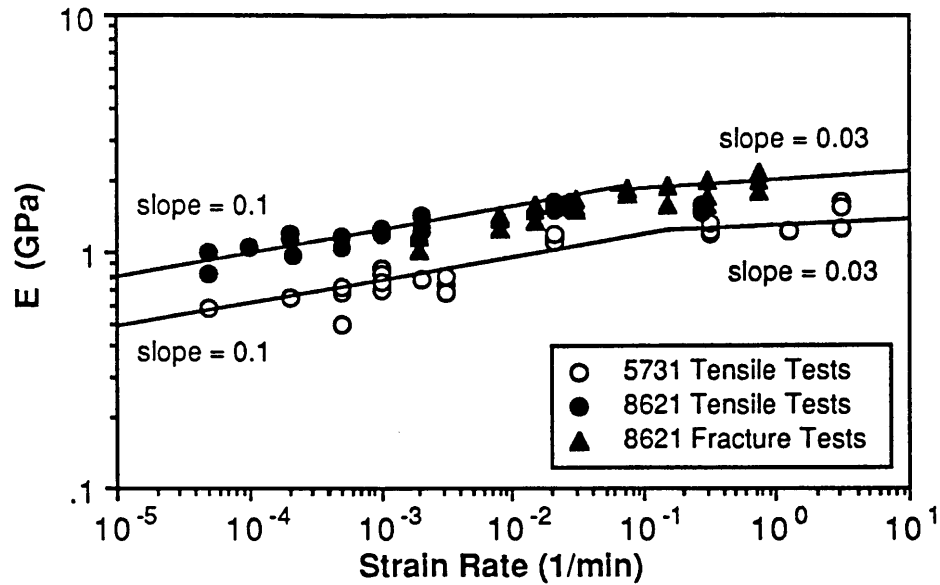


Fig.2.17 Strain rate dependence of modulus

in good agreement with those from the tensile tests. n was found to be 0.1 for both polyethylenes at $\dot{\epsilon}$ below 10^{-2}min^{-1} . From Figs.2.16 and 2.17, it can be noted the rate dependence of yield stress and modulus decreases at higher strain rates. The changes of m and n with $\dot{\epsilon}$ were also reported by Chan[1983]. Since polyethylene is a semi-crystalline polymer, the measured modulus and yield stress can be treated as the combined ones of the crystalline and amorphous regions. As the strain rate increases, there is less time for the molecules in the amorphous regions to disentangle. The crystalline regions will have a stronger influence on the properties and this will increase the σ_y and E of the material. In addition, this may also account for the change in rate dependence at higher strain rates. However, the exact deformation mechanisms are much more complex and the reason for the changes in m and n is far from clear.

2.8.2 K_{IC} and G_{IC} Testing

For the 5731 material at room temperature, the specimens suffered severe crack tip blunting and through-thickness contraction, and failed by gross yielding of the

remaining ligament. The test temperature was lowered to -100°C and -120°C in order to promote brittle failure. However, examination of the fracture surfaces revealed shear lips and lateral contraction on the sides of the specimens. Although the material behaved in a less ductile manner at low temperature, the tests were still invalid according to the ASTM size criteria, see Equation 2.19. Brown and Srawley[1966] suggested that for SEN bend specimens, the gross stress should be less than the yield stress for LEFM to be applicable. Fig.2.18 illustrates that the maximum loads recorded in the fracture tests are above the yield loci; the load at which net section yielding will occur. This renders the LEFM analysis inapplicable in this case.

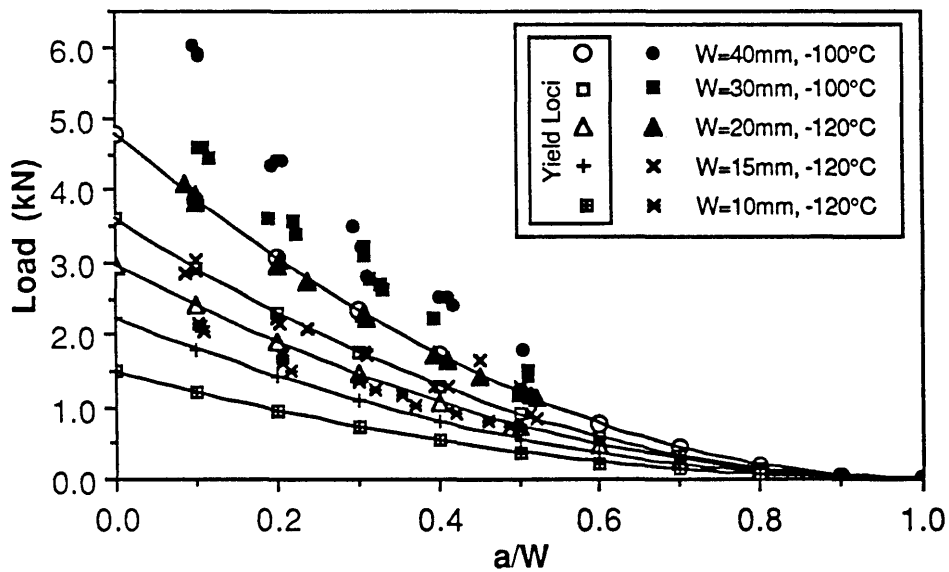


Fig.2.18 The fracture load for 12 mm thick 5731 specimens with various a/W ratios

Since the specimen size was limited by the thickness of the compression moulded sheet, it was not possible to fabricate specimens with thickness greater than 12 mm. Another approach, discussed in Chapter 3, has been adopted to measure the plane strain fracture toughness of this material. Due to the extreme toughness of the 5731 HDPE which invalidates the LEFM analysis, all the results reported here are on the 8621 material only.

2.8.2.1 Temperature Effect

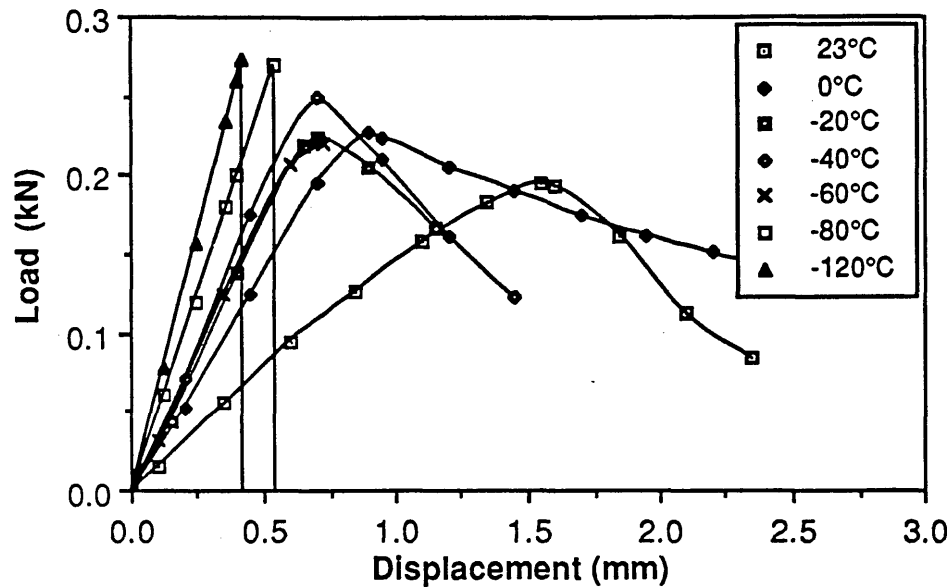


Fig.2.19 Load-displacement curves for the 8621 HDPE at various temperatures

The load-displacement records at different temperatures are shown in Fig.2.19. The load-displacement curve broadened, and the degree of non-linearity increased as temperature increased. At low temperatures, the curves were linear up to the maximum load and the peaks were well defined.

Stable crack growth after attainment of the maximum load was observed at temperatures above -60°C and crack front 'bowing' was found on the fracture surfaces. For test temperatures below -60°C , unstable fracture was obtained. Values of K_Q together with those calculated from the G_{IC} values via Equation (2.12) are plotted against temperature in Fig.2.20.

It can be seen that the results from these two approaches are in good agreement except for the tests performed at room temperature. It must be pointed out that the criterion of linearity of the load-displacement diagram shown in Equation (2.21) was

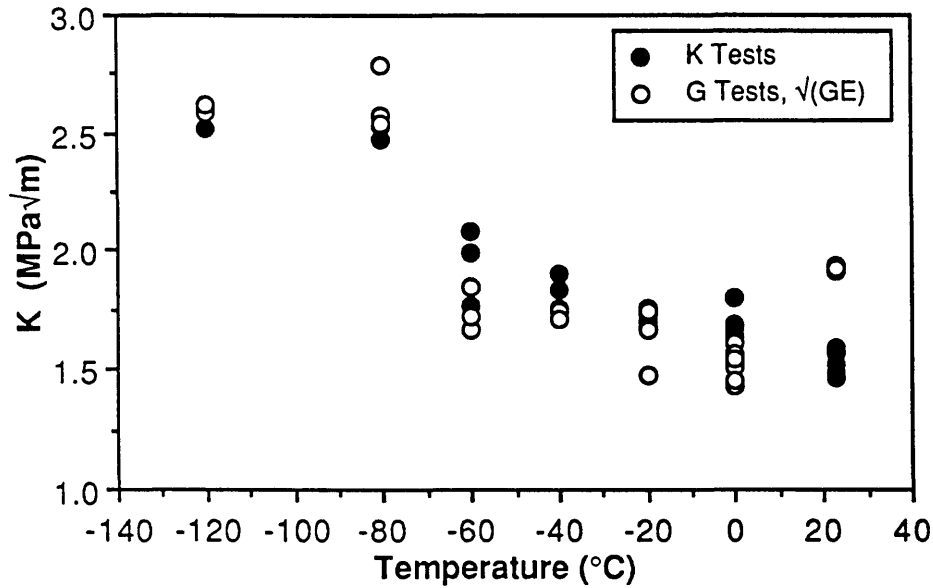


Fig.2.20 Variation of K_Q with temperature for 8621 HDPE

violated for the tests at room temperature. Since the $P_{5\%}$ load was too small, and the crack was observed to grow near the maximum load, the maximum load was used to evaluate the K_Q as K_{max} . This gave an indication of the fracture resistance of the material at room temperature.

K_Q was found to increase with decreasing temperature. A pronounced increase in K_Q could also be observed at temperatures below -60°C . Chan[1983] suggested that the increase in K_Q was due to the test temperature approaching the transition temperature of the material. Dynamic mechanical thermal analysis(DMTA) was performed on both grades of polyethylene at a frequency of 1Hz. A loss peak was found at -105°C which corresponded to the γ transition as shown in Fig.2.21. Young[1983] pointed out that the γ transition in the polyethylene was probably the glass transition in the amorphous region. It can be seen from Fig.2.20 that the peak in K_Q does not coincide with the loss peak. However, Kisbenyi et al.[1979] showed that the positions of loss peaks could depend on frequency, the difference between the frequency of the DMTA tests and the frequency of the fracture process might cause the inconsistency of the peaks.

A peak can also be observed in the plot of G_{IC} versus temperature shown in Fig.2.22. The equivalent G_{IC} values evaluated from the corresponding K_{IC} values are also included. Apart from the results of the tests at room temperature, there is good agreement. The G_{IC} value increases with increasing temperature except at the temperatures near the γ transition. Although the load capacity was increased by lowering the temperature, the energy absorbing capacity was considerably reduced. The increase in K_Q and G_{IC} values near the transition has been considered by Chan[1983] who suggested the phenomenon was caused by the increase in viscoelastic loss, $\tan\delta$. However, it has been pointed out by Kisbenyi et al.[1979] that the proportion of increase in $\tan\delta$ at the loss peak was too small to justify the large increase in the fracture energy if they were directly proportional to each other. Although this problem was not resolved, they suggested that the increase in G_{IC} was probably caused by the energy absorbing processes like crazing and formation of subsidiary crazes around the crack tip.

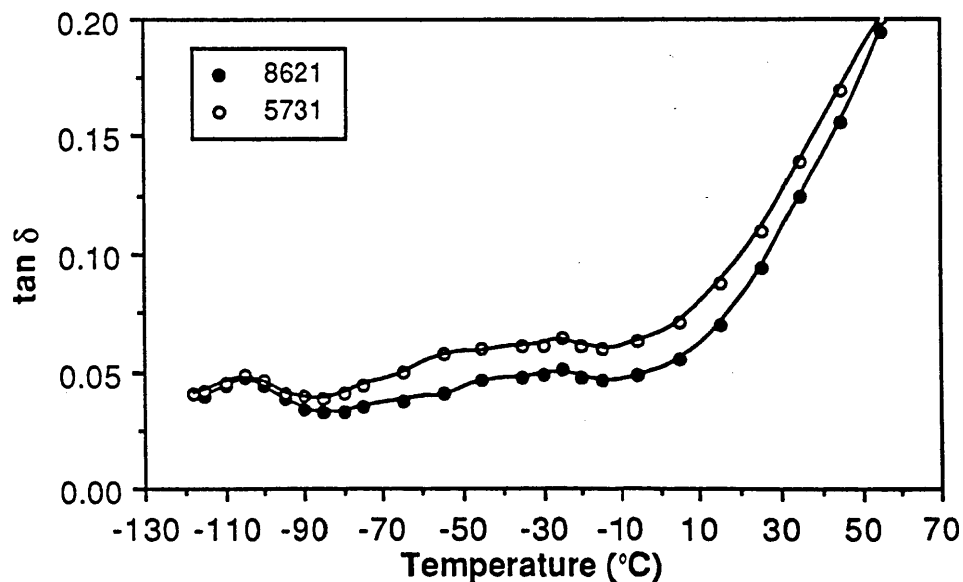


Fig.2.21 Variation of loss modulus with temperature

By examining the fracture surfaces using a scanning electron microscope, it is evident from Fig.2.23 that the fracture morphology changes from ductile to brittle as temperature decreases. Fibrils can be observed from the fracture surfaces and this form of micro-ductility may be one of the major sources of the toughness of the material. It can be seen that the size of the fibrils decreased with decreasing temperature, therefore the energy to fracture is also expected to decrease. At -120°C , the fracture surface is essentially brittle and it may be inferred that the fibrils were formed by the flow of the material in the amorphous regions. The motion of the molecules was inhibited at temperature below the γ transition and no fibrils were observed. It is interesting to note that the fracture surface at -80°C appears to be more brittle than that at -60°C , but the G_{IC} value of the former is about two times higher than the latter. Therefore, the energy absorbing process must be dominated by other mechanisms rather than the plastic deformation of fibrils. Nevertheless, it is still not clear whether the peaks in G_{IC} and K_Q are caused by the loss peak and more work will be necessary to clarify this point.

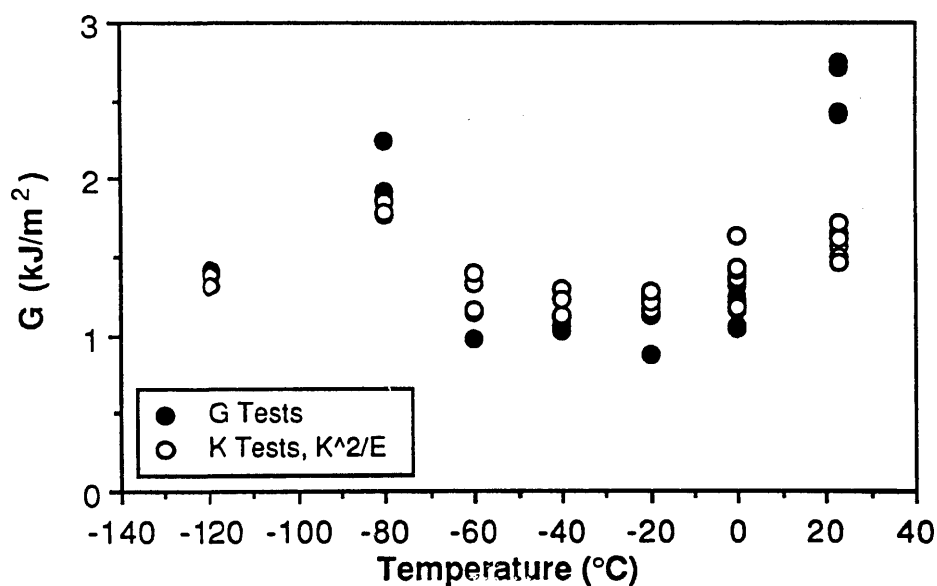
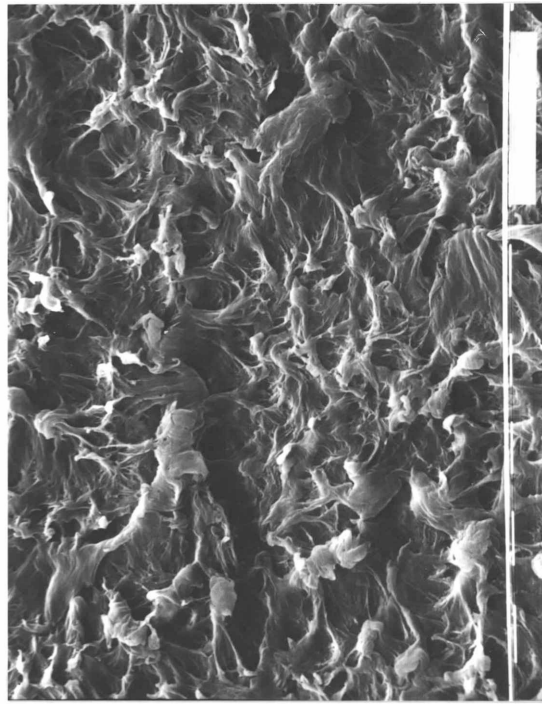
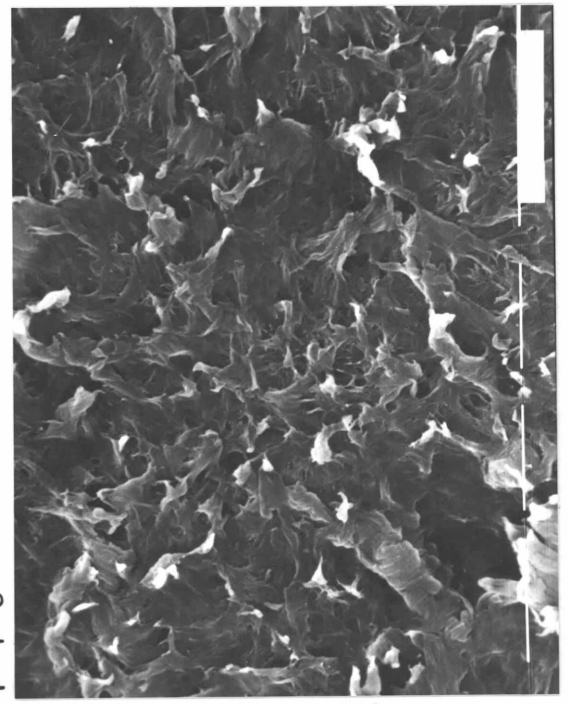


Fig.2.22 Variation of G_{IC} with temperature for 8621 HDPE

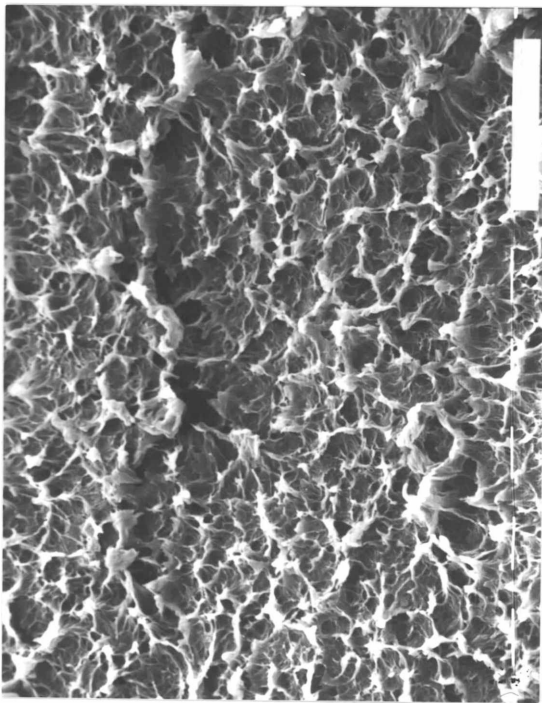


(a) 10 μm

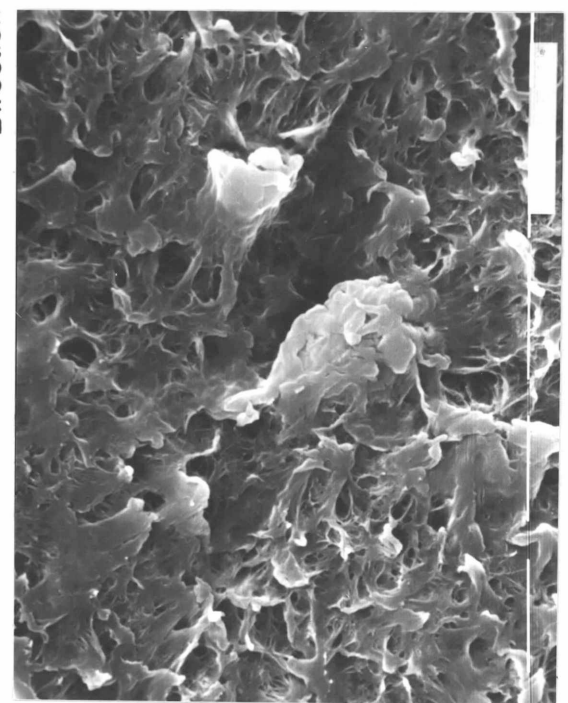


(b) 10 μm

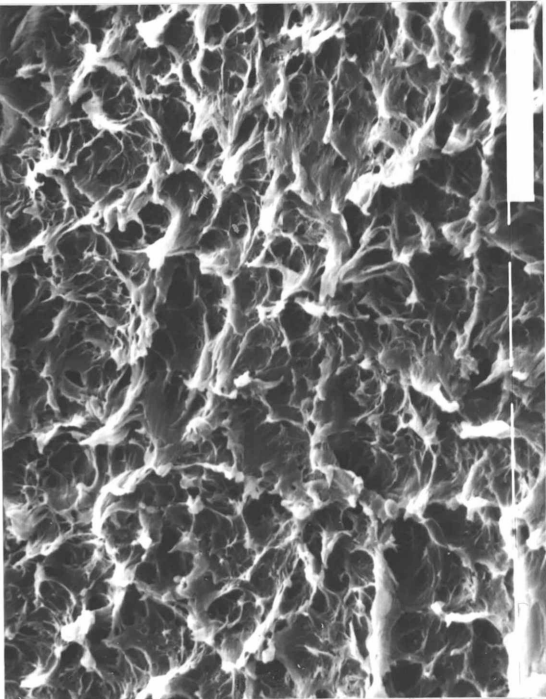
Direction of crack propagation ↑



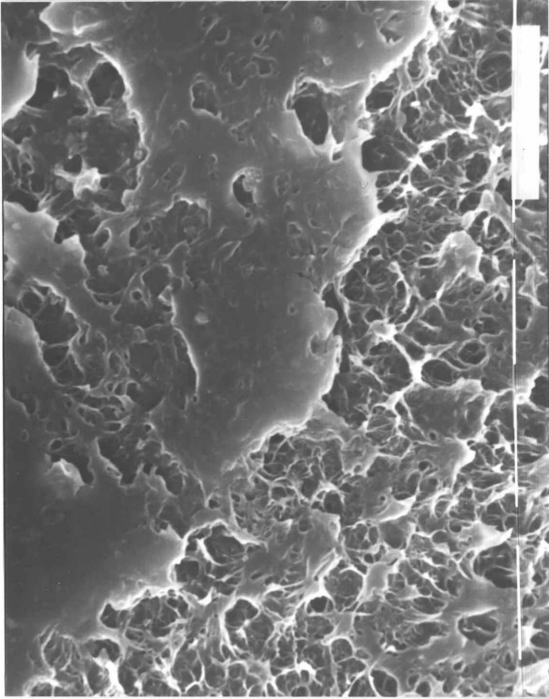
(c) 10 μm



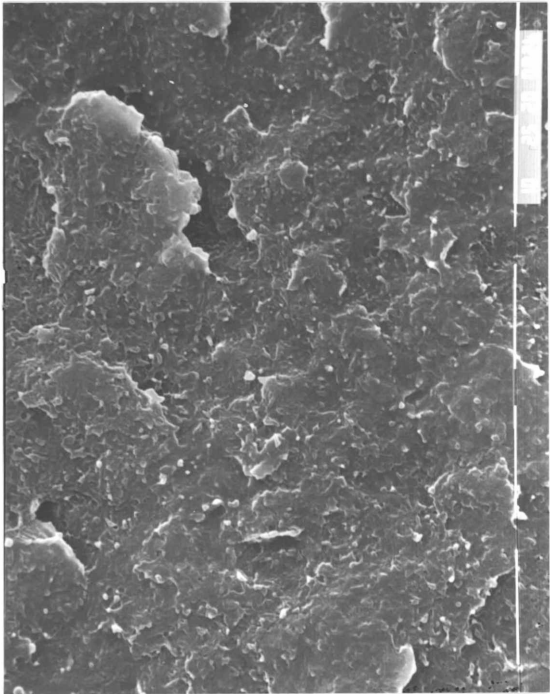
(d) 10 μm



(e) 10 μm



(f) 10 μm



(g) 10 μm

Direction of crack propagation $\uparrow$

Fig.2.23 Fracture surfaces for 8621 specimens at various temperatures. (a) 23°C, (b) 0°C, (c) -20°C, (d) -40°C, (e) -60°C, (f) -80°C, (g) -120°C

The minimum required thicknesses determined from Equation (2.19) for valid K_{IC} values are shown in Fig.2.24 for different test temperatures. All the tests satisfied the size criteria. For the tests below room temperature, the measured K_Q can be regarded as the K_{IC} of the material according to the EGF protocol. Near the transition temperature, a slight increase in the required specimen size is observed due to the increase in K_Q value.

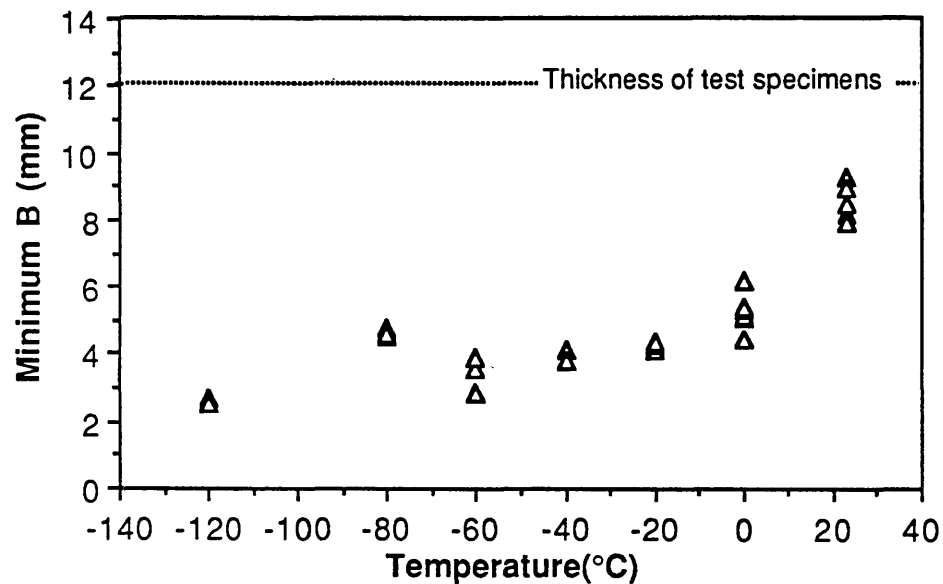


Fig.2.24 Minimum required thickness for valid K test for 8621 at different temperatures

It has been demonstrated that the discrepancy between K_{IC} and G_{IC} becomes significant at room temperature. The G_{IC} value at room temperature was higher than that obtained from the K_Q value. The K_Q value was calculated from the maximum recorded load, but crack initiation could occur at a load prior to the maximum load. The energy evaluated up to the maximum load would therefore include the energy for stable crack growth and plastic deformation at the shear lips after the crack initiated. This would lead to an over-estimation of the G_{IC} value. The linearity criterion has been overlooked by most researchers in the past because non-linearity in the load-displacement curve was expected for most polymers. The maximum load was commonly used for evaluation of K_Q and the value would be regarded as valid when

the ASTM size criteria were satisfied. Since the K_{IC} value is load determined, a small difference between the true initiation load, P_i , and the maximum load in a non-linear load-displacement curve will still yield an acceptable estimation of the K_{IC} value if the maximum load is used, but it may induce a large error in the evaluation of the fracture energy as illustrated in Fig.2.25. This indicates that the linearity criterion shown in Equation (2.21) must be strictly followed for the determination of G_{IC} .

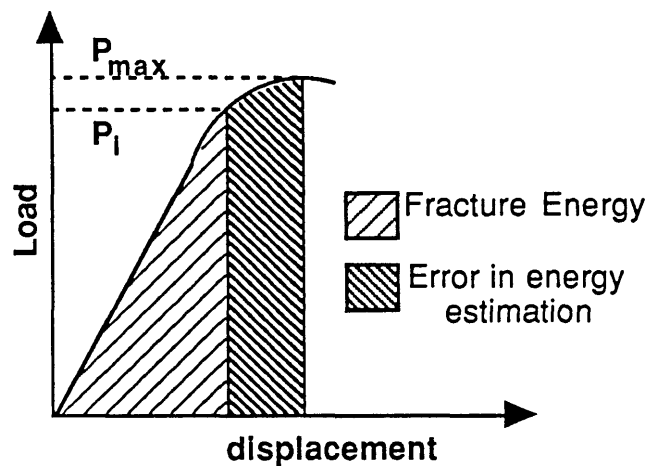


Fig.2.25 Error in energy estimation for the determination of G_{IC}

Although the K tests at room temperature are invalid on the ground of excessive non-linearity, it is believed that the measured K_{max} value is a reasonable estimate of the K_{IC} and the results from the elastic-plastic fracture tests, covered in Chapter 3, show good agreement with the K_{max} value.

2.8.2.2 Loading Rate Effect

The load-displacement diagrams for the 8621 HDPE at various loading rates at room temperature are shown in Fig.2.26. The maximum load increases with increasing cross head speed due to the increase in yield stress and modulus at higher strain rates.

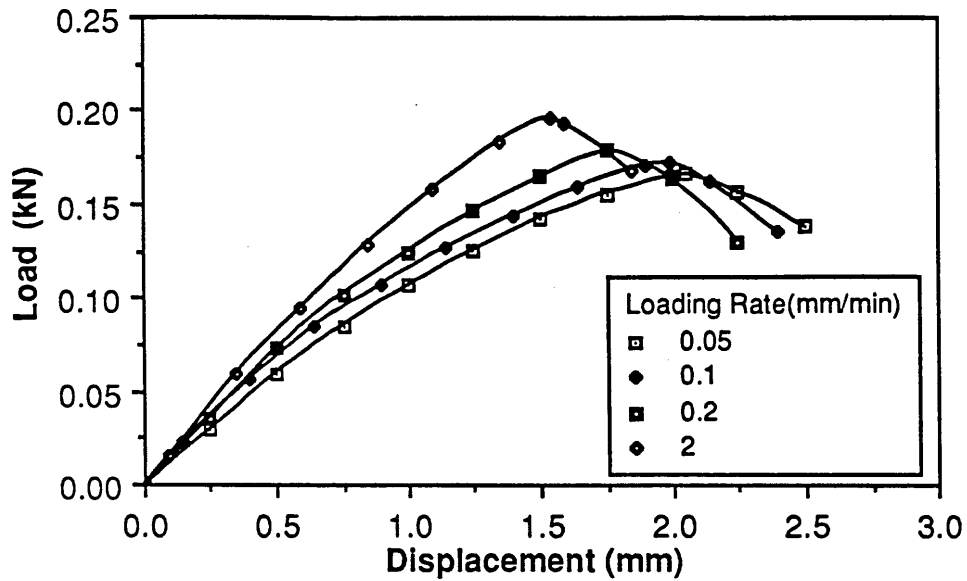


Fig.2.26 Load displacement diagrams for 8621 at various loading rates at 23°C

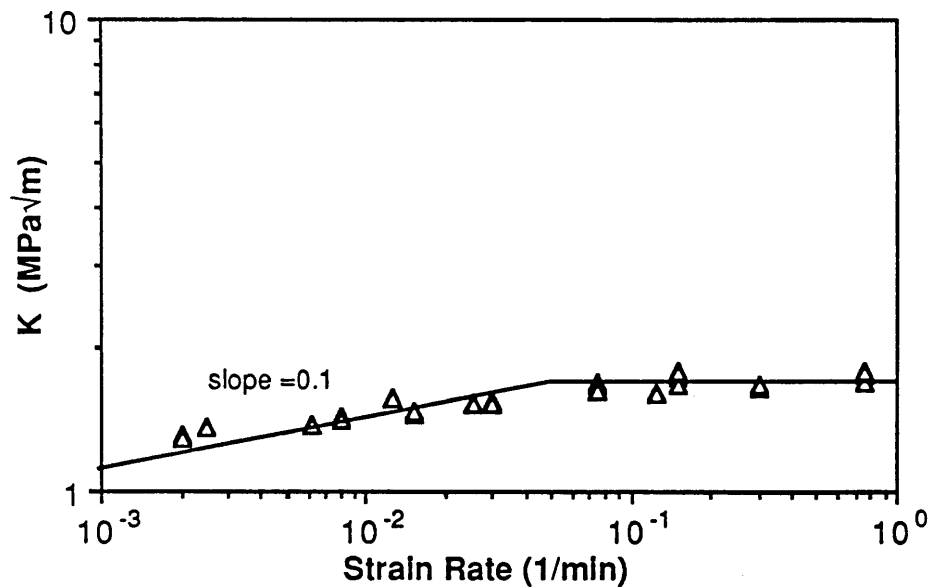


Fig.2.27 Strain rate dependence of K_{max} for 8621 at 23°C

It has been demonstrated that the G_{IC} approach is not appropriate for tests at room temperature due to the error in energy evaluation, therefore the G_{IC} values at different strain rates have not been measured. Fig.2.27 shows the change of K_{max} value with strain rate. The nominal strain rate was computed using Equation (2.20).

Since the strain rate dependence of the yield stress and modulus can be described by a power law, the same is expected for the rate dependence of K . Williams[1984] proposed that the rate dependence should be written as:

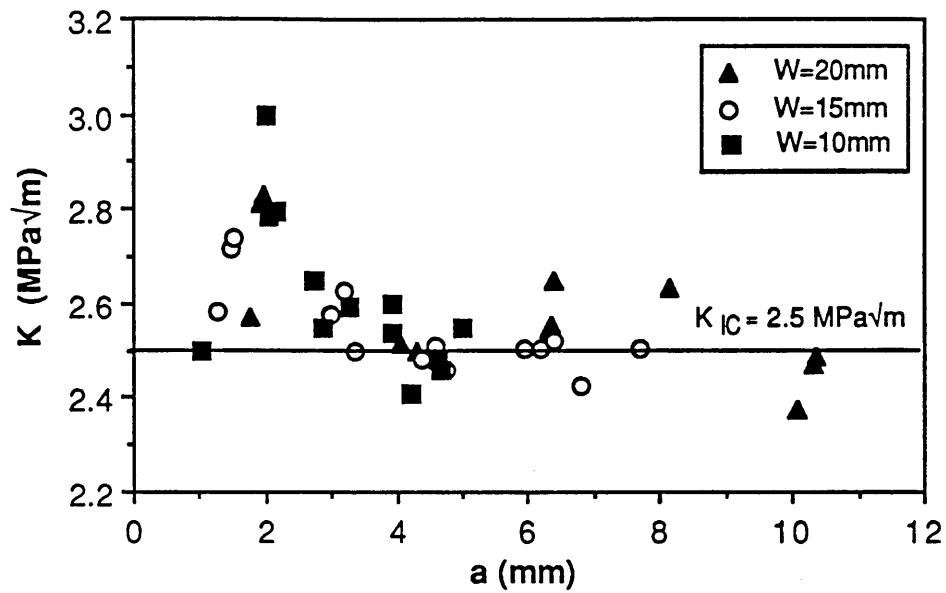
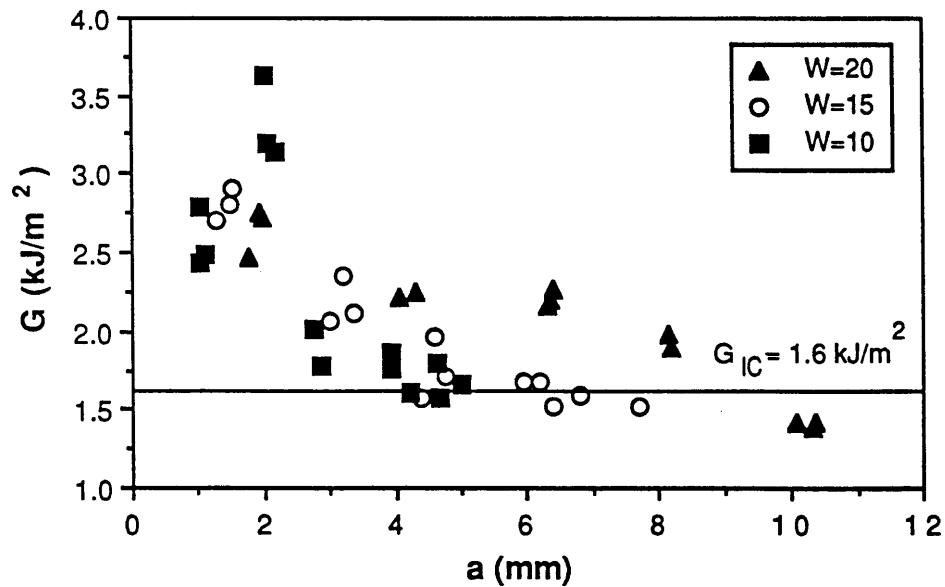
$$K \propto \dot{\epsilon}^{n/2} \quad \text{for } J = J_c \quad (2.28a)$$

$$K \propto \dot{\epsilon}^{(m+n)/2} \quad \text{for } \delta = \delta_c \quad (2.28b)$$

where m and n are the rate dependence of yield stress and modulus respectively. J is the J integral and δ is the crack opening displacement which will be discussed in Chapter 3. The subscript c denotes the critical value at which fracture initiates. Equations (2.28a) and (2.28b) represent the constant J and the constant δ fracture criteria respectively. Both criteria assume fracture to occur when δ and J reach the critical values of δ_c and J_c , respectively. From the results shown in Figs 2.16, 2.17 and 2.27, the change of rate dependence is observed in each figure. At a strain rate of 10^{-2} min^{-1} , $m \approx n \approx 0.1$ and the rate dependence of K_{max} also gives a value about 0.1 which satisfies Equation (2.28b). It appears that the critical crack opening displacement is the appropriate fracture criterion for this material. This criterion is particularly important when modelling the slow crack growth in polymers, and will be covered in Chapter 4. It is also noted from Fig.2.27 that K_{max} attains a fairly constant value at high strain rate. It is believed that this is due to the weak dependence of σ_y and E at high strain rate, and the K_{max} value is approaching the K value at instability which is a constant.

2.8.2.3 Effect of Initial Crack Length

Specimens with widths of 10, 15, and 20 mm and a thickness of 12 mm were tested at -120°C . Fig.2.28 and Fig.2.29 show the effects of initial crack size on K_{IC} and G_{IC} , respectively.

Fig.2.28 Variation of K_{IC} with a for 8621 HDPE at -120°C Fig.2.29 Variation of G_{IC} with a for 8621 HDPE at -120°C

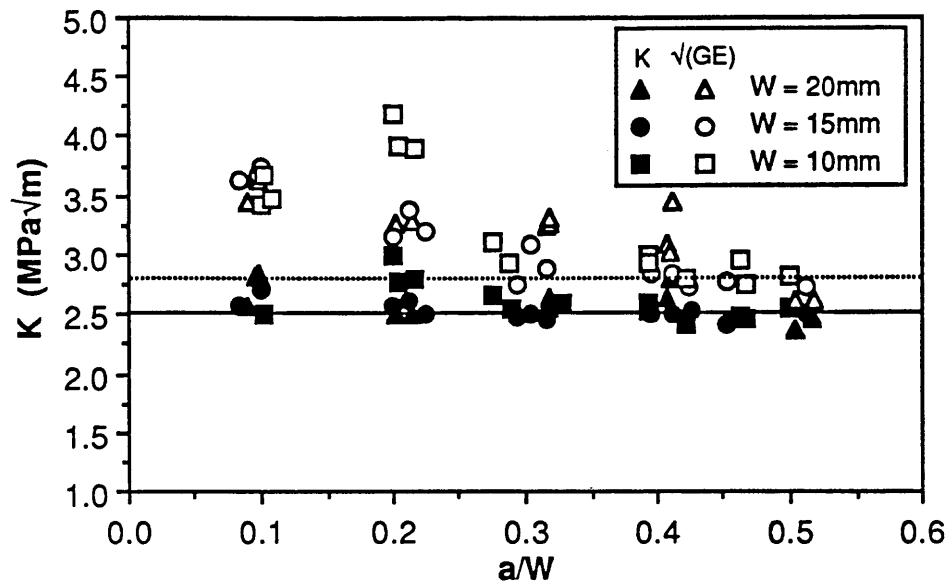
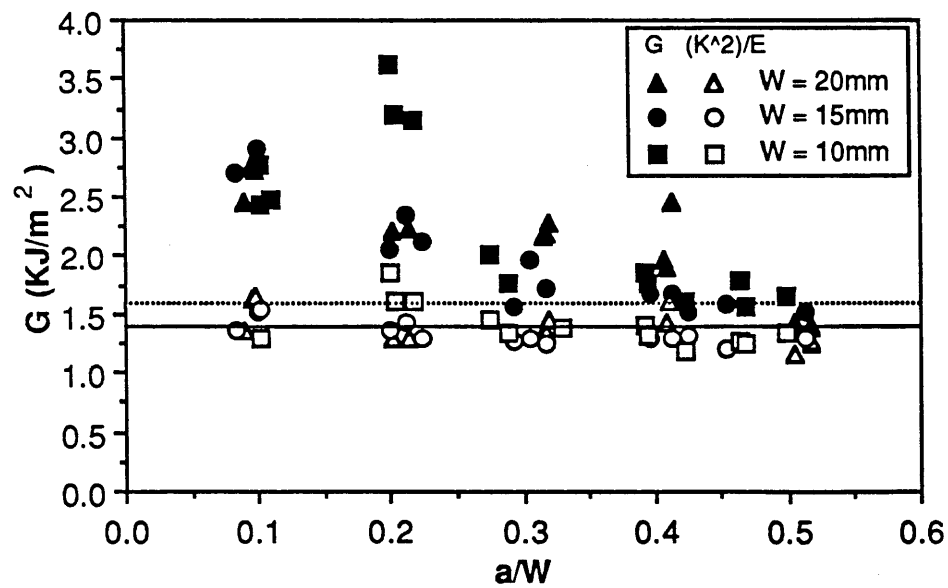
K_{IC} and G_{IC} for specimens with short initial cracks tend to be higher. It is evident from Figures 2.28 and 2.29 that K_{IC} and G_{IC} increase for the samples with cracks shorter than 4 mm. This is larger than the minimum required crack length calculated

according to the ASTM size criteria. Since the constant of proportionality of 2.5 in Equation (2.19) has been determined empirically for steels, it may not be appropriate for the polyethylene studied here. The results show that the constant should be about 4.0 in this case. Results from Mandell et al.[1983] also demonstrate that a constant of proportionality of 3.5 is more appropriate for polyethylene. This implies that the K tests at room temperature fail the size requirement, since the minimum required thickness is marginally larger than that of the specimens. However, tests on specimens of various sizes will be required in order to confirm the same constant of proportionality for other specimen dimensions, and the value of the constant may also depend on the test temperature.

It can be seen from Figs.2.28 and 2.29 that the degree of variation in G_{IC} with a is much larger than that in K_{IC} . The specimens with short cracks can give a two fold increase in the measured G_{IC} value, but the difference in the K_{IC} is only about 20%. It is suggested that the crack was too close to the edge of the sample and the crack blunted before fracture. Examination of the load-displacement diagrams of the short cracked samples reveals that there was a small amount of non-linearity prior to the maximum load although the diagrams were essentially linear. The same was not noticed for the samples with deep cracks. This non-linearity is believed to be caused by plastic deformation in the uncracked ligament. By considering the plastic collapse load of a beam, an approximation of the relationship between the net section stress at plastic collapse, σ_p , and the yield stress σ_y can be written as (Williams[1984]):

$$\frac{\sigma_p}{\sigma_y} = \frac{3}{2} \left[1 - \frac{a}{W} \right]^2 \quad (2.29)$$

According to Equation (2.29), the gross stress will be larger than the yield stress when a/W is less than 0.18. Therefore it is recommended that the crack size should be larger than $0.2W$ if SENB specimens are used.

Fig.2.30 Comparison of the measured and calculated K_{IC} values for 8621Fig.2.31 Comparison of the measured and calculated G_{IC} values for 8621

The values of K_{IC} calculated from the corresponding G_{IC} values and vice versa are illustrated in Fig.2.30 and 2.31, respectively. The results shows that the G approach gives a higher fracture toughness in both cases but the two parameters are

consistent at $a/W=0.5$. The rise in G_{IC} for short crack specimens may be because of the plastic deformation caused by the high applied stress. The EGF protocol recommends an a/W ratio of 0.5 which gives good agreement between the measured K_{IC} and G_{IC} values.

2.8.3 K_{IC} and G_{IC} as Fracture Parameters for Polymers

It has been demonstrated that K_{IC} and G_{IC} are interchangeable when all the constraints on the test are satisfied. However, the G_{IC} approach has several drawbacks when it is applied to polymers. The calculation of G_{IC} requires the evaluation of the energy to fracture which limits its use on fracture toughness characterisation to brittle polymers only. The fracture initiation point for tough polymers is seldom well defined and it is difficult to partition the energy for fracture from the total energy input. The $P_{5\%}$ and P_{max} suggested in the EGF protocol offers a guide-line for the definition of the initiation point but this is an arbitrary rule. This may give a possible source of error in energy estimation. This error cannot be quantified since it depends upon the shape of the load-displacement curve. In addition, high G_{IC} values obtained from the short crack samples which exhibit fairly linear load-displacement behaviour makes fracture characterisation using the G approach alone unattractive. On the other hand, the K_{IC} approach seems to be quite reliable in the determination of fracture toughness for polymers. Since it is determined from load, unlike the G_{IC} method, if the initiation load is within the bounds of $P_{5\%}$ and P_{max} , the maximum error in determination of K_{IC} is 10%. Provided that the specimen is thick enough to ensure a sufficient degree of plane strain, and plastic deformation is not severe, the K_Q calculated from these loads should not differ much from the true K_{IC} value since most polymers show little strain hardening.

Although in theory these two approaches should give identical fracture toughness values, the K approach seems to be more suitable for tough polymers. Nevertheless,

the G method still provides a useful tool for analysing the fracture process in more brittle polymers.

2.9 Conclusions

The σ_y and E of the two HDPEs were found to be temperature and strain rate dependent. The strain rate dependence could be described by a power law, but a weak dependence was observed at high strain rate. The fracture toughness of the 5731 HDPE could not be characterised using the LEFM approach due to limits on the size of specimens that could be used. The K_{IC} and G_{IC} values of the 8621 HDPE were measured and were shown to be compatible when all the test constraints were satisfied. For the G_{IC} tests, the linearity criterion must be rigorously followed. The K_{IC} and G_{IC} were also found to be dependent on temperature and strain rate and the fracture process followed a constant crack opening displacement criterion at low strain rates. K_{IC} and G_{IC} values were found to be constant for deeply notched SEN bend samples. The minimum required crack size determined according to the ASTM size criteria appeared to be too small to give constant K_{IC} and G_{IC} values for the 8621 polyethylene. The constant of proportionality for minimum required crack length was found to be 4.0 in this case. The K approach is suggested to be more suitable than the G approach for fracture characterisation of tough polymers because of the difficulty in evaluating the fracture energy accurately.

Plane Strain Fracture Toughness Study : Elastic-Plastic Fracture Mechanics Approach

3.1 Introduction

For cracked bodies with small scale yielding at the crack tip and fracture under elastic conditions, Linear Elastic Fracture Mechanics (LEFM) provides a single parameter failure criterion for this class of problems. Although LEFM can be modified by adding a plastic zone correction to deal with the limited plasticity at the crack tip, the size of the crack tip plastic region must still be at least an order of magnitude smaller than the physical dimensions of the specimen. A failure criterion which can describe the fracture behaviour involving both small and large scale plasticity is therefore desirable. This leads to the concept of Elastic-Plastic Fracture Mechanics (EPFM). EPFM is an extension of LEFM beyond the elastic regime, the two concepts are notionally similar in providing a single fracture parameter to characterise the critical condition at which fracture will initiate, but EPFM is expected to cope in the presence of plasticity. The two parameters which are widely adopted are *crack (tip) opening displacement COD* or *CTOD* and the *J contour integral*. The *COD* is a measure of crack tip deformation and the *J* integral describes the crack tip elastic-plastic field. Both approaches assume that the parameter will reach a critical value at the onset of fracture for a particular material. These two concepts are closely related but the analysis and application are different in many

ways. The subject was discussed in detail by Turner[1984], Williams[1984] and Atkins et al.[1985]. In this study, the work here is mainly based on the J integral approach, however, the COD criterion will also be covered briefly since these two parameters form the main stream of EPFM.

3.2 Crack Tip Opening Displacement δ

The concept of using the crack opening displacement as a fracture criterion was first proposed independently by Wells[1961] and Cottrell[1961]. They suggested that fracture would occur when the crack opening displacement δ attained a critical value δ_c even in the presence of large scale yielding at the crack tip. The advantage of the method is that the δ_c measured from a small specimen which shows considerable plastic deformation in the laboratory can be very useful in predicting failure in a large structure which is made of the same material. An analytical model proposed by Dugdale[1960] which accounted for the crack tip plasticity laid down the basis for the determination of δ . The model is also known as the strip yield or line zone model.

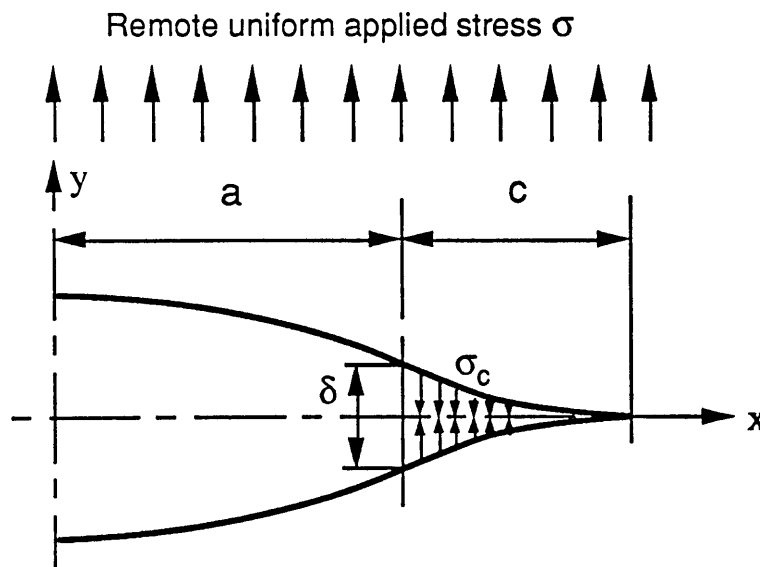


Fig.3.1 The Dugdale or line zone model of crack tip plasticity

It is considered that an infinite plate with a crack of length $2a$ should be loaded under a remote stress σ . The real crack is assumed to be extended by a plastic zone of length c at each end with a uniform cohesive stress σ_c acting over the zone as shown in Fig.3.1.

In this case the stress at the crack tip is limited by σ_c and the singularity could be removed if the size of the line zone c is:

$$c = a \left[\sec \left(\frac{\pi \sigma}{2 \sigma_c} \right) - 1 \right] \quad (3.1)$$

Buredekin and Stone[1966] derived an expression for δ of the Dugdale model as:

$$\delta_c = \frac{8 \sigma_c a}{\pi E} \ln \left(\sec \frac{\pi \sigma}{2 \sigma_c} \right) \quad (3.2)$$

where E is the Young's modulus. For a small increment of crack growth, Wells also suggested that in the elastic case, δ can be related to the energy release rate G as:

$$G = \sigma_y \delta_c \quad (3.3)$$

where σ_y is the yield stress. By expanding the $\ln$ term in Equation 3.2 and retaining the first term, the following expression is obtained:

$$\delta_c = \frac{\pi \sigma^2 a}{E \sigma_c} = \frac{K^2}{E \sigma_c} = \frac{G}{\sigma_c} \quad (3.4)$$

Equations (3.3) and (3.4) are identical if σ_c is replaced by σ_y . For small scale yielding, $\sigma/\sigma_y \ll 1$, by expanding the $\sec$ term in Equation 3.1, it can be reduced to

$$c = \frac{\pi}{8} \left(\frac{K_I}{\sigma_y} \right)^2 \quad (3.5)$$

The size of the plastic zone calculated according to the Dugdale model is larger than that given by Irwin, cf. Equation (2.13). It must be noted that the Dugdale analysis has been derived for the state of plane stress. For the plane strain case, it was suggested by Wells[1968] that σ_c or σ_y should be replaced by a constrained yield stress:

$$\sigma_c = M\sigma_y \quad (3.6)$$

where M is the plastic constraint factor, see Section 2.3.2. The *COD* approach has been widely used in Europe and Japan, but its application will not be pursued here. An excellent review on this subject has been given by Turner[1984].

