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Analysis Of Low Temperature Impact Fracture Data Of Thermoplastic Polymers

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ABSTRACT: Impact fracture toughness of polypropylene (PP) blends, high density polyethylene (HDPE) and rubber toughened polymethylmethacrylate (RTPMMA) has been studied by means of three-point bending falling weight impact testing at different temperatures ranging from –60ºC to room temperature using the cleavage fracture toughness, J_C parameter [ASTM E1820-99a]. The latter Fracture Mechanics methodology was chosen due to its simplicity [Fasce et al., 2003]. Traces of the impact tests were analyzed using an inverse methodology just proposed by Pettarin et al. (2003). This methodology makes it possible to obtain from a three-point bending instrumented impact test the mechanical response of the material, discarding the dynamic effects associated with the test. The results show that the average J_C values calculated with treated and untreated data are similar for a given material, while the standard deviations are larger when the calculations are made with the untreated data. It is clear that the inverse methodology used to correct the data reduces error propagation, giving place to more precise estimations, and therefore more reliable J_C values.

1. INTRODUCTION

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 to and because it also diminishes the viscoelastic effects. However, the difficulty of obtaining reliable data from instrumented impact tests at high speeds is well known and pointed out in the literature [see for example Kalthoff, 1985; Williams and Adams, 1987; Pavan and Draghi, 2000].

Brittle fracture toughness, J_C, is the methodology chosen to assess fracture toughness [Fasce et al., 2003]. Under three-point-bending conditions and a crack-depth to specimen-width close to 0.5, this methodology can be applied to polymers displaying either linear or non-linear unstable fracture pattern under dynamic conditions. 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.

This parameter is commonly calculated from the experimentally measured load versus time curves. However, these curves are not what theoretically should be used for this purpose, because the measured load is not equal to the load exerted on the tested specimen, the load from which the mechanical performance of the material should be evaluated. The recorded load is corrupted by other forces acting during the experimental run, which depend in part on the characteristics of the tester and in part on the properties and geometry of the tested material. A simple method which combines a model mechanically equivalent to the system specimen-impact instrument and the inverse problem concept [Pettarin et al., 2003] is used to obtain an accurate estimation of the actual flexural curve in impact testing.

Room and low temperature impact fracture toughness parameter, J_C, of PP with rubber, HDPE, MDPE and RTPMMA has been assessed making use of the inverse methodology to analyze data.

2. EXPERIMENTAL DETAILS

Experiments were conducted on different polymeric materials. Two novel blends based on PPH modified with 10% and 20% wt of an elastomeric polyolefin (PP+10%POEs, PP+20%POEs), a third generation bimodal PE (PE100), and a rubber toughened polymethylmethacrylate (RT-PMMA).

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. At least seven specimens having a crack-depth to width-ratio of $0.45 \leq a_0/W \leq 0.55$ were tested.

Impact testing was carried out using a non-commercial falling weight apparatus equipped with data acquisition system, in three-point-bending (mode I). The specimen thickness, B, and the span to depth ratio, S/W, were always kept equal to W/2 and 4, respectively. Final dimensions were length $L = 100mm$, thickness $B = 10mm$, width $W = 20mm$ and span $S = 80mm$. PE100 samples were side grooved in order to avoid bowing of the crack front and ductile propagation after initiation [Dekker and Bakker, 1994]. Energy values were computed from the neat and filtered load-displacement curves. Impact tests were carried out at different temperature (-60ºC, -30ºC, 0ºC, 20ºC) and at $V = 1\, m/s$ according to the recommendations given in ISO 17281:2002 but without damping the contact between the striker and the specimen.

In calculations the influence of a small confined plastic zone or subcritical crack growth r_p at the crack tip has been taken into account by substituting the original crack length a_0 by $a_{eff} = a_0 + r_p$.

