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Interlaboratory tests

Application of the load separation criterion in J-testing of ductile polymers: A round-robin testing exercise



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ABSTRACT

Round robin tests carried out under the direction of the Technical Committee 4 of the European Structural Integrity Society (ESIS TC4) have shown that, for determining the fracture resistance of ductile polymers at low loading rates, the multi-specimen methodology based on the construction of the material crack growth resistance curve often does not provide reliable data due to the uncertainties associated with the measurement of crack advancement (Δa). With the aim of strengthening this multi-specimen methodology, the ESIS TC4 attention has been recently focused on the analysis of a testing scheme based on the load separation criterion, which does not require the measurement of Δa .

The present work gives the results of a multi-laboratory round-robin testing exercise carried out by ESIS TC4 in order to assess the degree of reproducibility of the fracture parameters obtainable with the application of this load separation criterion based testing scheme. Encouraging results have been obtained.

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1. Introduction

The most used approach for determining the low-rate fracture resistance of ductile polymers that cannot be tested using standard linear elastic fracture mechanics (LEFM) tests is based on the construction of the material crack growth resistance (J_R) curve (J parameter vs crack

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http://dx.doi.org/10.1016/j.polymertesting.2015.03.019 0142-9418/© 2015 Elsevier Ltd. All rights reserved. extension, Δa). This is usually obtained by the application of a multi-specimen methodology (procedure [1] developed by the Technical Committee 4 of the European Structural Integrity Society, ESIS TC4, on "Polymers and Polymer Composites" [2] and ASTM D 6068 [3]). ESIS TC4 roundrobin, RR, tests have shown that often this methodology does not provide reliable data due to the uncertainties associated with the measurement of Δa . Further, in many cases, an initiation fracture resistance parameter, J_{Ic}, cannot be properly evaluated. With the aim of strengthening this multi-specimen methodology, the attention of ESIS TC4 has been recently paid to the analysis of a single-specimen testing scheme based on the load separation criterion (LSC) [4], which does not require the measurement of Δa . This would allow determination of: (i.) a material pseudoinitiation fracture resistance parameter (J_{I,lim}), and (ii.) a parameter (m_S) indicative of Δa produced, per unit of plastic displacement, during fracture propagation. Both these parameters can be very useful for the fracture characterization of ductile polymers: J_{I,lim} might be used as replacement of the material fracture resistance parameters computable from the J_R curve; m_S parameter could be used to classify fracture propagation processes by the amount of crack growth produced in the plastic region.

To employ this new approach in a standardized procedure, the degree of reproducibility of the results obtainable with the application of this testing scheme has to be assessed and, with this in mind, a specific multi-laboratory RR testing exercise started in September 2011 under the direction of ESIS TC4. Nine different laboratories participated in this activity and, after preliminary multilaboratory work, a reference draft protocol [5] was prepared by the coordinating laboratory (Università degli Studi di Brescia, Italy). Such a protocol also included the instructions for the RR test program, which was carried out on two materials, an acrylonitrile-butadiene-styrene (ABS) resin and a high-impact polystyrene (HIPS). This RR activity is shown in the present work; specifically, the test procedure is described and the main results obtained by the working group discussed.

2. Experimental

The nine laboratories involved in the ESIS TC4 roundrobin are indicated in the list of authors' affiliations.

The materials tested, kindly provided by Versalis (Mantova, I), are an ABS, in the form of 6 mm thick compression moulded plates, and an HIPS, in the form of injection moulded dumbbell specimens (central narrow portion dimensions: $80 \times 10 \times 4 \text{ mm}^3$). Single edge notched in bending, SE(B), configuration was adopted in the RR protocol, and both sharp-notched (sN) and blunt-notched (bN) specimens were used (see Fig. 1). Table 1 reports the nominal specimen dimensions for the two materials, selected according to the ESIS TC4 methodology for J-testing [1].

A preliminary mechanical characterization was carried out on the materials. All the tests were performed at room temperature and at 1 mm/min crosshead rate. Young's modulus, E, and tensile yield stress, σ_y , of HIPS were measured by uniaxial tensile tests performed on the dumbbell specimens. For ABS, E was measured by tensile tests on bars machined from the plates, whereas σ_y was calculated as 70% of the yield stress measured by compression tests. E and σ_y data are reported in Table 2.