3.3 The J Contour Integral

The J integral was originally proposed by Rice[1968] as an energy line integral around the crack tip to describe the near tip stress-strain field of an elastic (linear or nonlinear) body as shown in Fig.3.2. It is defined for two dimensional problems and the equation for the line integral is given as:

$$J = \int_{\Gamma} \Omega dy - T \left[\frac{\partial u}{\partial x} \right] ds \quad (3.7)$$

where Ω is the strain energy defined as:

$$\Omega = \int \sigma_{ij} d\epsilon_{ij} \quad (3.8)$$

σ_{ij} and ϵ_{ij} are the stress and strain tensors respectively. T is the traction vector defined by the outward bound vector n along Γ , s is the arc length along Γ and u is the displacement vector.

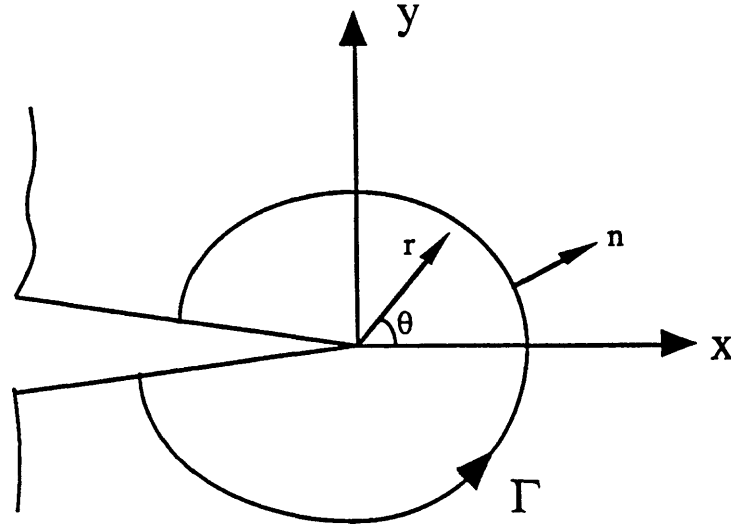


Fig.3.2 The crack tip coordinate system and the arbitrary contour for evaluation of J

Rice has also shown that the J integral is path independent; hence it offers the advantage of characterizing the near tip stress-strain field by evaluating the integral with the path far from the crack tip. It has also been pointed out by Rice that, for a nonlinear elastic material:

$$J = \frac{-1}{B} \frac{\partial U_p}{\partial a} \quad (3.9)$$

where U_p is the potential energy, B is the thickness of the body and a is the crack length. Considering a cracked body with a crack length a , the crack extends from a to $a + \Delta a$ during loading and the load deflection curve is illustrated in Fig.3.3. For loading imposed by a constant displacement v , Equation (3.9) becomes:

$$J = \left. \frac{-1}{B} \frac{\partial U}{\partial a} \right|_v \quad (3.10)$$

U is the strain energy and the change in potential energy is same as the change in strain energy. For the fixed load case, the change in potential energy is equal to the change in complimentary energy U_{co} , so that:

$$J = \frac{1}{B} \frac{\partial U_{co}}{\partial a} \bigg|_P \quad (3.11)$$

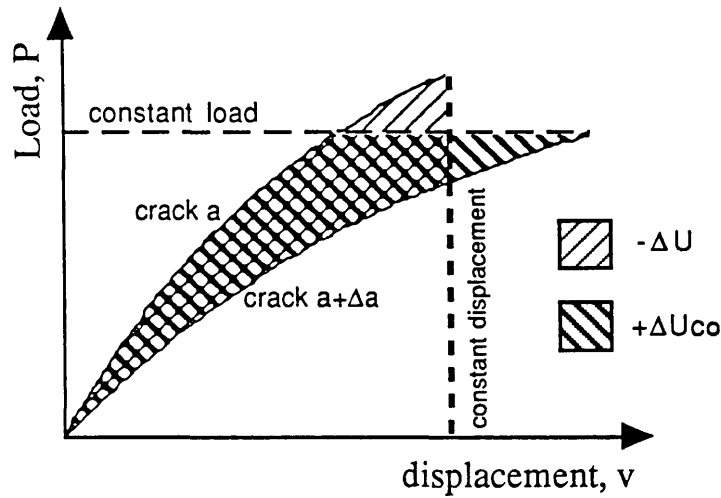


Fig.3.3 Load deflection diagrams for J evaluated at constant load or constant displacement

From Fig.3.3, it is obvious that for the elastic case, the elastic value of J , J_e , is equal to G . Rice also showed that for small scale yielding:

$$J_e = \frac{K^2}{E''} = G \quad (3.12)$$

$E'' = E/(1-\nu^2)$ for the plane strain case and $E'' = E$ for the plane stress case where ν is the Poisson's ratio.

The solutions for the stress and strain singularities at the crack tip have been given by Hutchinson [1968] and Rice & Rosengren [1968], and are known as the HRR

solutions. They assumed that the material should be non-linear elastic and hardened according to power law:

$$\frac{\varepsilon}{\varepsilon_y} = \alpha \left(\frac{\sigma}{\sigma_y} \right)^N \quad (3.13)$$

N and α are constants, and ε_y and σ_y are the yield strain and yield stress, respectively. By using the deformation theory, it was shown that the J integral could be used to characterise stress and strain singularities. The HRR solutions were later rewritten by McClintock[1971] in the form:

$$\frac{\sigma_{ij}}{\sigma_y}(r, \theta) = \left(\frac{JE}{I\alpha\sigma_y^2 a} \right)^{1/(N+1)} \frac{1}{(r/a)^{1/(N+1)}} \sigma_{ij}(\theta) \quad (3.14a)$$

$$\frac{\varepsilon_{ij}}{\varepsilon_y}(r, \theta) = \alpha \left(\frac{JE}{I\alpha\sigma_y^2 a} \right)^{N/(N+1)} \frac{1}{(r/a)^{N/(N+1)}} \sigma_{ij}(\theta) \quad (3.14b)$$

where I is a function of N and is dimensionless. For the linear elastic case with $N=1$, Equation (3.12) can be obtained:

$$(JE)^{1/2} = (GE)^{1/2} = K \quad (\text{c.f. 3.12})$$

It is also evident that the singularity of energy density, $\sigma\varepsilon$, is r^{-1} . Since the deformation theory of plasticity is mainly a nonlinear elastic theory, unloading either by reduction of applied load or crack extension is not permitted. Hence, in principle J should only be applicable up to the point of crack initiation. There are generally two aspects of J . J can be viewed as i) a measure of the intensity of surrounding stationary crack tip stress fields or ii) an energy interchange parameter at the crack tip. The former implies that equal J values mean identical stress and strain field at the crack tip for a given material and the latter can be interpreted as the energy available to increase the crack surface area.

3.3.1 J as a Characterising Parameter for Fracture

In order to establish J as a crack tip parameter, it is important to verify the path independence of the integral. Many computations for J with the aid of finite element methods have been achieved by using the incremental laws of plasticity. Hayes[1970] found that values of J were constant for different paths except those near the crack tip. Path independence was also confirmed by Sumpter[1973] and McMeeking[1977]. In the presence of plasticity and in the elastic-plastic regime, Paris[1977] showed that the role of J as a characterising parameter was maintained provided that gross yielding of specimen did not occur. This view was supported by the results from Sumpter and Turner[1976, i]. It was argued that the J description was acceptable if the fracture process zone was encompassed by the HRR field which resembled the K dominance in the LEFM case (Atkins et al.[1985], Turner[1984]) and a highly constrained specimen, such as deep notch bending, would be necessary.

In the presence of plasticity, it is apparent that J loses its meaning as the potential energy available to propagate the crack. Some of the energy input into the system will be dissipated in the plastic deformation at the crack tip. Landes et al.[1972] pointed out that the results of growing a crack under loading might not be the same as initially extending the crack and then reloading, since plastic deformation is irreversible. Therefore J as the energy release rate is not justified in the elastic-plastic regime. Hence, Equation (3.12) is only valid for linear or nonlinear elasticity. However, J can be regarded as the total energy input to the body or the overall resistance. J can be partitioned into two terms as (Sumpter et al.[1976, ii]):

$$J = J_e + J_{pl} \quad (3.15)$$

where J_e and J_{pl} are the elastic and plastic components, respectively. J_e is notionally equivalent to G , and J_{pl} accounts for the work done due to plasticity. It appears that the application of J is restricted to the analysis of a stationary crack, but Hutchinson et al.[1979] indicated that J could be used to study crack growth and stability problems provided that the '*J controlled growth*' conditions are achieved. In essence, the amount of crack growth is limited by the size of the crack tip singularity field, and Shih et al.[1979] showed that J remained path independent for a crack extension of 6% of the uncracked ligament. Thus J can be regarded as a measure of fracture resistance of materials. It is common practice to present the results in the form of an *R*-curve analysis in which the resistance, J in this case, is plotted against crack extension Δa . J can be related to the *COD* via Equations (3.3) and (3.6). By combining the two, it can be written as:

$$G = M\sigma_y\delta \quad (3.16)$$

it can also be generalised to:

$$J = M\sigma_y\delta \quad (3.17)$$

J will normally be greater than G due to plasticity and the value of M will depend on the geometry of the specimen and the degree of constraint at the crack tip.

3.3.2 Limitations of the J_{IC} Approach

There are several limitations of the J approach according to the analytical basis. They are summarised as follows:

- 1) The assumptions of deformation theory will restrict the use of J to monotonic loading and no subcritical crack growth before fracture is allowed since unloading is not permitted.

- 2) The analysis is basically a two dimensional analysis, it is limited to the plane strain or general plane stress cases.
- 3) The J integral as a fracture criteria is limited to the case of mode I (crack opening mode) and plane strain. Failure in plane stress is normally accompanied by some subcritical crack growth.
- 4) It is assumed that all materials show some degree of hardening. For perfectly plastic materials, the size of the HRR field will diminish as strain hardening vanishes, then the strain field of dominant singularity will not be unique.
- 5) The interpretation of J as the energy available for crack extension (Equation 3.12) is only valid for linear or nonlinear elastic materials.

Despite all the limitations imposed on the J analysis, the actual development of J as a fracture criteria is on an experimental basis. Extensive experiments have been carried out and a new set of limitations determined empirically is established which may not strictly adhere to the analytical restrictions.

3.4 Experimental Methods for J_{IC} Determination

3.4.1 Energy Rate Interpretation of J

The experimental procedure for determining J_{IC} was first proposed by Begley and Landes [1972]. The method is based on the energy rate interpretation of J as governed by Equation (3.10) and is illustrated graphically in Fig. 3.4. In this method, several specimens of different crack lengths are prepared and loaded to the start of fracture and beyond. For a given displacement, the energy input to each specimen, which is represented by the area under the load displacement curve, is plotted against crack length. From Equation (3.10), the slopes of these curves should represent the J values. The J values are then plotted as a function of displacement. By observing the critical displacement v_c at fracture initiation, v_c is determined as a

function of crack length. J_{IC} can be found from a plot of J versus v by knowing the critical displacement for a specific crack length.

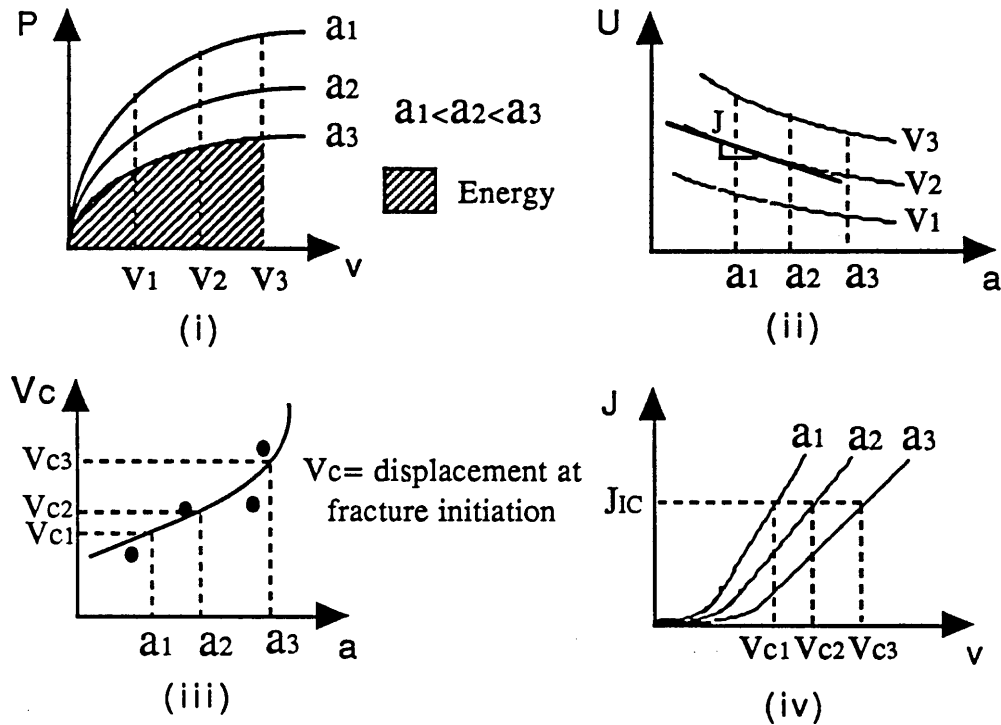


Fig.3.4 Procedures for determining J_{IC} using the energy rate concept

For intermediate to low strength steels, Begley and Landes found that the J_{IC} values for elastic and fully plastic cases were in good agreement with the G_{IC} values obtained from the LEFM tests. In the discussion, Paris (Begley and Landes[1972]) pointed out that J/σ_y is a size parameter which should represent the size of the fracture process zone. In order to maintain the plane strain condition, it was suggested that :

$$B \geq \alpha \frac{J_{IC}}{\sigma_y} \quad (3.18)$$

where α is a constant. Begley et al.[1972] suggested that the value of α should be about 25. Landes and Begley[1972] further investigated the effect of geometry on J_{IC} . They found that there were virtually no geometrical and thickness effects on the

values of J_{IC} for the materials being tested. They also showed that the difference in the slip line fields of the two geometries, compact tension and bending, had no effect on the J_{IC} values due to the dominance of the HRR crack tip field. Bending specimens provide sufficient constraint to achieve nearly plane strain results even in the fully plastic state, and a constant critical displacement is achieved. It was established that J is a successful fracture criterion for both elastic and plastic conditions. However, this method has a number of drawbacks. It requires a number of specimens and tedious calculations are necessary. The main disadvantage is the need to determine the initiation point by observation since all the specimens are loaded beyond initiation, this makes the determination of the initiation point difficult.

3.4.2 The Multiple Specimen Method

Rice et al.[1973] proposed a method for estimating J which formed the backbone of the current methods for laboratory determination of J_{IC} . They suggested that for a deeply cracked specimen under pure bending, J could be represented as a function of the work done on the cracked body:

$$J = \frac{2U_c}{Bb} \quad (3.19)$$

where U_c is the work done during loading in the presence of the crack and b is the remaining ligament. For very deeply cracked specimens, $0.5 \leq a/W \leq 0.75$, Landes et al.[1979] showed that U_c could be replaced by the total energy U_T , which is the total area A under the displacement curve. Therefore Equation (3.19) can be rewritten as:

$$J = \frac{2A}{Bb} \quad (3.20)$$

A more general version of Equation (3.19) was given by Sumpter and Turner[1976, ii] as:

$$J = J_e + J_p = \frac{\eta_e U_e}{Bb} + \frac{\eta_p U_p}{Bb} \quad (3.21)$$

The total energy is divided into elastic U_e and plastic components U_p as illustrated in Fig.3.5. η_e and η_p are geometry dependent constants. For $a/W > 0.5$ and $S/W=4$, $\eta_e \approx \eta_p \approx 2.0$, and Equation (3.21) reduces to Equation (3.20).

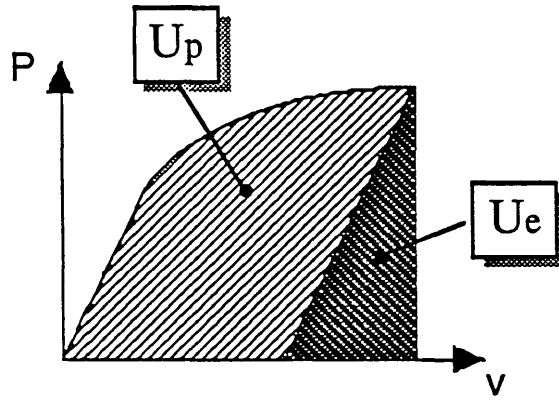


Fig.3.5 Partition of work done

A refined method for J_{IC} determination based on Equation (3.20) was given by Landes and Begley [1974]. This method is now adopted as the standard method for J_{IC} determination by the American Society for Testing and Materials, ASTM, designated ASTM E813. For this method, several identical deeply cracked specimens are loaded to various predetermined displacements to obtain different amounts of crack growth. After unloading, the specimen is broken open and the crack extension Δa is measured. The value of J at the point of unloading for each specimen is calculated using Equation (3.20). The results are presented in the form of an R -curve in which the value of J is plotted as a function of crack extension. In order to account for the apparent crack growth due to blunting before initiation, a *blunting line* is used to determine the J_{IC} value. By assuming smooth blunting at the crack tip, Δa can be approximated as:

$$\Delta a = \frac{\delta}{2} \quad (3.22)$$

where δ is the COD. Thereby Equation (3.17) can be rewritten as:

$$J = 2M\sigma_y\Delta a \quad (3.23)$$

In accordance with ASTM standard E813-81, $M=1$ and the intersection of the blunting line and the R -curve is regarded as the J_{IC} . This method is known as the *multiple specimen method*. The schematic diagram of the test method and illustration of the J - R curve are shown in Figs.3.6 and 3.7, respectively.

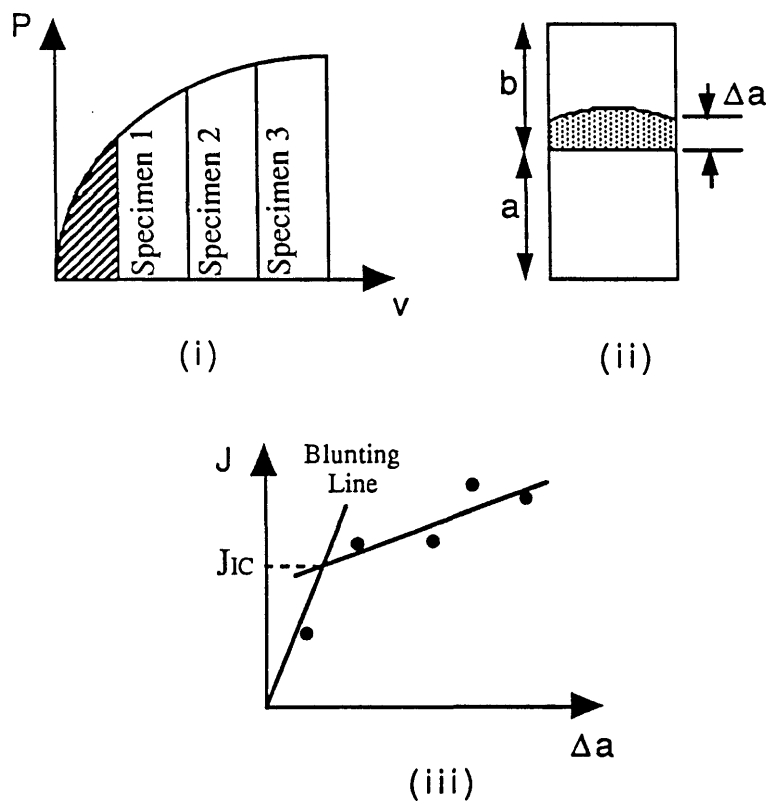
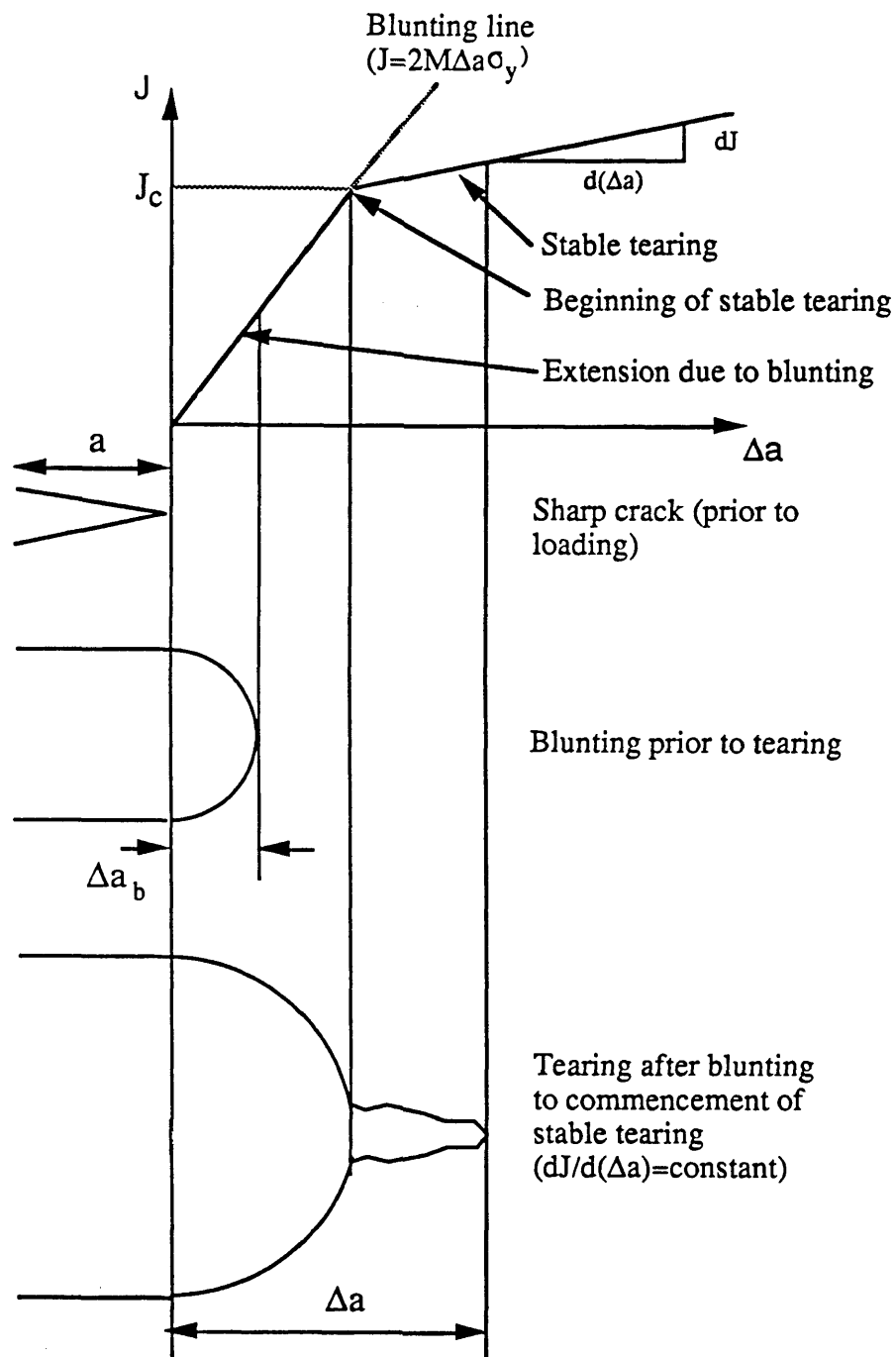


Fig.3.6 Schematic diagram of J_{IC} determination - multiple specimen method

Fig.3.7 Illustration of the J - R curve

3.4.3 The Single Specimen Method

The multiple specimen method is simple but a large number of specimens are required to determine the J_{IC} value. The latest method is an extension of the multiple specimen technique in which the whole J - R curve can be acquired from one specimen and the crack advance is monitored continuously during loading. This method is termed the single specimen method. The unloading compliance method is widely used for monitoring crack advance and this method was first demonstrated by Clarke et al.[1976]. Crack length is estimated from successive partial unloadings during the load history of the test specimen. The method assumes that the unloading process behaves in an elastic manner. For small amounts of unloading, it is assumed that the fracture process will not be affected and compliance changes can reflect the crack

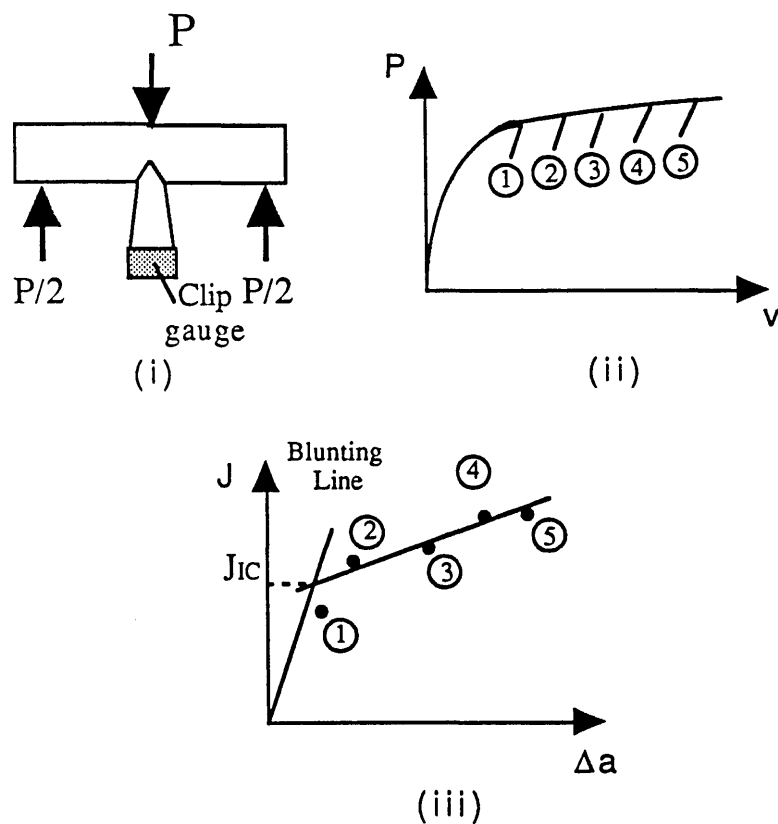


Fig.3.8 Illustration of the unloading compliance method

extension sufficiently accurately. The unloading compliance method is illustrated graphically in Fig.3.8. For deeply cracked three point bend specimens, crack extension is determined from the change of crack mouth opening compliance, and the J values at any unloading point can be evaluated using Equation (3.20).

Since the bend type specimen offers an advantage of high constraint at the crack tip, the plane strain condition can be achieved even if a test specimen of relatively small size is used. Thus three point bend specimens are used throughout this study. Both the unloading compliance and multiple specimen methods are widely accepted as the standard test methods for determination of J_{IC} nowadays.

3.5 Application of J to Polymers

The application and test technique for the J integral method are well established for metals, but the use of the J method is still in its infancy for polymers. Chan and Williams[1983] used J to characterise the plane strain fracture toughness of a tough high density polyethylene (HDPE) at room and sub-zero temperatures in the region where the conventional LEFM test method was inapplicable. The J values evaluated using different methods were compared and they found that the ASTM procedures worked well for the HDPE being tested. Similar results were obtained by Hashemi and Williams[1986,(i)] for the testing of a polypropylene. So et al.[1986] studied the fracture toughness of surface embrittled polymers using the J integral approach and found that the J method was useful in this aspect. Hashemi and Williams[1986,(ii)] applied the technique to measure the fracture toughness of a low density polyethylene and a linear low density polyethylene over a range of temperatures. They found that there was a transition from crack tip blunting to cracking in the materials as temperature decreased. They also indicated that the conventional blunting line given in Equation (3.23), with $M=1$, did not describe the data for the

whole temperature range, and the plastic constraint factor M varied from 3.0 at 20°C to 1.0 at -80°C and below. Hashemi and Williams[1986,(iii)] further investigated the effects of specimen configuration and notch tip radius on the J_{IC} values for various polymers. A constant value of J_{IC} was achieved for different specimen configurations but the shape of the R -curve was altered. It was also found that J_{IC} was insensitive to notch tip radius except for very large radii, where unstable failure would result. A series of J tests were performed by Huang and Williams[1987] on nylon at high loading rates. They demonstrated that the J integral technique was suitable for measuring the fracture toughness of toughened polymers, but an unexpected size effect was observed in which the J_{IC} values decreased with decreasing thickness. This was later proved to be an artefact caused by the test method (Huang [1989]), the data was reanalysed using the new protocol E813-87 and a constant J_{IC} value was obtained. However, there was a significant increase in the J_{IC} values when the data was processed using the new standard and Huang pointed out that it was not clear whether the J_{IC} value could be used as a single parameter for design purposes.

Narisawa[1987] used the J method to measure the fracture toughness of crystalline polymer solids. He showed that J was a useful parameter but the conventional blunting mechanism did not depict the actual deformation process at the crack tip before initiation, and this made the determination of the initiation point uncertain. Due to the ambiguity caused by the blunting mechanisms of polymers, which is always a complicated process and is material dependent, Narisawa et al.[1989] and Takemori et al.[1989] used a different approach in measuring the subcritical crack extension. The test specimen was severed into two half after unloading. One half was polished to produce a side view of the crack near the mid-plane of the sample, the crack tip and the associated damage zone ahead could be observed. The other half was broken open for fracture surface observation and crack growth, Δa , measurements. They claimed that the former method should give a more accurate

indication of the fracture initiation than the latter. They also noted that the fracture surface measurements were often ambiguous and the use of the conventional blunting line would sometimes highly overestimate the J_{IC} value. Jones et al.[1989] investigated the effect of notching methods and the loading displacement rate on J_{IC} of a HDPE. They showed that fatigue precracking would give a lower J_{IC} value than razor notching, and the shape of the R -curve was influenced by the loading rate. They proposed that the drop in J_{IC} was due to the presence of a pre-formed process zone in the fatigued specimen.

The multiple specimen method was used in all the studies discussed above and the test methods employed were based mainly on the original ASTM standard test method E813-81, except Huang[1989] who adopted the updated version of it, E813-87. It must be pointed out that the protocol was not strictly followed and various modifications to the standard were made by workers in order to accommodate polymers. However, the multiple specimen method has been proved to be effective and simple for the fracture toughness evaluation of polymers. The unloading compliance method discussed previously was first developed for metals and the technique is well established now, but it has rarely been applied to polymers and the validity of the method is still uncertain.

3.6 Determination of J_{IC} Values Using the E813 Method

Since the J_{IC} value calculated using the original or the updated E813 can differ significantly, it is important to discuss the distinction between the two methods. The updated version E813-87 is different from its predecessor E813-81 in many ways. The major differences are the inclusion of the single specimen method in the new standard, and the procedures for J_{IC} evaluation. The old standard is still used in

many studies of fracture toughness of polymers, but the new protocol has been shown to give more consistent J_{IC} values. In the old standard, the R -curve is represented by a linear regression line, and the point where the regression line meets the blunting line is regarded as the initiation point. A power law fitting to the data is proposed in the new standard, and the J_{IC} value is defined as the intersection of the R -curve and the 0.2 mm offset blunting line as shown in Fig.3.9. The 0.2 mm offset blunting line was originally suggested by Heerens et al.[1988] to account for the difficulties in defining the initiation point, since the transition from blunting to ductile tearing is always obscure. The offset blunting line is proposed as a well defined condition close to initiation of ductile tearing. J_{IC} must be a point on the R -curve and extrapolation is to be avoided. The amount of offset, 0.2 mm in this case, is based on empirical results.

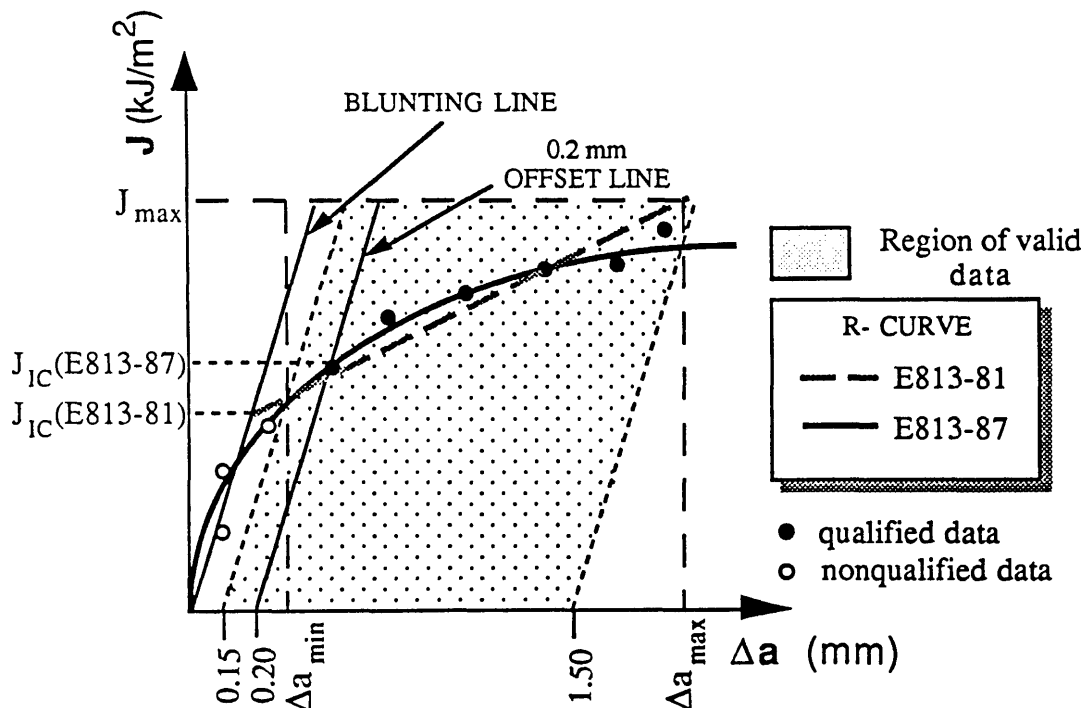


Fig.3.9 Definition of J_{IC} according to the ASTM E813 methods

For both standards, the experimental value of J_{IC} is considered to be valid only if it satisfies the size criteria:

$$B, b > 25 \frac{J_c}{\sigma_y} \quad (3.24)$$

which ensures that the crack growth occurs under plane strain conditions with the appropriate degree of constraint. The data must be enclosed by the area defined by J_{max} , $\Delta a_{(min)}$ and $\Delta a_{(max)}$ as shown in Fig.3.9, where:

$$J_{max} = b \frac{\sigma_y}{15} \quad (3.25)$$

$\Delta a_{(min)}$ and $\Delta a_{(max)}$ are the intersections of the 0.15 offset blunting line and the R -curve and 1.5 offset blunting line and the R -curve respectively. There are limitations on the distribution of data points, there must be at least one point between the 0.15 and 0.5 offset lines and one point between the 1.0 and 1.5 offset lines.

There are also other standard test methods, e.g. the European standard, in which a more sophisticated formulation of the blunting line is used accounting for power law hardening materials, but the test method is in essence the same as the ASTM method.

3.7 Experimental: J_{IC} Testing

3.7.1 Objectives

The J method provides a reduction of specimen size required for obtaining valid plane strain fracture toughness when compared to the K_c tests because the degree of plane strain that ensures the J_{IC} value is independent of component size is much lower than that in the LEFM case. This gives an advantage for testing polymers since

there are considerable difficulties in manufacturing thick sections. The single specimen method provides an alternative to the multiple specimen method, but the applicability of the method to polymers is not certain due to their viscoelastic nature. Here, an attempt is made to determine the J_{IC} value of several polymers using the unloading compliance method. In order to enable a level of confidence to be attached to the method, the results are compared with those obtained by the multiple specimen technique which has been demonstrated to be successful for polymers. The results have been analysed according to the ASTM E813-81 and the E813-87 standards. The advantages and drawbacks of each method are investigated. The effects of specimen size, loading rate and side-grooving on the J_{IC} and the R -curve for the high density polyethylene 8621 have also been examined. For the high density polyethylene 5731, the test temperature was lowered in order to reduce the minimum specimen size required for valid J_{IC} values.

3.7.2 Materials and Specimen Preparation

The two grades of high density polyethylene (HDPE), 8621 and 5731 were tested using the J method. In order to show the validity of the single specimen method on other polymers, a polyvinylidene difluoride (PVDF) and a medium density polyethylene (MDPE) supplied in the form of compression moulded sheets were also tested. The mechanical properties of the materials are shown in Table 3.1.

Supplier	Material	Designation	MFI (g/10min)	Density (kg/m ³)	Yield Stress (MPa)	Modulus (GPa)
DSM	HDPE	8621	0.16	958	27	1.7
DSM	HDPE	5731	0.03	945	23	1.1
Solvay & Cie	PVDF	1010	N/A	N/A	53	2.5
BP Chemicals	MDPE	Rigidex 002-50	N/A	950	17	0.8

Table 3.1 Properties of the materials

Single edge notched specimens were used and notched to a crack to width ratio of 0.5 to 0.6 and the specimen preparation procedures were same as those described in Section 2.7.1 in Chapter 2. For side-grooved samples, side-grooves were machined on both sides of the specimens after the notch had been introduced and the total reduction of the specimen thickness was less than 25% as suggested in the ASTM standard. All the specimens were loaded in three point bending with a span to width ratio of 4 and tests were performed on an Instron testing machine. The nominal strain rate, $\dot{\epsilon}$, of three point bend samples was determined using Equation (2.20).

Material	Width (mm)	Thickness (mm)	Span (mm)	Type of samples	Side Grooves, each side (mm)	a/W	Cross head speed $\dot{X}$ (mm/min)	Test temperature (°C)
HDPE 5731	30	12	120	NS	-	0.6	2	-120 to 20
HDPE 8621	40	12	160	NS,S	0.75	0.6	1	room
	30	12	120	NS,S	0.75	0.6	0.5, 1, 5	room
	25	12	100	NS,S	0.75	0.6	1	room
	20	12	80	NS,S	0.75	0.6	0.5	room
	15	12	60	NS,S	0.75	0.6	0.5	room
	30	5	120	NS,S	0.5	0.6	1	room
	30	7.5	120	NS,S	0.6	0.6	1	room
	30	9	120	NS,S	0.75	0.6	1	room
PVDF	22	10	88	NS	-	0.55	1	room
MDPE	50	25	200	S	1.5	0.6	0.5	room

Table 3.2 Specimen dimensions and loading conditions for J tests
(NS=non side-grooved, S= side-grooved)

The machine was adjusted to the appropriate cross head speed in order to maintain a constant $\dot{\epsilon}$ for specimens of different size. For the 8621 material, the effects of specimen size, loading rate and side-grooving were investigated. The dimensions of the specimens and the loading conditions are summarised in Table 3.2.

3.7.3 Multiple Specimen J Tests

Six or more specimens of each material were prepared and loaded to pre-determined displacements as described in section 3.4.2. The load-line displacement was measured using the transducer which was built into the machine. The load and displacement were registered by a personal computer connected to the testing machine and the area under the load-displacement curve was evaluated. The specimens were then broken open by impact after dipping into liquid nitrogen to reveal the crack extension. The crack size was measured at nine equally spaced points as suggested in the E813 method. The change of morphology, not whitening, on the fracture surface was taken as the extent of crack extension. The corresponding value of J at the unloading point was calculated using Equation (3.20) with the thickness B replaced by the net thickness B_N for side-grooved samples. The HDPE side-grooved specimens were loaded beyond initiation since shear lips and crack front 'bowing' should not occur prior to fracture initiation. It could be assumed that the blunting process was common for both the non side-grooved and side-grooved samples. Therefore, by using the data obtained from the non side-grooved samples, the number of side-grooved specimens required for obtaining a J - R curve could be reduced. Tests performed at sub-zero temperatures were carried out in an insulated box as described in Section 2.7.2.1. The energy was corrected for indentations of the specimen by the support rollers and loading pin. The procedures reported in Section 2.7.2.1 were followed.

3.7.4 Crack Length Prediction Using the Compliance Method

For the unloading compliance method, crack length can be predicted from the crack mouth opening compliance and various expressions relating the compliance and crack length for the three point bend specimen can be found in the literature. The one developed by Bakker[1990], rather than that suggested in the ASTM E813-87, has been used for crack length estimation since the accuracy has been shown to be better. The transfer function δ_{cm} developed by Kapp et al.[1985], for the inverse crack mouth opening compliance function was used by Bakker to calculate the first estimate of crack to width ratio as shown in Equation (3.26) and Equation (3.27).

$$\delta_{cm} = \frac{1}{1 + \sqrt{\frac{3.95S/W}{E'B_e C_{cm}(exp)}}} \quad (3.26)$$

$$\begin{aligned} \frac{a}{W} = & 9.56 \times 10^{-4} + 5.504 \times 10^{-2} \delta_{cm} - 1.0968 \delta_{cm}^2 + 9.9706 \delta_{cm}^3 - \\ & - 13.096 \delta_{cm}^4 + 5.1707 \delta_{cm}^5 \end{aligned} \quad (3.27)$$

$$B_e = B - \frac{(B - B_N)^2}{B_N} \quad (3.28)$$

$C_{cm}(exp)$ is the measured crack mouth opening compliance, B_e is the effective thickness and E' is the effective modulus(see Equation (3.30)). A correction to the first estimate was given by Bakker as shown in Equation (3.29) to improve the accuracy of the crack length prediction:

$$\left(\frac{a}{W} \right)_{corrected} = \frac{a}{W} + \frac{C_{cm}(exp) - C_{cm}(a/W)}{\frac{dC_{cm}(a/W)}{d(a/W)}} \quad (3.29)$$

$C_{cm}(a/W)$ is the value of compliance calculated from the first estimate of a/W using the following:

$$E'BC_{cm}(a/W) = \frac{S}{W} \frac{a/W}{(1-a/W)^2} [(8.737 - 8.681(a/W)^{0.5} + 3.321(a/W)) + 0.573(a/W)^{1.5}] \quad (3.30)$$

In order to account for various uncertainties in testing, an effective modulus E' is evaluated using Equation (3.30) by measuring the compliance of the first few unloadings where no crack extension occurs. The value of E' should not differ from the elastic modulus E by more than 10%.

3.7.5 Single Specimen J Tests

For the single specimen J tests, the load line displacement and the crack mouth displacement were monitored by a linear voltage displacement transducer (LVDT) and a clip gauge, respectively. The load and displacements were recorded on Hewlett Packard X-Y plotters. Knife edges were used to mount the clip gauge in order to obtain friction free seating of the gauge. The layout of the apparatus is shown in Fig.3.10. The energy for computation of J was obtained up to the point of unloading from the load-load line displacement graph, and compliance was measured graphically from the load-clip gauge displacement plot. The J value at the point of the i^{th} unloading is given as:

$$J_i = J_{i-1} + \frac{2U_{i,i-1}}{Bb} \quad (3.31)$$

where $U_{i,i-1}$ denotes the area under the load-load line displacement curve between two consecutive unloading points $i-1$ and i and $B=B_N$ for side-grooved specimens. Equations (3.26), (3.27), (3.29) and (3.30) were used to evaluate the crack length at the point of unloading. During the tests, each specimen was unloaded 12-18 times by

20-30% of the current load. Loading and reloading were carried out continuously at the same cross head speed. The effective modulus was estimated from the compliance measured at between 10% to 30% of the maximum load. For each material, specimen size, and loading rate, at least two replicates were tested, and all the tests were performed at room temperature. Crack extension was measured after the test using the nine point averaging technique, and it was compared with that predicted using the unloading compliance method. The energy was corrected for indentation as described previously.

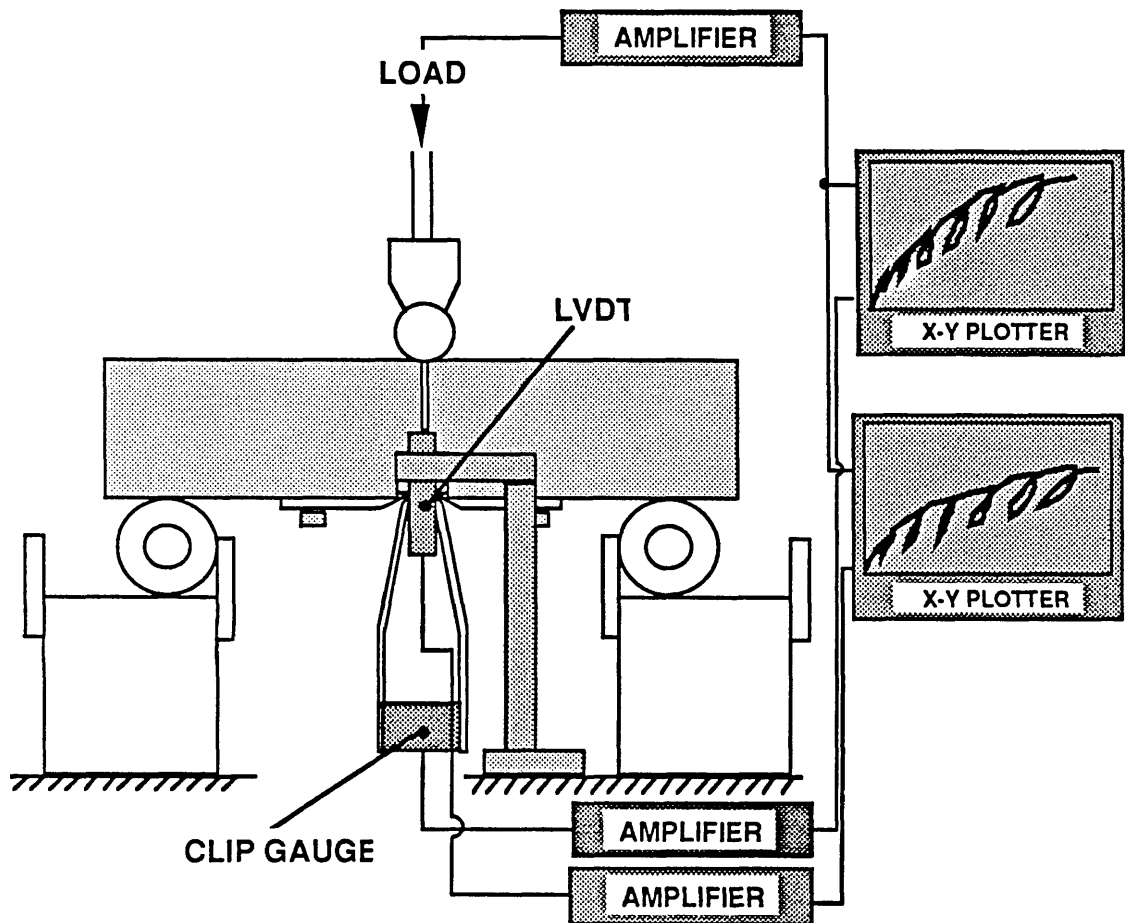


Fig.3.10 The layout of the apparatus for single specimen J tests

3.8 Results

3.8.1 Multiple Specimen J Tests

For the non side-grooved specimens, severe crack front curvature was found on the fracture surface and the crack extension at the centre was normally larger than that at the near surface by more than $\pm 0.02W$. However, straight crack fronts were obtained for side-grooved samples. The errors caused by the deformation of the testing fixture and specimens were found to be small, the maximum error was about 5% of the measured J values. This may be due to the relatively low stiffness of plastics compared to that of the steel testing fixture components.

3.8.1.1 HDPE, 5731

The multiple specimen J tests were performed over a temperature range from 20°C to -120°C. All specimens exhibited stable crack growth over the whole temperature range, except those at room temperature and 0°C, where the crack blunted severely

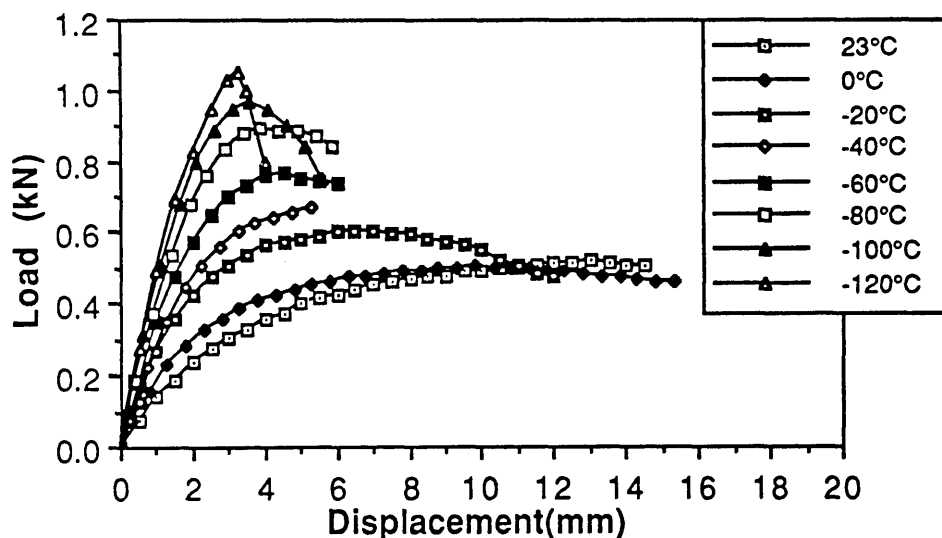
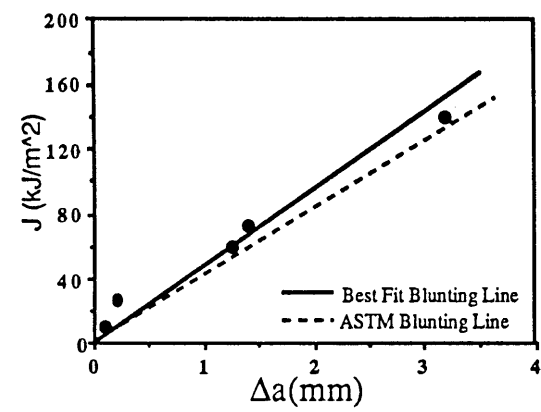
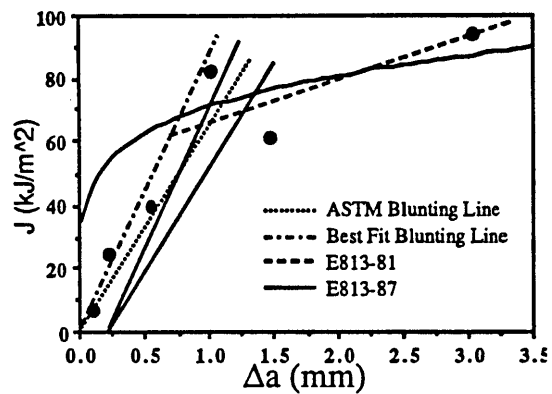
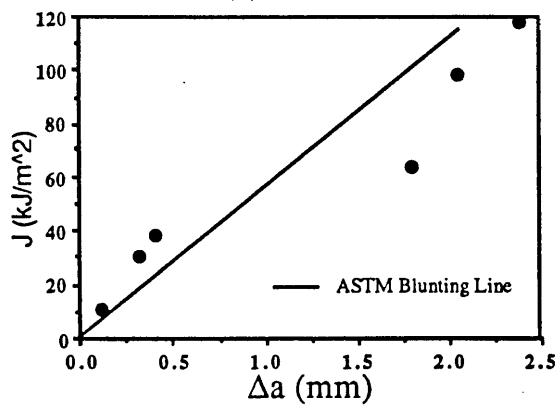
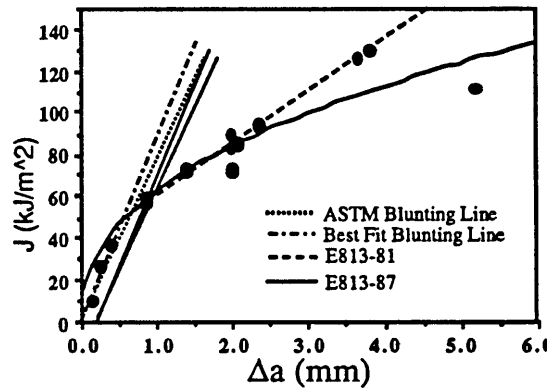


Fig.3.11 Load displacement diagrams for 5731 at various temperatures

and material separation at the crack was not observed. Load-displacement records at different temperatures are shown in Fig.3.11. The shape of the load-displacement curves change significantly as temperature is decreased. All curves are linear up to a certain load beyond which they depart from linearity, but the load continues to rise until a maximum is reached. For tests at temperatures below -80°C , the peak of the curves is well defined.

