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Experimental and numerical investigation of the influence of temperature on the fracture behavior of high impact polystyrene evaluated by the J-integral approach using multiple specimen method

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Abstract

The aim of the present work is to study the mechanical behavior in fracture of high impact polystyrene (HIPS) subjected to tensile tests under the effect of temperature to determine the R-curves. The theory of the J-integral contour has been used for the development of a characterization method of the fracture strength appropriate to the case of this non-linear elastoplastic polymer material. To this end, we used the method of multiple specimens (Single edge notch tension SENT) of thin thickness; we used several identical test pieces containing cracks of the same lengths. The tensile tests were conducted under different temperatures, ranging from 25°C to 100°C. The results suggested a very temperature-dependent behavior due to significant increase in crack resistance as demonstrated by a comparison of J_{IC} values related to initiation of crack propagation. The fracture energy absorbed as a function of the temperature suggested that the increase of the temperature is marked by the brittle-ductile transition in the material, which we confirmed numerically via a CASTEM software simulation.

1. Introduction

The mechanical behavior of the usual polymers is characterized by a great apparent diversity. Indeed, under the same conditions of service, and from a technological point of view, one can find polymers that are either rigid, fragile, ductile or rubbery. This diversity is found, for the same polymer, if one varies some of its characteristics, or simply its conditions of use. It can be rigid brittle, ductile or elastic etc. This does not mean that the behavior of a polymer is variable or even uncontrolled. Indeed, it is the parameters controlling its behavior and the considered elementary processes that are numerous.

These behavioral transitions are strongly related to the structure of the polymer and vary significantly from one polymer to another. The purpose of this work is to describe the changes in the fracture behavior of high impact polystyrene (HIPS) due to temperature variation.

Several authors have studied the propagation of cracks on fragile materials such as ceramics [1,2] and certain polymers such as PMMA [3]. In the existing work at [4], tests were conducted on PMMA specimens, to determine the toughness of the material; this toughness was measured for specimens cut into three different geometries that were selected for this purpose: specimens CT (Compact Tension Sample), Single Edge Notched Beams (SENB) and Double Edge Notched Tension (DENT) specimens.

In the literature, mechanical tests were carried out on certain types of amorphous polymers, the results obtained give a mean value of the critical stress intensity factor KIC = 1.7 MPa√m. This value is closer than the one measured by N. Saad-Gouider [5] who found a KIC value of 1 MPa√m, while Loya's work [6] was done on the same type of sample, the latter found that KIC is of the order of 3.2 MPa√m.

Some studies concerning the influence of the temperature of the test on the fracture parameters found in the literature are [7-11]. However, this work lacks unifying conclusions on this subject. Arkhireyeva et al. [7] offer a complete study of the breaking behavior of polyethylene naphthalate (PEN) over a wide temperature range. Three distinct regions were established, related to the values of elastic fracture work. Between 23°C and 80°C it appears constant, between 80°C and 120°C, it increases substantially, and finally decrease above 120°C. This study also shows that elastic fracture work takes a maximum value near the glass transition Tg (Tg = 120°C). The increase in temperature causes that of the value of plastic fracture work, this is attributed to a change of the geometry of the plastic zone at the end of the crack. Unlike Hashemi [10] and Fayolle [11], Arkhireyeva et al. [8] found that the two parameters of the fracture work test are not very sensitive to the temperature of 23°C to 60°C (T < Tg) in the case of PVC films.

For the quantitative evaluation of fracture behavior of fragile materials, i.e. materials with elastic or near-elastic behavior and essentially unstable crack propagation, the concepts of linear elastic fracture mechanics (LEFM) [12] have proven effective in both experimental and numerical studies [13-14]. However, if the size of

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the plastic zone is smaller compared to the initial crack length and the geometry of the specimen, the elastoplastic fracture mechanics (EPFM) concepts must be applied. In all these studies, the relationship between the J-integral parameter and incremental changes leading to transitional behaviors in polystyrene based materials due to temperature haven't been substantiated. Therefore, the parameter used in this work to describe the field of stress and strain close to the crack tip is the integral of J determined by the energy method using the multiple specimen method (MSM). The validity of the J-integral concept is guaranteed if large areas of flow are formed in the region of the crack tip, regardless of the behavior of the material. Therefore, this concept is well suited to the evaluation of the fracture mechanics of high impact polystyrene subjected to temperature variation.

