Evaluation of Impact Fracture Toughness of Polymeric Materials by Means of the J-Integral Approach

LAURA A. FASCE, VALERIA PETTARIN, ROCÍO SELTZER, *and* PATRICIA M. FRONTINI*

División Polímeros
Instituto de Investigaciones en Ciencia y Tecnología de Materiales
Av. J. B. Justo 4302, 7600, Mar del Plata, Argentina

The most commonly accepted method of determining impact fracture toughness of polymeric materials that exhibit small scale yielding and negligible influence of dynamic effects is given by the ISO/DIS 17281 Standard, which states that for brittle behavior, basically a linear relationship exists between the fracture energy, U, and the energy calibration factor, ϕ. This relationship allows calculation of the critical strain energy release rate, G_{IC}, from the slope of the U vs. $BW\phi$ plot. This paper describes a simpler alternative methodology capable of evaluating impact fracture toughness using the J_c parameter. The J-integral is evaluated at the instability load point, by calculating the fracture energy required to produce cleavage behavior of a pre-cracked specimen. The methodology is limited to single edge notched three-point-bending specimens with a crack to depth ratio equal to 0.5. Tests were carried out on an instrumented falling weight impact testing machine on the following materials: PP (polypropylene), HDPE (high-density polyethylene), MDPE (mid-density polyethylene) and RT-PMMA (rubber toughened polymethylmetacrylate). Results are in excellent agreement with the critical values determined by the ISO/DIS 17281 Standard.

INTRODUCTION

Williams and Adams (1) have pointed out the difficulty of obtaining reliable data from instrumented impact tests at high impact speeds. The growing use of polymeric materials in engineering applications demands new methodologies in order to assess their capability to withstand load. It is well known that thermoplastics, even the toughened grades, are relatively susceptible to impact fracture. Impact testing is widely used to characterize the fracture resistance of polymers in industry because it attempts to simulate the most severe loading conditions to which a material can be subjected and because it also diminishes the viscoelastic effects.

Instrumented impact testing is gaining considerable practical importance, as it allows the assessment of the material toughness under the most critical conditions, i.e. high strain rates and presence of notches. As is well known, the use of fracture mechanics allows one to describe the material toughness by parameters that are true material properties (2). The use of fracture mechanics to analyze such tests has greatly improved their utility, and, with the recent availability of good high speed recording equipment, there has been much progress reported.

In the framework of fracture mechanics theory, fracture toughness determination is conditioned by the behavior displayed by the material itself. To date, very few procedures have been standardized (3-5) in the polymer field and they are not still able to cover all possible behaviors.

Linear elastic fracture mechanics (LEFM) have been widely applied for the evaluation of the fracture behavior of brittle polymers when the size of the plastic zone is much smaller than the in-plane specimen dimensions. For these polymers, the initiation of unstable fracture can be accurately described by either K_{IC} or G_{IC}. The most widely accepted method to determine the high rate fracture toughness (around 1 ms^{-1}) for linear-elastic polymeric materials behavior is the G_{IC} methodology (6–8) since it avoids the need to determine the Young's modulus (E) at a reliable test rate. However, when materials are crack length sensitive, the determination of G_{IC} by the ISO/DIS

*To whom correspondence should be addressed.

17281 methodology may present some drawbacks. For stable fracture, elastic-plastic fracture mechanics (J-Integral methodology) and post yield fracture mechanics (essential work of fracture method) concepts have been introduced (9, 10). Because of the inherent difficulties of the J-R methodology, only non-general accepted protocols exist for polymers up to now. However, in many cases there is some non-linearity in the load-displacement diagram, and this can be due to either plastic deformation at the crack tip or stable crack growth after initiation but prior to instability. The first effect violates the LEFM assumption and the latter means that the true initiation is not defined by the maximum load. Hence, the so-called semibrittle behavior constitutes the most difficult problem.

In this paper we have proposed the use of the "cleavage fracture toughness," J_c, to assess fracture toughness of polymers displaying either linear or non-linear unstable fracture pattern under dynamic conditions. This parameter has been successfully applied to metals and polymers in the brittle-ductile transition regime (11–14). It only consists of calculating the J-Integral at the point of unstable fracture (instability load point), which may or may not be preceded by plastic deformation or very little slow crack growth. One of the first researchers who succeeded in applying the J_c method to calculate G_{IC} of a PP homopolymer was Bramuzzo (15) in 1989.

Impact fracture toughness of PP, HDPE, MDPE and RTPMMA has been assessed by the two mentioned approaches. The different fracture toughness parameters calculated from both approaches are compared and their pertinence is also discussed.

EXPERIMENTAL DETAILS

Experiments were conducted on different polymeric materials: a commercial polypropylene homopolymer, Cuyolem NX1100 (PPH), and a novel blend based on PPH modified with 30% wt of an elastomeric polyolefin (PP + 30% POEs), both kindly supplied by Petroquímica Cuyo SAIC. Three commercial grades of polyethylene (PE)—a high-density PE (HDPE), a discontinuous short glass fiber reinforced HDPE (DSGF-HDPE) with a fiber weight fraction of 3% and a mid-density PE (MDPE)—were kindly provided by Siderca. A third generation bimodal PE (PE100) was kindly supplied by Repsol and a rubber toughened polymethylmetacrylate (RT-PMMA) was kindly provided by Ineos Acrylics. Materials as well as their tensile properties are listed in *Table 1*.

Pellets of the materials were compression molded into 8–10 mm thick plaques.

Rectangular bars used in fracture experiments were cut from the compression molded plaques and then machined to reach the final dimensions and improve edge surface finishing. Sharp notches were introduced by scalpel-sliding, a razor blade having an on-edge tip radius of 0.13 mm. In the case of J_c determination, at least seven specimens of equal a_0/W ratio were tested, whereas more than fifteen specimens with different a_0/W ratio were used in G_{IC} determination.

Table 1. Tensile Properties of the Materials Under Study.

Material ID	E (GPa)	σ_y (MPa)
PPH	1.60	37
PP + 30% POEs	1.06	25
HDPE	0.71	25
DSGF-HDPE	0.87	31
MDPE	0.60	14
PE-100	0.94	21
RT-PMMA	2.01	65

Impact testing was carried out using a falling weight type machine, Fractovis 6789 by Ceast, in three-point-bending (mode I) at room temperature and at 1 m/s. For the sake of simplicity, pre-cracked specimens were impacted without cushioning. The specimen thickness, B, and the span to depth ratio, S/W, were always kept equal to $W/2$ and 4, respectively. PE100 samples were side grooved in order to avoid bowing of the crack front and ductile propagation after initiation (16). Energy values were computed from the non-filtered load-displacement curves. Spurious contributions to the measured energy due to machine compliance and specimen indentation were corrected following the procedure recommended in ISO/FDIS 17281 (Section 8.2.1) (3).

The original crack length, a_0, and the stress whitened zone length, r_p, were physically measured from the fracture surface using a profile projector with a magnification of 20×. Fracture surface appearance was examined using an Olympus SZH 10 Optical Microscope. A fracture surface appearance is shown schematically in *Fig. 1*.

DATA ANALYSIS

Linear Elastic Fracture Mechanics, G_{IC} Determination

Linear Elastic Fracture Mechanics, Energy Release Rate, G_{IC} Determination ISO/DIS 17281 Standard (3) states that for brittle behavior, basically a linear relationship exists between the fracture energy, U; and the energy calibration factor, ϕ. This relationship allows, by testing specimens having a crack depth ranging from 0.2 to 0.7 (a/W), calculation of the critical strain energy release rate G_{IC} for unstable fracture from the slope of the U vs. the product of the specimen dimensions and the energy calibration function $BW\phi$. U is the energy absorbed by the specimen during fracture, B and W are the specimen thickness and width, respectively, and the calibration factor, ϕ, depends on the length of crack size (a_0) of the sample.