All fracture surfaces showed characteristic thumb-nail features associated with the ductile tearing failure mode. The J - R curves determined according to the old and new E813 methods at various temperatures are shown in Fig.3.12. By referring to Fig.3.12, the data points obtained at 23°C and 0°C conform to the blunting line, and the J_{IC} values could not be determined. It is also apparent that the conventional blunting line given in Equation (3.23), with $M=1$, does not adequately describe the data points near the blunting region in most cases. As a result, a best straight line through those data was drawn as indicated in Fig.3.12. The J_{IC} values acquired from this experimental blunting line were compared with those evaluated using the conventional blunting line and the results are shown in Fig.3.13 and Table 3.3. It can be seen that the difference in the J_{IC} values obtained from these two types of blunting lines is not dramatic, and that the J_{IC} values increase with increasing temperature. The fracture surfaces were studied using a scanning electron microscope and the surfaces at different temperatures are shown in Fig.3.14. All fracture surfaces exhibited spherulitic features, and severe plastic deformation could be found near the boundaries of the spherulites. It can be observed that the fracture mode changes from ductile to brittle as temperature decreases, and this accounts for the decreasing J_{IC} with decreasing temperature. The J_{IC} values determined according to the new E813 procedures were found to be higher than those obtained from the old method despite the type of blunting line used in the analysis, but the discrepancy diminished as temperature decreased, as illustrated in Fig.3.13. The

minimum required thickness for valid J tests determined via Equation (3.24) at various temperatures is given in Fig.3.15. This shows that all the tests performed at temperatures above -120°C are invalid in accordance with the ASTM size criteria. Since an R -curve and a valid J_{IC} value cannot be achieved at room temperature, the single specimen method could not be applied to this material due to the limit of the working temperature of the transducers used.

(a) 23°C (c) -20°C (b) 0°C (d) -40°C

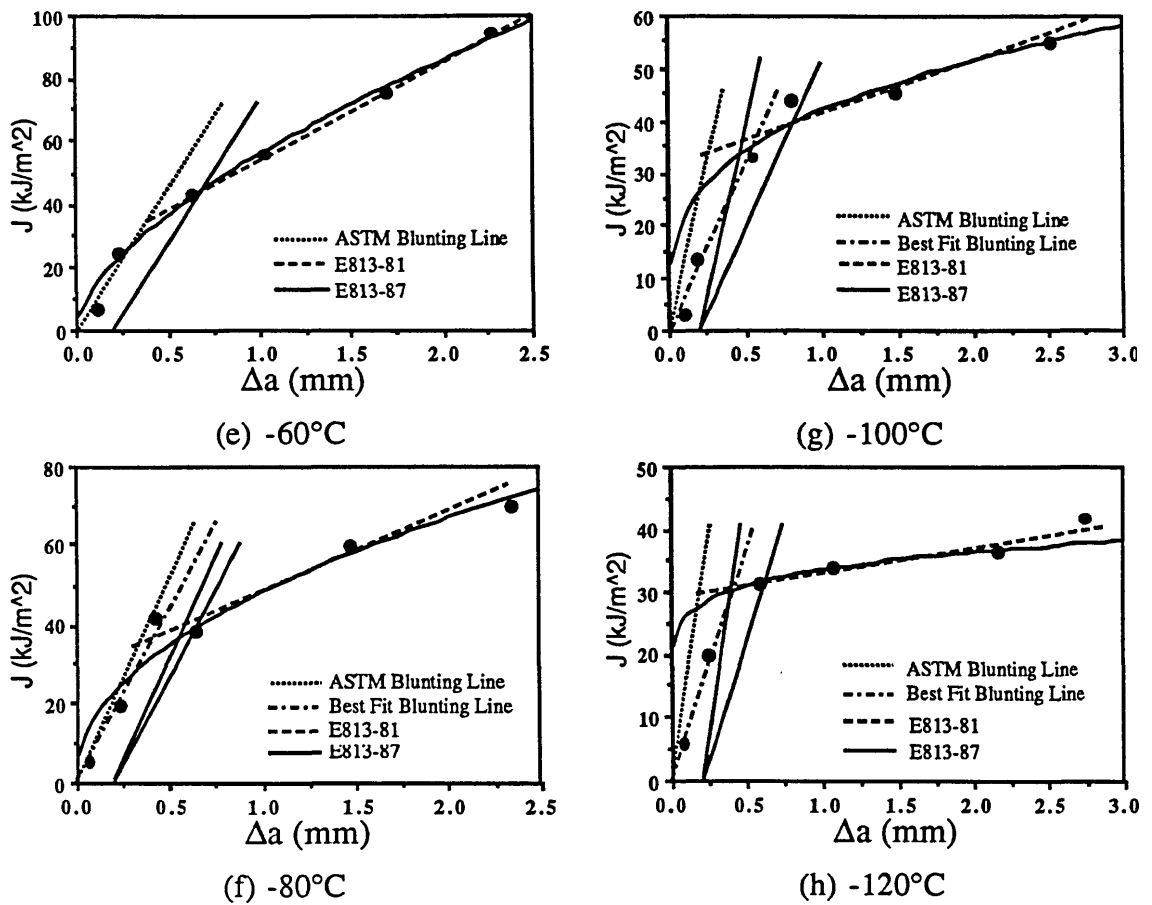


Fig.3.12 J - R curves for 5731 at various test temperatures; (a) 23°C, (b) 0°C, (c) -20°C, (d) -40°C, (e) -60°C, (f) -80°C, (g) -100°C, (h) -120°C

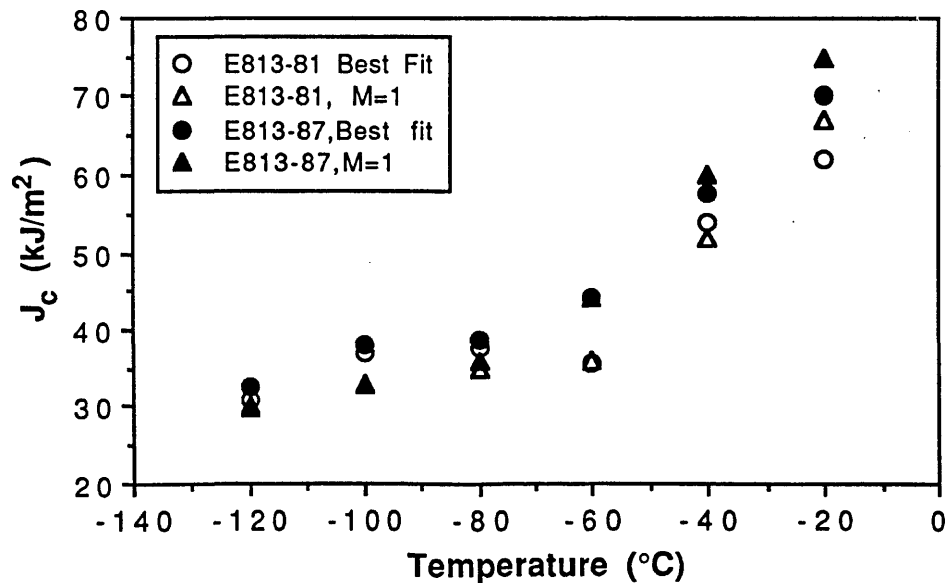
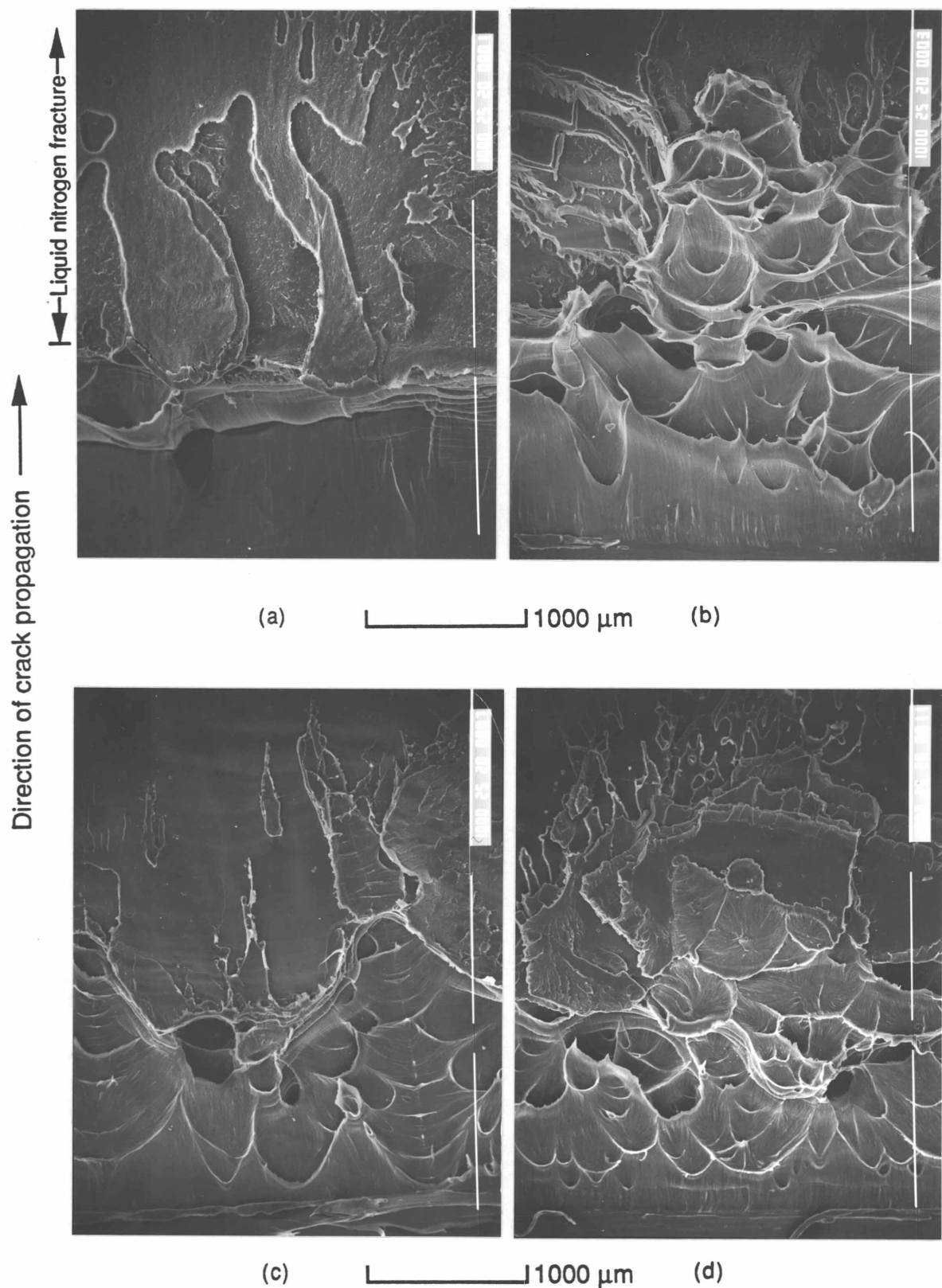


Fig.3.13 Variation of J_c with temperature for HDPE 5731



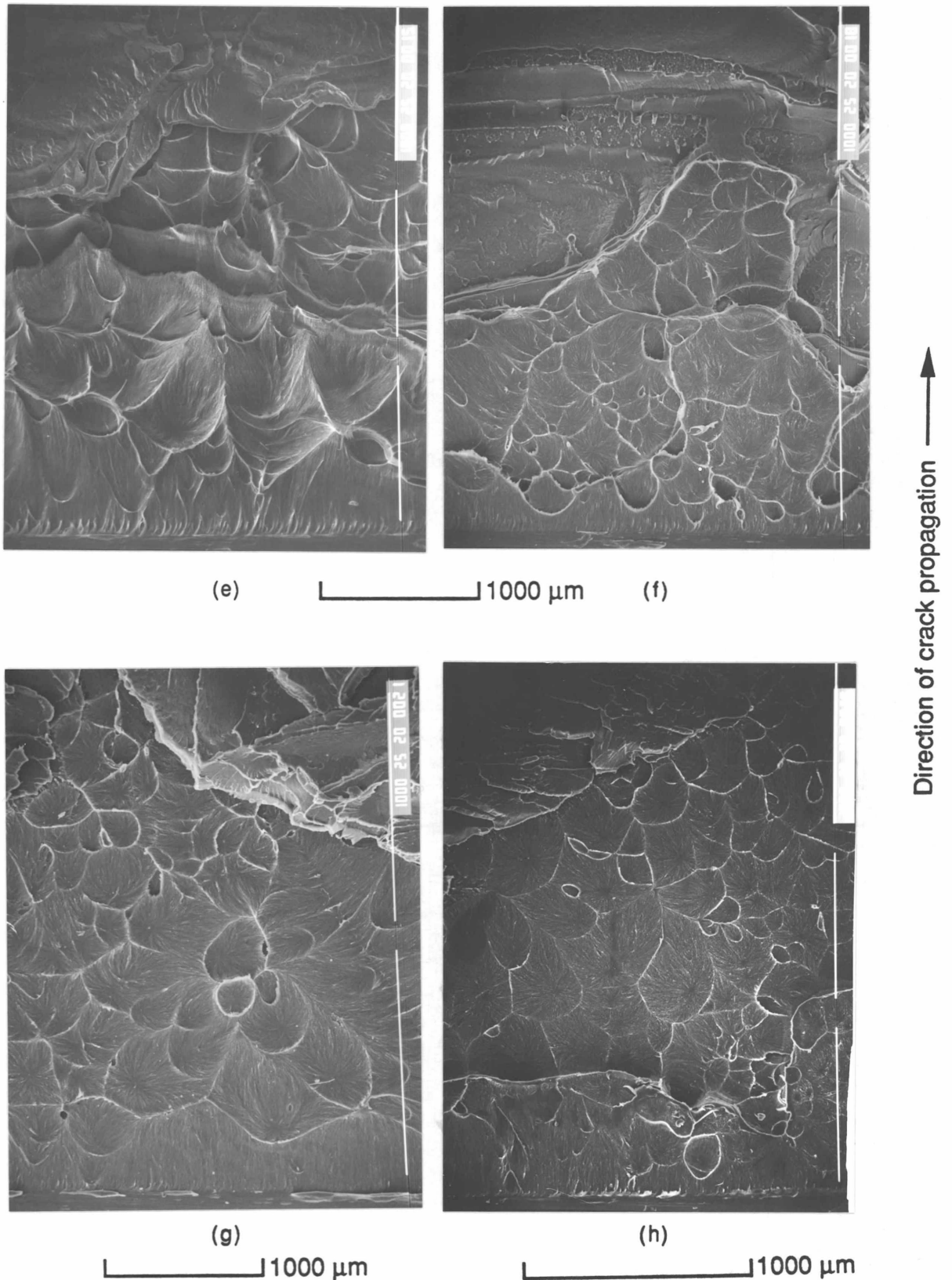


Fig.3.14 Fracture surfaces of HDPE 5731 at various temperatures. (a) 23°C, (b) 0°C, (c) -20°C, (d) -40°C, (e) -60°C, (f) -80°C, (g) -100°C, (h) -120°C

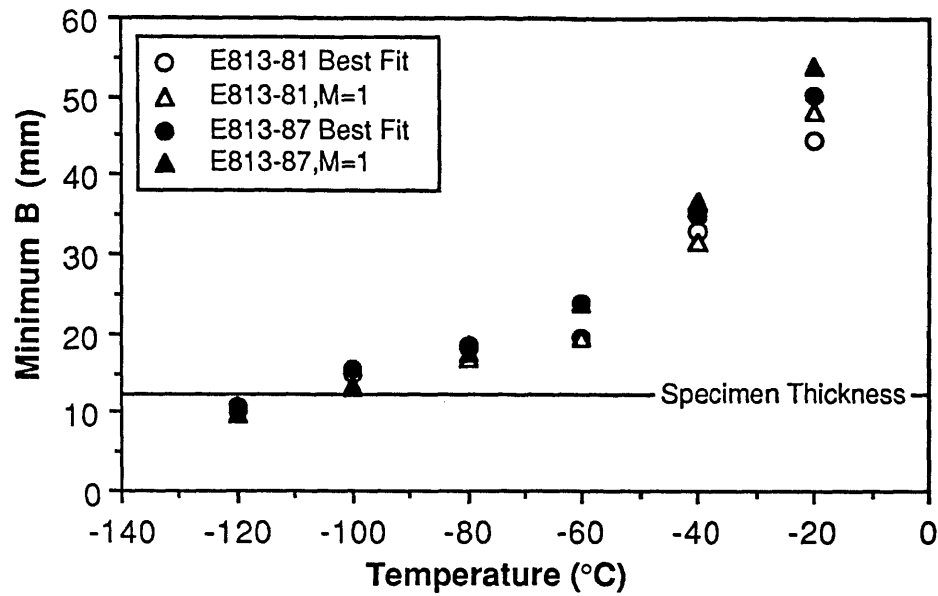


Fig.3.15 Minimum specimen thickness required for valid J tests for HDPE 5731 at various temperatures

Temperature (°C)	J_{IC} (kJ/m ²)			
	E813-81 Method		E813-87 Method	
	Best Fit Blunting Line	ASTM Blunting Line	Best Fit Blunting Line	ASTM Blunting Line
-20	62	67	70	75
-40	53.9	52	57.5	60
-60	35.5	35.5	44	44
-80	37.5	35	38.5	36
-100	37	33	38	32.8
-120	30.9	29.8	32.5	30

Table 3.3 Main results for J tests for HDPE 5731

3.8.1.2 HDPE, 8621

A typical J - R curve for 8621 obtained from the multiple specimen J test is given in Fig.3.16. It can be observed that the J_{IC} value evaluated using the new standard is higher than that acquired from the old standard. The data points near the blunting region again do not follow the conventional blunting line. The best fit blunting line is considered to be more appropriate for J_{IC} determination in this case. The effect of loading rate on the R -curve is illustrated in Fig.3.17 (the construction of the blunting line and R -curve is not shown for clarity). It can be seen that the J_{IC} values do not vary strongly with the loading rate but the R -curves tend to flatten slightly as the loading rate increases. However, due to the scatter of the data, additional tests would be needed in order to define the loading rate effect on the R -curve.

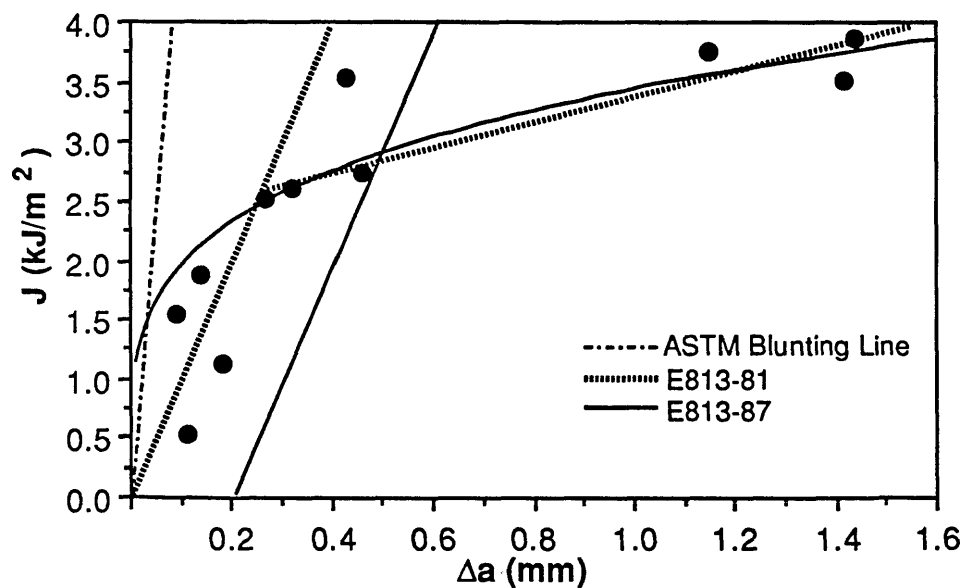


Fig.3.16 J - R curve for HDPE 8621 from multiple specimen J tests with loading rate of 1mm/min, $B=12$ mm and $W=30$ mm

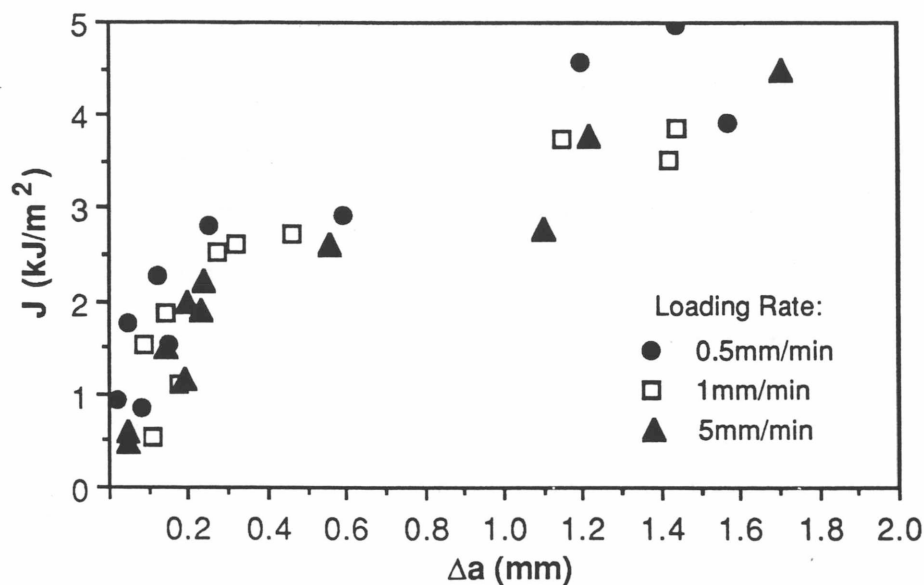


Fig.3.17 J - R curves for HDPE 8621 from multiple specimen J tests at various loading rate

Figures 3.18 and 3.19 show the fracture surfaces of the specimens with various widths and thicknesses. It is apparent that side-grooving reduces the crack front curvature by promoting the triaxiality of the stress at the crack tip, thus reducing the uncertainty in crack extension measurement.

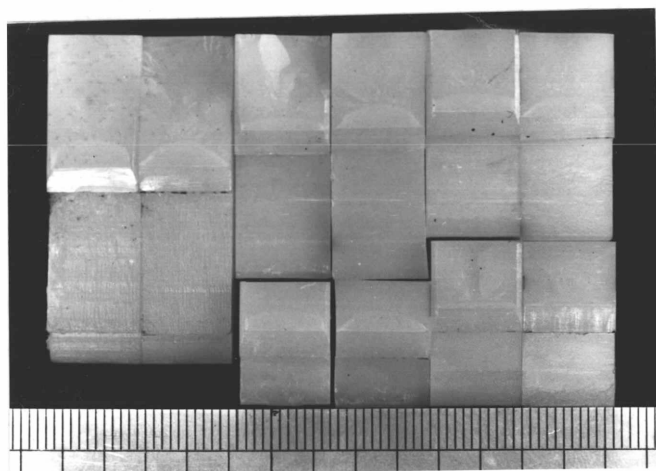


Fig.3.18 Fracture surfaces of 8621 specimens with various widths

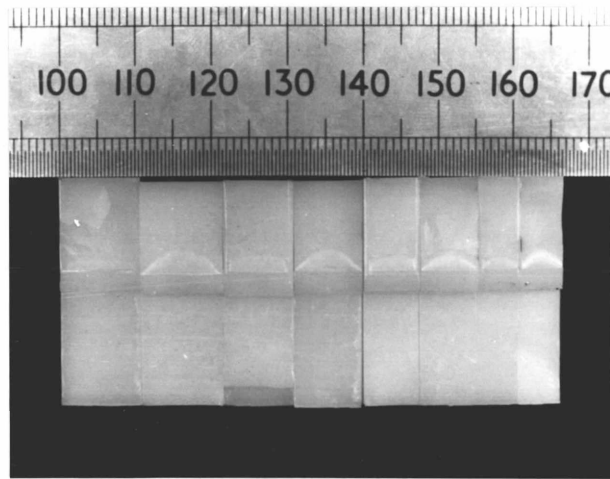


Fig.3.19 Fracture surfaces of 8621 specimens with various thicknesses

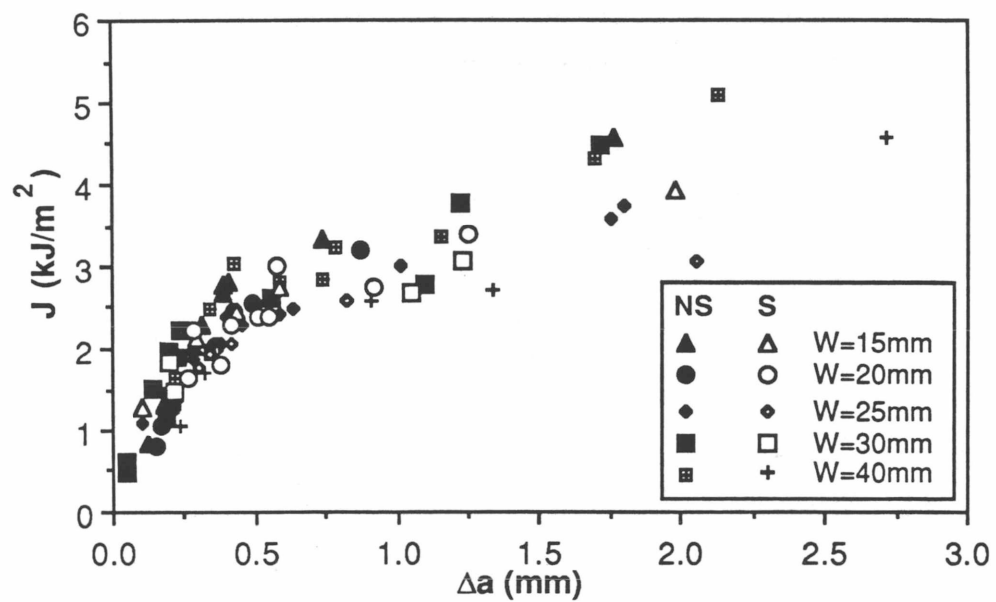


Fig.3.20 Effect of specimen width on the J - R curves obtained from multiple specimen J tests for HDPE 8621 (NS=Non side-grooved specimens, S=Side-grooved specimens)

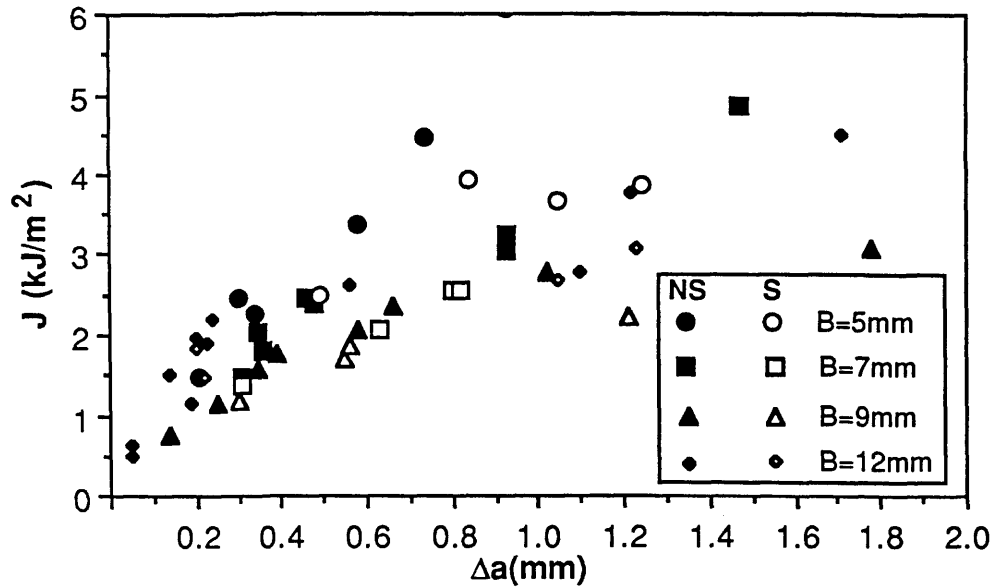


Fig.3.21 Effect of specimen thickness on the J - R curves obtained from multiple specimen J tests for HDPE 8621 (NS=Non side-grooved specimens, S=Side-grooved specimens)

The R -curves for specimens with different widths and thicknesses are shown in Fig.3.20 and Fig.3.21, respectively. From Fig.3.20, it is noted that the specimen width has little effect on the J_{IC} value and a fairly unique R -curve is suggested by the data. The results shown in Fig.3.21 indicate that the slope of the R -curve increases with decreasing specimen thickness but the J_{IC} value does not show a strong dependence on thickness except for the thinnest samples. It can also be seen from Figs. 3.20 and 3.21 that side-grooving tends to reduce the slope of the R -curve, and the thickness effect is less prominent in this case. The data from the side-grooved and non side-grooved specimens agrees well in the blunting region, and the J_{IC} values determined using the side-grooved specimens are also slightly lower than that using the non side-grooved samples. The scatter in the data may be caused by the error in crack length measurement. The results and the coefficients of the power law fitted R -curves are summarised in Table 3.4 and Table 3.5 in Section 3.8.3.2, respectively.

According to Equation (3.24), the minimum required thickness for valid J_{IC} was found to be approximately 2 mm for this material, and all the specimens satisfied this criterion. However, for the 5 mm thick specimens, the R -curve tends to rise steeply. When the crack was grown beyond 0.6 mm, severe plastic deformation was observed during the experiments in which the crack blunted without further crack extension. A stress whitening region at the crack front associated with the blunted crack can be seen in Fig.3.22. Therefore a J_{IC} value could not be evaluated for the 5 mm thick non side-grooved samples and the validity of Equation (3.24) became questionable for this material.

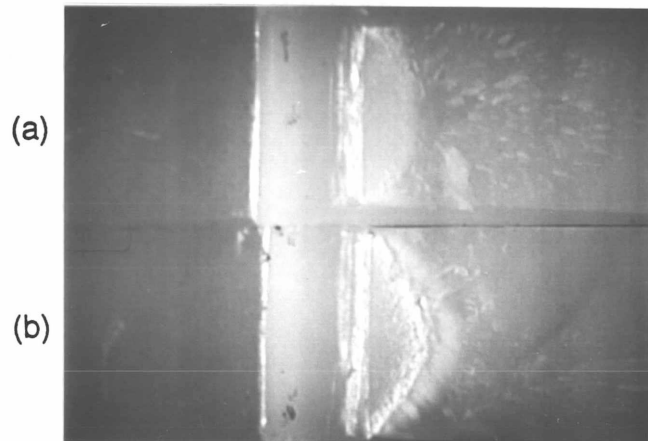


Fig.3.22 Fracture surfaces of 5 mm thick 8621 specimens
a) side-grooved, b) non side-grooved

The results are re-plotted on Figures.3.23 and 3.24 for non side-grooved and side-grooved specimens respectively. Despite the results from the 5 mm samples, it can be observed that side-grooving tends to reduce the amount of scatter in the data.

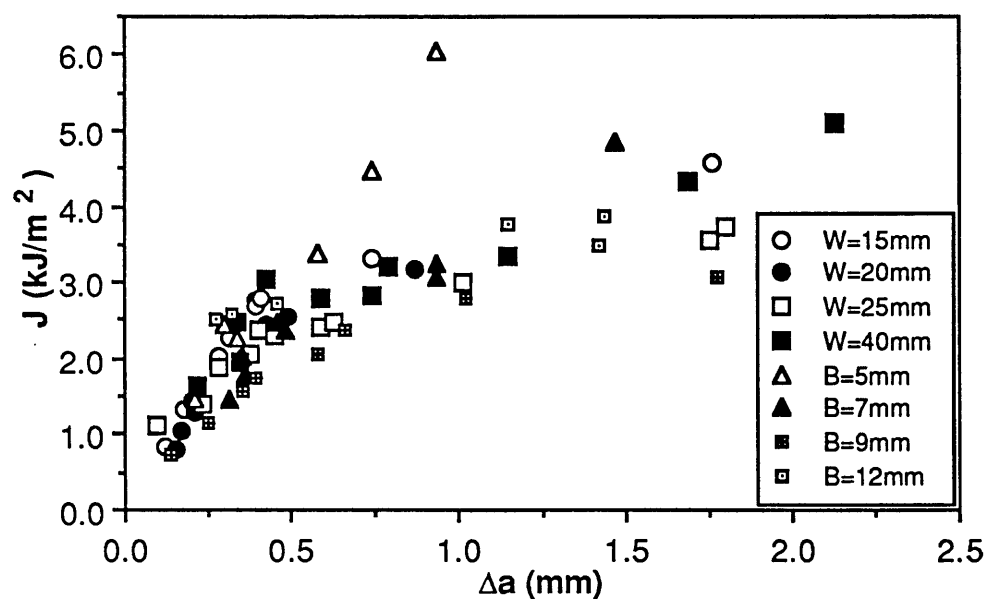


Fig.3.23 Effect of specimen size on the J - R curves obtained from non side-grooved specimens for HDPE 8621

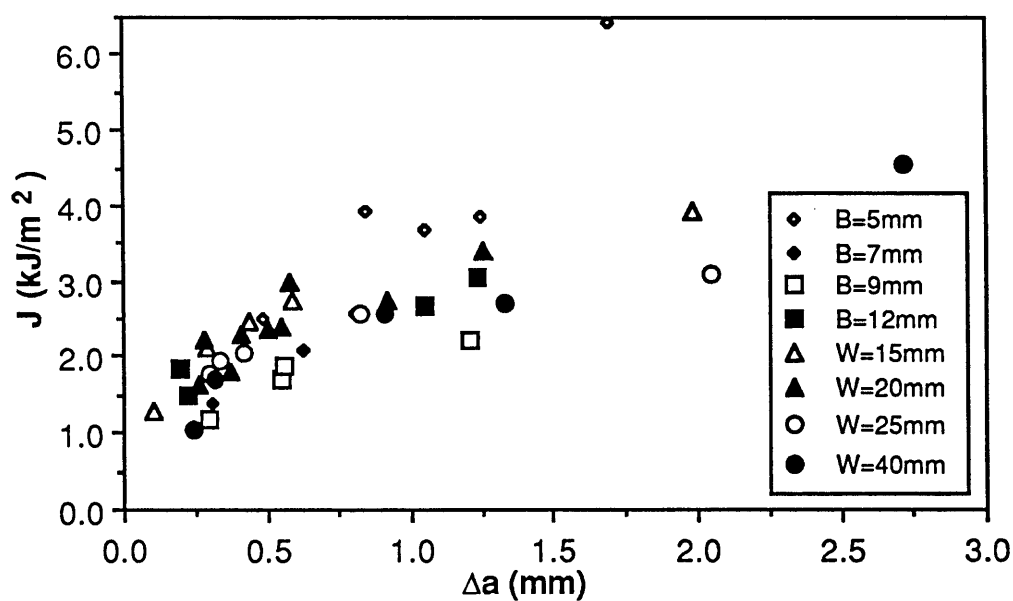


Fig.3.24 Effect of specimen size on the J - R curves obtained from side-grooved specimens for HDPE 8621

3.8.1.3 PVDF and MDPE

The J - R curves for PVDF and MDPE are shown in Fig.3.25 and 3.26 respectively. Higher J_{IC} values evaluated using the E813-87 method are also observed. From the results, the use of the conventional blunting line is again found to be inappropriate, and the experimentally determined blunting lines were used to evaluate the J_{IC} values. The results are shown in Table 3.6 in Section 3.8.3.2. It should be noted that the MDPE specimens did not meet the ASTM size criteria as suggested in Equation (3.24) and the measured J_{IC} value was invalid.

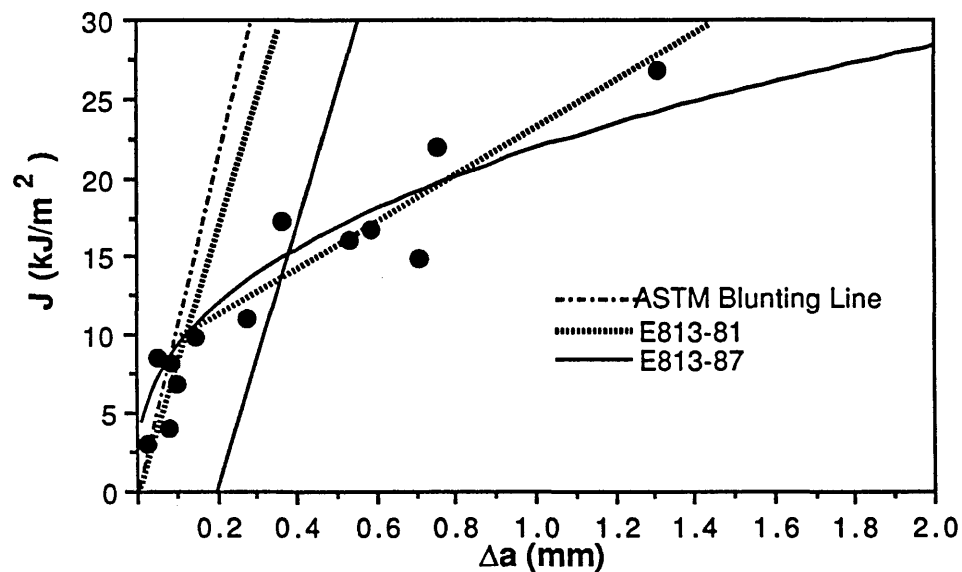


Fig.3.25 J - R curve for PVDF from multiple specimen J tests

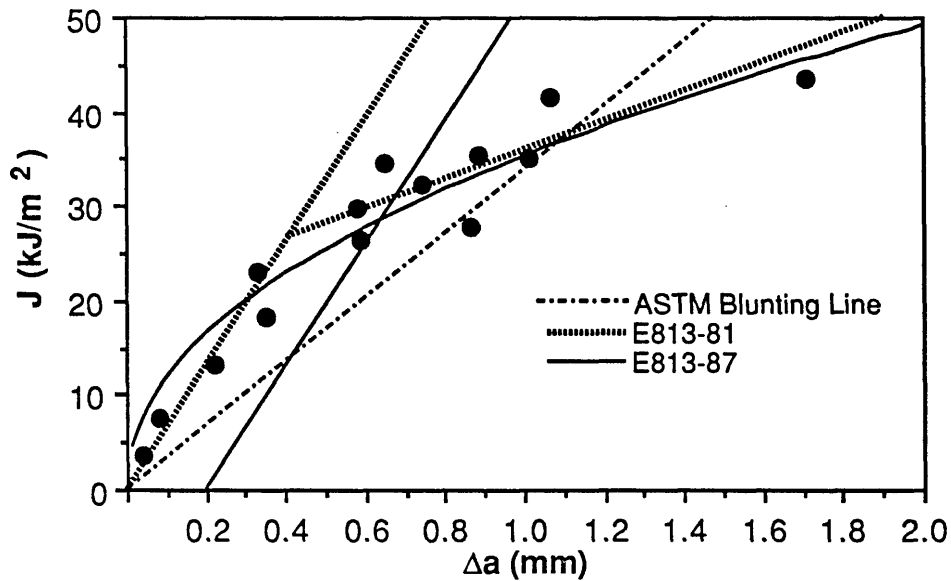


Fig.3.26 J - R curve for MDPE from multiple specimen J tests

3.8.2 Compliance Measurements

Typical load and clip gauge displacement curves for the HDPE 8621 are shown in Fig.3.27. Hysteresis loops resulting from unloading were observed in both the load-clip gauge displacement and load-load line displacement plots for all materials. If fixed support rollers were used, rhomboid-shaped hysteresis loops resulted as shown in Fig.3.28 and they appeared in both the load line and crack mouth compliance. This shows that the friction at the rollers has an effect on the specimen compliance for polymers, and the sharp corners indicate where the friction effect reverses. Thus free rollers must be used in order to minimize the error in compliance measurements.

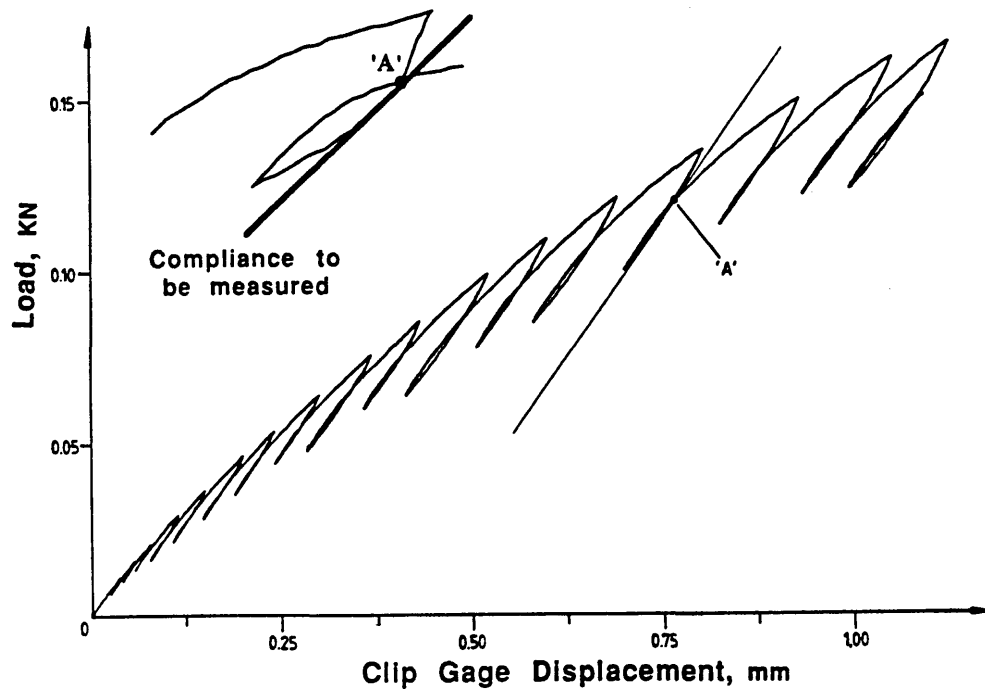


Fig.3.27 Typical load-clip gauge displacement curve for HDPE 8621 with free support rollers

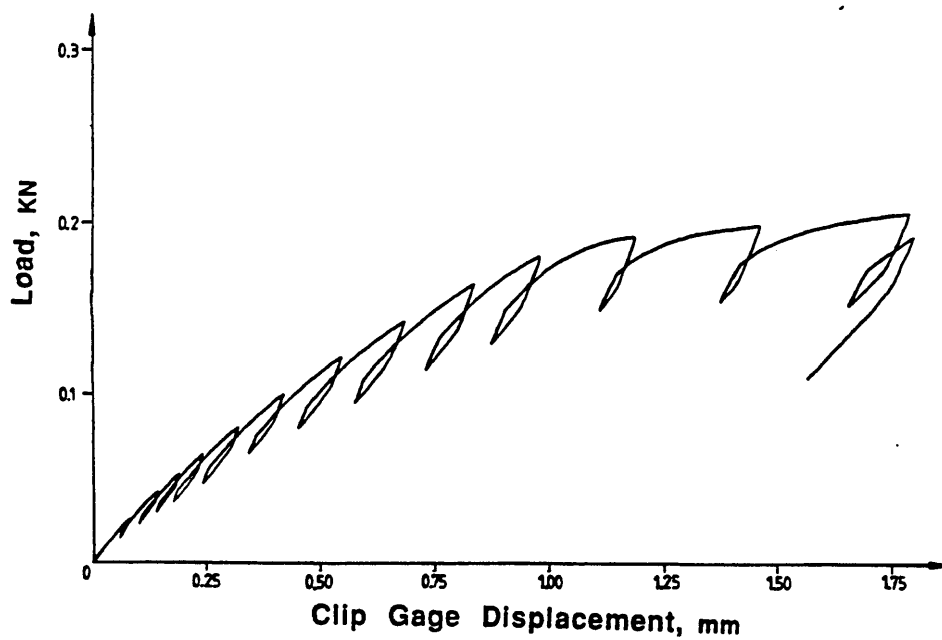


Fig.3.28 Typical load-clip gauge displacement curve for HDPE 8621 with fixed support rollers

In Fig.3.27 it can be seen that the main difficulty in crack length prediction is to determine the correct part of the unloading loop for the compliance measurement.

Since an elastic model was adopted to predict the viscoelastic behaviour of the materials, certain assumptions were made. Firstly, the material was assumed to behave elastically when it was subjected to sudden changes of loading condition or rate, and thus the slope taken for calculating the crack length should be close to the point of unloading. Secondly, the region immediately after the unloading point should not be included since pronounced curvature at that region was caused by the re-distribution of stress in the system after unloading. The position of the intersection point of the loading and unloading curves, marked as 'A' in Fig.3.27, was found to be fairly consistent and independent of the amount of unloading which had taken place and hence the point 'A' could be considered as the end of the stress re-distribution in the system. The first linear region starting from the point 'A' was used for crack length estimation as shown in Fig.3.27. The change of slope along the unloading loops for the HDPE and MDPE specimens is shown in Fig.3.29 and Fig.3.30 respectively, where both axes are plotted on arbitrary scales. Despite the scatter in the data, it can be seen that the slope attains a fairly constant value after the point 'A', where the material was assumed to commence behaving in an elastic manner, and hence the compliance measurements were taken in this region. The estimated final crack extensions using this procedure were compared with the measured ones and the results are shown in Fig.3.31. The *ASTM E813* standard suggests that the accuracy of the predicted crack extension should be within $\pm 15\%$ of the real value. However, for small amounts of crack extension, this requirement is difficult to achieve. It has been proposed by Heerens et al.[1988] that a margin of $\pm 0.1\text{mm}$ or $\pm 15\%$ in crack extension, whichever is greater, can be regarded as an appropriate requirement for crack length measurement accuracy. Based on these criteria, it can be seen from Fig.3.29 that most of the data satisfy this requirement, and those which do not are only marginally outside.

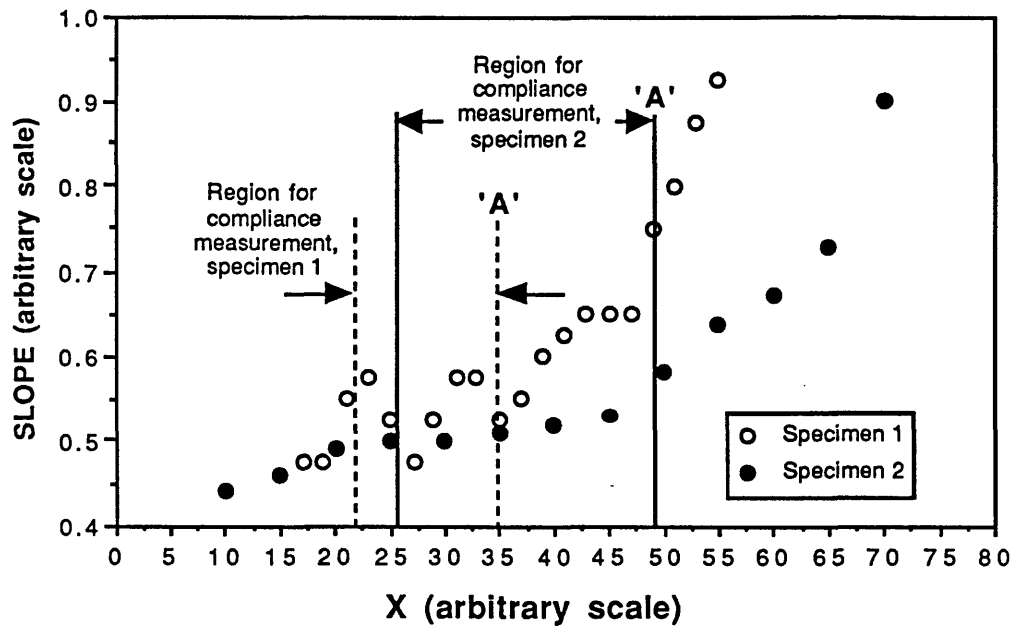


Fig.3.29 The change of slope along the unloading loop for HDPE 8621

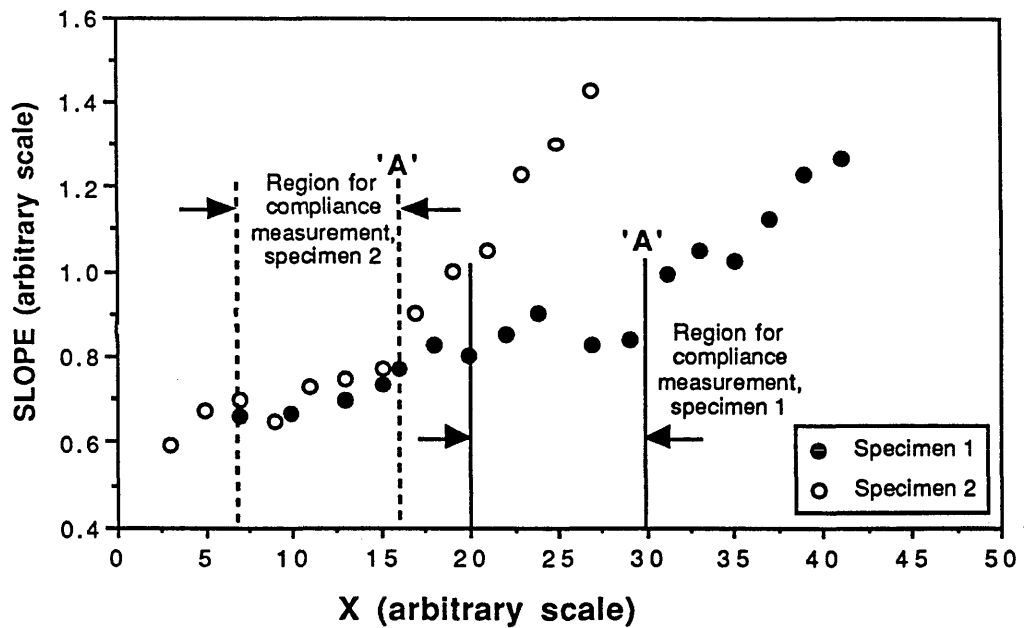


Fig.3.30 The change of slope along the unloading loop for MDPE

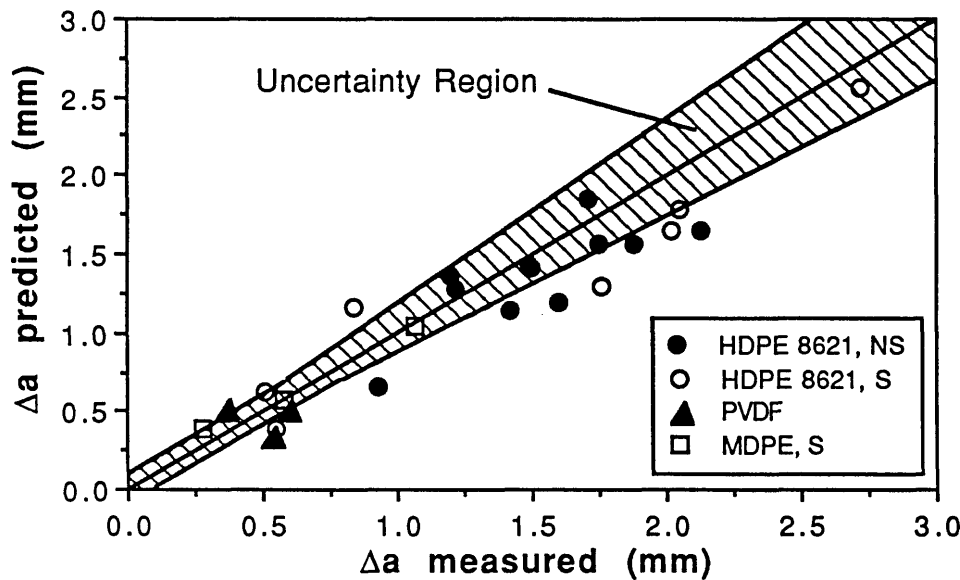


Fig.3.31 Comparison of measured and predicted Δa
(NS=non side-grooved samples, S= side-grooved samples)

3.8.3 Single Specimen J Tests

3.8.3.1 HDPE 8621

Figure 3.32 shows a typical J - R curve obtained from the single specimen J tests for HDPE 8621. The experimental blunting lines acquired from the multiple specimen J tests were used for evaluating the J_{IC} values. It can be seen that the agreement between the results from the multiple and single specimen methods is encouraging. The new E813 protocol again predicts higher J_{IC} values than the old one. The J - R curves for various loading rates are given in Fig.3.33 and the effect of flattening of the R -curve for high loading rates can also be observed.