2. Experimental

2.1 J-integral of rice

Using the results of the work of Eshelby [15], Cherepanov [16] and Rice [17] defined a contour integral called J-integral, characterizing the rate of energy release of a structure containing a crack, during its growth. In the particular case of a plane problem, illustrated by Figure l, the J-integral is defined for an oriented contour Γ, by the following relationship:

$$J = \int_{\Gamma} \left[w.dx_2 - T_i \frac{du_i}{dx_i}.ds \right] \quad (1)$$

- Γ is a contour passing through the material and surrounding the crack tip starting from the lower surface and ending on the upper surface.
- x_1 and x_2 are the coordinates of the point M of the contour from the crack tip.
- ds is the curvilinear element in M of the contour.

$$w = \int_0^{\varepsilon} \sigma_{ij}.d\varepsilon_{ij}$$

- w is the strain energy density, defined by:
- $\vec{T}$ is the vector force acting on ds in M.
- $\vec{n}$ is the unit vector normal to contour Γ, outwardly directed in M.
- $\vec{u}$ is the displacement vector of the point of application of $\vec{T}$ in M.

Rice has further shown that this integral in the case of a material with elastic behavior (linear or not) was independent of the integration contour. In the case of elastoplastic behavior, this property remains true in the case of radial loading.

On the other hand, any unloading (or any sudden variation in the direction of loading) makes it lose this property, thus indicating, for this type of behavior, that the description of the propagation of the crack by the J-integral cannot be envisaged [18]. In fact, the nonlinear elasticity supposes that the unloading is done following the same curve as at the rise, which is not the case of a real elastoplastic material.

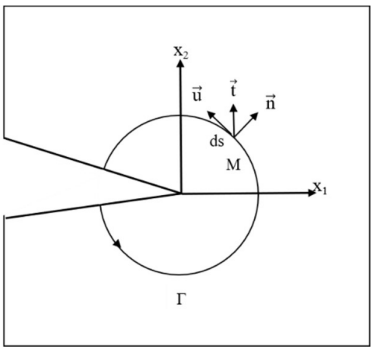

Figure 1. Definition of the J -integral contour.

Moreover, in the case of a linear elastic behavior, the integral J represents the rate of restitution of energy G:

$$J = G = -\frac{dP}{da} = \frac{K_I^2}{E} \quad (2)$$

Where $\overline{E} = E$ in plane stresses.

$\overline{E} = \frac{E}{(1-\nu^2)}$ in plane deformations.

With E: the Young's modulus, ν the Poisson's ratio, K_I the stress intensity factor,
P: the potential energy and the variation of the crack area.

The integral allows to define the fields of the stresses and deformations according to the Hutchinson formulations [19-20], Rice and Rosengen [21] in polar coordinates at the bottom of crack. These authors have shown, for materials obeying a law of flow of the form $\frac{\sigma}{\sigma_y} = \left(\frac{\varepsilon}{\varepsilon_y}\right)^n$ that there exists ,in the vicinity of the crack, a field of constraints and deformations having the following expression:

$$\frac{\sigma_{ij}}{\sigma_y} = \left(\frac{J}{\varepsilon_y.\sigma_y.I_n.r} \right)^{\frac{n}{n+1}} .f_{ij}(\theta) \quad \frac{\varepsilon_{ij}}{\varepsilon_y} = \left(\frac{J}{\varepsilon_y.\sigma_y.I_n.r} \right)^{\frac{n}{n+1}} .g_{ij}(\theta) \quad (3)$$

Where n is the coefficient of strain hardening, σ_y the yield strength, ε_y the strain related to σ_y, I_n a stress of integral depending on n, $f_{ij}(\theta)$ and $g_{ij}(\theta)$ dimensionless functions of θ and n.

The intensity of the stresses and deformations in the elastoplastic singularity is therefore characterized by the J-integral, which thus plays the same role as K in elastic linear mechanics.

Rice then established that the integral J could be related to the dissipated energy rate (Figure 2) by the following relationship [22,23]:

$$J = -\frac{1}{B}\left(\frac{\partial U}{\partial a}\right) \quad \text{at constant displacement.}$$

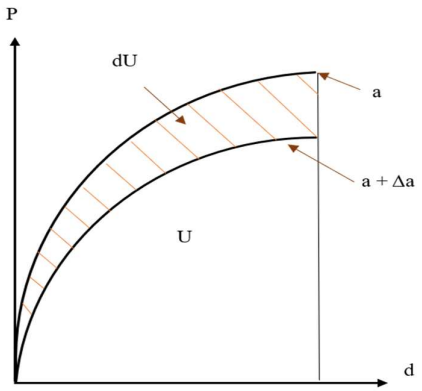

Figure 2. Variation of dissipated energy with constant displacement.