The ESIS TC4 multi-specimen approach [1] was also applied to the two materials. Fig. 2 shows the J_R curves obtained. The same specimen geometry (sN specimen, Table 1) and testing conditions (crosshead rate and temperature) of the RR tests were used. Fig. 2 displays, for each material, the experimental (Δa ; J) data points (full circles),



Fig. 1. Schematic representation of SE(B) sN specimen (a) and bN specimen (key-hole type notch) (b).

Table 1

Nominal dimensions of SE(B) specimens for the two materials tested in the RR activity.

	ABS	HIPS	
Thickness, B [mm]	6	4	
Width, W [mm]	12	10	
Length, L [mm]	53	44	
Span, S [mm]	48	40	
Initial crack length (a ₀) to W ratio,	0.6		
a ₀ /W (for both sN and bN specimens)			
notch tip radius of the sN specimen	as small as possible		
	(see [1])	1	
notch tip radius of the bN specimen [mm]		1	

Table 2

Young's modulus, E, tensile yield stress, σ_{y_i} and fracture resistance parameters (from the ESIS TC4 multi-specimen methodology [1]) of the two materials tested in the RR activity.

Material	E [MPa]	σ _y [MPa]	J _{0.2} [kJ/m ²]	J _{bl} [kJ/m ²]
ABS	2500	44	5.71	1.62
HIPS	1760	18	2.84	0.80

fitted by the power law best fitting curve (solid line); the exclusion lines (vertical lines); the blunting line with equation $J = 2 \cdot \sigma_y \cdot \Delta a$ (broken line); the point on the J_R fitting curve corresponding to $J_{0.2}$ parameter (at $\Delta a = 0.2$ mm, indicated with an asterisk); the point on the J_R fitting curve corresponding to J_{bl} parameter (at the intersection with the blunting line, indicated with "x"). The fracture resistance parameters obtained by the application of the ESIS TC4 multi-specimen methodology [1] ($J_{0.2}$ and J_{bl}) are reported in Table 2.

For the RR activity, six ABS bars (dimensions $53 \times 12 \times 6 \text{ mm}^3$), machined in Brescia from the ABS plates, and six HIPS dumbbell specimens were delivered to the various laboratories, together with the RR instructions. The



Fig. 2. J_{R} curves for ABS (a) and HIPS (b) specimens. For the symbols, refer to the text.

following steps sum up the RR testing procedure applied by each laboratory:

- i. cut bars with length of 44 mm from the central narrow portion of HIPS dumbbell specimens, for the preparation of the SE(B) specimens;
- ii. measurement of specimen dimensions (B and W), for three sN specimens and one bN specimen for each material;
- iii. notching of specimens, and measurement of actual a₀ of bN specimens;
- iv. execution of the low rate fracture tests (recording of load, P, vs displacement, u, curve), with a crosshead rate of 1 mm/min, at a temperature ≈ 23 °C;
- v. breaking-opening of sN specimens and measurement of actual a₀;
- vi. execution of the test for indentation correction (see [1]);
- vii. data processing according to the procedure described at Section 3;
- viii. forwarding of data (specimen dimensions, raw loading curves, resulting fracture parameters) and broken-open specimens to Brescia.

The data were collected and compared at the laboratory of Brescia.

3. The load separation criterion based testing method

3.1. Construction of the load separation parameter curve

The RR draft protocol [5] describes the procedure for the construction of the "load separation parameter curve" (S_{sb} curve) of a specimen of ductile polymer. It is based on the "load separation principle" proposed by Ernst [6], and derived from Sharobeam and Landes' works published in the early 90's [7,8] on metals. The load separation principle assumes that, for a defined specimen geometry, material and constraint, the load, P, recorded in a fracture test in the plastic region is mathematically equal to the product of two independent functions, as Equation 1 shows:

$$\mathbf{P} = \mathbf{G}\left(\frac{\mathbf{b}}{\mathbf{W}}\right) \cdot \mathbf{H}\left(\frac{\mathbf{u}_{\text{pl}}}{\mathbf{W}}\right) \tag{1}$$

where G(b/W) and $H(u_{pl}/W)$ are the geometry and the material deformation functions, respectively, b is the specimen ligament length (b = W-a₀), W the specimen width and u_{pl} is the plastic displacement. The validity of the load separation principle was verified for polymeric materials in both stationary and growing crack experiments [9–13].