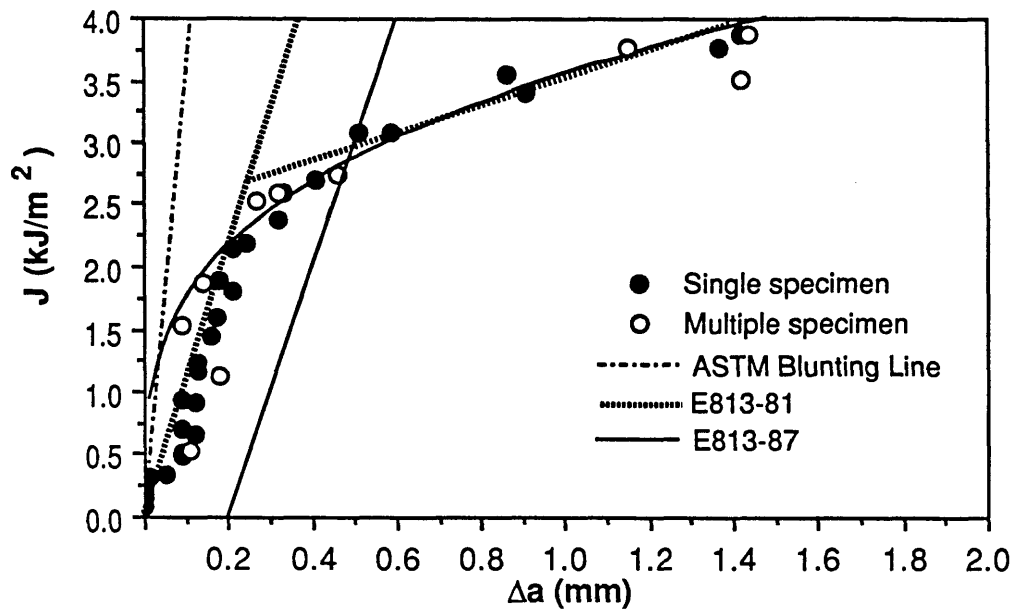


Fig.3.32 J - R curve for HDPE 8621 from single specimen J tests with loading rate of 1mm/min, $B=12$ mm and $W=30$ mm

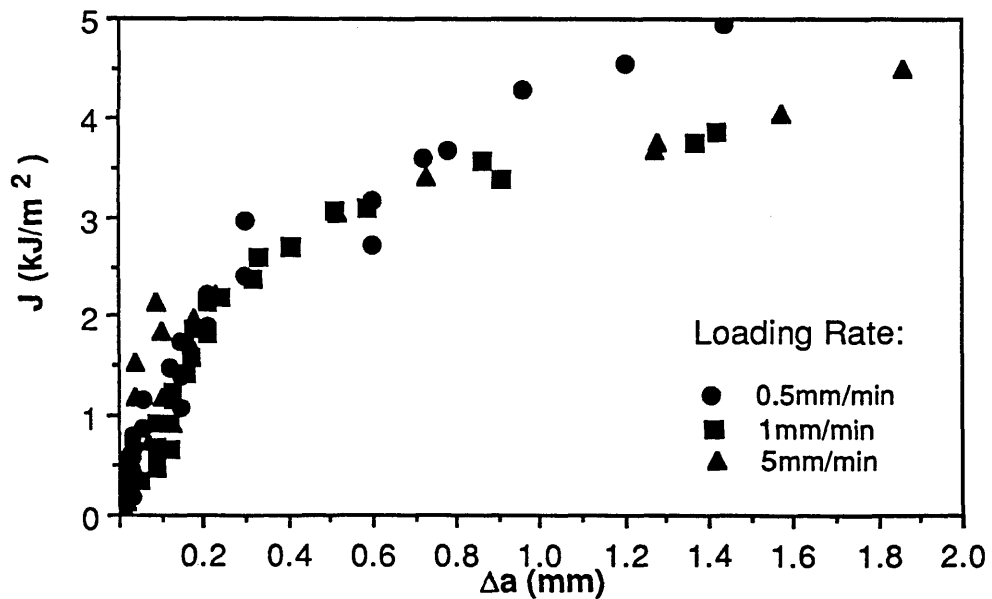


Fig.3.33 J - R curves for HDPE 8621 from single specimen J tests at various loading rates

The effect of specimen thickness and width on the J - R curves are illustrated in Fig.3.34 and Fig.3.35 respectively. The results from the single specimen J tests also indicate that the J - R curves are not affected by the variations in specimen width but the slope of the R -curves increases with decreasing specimen thickness. Side-

grooved samples again produce flattened R -curves and slightly lower J_{IC} values as observed previously.

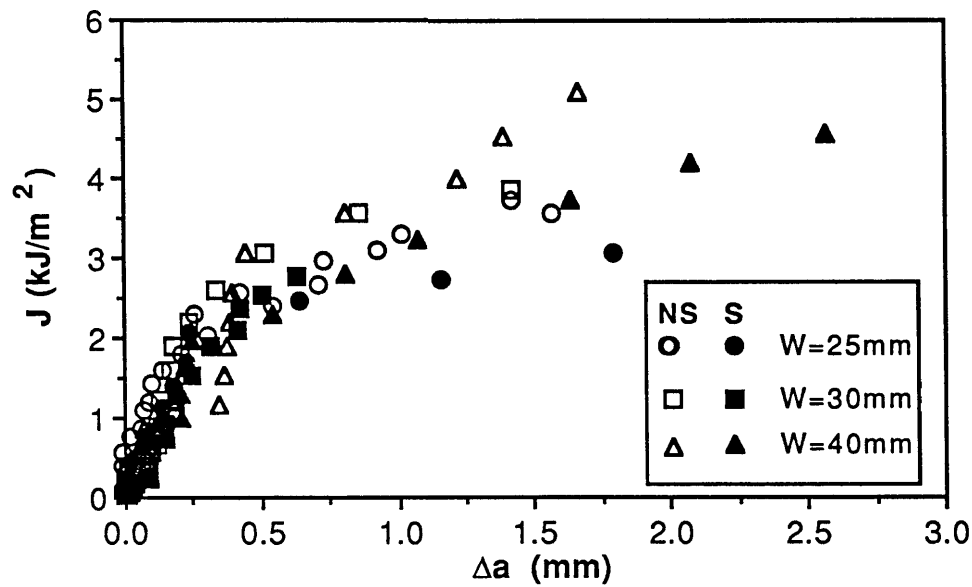


Fig.3.34 Effect specimen width on the J - R curves obtained from single specimen J tests for HDPE 8621 (NS=Non side-grooved specimens, S=Side-grooved specimens)

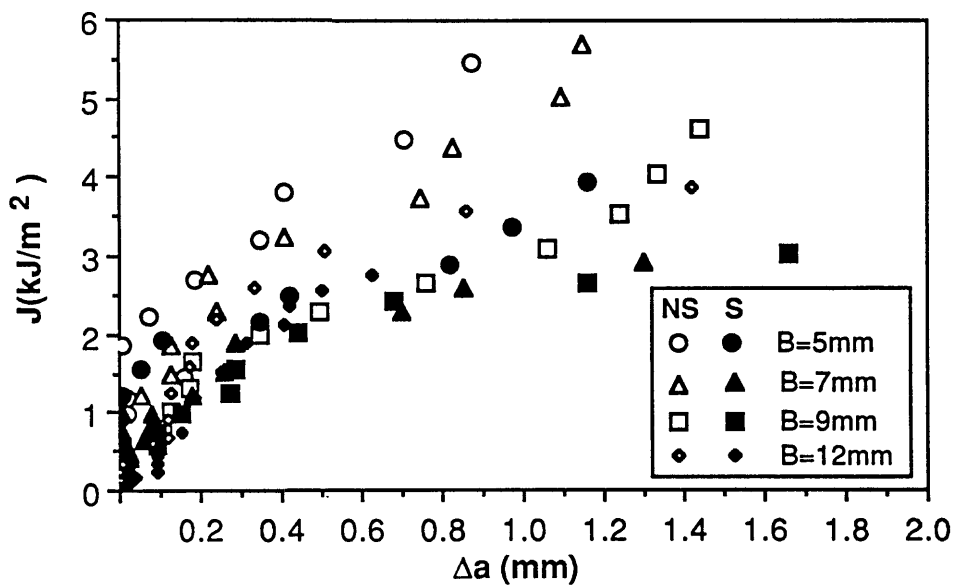


Fig.3.35 Effect of specimen thickness on the J - R curves obtained from single specimen J tests for HDPE 8621 (NS=Non side-grooved specimens, S=Side-grooved specimens)

The J - R curves obtained from the two methods are compared and shown in Figs 3.36 to 3.40. In spite of the scatter, the results from both methods show good agreement and are presented in Table 3.4. Nevertheless, for non side-grooved specimens with small thicknesses, 5 and 7 mm, discrepancies become significant as shown in Fig.3.38. It can be seen from Fig.3.39 and Fig.3.40 that side-grooving seems to improve the consistency of the results for both methods. The coefficients of the equation for the power law fitted R -curves are shown in Table 3.5 and they agree well with those obtained from the multiple specimen tests.

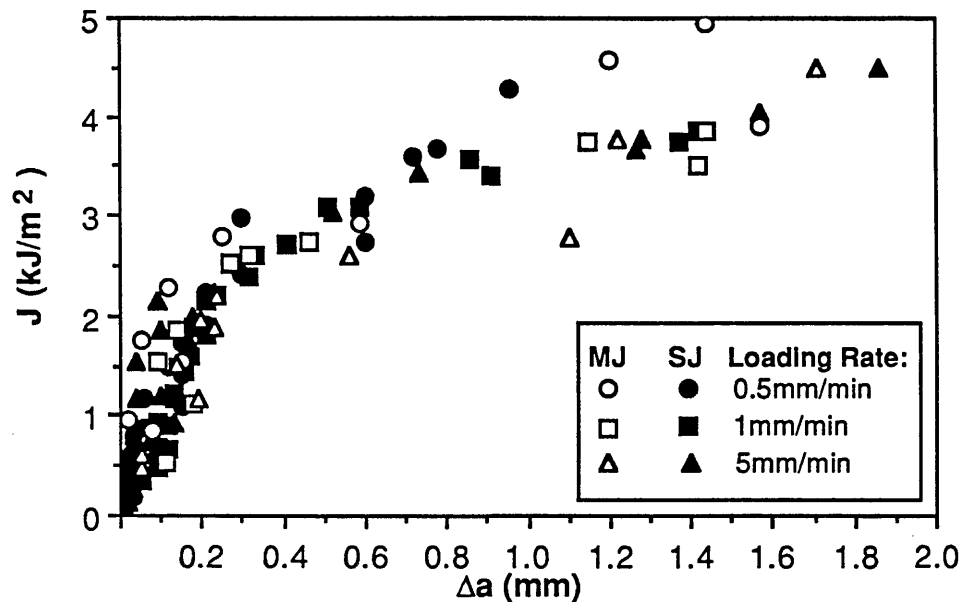


Fig.3.36 Comparison of the J - R curves from multiple(MJ) and single(SJ) specimen J tests for HDPE 8621 at various loading rates

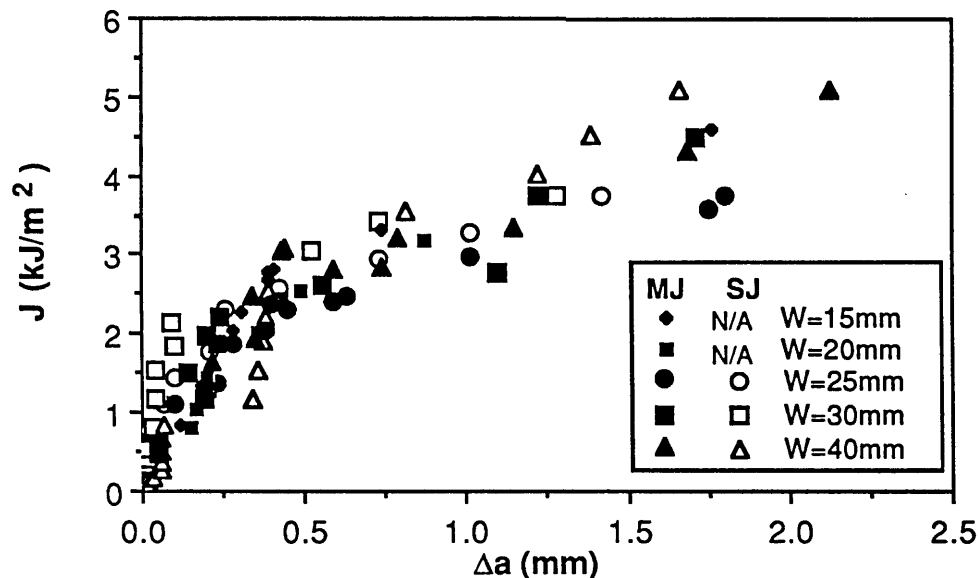


Fig.3.37 Comparison of the J - R curves from multiple(MJ) and single(SJ) specimen J tests for HDPE 8621non side-grooved specimen with various widths

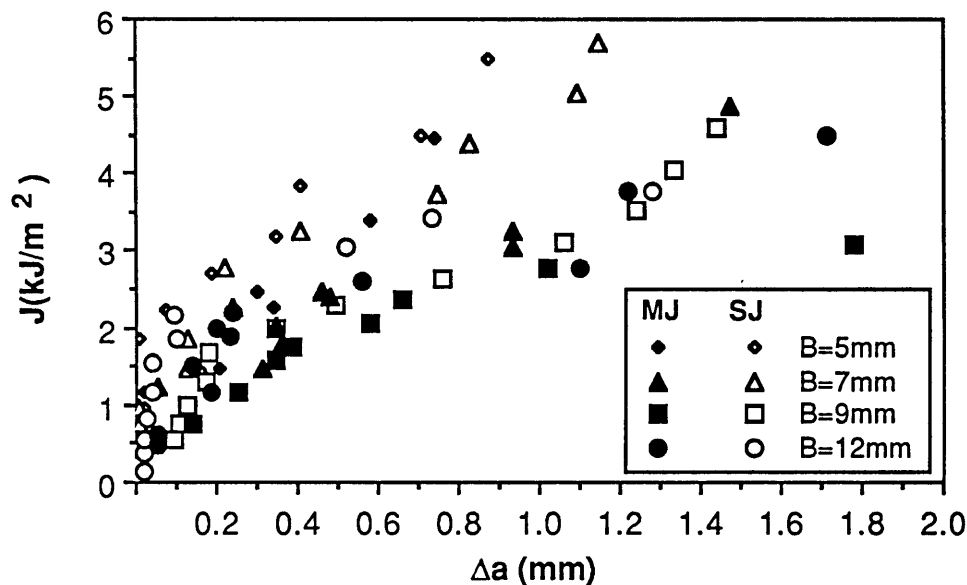


Fig.3.38 Comparison of the J - R curves from multiple(MJ) and single(SJ) specimen J tests for HDPE 8621non side-grooved specimen with various thicknesses

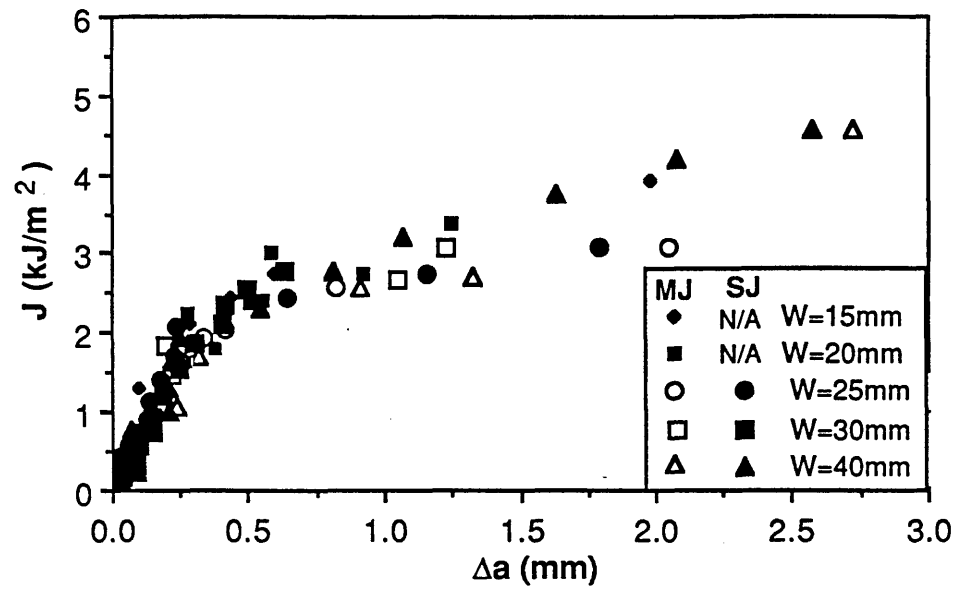


Fig.3.39 Comparison of the J - R curves from multiple(MJ) and single(SJ) specimen J tests for HDPE 8621 side-grooved specimen with various widths

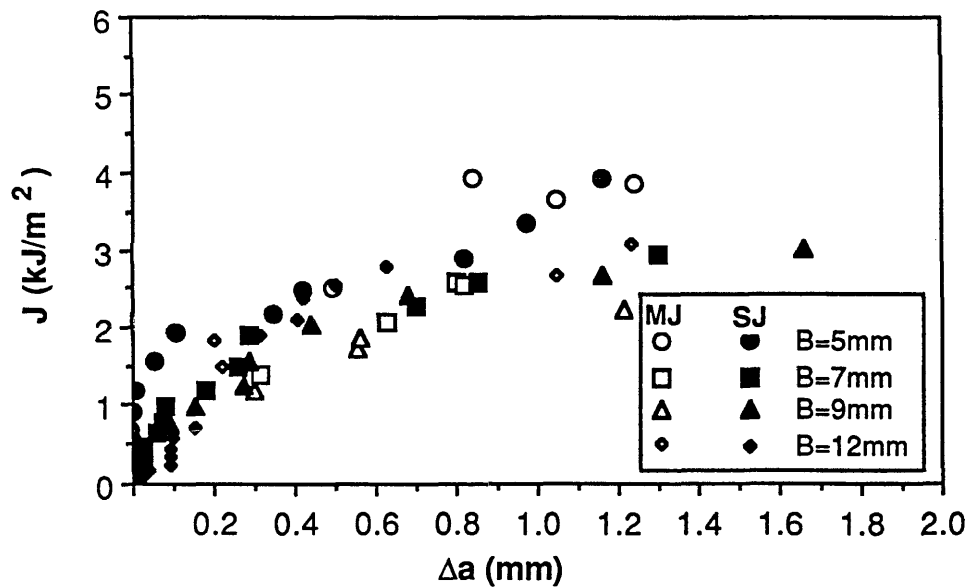


Fig.3.40 Comparison of the J - R curves from multiple(MJ) and single(SJ) specimen J tests for HDPE 8621 side-grooved specimen with various thicknesses

			J_{IC} (kJ/m ²)							
			E813-81 Method				E813-87 Method			
			Non side-grooved		Side-grooved		Non side-grooved		Side-grooved	
B (mm)	W (mm)	$\dot{X}$ (mm/min)	MJ	SJ	MJ	SJ	MJ	SJ	MJ	SJ
5	30	1.0	-	4.2	2.1	2.1	-	5.5	2.8	2.8
7	30	1.0	2.4	2.5	1.5	2.0	3.2	5.3	2.1	2.25
9	30	1.0	2.15	2.3	1.7	2.3	2.3	2.8	1.81	2.3
12	30	0.5	2.6	2.3	-	-	3.0	2.8	-	-
12	30	1.0	2.6	2.7	2.0	2.1	2.9	2.8	1.7	2.3
12	30	5.0	2.1	2.7	-	-	2.7	3.0	-	-
12	15	0.5	2.8	-	2.5	-	3.2	-	2.7	-
12	20	0.5	2.4	-	1.8	-	2.8	-	2.1	-
12	25	1.0	2.3	2.4	2.2	2.3	2.4	2.7	2.3	2.4
12	40	1	2.5	2.6	2.0	2.4	2.8	3.1	2.0	2.4

Table 3.4 Main results for J tests for HDPE 8621
(MJ = Multiple specimen J tests, SJ = Single specimen J tests)

3.8.3.2 PVDF and MDPE

The J - R curves gained from the single specimen J tests for PVDF and MDPE are shown in Fig.3.41 and Fig.3.42 respectively. The J_{IC} values were determined using the best fit blunting line obtained from the multiple specimen J tests and the results are included in Table 3.6. It can be seen that scatter of the data is more pronounced at the blunting region for the MDPE samples. The results agree well with those from the multiple specimen J tests within the scatter of the data. The constants for the power law fitted R -curves are shown in Table 3.7.

			$J = C \Delta a^n$							
			Non side-grooved				Side-grooved			
			C		n		C		n	
B (mm)	W (mm)	$\dot{X}$ (mm/min)	MJ	SJ	MJ	SJ	MJ	SJ	MJ	SJ
5	30	1.0	-	5.67	-	0.51	3.95	3.44	0.66	0.42
7	30	1.0	3.56	4.87	0.55	0.51	2.88	2.67	0.63	0.40
9	30	1.0	2.62	3.10	0.33	0.45	2.12	2.61	0.29	0.29
12	30	0.5	3.81	3.56	0.44	0.45	-	-	-	-
12	30	1.0	3.42	3.54	0.25	0.22	2.84	3.66	0.35	0.56
12	30	5.0	3.23	3.61	0.47	0.27	-	-	-	-
12	15	0.5	3.75	-	0.34	-	3.19	-	0.30	-
12	20	0.5	3.47	-	0.47	-	2.56	-	0.30	-
12	25	1.0	2.95	3.19	0.38	0.36	2.61	2.69	0.25	0.22
12	40	1	3.42	3.85	0.46	0.47	2.7	3.08	0.44	0.42

Table 3.5 Constants of the power law fitted R -curves for 8621HDPE

(MJ = Multiple specimen J tests, SJ = Single specimen J tests)

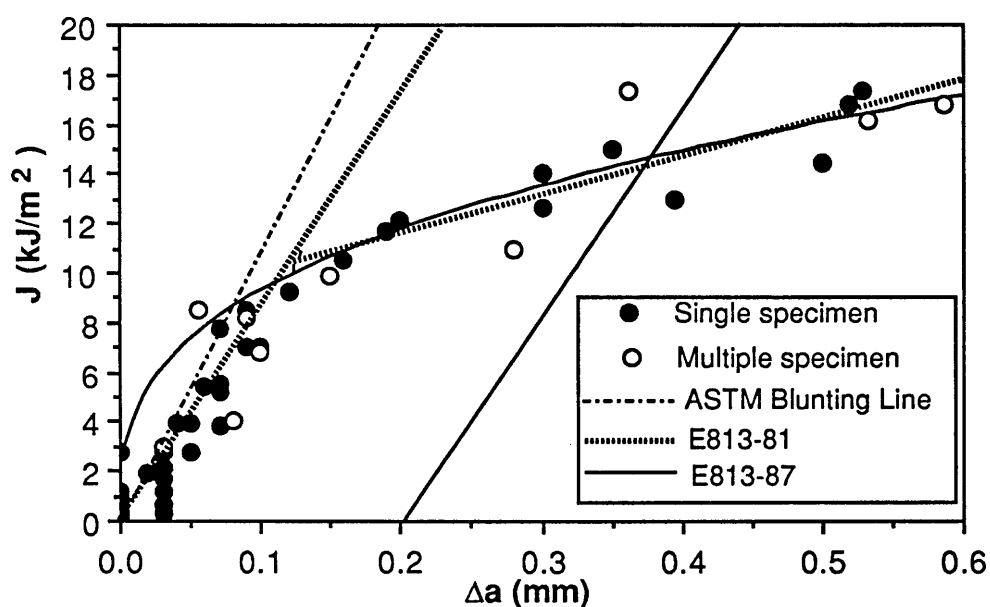
	J_{IC} (kJ/m ²)			
	Multiple Specimen Tests		Single Specimen Tests	
	E813-81	E813-87	E813-81	E813-87
PVDF	10.1	15	10.2	14.5
MDPE	26.5	28.5	26	31

Table 3.6 Main results for J tests for PVDF and MDPE

	$J=C\Delta a^n$			
	C		n	
	MJ	SJ	MJ	SJ
PVDF	21.8	20.4	0.38	0.34
MDPE	35.2	39.4	0.48	0.60

Table 3.7 Constants of the power law fitted *R*-curves for PVDF and MDPE

(MJ = Multiple specimen J tests, SJ = Single specimen J tests)

Fig.3.41 *J*-*R* curves for PVDF from single specimen *J* tests

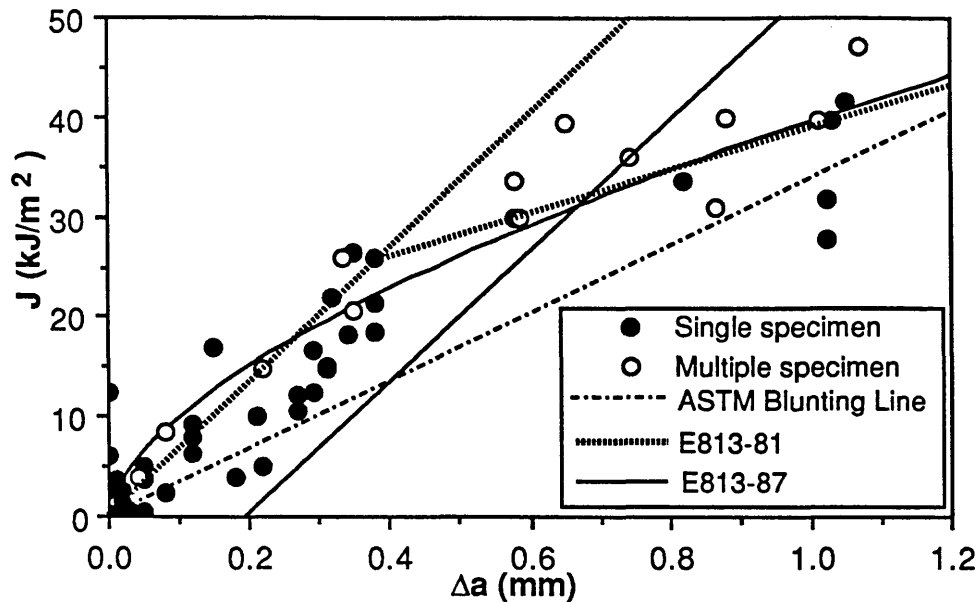


Fig.3.42 *J-R* curves for MDPE from single specimen *J* tests

3.9 Discussion

3.9.1 Application of the ASTM E813 Test Methods

For the single specimen method, the point where compliance was taken for crack length prediction was determined empirically, and it worked well for the materials tested here which represent the characteristics of a range of thermoplastics. The results agreed well with those from the multiple specimen method within the limits of the scatter. Since the compliance was measured manually, scatter in the data could not be avoided, and accuracy could not be evaluated quantitatively. Nevertheless, the single specimen method was found to be a possible alternative to the multiple specimen method.

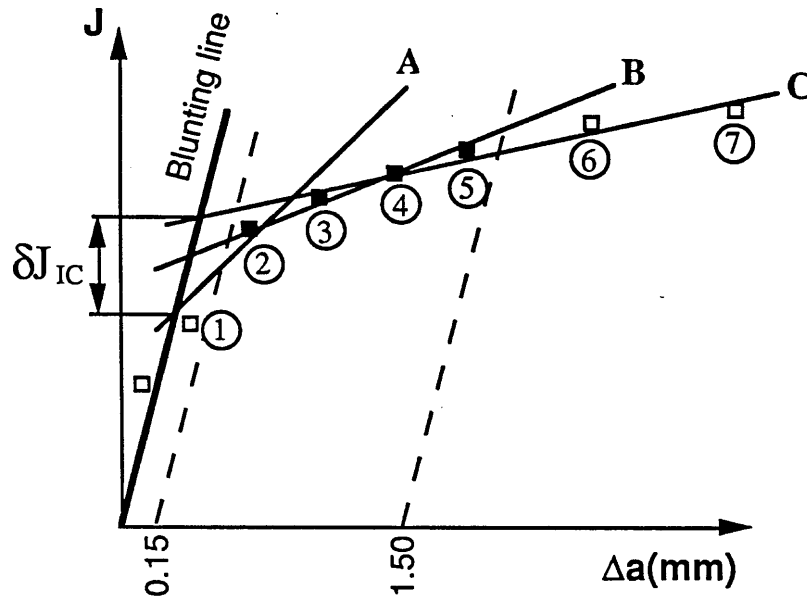
For results to be considered valid in this method, the *J* controlled growth condition must be fulfilled and this is achieved by limiting the maximum crack extension to less

than 6% of the uncracked ligament. In the ASTM E813 standard, the 1.5 mm offset blunting line is also used for data exclusion. Combining these two restrictions, it can be implied that the specimen should have a minimum width of 50 mm and a thickness of 25 mm. This may impose problems in producing specimens of such a size for polymers. It is suspected that the 6% criteria is too stringent, and on examination of the work performed by other authors (Chan et al.[1983], Hashemi et al.[1986 i, ii, iii] and Huang et al.[1987]) and the results from this study, it is apparent that a geometry independent R -curve can exist for crack growth of up to about 10% of the uncracked ligament. Gordon et al.[1989] has also proposed that the limit can be relaxed to 15% for metals and a size independent R -curve still be obtained. Therefore, it is suggested here that a maximum crack extension of 10% of the ligament is considered to be acceptable for polymers.

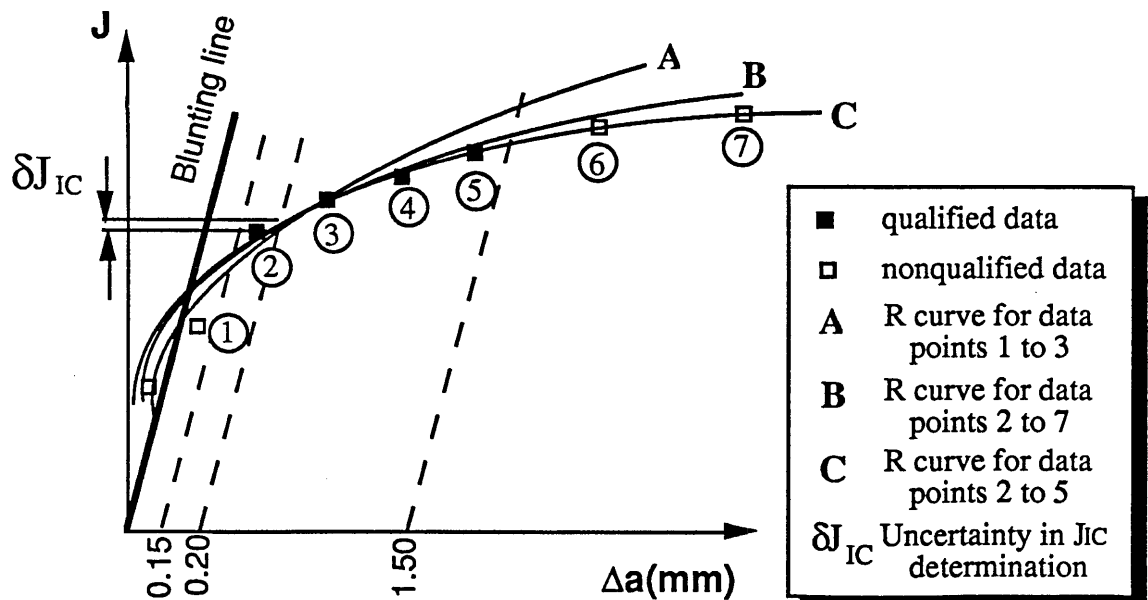
The nine point averaging technique used for crack extension measurements is considered to be more appropriate than the maximum point taken as the Δa by most other investigators in the past. Since J is a two dimensional analysis a straight crack front is assumed, so that the fracture area can be represented by the crack length. However, J is the energy per unit area and this area change is better described by the nine point averaging technique if the amount of curvature changes with Δa .

The data extrapolation procedure in the E813-81 method can lead to an artificial size effect on the J_{IC} value as observed by Huang et al.[1987]. The same effect could also result if the amount of crack extension is small as the results reported by Narisawa et al.[1989] and this is illustrated in Fig.3.43a. It can be seen that the J_{IC} value depends on the amount of crack extension and the distribution of the data points. In order to reduce the bias on the J_{IC} determination, δJ_{IC} , it is suggested that the data exclusion and distribution schemes recommended in the standards be followed if the E813-81 protocol is adopted. However, the curve fitting practice in the E813-87

method will provide a more consistent J_{IC} value (Huang [1989]) as shown in Fig.3.43b.



a) E813-81



b) E813-87

Fig.3.43 Error in the determination of the J_{IC} value. a) E813-81, b) E813-87

3.9.2 Blunting Line

In E813, the blunting line plays a crucial role in defining the J_{IC} value. However, it has been reported that the conventional blunting line does not adequately reflect the blunting mechanisms for polymers (Chan et al.[1983], Hashemi et al.[1986(i)], Narisawa et al.[1989]) and the same phenomenon can be seen in the results presented here. The reasons for this are still uncertain but it has been explained by Hashemi et al.[1986(i)] as the effect of plastic constraint at the crack tip. From the results, this is considered to be inappropriate for the materials tested here since the constraint factor will be less than 1 in the cases of HDPE 8621, HDPE 5731 at low temperature, and PVDF. It is believed that the problem lies with the crack length measurement and the properties of the materials. Crack extension measurements from the fracture surfaces are always difficult since the changes of fracture surface morphology are taken as crack advance. For materials such as HDPE which craze before fracture, the craze zone is usually included in the crack length measurements and this is bound to give a less steep blunting line as shown in Fig.3.44a in which the crack extension due to blunting Δa_b is greater than half of the crack opening displacement, δ . In the case of the tougher polymers such as MDPE, the material at the crack tip flows due to viscoelasticity and a layer of plastically deformed and strain hardened material will be formed. A larger crack opening displacement and a steeper blunting line will result as shown in Fig.3.44b in which $\Delta a_b < \delta/2$. Fig.3.44c shows the usual assumption of smooth blunting in which $\Delta a_b = \delta/2$ together with the three types of blunting lines.

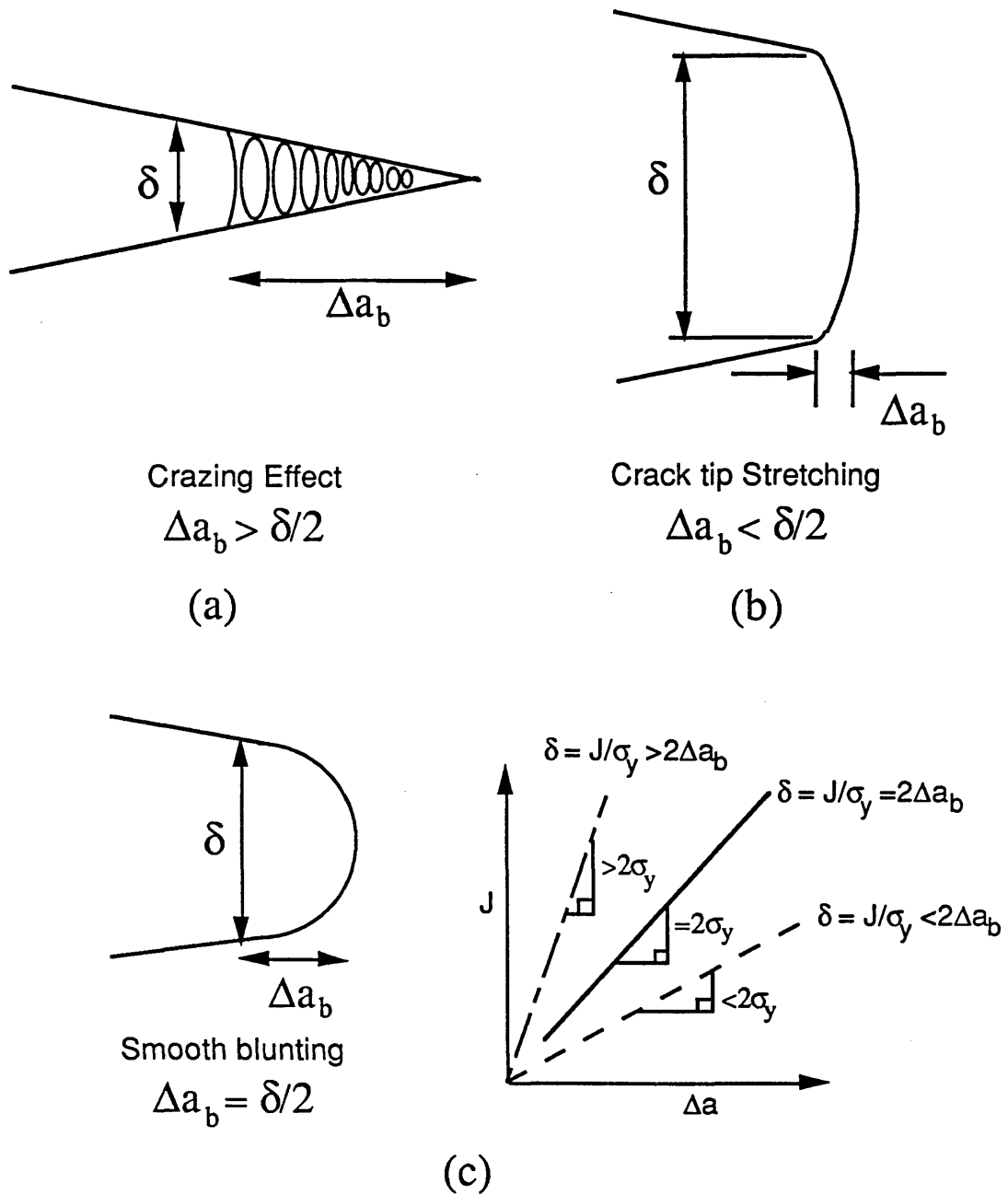


Fig.3.44 Illustration of blunting processes for polymers

The results from the HDPE 5731 show that the blunting process changes from crack tip stretching at high temperature, due to small specimen size and lack of constraint at the crack tip, to the formation of crazes ahead of the crack tip at very low temperatures. Crack blunting measured from the side of the mid-section by sectioning of the specimen as suggested by Narisawa et al.[1989] provides an alternative, but the crack tip must be opened to attain the same δ as before

unloading took place since the crack tip will close up in the relaxed state and no crack extension or a sharp crack will be observed. Since the blunting process is material dependent, a formulation of the blunting line for polymers is difficult to establish. As long as the blunting line still plays a role in defining the J_{IC} , further investigation of the blunting process is necessary.

3.9.3 Definition of J_{IC} According to the ASTM E813 Standards

By comparing the definitions of J_{IC} in the new and old *ASTM E813* protocols, it is apparent that each has its own advantages and disadvantages. The old standard seems to identify the initiation point more correctly, but the representation of the R -curve by a regression line causes some degree of uncertainty in determining J_{IC} , as shown earlier in Fig.3.43a. This is especially true for toughened polymers where the R -curve rises gently, and a distinct change of slope as expected in the protocol will not be observed. For the new E813 procedures, extrapolation of data is avoided and the J_{IC} is defined as a point on the R -curve. The 0.2 mm offset blunting line offers the advantage of excluding data in the blunting region, which are often too small to be measured accurately. The power law fit gives a better description of the R -curve, but the 0.2 mm offset blunting line for the J_{IC} determination actually describes the fracture toughness at 0.2 mm ductile crack growth which will always give a higher J_{IC} value. The new E813 standard gives more consistent J_{IC} values, although it does not characterize the initiation point satisfactorily.

In both protocols, the blunting line is not required to be measured during the J tests but the use of the theoretical blunting line is suggested. However, it has been shown that the conventional blunting line is not adequate for modelling the blunting mechanisms of polymers. The use of the blunting line concept for defining the initiation point is questionable. Direct visualization of the initiation point is often

impossible for opaque materials and crack growth measurements from the mid-section of the specimen are dubious since the crack tends to initiate in the middle. Initiation defined using this technique will result in a very low J_{IC} value because ductile tearing may not have occurred along the whole crack front. An alternative J_{IC} definition is given in the European standard; the J value at 0.2 mm absolute crack growth is taken as the J_{IC} value. The advantage of this method is that it avoids the use of a blunting line, but the amount of crack extension for J_{IC} determination seems to be arbitrary. The major difficulty with the application of the J method to polymers is to define a suitable measurement point for J_{IC} and further investigation of the initiation process in polymers would be desirable before the measured J_{IC} value can be successfully applied for design purposes.

3.9.4 Effects of Loading Rate and Side-grooving

The J_{IC} values and the J - R curve for the HDPE 8621 did not show a strong dependence on the loading rate. However, it can be seen from the results that the J - R -curves tend to flatten with increasing loading rate. This indicates that the energy for crack propagation reduces as strain rate increases. However, additional experiments are considered to be necessary owing to the restricted range of loading rates used in the tests and the amount of scatter in the data.

Side-grooving is used to remove the shear lips and promote the triaxiality of stress at the crack tip which in turn will reduce the crack front curvature. From the results, it can be seen that side-grooving reduces the slope of the R -curve and the value of J_{IC} . The decrease in slope indicates that the plastic deformation at the shear lips accounts for a substantial part of the total energy input into the system, and the lower R -curve will cause a lower J_{IC} value. If the actual initiation point is on the blunting line, a difference in the J_{IC} value between the side-grooved and non side-grooved specimens will result due to the rather low resolution of the data, and the

intrinsic problems associated with the definition of the initiation point, as shown in Fig.3.45.

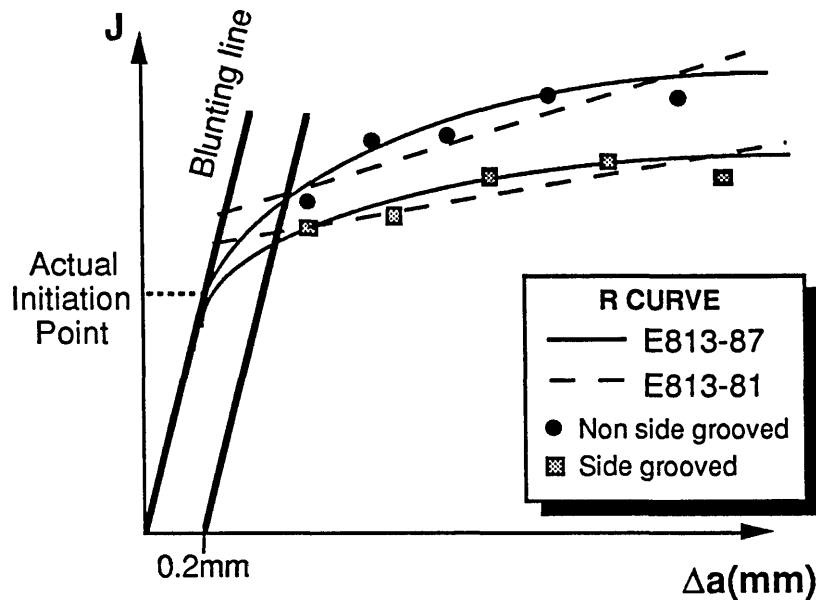


Fig.3.45 Illustration of the effect of side-grooving on the R -curve and the J_{IC} value

The J_{IC} value evaluated using the side-grooved samples should reflect the actual initiation point more closely since the blunting process for both types of specimen is the same. The difference between the J_{IC} values evaluated using the old and new protocols was reduced for a flat R -curve and this trend can be observed from the results for the side-grooved samples and the HDPE 5731 at low temperature. Side-grooving also improved the reproducibility of data, and the unloading compliance method worked better on side-grooved specimens.

It can be seen from Fig.3.38 that the R -curves for the 5 and 7 mm thick specimens obtained from both methods are not comparable, but the same effect is not observed for the R -curves from side-grooved samples shown in Fig.3.40. This shows that the size of the plastic zone relative to the specimen thickness has a direct effect on the compliance of the specimens. The fracture surfaces of the 7 mm thick samples are shown in Fig.3.46. By referring to Figs.3.22 and 3.46, it is obvious that there is more

plastic deformation at the crack fronts of the non side-grooved samples. This is probably due to the lack of constraint at the crack tip. Since the single specimen method assumes elastic unloading, if the size of the plastic region is large relative to the specimen thickness, the unloading response will be controlled by the plastically deformed material which behaves in a non-linear manner. The compliance measurements will be subject to error in this case.

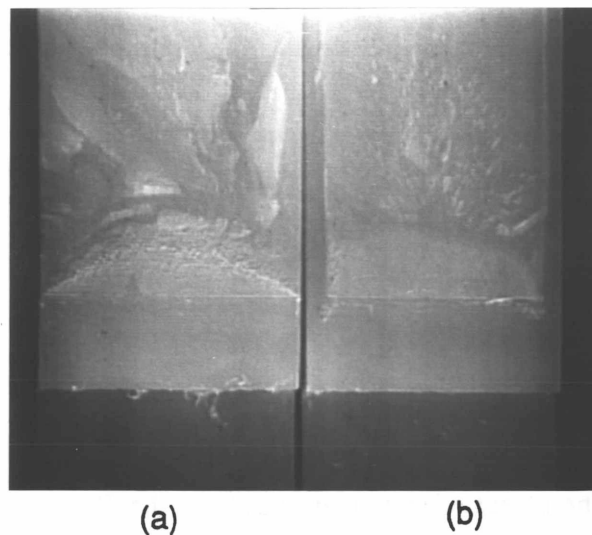


Fig.3.46 Fracture surfaces of 7 mm thick 8621 specimens
a) non side-grooved, b) side-grooved

It has been suggested that the ASTM size criteria may not be appropriate for this material. By examining the results shown in Figs.3.38 and 3.40, little change in the slope of the R -curves is observed for specimens with thickness greater than 5 mm. If the minimum required dimension is assumed to be 5 mm, the specimens with the shortest ligament will still satisfy this requirement. It is believed that if the ligament is further reduced to less than 5 mm, a steeply rising R -curve may be obtained due to the short ligament failing in the post yield regime. This corresponds to a value of 50 for the constant α in Equation (3.18). This observation is consistent with the results presented in Section 2.8.2.3. It has been shown that the minimum required crack length for achieving a constant K_{IC} value for this material is larger than that

calculated from the ASTM size criteria. Therefore, the ASTM size criteria for both K_{IC} and J_{IC} tests may not be appropriate for this material.

The side-grooving technique is recommended here especially for testing thin samples using the unloading compliance method. In terms of design usage, data generated from side-grooved specimens will be more conservative since most of the plastic work is not taken into account. On the other hand, it may be unfair to judge the material performance if the plastic work is disregarded. However, partition of the elastic and plastic work is still difficult to achieve, and information provided by the side-grooved specimens seems to be more appropriate on the grounds of safety.

3.9.5 Effects of Specimen Size

It has been shown that variation of specimen dimensions has little effect on the J_{IC} value for the HDPE 8621 material but the shape of the R -curve has been observed to be size dependent. The effect of specimen width on the R -curve was small but the R -curve became steeper as specimen thickness decreased. The latter suggests that the increase in slope is due to the plastic work dissipated at the shear lips. It has been indicated by Etemad et al.[1988] that a unique R -curve can be obtained by normalizing the abscissa, Δa , with the appropriate size parameter. They have shown that this method works well for metal specimens with large amounts of crack extension. Based on this method, the abscissa, Δa , of the R -curves for specimens with various thickness was normalised by B . However, for side-grooved samples, Δa was normalised by B^2/B_N instead of B_N as suggested by Etemad et al.[1988]. It is thought that B actually indicates the degree of constraint and it may be represented more closely by the ratio of the specimen thickness and the shear lip size, r_s , i.e. B/r_s . If the shear lip size is approximately the same for all specimens, Δa can be normalised with B only. Since the shear lips are much smaller for the side-grooved specimens, a larger B will be required if B is the normalising parameter. It is

proposed that for side-grooving less than 20% of total specimen thickness, B^2/B_N should provide a good approximation of the actual normalising thickness. The normalised R -curves are given in Fig.3.47. It can be seen that the R -curves are nearly thickness independent, and it must be pointed out that the R -curves for the 5 and 7 mm thick plain-sided specimens obtained from single specimen method are excluded here, since it has been demonstrated that the crack length prediction is erroneous due to insufficient constraint at the crack tip. It can also be noted that the scatter near the blunting region is more pronounced.

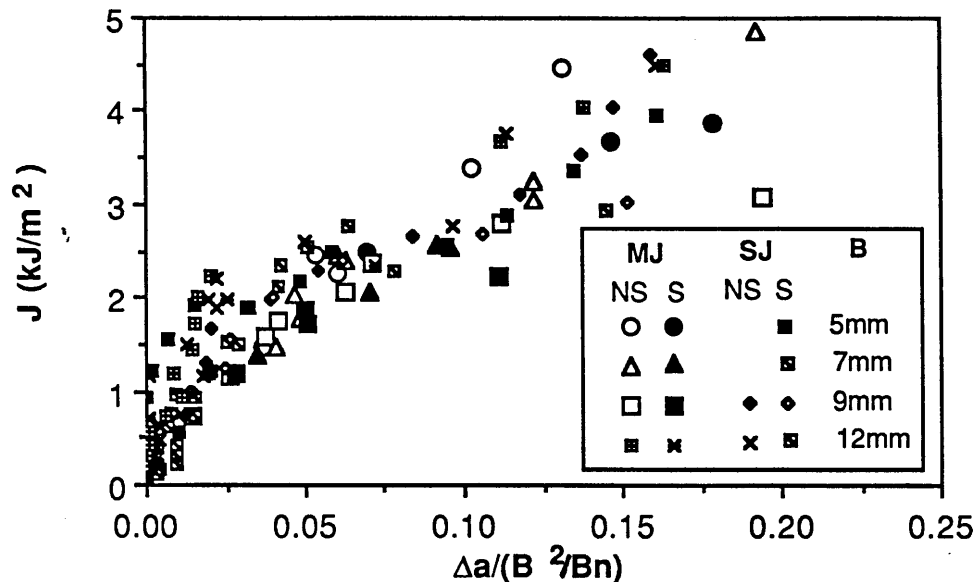


Fig.3.47 Normalised R -curves for HDPE 8621 specimens with various thickness
(MJ=multiple specimen test data, SJ=single specimen test data,
NS=non side-grooved specimens, S=side-grooved specimens)

Although the improvement on the uniqueness of the R -curve gained by the normalisation procedure is promising, it must be realised that the extent of crack extension obtained here is relatively small and only represents a fraction of the whole R -curve. Based on the results presented here, it is difficult to comment on the applicability of the method to polymers. It would be desirable to perform extra tests to obtain R -curves with larger amounts of Δa in order to verify the utility of the

normalisation procedure to polymers. This would provide valuable information on the crack growth resistance of the material.

3.9.6 Significance and Use of the K_{IC} , G_{IC} and J_{IC}

It is a common practice to equate the J_{IC} with K_{IC} via Equation (3.12). This equation is strictly only true for the linear elastic case. The J_{IC} value determined from side-grooved samples using the E813-81 method gives an value of 2.0 kJ/m^2 which is equivalent to $1.8 \text{ MPa}\sqrt{\text{m}}$ in terms of K_{IC} . K_{max} measured at room temperature was found to be $1.6 \text{ MPa}\sqrt{\text{m}}$. So that the agreement between the two values is good.

The current standard test methods provide a means for characterising the toughness of polymers near the initiation point, but refinement of certain notions such as the initiation point and blunting line are necessary. Theoretically, K_{IC} , G_{IC} and J_{IC} should be single parameters which are material properties. In fact, they do vary with test conditions for polymers, and it is possible to obtain crack growth under a constant load well below the failure load predicted from these measured toughnesses. In terms of the practical application of these plane strain toughness values for polymers, the loading conditions during the test should resemble that in actual operation. Data obtained from tests at sub-zero temperature will provide little information on the performance of the material at room temperature. Such data can only be used to compare the characteristics of different materials for quality control purposes. Due to the viscoelastic and time dependent nature of polymers, the validity of applying these values to predict the long term performance is not certain. In applications where time dependence is not a major factor, these values will be useful in establishing the flaw size limits for service evaluation or for failure load prediction.

3.10 Conclusions

The results from the multiple specimen tests and the single specimen tests were in good agreement. The regions on the unloading and loading hysteresis loops for compliance measurements were determined empirically and the friction of support rollers would change the appearance of the loops. The crack extension predicted using this scheme agreed well with that measured directly from the fracture surfaces. The data exclusion and distribution scheme suggested in the E813 standards must be followed in order to gain an unbiased estimate of J_{IC} values. The maximum Δa of 6% of uncracked ligament recommended for attaining J controlled growth conditions is considered to be too stringent for polymers. The conventional blunting line which plays an important role in J_{IC} determination was found to be inadequate for predicting the crack blunting behaviour of polymers, and the concept of blunting line needs further revision. The J - R curves for HDPE 8621 tended to flatten slightly as the loading rate increased, but the effect of it is still not clear due to insufficient data and the limited range of loading rates used in this study. Side-grooving was found to reduce the slope of the R -curve and the J_{IC} value and to improve the consistency of results by reducing the amount of scatter in the data. The J - R curve was found to be thickness dependent for the HDPE 8621 material, but the J_{IC} value was fairly consistent despite the size of the specimens. It was found that the ASTM size criteria might be inappropriate for the 8621 polyethylene. A thickness independent R -curve is suggested by the data when the abscissa is normalised with the thickness. Due to the restricted amount of crack extension obtained in the tests, it was difficult to assess the appropriateness of the normalisation procedures for polymers. However, such a scheme would provide important information on crack growth resistance of materials provided that a unique normalised R -curve was obtained. Further investigation of this method would be desirable.