This energetic interpretation of J is the starting point for all the experimental methods for determining the parameter J.

In the case of a ductile material, the crack growth, under the action of an increasing load is characterized macroscopically, by the following steps (Figure 3).

- Blunting of the front of the crack initially acute. The radius of curvature in the crack tip obtained by pre-cracking of the specimen is very small and will rapidly increase during loading, by plastic deformation, before propagation;
- Stable growth resulting from tearing at the bottom of the crack;
- Brutal instability.

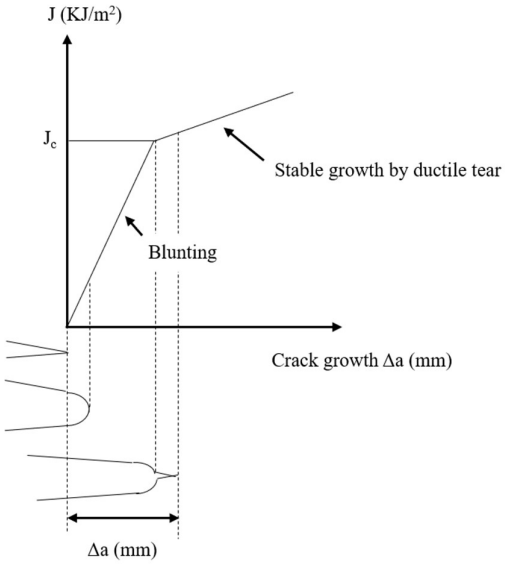

Figure 3. Schematization of the process of ductile tearing.

Such behavior is generally characterized by an experimental curve, expressing the variation of the parameter J as a function of the advance of the crack Δa.

Experimentally, it is not possible to measure the integral J, but rather the energy parameter J that can be identified with the integral under certain conditions. There are several techniques for determining this parameter J, one of them is the multi-specimen technique.

2.2 Materials and methods

2.2.1 Experimental measurement of J

It is through a series of articles [24-26] that Landes and Begley contributed to the development of a first experimental method for measuring the critical value J_{IC}. Their technique requires the use of several test pieces. These test pieces must also have an initial crack of the same lengths. The preparation of the specimens must therefore be more difficult because of the need to cut cracks of the same lengths in each of the specimens. This first method does not allow to draw a J-R resistance curve.

Rice, Paris and Merkle [27] later develop an improved method for experimentally measuring J_{IC}. This new method allows to connect by an equation J and the area under the tensile curve, which is the energy. For a 3-point bend test, they found equation 4.

$$J=\frac{2}{Bb_0}\int_0^{\Delta l} Pd\Delta l \qquad (4)$$

Where: B = thickness of the specimen.
b_o = initial length of the uncracked ligament.
P = load applied to the specimen.
Δl = lengthening of the test piece.

Merkle and Corten [28] showed that a correction to Equation 4 is necessary in order to accommodate compact tensile specimens (CT) as well as other types of specimens that are not subjected to pure bending. The correction to be used depends on the length of the un-cracked ligament b_o, the applied load P and the displacement Δl. The effect of this correction was to replace the 2 in equation 4 with a parameter η which depends on the geometry of the specimen. Normally, the value of η is between 1,0 and 2,4. In ASTM standardized tests, J is calculated using Equation 5.

$$J=\frac{\eta U}{Bb_0} \qquad (5)$$

Where $U = \int_0^{\Delta l} Pd\Delta l$

Where η is the Merkle-Corten parameter [28] and b_o is the initial length of the uncracked ligament The value of the Merkle-Corten parameter is known for ASTM-standardized specimens, otherwise, The Merkle-Corten parameters can be deduced if the integral J, the stored energy and the geometry of the specimen are known. According to ASTM E813, for SENT specimens η = 2.

2.2.2 Criteria of crack initiation

As opposed to brittle fractures, characterized by a sudden instability of the crack, fractures in elasto-plasticity are often preceded by a phase of stable propagation of the defect (ductile propagation). When the plasticization is noticeable, it is no longer possible to measure KIC validly. The toughness of the material must be measured by the J_{IC} characteristic. The critical value J_{IC} is proposed as a criterion of initiation and a curve J-Δa is proposed for the description of the propagation. At the beginning of loading, the crack does not spread yet but dulls in crack tip. The blunting is connected to J by a linear relationship of the type:

$$J = \alpha \Delta a \quad (6)$$

Where α is a constant depending on the characteristics of the material. Lautridou [29] proposes a value equal to $4\sigma_y$ (σ_y is the elastic limit) for this coefficient α.