For the construction of the S_{sb} curve, only two tests – one on a sN specimen and one on a bN specimen – have to be performed. According to the RR protocol, both specimens have the same dimensions, except for the notch tip radius, which is as small as possible for the sN specimen, according to the usual requirements of fracture mechanics tests, and large enough to hinder crack propagation for the bN specimen.

From the load, P, vs displacement, u, curves measured by testing sN and bN specimens in quasi-static conditions, it is possible to calculate the separation parameter and draw the S_{sb} curve, i.e. S_{sb} vs u_{pl} , by the following equations:

$$u_{pl} = u - P \cdot C_0 \tag{2}$$

$$S_{sb}(u_{pl}) = \frac{P_s}{P_b}\Big|_{u_{pl}}$$
(3)

where C_0 is the initial elastic compliance evaluated for each specimen on the loading curve, P_s and P_b are the values of the load read, at the same u_{pl} , on the P vs u_{pl} curve of the sN specimen and of the bN specimen, respectively. It is worth pointing out that, in the presence of fracture propagation, u_{pl} evaluated according to Equation 2 has a nominal character; the true plastic displacement should be evaluated using the actual compliance, which increases during fracture propagation but is not known. Preliminary investigations showed that the use of u_{pl} data evaluated from C_0 does not impair the application of this procedure to the aims of the RR draft protocol.

Fig. 3a shows a typical separation parameter curve, obtained by testing an ABS with 15% of rubber [SE(B) specimens with B = 3 mm, W = 13 mm, $a_0/W = 0.6$; testing conditions: room temperature, crosshead rate of 1 mm/ min]. In principle, according to the LSC, a separation parameter curve shows three distinct zones [8]:



Fig. 3. Examples of S_{sb} (a) and R_S (b) curve (ABS with 15% rubber). The limit point (grey circle), the plateau level (horizontal segment) and the three regions of the S_{sb} curve are displayed (see section 3.2 for limit point and 3.3 for R_S curve).

- region I: at very small plastic displacements, termed the "unseparable" region and characterized by a highly unstable S_{sb} parameter; here the load separation principle is not valid;
- region II: the "plateau" region, characterized by an almost constant value of S_{sb} (S_{sb,plateau}); it should correspond to the blunting process of the sN specimen;
- region III: the fracture propagation region, characterized by decreasing S_{sb} values; it corresponds to the crack propagation process of the sN specimen.

3.2. Determination of J_{I,lim} parameter

According to the LSC, the point between region II and region III (limit point), at $u_{pl} = u_{pl,lim}$, corresponds to fracture initiation. Whether this point is really representative of fracture initiation is questionable, and some considerations are needed. A previous work by the researchers of the coordinating laboratory [4] showed that, for an ABS resin, the LSC-based technique here described detects fracture initiation with a slight "delay". In fact, the onset of fracture initiation was visually detected by a multi-

specimen approach, i.e. by visual inspection of subsequent smaller and smaller crack extensions produced in several nominally identical specimens, and it was found that upl value at the visually observed initiation point was lower than the uplim value. The limit point was indicative of a small but measurable amount of crack extension (around the 3% of the initial uncracked ligament length). In agreement with Laiarinandrasana et al. [14], Baldi et al. [4] pointed out that, for ductile polymers, fracture initiation can be a complex progressive process, characterized by the slow development of the crack front across the thickness of the pre-cracked specimen, and not a sharp blunting-tofracture transition. Hence, the determination of an initiation point can turn into an arbitrary issue. Therefore, based on these findings, this limit point is considered here only as a point of fracture pseudo-initiation.