For G_{IC} determination, U was taken as the energy absorbed up to the maximum load F_{max}, as schematically shown in *Fig. 2*. In the case of limited plasticity, the Corrected Elastic Fracture Mechanics concept was applied (17). It basically consists of replacing a_0 by an

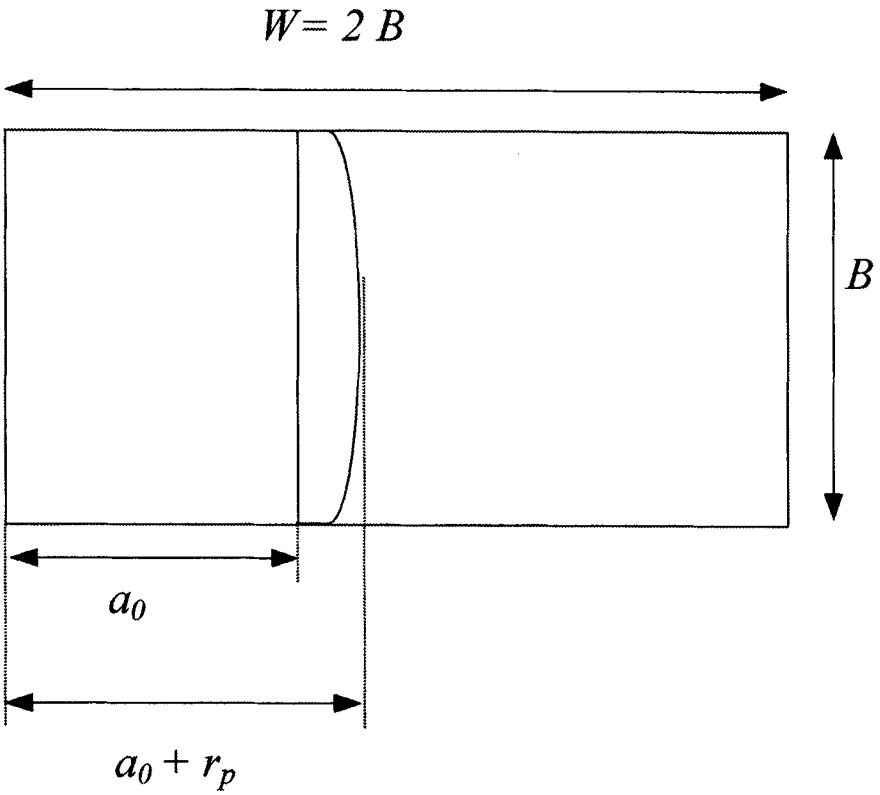


Fig. 1. Scheme of a fracture surface.

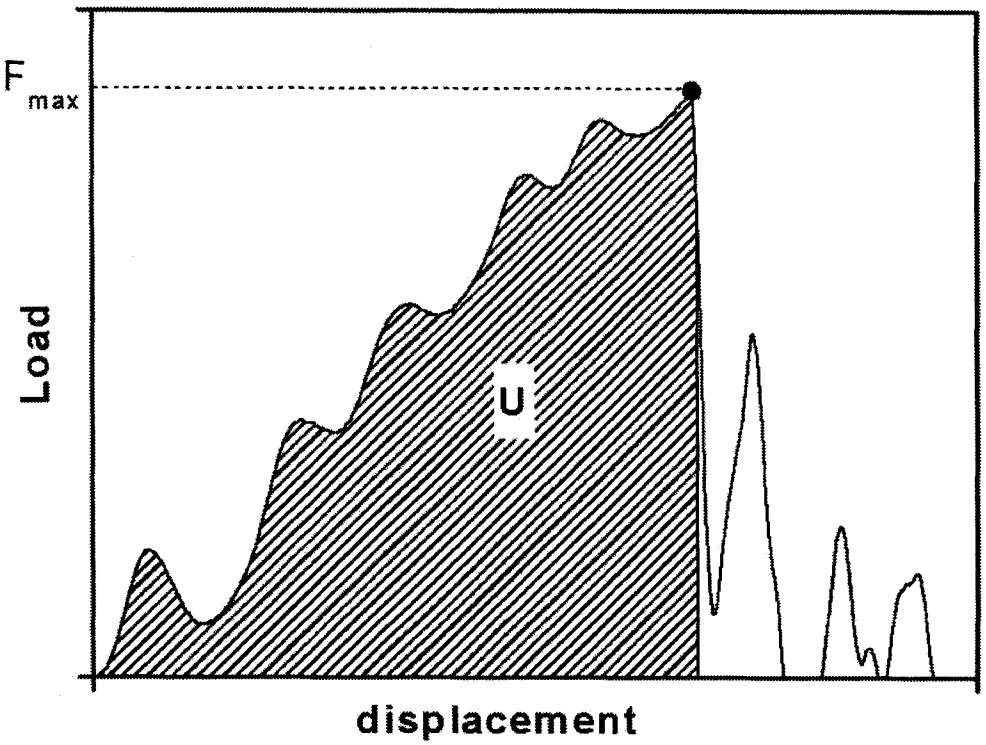


Fig. 2. Fracture energy, U, determination from a load-displacement record.

effective crack length $a_{eff} = a_0 + r_p$, and then calculating G_{IC} following the standard procedure. For the latter calculations, r_p was taken to be the white halo denoted on the post mortem fracture surfaces without distinguishing between sub-critical crack growth and true plastic deformation zone (*Figs. 3b, d, e, f, g*).

Non-Linear Elastic Fracture Mechanics, J_c Determination

The *J*-Integral is conventionally defined for non-linear elastic materials as a path independent line integral. In fact, the single-specimen *J* formulation has been extensively used in the past to characterize ductile polymer fracture under impact conditions (17–19). Although ASTM E813-87 and ASTM E1152-87 apply only to ductile fracture, more recent standards permit *J* testing of materials that fail by cleavage. The J_c parameter (17, 20, 21) as defined here is applicable to characterize quasi-brittle failure behavior (quasi-linear load–displacement curves with sharp load drop at the point of fracture) provided that the specimens used are single-edge-notched three-point-bending specimens with a crack to depth ratio close to 0.5. Under the former condition, the factors (η_{el} and η_{Pl}) relating *J* with the work done on the specimen by the applied load can be considered equal to 2. The *J*-Integral was evaluated at the instability load point (*Eq 1*), by calculating the fracture energy required to produce cleavage behavior (*Fig. 2*) of pre-cracked specimens having a crack depth to width ratio of $0.45 \leq a/W \leq 0.55$ in order to determine "cleavage fracture toughness" (J_c).