In general, the moment when the crack starts is not easy to determine experimentally. A value Δa = 0.2 mm is proposed by the ASTM [30] standard as the detection limit of crack propagation initiation. The starting toughness at this time noted $J_{0.2}$ is defined by the intersection of the J-Δa curve with the translational blunting line of 0.2 mm, as shown in Figure 4.

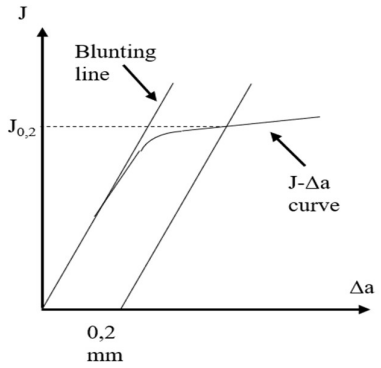

Figure 4. Determination of $J_{0.2}$.

2.2.3 Multi-specimen technique

The energetic interpretation of the integral led Begley and Landes [31] to propose the first experimental method of evaluating the parameter J. It requires the use of several identical test pieces containing cracks of the same lengths.

The determination of J then takes place in three stages:

- Extend the specimen (by moving the deck of the tensile testing machine) by a Δl displacement that should produce a crack elongation Δa. A good way to do this is to stretch the test piece until there is a significant drop in the recorded load.
- Unload the specimen and measure the length of the crack. In the case of high impact polystyrene, we let the specimen cool in ambient air.
- Measure the length of the crack using a measuring device taking care not to tighten the specimen, as it deforms very easily, especially after being heated.

2.2.4 Characterization test

The fracture tests required to plot the J-R curves were made from standard test pieces. The type of test specimen is; single edge notched tensile (SENT) standard specimen as shown in Figure 5.

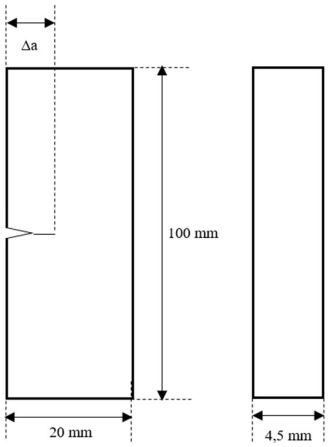

Figure 5. Cracked tension test piece on the side (SENT).

All SENT test specimens are obtained from thin sheets of commercial high impact polystyrene.

Figure 6. High impact polystyrene sheets.

At first we cut from the tubes, samples using a cutter (Figure 7) equipped with a blade thickness of 0.9 mm, this operation was carried out in cooperation with LPEE team from CASABLANCA. The test specimens shall be of dimensions in accordance with the ASTM E1820 standard (length L = 100 mm and width W = 20 mm).

The specimens obtained by cutting have rough edges, to have test specimens of high quality and dimensions in accordance with ASTM E1820, it is necessary to perform a grinding operation using glass-paper discs.

The realization of acute cuts is necessary for the measurement of tenacity of the chosen material. At a given thickness, the more acute the notch, the more flat-deformation conditions prevail at the crack tip over

those of plane stress. This sharpness is achieved using a razor blade.

In the case of amorphous polymer materials, in particular high impact polystyrene, for which a significant plastic deformation is observed before fracture, it is necessary to use a geometry in which the stress gradient is greater. To do this we adopt a SENT configuration (Simple Edge Notched Tension).

To check the machining condition and the dimensions of the same notches lengths on the flat test pieces we used KSM off-line measuring device that measure thinly cut cross sections (slices) of specimens.

KSM is a cable-measuring unit to "off-line" measure cable slices, tubes and profiles. It replaces manual profile projectors and measuring microscopes and gives far better repeat accuracy (0.2 %). KSM measure is very fast (1-3 s) and using imaging technology so that the measuring time only takes seconds.

After all these steps, we obtained SENT (Simple Edge Notched Tension) specimens of the same lengths of notches for the fracture tests. Once the verification operation is complete, all SENT test pieces have been indexed and numbered, taking care to note the notch length of each test specimen.