3. DATA ANALYSIS

3.1. Filtering of load-displacement traces: Inverse method

The bending force, P_b, acting in the specimen is the one that ideally should be determined from an impact test. Therefore, the problem to be solved is that of finding that force, P_b, from the knowledge of the force sensed in the striker and measured by the instruments, P_t. For that purpose the part of the model proposed by Pavan and Draghi (2000) which takes into account the striker and the viscoelastic contact between the striker and the specimen, was used (Figure 1) to develop an inverse methodology [Pettarin et al, 2003]. The interplay between P_t and P_b allows one then to define a direct problem and in inverse problem. The direct problem is that of obtaining P_t from P_b, something that can be done by direct integration of the differential equations of the model [Pavan and Draghi, 2000] for a given P_b. The inverse problem is the one posed when P_b is to be obtained from P_t, and is the relevant problem here.

The solution of the inverse problem is not as direct as the solution of the direct problem and was solved following the approach of transforming the differential equations into a first order Fredholm integral equation of the first class [Rust and Burrus, 1972] in the two functions, P_t and P_b, resulting in matrix form in:

$$\mathbf{p}'_t = \mathbf{A}\mathbf{p}_b \tag{1}$$

where $\mathbf{p}'_t$ is a vector containing the values of $P_t(t)$ at the discretization times, **A** is a matrix result of the quadrature process used, and $\mathbf{p}_b$ is a vector containing the unknown values of $P_b(t)$ at the discretization times. **A** is a function of the parameters of the dynamic model.

The direct solution of Eq (1) is known to suffer from the error amplification caused by the ill-posed nature of the problem [Twomey, 1996]. This undesirable feature of inverse problems has been tackled by solving Eq (1) in a form such that exact agreement between the experimental data and the model is sacrificed to reduce the oscillatory error that usually appears in the solution when no precautions are taken [Phillips, 1962]. Thus, the solution of Eq (1) is obtained as the solution of the following minimization problem:

$$\min_{\hat{\mathbf{p}}_b,\mathbf{p}} \left\{ \left| \mathbf{p}_t'(\mathbf{p}) - \mathbf{A}(\mathbf{p})\hat{\mathbf{p}}_b \right|^2 + \gamma\, q(\hat{\mathbf{p}}_b) \right\} \qquad (2)$$

where $\mathbf{p} = \left[k_t \quad m_t \quad k_c \quad r_c \quad m_{sc} \right]$. The model considered here, even though it is purely phenomenological, allows a direct experimental calibration of most of its parameters, with *ad hoc* experiments performed on the same specimen/tester system, using special arrangements of the test set-up [Pavan and Draghi, 2000]. Therefore, the values of k_t, m_t, k_c and r_c are estimated through independent experiments and m_{sc} is estimated together with $\mathbf{p}_b$ from Eq (2). $q(\hat{\mathbf{p}}_b)$ is a function that penalizes oscillations in $\mathbf{p}_b$.

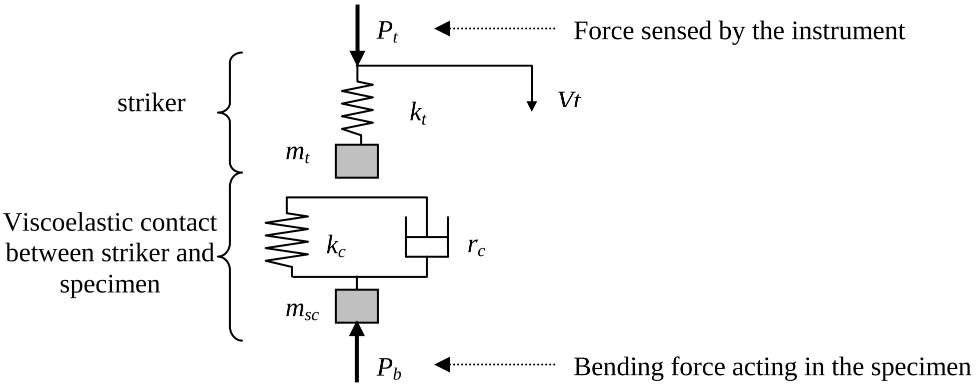

Figure 1. Schematic representation of the model involved in the inverse method.