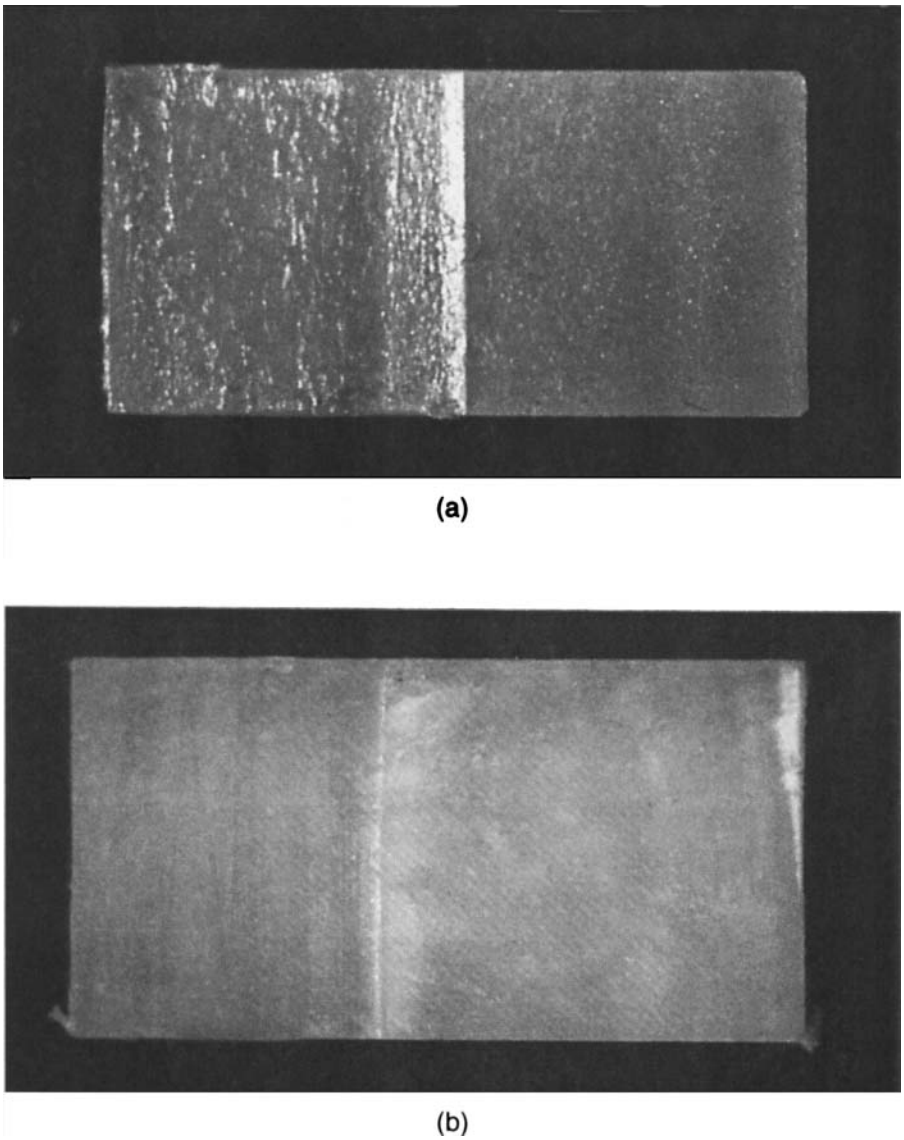
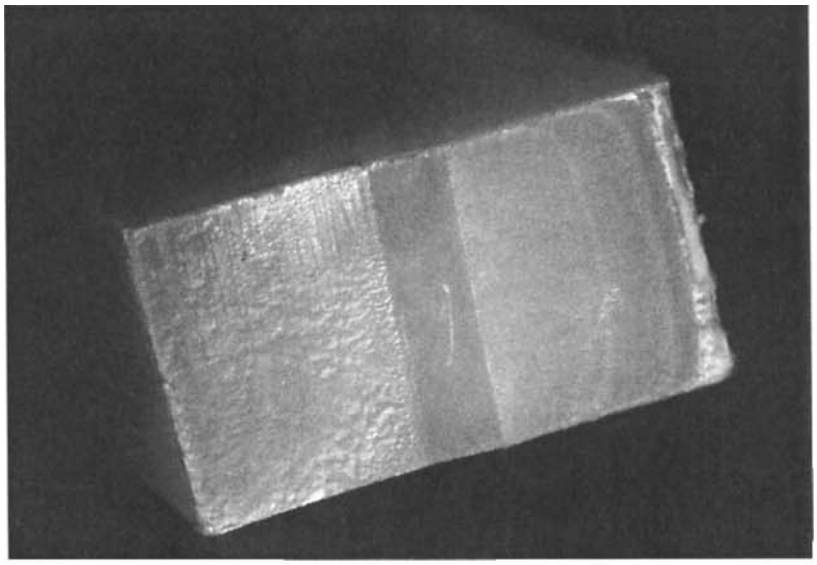
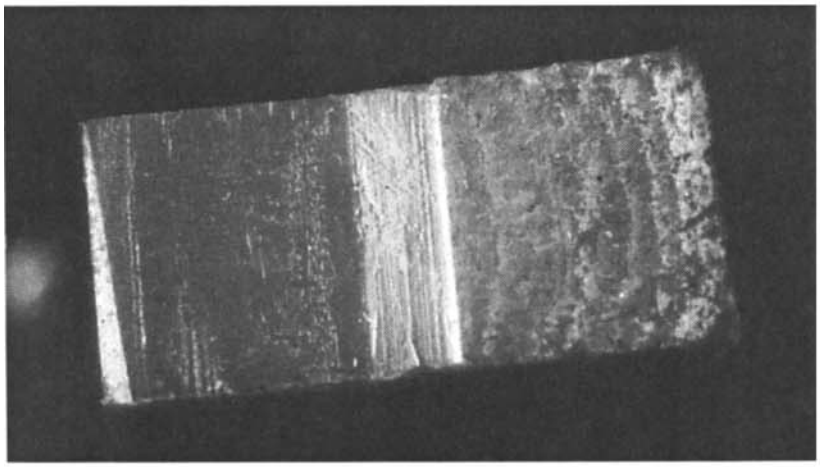


Fig. 3. Fracture surfaces. a) PPH; b) PP + 30% POEs; c) HDPE; d) DSGF-HDPE; e) MDPE; f) PE-100; g) RT-PMMA. In all cases, crack propagation is from left to right.



(c)



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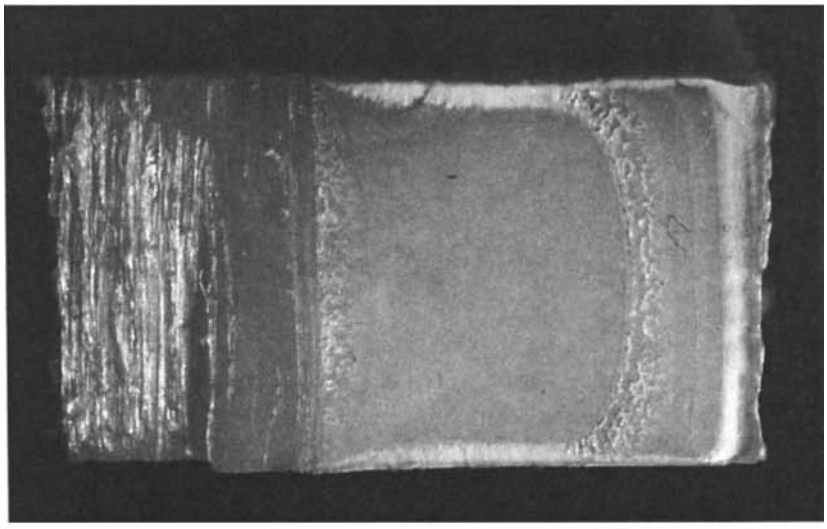
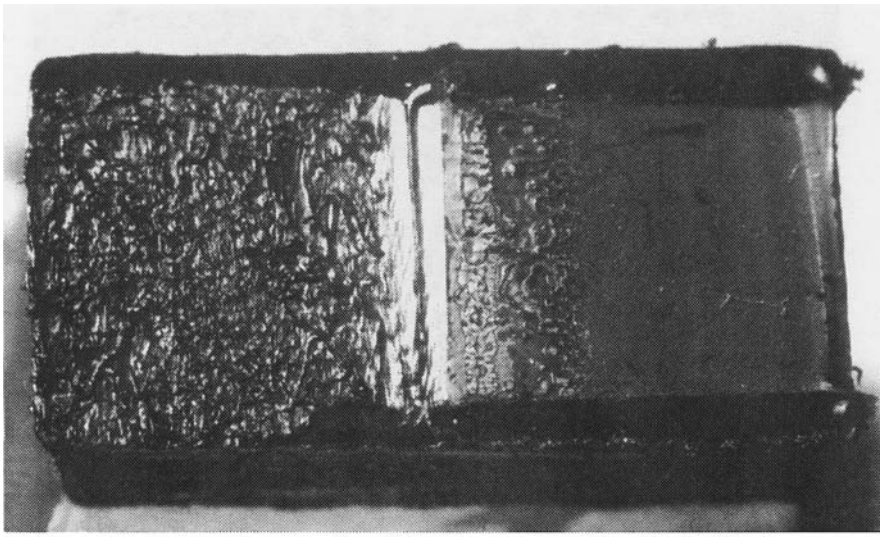


(e)

Fig. 3. Continued.