2.2.5 Acquisition of experimental data

Some methods of characterization of the breaking strength found in the literature can be used to draw resistance curves. Unlike single-break parameter methods, the resistance curves do not only predict the conditions under which a crack will propagate. They also predict the advance of the crack as the total energy stored at the tip of the crack is released by the creation of new surfaces. The resistance curves are often called "J-R curve" where J represents the value of the contour integral J and R represents the breaking strength. Figure 7 illustrates a typical J-R resistance curve.

Since J is a measure of the energy available at the creation of new surfaces, it can be understood from Figure 9 that this energy must be constantly increased to further lengthen the crack. Figure 9 also shows a unique break parameter named JIC, which corresponds to the point at which stable crack propagation begins. A detailed procedure for measuring this failure parameter is given in the ASTM E813 Standard Test Documentation [32-34]. Although this parameter can tell us the value of J for which the crack will begin to propagate, it does not give us any information as to the future progression of the crack. It is for this reason that our characterization methodology is based on the use of J-R resistance curves.

To obtain a J-R resistance curve, it is necessary to carry out experiments with cracked test pieces. Some methods found in the literature suggest standardized specimens. For these specimens, there are formulas for calculating J from experimental measurements. These measurements are the dissipated energy U per test piece during the test and the measurement of the elongation of the crack obtained. Figure 8 shows the dissipated energy U by a cracked specimen during a tensile test where it is subjected to a load P causing elongation Δl (from point 1 to point 2). When the load is removed, the tensile curve moves to point 3 rather than point 1 because of crack elongation and plastic damage in the material. The area under the tensile curve is the energy dissipated by the cracked specimen.

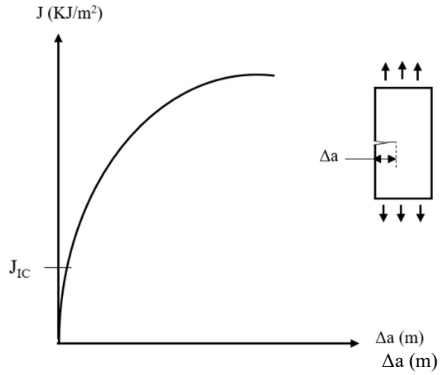

Figure 7. Typical J-R resistance curve.

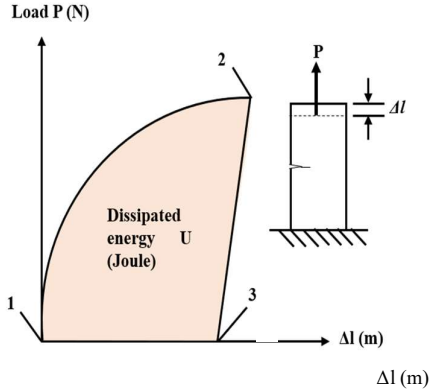

Figure 8. Tensile curve of a cracked specimen.

The mechanical uniaxial tensile tests were carried out at the Public Laboratory of Experiments and Studies (LPEE) of Casablanca. The laboratory has a ZwickRoell tensile machine with a 2.5 kN load cell, which made possible to obtain more precision in our tests, given the nature of the test material and the geometry of the specimens which have a small thickness.

- Thermal enclosure

The tests can also be performed under different temperature conditions. To do this, the necessary thermal chamber is also controlled by TestXpert II. The enclosure has a temperature range of -80 to +250°C.

We subjected each cracked specimen to a different Δl elongation using a tensile test machine. After discharging and cooling the specimen to ambient air, the elongation of the crack caused was measured using the KSM measuring device.

The multi-specimen technique consists of slitting the specimens at the same elongations a0 and measuring the length of the growth of the crack Δa. ASTM D6068-96 suggests preparing a minimum of 5 test pieces. We have prepared 10 for each series of tests, it is important to take care

to make them so that they all have the same dimensions. Initial cracks should also be the same length for each specimen. Each test follows a step procedure.

Each test piece is thus subjected to a tensile test to obtain the curve P-Δl and calculate the parameter J for each Δa according to the Equation (5).

The curve J-Δa is then established from the results of the previous equation to deduce J_{IC} (Figure 9).

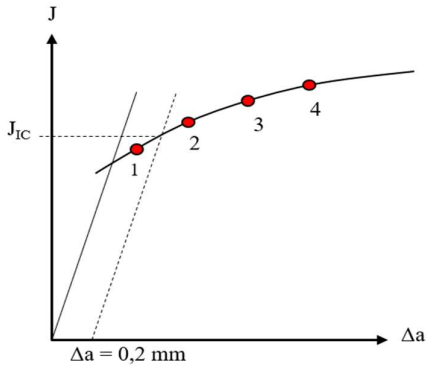

Figure 9. Procedure to create a crack resistance curve using the multi-sample method. Measurement of charge-displacement recordings of different specimens and calculated J-R curve for different crack growths according to [35].