Creep Fracture: The Fracture Mechanics Approach

4.1 Introduction

In recent years, there has been considerable interest in the fracture behaviour of polyethylene. It is largely due to its increasing use in engineering applications such as water and gas pipes installations. The standard laboratory tests described in previous chapters have been widely accepted for quality control and design purposes. The materials always exhibit high ductility at room temperature in these tests. However, brittle failure can occur under a constant load which is well below

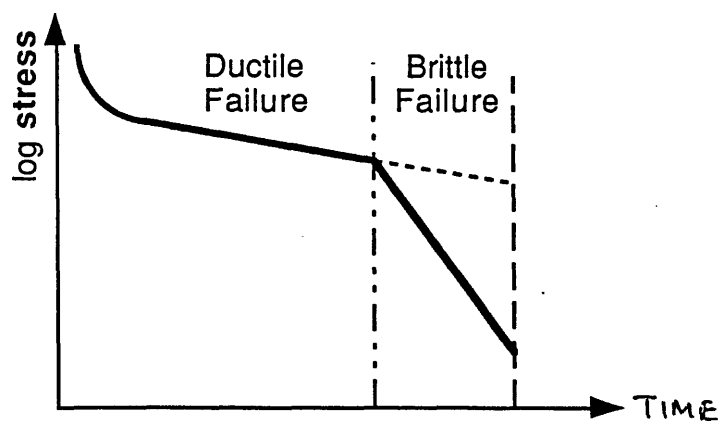


Fig.4.1 Schematic diagram of stress-lifetime curve of polyethylenes

the failure load predicted from conventional fracture tests. This phenomenon is attributed to creep and stress relaxation of the materials and is often referred to as *slow crack growth* or *sub-critical crack growth*. These factors are not considered in the time-independent mechanics of fracture which states that no cracking should occur before the critical stress intensity factor is reached. Life prediction of stressed components using the ductile failure data obtained from short term tests can overestimate the actual failure time significantly as shown in Fig.4.1. The conventional stress against lifetime curve used for long term performance evaluation tends to be unreliable due to the inherent defects in the material and the complex loading in the actual application. Therefore, it is important to understand the mechanics of slow crack growth and mode of failure. The presence of hostile environments will also assist slow crack growth. The environmental effects superimpose on the relaxation behaviour and is commonly known as *environmental stress cracking*. The concepts of fracture mechanics have been used to study fracture initiation and the kinetics of slow crack propagation. It also provides a means to evaluate the long term performance of the material under creep loading.

4.2 Slow Crack Growth

Since polymers creep under constant applied stress, the modulus and yield stress of the materials decrease with time. If a crack is present in the material, the crack will initiate when the yield stress reduces to the same value as the crack tip stress. Since the fracture is essentially brittle, the concept of linear elastic fracture mechanics can be adopted and the stresses at the crack tip can be characterised using the parameter K . The sub-critical fracture toughness is denoted as K_c here.

4.2.1 Relaxation Controlled Crack Growth

Marshall et al.[1972, 1973] showed experimentally that K_c was a function of crack growth rate, $\dot{a}$. Williams and Marshall[1975] further modelled the slow crack growth behaviour of polymers using the crack opening displacement, COD, analysis and the Dugdale line zone model. They postulated that a craze zone of size c existed ahead the crack tip and fracture occurred when the COD attained a critical value δ_c as shown in Fig.4.2. It was assumed that the stress in the craze zone σ_c was constant along the zone and that δ_c would remain constant as the crack propagated.

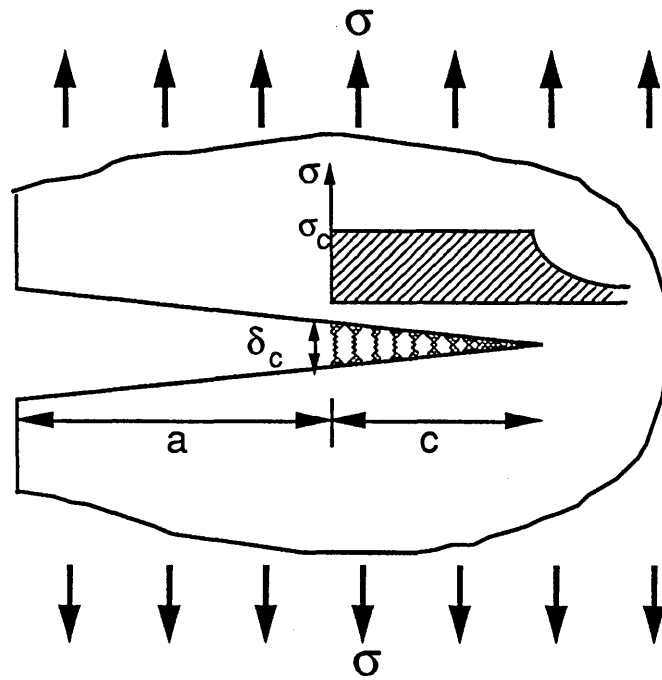


Fig.4.2 Stress distribution and line zone at a crack tip

The time dependence of the modulus E and craze stress σ_c can be described by simple power laws as:

$$\sigma_c(t) = \sigma_o t^{-m} \quad (4.1a)$$

$$E(t) = E_o t^{-n} \quad (4.1b)$$

where σ_o and E_o are the unit time values, t is a suitable time scale for the controlling process at the crack tip, m and n are the time dependence indices and should be equal to the corresponding rate dependence shown in Equations (2.26) and (2.27) if the material is linear viscoelastic. By taking the time dependence into account, the expression for the line zone size and K_c can be written as:

$$c = \frac{\pi}{8} \frac{K_c^2}{\sigma_c^2(t)} \quad (4.2)$$

$$K_c^2 = \delta_c \sigma_c(t) E(t) \quad (4.3)$$

For relaxation controlled fracture, the time scale at the crack tip can be assumed to be:

$$t = \frac{c}{\dot{a}} \quad (4.4)$$

By combining the above equations, an expression of the following form can result:

$$K_c = D \dot{a}^{\left(\frac{m+n}{2(1-m+n)}\right)} \quad (4.5)$$

where D is a constant. Schapery[1975 i, ii, iii] also showed that the relationship between K_c and $\dot{a}$ followed the same form as Equation (4.5).

4.2.2 Flow Controlled Crack Growth

Williams and Marshall[1975] also considered the environmental effect in their model. In the presence of an aggressive environment, plasticization of the crack tip zone will occur. They suggested that the crack tip zone is completely plasticized at low crack speeds and m will be greater than n . However, at high speeds, there may not be sufficient time for the fluid to penetrate into the craze region, then the crack

growth occurs in an unplasticized state. The K_c - $\dot{a}$ characteristic will resemble the one in inert environment with $m \simeq n$. Both crack growth processes at high and low crack speeds are relaxation controlled. The amount of plasticization will be limited by the flow of the fluid within the craze in the transition region. By using Darcy's law, where the flow velocity, V_f , is given as:

$$V_f = \frac{A}{12\mu} \frac{dP}{dx} \quad (4.6)$$

where A is the pore area in the craze, μ is the viscosity and dP/dx is the pressure gradient. A is assumed to be proportional to δ ($A = k\delta$, $k = \text{constant}$) and dP/dx is taken as the atmospheric pressure, P_a , divided by a constant length l which depends on the crack tip geometry and specimen thickness. Equation (4.6) can be written as:

$$V_f = \frac{k\delta}{12\mu} \frac{P_a}{l} \quad (4.7)$$

When δ attains δ_c , which has been assumed to be constant at failure, V_f will be constant and equal to the transition crack speed $\dot{a}_t$. For crack speeds exceeding $\dot{a}_t$, equilibrium can only be achieved by increasing δ until $V_f = \dot{a}$. δ_c varies with $\dot{a}$ in this case. From Equation (4.7) and Equations (4.1) to (4.4), the following relationship between K_c and $\dot{a}$ can be obtained:

$$K = Q\dot{a}^{0.5} \quad (4.8)$$

where Q is a constant. It should be noted that the flow controlled crack growth is independent of the relaxation parameters m and n . The three regions of the K_c - $\dot{a}$ curve are shown in Fig.4.3.

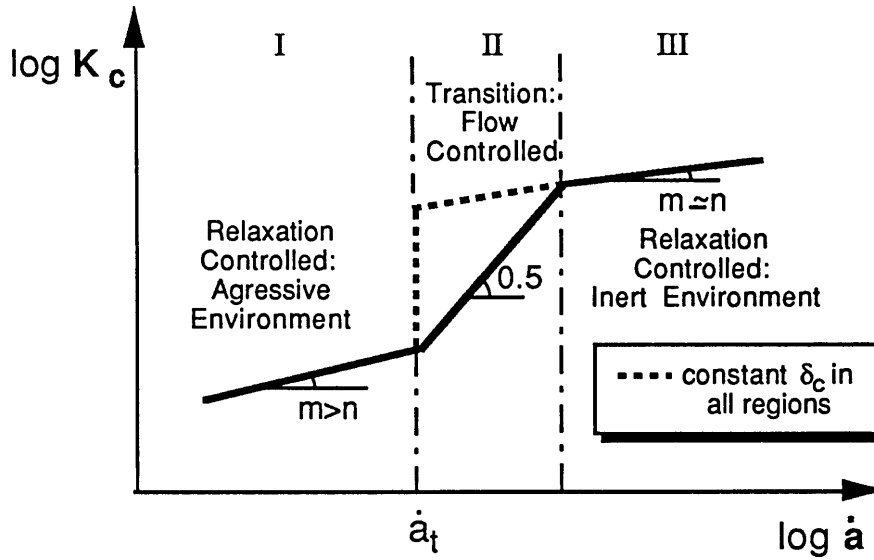


Fig.4.3 Three regions of the K_c - $\dot{a}$ curve for environmental stress cracking on polymers.

Williams [1978] later postulated that the craze zone might consist of two stages, a completely plasticized region near the crack front and an unplasticized part near the craze tip. He assumed that the fracture process was governed by the same δ_c in all regions. In this model, the transition speed is constant and is shown as the hatched line in Fig.4.3.

4.2.3 Incubation Time and Failure Time

According to the constant crack opening displacement failure criterion, the crack will not propagate if δ is less than δ_c . Due to the effect of creep, δ will eventually reach δ_c as time increases. Therefore, the relationship between the initial applied K_c , K_i , and the initiation time t_i can be found via Equation (4.3) as:

$$K_i = \delta_c \sigma_o E_o t_i^{\frac{-(m+n)}{2}} \quad (4.9)$$

The propagation time t_p of the crack from initiation to fast failure can be evaluated by integrating Equation (4.5):

$$t_p = \int_{a_i}^{a_f} \frac{D^{1/q}}{K_c^{1/q}} da \quad \text{where } q = \frac{m+n}{2(1-m+n)} \quad (4.10)$$

where a_i is the initial crack length and a_f is the crack size at fast failure. If $K = \sigma_a Y \sqrt{a}$, where σ_a is the applied stress, t_p can be written approximately as:

$$t_p = \frac{2D^{1/q}}{Y^2 \sigma_a^2} \frac{q}{1-2q} \left[K_i^{(2q-1)/q} - K_{cf}^{(2q-1)/q} \right] \quad (4.11)$$

where K_{cf} is the value of K_c at failure. Although Y is generally a function of a/W , it has been pointed out by Atkins et al.[1985] that t_p is mainly controlled by a_i and σ_a . The error introduced by keeping Y constant and equal to the value of Y at a_i is negligible. For $K_i \leq 0.9K_{cf}$, Equation (4.11) can be rewritten as:

$$t_p \simeq \frac{2q}{1-2q} \frac{D^{1/q}}{Y^2 \sigma_a^2} K_i^{(2q-1)/q} = \frac{2q D^{1/q} \sigma_a^{-1/q} Y^{-1/q} (\sqrt{a_i})^{(2q-1)/2q}}{1-2q} \quad (4.12)$$

If $m \simeq n$, then $q \simeq n$. The total failure time is the sum of t_i and t_p . The incubation time is sometimes ignored for a conservative prediction of lifetime of the materials.

4.3 Review on the Application of Fracture Mechanics to Creep Fracture of Polyethylene

4.3.1 Inert Environments

The susceptibility to creep crack growth of polyethylene has become an important consideration in evaluating its performance under constant stress. Due to its extensive use in natural gas distribution networks operating at low pressure, it is crucial to ensure the integrity of the systems and an adequate service lifetime. A

specification has been adopted by British Gas to ensure a minimum of 50 years operation life of the pipe systems. The subject of fracture mechanics has been regarded as the best way of studying the slow crack growth mechanisms and it is supported by generous experimental data. The aim of the slow crack growth tests is to predict the long term performance of the material using relatively short term laboratory tests. Various test methods have been employed by different researchers. The data are normally extrapolated to the long term loading situation to estimate the permissible load levels. Since the time relaxation parameters, m and n , of σ_y and E vary little with temperature, higher test temperatures are often used to accelerate the crack growth and reduce the duration of the tests.

Birch et al.[1979] showed that an accurate prediction of the failure time of PVC pipes could be made by using the fracture mechanics approach. The fracture behaviour of a HDPE and a MDPE were studied by Gray et al.[1981]. They showed that Equation (4.9) provided a good model for lifetime estimation of the materials. However, they pointed out that the crack growth behaviour of the MDPE could not be described by the power law relation shown in Equation (4.5). Greig[1981] later observed that, at any temperature, the K_c approached a threshold value at slow crack speeds. He suggested that this value of K_c could be used as a design limit. The value of K_c at a crack velocity of 10^{-4} mm/hr were chosen as the threshold value. In a review of the role of fracture mechanics for design, Marshall[1982] showed that most laboratory tests would lead to ductile failure of the materials. The data obtained from such tests were not suitable for prediction of long term performance. The materials under constant loading tended to fail in a brittle manner. Marshall et al.[1982] further indicated that the residual stresses in the pipes which were caused by the manufacturing process could reduce the service lifetime.

The effects of temperature on the K_c - $\dot{a}$ characteristic of polyethylenes in distilled water were studied by Chan and Williams[1983]. They showed that the slopes of the K_c - $\dot{a}$ curves were the same for different temperatures. However, a slope of 0.25 was obtained instead of 0.1 as predicted from Equation (4.5). This high slope was attributed to the effect of different crack growth mechanisms. Notch ring tests have been used by Harnett et al.[1985] to estimate the long term performance of polyethylene pipe materials. They found that the results obtained from the notched ring tests at 23°C were not comparable with those obtained from stress rupture tests at 80°C. They concluded that the K_c - t_f curve from the notched ring tests had no value as a means of quality control on pipe production. Bhattacharya and Brown[1985] examined the shape and size of the damaged zone ahead the crack tip of a polyethylene during slow crack growth. The zone was found to be essentially planar at an applied stress below 12 MPa. The width of the zone increased rapidly above this value. The dimensions of the zone also increased with time. They suggested that the relationship between the velocity of the zone and the applied stress is governed by a simple power law. The effect of the flaw size on service lifetime has been investigated by Sandilands and Bowman[1986]. They showed that the lifetime decreased with increasing flaw size. The influence of residual stresses in polyethylene pipes was studied by Chaoui et al.[1987]. The stress components in the outer and inner shell were found to be compressive and tensile respectively. The tensile component tended to assist the outward crack propagation. The crack growth behaviour of the outward and inward crack was not a unique function of the energy release rate.

A different approach was adopted by Brown and co-workers. They argued that the initiation stage was important because the incubation time contributed a significant portion of the total failure time. The same observation has been noted by Gray et al.[1981]. Lu and Brown[1986 i,ii] related the applied K_c to the initial notch opening

rate. They showed empirically that the time to failure could be predicted from the initial notch opening rate, and the size of the damaged zone could be calculated from the crack opening displacement. They proposed that initiation and crack growth could be based on the same underlying mechanism. Brown et al.[1987] later estimated the failure time at the transition from ductile to brittle failure based on the empirical expressions. They indicated that the crack blunted at the transition due to a creep process as the failure time decreased. An 'overshoot' in the transition region was recorded because of the effect of crack tip blunting.

Bassini et al.[1988] and Lu et al.[1989] argued that the fracture parameter J should be used in characterising the slow crack growth of polyethylenes since the material was non-linear elastic. A better correlation between J and the initial notch opening rate was obtained. However, it must be pointed out that the strain rate sensitivity of the material was ignored in their analysis. The stress and strain field at the vicinity of the notch was measured by Wang and Brown[1989]. The results suggested that a constant stress field from the notch to the craze tip was obtained which agreed well with the assumption of the Dugdale model. The strain attained a maximum at the notch tip or the craze tip but the behaviour was complicated and not uniform. Qian et al.[1989] showed that there existed a threshold K_c at which no nucleation of damage at the crack tip was observed.

4.3.2 Aggressive Environments

It has been well known that cracks will initiate at a polymer surface when the material is under stress and in contact with an aggressive environment. The term *Environmental Stress Cracking* (ESC) was first introduced by DeCoste et al.[1951]. They studied the effect of environments in cracking of polyethylene cable sheath. They also developed a test method to determine the crack resistance. A number of bent specimens with surface flaws were placed in a bending clamp. The specimens

were then immersed into the environment at the desired temperature. The crack resistance was indicated by the number of specimens failed at a given time. This method was later accepted by the ASTM as a standard test method (ASTM D 1963-70). However, results obtained from these tests have little value in application to engineering problems. The application of fracture mechanics to ESC problems was demonstrated by Marshall et al. [1972]. It was proposed that the crack speed provided a suitable reference scale and controlled the K_c values. They showed that the K_c - $\dot{a}$ curves of polyethylenes were independent of thickness and loading. A mathematical model of slow crack growth was later developed by Williams and Marshall [1975] as described in Section 4.2.2. The experimental results and theoretical predictions were in good agreement.

Bandyopadhyay and Brown [1979] observed that for polyethylenes with low melt flow indices (MFI), $\dot{a}$ did not increase steadily with time. After the initial stage, the crack speed became constant with time and then started decreasing. They suggested that the crack speeds were only controlled by K_c in this case. The decreasing crack speeds were explained as the effect of transition from plane strain to plane stress failure. The results from further experiments (Bandyopadhyay and Brown [1981]) showed that the crack growth behaviour agreed well with the two stage craze model proposed by Williams [1978] which predicted a constant $\dot{a}$ at the transition region. In a study of the kinetics of ESC in high density polyethylene, Bubeck [1981] showed that the incubation time was more significant than the crack propagation time. He also found that the incubation time increased with decreasing applied stress. Chan and Williams [1983] performed a series of tests on ESC in polyethylenes. The slopes of the K_c - $\dot{a}$ curves determined from the experimental results were different from that predicted from Williams' model. They postulated that the failure might be controlled by a mechanism other than the critical COD failure criterion.

The effect of different environments, temperature and loading conditions on ESC of polyethylenes were investigated by Ogata et al.[1986 i,ii]. The K_c - $\dot{a}$ characteristics were found to vary with the type of environment, loading conditions and temperature. They concluded that the diffusion mechanism at the crack region should be controlled by a process of thermally activated molecular motion rather than hydrodynamics as described in the Williams model. The applicability of LEFM to ESC of low density polyethylenes has been reviewed by Tonyali et al.[1986]. They strongly argued against Williams' model and showed that a unique K_c - $\dot{a}$ curve could only be achieved at low K_c values. A deceleration of $\dot{a}$ at high K_c levels was also observed. In a later publication, Tonyali et al.[1987 i, ii] demonstrated that the transition crack speed could increase with increasing viscosity of the environment. Based on this, they deduced that the flow model was inadequate to describe the crack growth behaviour of low density polyethylene in organic liquids. They also proposed that the crack growth mechanisms depended on the flow rate of the swollen material and absorption properties of the polymers. Tonyali et al.[1987, iii] later studied the effects of detergent concentration on ESC in high density polyethylene. Their results revealed that the crack tended to propagate faster in environments with higher detergent concentration. Since the viscosity increased with increasing concentration, they inferred that crack growth in the transition region was not controlled by the hydrodynamic properties of the environment.

4.3.3 Problems Associated with Slow Crack Growth Testing

The use of fracture mechanics in studying slow crack growth problems has been generally supported in most of the literature. The relaxation and flow controlled crack growth models established by Williams seems to be commonly accepted by various workers. However, other crack growth mechanisms have also been proposed to account for the unexpected behaviour of the materials which cannot be described by Williams' model. The actual failure mechanisms are still far from clear. It is noticed

from the literature that the failure time tends to vary with the size of the specimen and the initial crack length. The propagation time of the crack should be proportional to the ligament size of the samples. Thus the failure time should be geometry dependent. It may be more appropriate to characterise the lifetime using the initiation time if it constitutes a significant portion of total failure time. In addition, the initiation time should depend on K_I only if the plane strain conditions are achieved. The specimen size used by other workers is generally small with a cross section area not greater than 5 mm X 15 mm. It is questionable that failure time measured by testing such small specimens can be used to predict the performance of a large structure. The duration of the laboratory tests have usually been reduced by increasing the test temperatures. Since the properties of polymers are sensitive to temperature changes, the mode of failure may be different from that at the actual service temperature.

The scatter of data from long term tests is usually considerable. Factors such as the sharpness of the notch, residual stresses in the samples and non-uniform density distribution across the plaques have direct effects on the experimental results. The detergent should have little effect on polyethylenes in a stress free state. It is still not clear how the detergent reacts with the polyethylenes under stress, but it is envisaged that in the presence of a detergent medium, the residual stresses in the material are sometimes sufficiently large to induce failure. In this study, some of these problems will be addressed but the subject as a whole is still very much in the development stage.

4.4 Micromechanisms of Slow Crack Growth Failure

The microscopic failure mechanism is crucial in understanding the cause of creep fracture in polymers. In order to ensure that the mode of failure is the same for the laboratory specimen and the actual structure, fractographic studies on the fracture

surfaces are often employed. This technique also provides further information about the fracture process.

4.4.1 Failure in Inert Environments

Lee and Epstein[1982] studied the fracture surfaces of polyethylene pipe samples. They showed that the fracture surfaces were macroscopically brittle but microscopically ductile. A fibrous texture was observed at the slow crack growth region. The texture changed from fine to coarse as the crack speed increased. They measured the height of the tufts and assumed that it was equal to half of the crack opening displacement δ . A power law relationship between δ and K_c was obtained. Such relationship could also be determined from the Dugdale model. Nevertheless, the measured fibril length was shorter than that predicted from the Dugdale model. It is suggested here that the fibrils may contract after failure due to recovery and this will cause shorter fibrils as observed in their results. Lustiger and Markham[1983] proposed that the brittle type slow crack growth behaviour was due to interlamellar failure. They claimed that the tie molecules (polymer chains which start and end in adjacent lamellae) are the major factor in determining crack growth resistance. In their model, the tie molecules disentangled and relaxed under long term stress and subsequently separated as the stress increased. The fibril structures were caused by pull-out of lamellae. They showed that polyethylenes with higher molecular weight were more resistant to crack growth because of an increase in the number of tie molecules. They also pointed out that preferential orientation of lamellae, increase in crystallinity and longer chain branches would reduce the fracture resistance of polyethylenes.

The micromechanisms of crack initiation in a polyethylene have been examined by Bhattacharya and Brown[1984]. They observed a dark halo at the crack tip under transmitted light. This dark region was due to the presence of pores in the deformed

material. The size of the halo increased with increasing applied stress but decreased with increasing specimen thickness. The halo size could not be calculated using the prevailing yield criteria. In thick sections, the porous region transformed to a fibrillar pattern. They suggested that the fibrils failed by localized shear yielding. Lustiger et al.[1986] catalogued various types of failure of polyethylene gas pipes operating in the field. The fracture morphology of the slow crack growth regions was similar to those obtained from laboratory test specimens. A craze zone ahead of the propagating crack was shown by Lustiger and Corneliussen[1987]. Lu et al.[1987] explained the fracture of polyethylene at room temperature as the result of disentanglement of the molecules in the fibrils. Brown et al.[1987] revealed that the damaged zone at the crack tip broadened at high applied stress. Crack blunting and microcracks were also evident in the damaged zone under this condition.

4.4.2 Failure in Aggressive Environments

Hannon[1974] studied the microscopic failure mechanisms of ESC in polyethylenes in detergent. The fracture surfaces appeared to be fibrous. He suggested that the failure proceeded by a void nucleation and growth process. The craze was stabilised by the plastically deformed fibrils between voids. Copolymers were found to be more resistant to crack growth than homopolymers. He pointed out that the large number of tie molecules in the copolymer led to its high fracture resistance. Since the detergent molecules were too large to enter the crystalline structure, he believed that the detergent penetrated into the intercrystalline regions and the voids. The amount of plastic deformation and strain hardening around the voids were reduced in the presence of detergent. Singleton et al.[1977] suggested that aggressive environments tend to weaken the cohesion between fibrils as well as the cohesion between lamellae. Brown[1978] considered that ESC was caused by stress induced swelling and plasticization of the amorphous regions where the bonding lamellae were normal to the applied stress. He indicated that the failure was interlamellar and

the amount of detergent absorbed increased under stress. The force required to drive the environment into the crack tip was considered to be a combination of the spreading coefficient of the environment on the polymer and the atmospheric pressure by Shanahan and Schultz[1978].

Interlamellar failure was observed by Bandyopadhyay and Brown[1978] on the fracture surfaces of a high density polyethylene under low applied stress. They showed that the failure was completely brittle and the banded structure of spherulites was sometimes misinterpreted as plastic deformation by other workers. The deformation of high density polyethylene single crystals in alcohol was investigated by Soni and Geil[1979]. The amorphous regions were 'solvated' and micro cracks were found in the crystalline structures. The amount of alcohol absorbed by the polyethylene was observed to increase with increasing applied stress. Bubeck and Baker[1982] demonstrated that polyethylenes with small spherulites had poor ESC resistance due to greater spherulitic boundary slip. The change of fracture morphology from interlamellar to fibrillar as K_{Ic} increased was observed by Chan and Williams[1983]. They suggested that the crack growth was due to a diffusion void growth process. Small interlamellar cracks in polyethylenes under stress were reported by Shinozaki and Howe[1986]. The microcracks were considered to nucleate to form large scale fracture. The surface energy and the stress necessary to open the cracks were assumed to be lowered in the presence of detergent.

It can be inferred from the literature that interlamellar failure accounts for the brittle type slow crack growth fracture of polyethylenes. An environment may act as a catalyst to accelerate the failure by plasticizing the intercrystalline regions or tie molecules. However, little has been revealed concerning the reactions between

environments and intercrystalline regions and how the effectiveness of the environment as a plasticizing agent changes with loading conditions.

4.5 Effects of Residual Stresses

It will be noticed from the review that residual stresses can considerably affect the slow crack growth behaviour in polyethylenes (Chaoui et al.[1987]). Residual stresses are commonly caused by non-uniform solidification of the polyethylene during the cooling process. Since residual stresses can sometimes be significant enough to initiate stress cracking in polyethylenes, it is necessary to measure the residual stresses in the plaques which are used for the slow crack growth tests. The residual stress distribution across the thickness of the plaques was measured using the progressive removal layer technique(see Appendix I). Removal of a thin layer from the surface of a beam will offset its stress equilibrium. The residual stresses can be evaluated from the changes of curvature of the beam. The materials tested were the 5731 and 8621 graded high density polyethylenes. The residual stresses in plaques of various thickness are shown in Fig.4.4 and Fig.4.5. The stresses were found to be compressive on the surfaces and tensile in the middle. It can be seen that the magnitude of the stresses reduces as specimen thickness decreases. For specimen thickness less than 10 mm, no noticeable residual stresses were recorded for the 8621 specimens. The maximum compressive stresses measured were 2 MPa and 1.5 MPa for the 5731 and 8621 samples respectively. The maximum tensile residual stress was found to be approximately 1MPa for both materials.

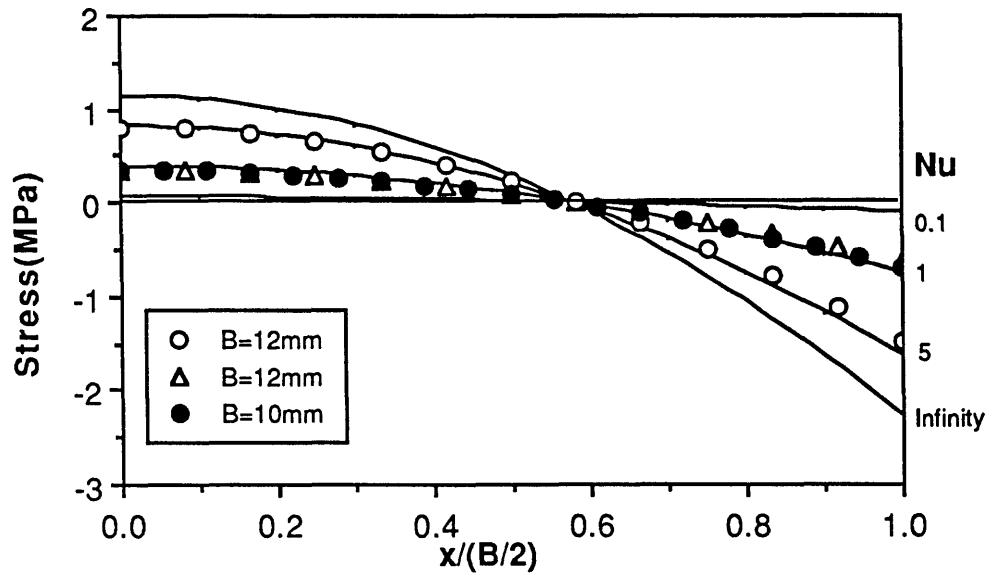


Fig.4.4 Residual stress distribution of 8621 samples
(x =distance measured from the centre of the plaque, B =specimen thickness)

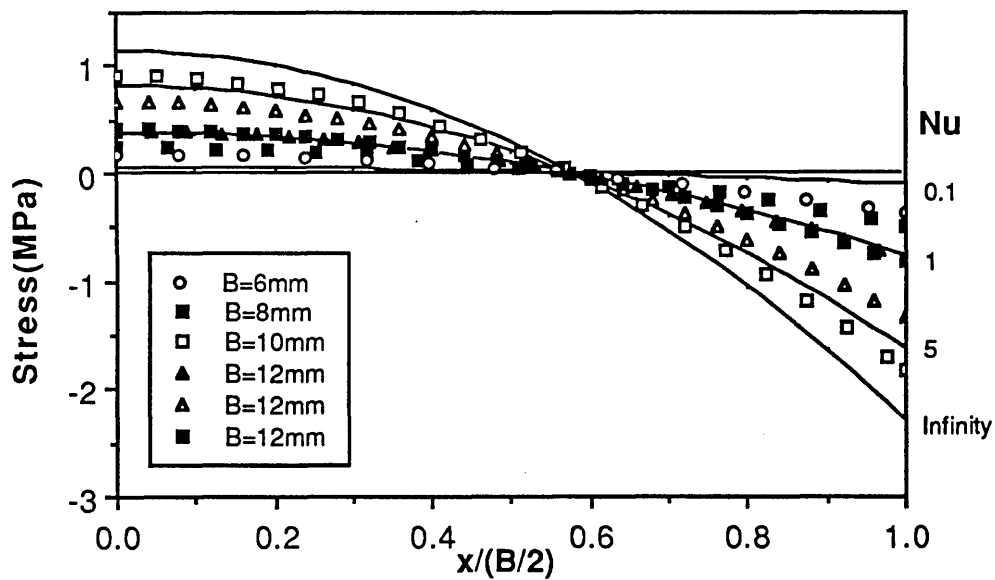


Fig.4.5 Residual stress distribution of 5731 samples
(x =distance measured from the centre of the plaque, B =specimen thickness)

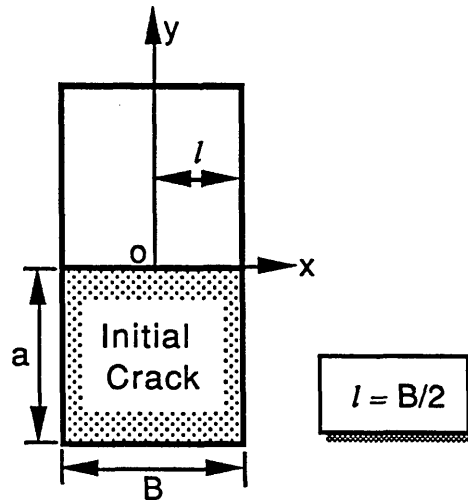


Fig.4.6 The co-ordinates of a cracked specimen

Due to the presence of the residual stresses, the actual K_c value, K_s , of a notched sample will be different from the apparent K_c value, K_{app} , calculated from the constant applied stress, σ_a . If the specimen is fabricated as shown in Fig.2.8, the value of K_s can be estimated by simply considering the stress distribution across the specimen as:

$$\sigma_s = \sigma_a + \sigma_r(x) \quad (4.13)$$

σ_s is the actual stress in the specimen and $\sigma_r(x)$ is the residual stress at a distance x from the centre of the specimen as illustrated in Fig.4.6. From Equation (2.12) and (2.14),

$$G_s = \frac{K_s^2}{E} = \frac{Y^2 \sigma_s^2 a}{E} \quad (4.14)$$

By assuming the crack shape is self-similar, the average value of K_s , $\tilde{K}_s$, can be calculated as:

$$\tilde{K}_s^2 = \frac{Y^2 a}{l} \int_0^l [\sigma_a + \sigma_r(x)]^2 dx \quad (4.15)$$

where $l=B/2$.

An analytical solution of $\sigma_r(x)$ has been developed by Williams[1981]. The solution can be simply expressed in the form:

$$\sigma_r(x) = \chi \left[\frac{l}{3} - \left(\frac{x}{l} \right)^2 \right] \quad (4.16)$$

χ is mainly a function of Nusselt number, $Nu=hl/k$ (h =surface heat transfer coefficient, k =thermal conductivity). The calculated residual stress distributions for typical high density polyethylenes at various values of Nu are also shown in Figures 4.4 and 4.5. By substituting Equation (4.16) into (4.15), the following expression can be obtained:

$$\frac{\tilde{K}_s}{K_{app}} = \sqrt{1 + \frac{4}{45} \left(\frac{\chi}{\sigma_a} \right)^2} \quad (4.17)$$

The full analysis is given in Appendix II. The variations of $\tilde{K}_s/K_{app}$ with Nu are plotted on Fig.4.7 for different applied stress levels. It can be found from Fig.4.4 and Fig.4.5 that the values of Nu are between 1 to 5 for both polyethylenes. The difference between $\tilde{K}_s$ and K_{app} is small within this range of Nu values unless σ_a is below 2 MPa. Bhattacharya and Brown[1985] suggested that σ_a should be less than one-half of the yield stress in order to achieve the characteristic long term brittle failure. As the yield stress of both materials are about 20 MPa, the error in the calculation of K_c due to the effect of residual stresses is small. However, it must be pointed out that this approximation is an average of the local K_c values across the specimen thickness and the crack is assumed to be straight. In reality, the tensile residual stress gives a higher local K_c value in the middle of the specimen. On the other hand, the compressive stress on the specimen surfaces tends to close the crack. Such a stress distribution will promote crack tunnelling as the crack propagates, further increasing the local K_c value at the centre of the specimen.

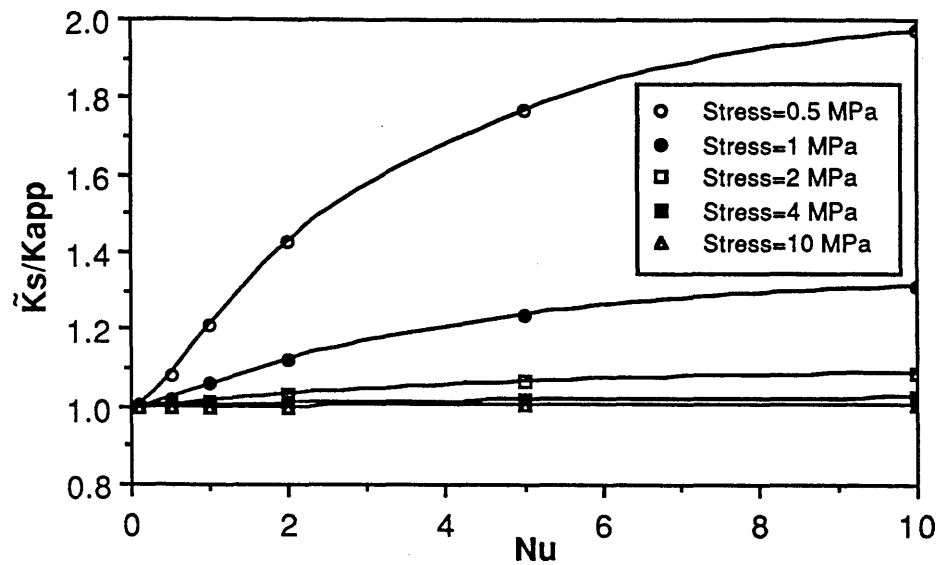


Fig.4.7 Variation of $\tilde{K}_s/K_{app}$ with Nusselt number

4.6 Experimental Considerations

4.6.1 Notching Techniques

A typical K_c - $\dot{a}$ curve for polyethylene is shown schematically in Fig.4.8. Since the K_c - $\dot{a}$ relationship is assumed to be a material property, it should be unique for a particular material. The relaxation and flow controlled crack growth models can only be used to describe the crack growth behaviour in region B. The sudden increase in $\dot{a}$ in region C is due to fast failure. The region A has been referred as the *anomalous* region by Chan[1983] where the K_c - $\dot{a}$ curve varies with applied K_c level. He suggested that this is due to crack tip development.

Since cracks commonly grow from the inherent flaws of the material, thus it is necessary to introduce an initial notch with sufficient sharpness in order to simulate a *natural* crack. The incubation time of the crack will also depend on the acuity of the

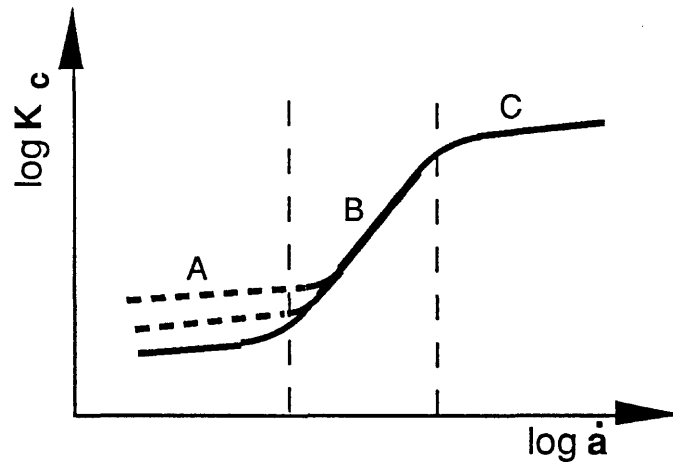


Fig.4.8 Schematic diagram of a K_c - a curve for polyethylene

notch but the exact relationship is not known. Chan[1983] pre-developed the razor notched crack by overloading the specimen and then reloaded at the required K_c level. Although this method appears to be well founded, it is not certain that the damaged zone developed by overloading will be the same as that developed at a lower K_c level. In addition, it is difficult to control the size of the pre-developed damaged zone. If it is larger than the zone size at low K_c levels, the residual strain and stresses in the zone will affect the subsequent crack initiation. The advantages of this method are overshadowed by the uncertainties. A *natural* crack can be induced by cooling the specimen to a temperature at which brittle fracture can be obtained, a razor blade is then tapped into the machined notch to propagate the crack in a brittle manner. This method will produce a nearly damaged zone free crack. However, the crack propagation is difficult to control and the success rate is extremely low.

The razor notching technique is widely used for introducing an artificial crack. This method provides a higher degree of control over the crack sharpness and crack size. Nevertheless, Marshall[1987] argued that the razor actually forced the material to open which would cause plastic deformation at the crack tip and a blunted notch would be resulted. Leis et al.[1987] showed that all razor notched specimens need some initial force to open the notch wedge. This indicates that internal stresses are

induced at the notch faces by razor notching techniques. Three notching methods have been investigated here. The final 3 mm of a machined notch was further sharpened i) with a single point flycutter, ii) by sliding a new razor blade across the notch, iii) by pushing/tapping the razor blade into the notch. All specimens were loaded at the same K_I level. The micrographs of the notches are shown Fig.4.9, Fig.4.10 and Fig.4.11. The flycutter notch and the sliding razor notch appear to be much sharper and deformation at the notch root is small. The pushing razor notch illustrated in Fig.4.11 appears to be inferior to the other notches. Therefore, the flycutter and sliding razor techniques have been used to introduce the initial cracks.

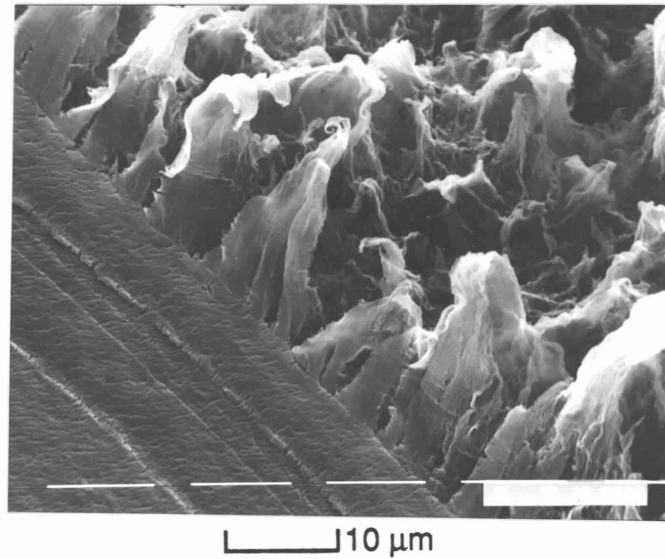
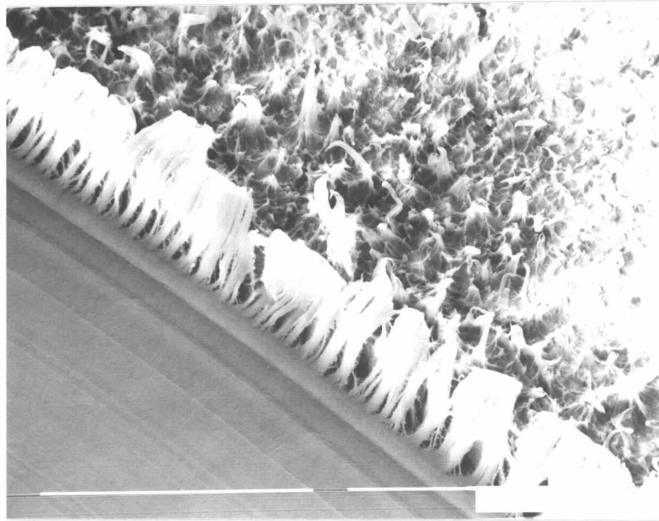
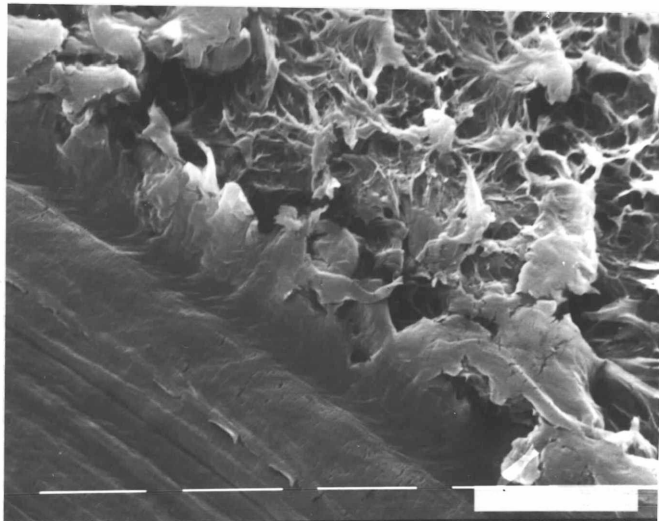


Fig.4.9 Micrograph of an initial notch induced by sliding a razor



100 μm

Fig.4.10 Micrograph of an initial notch induced by a flycutter



10 μm

Fig.4.11 Micrograph of an initial notch induced by pushing a razor

4.6.2 Crack Length Measurements

Crack length is one of the crucial parameters in the analysis. During the loading period, the crack length was usually monitored using a travelling microscope or time lapse photography by other workers. These methods involve crack length

measurements from the sides of the sample. Since plane stress conditions prevail near the sides of the specimen, while the stress state at the centre is plane strain, crack initiation will take place first at the centre and then spread to the sides. This causes crack front bowing which is also promoted by residual stresses in the specimen. For opaque materials, this renders crack lengths measured from the sides of the specimen inaccurate. Bandyopadhyay et al.[1981] and Chan[1983] observed a *dark zone* in front of the crack, which they interpreted as the crack tip. They found that it tunnelled a distance at the centre of the specimen as the crack at the sides appeared to be stationary. The *dark zone* is believed to be comprised of the crack, the craze zone and the deformed material ahead the craze tip. If the crack shape is not self-similar and the damaged zone size increases as the crack propagates, the growth speed of the *dark zone* will overestimate the crack speed.

An alternative is to measure the crack growth indirectly from the change of compliance of the specimen. The compliance method has been adopted by Taylor[1983] and Leis et al.[1987], and close agreement between the final predicted and measured crack length was obtained. If the change of compliance is assumed to be due to crack growth and creep only, crack lengths can be evaluated using the inverse load-line compliance function developed by Bakker[1990]:

$$\delta_{cr} = \frac{I}{1 + \sqrt{\frac{0.9875(S/W)^2}{E^*B_e C_{cr}(exp)}}} \quad (4.18)$$

$$\frac{a}{W} = 2.24 \times 10^{-3} + 0.2904\delta_{cr} - 0.4573 \delta_{cr}^2 + 1.3756 \delta_{cr}^3 - 1.1319\delta_{cr}^4 \quad (4.19)$$

where B_e is the effective thickness as defined in Equation (3.28) and E^* is the effective creep modulus (see Equation(4.22)). $C_{cr}(exp)$ is the measured compliance

due to the the presence of the crack only, i.e. $C_{cr}(exp) = C_{measured} - C_o \cdot C_{measured}$ and C_o are the measured load line compliance and load line compliance of the non-cracked beam. C_o can be evaluated using:

$$C_o = \frac{I}{4E^*B_e} \left(\frac{S}{W} \right)^3 \left[1 + 2.85 \left(\frac{W}{S} \right)^2 - 0.84 \left(\frac{W}{S} \right)^3 \right] \quad (4.20)$$

A correction procedure is employed to improve the accuracy of the crack length prediction and is given as:

$$\left(\frac{a}{W} \right)_{corrected} = \frac{a}{W} + \frac{C_{cr}(exp) - C_{cr}(a/W)}{\frac{dC_{cr}(a/W)}{d(a/W)}} \quad (4.21)$$

The first estimate of a/W from Equations (4.18) and (4.19) is used to compute $C_{cr}(a/W)$ via the following expression:

$$E^*B_eC_{cr}(a/W) = \left(\frac{S}{W} \right)^2 \frac{(a/W)^2}{(1-a/W)^2} [8.9 - 33.717(a/W) + 79.616(a/W)^2 - 112.952(a/W)^3 + 84.515(a/W)^4 - 25.672(a/W)^5] \quad (4.22)$$

E^* is equivalent to the time dependent E defined in Equation (4.1b). The time dependence, n , can be estimated from the initial part of the compliance versus time curve. Equations (4.20) and (4.22) can be combined, and, if no crack growth is assumed; written in the form:

$$C_{measured} = C_{cr} + C_o = \frac{t^n}{E_o} [\text{Constants}] \quad (4.23)$$

Then the slope of the $C_{measured}$ versus time curve should correspond to the value of n as shown in Fig.4.12. Assuming the polyethylenes are linear viscoelastic, the value of n will be equal to 0.1. Since the change of creep compliance is due to crack growth

only, the point where deviation from linearity occurs can be considered as the initiation point, see Fig.4.12. By measuring the initial crack length after completion of individual tests, the value of E_o can be computed from Equation (4.23) with the corresponding compliance and time. The procedures are similar to those described in Section 3.7.4.

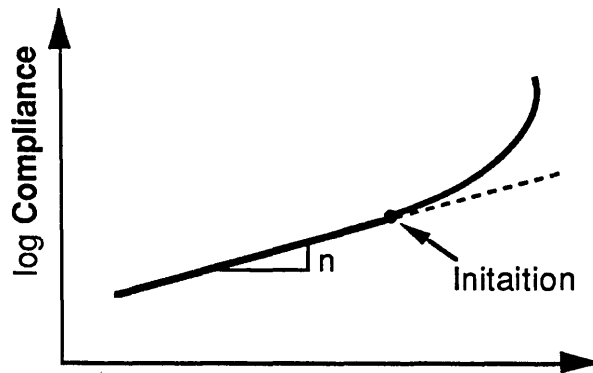


Fig.4.12 Schematic diagram of the compliance versus time curve for a notched specimen

Since the crack length is estimated from the resultant compliance of the specimen, it can be regarded as an average value across the specimen thickness. Hence the effect of crack front curvature has been taken into account. Since optical methods are tedious and the interpretation of crack size is subjective, the compliance technique is considered to be more appropriate.

4.7 Experimental Details

An experimental programme has been undertaken to elucidate the validity of the LEFM analysis to slow crack growth problems in polyethylenes. The effects of specimen size and side grooving on the initiation time and the kinematics of slow crack growth have been investigated. One of the aims of this study is to predict the long term performance of the polyethylenes under static loading conditions in an inert environment. Although ESC is an important consideration, detergent has been used

here simply for accelerating the failure of specimens, and it will be covered only briefly in this study.

4.7.1 Materials and Specimen Preparation

Two grades of polyethylenes considered were 8621 and 5731 used in the fracture toughness studies, see Table 3.1 in Chapter 3. The molecular weight distributions for both polyethylenes have been kindly measured by the supplier, DSM Kunststoffen BV. Light scattering and gel permeation chromatography (GPC) techniques were employed. The results are given in Table 4.1.

Designation	Weight Average Molecular Mass M_W (kg/mol) Light-scattering	Average Number Molecular Mass M_N (kg/mol) GPC
8621	200	10
5731	190	15

Table 4.1 Molecular weight of the polyethylenes

They pointed out that the degree of scatter in the data from both methods was considerable. They suggested that $M_W = 200$ kg/mol and $M_N = 20$ kg/mol for both materials.

SEN bend specimens were used and the preparation procedures described in Section 2.7.1 were followed. Specimens were cut from sheets of various thicknesses and notched to different a/W ratios. For the side-grooved specimens, the total thickness reduction was less than 20%. All specimens were loaded in three point bending with a span to width ratio of 4. The range of specimen size used is given in Table 4.2.

Thickness, B (mm)	Width, W (mm)	a/W
5	30	0.5
7	30	0.5
9	30	0.5
10	30	0.1 - 0.7
10	40	0.5
10	25	0.5
10	15	0.5

Table 4.2 Range of specimen size used for the long term tests

In order to relax the stresses at the crack tip induced by the notching process, the notched specimens were stored at room temperature for at least one week before testing took place. The detergent used for assisting failure was Arkopal N-090 supplied by Hoechst U.K.. The detergent is non-ionic and the structure is shown in Fig.4.13.