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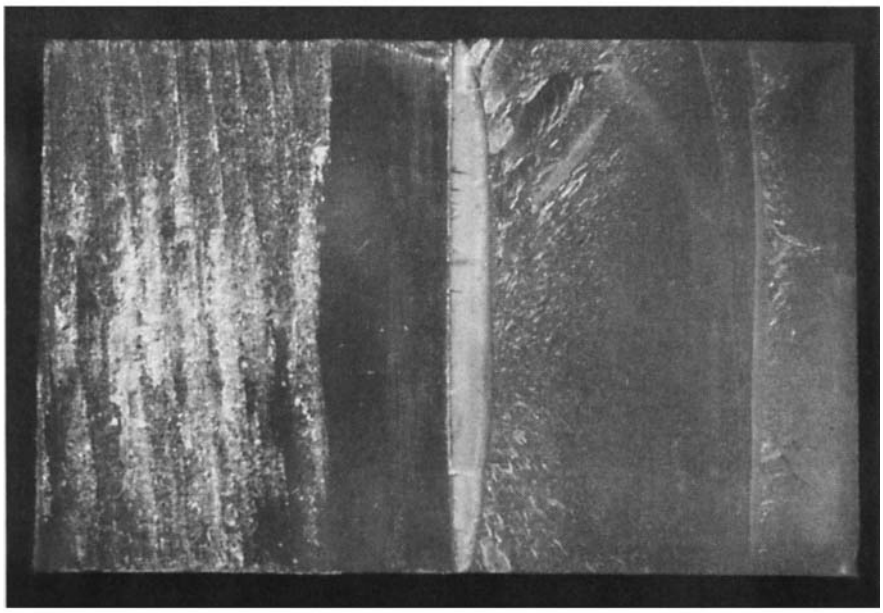


(g)

Fig. 3. Continued.

$$J_c = \frac{2U_{tot}}{B(W - a)} \quad (1)$$

RESULTS AND DISCUSSION

Fracture Patterns

Typical load-time curves of materials obtained during instrumented impact tests are given in *Fig. 4*. Superimposed oscillations of the force signal are due to the well-known dynamic effects in impact testing (1, 20). However, they are not significant when fracture toughness is intended to characterize by means of energy-based methodologies (22).

All samples fractured in an unstable manner but some differences in fracture behavior were observed for several materials.

PPH and HDPE exhibited complete brittle fracture as judged from the linearity of the load deflection records (*Figs. 4a, c*) and the feature of the fracture surface (*Figs. 3a, c*). Load-time curves dropped to zero instantaneously upon reaching the maximum load at relatively short time levels. Consistently, the fracture surface features were mirror-smooth without stress whitening.

Elastomer modification of the PPH matrix, as well as low incorporation of glass fiber to HDPE, caused the materials to develop limited plasticity ahead of the

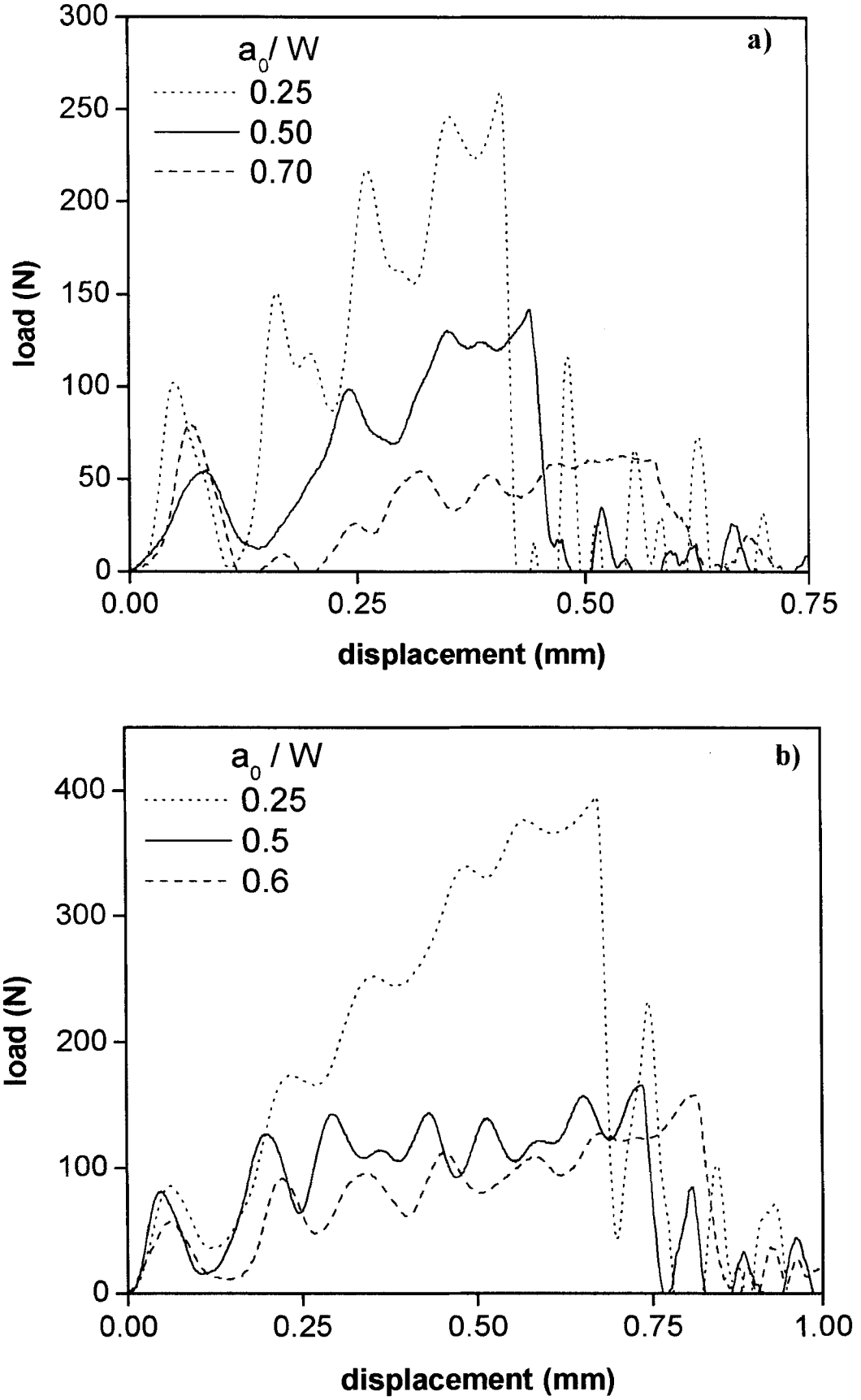
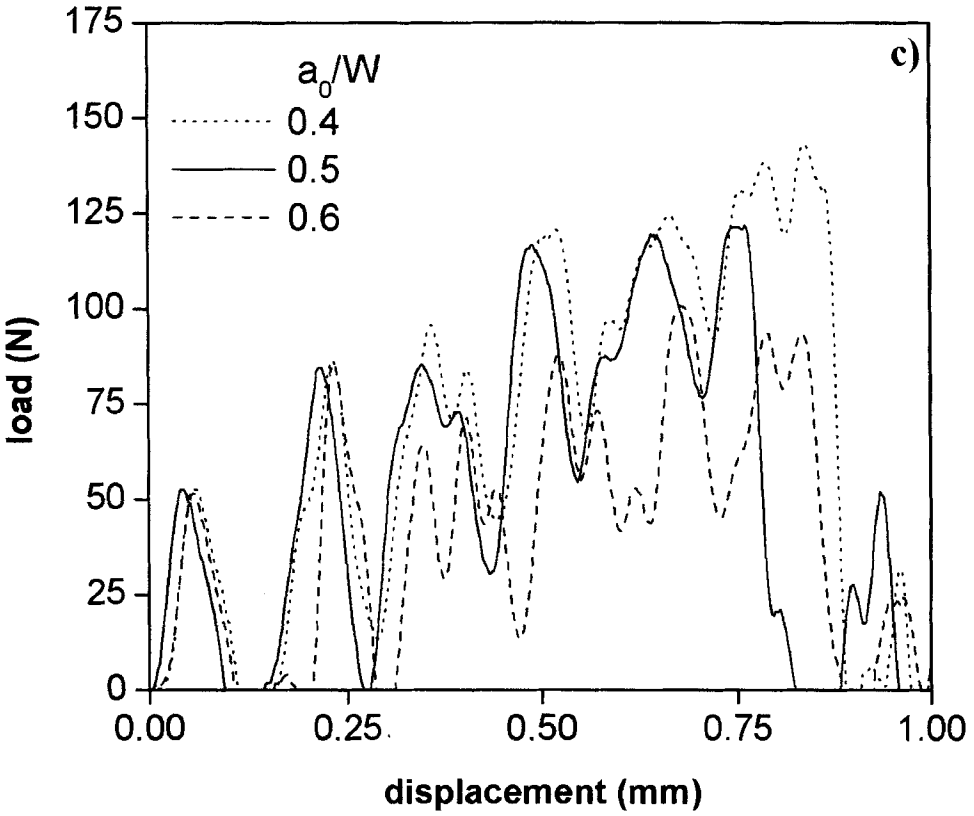


Fig. 4. Load vs. displacement curves. a) PPH; b) PP + 30% POEs; c) HDPE; d) DSGF-HDPE; e) MDPE; f) PE-100; g) RT-PMMA.