4. Results and discussion

The following curves illustrate the effect of temperature increase on the tensile strength of HIPS specimens according to the multiple specimen method according to ASTM E813.

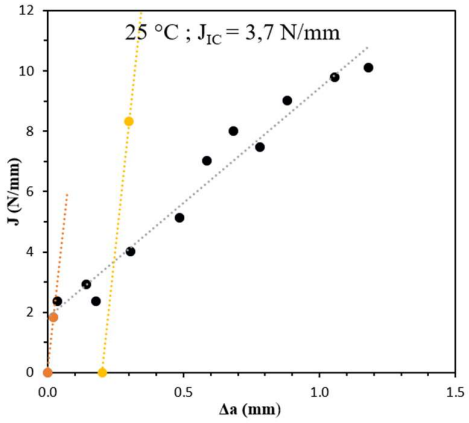

Figure 10. The J-Δa curve of the HIPS at 25°C according to the MSM.

From the curves, it is noted that the crack propagation depends on the temperature at higher temperatures, because the slippage of the molecules takes time. At room temperature, in the brittle state, chain scission of

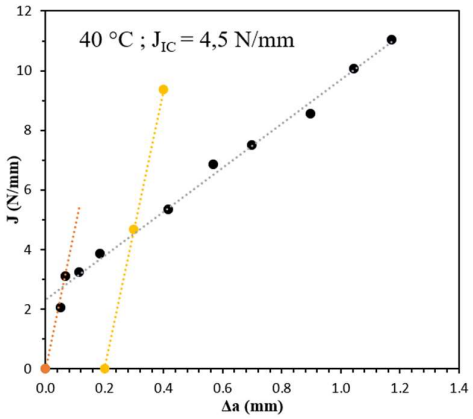

Figure 11. The J-Δa curve of the HIPS at 40°C according to the MSM.

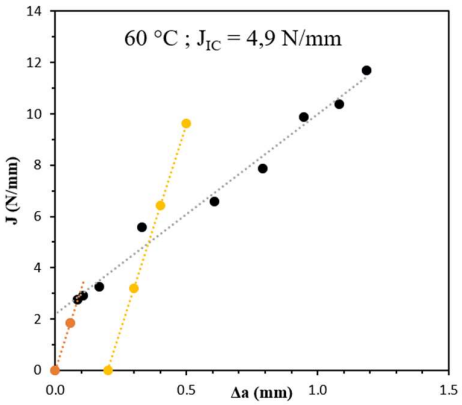

Figure 12. The J-Δa HIPS curve at 60°C according to the MSM.

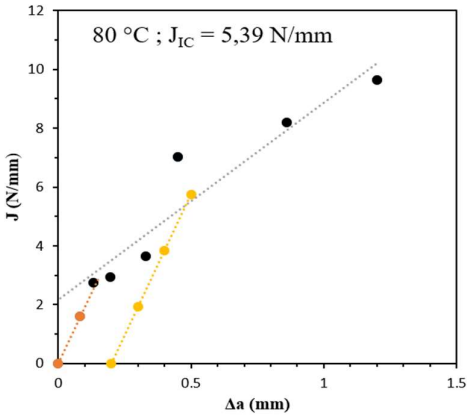

Figure 13. The J-Δa HIPS curve at 80°C according to the MSM.

the polymer predominates, which depends only on the activation energy of the bond in the main chain. Consistent with this logic, the critical energy release rate JIC of HIPS increases with molecular weight at

higher temperatures since the molecular chains tend to become entangled thus requiring more energy to break up.

We can say that the increase of the temperature is marked by the brittle-ductile transition in the material, in fact, the fracture energy absorbed as a function of the temperature is illustrated in the Figure 17 and one clearly finds that a weak absorption of energy accompanied by brittle fracture would occur at temperatures below the glass transition temperature. On the other hand, high energy absorption with ductile fractures occurs at high temperatures and a ductile-brittle transition will occur at an intermediate temperature, which means crack resistance related to these transitions, although the material is generally weakened.