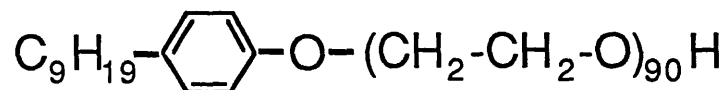


Fig.4.13 Structure of the detergent

4.7.2 Apparatus and Testing Procedures

The layout of the apparatus for slow crack growth tests is shown schematically in Fig.4.14. The load was applied centrally to the specimen. The radii of the supports and the loading pin were 5 mm. Some of the loading brackets were modified to provide a side view of the crack growth as illustrated in Fig.4.15. A front coated silver mirror was mounted on the loading fixture underneath the specimen at an angle of 45°, so that the crack tip could be observed directly using a microscope. The required load, P , for a particular applied K level was calculated using Bakker's K -expression[1990]:

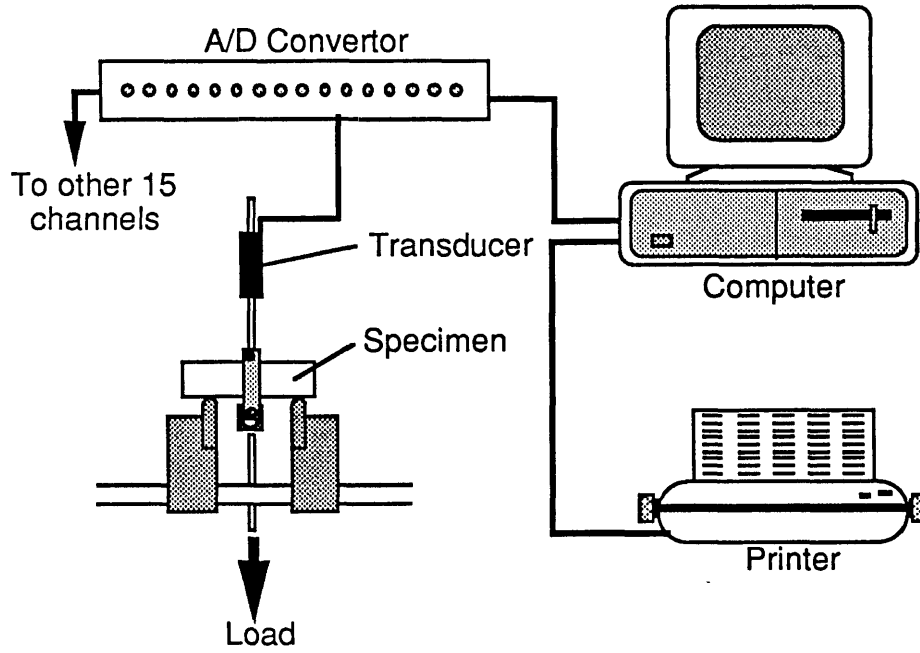


Fig.4.14 The layout of the apparatus for slow crack growth tests

$$\frac{K_c B \sqrt{W}}{P} = \frac{S}{W} \frac{\sqrt{\alpha}}{(1-\alpha)^{3/2}} [8.9 - 50.576\alpha + 176.091\alpha^2 - 361.995\alpha^3 + 423.873\alpha^4 - 259.484\alpha^5 + 64.181\alpha^6]^{1/2} \quad (4.24)$$

where $\alpha = a/W$.

The load-line displacements were monitored by a LVDT. The plunger of the transducer was placed on the top of the loading pin and located immediately above the loading point. The load was supported by a laboratory jack prior to commencement of the experiment. The initial displacement of the transducer was recorded and taken as the threshold displacement. The subsequent deflection of the sample during loading was obtained by subtracting the threshold displacement from the measured displacement. There were sixteen experimental stations in total and each transducer was calibrated independently using a set of engineering slip gauges. By plotting the output voltage of the transducer versus known deflections, the calibration factor could be determined from the slope of the curve. The resolution of the transducers was 0.04 mm.

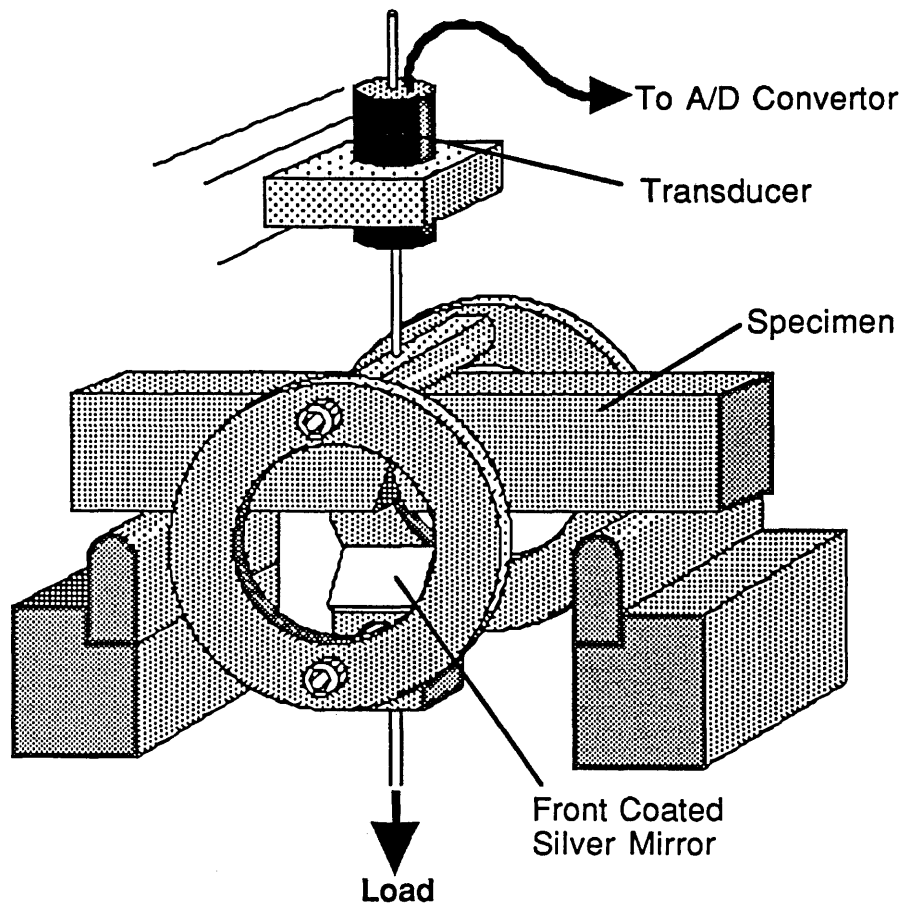


Fig.4.15 Test rig with a modified loading bracket

The transducer outputs were fed to a microcomputer via an analog to digital (A/D) convertor. The necessary software was developed by the Polymer Engineering Group at Manchester Polytechnic, England. The computer scanned through all the channels and the approximate scan time for each channel was 0.1 sec. During the course of the scan, the computer was programmed to compare the current output of the transducers with that it last recorded. If the current displacement was larger than the prescribed increment, 0.05 mm to 0.1 mm here, the computer would register the new displacement and time. The last record would be updated with the new one. If two consecutive increments were recorded in the same channel within 0.01 hr, the computer would concentrate on that channel until the readings became steady. This

would improve the resolution of the data when fast fracture occurred. Crack lengths were evaluated from the load-line compliance as described in Section 4.6.2. The corresponding K_c values were computed using Equation (4.24). From the crack length versus time curve, a straight line was fitted using the least square method through every five data points along the curve and the crack speeds were determined from the slopes of these lines.

If the initiation time was greater than 2000 hrs, then detergent would be used to reduce the lifetime of the sample. The specimen and detergent were sealed inside a thin polyethylene bag. All the tests were performed at room temperature.

4.8 Results and Discussion

4.8.1 General Observation

The 5731 polyethylene showed much stronger resistance to slow crack growth than the 8621 material. This may be due to the low MFI of the 5731 as suggested by Bandyopadhyay et al.[1979]. The life span of a 5731 specimen was normally over 5000 hours in air in order to obtain brittle failure. Although detergent was employed to accelerate the crack growth rate, the duration of the tests still exceeded 4000 hours. Due to the extreme toughness of this material, little data could be gathered. Most of the results reported here are based on the 8621 material.

Typical compliance versus time curves are shown in Fig.4.16. The shape of the curves is the same as that illustrated in Fig.4.12. A crack incubation period was observed before the crack initiated. Direct observation at the crack tip revealed that the original crack was first stretched and a fibrillar or craze-like structure was then developed during the incubation period. The point of deviation from linearity as illustrated in Fig.4.16 coincided with the first sign of breakage of fibrils at the

stretched crack tip. The crack was observed to initiate first at the centre and extended to the sides of the specimen. Crack tunnelling occurred in all plain-sided specimens. A layer of highly drawn material was formed on both sides of the specimen or the notch roots of the side grooves, see Figs.4.17. The size of the shear lips was smaller in the side-grooved specimens.

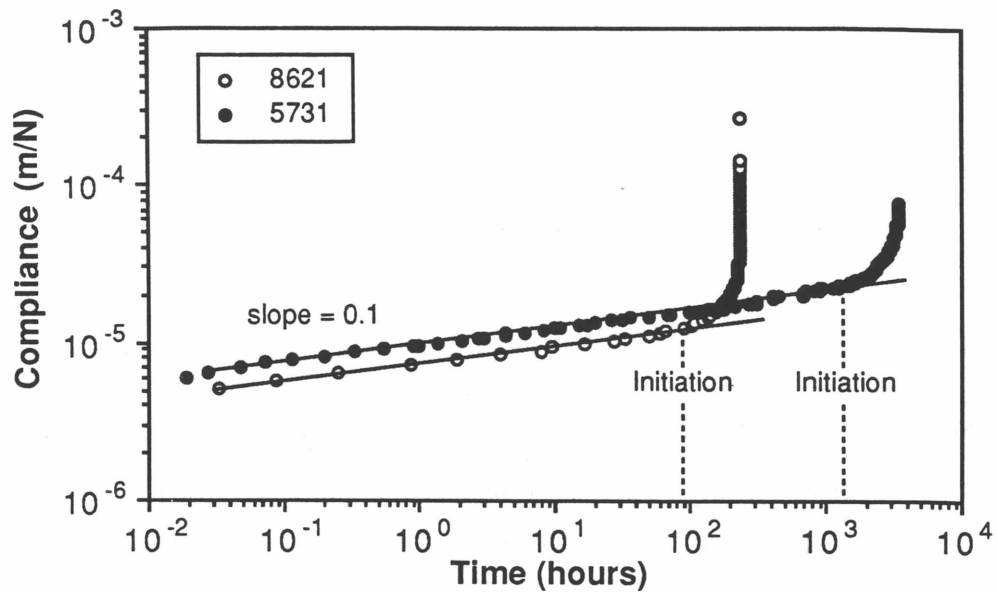
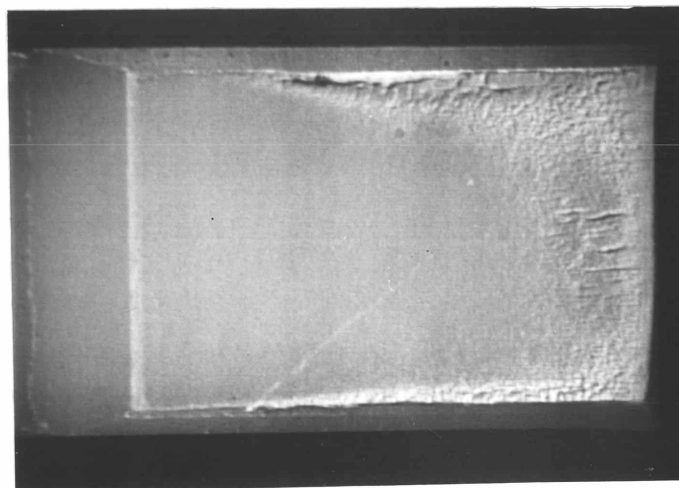
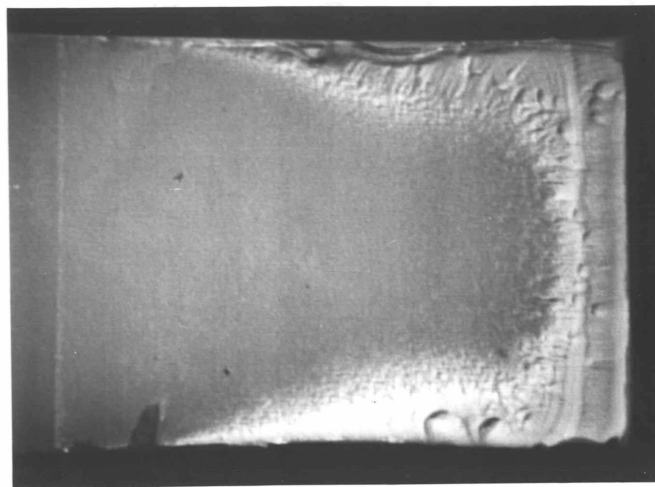


Fig.4.16 Experimental compliance versus time curves



(a)



(b)

Fig.4.17 Fracture surface of 8621 specimens. a) Side-grooved, b) Plain-sided.

Some tests were terminated before fast failure occurred and the specimens were broken open by impact after immersion in liquid nitrogen. Thumb nail shaped cracks were obtained for plain-sided specimens. Side-grooving tended to reduce the crack front curvature. This indicated that the crack propagated at a much slower speed at the sides of the specimen than at the centre. Nine measurements were taken at equidistant points across the thickness of the specimen. The average value was regarded as the measured crack length. The correlation of the measured and predicted crack lengths is shown in Fig.4.18. It can be seen that the agreement is good. The maximum error in crack length prediction is about 10%, which in turn gives a 5% error in the calculation of K_c . The inconsistency was, partly if not mainly, caused by the presence of shear lips. A smaller crack size was often predicted since the shear lips provided a measure of support which effectively reduced the compliance of the specimen. Nevertheless, the errors are considered to be small and are overlooked in the subsequent analysis.

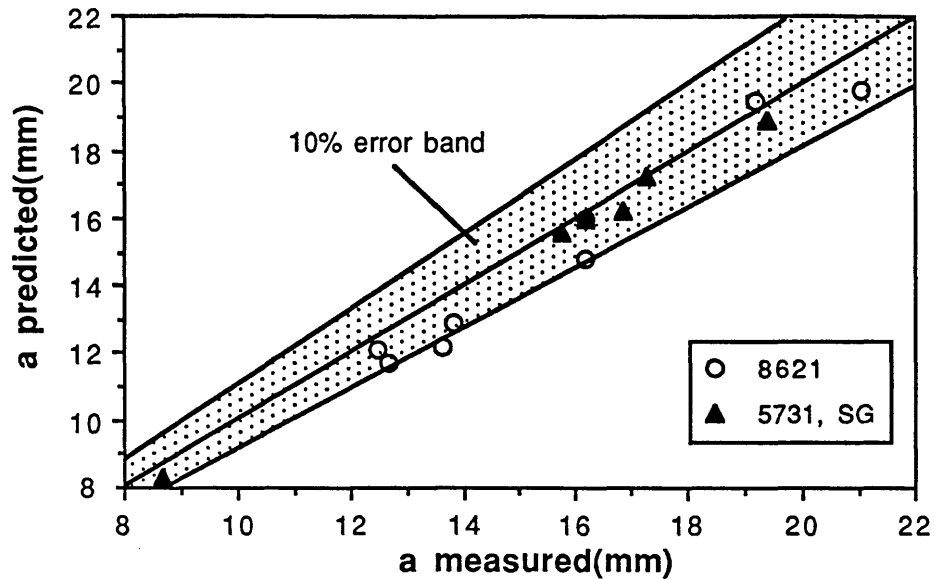


Fig.4.18 Correlation of the measured and predicted crack lengths
(SG =side grooved samples)

4.8.2 Initiation and Failure Time

The effect of applied K_c levels, K_i , on initiation time, t_i , and failure time, t_f , has been examined. Specimens with the same dimensions ($B=10$ mm, $W=30$ mm, $S=120$ mm) were loaded at different K_i levels. The 5731 specimens were side-grooved and the 8621 ones were plain-sided. The variation of t_i with K_i and t_f with σ_a are given in Fig.4.19 and Fig.4.20 respectively. Specimens with high K_i failed in a ductile manner. At a K_i level of $0.3 \text{ MPa}\sqrt{\text{m}}$ for the 8621 samples and $0.5 \text{ MPa}\sqrt{\text{m}}$ for the 5731 samples, no noticeable crack growth was found after 10000 hours. It can also be noted that the data obtained from the flycutter notched samples are consistent with those from slicing razor notched specimens. For the 8621 data the curves were separated into ductile and brittle regions. The transition regions are shown as hatched lines because the exact form of the curves in these regions is not known due to lack of data. The slopes of the K_i versus t_i curve and σ_a versus t_i curve in the brittle region are -0.11 and -0.12 respectively. These values agree well with those predicted from Equation (4.9) and Equation (4.12). Further, the $\sigma_a - t_f$ curves are

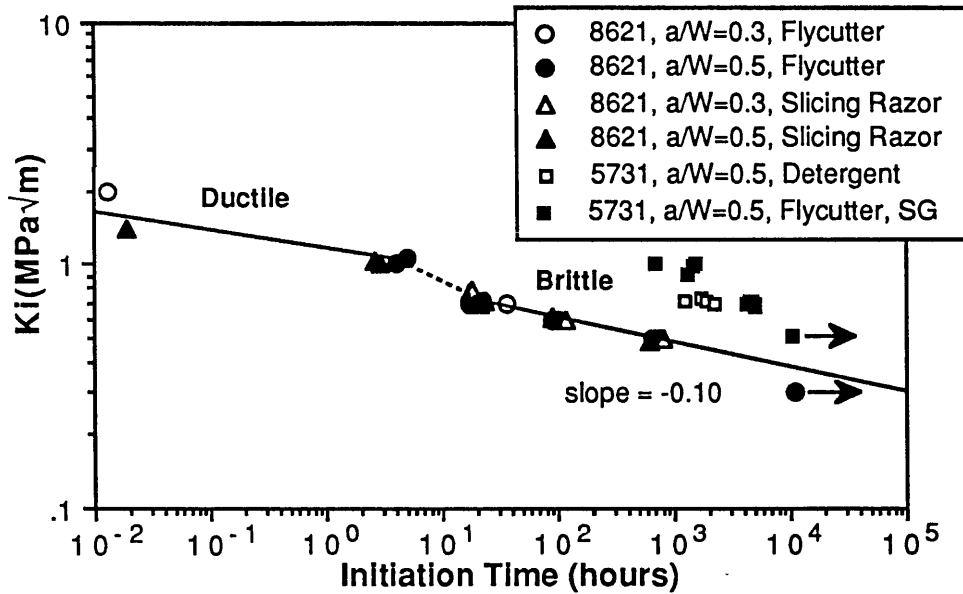


Fig.4.19 Applied stress intensity factor against initiation time
(SG=side-grooved samples)

crack length dependent as suggested in Equation (4.12). This crack length dependence has also been observed by Brown et al.[1987]. However, it will be shown in a later section that the slope of the $\sigma_a - t_f$ curve is dominated by the power dependences of the initiation time and crack tip development time. t_i and t_f for the 5731 specimens were much higher than those of the 8621 samples. The presence of detergent reduced t_i and t_f and the variations of t_i and t_f are due to the difference in detergent concentration which will be discussed in a later section. Although the slopes of the curves appear to be steeper, there are insufficient data to comment on the behaviour of the 5731 material.

4.8.2.1 Effects of Specimen Size and Side-Grooving

The effects of specimen size and side-grooving were investigated by loading plain-sided and side-grooved specimens of various sizes at the same K_i level. The range of specimen sizes tested is given in Table 4.2. By referring to Fig.4.19, a K_i level of $0.6 \text{ MPa}\sqrt{\text{m}}$ was chosen for the 8621 material since brittle failure could be obtained. The effects of specimen width W , initial crack length a_i , and thickness B on t_i and t_f

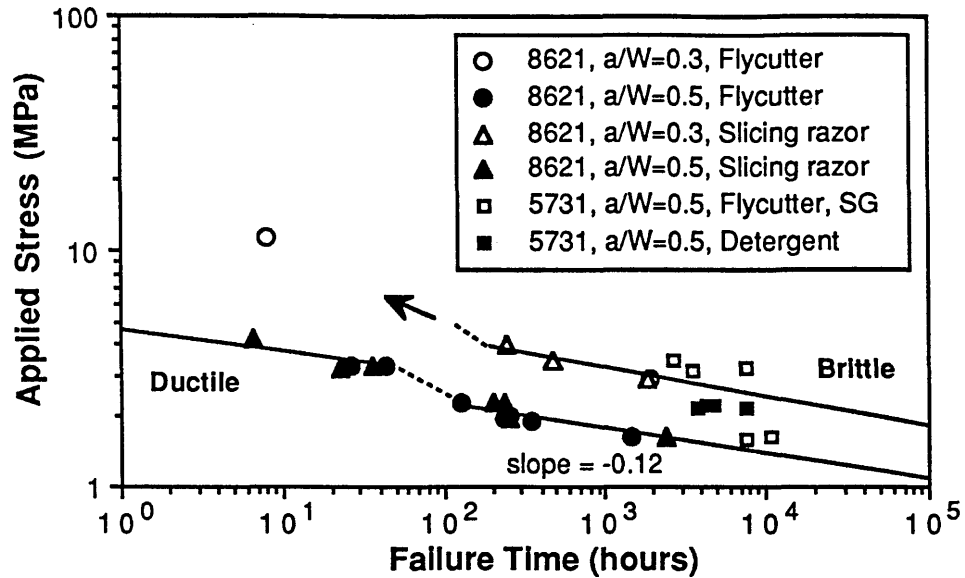


Fig.4.20 Applied stress against failure time (SG=side-grooved samples)

for 8621 are shown in Fig.4.21, Fig.4.22 and Fig.4.23 respectively. Changes in W and a_i actually reflect the changes in uncracked ligament size. It can be found from Fig.4.21 and Fig.4.22 that t_i and t_f increase with increasing ligament length. From Fig.4.23, t_i remains fairly constant as B changes. This implies that the specimens used here were sufficiently thick for the crack to initiate under plane strain conditions. t_f increases as specimen thickness decreases but it decreases for the thinnest samples. In all cases, side-grooving tends to reduce the failure time but it has little effect on the initiation time. It will be shown later that the crack travels faster for the side-grooved samples because of the reduction in shear lip size. In addition, the displacement at failure reduced with increasing specimen thickness and with side-grooving, but it rose with increasing ligament length. It is interesting to note that, for the same K_i level, the difference of the t_i and t_f between samples of varied size can be more than 80%. This suggests that K_i is not the only parameter which controls t_i and t_f , and Equation (4.9) is only applicable when all the specimens have the same size.

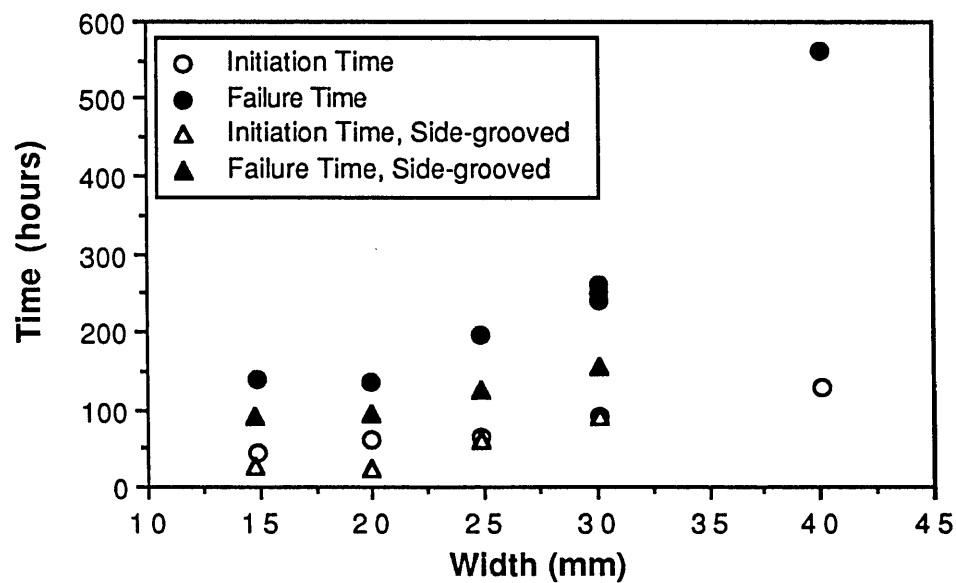


Fig.4.21 Variation of t_i and t_f with specimen width for 8621.

$a/W=0.5$, $B=12$ mm, $K_I=0.6$ MPa $\sqrt{m}$.

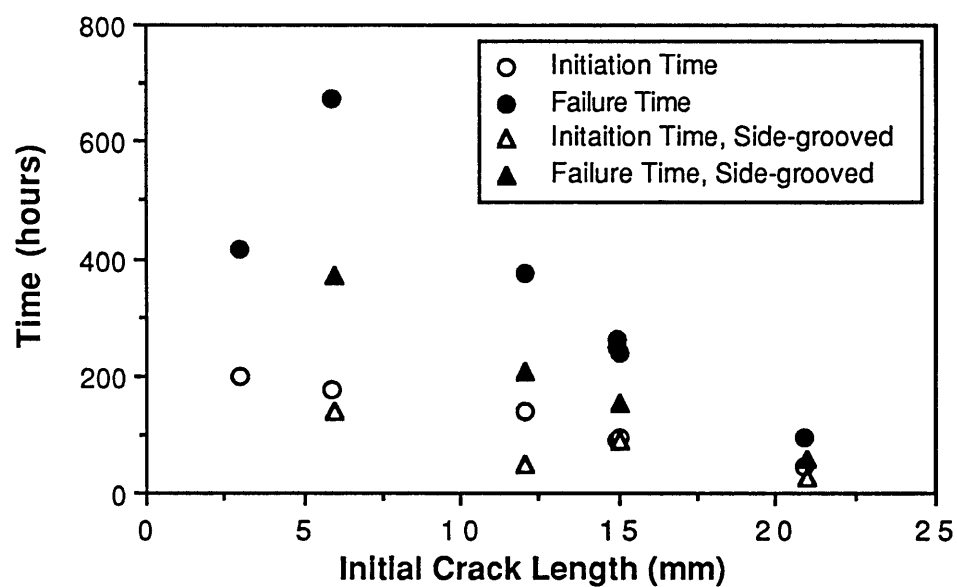


Fig.4.22 Variation of t_i and t_f with initial crack length for 8621.

$W=30$ mm, $B=12$ mm, $K_I=0.6$ MPa $\sqrt{m}$.

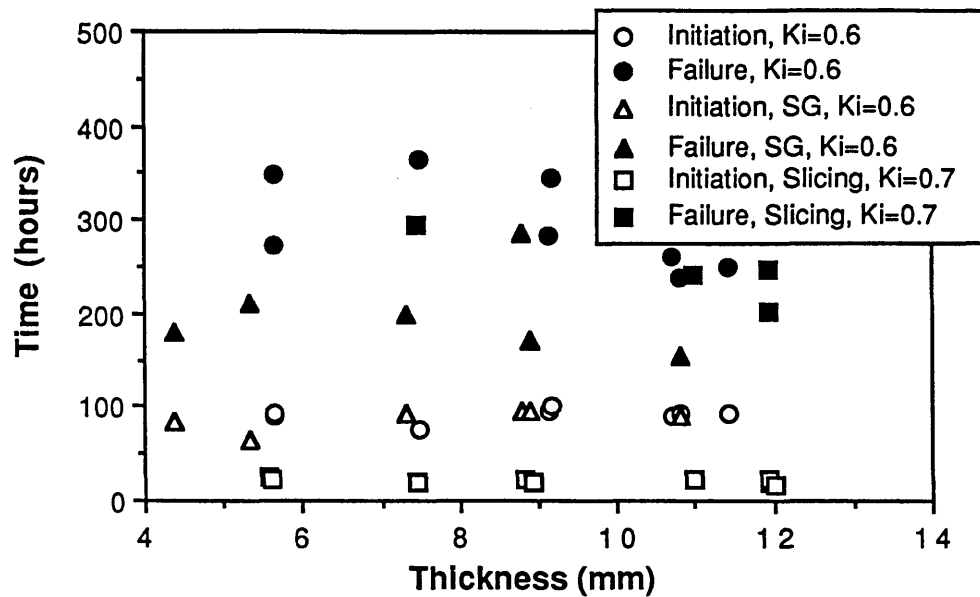


Fig.4.23 Variation of t_i and t_f with specimen thickness for 8621.
 $a/W=0.5$, $W=30$ mm, SG=side grooved samples.

K_I (MPa $\sqrt{m}$)	B (mm)	B _N (mm)	W (mm)	a/W (mm)	t_i (hrs)	t_f (hrs)	σ_a (MPa)	σ_N (MPa)	Final a (mm)
1.0	11.5	9.9	25.0	0.5	670	2701	3.5	14.1	failure
	11.1	9.2	30.0	0.5	1548	3479	3.2	12.6	failure
	5.8	5.8	29.8	0.5	1477	7003+	3.14	12.8	17.25
0.7	12.9	11.5	30.0	0.6	4364	7565	1.61	10.1	failure
	12.9	12.9	30.1	0.6	4799	10922+	1.62	10.0	19.39
	11.1	9.6	30.0	0.5	5012+	10800+	2.3	9.1	15.76
	11.8	10.2	15.2	0.5	5043	7479	3.2	12.6	failure
	11.7	11.7	15.3	0.5	4172	7675+	3.3	12.7	8.67

Table 4.3. Summary of specimen size effect for 5731 samples

Due to the extreme crack growth resistance of the 5731, the intended experimental programme for this material was far from completed. The results are summarised in Table 4.3. For the same K_I level, the results indicate the same trend on the size

effects as observed for the 8621 samples. Side-grooving tended to reduce the failure time considerably for this material. However, more tests will be necessary before any conclusions on the size effects on t_i and t_f of this material can be drawn.

4.8.2.2 The Net Section Stress as a Characterising Parameter

It has been suggested by the data that t_i and t_f are not solely governed by the K_i levels. By keeping the same K_i level, σ_a and the net section stress σ_N were the parameters that changed as specimen dimensions varied. σ_N is defined as $\sigma_N = 3PS/[2B(W-a)^2]$. Variations of σ_a and σ_N with W , a_i and B for 8621 are shown in Fig.4.24, Fig.4.25. Fig.4.26 respectively.

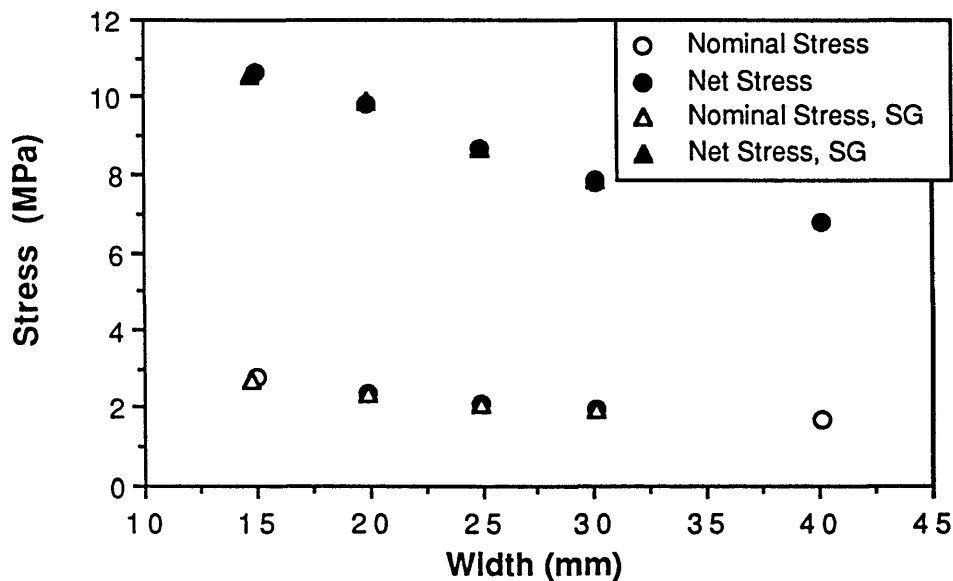


Fig.4.24 Variations of nominal stress and net section stress with specimen width for 8621.
($a/W=0.5$, $B=12$ mm, $K_i=0.6$ MPa $\sqrt{m}$, SG=side-grooved samples)

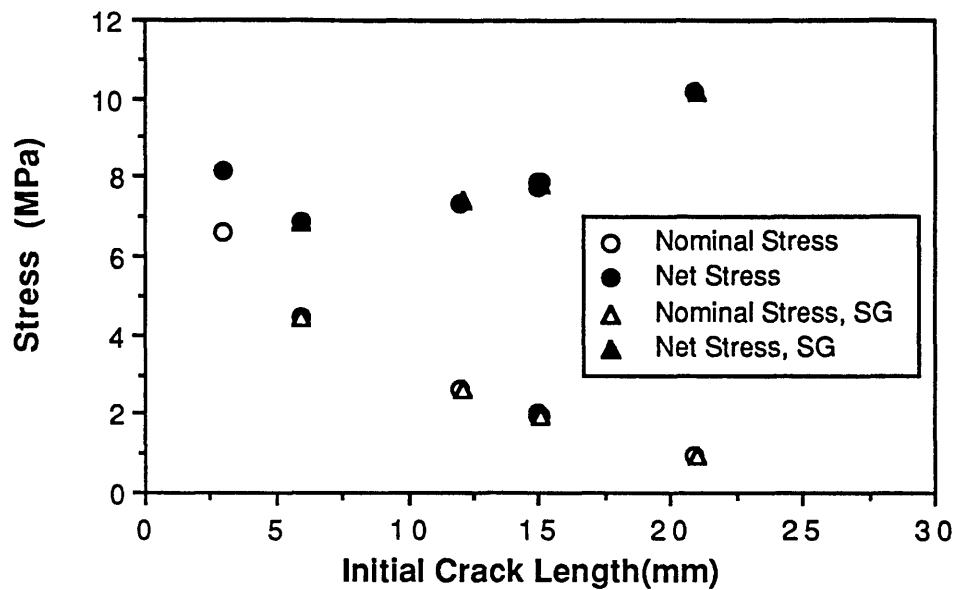


Fig.4.25 Variations of nominal stress and net section stress with initial crack length for 8621 ($W=30$ mm, $B=12$ mm, $K_I=0.6$ MPa $\sqrt{m}$, SG=side-grooved samples)

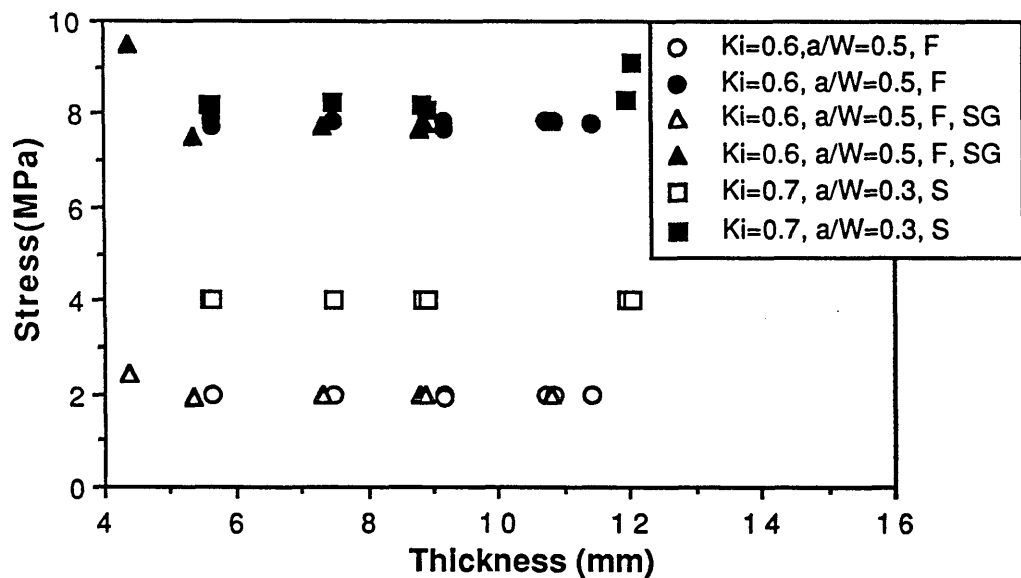


Fig.4.26 Variations of nominal stress and net section stress with specimen thickness for 8621. ($a/W=0.5$, $W=30$ mm, SG=side-grooved samples, F=Flycutter, S=Slicing razor. open symbols=Nominal stress, closed symbols=Net section stress)

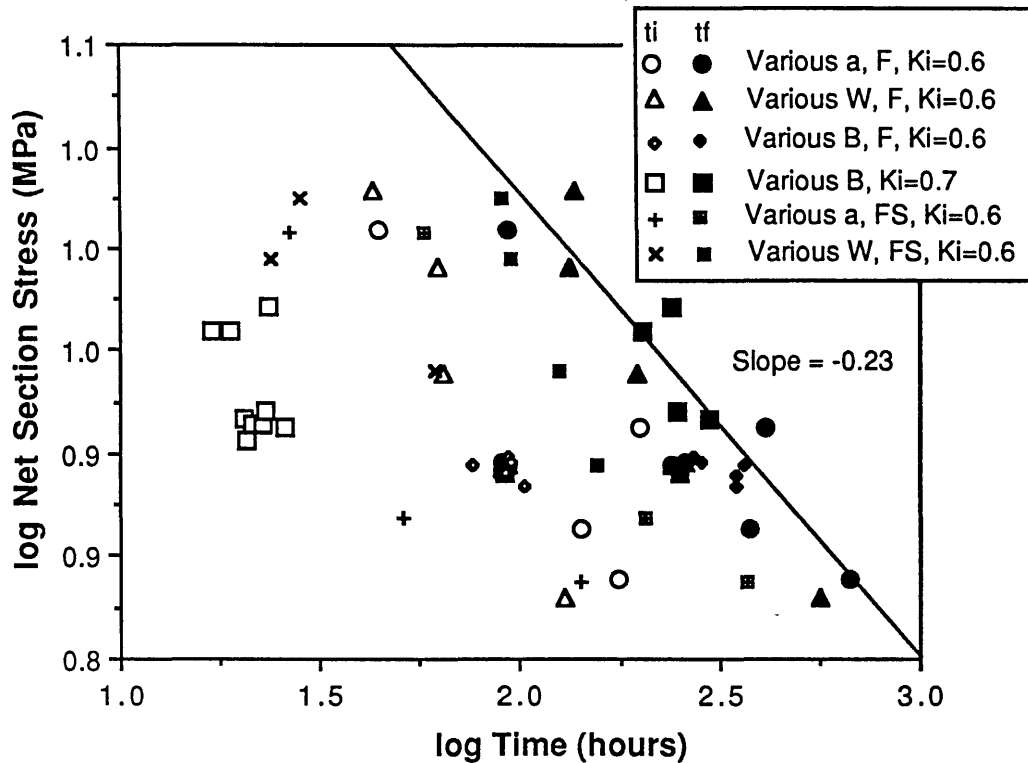


Fig.4.27 Variations of the initiation time(t_i) and failure time(t_f) with the net section stress of 8621 samples with different sizes.
(F=Flycutter, FS=Flycutter, side-grooved samples)

By comparing Fig.4.25 with Fig.4.22, it can be seen that t_i and t_f depend on the magnitude of σ_N instead of σ_a . For the specimens with the deepest cracks, t_i and t_f decrease due to the high σ_N in the uncracked ligament but σ_a tends to decrease with increasing crack length. Comparisons of Figures 4.21 with 4.24 and Figures 4.23 with 4.26 also show that the variations of t_i and t_f follow the trend of σ_N with specimen dimensions. By plotting σ_N versus t_i and t_f as shown in Fig.4.27, the results show that the correlation between σ_N and t_f is better than that between σ_N and t_i . The positions of the σ_N versus t_i plots depend on the K_i levels and side-grooving. Side-grooved specimens tend to give a lower $\sigma_N - t_f$ curve, but the curve is essentially parallel to the one obtained from the plain-sided samples which shows no effect on the K_i levels. A common slope of -0.23 was found for the $\sigma_N - t_f$ curves. The net section stresses of all the specimens tested are plotted against the

corresponding failure time as illustrated in Fig.4.28. It appears that σ_N can describe the failure time adequately well. The slope of the curve is measured to be -0.23 which agrees well with the value of -0.25 obtained from the results of hydrostatic pressure tests for HDPE pipes performed by BP (Williams[1984]).

The influence of σ_N provides an explanation for the short failure time of small specimens at low K_i levels. Brown[1987] and Tonyali[1986, 1987] used specimens with very small ligament length and the samples failed at low K_i level ($K_i = 0.3 \text{ MPa}\sqrt{\text{m}}$) with a reasonable duration, in terms of minutes at least. The effect of σ_N on t_i and t_f increases as the ligament becomes shorter. A small increase in crack length increases σ_N significantly. Consider two samples are loaded at the same K_i level, the dimensions of one sample are L times smaller than the other. The gross and net section stress of the smaller sample will be $\sqrt{L}$ times higher than the large specimen. Therefore, for the same applied K_i , the smaller the specimen size, the greater the effect of σ_N on t_i and t_f will be. It is considered that for very small

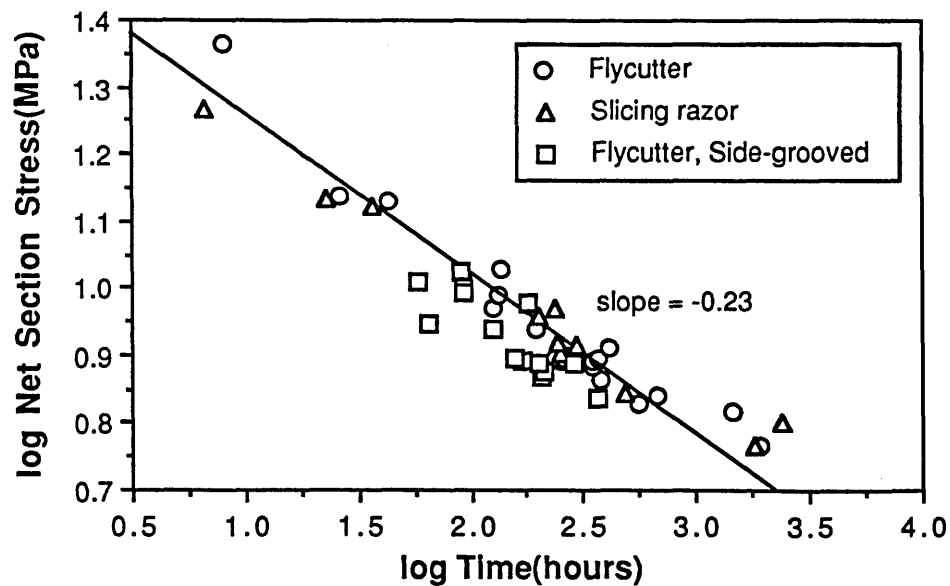


Fig.4.28 Net section stress against failure time for 8621

specimens, the fracture event will be dominated by the level of σ_N in the ligament even when the crack is initiated under brittle conditions and this point will be addressed again later. Although σ_N shows a good correlation with t_f , more work will be required to confirm the utility of σ_N for failure time characterisation.

4.8.2.3 The J -Integral as a Characterising Parameter

It has been shown in Chapter 3 that the J -integral can accommodate a higher degree of plasticity when compared to K . Williams[1984] proposed that the value of J at initiation J_c could be related to t_i by the expression:

$$\frac{J_c}{J_o} = \left(\frac{t_i}{\tau_o} \right)^n \quad (4.25)$$

where $J_o = K_i^2 / E_o$ and τ_o is the reference time. The definition of J used here is slightly different from that given in Equation (3.9). The dissipated energy, U_{dis} , is employed instead of the potential energy, U_p . For crack growth under constant load, U_{dis} is defined as the total energy, U_T , minus the energy for deflecting the beam, U_o .

$$U_{dis} = U_T - U_o = P^2(C - C_o) \quad (4.26)$$

where P is the load, C is the measured compliance, and C_o is the compliance due to the beam, see Equation (4.20). J is written as :

$$J = \frac{1}{B} \frac{dU_{dis}}{da} \quad (4.27)$$

J_c is taken as the value of J at the initiation point. Fig.4.29 shows the variations of (J_c/J_o) with t_i and t_f for specimens with different sizes loading at different K_i levels. A power dependence of 0.13 was found for t_i which agreed well with the value of 0.1 obtained from Equation (4.25). The curve seems to be independent of specimen sizes and loading conditions but the scatter of the data is quite significant. Since the

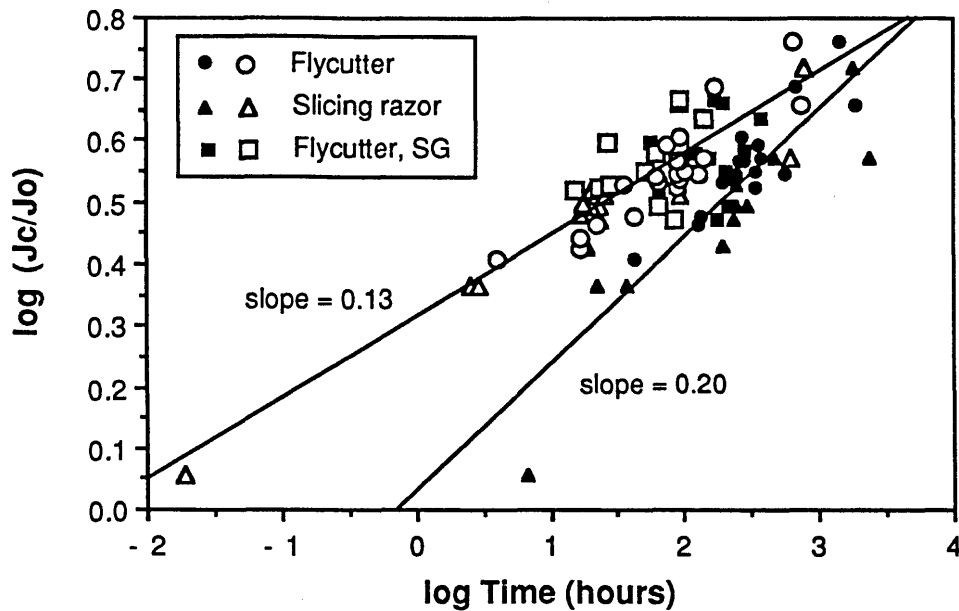


Fig.4.29 J_c/J_o versus initiation and failure time for 8621 specimens
(closed symbols= failure time, open symbols= initiation time)

J -integral can cope with some degree of plasticity, the $(J_c/J_o) - t_i$ curve may not show the ductile brittle transition. It must be noted that the relation between (J_c/J_o) and t_f is not known here. It is clear from Fig.4.29 that the failure time of the side-grooved samples does not follow the trend of the data obtained from the plain-sided specimens. The curve would give a slope of 0.2 if the side-grooved data were ignored. The value of the slope is close to that measured from the $\sigma_N - t_f$ curve but it is not certain if there is a link between the two approaches. Despite the scatter of the data, the J -integral is one of the possible candidates to characterise t_i for the 8621 polyethylene.

4.8.2.4 Effects of Detergent Concentration

The effects of detergent on t_i and t_f of 5731 material have been investigated. Referring to Fig.4.19 and Fig.4.20, t_i and t_f can be halved in the presence of a detergent. The detergent was diluted using distilled water. All the specimens were 30 mm wide, 11 mm thick and with a a/W ratio of 0.5. The samples were side-

grooved and the total thickness reduction was 15% of the original thickness. They were loaded at a K_i level of $0.7 \text{ MPa}\sqrt{\text{m}}$. Although detergent was employed, the duration of each experiment was normally over 4000 hours which is considered to be impractical in a laboratory environment. A very limited amount of data has been collected and the results are shown in Table 4.4.

Concentration (% by volume)	t_i (hours)	t_f (hours)	Final crack length (mm)
10	1698	4367	failure
25	1207	3747	failure
50	2234	4853	failure
50	1877	4271	failure
100	4465	7659+	16.23

Table 4.4 Effects of detergent concentration on t_i and t_f

The results show that both t_i and t_f decrease at first with increasing detergent concentration, then increase as the concentration moves above 25%. The detergent is a gel-like liquid when the concentration is over 50%. Increase in t_i and t_f may be caused by the inability of the fluid to penetrate into the porous region in front of the crack tip because of its high viscosity. Based on the amount of data gathered here, the effect of detergent concentration on t_i and t_f is not conclusive.

4.8.3 Crack Propagation

4.8.3.1 Effect of Side-Grooving and Prediction of t_f from K_C - $\dot{a}$ Curves

After the crack has initiated, it grows in a stable manner. When the ligament is too short to sustain the applied load, fast failure will occur. Typical K_C - $\dot{a}$ curves for 8621 and 5731 specimens of the same size are shown in Fig.4.30.

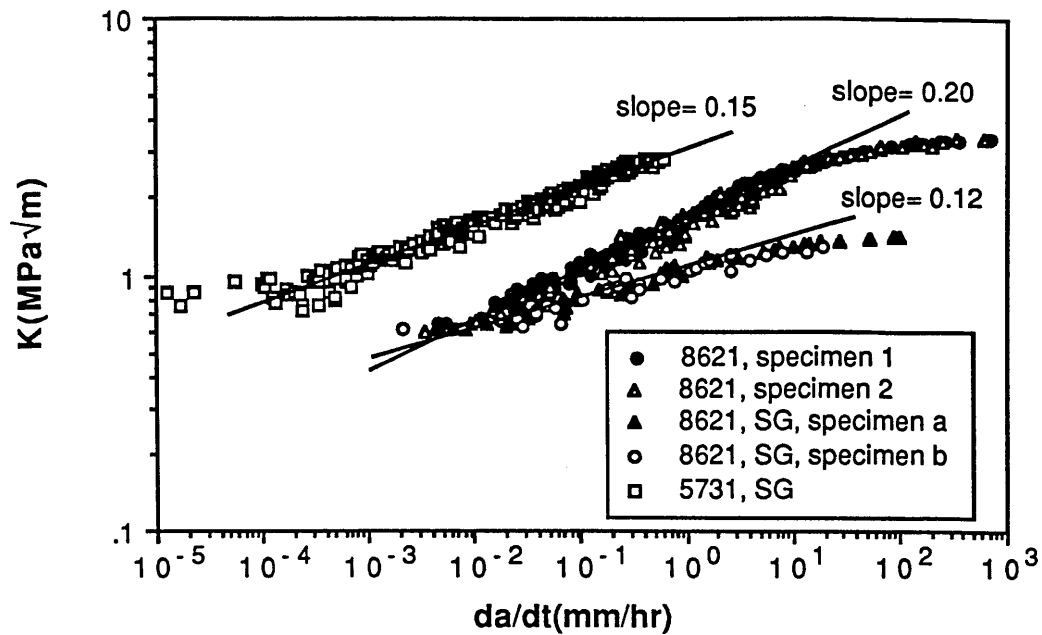


Fig.4.30 Typical $K_C - \dot{a}$ curves. $K_i=0.6 \text{ MPa}\sqrt{\text{m}}$ for 8621 samples. $K_i=0.7 \text{ MPa}\sqrt{\text{m}}$ for 5731 samples ($B=10 \text{ mm}$, $W=30 \text{ mm}$, $S/W=4$, SG=side-grooved samples)

It can be seen that the reproducibility of data from different specimens is good. The curves first rise gently then attain a linear region and finally flatten when fast failure occurs. Equations (4.5) is only capable of modelling the linear part of the curve. It is apparent that the crack travels at a much slower speed in the 5731 than in the 8621. The $K_C - \dot{a}$ curves for the plain-sided specimens are steeper than that of the side-grooved specimens, this means that the crack speeds are higher for side-grooved samples. As side-grooving tends to reduce the size of shear lips, it is believed that the crack speed reduces with increasing shear lip size. It is evident from Fig.4.17 that the shear lip size increases as the crack grows. The material near the edges can sustain larger elongation before separation takes place because the plane stress condition prevails near the surfaces. This results in a lower crack growth rate and a steeper $K_C - \dot{a}$ curve. For the 5731 samples, the amount of plastic deformation near roots of the side-grooves are still significant as shown in Fig.4.31.

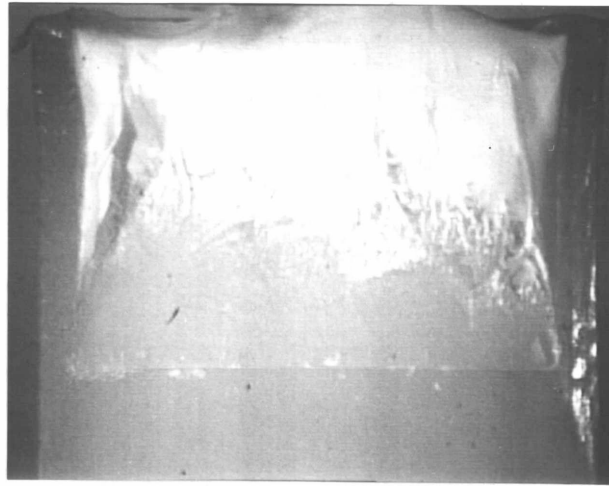


Fig.4.31 Fracture surfaces of a 5731 side-grooved sample

According to Equation (4.5), the slope of the $K_c - \dot{a}$ curve should be 0.1 for both polyethylenes. The slope of the $K_c - \dot{a}$ curve for the 5731 side-grooved specimens was found to be 0.15. The discrepancy is believed to be due to the presence of the shear lips. The $K_c - \dot{a}$ curves of the 8621 side-grooved specimen gave a value of 0.12 which agrees fairly well with the predicted value of 0.1. However, a slope of 0.2 was obtained from the $K_c - \dot{a}$ curves of the plain-sided 8621 specimens which is twice the theoretical value. This seems to contradict the results shown in Fig.4.20 because Equation (4.12) has been derived from Equation (4.5). From Equation (4.12), if q is measured from the $K_c - \dot{a}$ curve, the slope of the curves illustrated in Fig.4.20 should have a value of -0.2. The reason lies in the short propagation time relative to total failure time. Data for specimen 1 in Fig.4.30 is re-plotted on Fig.4.32 with the corresponding loading time at various propagation stages.