The solution to the minimization problem of Eq (2) is given, for the case in which only m_{sc} is unknown, by:

$$\hat{\mathbf{p}}_b = [\mathbf{A}^T(m_{sc})\mathbf{A}(m_{sc}) + \gamma\mathbf{H}]^{-1}\mathbf{A}^T(m_{sc})\mathbf{p}_t'(m_{sc}) \qquad (3)$$

where the value of m_{sc} is the one that minimizes the single variable equation obtained after replacing Eq (3) in Eq (2), i.e.:

$$\phi(m_{sc}) = \left\{ \left| \mathbf{p}_t'(m_{sc}) - \mathbf{A}(m_{sc})\hat{\mathbf{p}}_b \right|^2 + \gamma q(\hat{\mathbf{p}}_b) \right\} \qquad (4)$$

H is a matrix given by the chosen penalty function. The value of γ, that establishes the smoothness degree of the sought solution, was calculated using the Generalized Cross Validation (GCV) technique [Golub et al, 1979]. The value of γ is automatically calculated for each test based only on the measured load-time curve and the model.

3.2. Non -Linear Elastic Fracture Mechanics: J_C Determination

The most widely accepted method to determine the high rate fracture toughness (around 1 m/s) for linear-elastic polymeric materials behavior is the critical energy release rate methodology [Pavan and Williams, 1999]. In a previous paper it have been demonstrated that Critical Energy Release Rate, G_{IC}, and Cleavage Fracture Toughness, J_C, appeared equivalent for a number of polymers displaying either linear or non-linear unstable fracture patterns [Fasce et al., 2003]. Thus, J_C testing, based only on direct determination of the total energy value of a set of similar samples may become the more attractive method due to its inherent simplicity.

The *J*-Integral is conventionally defined for non-linear elastic materials as a path independent line integral. Although ASTM E813-87 and ASTM E1152-87 apply only to ductile fracture, more recent standards permit *J* testing of materials that fail in a brittle manner. The J_C parameter [ASTM E1820-99a, 1999] as defined here is applicable to characterize brittle and 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 5), by calculating the fracture energy, U_{tot}, required to produce brittle behavior of pre-cracked specimens having a crack depth to width ratio of a nominal *a/W* equal to 0.5 in order to determine "cleavage fracture toughness" (J_C).

$$J_c = \frac{2U_{tot}}{B(W-a)} \tag{5}$$

4. RESULTS AND DISCUSSION

Typical load-time curves of materials obtained during instrumented impact tests are given in Figure 2. They showed superimposed oscillations of the force signal due to the well-known dynamic effects in impact testing [Williams and Adams, 1987].