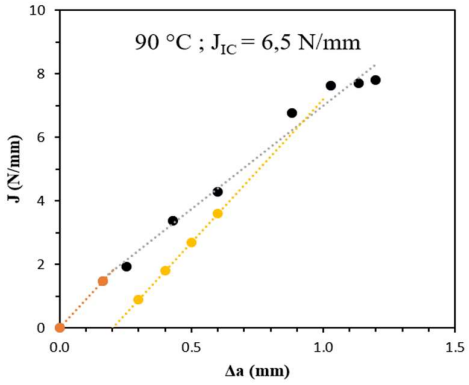

Figure 14. The J-Δa curve of the HIPS at 90°C according to the MSM.

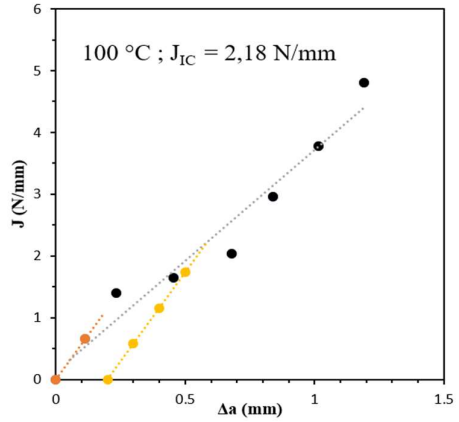

Figure 15. The J-Δa HIPS curve at 100°C according to the MSM.

On the other hand, beyond the glass transition temperature Tg (in this case Tg is around 96°C [36]), there is a remarkable decrease in the JIC, this is explained by the sufficient thermal activation at this stage to overcome the intermolecular bonds, and increase the mobility of the molecules at the same time in the material. At this stage the molecules become more and more mobile and can rearrange or slide easily on loading, it is perceived that when the transition temperature is exceeded, the material reaches the viscous state, the increase in temperature has little effect on intermolecular links completely degraded, so little cracking energy is needed to create rips within the material.

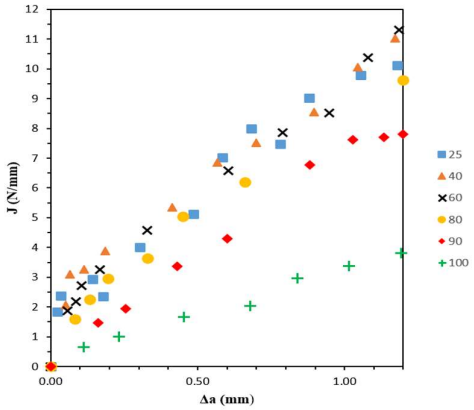

Figure 16. Th J-Δa HIPS curve at all temperatures according to the MSM.

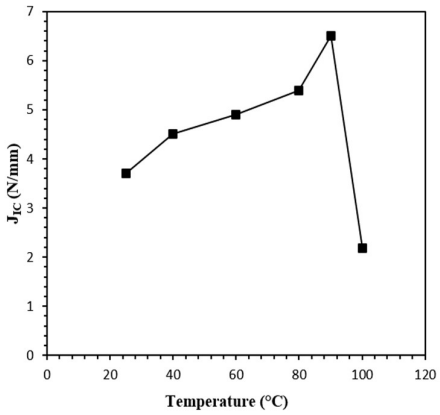

Figure 17. The evolution of JIC as a function of temperature T.

In addition, we note from T = 90°C, the absence of the initially acute cracking of the front of the crack as shown in Figure 3 indicating what clearly illustrates a transition of an unstable behavior of the progress of the crack towards a behavior dominated by the stable advancement of the crack thus a resistance to the cracking, which confirms the increase of the parameter J_{IC}.

4. Comparison with numerical analysis using Finite Element Method.

The CAST3M 2017 software was used for modeling and simulation. It incorporates G-Theta method for calculating the J integral along the crack front.

The Figures 18 and 19 show the specimen deformation under maximum loading in mechanical and thermo-mechanical calculation. We clearly see the effect of thermal dilatation in the deformation.

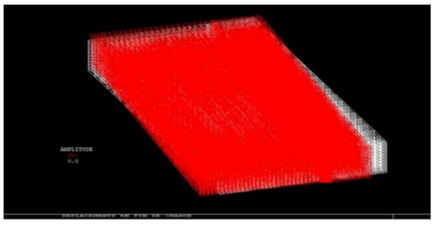

Figure 18. Mechanical calculation of the deformation at maximum load.

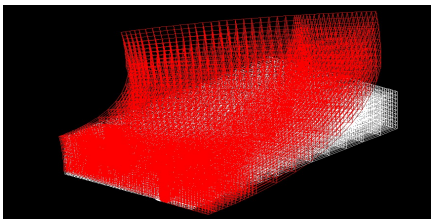

Figure 19. Thermomechanical calculation of the deformation at maximum load.

