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Determination of the fracture resistance of ductile polymers: the ESIS TC4 recent experience

Francesco Baldi, Università degli Studi di Brescia, Brescia, Italy

Silvia Agnelli, Università degli Studi di Brescia, Brescia, Italy; Luca Andena, Politecnico di Milano, Milano, Italy; Bamber R.K. Blackman, Imperial College London, London, United Kingdom; Leonardo Castellani, Versalis S.p.A., Mantova, Italy; Patricia M. Frontini, Universidad Nacional de Mar del Plata, Mar del Plata, Argentina; Jaroslav Kucera, Polymer Institute Brno, Brno, Czech Republic; Lucien Laiarinandrasana, MINES ParisTech, Evry Cedex, France; Alessandro Pegoretti, Università degli Studi di Trento, Trento, Italy; Alicia Salazar, Universidad Rey Juan Carlos, Madrid, Spain; Laurent Warnet, University of Twente, Enschede, The Netherlands

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Round-robin (RR) tests carried out under the direction of the Technical Committee 4, "Polymers, Polymer Composites and Adhesives", of the European Structural Integrity Society (ESIS TC4) showed that the multi-specimen methodology employed for the construction of the crack growth resistance curve (J vs crack extension, Δa) of polymers often does not provide reliable data, because of the uncertainties associated with the measurement of Δa. With this in mind, the ESIS TC4 attention has been more recently focused on the analysis of a testing scheme based on the load separation criterion, which does not require the measurement of Δa. With the aim to employ this new approach into a standardized procedure, the degree of reproducibility of the results obtainable with the application of this testing scheme to ductile polymers has been assessed by means of multi-laboratory RR testing exercises, started in 2011. An ESIS TC4 reference draft protocol was prepared and ten laboratories participated in the RR activities. The present work describes the load separation criterion-based testing procedure recently examined by ESIS TC4, and gives a summary of the results obtained in the RR activities, which appear encouraging.

(Extended-Abstract – not to exceed 5 pages)

INTRODUCTION

For determining the low-rate fracture resistance of ductile polymers, for which the standard linear elastic fracture mechanics tests fail, the material crack growth resistance (J_R) curve (J *vs* crack extension, Δa) is generally employed. This is usually constructed by means of a multi-specimen approach (procedure [1] developed by ESIS TC4, that is the Technical Committee 4, "Polymers, Polymer Composites and Adhesives", of the European Structural Integrity Society [2], and ASTM D6068 [3]). Specific ESIS TC4 round-robin, RR, tests showed that the uncertainties associated with the measurement of Δa often make this approach unreliable. Further, in many cases, an initiation fracture resistance parameter, J_{Ic}, cannot be obtained. With this in mind, 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), which does not require the measurement of Δa [4]. This approach would allow to: i) determine a material initiation fracture resistance parameter, $J_{I,lim}$; ii) provide a rough measure of Δa produced during the fracture test, in the plastic region. In order to assess the degree of reproducibility of the results obtainable with the application of this method, in view of its possible employment in a standardized procedure, a multi-laboratory activity has started in September 2011 under the direction of ESIS TC4, with Università degli Studi di Brescia (I) as the coordinating laboratory. Ten laboratories (indicated in the authors' list) have participated in this activity, organized on three consecutive RR testing exercises (RR1 to RR3). RR1 consisted in a preliminary work aimed at setting the key-points for the preparation of the reference draft testing protocol [5]. In RR2 and RR3, this protocol was applied to the fracture characterization of polymeric materials with different degrees of ductility, and the outcomes used to enhance the robustness of the method and to improve the protocol itself. The examined materials are: an acrylonitrile-butadiene-styrene (ABS) resin and a high-impact polystyrene (HIPS), in RR2; a rubber-toughened polybutylene terephthalate (RT-PBT) and a linear low-density polyethylene (LLDPE), both exhibiting a very high degree of ductility, in RR3. The present work describes the LSC-based testing procedure examined by ESIS TC4, and gives a summary of the results obtained during the RR activities.