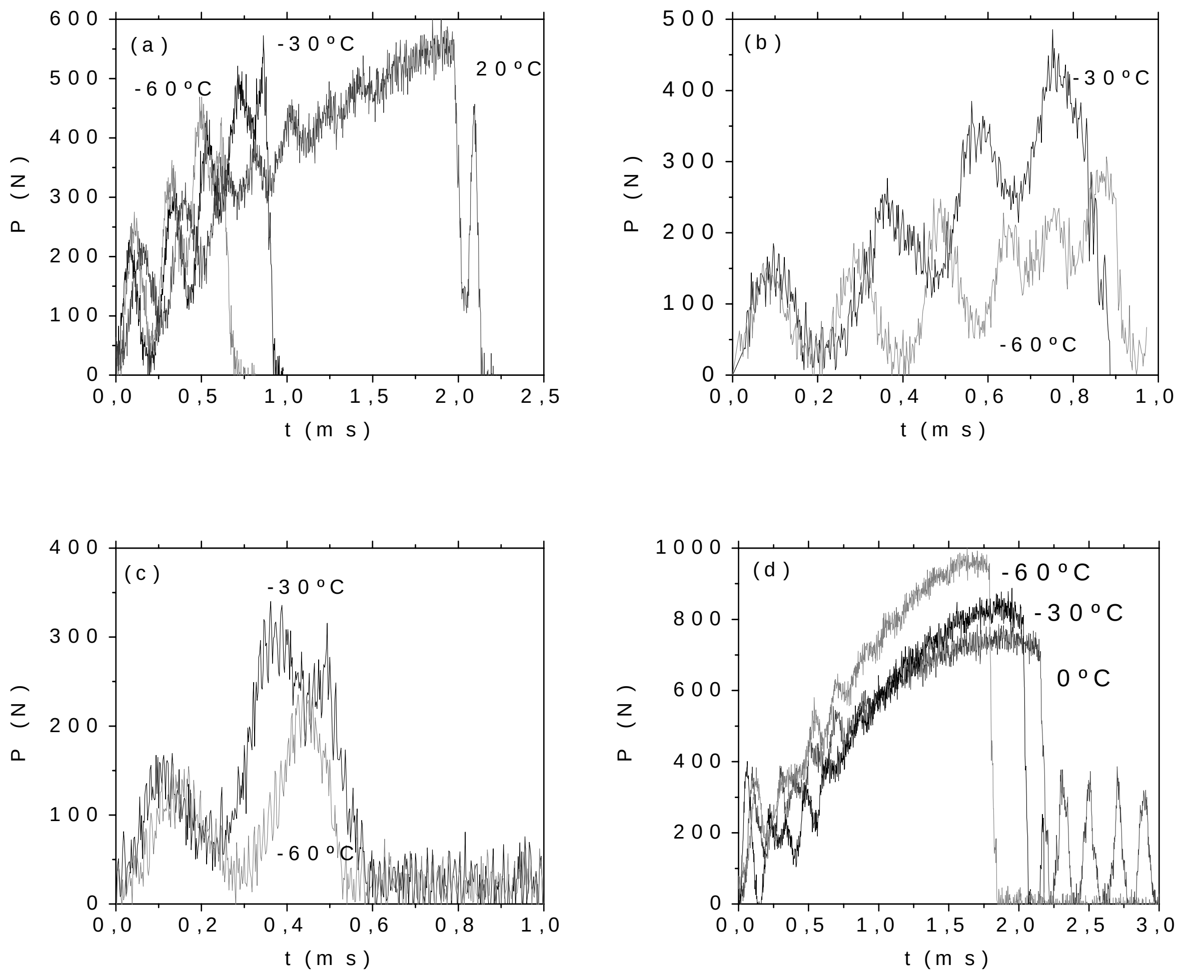

Figure 2. Load vs. time curves for tested materials. (a) PP+10%POEs (b) PP+20%POEs (c) RT-PMMA (d) PE-100

All materials fractured in an unstable manner but clear differences in behavior were observed among them. Modified PP and RT-PMMA exhibited complete brittle fracture as judged from the linearity of the load deflection records (Figures 2a,b,c) and the features of the fracture surface. Load-time curves dropped to zero instantaneously upon reaching the maximum load at relatively short time levels. PE100 exhibited semi-brittle behavior and developed limited plasticity ahead of the crack tip. The load increased non-linearly and displayed a drastic drop in coincidence with the sample failure (Figure 2d). Curves were influenced by temperature. Modified PP and RT-PMMA reached lower forces with decreasing temperatures, while PE100 reached higher forces but lower times to fracture with decreasing temperatures.

Original traces of Figure 2, which represent the forces sensed by the force transducers of the impact tester, were inverted using the proposed inverse methodology. Figure 3 show, for all materials, typical examples of the forces obtained through the inverse methodology. It is evident that the behavior of P_b is in all cases much less oscillatory than the original records, as noted previously [Pettarin et al., 2003; Pettarin et al., 2004], and the behavior of the samples is much more easy to recognize.