It can be noted that the propagation time only occupies a fraction of the total failure time. From Fig.4.32, the initiation time is 93 hours but the relaxation crack growth process takes place after 191 hours. The time between initiation and crack propagation is believed to be the time for the damaged zone to become fully developed and is termed as the crack tip development time, t_{dev} , here. The total

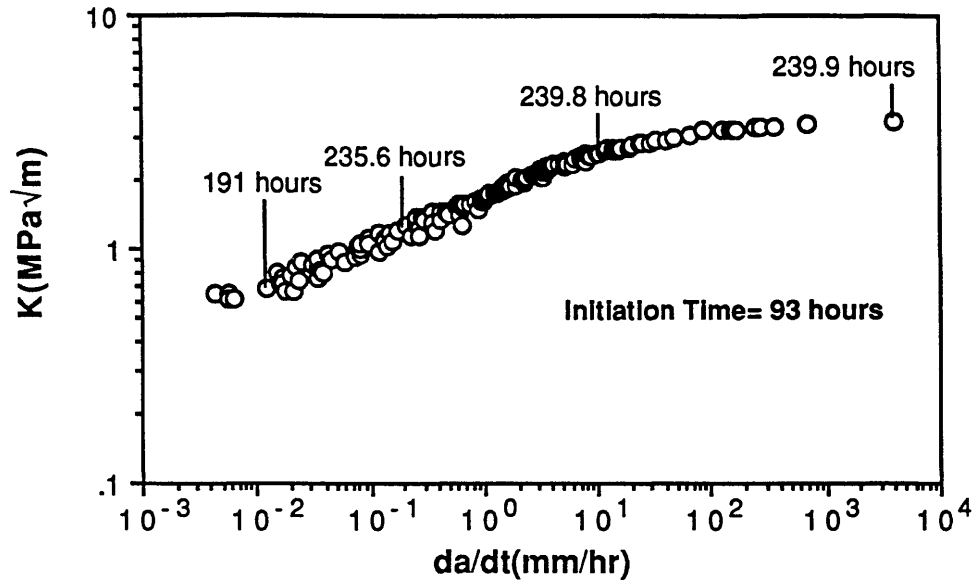


Fig.4.32 K_c - $\dot{a}$ curve for a 8621 plain-sided specimen

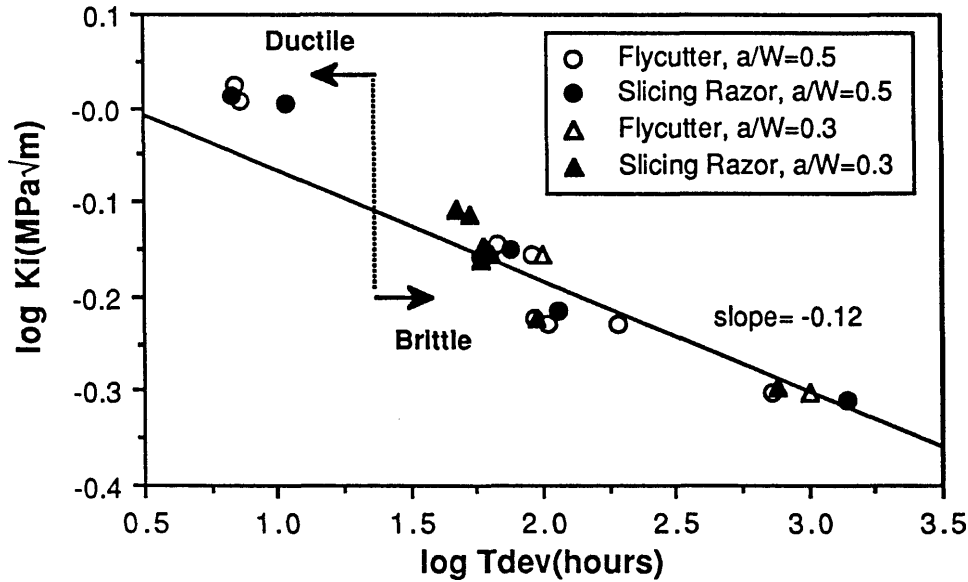
failure time is probably reflected more appropriately by the total incubation time, $t_i + t_{dev}$. Using Equation (4.3), the length of the damaged zone, c , can be written as:

$$c = \frac{\pi}{8} \left(\frac{K_c}{\sigma_o} \right)^2 t^{2m} \quad (4.28)$$

t is measured from the initiation time onward. If the size of the fully developed zone is assumed to be constant at the onset of the relaxation controlled crack propagation, t_{dev} can be related to K_i as:

$$K_i \propto t_{dev}^{-m} \quad (4.29)$$

provided that the change in K_c is small. This will also yield a time dependence of -0.1. t_{dev} at various K_i levels are given in Fig.4.33 and a slope of -0.12 is obtained. Therefore, the σ_a versus t_f plot gives a slope of about -0.1 as shown in Fig.4.20 due to the dominance of t_i and t_{dev} on the total failure time.

Fig.4.33 K_I versus t_{dev} for 8621 specimens

It must be emphasised that the dashed line in Fig.4.33 does not actually represent the position of a ductile-brittle transition. t_f calculated from Equation (4.11) may heavily underestimate the actual lifetime. Atkins and Mai[1984, Ch.6] showed some experimental $K_c - t_f$ data for PMMA and the measured t_f was an order of magnitude higher than that predicted from Equation (4.11). It is therefore questionable if the $K_c - \dot{a}$ characteristic can be related to the results obtained from the hydrostatic stress rupture tests. This will be true only if the crack incubation time is small when compared to the total failure time.

4.8.3.2 Effects of Specimen Size and Applied K_I Levels

The behaviour of slow crack growth in the polyethylenes has been shown to obey an equation of the form:

$$K_c = D\dot{a}^q \quad (4.30)$$

where D is a material constant but q has been found to be affected by the presence of shear lips, and is not a constant as suggested in Equations (4.5). The effects of specimen size and K_I levels on K_{IC} - $\dot{a}$ characteristics have been examined. The range of specimen sizes used are given in Table 4.2. Fig.4.34 shows typical K_{IC} - $\dot{a}$ curves for 8621 specimens with various thicknesses.

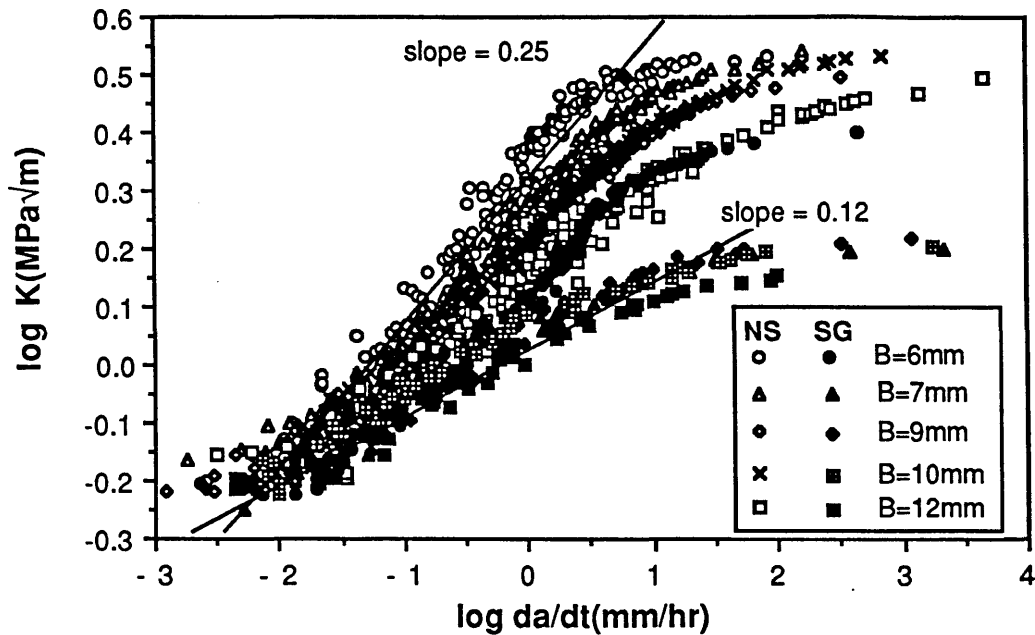


Fig.4.34 Effect of specimen thickness, B , on K_{IC} - $\dot{a}$ curves for 8621 specimens
(NS=non side-grooved specimens, SG=side-grooved specimens,
 $W=30\text{mm}$ and $S/W=4$, $K_I=0.6\text{MPa}\sqrt{\text{m}}$)

The slope of the K_{IC} - $\dot{a}$ curve is found to increase with decreasing specimen thickness. The curves for the side-grooved specimens are lower than those of the plain-sided samples as observed previously. The effect of specimen thickness on K_{IC} - $\dot{a}$ curves of the side-grooved samples is small, except for the 6 mm thick specimens, when compared with that for plain-sided samples. The slope attains a maximum value of 0.25 for the 6 mm thick plain-sided specimens. It is believed that the increase in slope is due to an increase in the relative size of the shear lips to the specimen thickness as shown in Fig.4.35. The fracture surfaces of the 6 mm thick specimens

revealed large shear lips and layers of tearing film at both sides of the specimens as shown in Fig.4.36, and the crack growth rate is affected strongly by the shear lips.

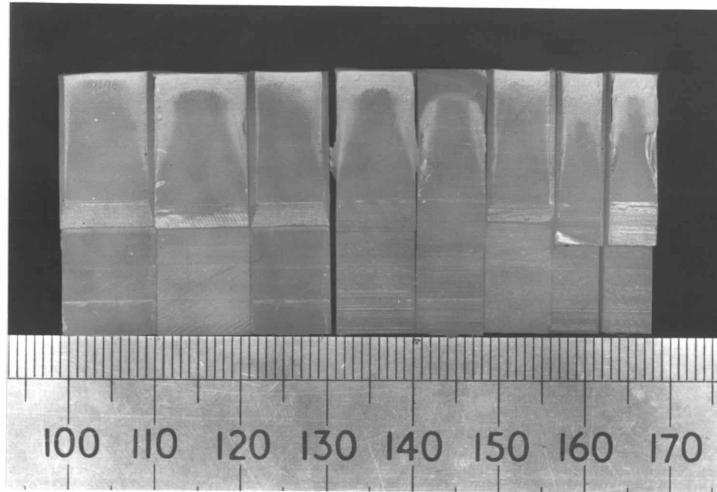


Fig.4.35 Fracture surfaces of 8621 specimens with various thicknesses

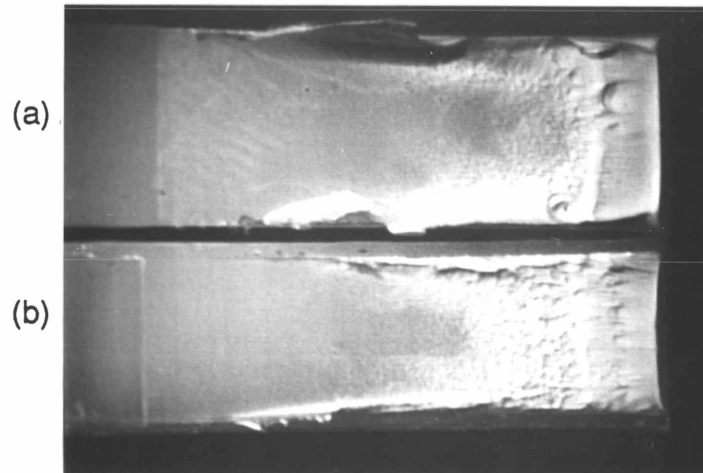


Fig.4.36 Fracture surfaces of 6 mm thick 8621 specimens
a) non side-grooved, b) side-grooved

The thickness dependence of the K_c - $\dot{a}$ curve can be modelled using a equation similar to Equation (2.18). If the size of the shear lips is considered to be the same as the plastic zone size, r_p , the measured K_c , K_c' , is an average of the distribution of

the plane strain and plane stress K_c values, termed as K_{c1} and K_{c2} respectively. K_c' can be estimated from the equation:

$$K_c' = K_{c1} + \frac{2(r_p - g)}{B} [K_{c2} - K_{c1}] \quad (4.31)$$

where $r_p = K_c'^2 / (2\pi\sigma_y^2)$ and g is the depth of the side-grooves. For a particular $\dot{a}$, the K_c' values for the plain-sided samples with different thicknesses are plotted against $(K_c'^2/B)$, with $g=0$, as shown in Fig.4.37. The values of K_{c1} at various crack speeds are evaluated from the intercepts of the lines and the K_c' -axis. The corresponding K_{c2} values can be determined from the slopes of the lines via Equation (4.31) and σ_y is assumed to be 26 MPa.

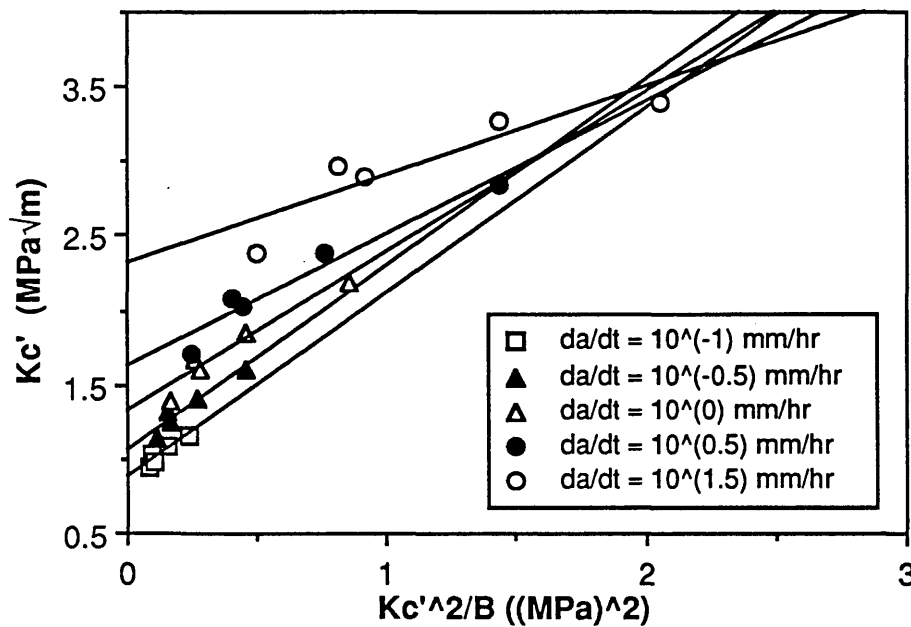


Fig.4.37 K_c' versus $K_c'^2/B$ plots at various crack speeds for 8621 plain-sided specimens.

For the side-grooved specimens, it was found that g is greater than r_p , except for the 6 mm thick sample. Therefore the K_c' values for samples with thicknesses greater than 6 mm can be regarded as the K_{c1} values. The average of these K_c' values is taken as the K_{c1} value. A plot equivalent to Fig.4.37 cannot be obtained for

the side-grooved specimens since the calculations show that the shear lip effect only occurs in 6 mm thick specimens.

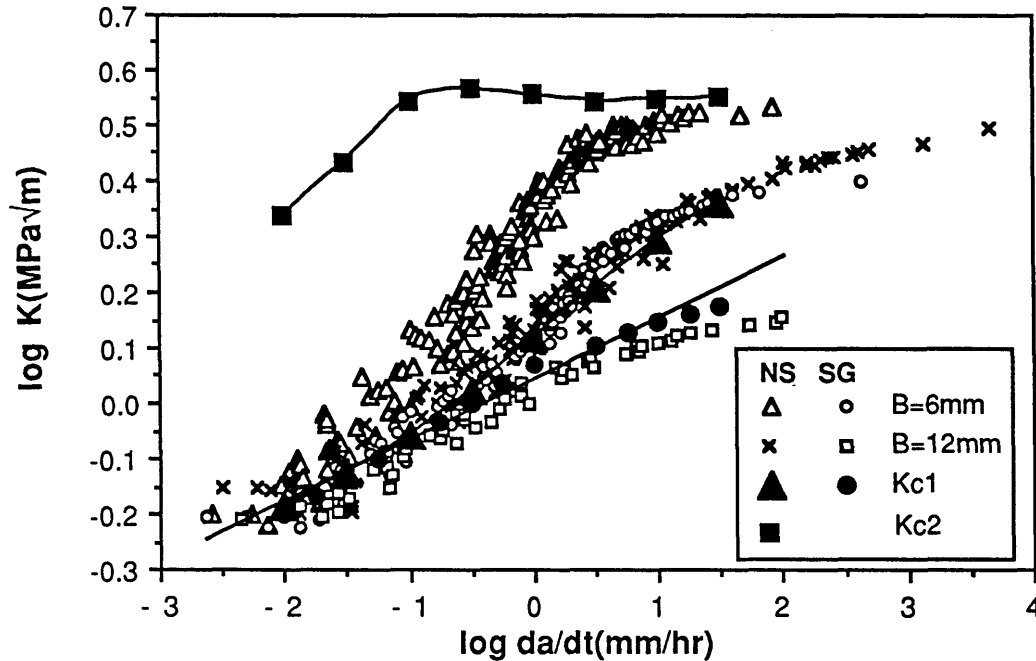


Fig.4.38 $K_{c1} - \dot{a}$ and $K_{c2} - \dot{a}$ curves for the 8621 specimens
(NS = plain-sided specimen, SG = side-grooved specimens)

The values of K_{c1} and K_{c2} at different $\dot{a}$ are shown in Fig.4.38. The $K_c - \dot{a}$ curves for the 6 mm and 12 mm thick specimens are also included for comparison. It can be seen that K_{c2} is nearly constant at high $\dot{a}$. The $K_{c1} - \dot{a}$ curve for side-grooved samples give a slope of 0.11 which agrees well with the theoretical value. It can be noticed that the $K_{c1} - \dot{a}$ curves for side-grooved and plain-sided samples have a common slope when K_c is low. This indicates that fully plane strain conditions are achieved in the initial crack propagation stage. By referring to Fig.4.17, it is evident that the size of the shear lips for the plain-sided sample is comparable to that of the side-grooved sample for small amounts of crack growth. Thus, the $K_c - \dot{a}$ curves in this region are expected to be the same. It can be assumed that the $K_{c1} - \dot{a}$ curve for the side-grooved specimens and the $K_{c2} - \dot{a}$ curve for the plain-sided specimens represent the lower and upper bounds of the measured $K_c - \dot{a}$ curves respectively. The $K_c - \dot{a}$

characteristic for any specimen which exhibits shear lips should fall between these two limits. The results from Chan[1983] showed that the slope of the K_c - $\dot{a}$ curve was 0.25 for HDPE. The thickness of the specimens used in that case was in excess of 12 mm. It is not certain whether it is the genuine behaviour of that material, or the growth of the shear lips was mistakenly measured as the crack growth.

The variations of K_c - $\dot{a}$ curves with specimen width and initial crack length are shown in Fig.4.39 and Fig.4.40 respectively. The K_c - $\dot{a}$ curves becomes steeper as the ligament size increases. According to LEFM, the slope of the K_c - $\dot{a}$ curve is expected to decrease with increasing ligament size since the ratio of r_p to ligament decreases. However, by comparing the fracture surface of the 15 mm wide specimen, shown in Fig.4.41, with that of the 30 mm wide specimen in Fig.4.17b, it is found that the shear lips are smaller in the 15 mm sample. The increase in shear lip size of the wide

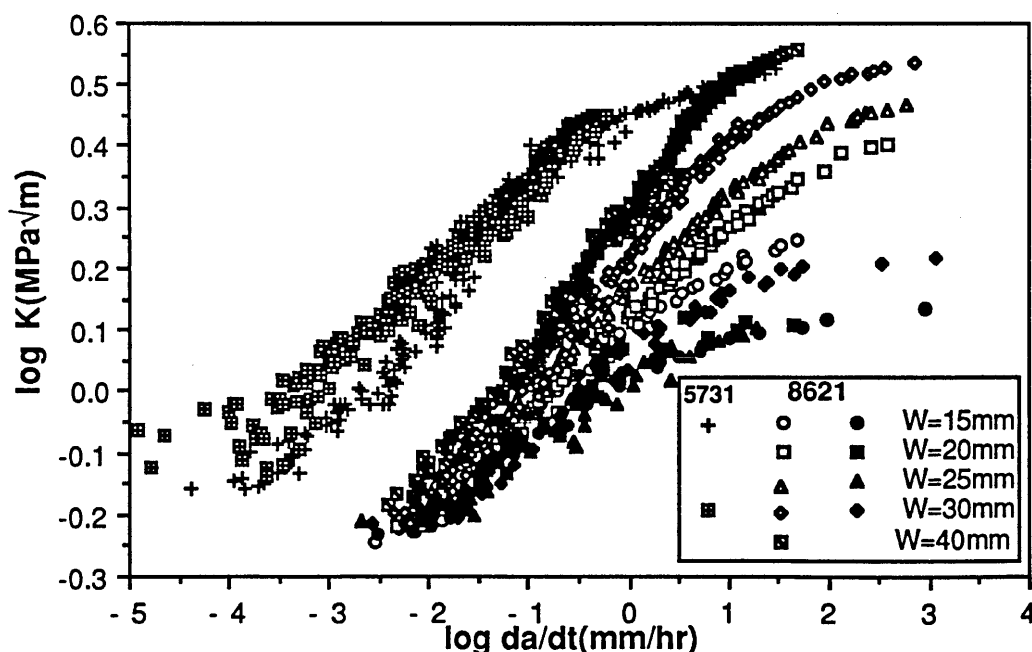


Fig.4.39 Effect of specimen width, W , on K_c - $\dot{a}$ curves. $B=10$ mm, $S/W=4$.

(8621: open symbols=plain-sided specimens, closed symbols=side-grooved specimens, $K_j=0.6\text{MPa}\sqrt{\text{m}}$. 5731: side grooved specimens only, $K_j=0.7\text{MPa}\sqrt{\text{m}}$.)

sample is suggested to be caused by crack tunnelling. Broken specimens with various initial crack lengths and various widths are shown in Fig.4.42 and Fig.4.43 respectively. It is clear from these figures that the degree of crack tunnelling increases with increasing ligament size. For specimens with large ligament size, the crack is able to tunnel a distance before plastic collapse in the ligament occurs. The size of the shear lips increases due to the high local K_c value at the centre of the specimen. The crack tunnelling effect is not prominent for specimens with short ligament because fast failure takes place before the crack starts tunnelling and, for side-grooved specimens, the shear lips are almost completely removed by side-grooving, see Fig.4.43. This accounts for the width independence of the K_c - $\dot{a}$ curves for the side-grooved samples with specimen width smaller than 30 mm. All K_c - $\dot{a}$ curves share the same slope at low K_c levels since the shear lips are small, and then diverge as crack tunnelling develops. Equation (4.31) could be used to describe the width effect on the K_c - $\dot{a}$ curves if the relationship between the shear lip size and the crack tunnelling mechanisms was known. Fig.4.44 shows the K_c - $\dot{a}$ curves of specimens loaded at different K_i levels. The curve rises slightly as K_i increases. Such a characteristic is caused by the increase in shear lip size due to the high K_c level.

It can be inferred from the results that the K_c - $\dot{a}$ approach should only be used when fully plane strain conditions are achieved and the K_c - $\dot{a}$ curves would be independent of specimen size and side-grooving under such conditions.

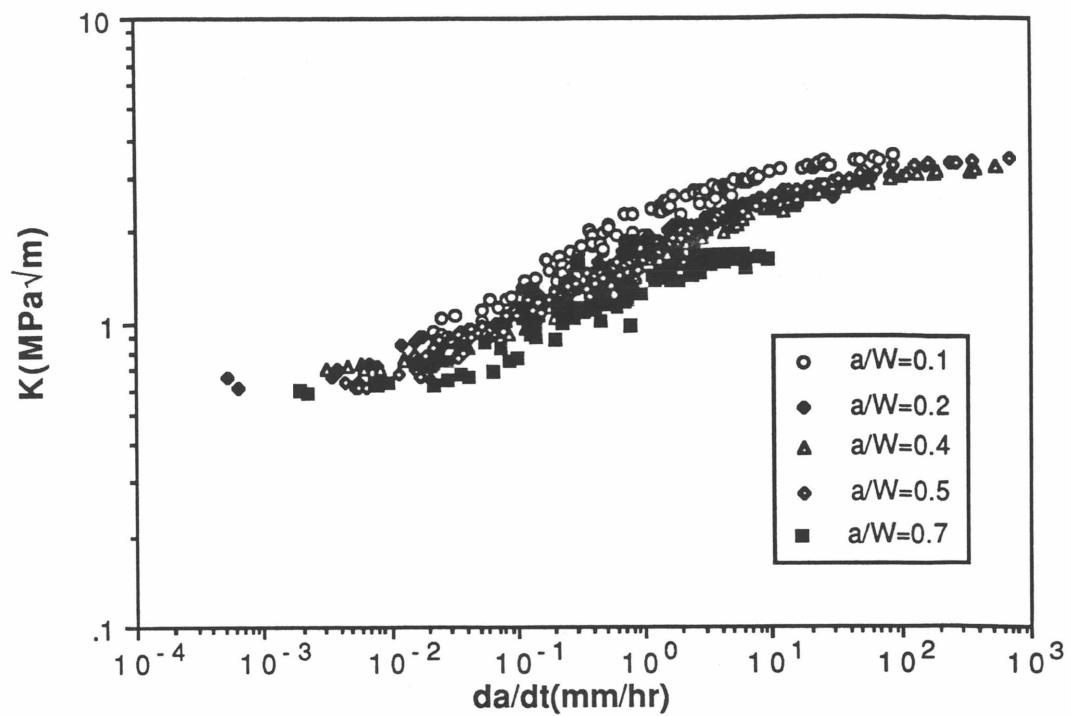


Fig.4.40 Effect of initial crack length on $K_C - \dot{a}$ curves for 8621.
($B=10$ mm, $W=30$ mm, $S/W=4$, $K_I=0.6\text{MPa}\sqrt{\text{m}}$ and all plain-sided specimen)

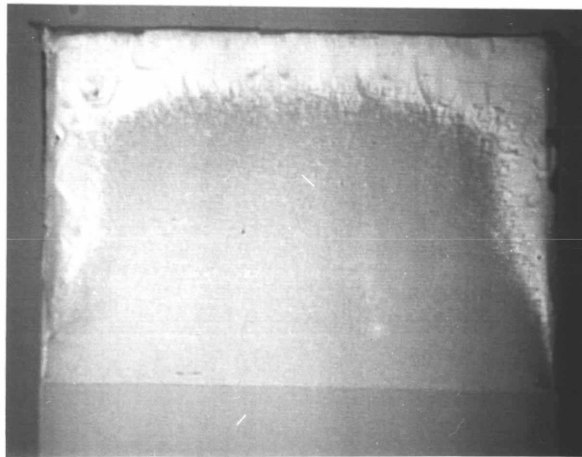


Fig.4.41 Fracture surfaces of the 15 mm wide 8621 specimens

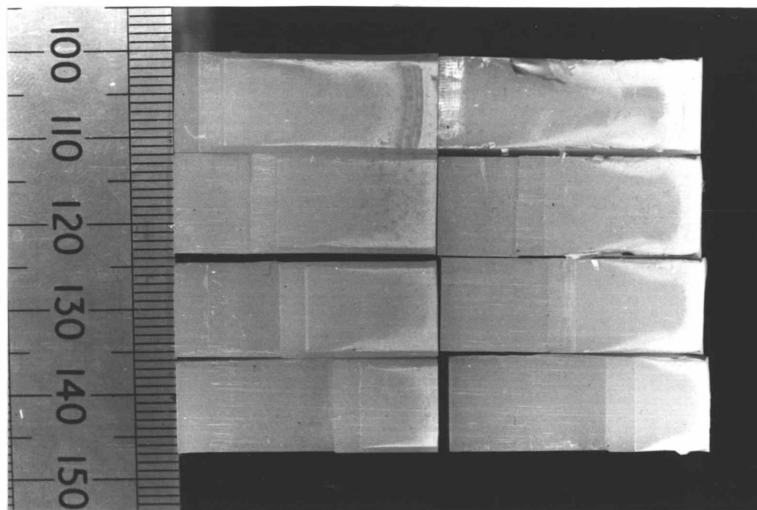


Fig.4.42 Fracture surfaces of 8621 specimens with various initial crack lengths

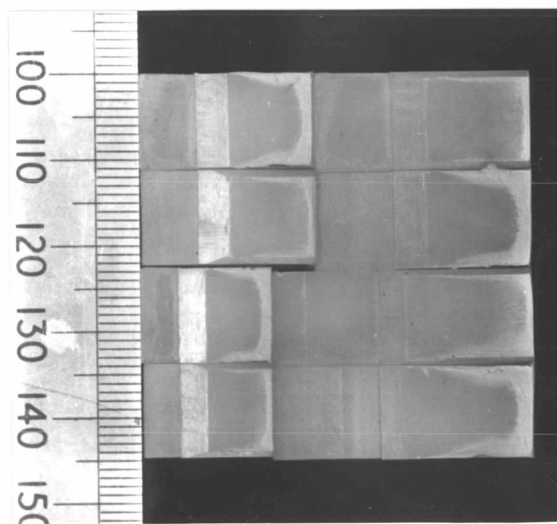


Fig.4.43 Fracture surfaces of 8621 specimens with various specimen widths

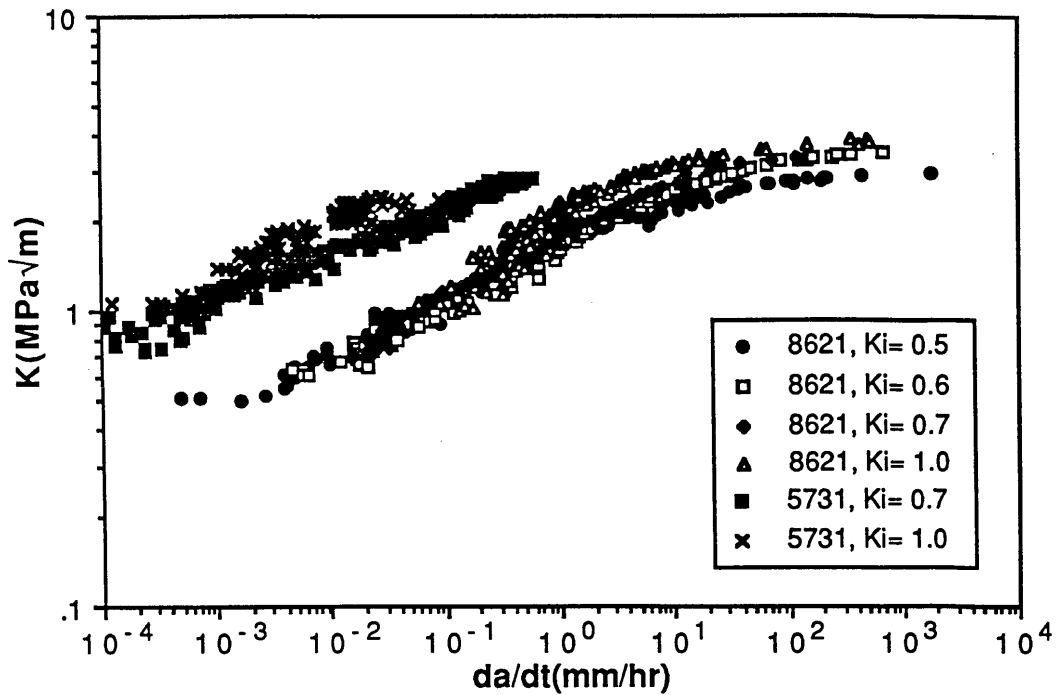


Fig.4.44 Effect of K_i on $K_C - \dot{a}$ curves

($B=10$ mm, $W=30$ mm, $S/W=4$, $a/W=0.5$ and all plain-sided specimen for 8621 and side-grooved samples for 5731)

4.8.3.3 Effect of Detergent on $K_C - \dot{a}$ Curves of 5731

Side-grooved specimens of the same size ($B=12$ mm, $W=30$ mm, $a/W=0.5$ and $S/W=4$) were loaded at a K_i level of $0.7 \text{ MPa}\sqrt{\text{m}}$. All the specimens showed crack arrests during the experiments but the same was not observed in the absence of detergent. Crack arrest marks can be observed easily on the fracture surfaces as shown in Fig.4.45.

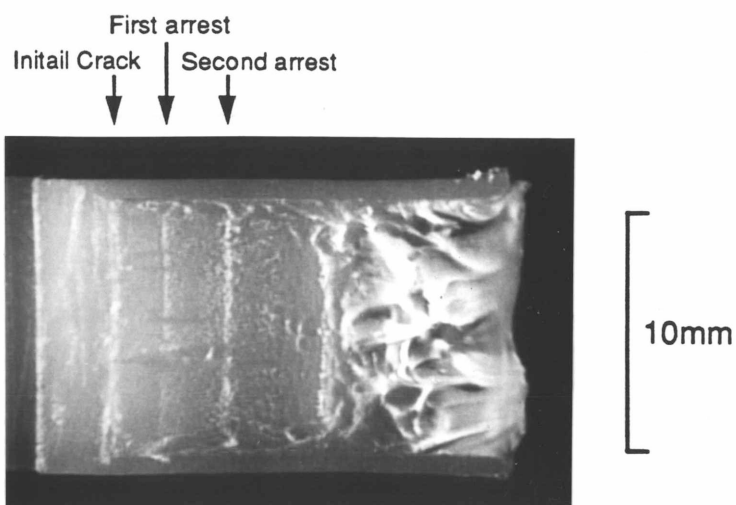


Fig.4.45 Fracture surface of 5731 specimen in 50% Arkopal (Initial crack length=15mm)

It can be seen that the crack front remained straight while the crack propagated. Fig.4.46 shows the predicted crack length at various times during the test for the sample illustrated in Fig.4.45. The positions of the crack arrest marks measured from Fig.4.45 coincide with the points where sudden jump in crack growth rate are recorded. These changes in crack growth rate induces 'zigzag' behaviour in the K_c - $\dot{a}$ curve. The K_c - $\dot{a}$ curves for specimens in different concentration of Arkopal are shown in Fig.4.47. It can be noted that crack arrests appear to occur at the same K_c levels despite the difference in detergent concentration. The transition crack speeds at the flow controlled region are found to be constant, which agrees with the Williams' flow controlled model (Williams et al.[1975]). The transition speeds are between 0.01 mm/hr to 0.02 mm/hr. However, there are insufficient data to comment on the effect of detergent concentration on the crack growth behaviour.

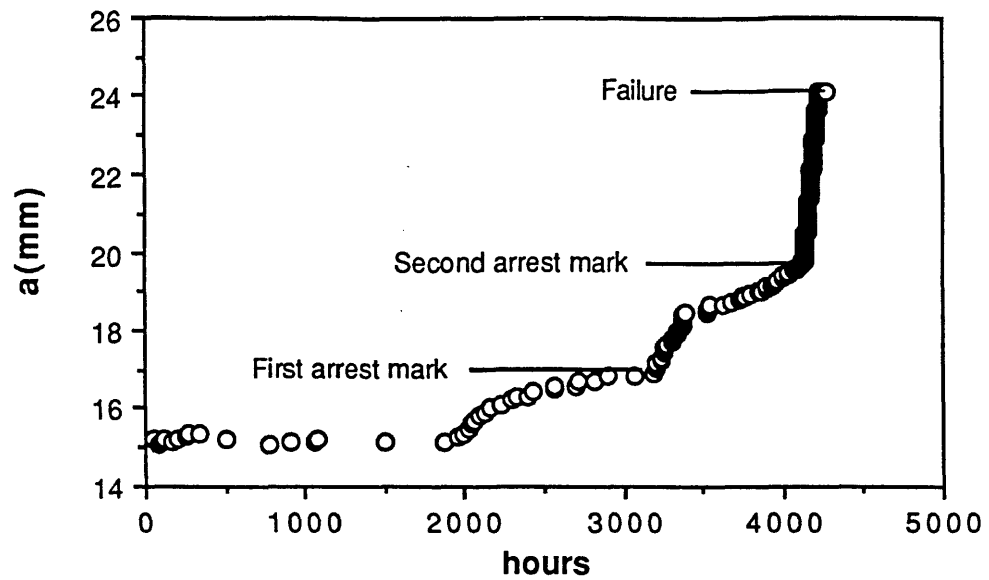


Fig.4.46 Variation of crack length during experiment for 5731 specimen in 50% Arkopal

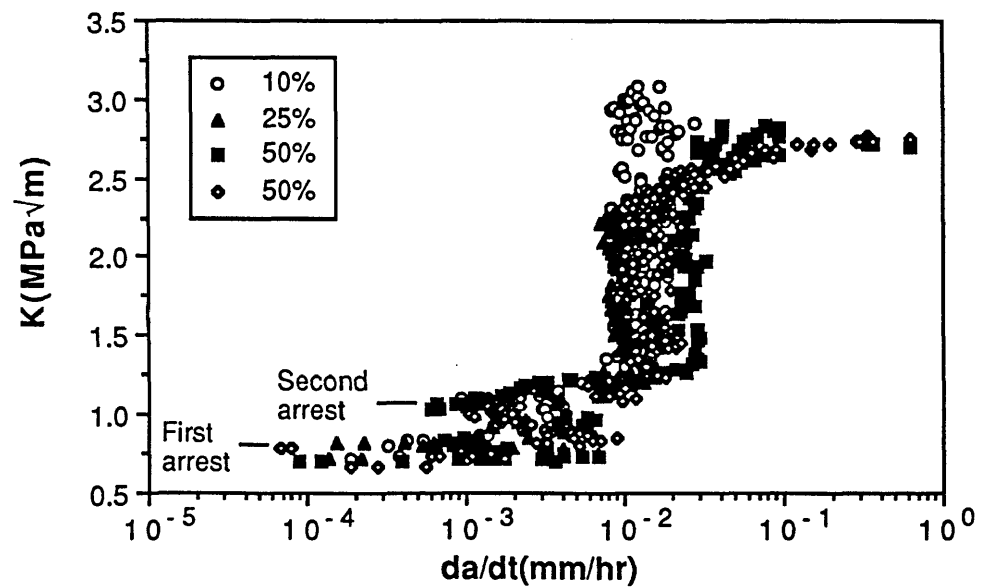


Fig.4.47 K_I - a curves of 5731 specimens in various concentration of Arkopal

4.9 Fractographic Studies and Crack Growth Mechanisms

4.9.1 Crack Growth in Inert Environment

Fractographic analysis of fracture surfaces always provides valuable information about crack growth mechanisms. A scanning electron microscope was used for microscopic examination of the fracture surfaces and the damaged zone ahead of the crack tip. Samples were unloaded during the experiments and sectioned at the mid-thickness, the surfaces were then polished. The sectioned samples were loaded in order to open the crack tip before fitting into the microscope. Care had been taken not to overload the specimens. The damaged zones for the polyethylenes are shown in Fig.4.48. The region near the crack tip is porous where voids and fibrils can be seen. Further ahead of the crack tip, the zone is comprised of deformed material where the voids and fibrils begin to form. The fibrils bridge the crack surfaces and are capable of carrying load. This helps to stabilize the plastically deformed material at the tip of the damaged zone. Fibrillar fracture surfaces were created by the breakage of fibrils. Fig.4.49 and Fig.4.50 show the fibrillar structure of the fracture surfaces for the 8621 and 5731 respectively. The size of the fibrils is much larger for the 5731 material and there are also fewer fibrils on the fracture surface. It has been suggested by Lustiger et al.[1983] that fibrils are formed by the pull-out of lamellae and the fracture is interlamellar. Not all fibrils are necessarily formed by the pull-out of lamellae, they may also be formed by the growth of voids. Since a spherulitic structure cannot be observed on the fracture surfaces here, it is not clear whether the failure process is interlamellar in this case. The crack growth process is thought to be caused by the nucleation of voids and the failure of fibrils.

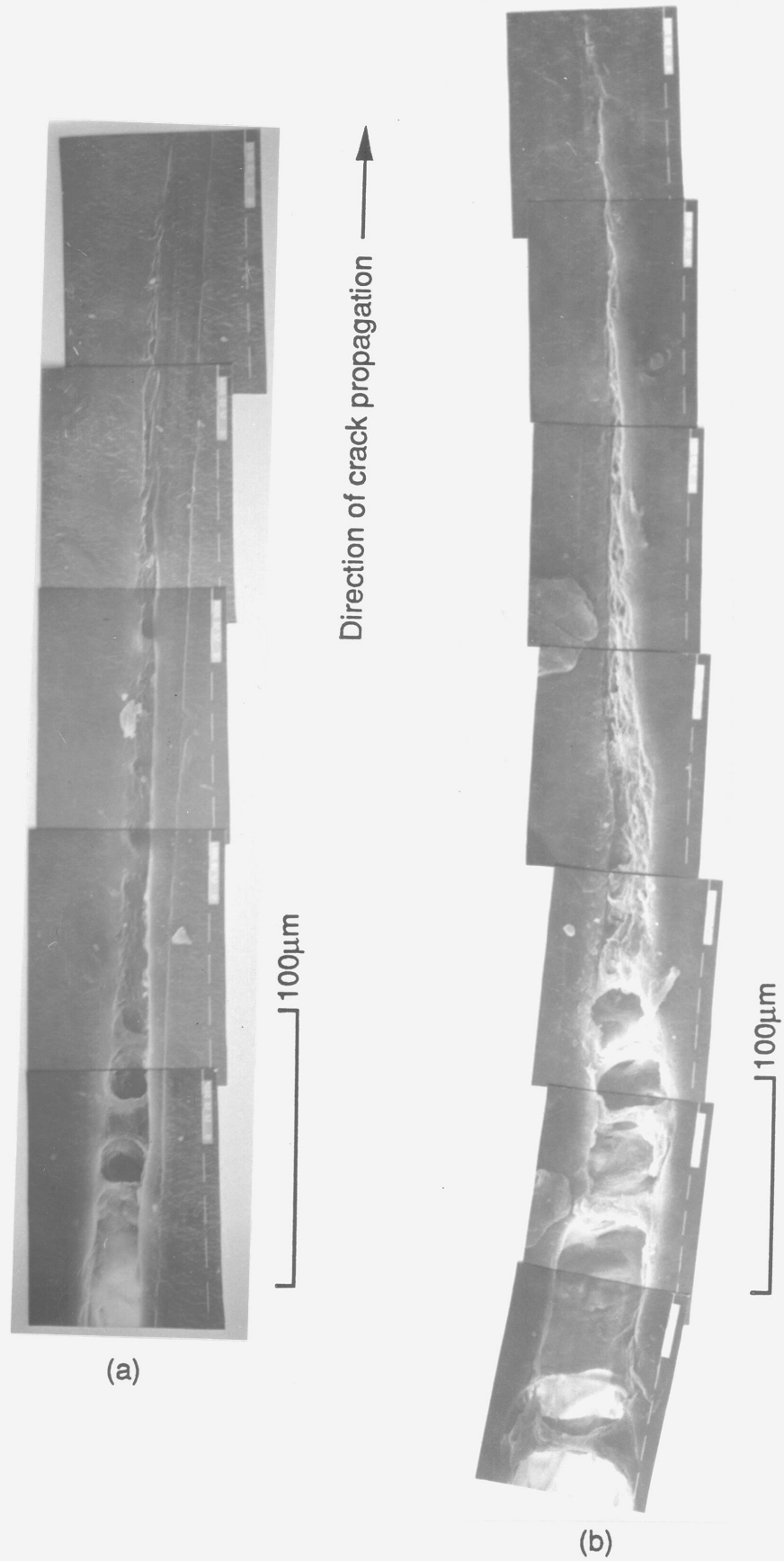


Fig.4.48 The damaged zone ahead of the crack tip. (a) 8621, (b) 5731

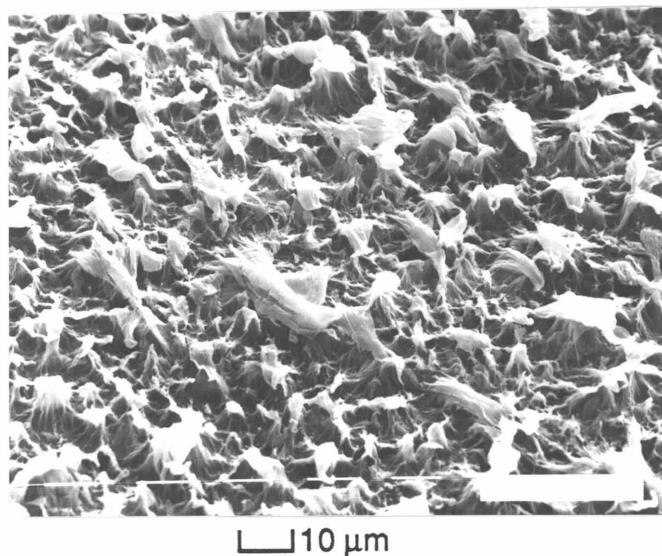


Fig.4.49 Micrograph of the fracture surface of a 8621 sample

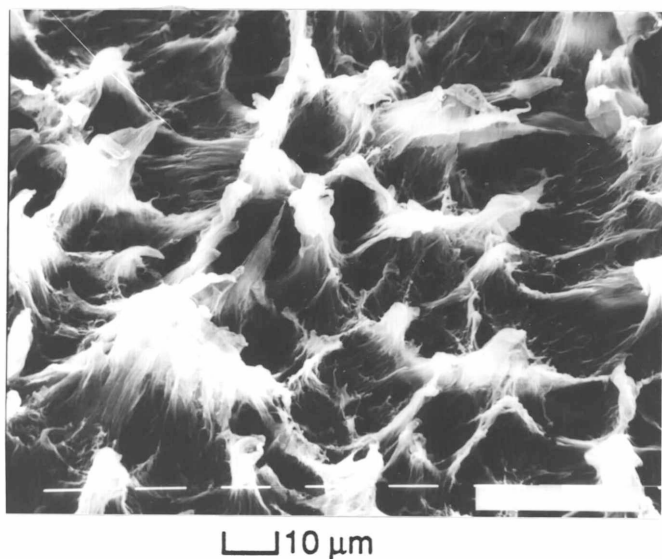


Fig.4.50 Micrograph of the fracture surface of a 5731 sample

Direction of crack propagation →

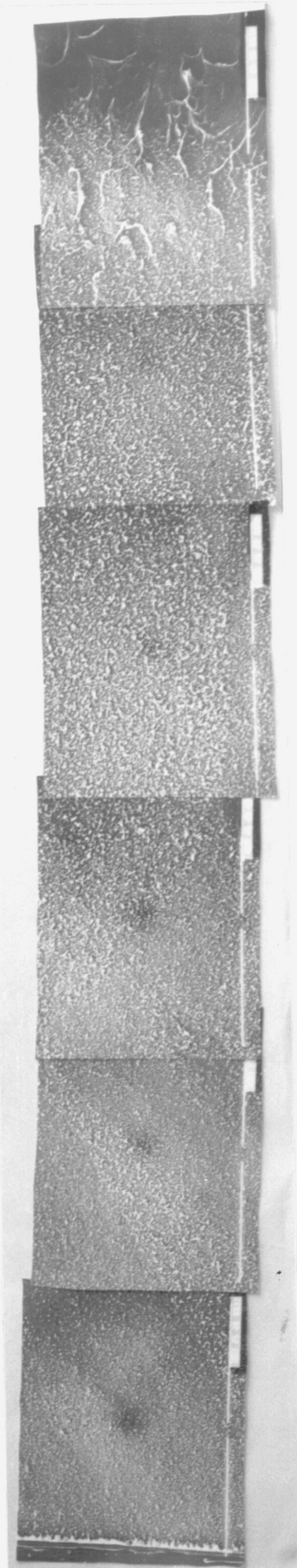


Fig.4.51 Overview of the fracture surface
of a 8621 specimen
└──────────┘ 1000 μm

Direction of crack propagation $\uparrow$

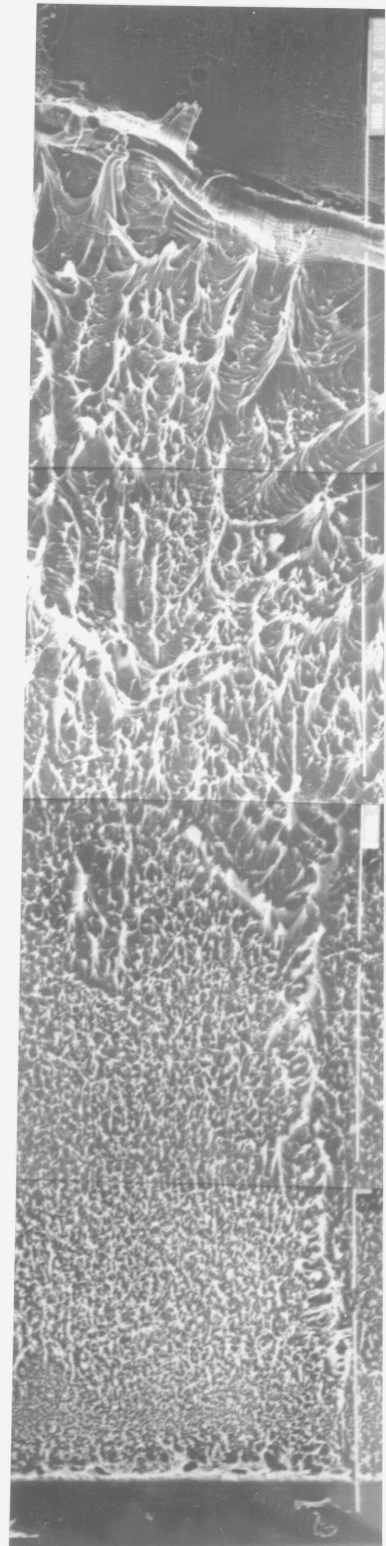


Fig.4.52 Overview of the fracture surface
of a 5731 specimen
└──────────┘ 1000 μm

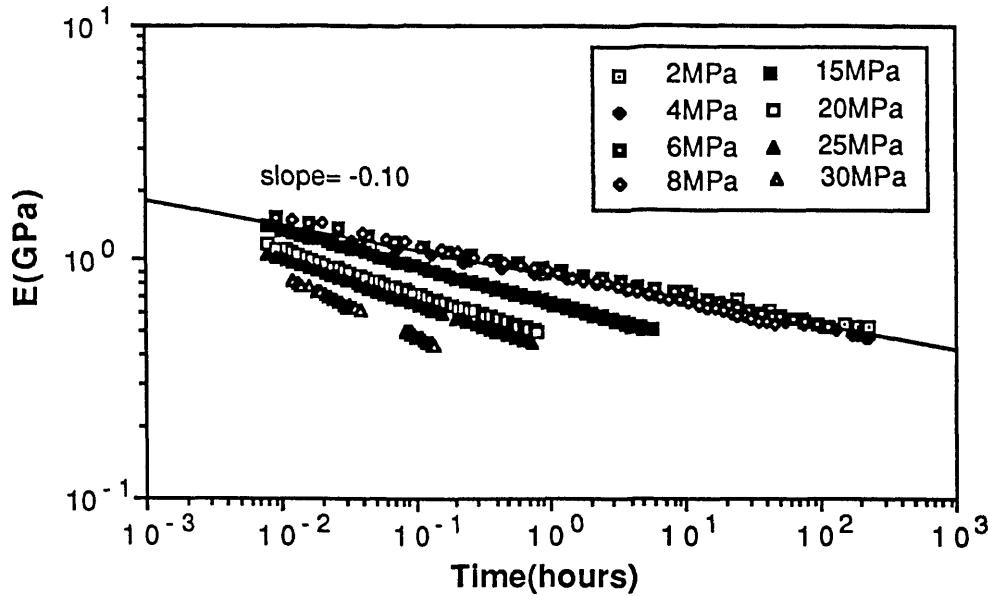


Fig.4.53 Creep curves for 8621

Fig.4.51 and Fig.4.52 show the overviews of the fracture surfaces of typical 8621 and 5731 specimens respectively. It is interesting to note that the fibrillar structure becomes coarser as the crack propagates through the ligament. This feature has also been reported by Chan[1983] but the exact cause is not clear. It is suggested here that the increase in fibril length is caused by the nonlinear viscoelastic response of the materials. In order to clarify this point, unnotched bars of the same size as the fracture test samples were loaded in three point bending at various constant load levels. The modulus was calculated using Equation (4.20). Results for the 8621 and 5731 specimens are given in Fig.4.53 and Fig.4.54 respectively. The time dependences of the materials were found to be constant only when the nominal applied stress did not exceed 10 MPa. By assuming the nominal strain ϵ_{nom} is σ_a/E , the nominal isochronous curves at different times are illustrated in Fig.4.55. It can be seen that the behaviour is non-linear and ϵ_{nom} increases rapidly when the time or stress is high. This explains the formation of a layer of highly drawn fibrils observed at the initial notch tip (see Figs.4.9 - 4.11). The material at the initial notch deformed in a nonlinear elastic manner due to the relatively long initiation time though the stress may be low.