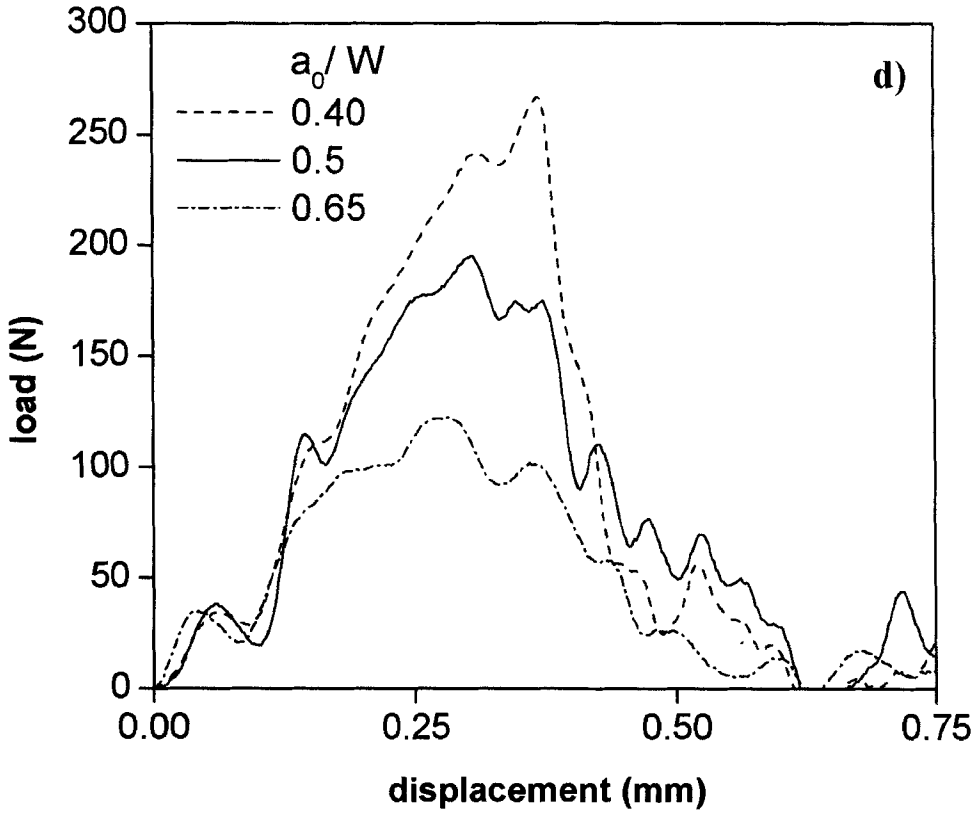
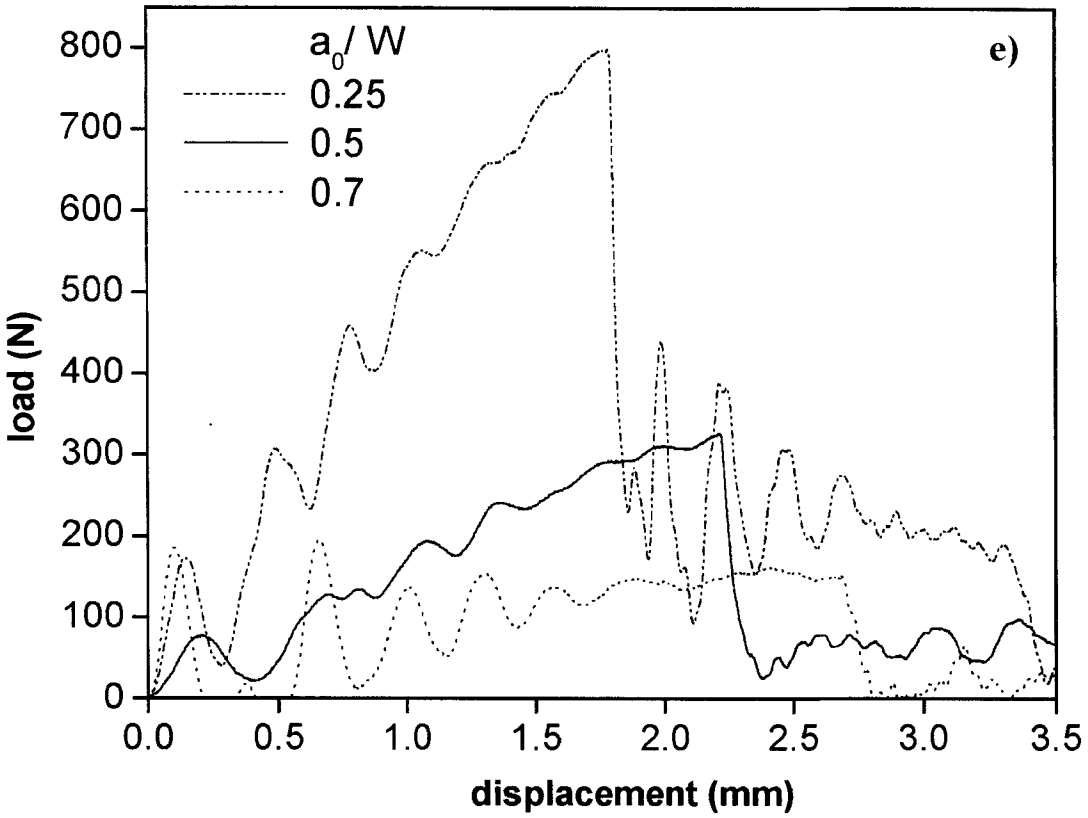
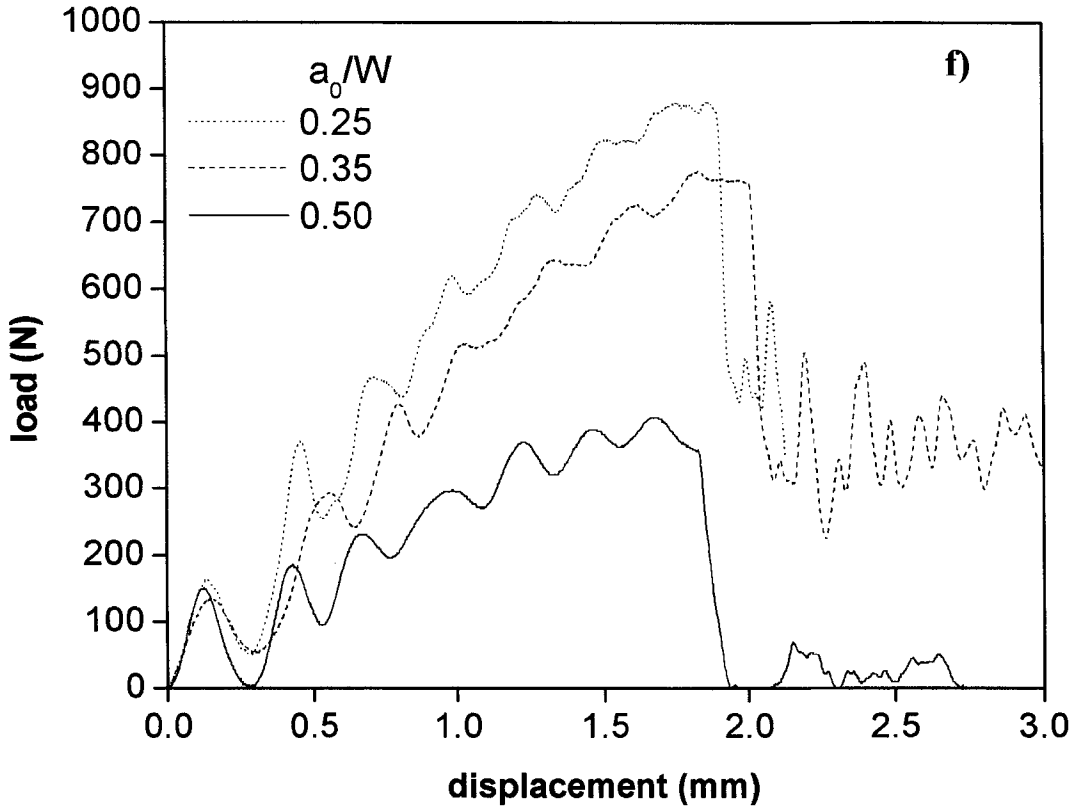


Fig. 4. Continued.