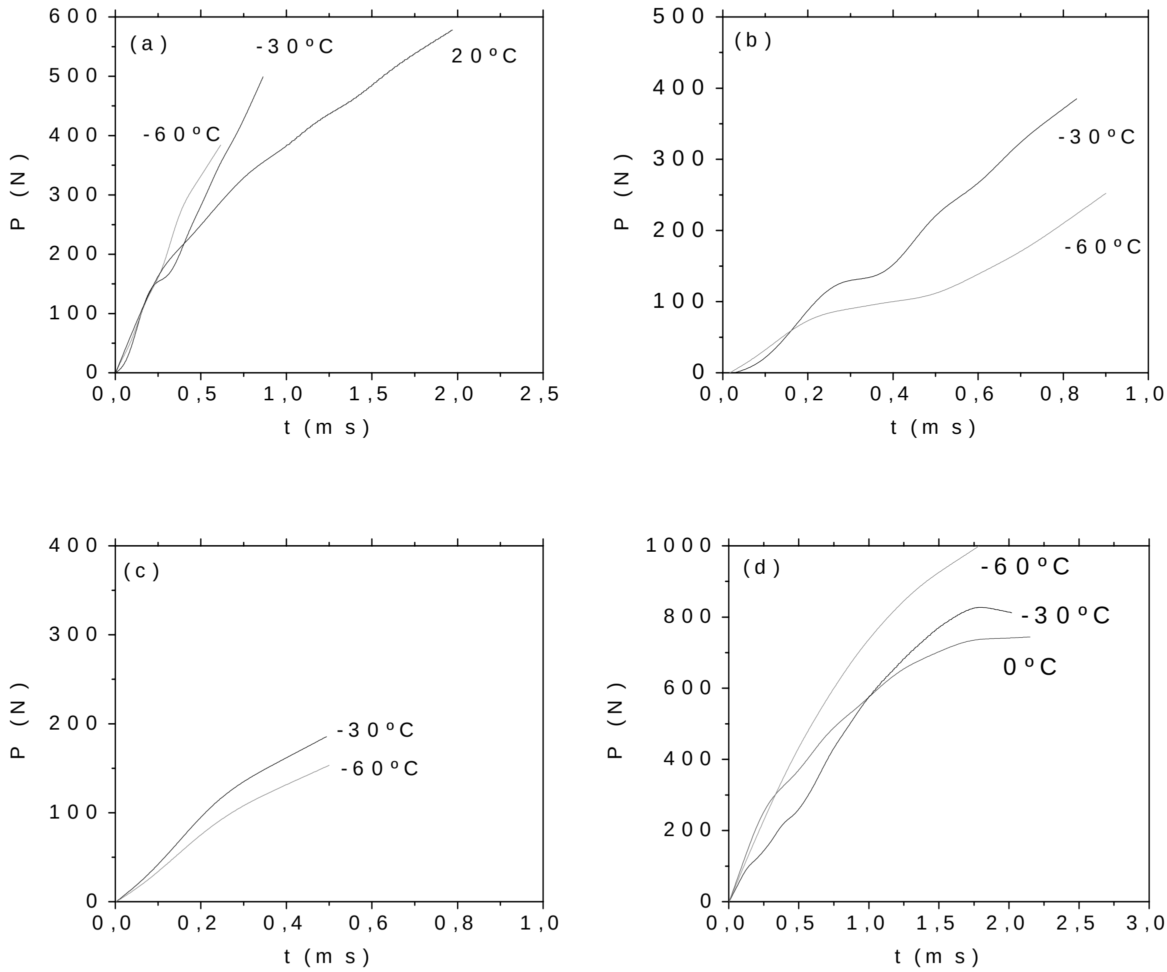

Figure 3. Estimated bending load P_b versus time for (a) PP+10%POEs (b) PP+20%POEs (c) RT-PMMA (d) PE100

The values of the mass of the sample that is in first contact with the striker, m_{sc}, appear to be material and temperature dependent (Figure 4). This result differs from other works exploring a wide range of test materials and geometries and striker material and shape [Maurer and Breuer, 1995] which stated that the "contact volume" (i.e. the contact mass times the density of the materials tested) is constant and has a magnitude of about 1.8 cm^3. This value was obtained considering only the (first) inertial peak of the recorded traces and modelling the contact occurring in the first part of the impact event by a simple one mass-one spring system. The more complex model used here, although it attaches the same meaning to the contact mass parameter, may be responsible of the different result obtained. Moreover, dependence of m_{sc} with material is in agreement with previous results [Pettarin et al., 2003; Pettarin et al., 2004].