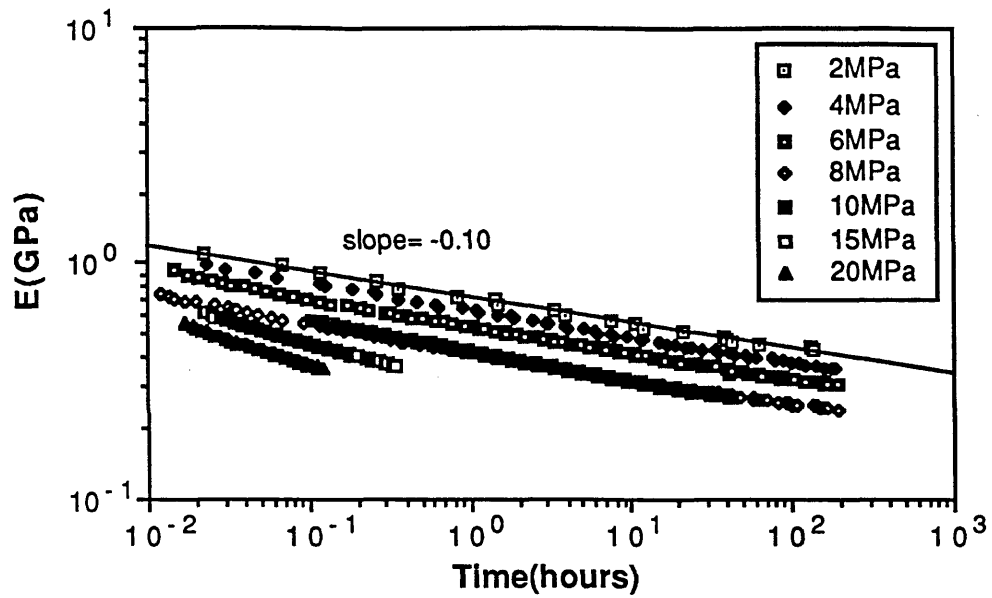
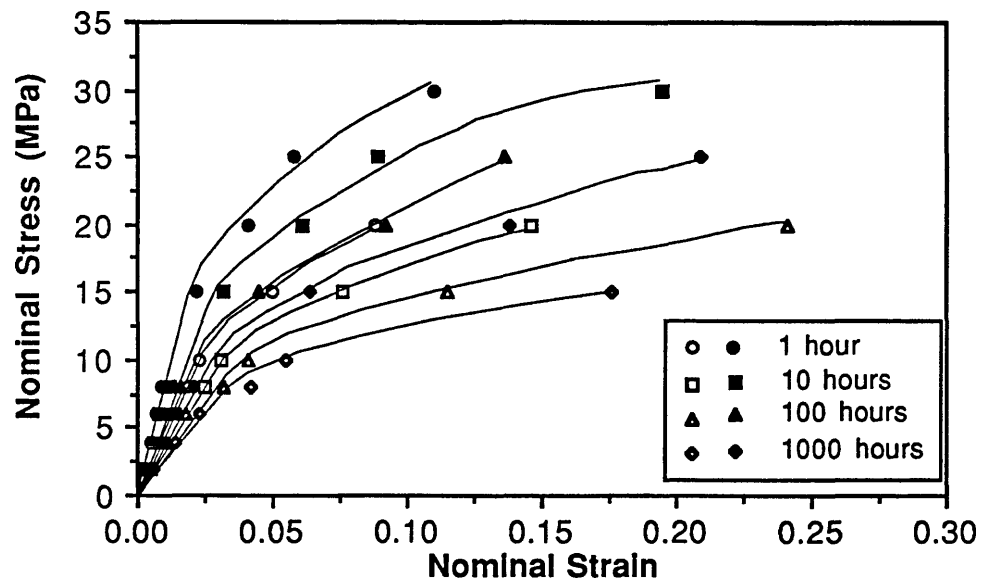


Fig.4.54 Creep curves for 5731

Fig.4.55 Nominal isochronous curves for 8621 and 5731 polyethylenes
(open symbols=5731, close symbols=8621)

Considering the stress in the remaining ligament of a bend specimen, the stress distribution in the uncracked ligament can be modelled as shown in Fig.4.56.

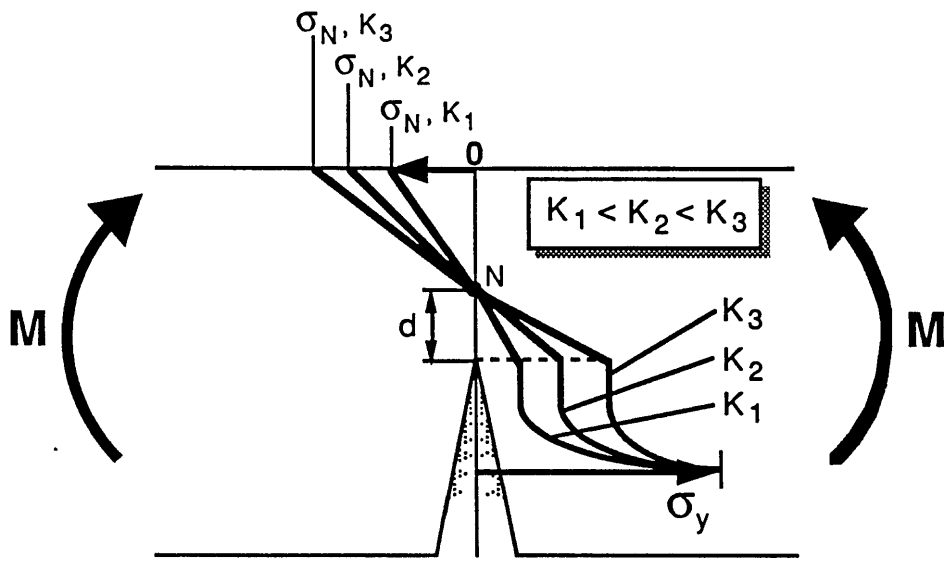


Fig.4.56 Stress distribution at the remaining ligament of a bend specimen

The fibrils are assumed to fail when the stress reaches σ_y . The stress at the damaged zone is assumed to be constant only in the region where voids have not been formed. The stress should increase sharply when voids appear. Tri-axial stresses and strain hardening have not been taken into account. Since σ_y is higher than σ_N , in order to maintain stress and moment equilibrium, the distance d must be small compared to the ligament length. d is conceptually shown as the same in Fig.4.56 for different K_c levels but it is believed to vary with K_c . The stress at the damaged zone will increase as K_c increases. Referring to Fig.4.55, the materials are still fairly linear viscoelastic when the stress is low and the time is short. The time scale for the crack growth process is given in Equation (4.4) and is small when compared to the initiation time. As a result, the deformation process at the damaged zone of a propagating crack can be reasonably assumed as linear viscoelastic if K_c is low. However, when K_c is sufficiently high that the material behaves nonlinear viscoelastically, the failure strain will increase considerably leading to the formation of larger fibrils.

The stress increases as the crack grows. In order to sustain stability, the neutral point N should also travel in the same direction as the crack propagation. However, instability will occur when the crack is very close to the neutral point or the stress in the damaged zone reaches σ_y . Since the distance d should be proportional to the ligament size, a small increase in crack length for specimens with short ligament size will trigger instability. Therefore, fast failure occurs shortly after the crack has initiated but the deformation process may still be linear viscoelastic. This accounts for the short failure time and the low K_c - $\dot{a}$ curves for the samples with small ligament size.

It has been shown that the shear lips play a role in determining the slope of the K_c - $\dot{a}$ curve. By comparing the micrographs illustrated in Fig.4.57, it is obvious that the plastic deformation at the surfaces of the plain-sided sample is more severe. The plastically deformed material will reduce the crack speed as explained earlier.

4.9.2 Crack Growth in Detergent

The fracture surface of a 5731 specimen failed in 50% Arkopal is shown in Fig.4.58 and two distinct crack arrest marks can be clearly seen. The fibrillar structure at region A is shown in Fig.4.59. The fibrils are much longer compared with those observed in Fig.4.50. The detergent appears to provide a 'thinning' effect on the fibrils. It can be seen that in both regions A and B, the size of the fibrils decreases as crack length increases. This is contrary to the crack arrest phenomenon due to crack blunting in which the blunted crack relieves the stress at the crack tip and subsequently causes crack arrest. In such a case, the size of fibrils is expected to increase before crack arrest occurs.

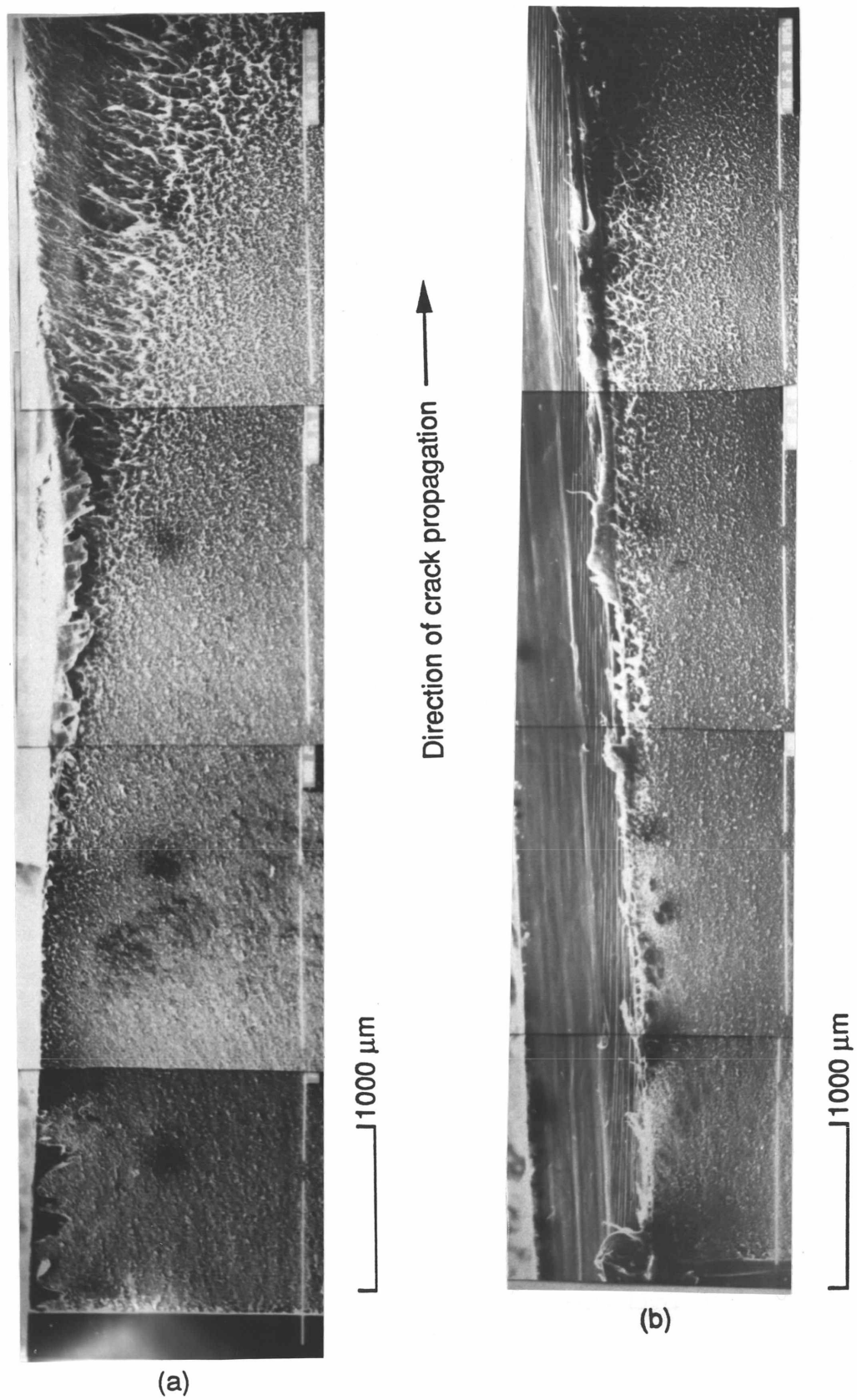


Fig.4.57 The shear lips near the side of 8621 samples. a) Plain-sided, b) Side-grooved

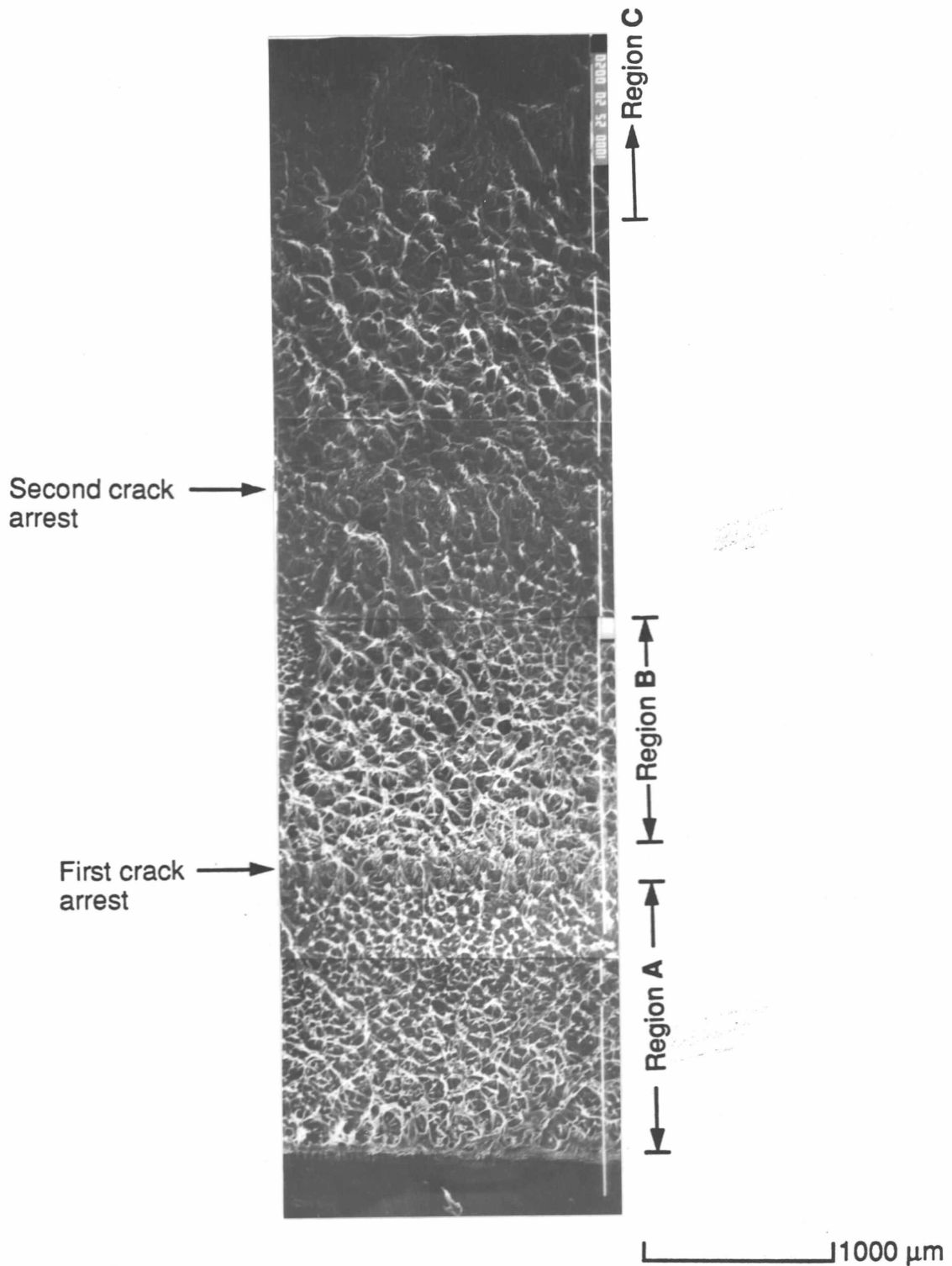


Fig.4.58 Overview of the fracture surface of a 5731 sample failed in 50% of Arkopal

The exact crack arrest mechanisms are not known, but it is suggested that crack arrest is due to the non-equilibrium between the fluid flow speed and the crack propagation rate. When the crack is stationary, there is sufficient time for the detergent to diffuse into the fibrillar region and to be absorbed by the stressed

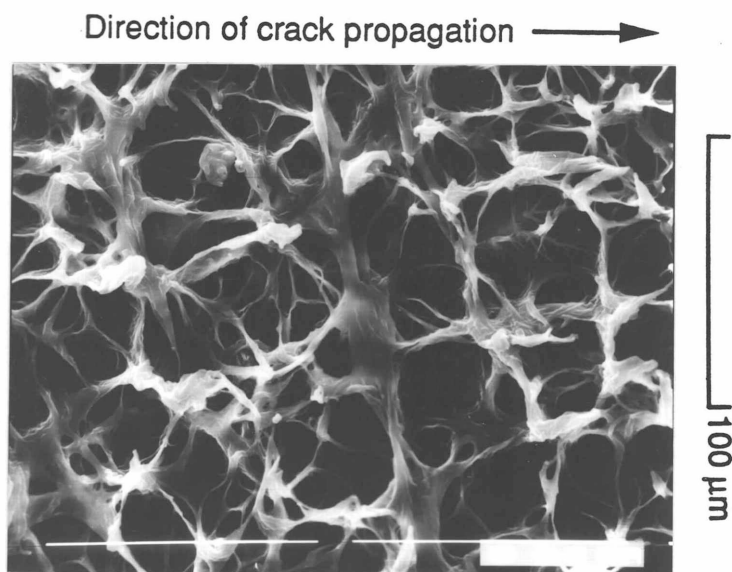


Fig.4.59 Fracture surface of the 5731 specimen in region A

material. The material is plasticized and the crack is able to grow at a lower K_c level. Longer fibrils will also form. Once the crack begins to propagate, there will be less time for the detergent to penetrate into the deformed material, the fibril size and COD will be smaller since plasticization of fibrils is less effective. This further restricts the flow of the detergent. If the crack growth rate reaches a value at which the plasticization effect is too small and the current crack driving force is unable to maintain the crack growth rate, the crack will be arrested or decelerated. The arrest time is normally over 500 hours and there is ample time for the detergent to diffuse into the fine porous structure. Then the whole process starts over again. This leaves a fracture surface with decreasing size of fibrils along the crack growth direction before the crack is arrested.

Careful examination of Fig.4.58 reveals that the fracture morphology at the initial crack, arrest marks and the flow controlled region, region C, is very similar but remarkably different from the fibrillar structure in regions A and B. Micrographs of higher magnification taken at the initial crack tip and the flow controlled region are shown in Fig.4.60 and Fig.4.61. The appearance of these fracture surfaces is astonishing. They give the impression that some of the material has been melted and blister-like fibrils are left. The actual causes of the blisters are far from clear. It is



Fig.4.60 Micrograph of the fracture morphology at the notch tip

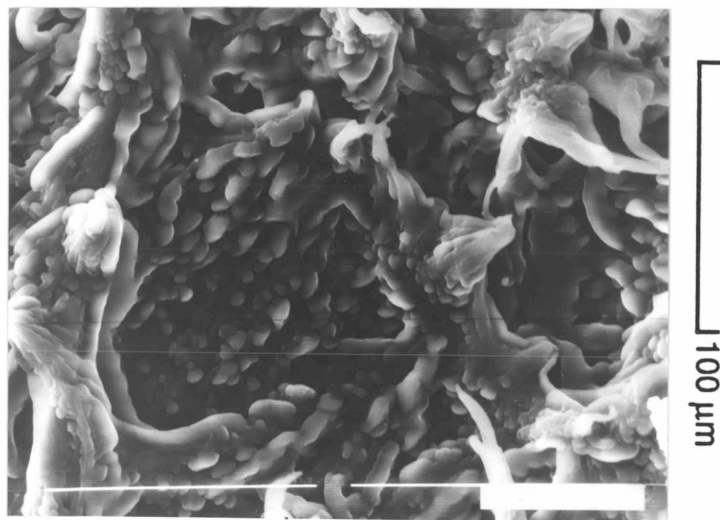


Fig.4.61 Micrograph of the fracture morphology at the flow controlled region

assumed that such a structure will only occur when the plasticization effect of the detergent is at its peak. The effectiveness of the plasticization effect is thought to be proportional to time and stress. The former accounts for the existence of such a morphology at the initial notch and arrest line since the initiation and arrest time is long. At the flow controlled region, the stress is higher and the large *COD* also assists the flow of the fluid, so the material is completely plasticized. Nonetheless, all the crack growth mechanisms proposed are superficial and a more comprehensive study of the micromechanisms is desirable.

4.10 Alternative Approaches for Characterisation of Slow Crack Growth

4.10.1 J -Integral and Net Section Stress

It is known that the J -integral can cope with a higher degree of plasticity during fracture than can K . The thickness effect observed in the K_c - a data may be eliminated by replacing K_c with the parameter J . The J definition described in Section 4.8.2.3 is employed here. The J -integral was evaluated using Equations (4.26) and (4.27). The J - $\dot{a}$ diagrams for specimens with different thicknesses are given in Fig.4.62. The thickness effect of the curves is apparent. Normalization procedures suggested in Section 3.9.5 are not applicable since the curves are not parallel on the log-log basis. Although J can tolerate some form of plasticity, it is still determined from the energy input into the system. For a thin specimen, failure is dominated by plastic deformation at the shear lips, a high J and a slow crack growth rate are

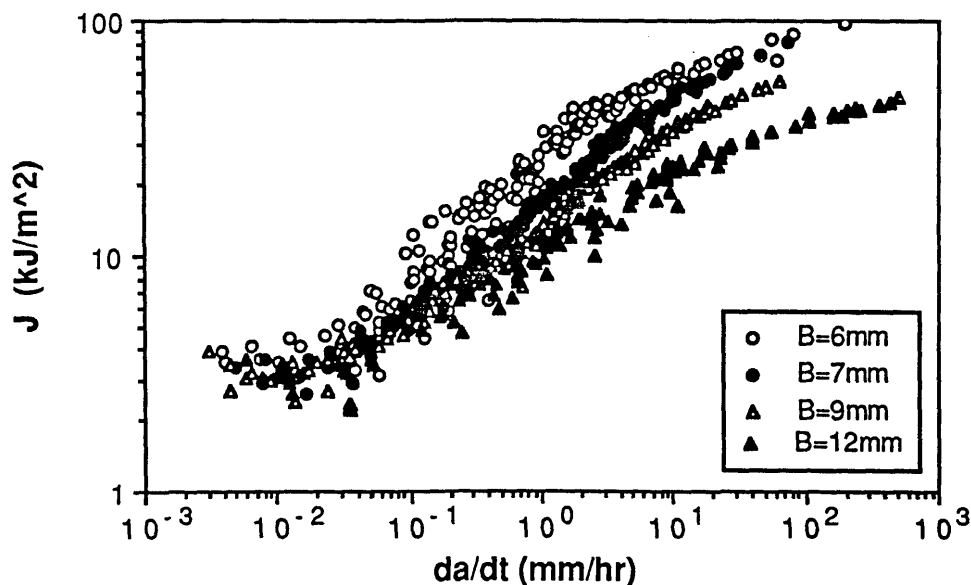


Fig.4.62 J - $\dot{a}$ curves for 8621 specimens with various thicknesses

inevitable. Therefore J is not an appropriate parameter to describe the slow crack growth process. Taylor[1983] proposed that net section stress can be used to correlate the slow crack growth data when the failure is ductile. The variation of $\sigma_N - \dot{a}$ curves with specimen thickness is shown in Fig.4.63. Again the slow crack data do not correlate with each other.

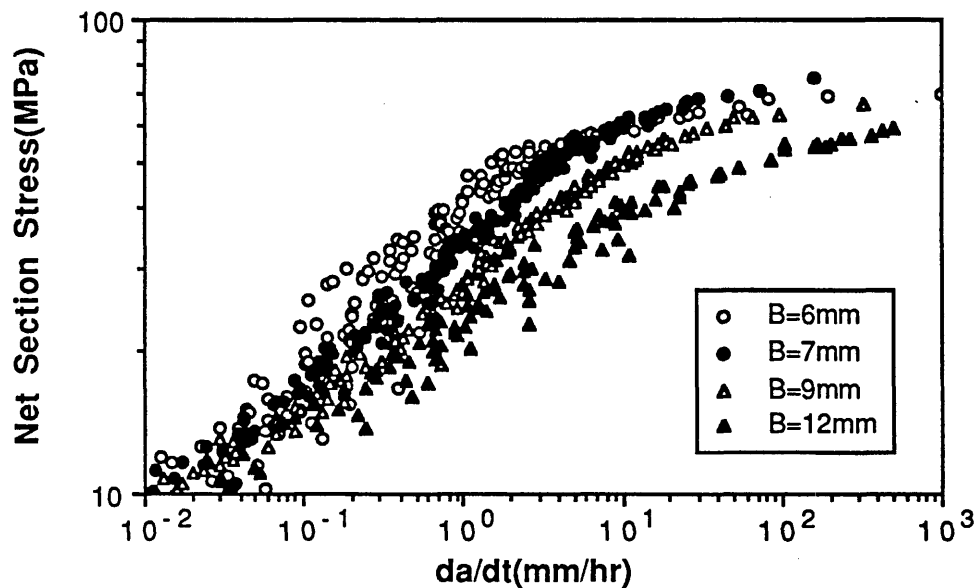


Fig.4.63 Net section stress- $\dot{a}$ curves for 8621 specimens with various thicknesses

4.10.2 The Work Rate Approach

It is believed that the slow crack growth behaviour should be controlled by the rate of irrecoverable work done. The work per unit crack growth area needed to propagate a crack in a thin sample will be higher than that of a thick sample due to the dominance of the shear lip deformation. However the crack growth rate and the rate of work done will be lower because plastic deformation is a rather slow process compared to brittle failure. Thus it is reasonable to assume that the crack speed is

proportional to the rate of irrecoverable work. The work rate per unit crack area can be represented by the parameter C^* . The parameter C^* was originally proposed by Landes and Begley[1976] and is commonly used for characterising the slow crack growth behaviour of metals. C^* can be viewed as the time derivative of the J -integral. The definition of C^* is given as:

$$C^* = \frac{1}{B} \frac{d\dot{U}_P}{da} \quad (4.32)$$

where $\dot{U}_P$ is the time derivative of the potential energy U_P . Riedel[1987] showed that the correlation between C^* and $\dot{a}$ for a power law viscous material with the stress-strain relation of $\sigma \propto \dot{\epsilon}^m$ can be written as:

$$C^* \propto \dot{a}^{m+1} \quad (4.33)$$

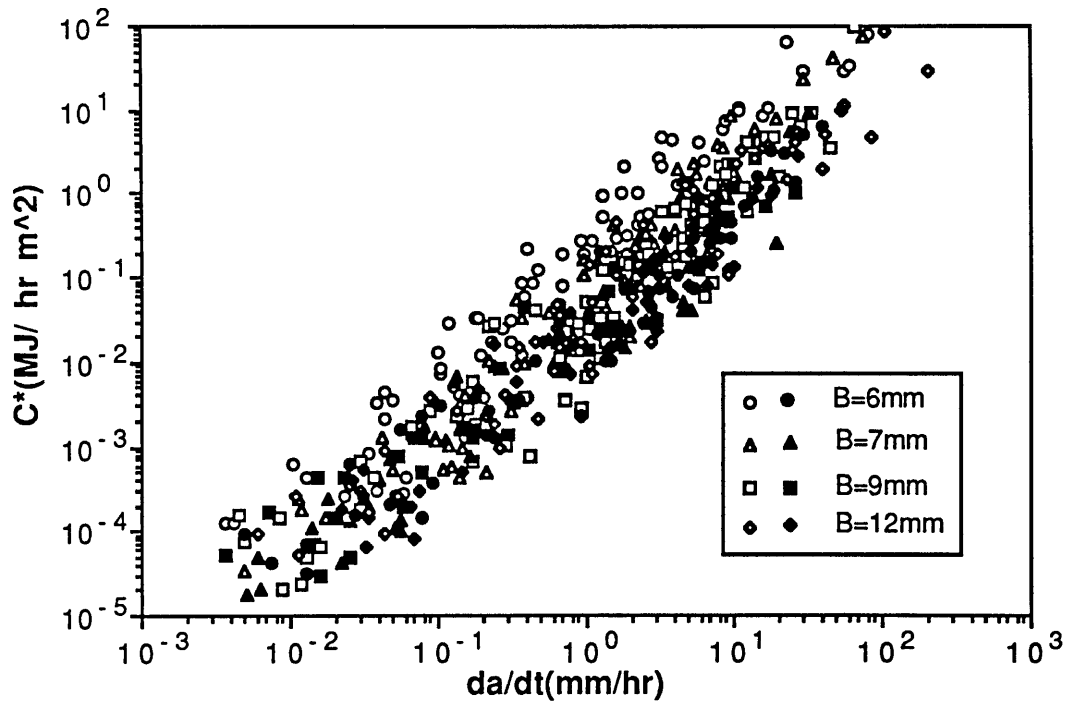


Fig.4.64 C^* - $\dot{a}$ curves for 8621 specimens with various thicknesses
(open symbols=plain-sided specimens, closed symbols=side-grooved specimens)

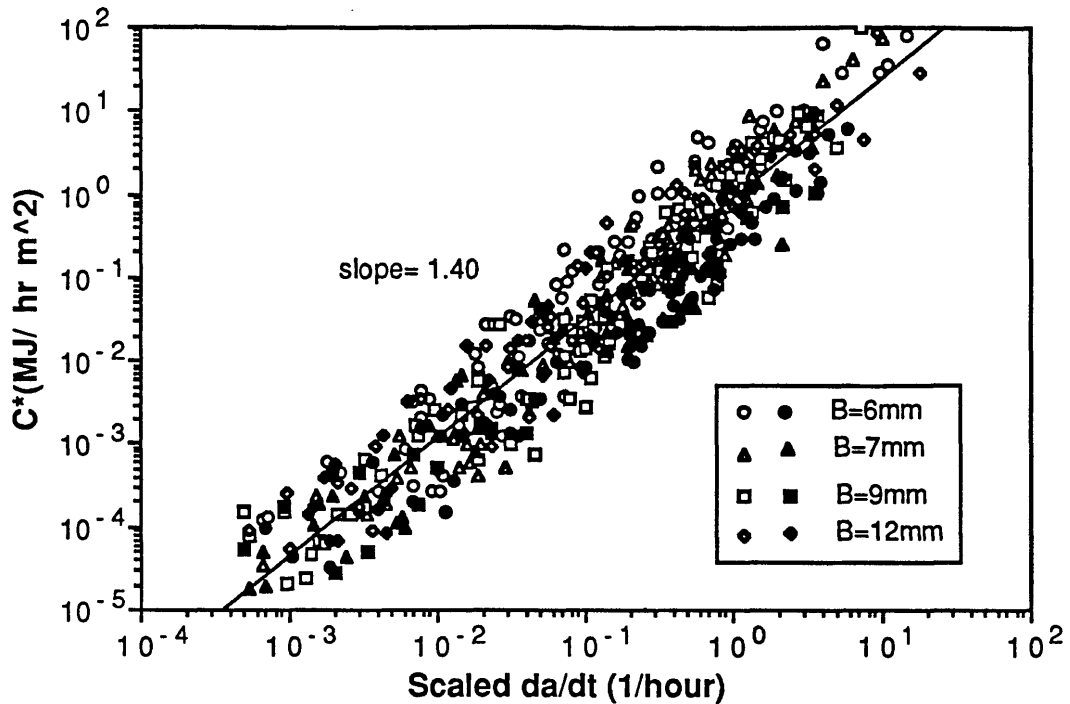


Fig.4.65 Scaled C^* - $\dot{a}$ curves for 8621 specimens with various thicknesses
(open symbols=plain-sided specimens, closed symbols=side-grooved specimens)

Therefore a straight line should be obtained by plotting C^* versus $\dot{a}$ on a log-log basis and the power dependence can be measured from the slope of the line.

The energy was computed using Equations (4.26) and (4.27), then it was differentiated numerically with respect to time and crack length. The slow crack growth data based on the parameter C^* are shown in Fig.4.64. It can be seen that all the curves are parallel and lie very close to each other. The curves give a common slope of 1.4. The C^* - $\dot{a}$ curves for the side-grooved samples are slightly lower than those of the non side-grooved samples. It can also be observed that the 6 mm thick samples give higher C^* - $\dot{a}$ curves. This resembles the thickness effect observed in the J - R curves shown in Chapter 3. Following the argument given in Section 3.9.5, the abscissa is scaled by dividing $\dot{a}$ with (B^2/B_N) . The scaled C^* - $\dot{a}$ curves are illustrated in Fig.4.65. The uniqueness of the curves is improved and the value of slope is also 1.4. The scatter is believed to be caused by the numerical estimation of C^* and $\dot{a}$. The scaled C^* - $\dot{a}$ curves for specimens with different widths and initial K_I

levels are illustrated in Fig.4.66. The scaled C^* - $\dot{a}$ curves for the 5731 samples flatten at high crack speeds as fast failure occurs. The results suggest that the scaled C^* - $\dot{a}$ curves are geometry and K_I level independent. It is also interesting to note that the scaled C^* - $\dot{a}$ curves for both polyethylenes bear the same slope. It may signify that the crack growth mechanisms for both materials are similar and are controlled by the work rate.

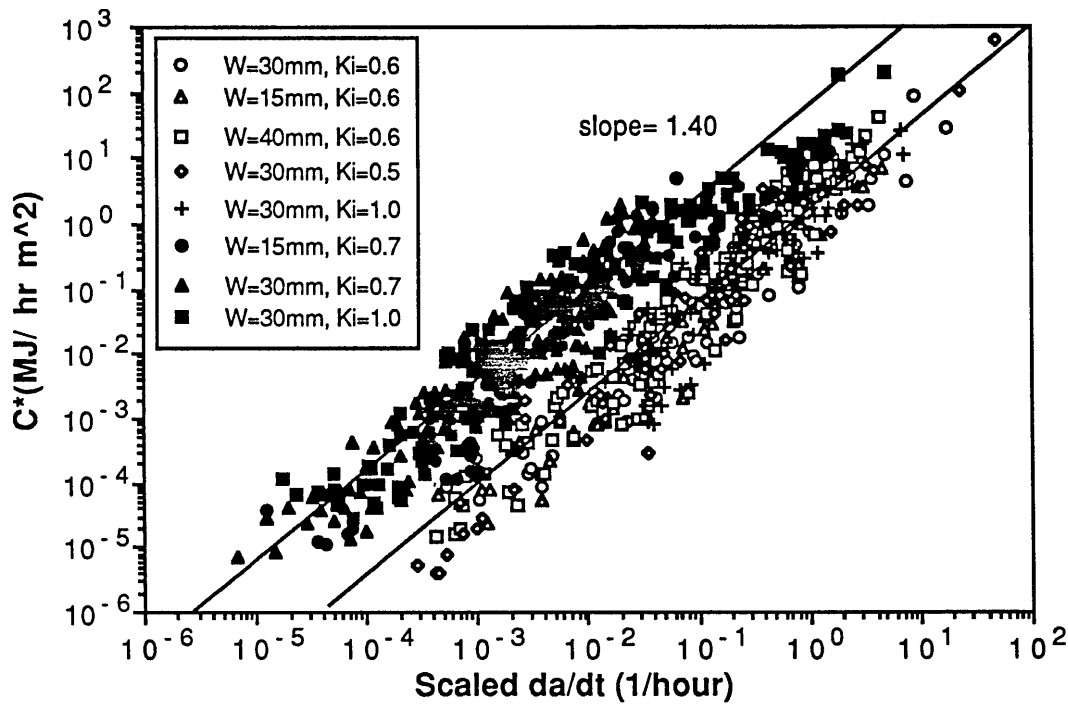


Fig.4.66 Effects of specimen width and K_I levels on scaled C^* - $\dot{a}$ curves
(open symbols=8621 samples, closed symbols=5731 samples)

From the results shown in Fig.2.16, the value of m for the two polyethylenes at low strain rate is 0.1. According to Equation (4.33), a slope of 1.1 is predicted which agrees reasonably well with the measured value of 1.4. It must be pointed out that m will be over-estimated if it is evaluated from the slope of the C^* - $\dot{a}$ curve. This may indicate that Equation (4.33) does not actually describe the slow crack growth of polymers but C^* and $\dot{a}$ follow a power law relationship of the form:

$$C^* \propto \dot{a}^\lambda \quad (4.34)$$

where λ is a constant. However, the correlation is derived from empirical results and it is not clear if Equation (4.34) will hold for all plastics. In order to establish C^* as a controlling parameter for the creep fracture of polymers, experiments on other plastics will be necessary. All in all, application of C^* on slow crack growth in polymers deserves further exploration in the future.

4.11 Conclusions

The compliance method has been found to be an effective means for monitoring the crack length during creep fracture tests. The residual stresses in the polyethylene plaques caused little effect on the determination of K_c unless the magnitude of the residual stresses was comparable with the applied stress. A strong specimen size dependence on the initiation and failure times was observed. It is suggested that K_i is not the only controlling parameter which governs t_i and t_f . The size effect has been proposed to be caused by the variation of net section stress in the uncracked ligament. It has been shown empirically that t_f can be described reasonably well by the net section stress but the exact relationship between the two is still not clear. Despite the scatter in the data, t_i can be predicted using the J -integral approach for the 8621 polyethylene. The changes in the shape of the K_c - $\dot{a}$ characteristics with side-grooving and specimen size have been suggested to be the result of the formation of shear lips and the crack tunnelling effect. It is believed that the K_c - $\dot{a}$ curves will be independent of specimen size and side-grooving if fully plane strain conditions are attained. It has been demonstrated that the slow crack growth in the polyethylenes studied here can be characterised adequately using the parameter C^* . The slopes of the C^* - $\dot{a}$ curves were not affected by changes in specimen geometry and loading conditions, but the positions of the curves were affected by side-grooving and specimen thickness. The uniqueness of the curves could be improved by scaling $\dot{a}$ with the specimen thickness. In the presence of detergent, crack arrests have been

observed for the 5731 specimens. The crack arrest is considered to be due to the inequilibrium of the fluid flow velocity and the crack growth rate.

Concluding Remarks

5.1 Conclusions on the Studies

The plane strain fracture toughness of two high density polyethylenes, designated 5731 and 8621, have been determined using different characterising fracture parameters. Standard test methods were adopted and the applicability of these methods for polymers was examined. The three fracture parameters employed were K_{IC} , G_{IC} and J_{IC} . The specimen size required for obtaining valid plane strain fracture toughness was significantly reduced when the J_{IC} approach was adopted. However, the 5731 polyethylene was so tough that plane strain conditions could only be achieved at extremely low temperature. The mechanical properties of the polyethylenes were found to vary with temperature and loading rate. Hence, K_{IC} and G_{IC} were also found to be temperature and strain rate dependent. The strain rate dependence of the fracture toughness and material properties could be described adequately by simple power laws. K_{IC} and G_{IC} have been shown to be compatible if the requirements for plane strain conditions and the linearity criterion for the load-displacement curve are satisfied. The linearity criterion was particularly important in the determination of G_{IC} . The results from the 8621 polyethylene suggested that the constant crack opening displacement was the appropriate failure criterion for this material. The fracture toughness of this material evaluated using the J -integral

approach was consistent with that obtained from K method. The J_{IC} value showed very little variation with specimen size and side-grooving except for the very thin samples. Based on the results obtained from the plane strain fracture toughness studies, it has suggested that the ASTM size criteria are not adequate for the 8621 polyethylene, the minimum required specimen dimensions are larger than those evaluated from these criteria.

It has been demonstrated that the unloading compliance method is feasible for determining the J - R curves for polymers. The results obtained from the unloading compliance method and the multiple specimen method were in good agreement. However, the unloading compliance method was found to be inappropriate for thin specimens since the compliance response was controlled by the plastically deformed material in the shear lips. The R -curves were found to be affected by the specimen thickness and side-grooving. The curve showed a tendency to rise with decreasing specimen thickness. This was due to the increase in the size of the shear lips relative to the specimen thickness. Side-grooved specimens tended to give lower R -curves but the thickness effect was less prominent. A thickness independent R -curve could be obtained by normalising the abscissa with the specimen thickness.

Information obtained from the short term tests could not be used to predict the long term performance of the materials since the time scales involved were different and cracks could initiate at K levels below K_{IC} . For the creep fracture tests, it was found that the crack lengths could be monitored satisfactorily using the compliance method. The crack initiation time, t_i , and failure time, t_f , of the polyethylenes under constant loading were measured. For the same initial applied K level, K_i , it was found that t_i and t_f for specimens with various sizes were different and the results could not be correlated. Therefore, it was proposed that t_i and t_f depend not only on K_i . A good correlation between t_f and the net section stress was obtained but the exact

mechanisms are still not clear. The J -integral approach was used to characterise the initiation time for the 8621 polyethylene. A unique relationship independent of the specimen size and loading conditions between t_i and the J value at initiation was found. It agreed well with the theoretical model proposed by Williams.

The K_c - $\dot{a}$ characteristics were found to vary with side-grooving, specimen size and K_i level. These effects were attributed to the increase in the shear lip size. The K_c - $\dot{a}$ curve became steeper as the shear lip size increased. The results suggested that the K_c - $\dot{a}$ curve would only be size independent if fully plane strain conditions were achieved. The slow crack growth behaviour could be neither characterised by the J -integral nor the net section stress. It was found that the parameter C^* offered a better description of the slow crack growth behaviour of the polyethylenes. All the C^* - $\dot{a}$ curves showed the same slope irrespective of specimen size and loading conditions. The positions of the curves were slightly thickness dependent, but the uniqueness of the curves could be improved by scaling the abscissa with the specimen thickness. Furthermore, the slopes of the C^* - $\dot{a}$ curves for both polyethylenes were the same. This indicated that crack growth in both polyethylenes might share the same underlying mechanisms.

Crack arrests were observed when the 5731 specimens were loaded in the presence of detergent. The crack arrests were proposed to be caused by the inequilibrium of fluid flow speed and crack speed. The crack speeds at the flow controlled transition were found to be constant, which agreed with Williams' flow controlled model.

One of the problems associated with all these tests is the arbitrary definitions of the crack initiation point. The crack was observed to initiate first at the centre and then spread to the sides of the specimen. The initiation point in the J - R curve is never well defined, and the first sign of fibril breakage may not fully describe the initiation event

in the long term tests. The definition of initiation point can affect the results remarkably, and more stringent definitions are necessary for both short and long term testing. It is also interesting to note that some of the results can be scaled or normalised to eradicate size effects. Consider the J - R curve, the ordinate is the energy required for a unit crack growth area which is a relative quantity, but the abscissa is the amount of crack growth which is an absolute quantity. The amount of crack growth in a large specimen will be quite different from that in a small specimen even though the energy per unit area is the same. The number of defects and the degree of constraint in a large structure will be higher those that in a small laboratory specimen. When J_{IC} is reached, the consequence of having all the defects in the large structure growing at one time is by no means the same as that of growing the defects in the small specimen. Therefore it is felt that the abscissa should also be a relative quantity rather than an absolute quantity.

5.2 Recommendations for Future Research

The possibilities of extending the current studies are almost infinite, but there are several areas to which attention should be drawn.

The use of J_{IC} in characterising the fracture resistance is questionable due to its ambiguous definition and the complicated blunting mechanisms of polymers. Alternatively, it may be possible to characterise the fracture resistance using the complete J - R curve. Although the J - R curve is apparently size dependent, the normalization procedure may serve as a solution to this problem. If the normalised R -curve is unique and is a material property, it will be invaluable in evaluating the fracture resistance of materials. Further research on investigating the validity of the normalisation procedures would be desirable. It has been shown that the single specimen method can be applied to polymers for determining the J - R curves. The

current procedures for locating the linear part of the unloading loop is tedious and sometimes may be subjective. It would be advantageous to computerise the whole process in the future, so that the exact location of the elastic region can be sought, and the accuracy in crack length prediction may be improved.

The time for the crack to initiate under creep loading should be affected by the sharpness of the initial crack. It would be interesting to investigate the notching methods further and try to find the best way to produce an artificial crack which is more similar to the natural crack. Although the J -integral and net section stress appear to be adequate in describing the initiation and failure times respectively, there are still insufficient data to support the applicability of these approaches to other polymers. This question will require further clarification. In order to establish C^* as the controlling parameter for slow crack growth in polymers, cross-examination with other polymers is mandatory. Finally, it is still not known whether threshold K_I levels exist for polymers, this would be an interesting area of exploration for future research.

Appendix I

Experimental Determination of Residual Stresses

The residual stresses in a planar body can be measured using the layer removal technique. The concept is based on the disturbance of stress and moment equilibrium within the body. The technique has been described in details by Russell et al.[1980] and So et al.[1976]. Consider a rectangular bar of thickness $2l$ containing only longitudinal stresses and behaving elastically. The stresses are assumed to be compressive in the surface and tensile in the center as shown in Fig.A1. Removal of a thin layer from the surface will disturb the force and moment equilibrium of the beam and the curvature of the bar will change due to the redistribution. The residual stress can be evaluated from the change of curvature.

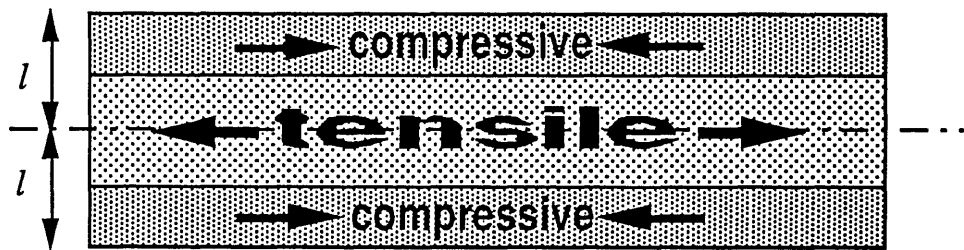


Fig.A1 The residual stresses in a rectangular bar

The layout of the apparatus is shown in Fig.A2. The deflection of the beam is measured using a dial gauge. The curvature of the beam is given as:

$$\Phi(x) = \frac{\delta f}{L^2} \quad (A1)$$

where x is the distance measured from the center of the beam. If the curvature of the bar is $\Phi(x_1)$ after reducing its thickness to $l + x_1$ as illustrated in Fig.A3, then the residual stress which existed originally at the layer x_1 , $\sigma_r(x_1)$, can be written as:

$$\sigma_r(x_1) = \frac{-E}{6(1-\nu^2)} \left[(l+x_1)^2 \frac{d\Phi(x_1)}{dx} + 4(l+x_1)\Phi(x_1) - 2 \int_{x_1}^l \Phi(x_1) dx \right] \quad (A2)$$

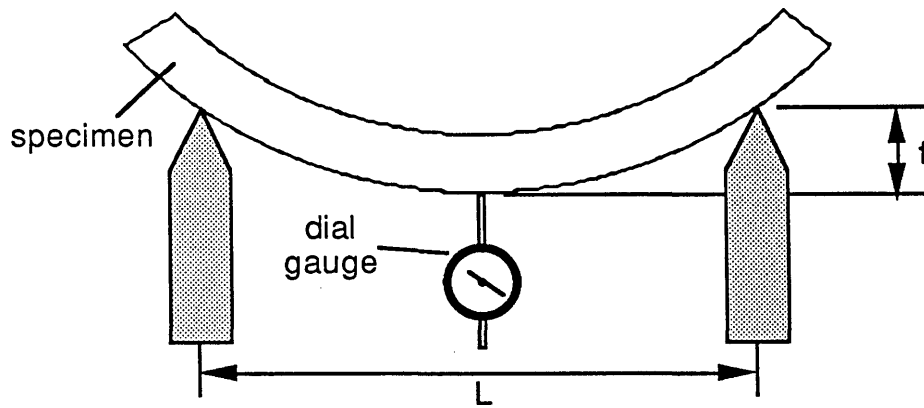


Fig.A2 The layout of the apparatus for measuring beam curvature

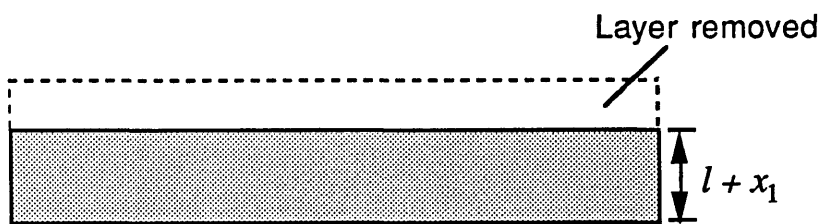


Fig.A3 Removal of a layer of material from the bar

From Equation (A1), Equation (A2) can be expressed as:

$$\sigma_r(x_1) = \frac{-4E}{3(1-\nu^2)L^2} \left[(l+x_1)^2 \frac{df(x_1)}{dx} + 4(l+x_1)f(x_1) - 2 \int_{x_1}^l f(x_1) dx \right] \quad (A3)$$

where E is the elastic modulus and ν is the Poisson's ratio. By plotting the specimen deflection against the thickness of removed materials as shown schematically in Fig.A4, all the terms in Equation (A3) can be evaluated. Hence the residual stress distribution across the thickness can be found.

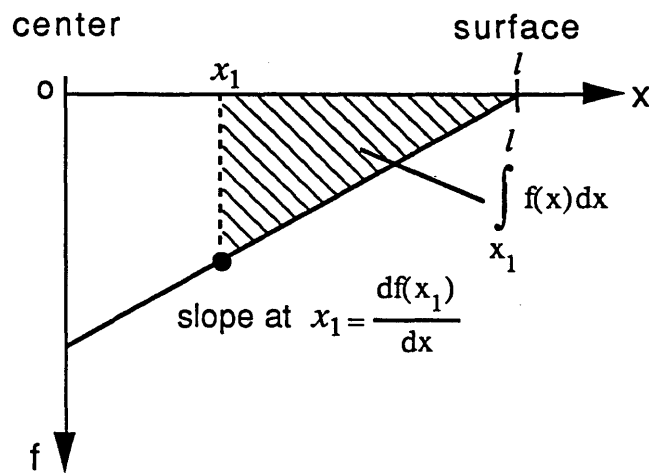


Fig.A4 Schematic diagrams of the deflection against removed thickness curve

Appendix II

Effect of Residual Stresses on K_c

Referring to Section 4.5 in Chapter 4, the average value of the actual K_c value across the specimen thickness is given as:

$$\begin{aligned}\tilde{K}_s^2 &= \frac{Y^2 a}{l} \int_0^l [\sigma_a + \sigma_r(x)]^2 dx \\ &= Y^2 \sigma_a^2 a + \frac{Y^2 a}{l} \int_0^l \sigma_r(x)^2 dx\end{aligned}\quad (B1)$$

An analytical solution of $\sigma_r(x)$ of a sheet with thickness $2l$ has been developed by Williams[1981] :

$$\sigma_r(x) = \frac{\alpha E(t')}{l-\nu} (T_m - T_c) \left(\frac{Nu}{2+Nu} \right) \left[\frac{1}{3} - \left(\frac{x}{l} \right)^2 \right] \quad (B2)$$

$$= \chi \left[\frac{1}{3} - \left(\frac{x}{l} \right)^2 \right] \quad (B3)$$

$$\text{and } \chi = \frac{\alpha E(t')}{l-\nu} (T_m - T_c) \left(\frac{Nu}{2+Nu} \right) \quad (B4)$$

where $E(t')$ is the modulus value after long time from moulding process. T_m and T_c are the softening temperature and the temperature at which the body cools respectively. Nu is the Nusselt number defined as hl/k , where h is the surface heat coefficient and k is the thermal conductivity. For a particular Nu , χ is constant. Substituting Equation (B3) into Equation (B1),

$$\tilde{K}_s^2 = Y^2 \sigma_a^2 a + \frac{Y^2 \chi^2 a}{l} \int_0^l \left[\frac{l}{3} - \left(\frac{x}{l} \right)^2 \right]^2 dx$$

$$\tilde{K}_s^2 = Y^2 \sigma_a^2 a + \frac{4}{45} Y^2 \chi^2 a$$

Since $K_{app} = \sigma_a Y \sqrt{a}$, then

$$\frac{\tilde{K}_s}{K_{app}} = \sqrt{1 + \frac{4}{45} \left(\frac{\chi}{\sigma_a} \right)^2} \quad (B5)$$

The material properties of a typical high density polyethylene and the variation of χ with Nu is given in Table-B1.

$T_m=60^\circ\text{C}$, $T_c=23^\circ\text{C}$, $E(t')=0.5 \text{ GPa}$, $\alpha = 12 \times 10^{-5} \text{ }^\circ\text{C}^{-1}$ and $\nu=0.35$	
Nu	$\chi \text{ (MPa)}$
∞	3.4155
10	2.8461
5	2.4396
2	1.7076
1	1.1385
0.5	0.6831
0.1	0.1626

Table-A1 Variation of χ with Nu for a high density polyethylene

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