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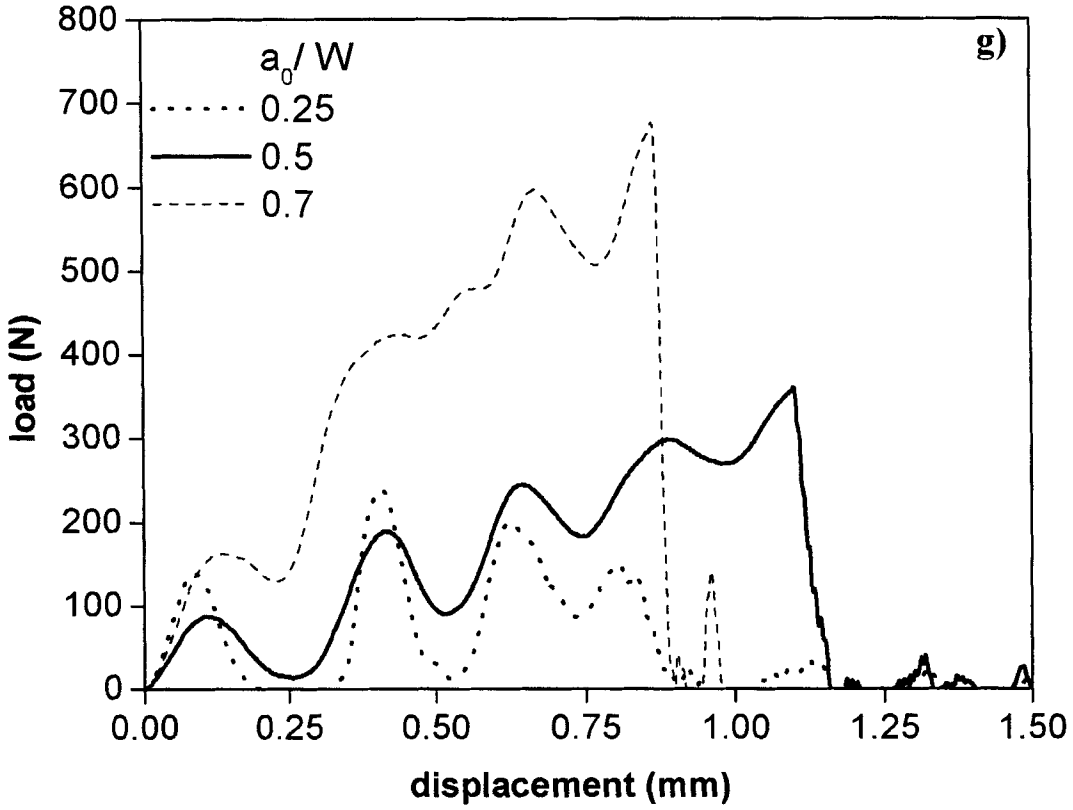


Fig. 4. Continued.

crack tip and concomitant semi-brittle behavior (23–25). This same behavior was exhibited by the PE100, the MDPE and the RT-PMMA. The load increased non-linearly and displayed a drastic drop in coincidence with the sample failure (*Figs. 4b, d, e, f, g*). Post-mortem fracture surface analysis revealed two distinct zones: a rough and whitened zone developed ahead of the crack tip followed by a smooth mirror like zone (*Figs. 3b, d, e, f, g*). The shape of the curves was influenced by crack length. Note that as the notch size (a_0) increases, the load rolls over a maximum and starts to fall before showing the abrupt drop, which corresponds to crack initiation (*Figs. 4a, b, c, d, e, f, g*). Plasticity, sub-critical crack growth, or both phenomena may cause a whitened zone. The whitened zone did not exceed 5% of the ligament length, except for the PE100, where it was 10%.

Fracture Toughness Determination

Fracture Toughness results are shown in *Table 2* and *Fig. 5*. Both methods lead to practically the same fracture parameter and comparable goodness of fit as judged by the statistical analysis.

G_{IC} values in PPH and HDPE were directly determined by applying the standard procedure. For the other materials, which display a certain degree of ductility, the corrected linear elastic method was preferred. When the post-peak ductility effects were significant or

Table 2. G_{IC} and J_c Results, and Standard Deviations.

Material ID	G_{IC} (N/mm)	Standard deviation of the slope	J_c (N/mm)	Standard deviation of the average
PPH	1.03	0.05	1.06	0.06
PP + 30% POEs	2.85	0.53	2.87	0.36
HDPE	3.36	0.10	3.43	0.41
DSGF-HDPE	3.15	0.13	3.13	0.23
MDPE	6.43	0.17	6.57	0.20
PE-100	11.17	0.37	11.18	1.15
RT-PMMA	3.65	0.11	3.46	0.26