For the identification of the limit point on the S_{sb} curve, each laboratory was asked to use a specific procedure, which is based on the determination of the level of $S_{sb,plateau}$. Once the limit point, at $u_{pl} = u_{pl,lim}$ was identified, the corresponding J value, $J_{l,lim}$, is evaluated by the following singlespecimen J-integral form for SE(B) specimens [1]:

$$J_{I,lim} = \frac{2 \cdot U_{lim,c}}{B \cdot (W - a_0)}$$
(4)

where $U_{\text{lim},c}$ is the energy up to the limit point, corrected for indentation.

As underlined in [4], $J_{I,lim}$ might be used as substitution for the parameter $J_{0.2}$ determined from the material J_R curve, and conventionally taken as the material fracture resistance parameter [1]. With respect to this latter parameter, $J_{I,lim}$ is much more closely related to the physics of the fracture process, and typically smaller.

3.3. Determination of m_s parameter

The RR protocol [5] describes the procedure for the determination of a second parameter: m_S . It is evaluated from the normalized separation parameter curve, i.e. $R_S vs u_{pl}$ [4] (see Fig. 3b). R_S is termed "normalized separation parameter" since it is defined as the ratio of the separation parameter over the plateau level of the same curve:

$$R_{S}(u_{pl}) = \frac{S_{sb}(u_{pl})}{S_{sb,plateau}}$$
(5)

The parameter m_S is evaluated as the opposite of the slope of the R_S curve in the region of stable fracture propagation (region III in Fig. 3), where a linear trend is usually observed, according to Equation 6:

$$m_{\rm S} = -\frac{dR_{\rm S}}{du_{\rm pl}}\Big|_{u_{\rm pl} > u_{\rm pl,lim}} \tag{6}$$

Such a parameter provides an indication of the crack advancement produced per unit of plastic displacement (if $m_S = 0$ the process is governed by crack blunting), as analytically shown in the following demonstration.

By taking into account the load separation principle (Equation 1), the load separation parameter (see Equation 3) can be written as follows:

$$\begin{split} S_{sb}(u_{pl}) &= \frac{P_s}{P_b} \Big|_{u_{pl}} = \frac{G_s(b_s/W) \cdot H_s(u_{pl}/W)}{G_b(b_b/W) \cdot H_b(u_{pl}/W)} \Big|_{u_{pl}} \\ &= \frac{G_s(b_s/W)}{G_b(b_b/W)} \Big|_{u_{pl}} = \frac{G_s(b_s/W)}{G_b(b_{b,0}/W)} \end{split}$$
(7)

where subscripts *s* and *b* refer to sN and bN specimen, respectively, and the subscript *0* refers to the initial specimen dimensions. Equation 7 is derived under the hypothesis that the material deformation function in the sN specimen (in which crack is allowed to propagate) and in the bN specimen (in which only blunting occurs) is the same ($H_s = H_b$); that is H depends only on the material deformational properties and on constraint. Such hypothesis was experimentally verified for both metals [8] and plastics [10,11,13]. Moreover, b_b is approximately independent of plastic displacement, since crack advancement should not occur during the experiment, therefore it is assumed equal to the initial ligament length ($b_b = b_{b,0}$), whereas b_s is dependent on the plastic displacement, since the crack can grow in the sN specimen.

In the plateau region, i.e. during the blunting process of the sN specimen, the crack advancement due to blunting in the sN specimen can be disregarded ($b_s \approx b_{s,0}$), therefore the separation parameter in the plateau region, $S_{sb,plateau}$, can be written as:

$$S_{sb,plateau} = \frac{P_s}{P_b}\Big|_{u_{pl} \in plateau} = \frac{G_s(b_{s,0}/W)}{G_b(b_{b,0}/W)}$$
(8)

By introducing Equations 7 and 8 into Equation 5, a form for the normalized separation parameter R_S can be derived, in which the dependency on specimen dimensions is made explicit:

$$\begin{split} R_{S}(u_{pl}) &= \frac{S_{sb}(u_{pl})}{S_{sb,plateau}} = \frac{\frac{G_{s}(b_{s}/W)}{G_{b}(b_{b,0}/W)}}{\frac{G_{s}(b_{s,0}/W)}{G_{b}(b_{b,0}/W)}} = \frac{G_{s}(b_{s}/W)}{G_{s}(b_{s,0}/W)} \\ &= \frac{\left[b_{s}(u_{pl})/W\right]^{\eta_{pl}}}{(b_{s,0}/W)^{\eta_{pl}}} = \left[\frac{W - a(u_{pl})}{W - a_{0}}\right]^{2} \end{split}$$
(9)

In Equation 9, the geometry function has been replaced by the corresponding analytical function (power law) proposed in literature for SE(B) specimens [6], i.e. $G_s(b_s/W) = A \cdot [b_s(u_{pl})/W]^{\eta_{pl}}$, where A is a constant parameter and η_{pl} is the plastic geometry factor, assumed to be 2, as analytically derived [15] and widely adopted in literature.