Average values of calculated J_C are reported in Table I and Figure 5. The standard deviation of these values is also listed. Table I includes these two parameters for each one of the materials, calculated in two ways: from the original load-time data, and from the inverted load-time data. Results show that the average values calculated with both types of data are similar for a given material, while the standard deviations are larger when the calculations are made with the untreated data. Although these results are not conclusive in terms of the validity of the J_C values obtained with the corrected data, it is clear that if the dispersion of these calculations is attributed to random error, the inverse methodology used to correct the data reduces error propagation, giving place to more precise estimations, and therefore more reliable J_C values. As clearly seen in Table I and Figure 5, J_C values increases with temperature. Low temperature inhibits some failure mechanisms responsible of energy absorption (such as shear yielding) and therefore materials toughness are lower.

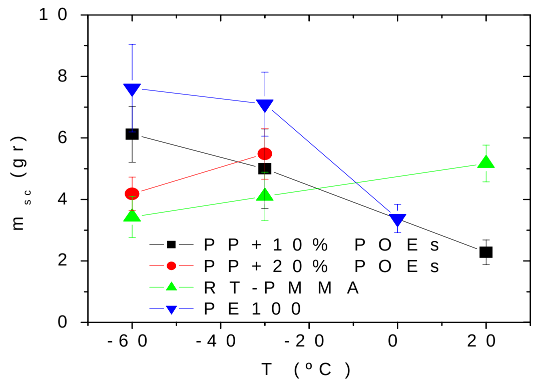

Figure 4. Estimated contact mass m_{sc} versus temperature for all materials.

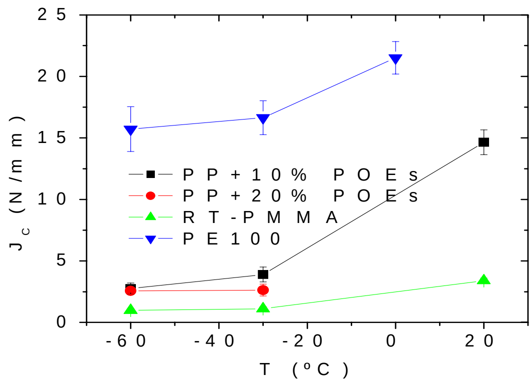

Figure 5. J_C values versus temperature obtained from filtered load-time data for tested materials.

Table I. Fracture parameters for all materials calculated from original and inverted data.

Material	Temp (ºC)	From P_t			From P_b		
		J_C (N/mm)	SD (abs)	SD (%)	J_C (N/mm)	SD (abs)	SD (%)
PP+10%POEs	-60	2.97	0.54	18.20	2.75	0.46	16.87
	-30	4.04	0.67	16.66	3.90	0.61	15.27
	20	14.82	1.14	7.67	14.65	1.01	6.89
PP+20%POEs	-60	2.68	0.36	13.37	2.56	0.28	10.82
	-30	2.87	0.59	20.47	2.63	0.48	18.33
RT-PMMA	-60	1.15	0.20	17.18	0.98	0.15	15.26
	-30	1.19	0.22	18.39	1.11	0.18	16.90
	20	3.46	0.26	7.51	3.40	0.21	6.17
PE100	-60	16.12	1.91	11.83	15.72	1.82	11.60
	-30	17.64	1.50	8.51	16.64	1.38	8.29
	0	21.06	1.74	8.27	21.51	1.32	6.18

5. CONCLUSIONS

The low temperature impact fracture data of thermoplastic polymers has been assessed by means of a novel inverse methodology developed for three-point bending testing [Pettarin et al., 2003]. This methodology is suitable to estimate the actual flexural curve in an impact-bending test which can be useful to calculate stress intensity factor curves, $K_I(t)$ vs t, and its dependence with time, dK/dt [Gensler et al., 2000; Morita et al., 2002]. Note that the use of the inverse methodology avoids the use of cushioning which can alter results if not done properly.

Parallely, the mass of the probe that is in first contact with the striker, m_{sc}, is also estimated and it appeared to be material and temperature dependent.

Fracture toughness was assessed by means of the J-Integral evaluated at the instability load point. J_C values have been estimated from raw and filtered data. There are no noteworthy differences between J_C values arising from both types of data, even if the inverse methodology used to correct the recorded curves reduces error propagation, giving place to more precise estimations, and therefore more reliable J_C values.

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