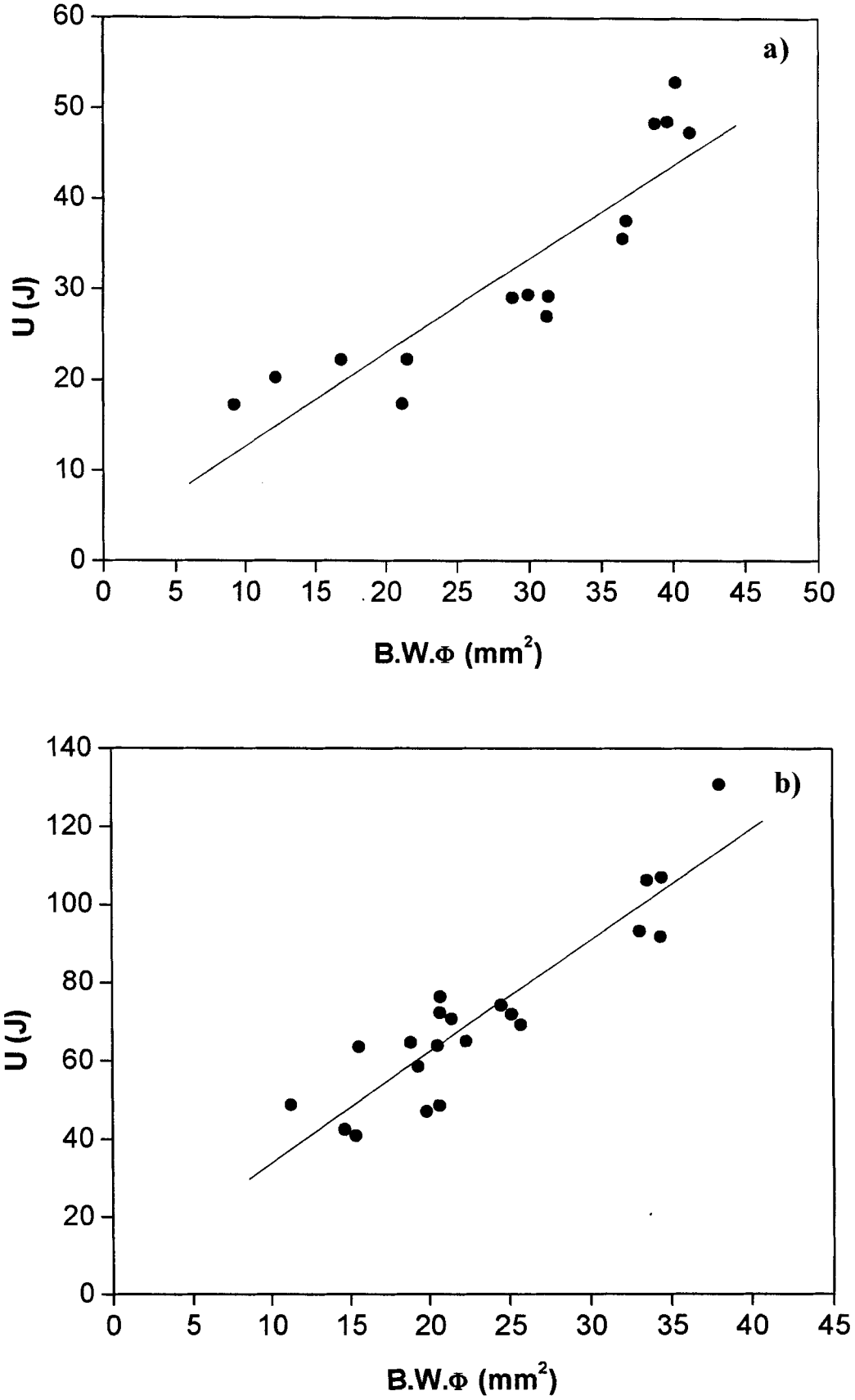
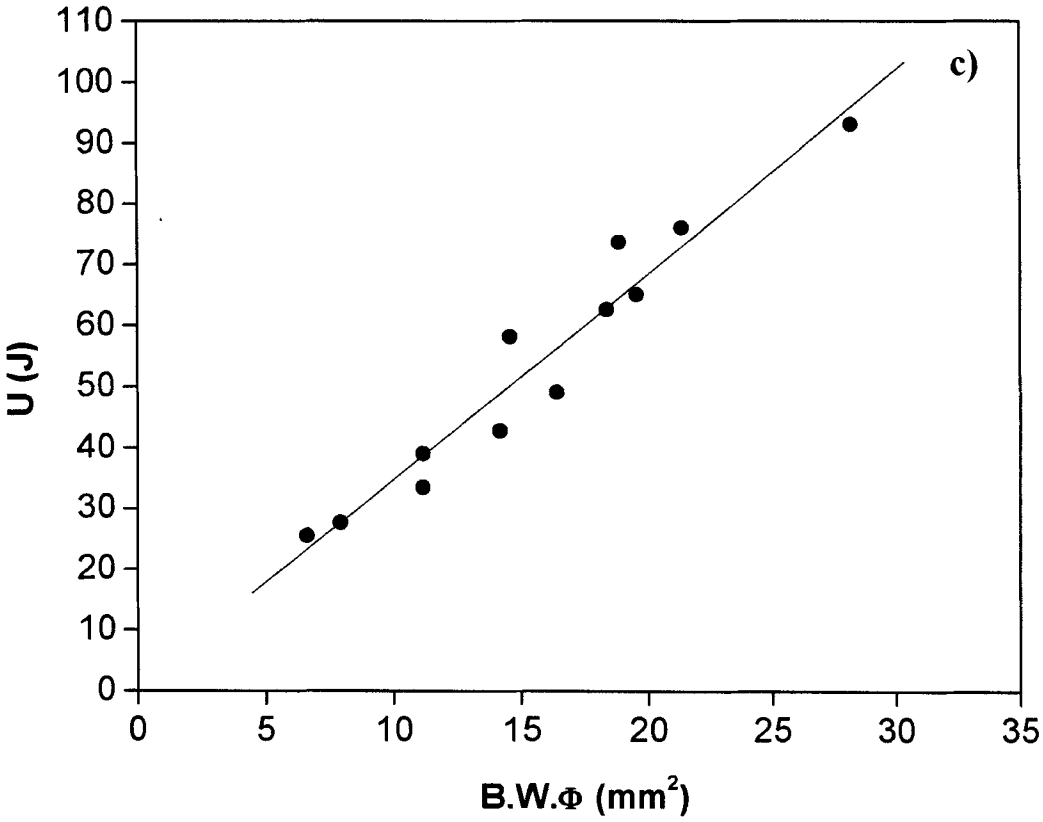
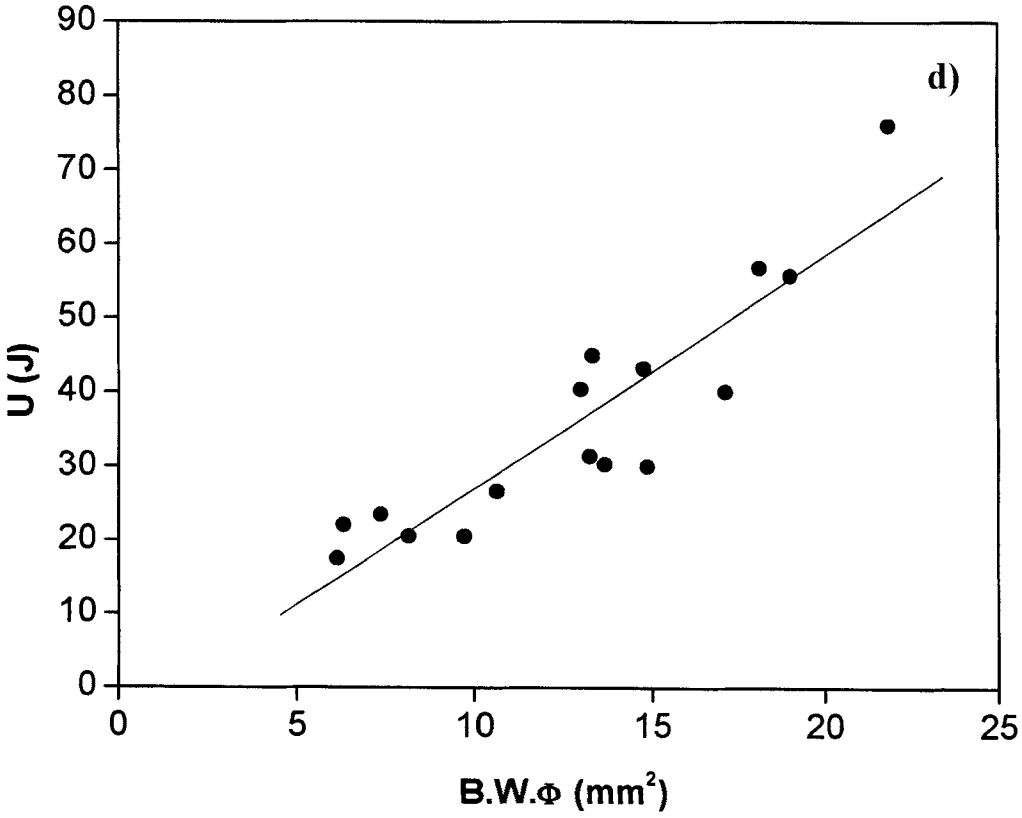


Fig. 5. G_{IC} determination. a) PPH; b) PP+30% POEs; c) HDPE; d) DSGF-HDPE; e) MDPE; f) PE-100; g) RT-PMMA.





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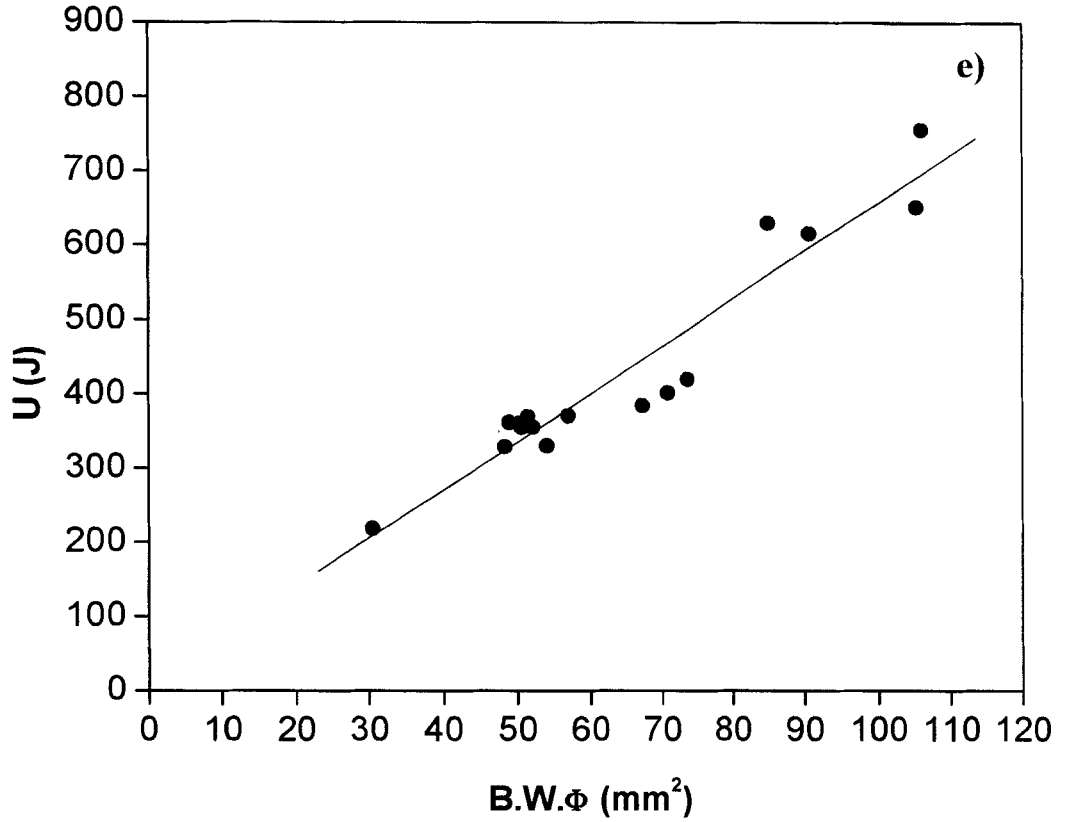