Before showing how Equation 9 can help to understand the physical meaning of m_S , some remarks need to be made on Equation 9. R_S curves evaluated from S_{sb} curves according to Equation 5 were compared with the trends of R_a data points plotted as a function of u_{pl} , where the values of R_a were evaluated from data of actual crack length (a), measured during fracture propagation tests, according to Equation 10:

$$R_{a}(u_{pl}) = \left[\frac{W-a(u_{pl})}{W-a_{0}}\right]^{2} \tag{10}$$

This comparison was carried out for several materials and specimen dimensions at the laboratory of Brescia: in all these cases results leading to the same conclusion were obtained. In this work, only the results obtained for the two materials used in the RR exercise (ABS and HIPS) are shown. These results were obtained from the elaboration of the data of the fracture tests carried out to evaluate the J_R curves reported in Fig. 2 (for ABS, also the data at Δa levels higher than those used for the J_R curve construction are taken into account). Several nominally identical sN specimens were tested according to the testing conditions of the RR exercise. The tests were stopped at different levels of displacement, in order to produce, at the final points, different levels of crack extension, Δa , which were then optically measured on crvo-fractured surfaces. These different crack growths were treated as consecutive frames of a unique fracture propagation process. From each sN specimen, a single $(u_{nl}; R_a)$ point, corresponding to the final point, was obtained (R_a and u_{pl} were evaluated according to Equations 10 and 2, respectively). The final crack length, $a(u_{pl})$ in Equation 10, was evaluated as $a_0+\Delta a$. Moreover, for each sN specimen, a R_S curve was constructed according to the LSC testing procedure described above (i.e. by using Equation 5), after a bN specimen was tested for this purpose.

Fig. 4a and b show both R_S curves and the series of the $(u_{pl}; R_a)$ data points for the ABS and HIPS specimens, respectively. It clearly emerges that, for a given



Fig. 4. R_S curves (solid lines) and R_a data points (full squares) plotted against u_{pl} for ABS (a) and HIPS (b) specimens. The least square regression straight line fitted to $(u_{pl}; R_a)$ data points is also displayed (broken line), together with fitting line equation and correlation coefficient.

combination of material and specimen geometry, the series of R_a data points and the R_S curves do not overlap, indicating that Equation 9 cannot provide an exact relationship and be used for the indirect evaluation of the actual crack length, a, during fracture propagation. Before discussing the basic reasons for this lack of overlap, it has to be underlined that even the various R_S curves constructed for the different sN specimens of a given material do not overlap perfectly in the region of fracture propagation. The degree of scattering observed for the R_S curves of HIPS specimens appears higher than that of ABS specimens, suggesting that this level of scatter can vary with the material analysed. This scattering might be ascribed to small differences in the fracture propagation process of the ("nominally" identical) sN specimens tested, and it clearly indicates that the description of a fracture propagation process during a fracture test by means of the R_S curve can have a small degree of uncertainty. Interestingly, for a given material, the slope of the R_S curve in the fracture propagation region appears to be only slightly dependent on the sN specimen considered.

The fact that R_a data points and the R_S curves do not overlap can be ascribed to different aspects, related to R_S and/or R_a evaluation:

- crack propagation started in the plateau region (as shown in [4]); in this case in the plateau region b_s ≠ b_{s,0}, differently from that supposed in Equation 8;
- incorrect value of S_{sb,plateau} used for the calculation of R_S;
- $\eta_{pl} \neq 2$ (examples: $\eta_{pl} = 1.9 \div 2.2$ [10]; 2.3 [13]), differently from that supposed in Equation 9 and used in Equation 10 for R_a evaluation;
- uncertainties associated with the measurement of Δa (used for R_a evaluation).