TEST METHOD – EXPERIMENTAL

The method described in the RR protocol, founded on the LSC proposed by Ernst [6], derives from Sharobeam and Landes' works published in the early 90's on metals [7,8]. The applicability of the LSC to polymeric materials, during both blunting and crack propagation phase, has been demonstrated (see [9] and referenced papers). The RR procedure is based on the construction of a "load separation parameter curve", S_{sb} curve, which requires the execution of two tests, on a sharp-notched specimen (sN, crack growth allowed) and on a blunt-notched specimen (bN, crack growth hindered). Single edge notched in bending, SE(B), configuration is adopted. From the load, P, *vs* displacement, u, curves obtained from quasi-static tests on a sN and a bN specimen, the separation parameter, S_{sb}, is determined as:

$$S_{sb}(u_{pl}) = \left.\frac{P_s}{P_b}\right|_{u_{pl}} \tag{1}$$

where P_s and P_b are load values read on P *vs* u_{pl} curves of sN and bN specimen, respectively, at a given value of (nominal) plastic displacement, u_{pl}, which is determined for each specimen as:

$$u_{pl} = u - P \cdot C_0 \quad (2)$$

C_0 being the initial elastic compliance of the specimen. It is expected that S_{sb} curve, that is S_{sb} plotted against u_{pl}, shows a "plateau" region, where S_{sb} maintains an almost constant value ($S_{sb,plateau}$), followed by a decreasing S_{sb} region. The former region corresponds to the crack blunting phase and the latter to the crack propagation phase, in the fracture process of the sN specimen. The point between these two regions (limit point), at $u_{pl} = u_{pl,lim}$, corresponds to fracture initiation (or pseudo-initiation, by considering that for ductile polymers fracture initiation can be a complex progressive process [10]). J-integral value at $u_{pl,lim}$, that is $J_{I,lim}$, which can be taken as a material pseudo-initiation fracture resistance parameter in place of the more conventional $J_{0,2}$ computed by the J_R curve (see [1]), is evaluated as:

$$J_{I,lim} = \frac{2 \cdot U_{lim,c}}{B \cdot (W - a_0)} \quad (3)$$

where $U_{lim,c}$ is the energy up to the limit point, corrected for indentation (see [1]), and B, W and a_0, the thickness, width and initial crack length of the sN specimen, respectively. Further, the normalized separation parameter curve, i.e. R_S *vs* u_{pl}, is traced (R_S is obtained as the ratio of S_{sb} over $S_{sb,plateau}$) and the parameter m_S determined in the region of fracture propagation as (see also Figure 1):

$$m_S = -\left.\frac{dR_S}{du_{pl}}\right|_{u_{pl} > u_{pl,lim}} \quad (4)$$

Parameter m_S, which is a specimen characteristic (i.e. dependent on both specimen geometry and material), provides an indication of the crack advancement produced per unit of u_{pl} and could be used, as a "ductility index", to classify the fracture propagation processes by the amount of crack growth occurring within the plastic region (if $m_S = 0$ the process is governed by crack blunting). For further details concerning this methodology, ref. to [4,5,10].

Materials ABS, HIPS and LLDPE were provided by Versalis SpA (Mantova, I), whereas RT-PBT by Radici Novacips SpA (Villa d'Ogna, Bergamo, I). Table 1 reports the as-supplied form of the materials, their basic mechanical properties, the nominal dimensions of the SE(B) specimens used in the RR fracture tests, and the fracture resistance $J_{0.2}$ data (from the J_R curve constructed by the application of the ESIS TC4 multi-specimen approach [1] on specimens having same geometry and dimensions of the RR tests). Each laboratory prepared and tested at least three sN and one bN specimens for each of the materials considered. The notching techniques were freely chosen by the laboratory. For LLDPE, two sets of specimens (#a and #b), differing in size, were examined. The experiments were performed by means of universal testing machines, at ≈ 23°C and with a crosshead rate of 1 mm/min. The data were processed according to the RR protocol [5], and the results (consisting in S_{sb} curve and data of $J_{I,lim}$ and m_S, for each sN specimen tested) sent to the laboratory of Brescia for the comparative analysis.