The distribution of the Von-set stress constraints (σ) along the axis of the test specimen modeled SENT in 3D is shown in Figure 20.

For non-linear calculations, they are performed in CASTEM with the operator "PASAPAS" (Step by step). In mechanics, the latter allows to perform incremental linear calculations. Nonlinearity can come from material (plasticity), large displacements or both at the same time. The results are calculated at values of the evolution parameter (pseudo time or real time) defined by the user. When thermally, this operator makes it possible to carry out nonlinear calculations taking into account conduction, convection and radiation. It is also possible to perform thermo-mechanical coupled calculations.

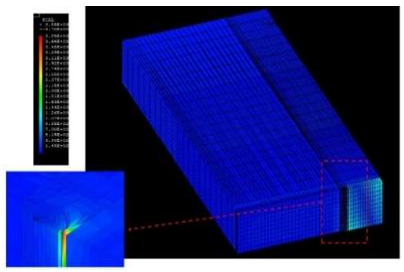

Figure 20. Von Mises equivalent stress.

We also calculated the values of the integral of contour J for each advancement of the crack and for each change of the temperature; the results are compared with the experimental ones for two temperatures (20°C and 60°C) in Figure 21.

According to numerical results, even though very close to the experimental trend-line studies, the crack required less energy to grow, furthermore, the blunting area is not as visible as in experimental studies. This is because Finite Element method is based on infinite deformations within the cracked a rea, and the crack tip is usually modeled with a perfect acute angle as shown in Figure 20, which is not the case in reality.

The good concordance between the two analysis however suggests the efficacy of "PASAPAS" operator within CASTEM in simulating the effect of temperature changing on J-integral calculation using Global Approach G-Theta Method (G_θ).

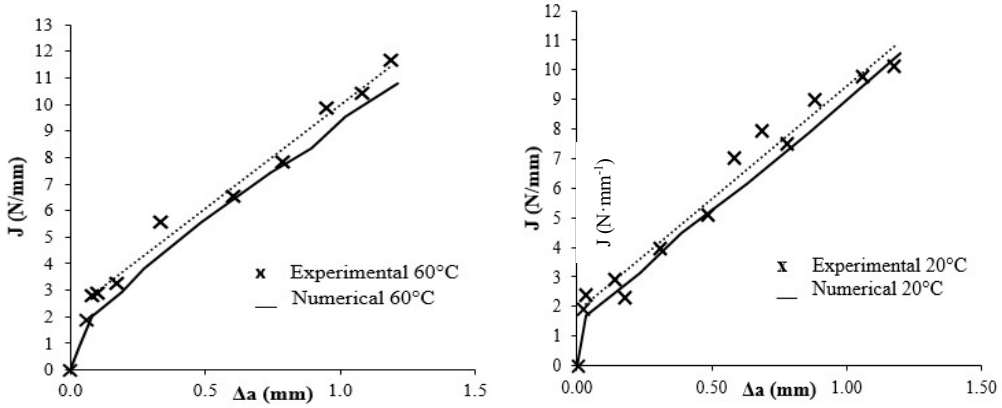

Figure 21. Numerical and experimental comparison of J-integral curves as a function of Δa at temperatures 20°C and 60°C.

5. Conclusions

The fracture behavior of semi-crystalline polymers such as high impact polystyrene can be described by geometry-independent fracture parameters. In addition, using the investigations of fracture mechanics, a correlation between structure and toughness properties, as well as initiation and crack propagation mechanisms, is possible. The use of the multiple specimen method for obtaining J-Δa curves is recommended for the general characterization of the fracture behavior of unstable growth toughness, for all the reinforced polystyrenes.

In conclusion, a clear transition in term of thermomechanical behavior was noticed in this work regarding High Impact Polystyrene, it can be said that in order to optimize the toughness properties by a suitable choice of the processing conditions and the choice of the material, it is necessary to take into account the influence of the temperature on the fracture behavior. Due to the difficult behavior of butadiene-reinforced polystyrenes in the presence of this environmental factor, which leads to a stable growth of cracks, the estimation of the resistance to stable growth of cracks is of great importance, in addition taking into account the resistance to the unstable growth of cracks. Comparing the J_{IC} values at each temperature we found a brittle-ductile transition in the material in terms of toughness, marked by an increase in crack resistance. This behavior is verified numerically via a simulation using CASTEM software.

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