However, from Fig. 4 it clearly emerges that, for a given material, in spite of the differences between R_S and R_a data series, their slopes in the fracture propagation region are very similar. From R_S curves, a value of m_S was calculated for each curve and an average m_S evaluated. From the series of R_a data points, a value of m_a was evaluated as the opposite of the slope of the least square regression straight line fitted to the experimental (u_{pl} ; R_a) data points, displayed in Fig. 4. It turned out that m_S and m_a values are practically equal (by taking into account the standard deviation for m_S): for ABS, $m_S = 0.258 \pm 0.009 \text{ mm}^{-1}$ and $m_a = 0.249 \text{ mm}^{-1}$; for HIPS, $m_S = 0.295 \pm 0.013 \text{ mm}^{-1}$ and $m_a = 0.303 \text{ mm}^{-1}$.

The results indicate that m_s is a valid approximation of m_a ; this can be equivalently formulated as in Equation 11, valid for $u_{pl} > u_{pl,lim}$:

$$m_{S} \cong m_{a} = -\frac{dR_{a}}{du_{pl}} = -\frac{d\left[\frac{W-a(u_{pl})}{W-a_{0}}\right]^{2}}{du_{pl}}$$
(11)

By developing the right-hand member of Equation 11 and neglecting the second order terms, Equation 11 becomes Equation 12:

$$m_{\rm S} \cong \frac{2}{b_0} \frac{da}{du_{\rm pl}} \cong \frac{2}{b_0} \frac{\Delta a}{\Delta u_{\rm pl}}$$
(12)

In Equation 12, the differentials are replaced by differences, which is an acceptable approximation, since the trend of R_S and R_a in the fracture propagation region is fairly linear. Since b_0 is a constant value, Equation 12 shows that m_S is approximately proportional to the crack advancement produced per unit of plastic displacement in the fracture propagation process.

In consideration of this, the m_S parameter, which is a specimen characteristic (i.e. dependent on both specimen geometry and material), could be used to draw a classification of fracture propagation processes as a function of the amount of crack growth in the plastic region. It could guide the choice of the most appropriate methodology for the fracture characterization of a polymeric material. This idea is supported by Fig. 5 that shows R_S curves of different polymeric SE(B) specimens (see Table 3 for the materials and specimen dimensions), constructed at the laboratory of Brescia according to the procedure here examined. From



Fig. 5. R_S curves obtained from the fracture tests on the specimens of Table 3. The specimens compared are different for both material and geometry dimensions (a), or only for geometry dimensions (b).

Table 3

Materials examined to obtain the curves of Fig. 5, with basic mechanical properties (Young's modulus, E, and tensile yield stress, σ_y), and dimensions of SE(B) specimens tested (refer to Table 1 for the acronyms of dimensions).

Material	Mechanical properties		Specimen dimensions		
	E [MPa]	σ _y [MPa]	B [mm]	W [mm]	a ₀ /W
Linear low-density polyethylene, LLDPE	90	6	4	13	0.6
ABS (very tough grade)	1360	31	6	22	0.65
Rubber toughened polyamide 66, RT-PA66	940	31	4	10	0.6
Rubber toughened polymethylmethacrylate, RT-PMMA	2050	51	4	10	0.6
ABS used in this RR, 6x12	_a	a	6	12	0.6
ABS used in this RR, 13x26	_a	a	13	26	0.5
ABS used in this RR, 13x13	_a	_a	13	13	0.5
HIPS used in this RR	_ ^a	_a	4	10	0.6

^a Refer to Table 2.