RESULTS AND DISCUSSION

Table 2 reports $J_{I,lim}$ and m_S results for the various materials examined. Each datum is the mean value obtained by averaging all the data from the various laboratories. The datum considered for

Table 1 – Supply-form of the materials examined and their basic mechanical properties (Young's modulus, E, and tensile yield stress, σ_y), nominal dimensions of SE(B) specimens, $J_{0.2}$ data.

Material (RR)	As-supplied form	Basic mechanical properties[a]		SE(B) specimen dimensions[b]			$J_{0.2}$ [kJ/m^2]
		E [MPa]	σ_y [MPa]	B [mm]	W [mm]	a_0/W	
HIPS (RR2)	inject. moulded dumb-bells[c]	1760	18	4	10	0.6	2.84
ABS (RR2)	6 mm thick compression moulded plates	2500	44	6	12	0.6	5.71
RT-PBT (RR3)	inject. moulded dumb-bells[c]	1450	31	4	10	0.6	6.58
LLDPE[d] (RR3)	10 mm thick inject. moulded plates	250	-[e]	10	10 (#a) 20 (#b)	0.6	-[e]

[a] from quasi-static tests at room temperature.
[b] B, thickness; W, width; a_0, initial crack length (in bN specimen, notch tip radius of 1 mm); span used in fracture tests, S = 4W.
[c] acc. to ISO 3167; central narrow portion (with dimensions 80x10x4 mm^3) used for SE(B) specimen preparation.
[d] two sets of SE(B) specimens (#a and #b) used.
[e] not measured (J_R curve not constructed).

Table 2 – Mean values (± standard deviation, δ) of $J_{I,lim}$ and m_S obtained by averaging all the (mean) data from the different laboratories. Values between {} indicate δ expressed as percentage of the corresponding mean value.

Material	$J_{I,lim}$ [kJ/m^2]	m_S [mm^{-1}]
HIPS	1.53 ± 0.251 {16%}	0.325 ± 0.0077 {2%}
ABS	4.09 ± 0.630 {15%}	0.244 ± 0.0132 {5%}
RT-PBT	4.6 ± 1.03 {22%}	0.163 ± 0.0045 {3%}
LLDPE #a	-[a]	0.025 ± 0.0096 {38%}
LLDPE #b	-[a]	0.010 ± 0.0052 {52%}

[a] reliable data not obtained.

each laboratory is the average of the data obtained from the various sN specimens tested. The degree of repeatability of the results within the same laboratory (index of repeatability not shown here) was generally higher than that of reproducibility (represented by the data of standard deviation in Table 2). Results with a very high degree of repeatability could be obtained. As an example, Figure 1 shows the R_S curves obtained by the same laboratory (Brescia) from six nominally identical sN specimens of RT-PBT, tested up to different levels of displacement. The curves overlap well, and they practically draw one single curve in which the plateau region that extends up to $u_{pl,lim}$ is clearly visible.