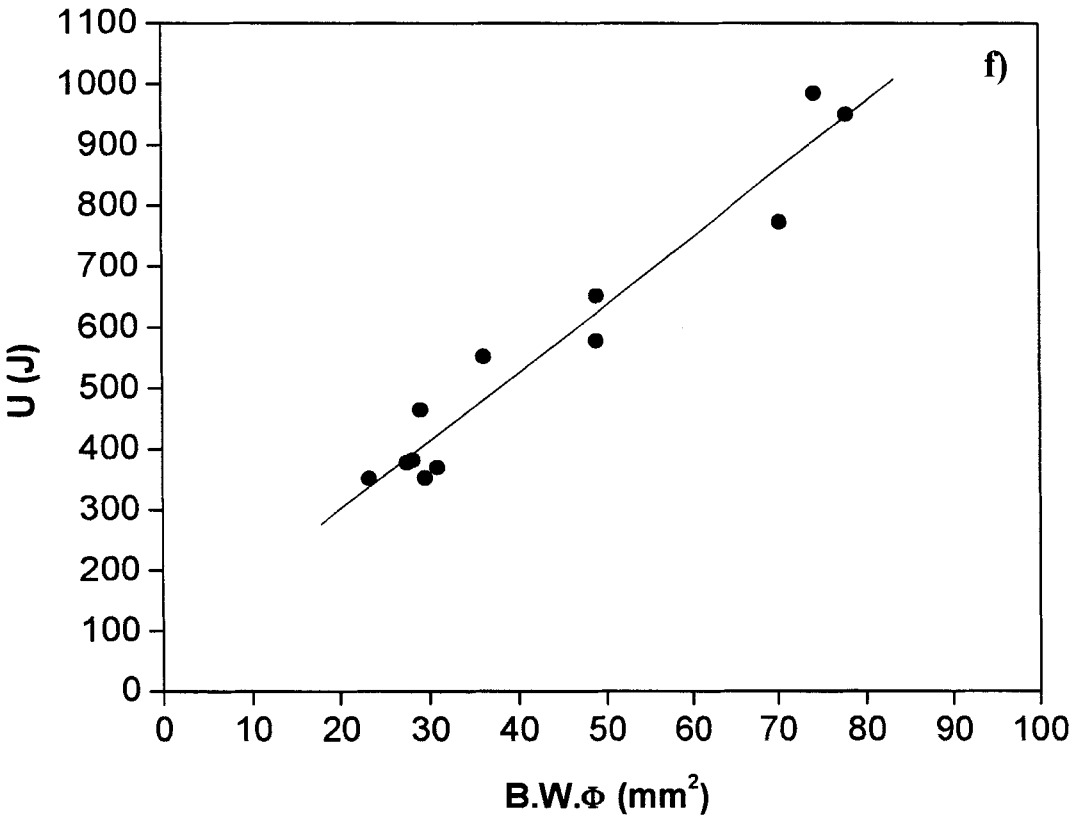

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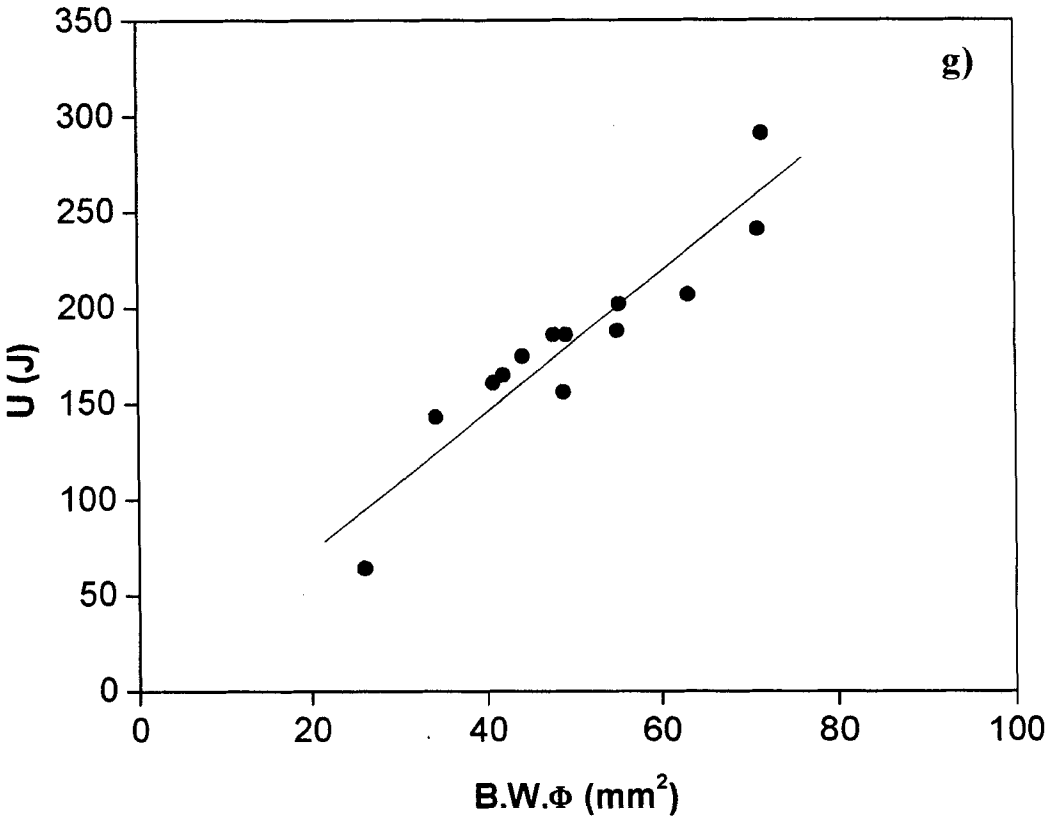

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the fracture behavior showed certain degree of dependency on the crack length (25), the range of a_0/W in which methodology requirements are met was very limited.

CONCLUSIONS

In this work, two methods have been used to assess impact fracture toughness of several polymers displaying either linear or non-linear unstable fracture patterns.

The experimental results presented here demonstrate that Critical Energy Release Rate, G_{IC}, and Cleavage Fracture Toughness, J_c, appeared equivalent. Thus, J_c testing, based only on direct determination of the total energy value (*Eq 1*) of a set of similar samples, may become the more attractive method because of its inherent simplicity.

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ABBREVIATIONS

ϕ = Calibration factor
σ_y = Yield point
A = Crack length
a_{eff} = Effective crack length
A_0 = Initial crack length
B = Specimen thickness
E = Young Modulus
F_{max} = Maximum load in a load-displacement curve
G_{IC} = Critical strain energy release rate
J_c = Cleavage fracture toughness
r_p = Stress whitened zone length
S = Span
U = Fracture energy
W = Specimen width

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