Fig. 5a, it emerges that m_s can cover a wide range of values (it varies from nearly zero for the LLDPE to 0.83 mm⁻¹ for the RT-PMMA). The LLDPE curve is an extreme: its ms value approaches zero, indicating that crack does not propagate with increasing plastic displacement. This is in agreement with the experimental observation that in such specimens the fracture process is dominated by crack tip blunting. For the evaluation of the fracture resistance of this LLDPE, the application of a testing scheme based on the propagation of an intentionally produced crack (such as the multispecimen approach for I-testing [1]) to SE(B) specimens with the dimensions indicated in Table 3 is likely to fail. It is necessary to use a different testing geometry, or even to resort to another testing scheme, such as cutting [16]. At the other extreme, with a relatively high m_S value, is the RT-PMMA curve; its high ms value indicates that the crack advancement per unit of plastic displacement is quite high, although fracture propagation is still stable. For this RT-PMMA, a valid J_R curve was constructed according to [1] (by using specimens with the dimensions indicated in Table 3), but it is worth noting that its fracture resistance could also be successfully evaluated by the LEFM approach [17]. In Fig. 5b, three R_S curves obtained for the same material (ABS used in this RR) from specimens with different dimensions are reported. It is found that crack advancement produced per unit of plastic displacement (represented by m_S) is dependent on the specimen dimensions (at a fixed thickness, B = 13 mm, a 42% decrease in m_S is observed by doubling the width from 13 to 26 mm). In spite of this, the fact that m_S keeps values considerably higher than zero suggests that crack tip blunting does not dominate the fracture process, and that a method based on the measurement of the crack growth might be successfully applied for the fracture resistance evaluation.

These findings suggest that m_S could be used as a key parameter for the development of a criterion to check *a*-*priori* the applicability of the ESIS TC4 multi-specimen approach for J-testing [1] to a given ductile polymeric material. New activities aimed at examining carefully this idea are being planned.

4. Results and discussion

Fig. 6 shows the R_S curves obtained by the different laboratories involved in the RR exercise for ABS (Fig. 6a)

and HIPS (Fig. 6b) specimens. The curves are vertically shifted for clarity, and the limit point identified by the laboratory (at $u_{pl} = u_{pl,lim}$) is displayed by a grey full circle on each curve. Only one representative curve from the three R_S curves constructed from the tests on the three different sN specimens is shown for each laboratory.

The results of a few laboratories were disregarded, since affected by experimental or computational problems. More specifically, the results provided by Lab. 8 for both ABS and HIPS specimens were disregarded since errors were introduced in data processing due to the incorrect determination of the initial elastic compliance data; moreover, in the bN specimen, fracture propagation occurred during the fracture test. The results provided by Lab. 9 for both ABS and HIPS specimens were disregarded because of the presence of relatively high fluctuations in the loaddisplacement curves (with an amplitude higher than 1 N), which impaired data processing. Although these laboratories did not contribute to the assessment of the reproducibility level of the results, their outcomes were useful to demonstrate the major difficulties in the application of the protocol procedure, which deserve special attention. It is worth noting also that Lab. 2 performed the tests on only two sN specimens of each material, since it had difficulty in producing defect-free sharp notches. Finally, the critical points are: the acquisition of loading curves with sufficiently high data resolution and devoid of load fluctuations, and the notching of both sN and bN specimens (relating to experimental aspects); the determination of the specimen initial elastic compliance from the loading curve (relating to computational aspects).

From Fig. 6, it emerges that, for a given material, the plastic displacement of the limit point $(u_{pl,lim})$, which should indicate the point of fracture initiation, shows appreciable variability, whereas there is a good agreement among the slopes of the R_S curves in the fracture propagation region.

Fig. 7a and b show $J_{I,lim}$ results for ABS and HIPS specimens, respectively, obtained as described at section 3.2 (for each laboratory, the average of the three $J_{I,lim}$ data obtained from the three sN specimens tested is indicated with the corresponding standard deviation bar). With reference to ABS specimens: (i.) $J_{I,lim}$ datum reported for Lab. 2 is the average (without standard deviation) of only two values, evaluated without applying the indentation correction; (ii.)



Fig. 6. R_S curves of ABS (a) and HIPS (b) specimens obtained at the various laboratories. The curves are vertically shifted by an h-factor (h is a multiple of 0.1).