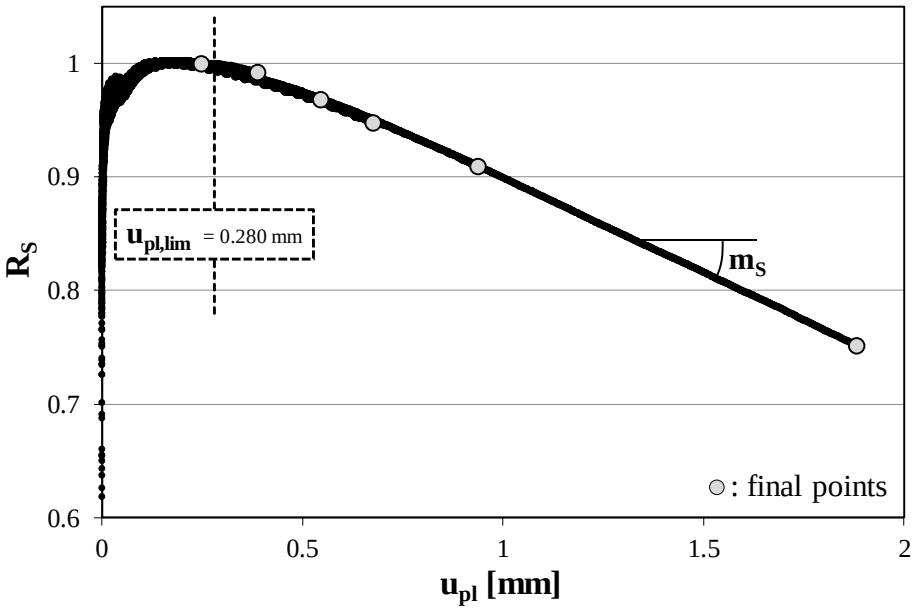

Figure 1 – R_S curves obtained at Brescia laboratory from six nominally identical sN specimens of RT-PBT, tested up to different levels of displacement (final points). $u_{pl,lim}$ is indicated by the vertical dashed line. m_S is also represented.

For HIPS, ABS and RT-PBT, the degree of reproducibility obtained for the m_S data is higher than that of $J_{I,lim}$ data. This suggests that, with respect to the fracture propagation phase, to which m_S parameter refers to, fracture initiation/early-stages of crack growth are less reproducible (at a macroscopic scale). Analysis of the data collected from the various laboratories seem to indicate that the scattering observed for $J_{I,lim}$ data is related to a combination of testing and data analysis aspects. In relation to testing, the quality of the notch, both sharp and blunt, seems to play an important role. With regard to data analysis, a crucial role is played by the determination of the initial specimen compliance, C_0; in addition, the procedure proposed for the identification of the limit point on the S_{sb} curve can be further improved. Within ESIS TC4, new activities aimed at examining carefully these aspects are in progress. It is worth noting, however, that the degree of scattering observed for $J_{I,lim}$ data could be acceptable within the field of fracture mechanics tests. Interestingly, for HIPS, ABS and RT-PBT, $J_{I,lim}$ value is lower than the technological $J_{0.2}$ parameter read on the J_R curve (see Table 1 and 2).

Among the various materials, LLDPE (irrespective of specimen size, either #a or #b) was largely the most hard-to-characterize material. A reliable plateau region could not be identified in the S_{sb} curves, indicating that crack blunting phase cannot be distinguished from crack propagation. The low values of m_S obtained (see Table 2) clearly suggest that fracture process is governed by blunting (irrespective of the specimen size considered, crack growth produced per unit of u_{pl} is very small). LLDPE shows a high degree of scattering for m_S data, and this suggests that its fracture process is not easily reproducible, contrary to what observed for the other materials for which standard deviations of m_S are quite small. This is one of the reason why a reliable J_R curve could not be constructed for this material (see Table 1), for which, furthermore, valid data of Δa could not be obtained by the inspection of the fracture surface produced. Even if valid fracture resistance data ($J_{I,lim}$) have not been determined for LLDPE, the testing procedure here examined was able to

highlight, through m_S determination, that a testing scheme based on the propagation of a crack cannot be successfully applied to this material, working on SE(B) specimens with dimensions as in Table 1. It would be necessary to use a different testing geometry, or to resort to another testing approach, such as cutting [11].

CONCLUSIONS

The results obtained in the ESIS TC4 RR activity on the use of LSC in J-testing of ductile polymers are encouraging. The method examined has been successfully applied to polymers with different degrees of ductility. It allows determining a material pseudo-initiation fracture resistance parameter ($J_{I,lim}$), as well as a parameter (m_S) that can have a key-role in a criterion to check *a priori* if the multi-specimen approach for J-testing [1,3] applied to a ductile polymer (with given specimen geometry and dimensions) is likely to fail. The RR activity is still in progress, and special attention is given to this latter outcome.

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