Lab. 3 tested five sN specimens and the J_{I,lim} datum reported is the average of five values; (iii.) Lab. 7 did not provide J_{I,lim} results. With reference to HIPS specimens: (i.) J_{I,lim} datum reported for Lab. 2 is the average (without standard deviation) of only two values; (ii.) the J_{I,lim} datum reported for Lab. 5 is the average (without standard deviation) of only two values; (ii.) Lab. 7 did not provide Jn Lab. 5 is the average (without standard deviation) of only two data since a valid S_{sb} plateau region could not be identified for one sN specimen; (iii.) Lab. 7 did not provide J_{I,lim} results. The comparison among the results from the different laboratories provides the reproducibility level of this parameter. The mean value of J_{I,lim} obtained by averaging all the mean data from the different laboratories is 4.09 \pm 0.630 kJ/m² for ABS and 1.53 \pm 0.251 kJ/m² for HIPS. The standard deviations correspond to a degree of scattering for J_{Llim} results of 15% for ABS and 16% for HIPS.

Such values could be acceptable within the field of fracture mechanics tests; however the procedure described in the draft protocol [5] for the identification of the limit point on the S_{sb} curve can be modified in order to lower the scattering of this parameter. Fig. 7 shows also $J_{0.2}$ and J_{bl} levels, evaluated from the application of the ESIS TC4 multispecimen procedure [1]. $J_{l,lim}$ values are lower than the technological $J_{0.2}$ parameter, as already pointed out, and slightly higher than J_{bl} , which is supposed to be more representative of the actual fracture onset.

Fig. 8 shows m_S results obtained from each laboratory participating in the ESIS RR activity (for each laboratory, the average of the three m_S data obtained from the three sN specimens tested is indicated with the corresponding standard deviation bar) – the additional details reported



Fig. 7. J_{I,lim} results (average and standard deviation within the same laboratory) for ABS (a) and HIPS (b) specimens. J_{0.2} and J_{bl} levels, evaluated from the application of the ESIS TC4 multi-specimen procedure [1], are also displayed.

above for $J_{I,lim}$ data, with reference to ABS and HIPS specimens, are valid also for m_S data. It emerges that not only the degree of repeatability within the same laboratory (represented by the standard deviation bar indicated on each column), but also the degree of reproducibility of such data is much higher than that of $J_{I,lim}$ data. By averaging all the mean data over all the laboratories, mean m_S values of 0.244 \pm 0.0132 mm⁻¹ for ABS and of 0.325 \pm 0.0077 mm⁻¹ for HIPS are obtained, corresponding to a degree of scattering of about 5% and 2% for ABS and HIPS, respectively. The very high reproducibility level obtained for m_S supports the idea to attribute a key role to this parameter in the fracture characterization of ductile polymers.

5. Concluding remarks

In this work, the results of a RR testing exercise carried out under the direction of the Technical Committee 4 of the ESIS are shown. This RR activity examines a singlespecimen testing scheme based on the load separation criterion (LSC), which does not require the measurement of crack advancement, the weak point of the multi-specimen approach for the construction of the J_R curve of ductile



Fig. 8. m_S results (average and standard deviation within the same laboratory) for ABS (a) and HIPS (b) specimens.

polymers [1]. More specifically, the aim of this exercise is the assessment of the degree of reproducibility of the two parameters evaluated by this LSC-based testing method, i.e. $J_{l,lim}$ and m_S .

The $J_{l,lim}$ parameter is put forward as a pseudo-initiation fracture resistance parameter. The degree of reproducibility obtained in the RR tests is acceptable. However, for a further reduction of the level of scattering, new data elaboration schemes for the evaluation of the limit point, that is the point on the loading curve at which $J_{l,lim}$ is determined, are under analysis.

The m_S parameter, dependent on both specimen geometry and material, is indicative of crack advancement produced per unit of plastic displacement in the fracture propagation process. The degree of reproducibility obtained for this parameter in the RR tests is high. This backs the idea to attribute a key role to the m_S parameter in the fracture characterization of ductile polymers. In this work, the possibility of using this parameter for the development of a criterion able to indicate whether the multi-specimen approach for J-testing [1] can be successfully applied to a ductile polymer is proposed. In support of this idea are the following outcomes: (i.) the testing scheme here examined has been applied with success to various polymers, different in both stiffness and yield stress; (ii.) the m_S parameter is effective at distinguishing fracture processes governed by blunting (with $m_S = 0$), which cannot be tested using the multi-specimen approach for J-testing [1], from those where generation of new fracture surfaces actually occurs (with $m_S > 0$).

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