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Application of the miniature small punch test for the mechanical characterization of polymer materials

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ABSTRACT

The aim of this paper is to demonstrate the applicability of the Small Punch Test (SPT) in the mechanical characterization of polymers, following previous achievements in metallic materials. For this purpose, different polymers with a wide variation of tensile properties were examined. The applicability of this type of test to characterize polymeric materials is especially interesting when these products are in the form of films, as their greatly reduced thickness enables an easy preparation of the SPT specimens.

All the polymer materials were characterized by means of small punch and tensile tests. The tensile stress-strain curves were compared with the load-displacement SPT curves and the representative SPT parameters were defined accordingly. A good correspondence was found between the tensile elastic modulus and the initial slope of the SPT plot ($Slope_{ini}/t$), with t being the specimen thickness, and a good correlation was also observed between the tensile yield strength and the SPT P_{ym}/t^2 parameter. On the contrary, no corresponding relationship was found to predict the ultimate tensile strength or the failure elongation, since these properties depend greatly on the stress state of the test (uniaxial or biaxial).

1. Introduction

Small punch tests (SPTs) have been successfully employed for decades for the mechanical characterization of different metallic [1–6] and non-metallic materials [7,8]. One of the advantages of this test lies in the very small size of the specimens employed, with a characteristic length (side or diameter) smaller than 10 mm and thickness of 0.5 mm or lower. These specimens are firmly clamped between two circular dies and are bi-axially deformed into a circular hole until failure by means of an hemispherical punch, as displayed in Fig. 1.

The "load-punch displacement" record obtained from the test can be used to estimate several "conventional" mechanical parameters [5,9]. Fig. 2 shows a typical load-displacement response obtained with a ductile steel specimen, identifying different zones (I–IV), where several points of the curve, relevant to tensile parameter correlation, are highlighted. The initial slope of the curve could be related to the elastic modulus [3]; the P_y load, located in the transition between the elastic (I) and the plastic (II) zones, related to the yield stress, σ_y [5,9]; the maximum load, P_m, related to the ultimate tensile strength, σ_u [5,9]; the maximum displacement, d_m, related to the tensile elongation, $e(\%)$ [10,11]; and the area under the load-displacement curve, W_u, related to the fracture toughness [9,12]. Some of the typical relationships between the aforementioned SPT parameters and the tensile properties are:

$$\sigma_y = \alpha \frac{P_y}{t^2} \quad (1)$$

$$\sigma_u = \beta \frac{P_m}{t^2} \quad (2)$$

$$e(\%) = \gamma \frac{d_m}{t} \quad (3)$$

being t the initial specimen thickness and α, β and γ the characteristic material coefficients.

To a much lesser extent, the SPT has also been employed in the characterization of polymeric materials [13–19]. Most of the studies on these materials have been purely devoted to analyze the form of the SPT curves [13,15], and correlations between the tensile properties of polymers and their corresponding SPT parameters are yet to be identified. Therefore, it seems to be particularly appealing to investigate whether or not it is possible to derive any correlation between the SPT parameters and those commonly employed to characterize their mechanical properties.

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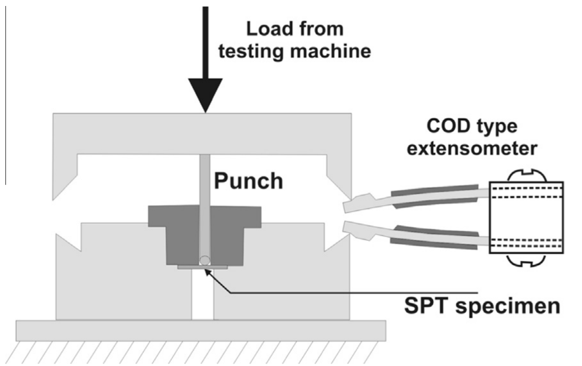

Fig. 1. Small punch test configuration.

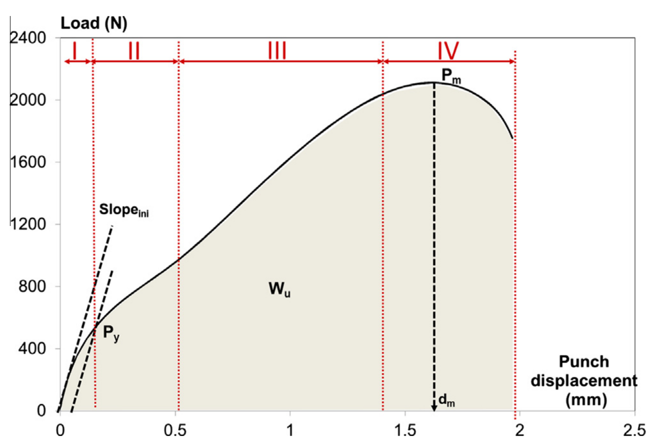

Fig. 2. Typical SPT plot obtained with a ductile steel specimen.

In a recent study [16] using polylactide acid (PLA) modified with montmorillonite as specimens, the relationship between the yield strength and the P_{ym}/t^2 SPT parameter was established (being P_{ym} the load corresponding to the first maximum obtained in the SPT record). In this same study, the area under the SPT curve was also related to the essential work of fracture.

More recently, Maspoch et al. [17] have also investigated the modification of the ductile-to-brittle transition of the PLA related to the time elapsed after the application of a de-aged thermal treatment by means of the SPT. They also established a correlation between the slope of the SPT curve (defined in the zone just before the first local maximum of the curve) divided by the specimen thickness (Slope/t) and the tensile elastic modulus.

All these correlations were corroborated in a further study [18] using other type of polymers. Good correlations were obtained between the elastic modulus and yield strength with the respective SPT parameters, Slope/t and P_{ym}/t^2, but the corresponding relations were markedly different than the ones obtained [17], so that a systematic experimental program is still necessary to ensure the applicability of the SPT to the mechanical characterization of polymers.

Consequently, the aim of this paper is to analyze whether SPT is a useful tool for the assessment of the most characteristic tensile mechanical properties of polymers. A possible correlation between SPT results and tensile properties is also discussed.

2. Materials

Four different types of polymers commonly used as films and laminates were studied in this work, namely polypropylene (PP), polyethylene terephthalate (PET), poly(lactic acid) and ethylene vinyl alcohol (EVOH). The aim was to study the influence of the molecular structure and morphology on the SPT properties. Therefore, two amorphous (PET and PLA) as well as two semicrystalline materials (PP and EVOH) were selected. Moreover, the effect of the dispersion state of a nanofiller (montmorillonite, MMT) on an amorphous (PLA) and on a semicrystalline polymer (EVOH) was assessed. This type of nanofiller is incorporated into polymers in order to improve certain properties, e.g. in the case of PLA and EVOH it is well known that the barrier properties improve upon MMT incorporation which is beneficial for food packaging.

Injected PP, which had been used in [19] to study the effect on mechanical strength of adding different percentages of recycled material, corresponded to grade BASELL X9077. PET and PETg were supplied by NUDEC in form of extrusion-calendered laminates. For the synthesis of PETg, CHDM is used to introduce discontinuities in the polymer chain in order to make the crystallization process more difficult, this material being nearly amorphous even in the form of thick plates.

PLA 2002D (96% L-lactide isomer) was supplied by NatureWorks and a commercial EVOH (Soarnol®AT44 03B) containing 44% mol of ethylene was used. Organically modified montmorillonite (Cloisite 30B), with 30 wt.% organic modifier, was supplied by Southern Clay Products.

Neat PLA (PLA_0%) and EVOH (EVOH_0%) laminates as well as their nanocomposites were prepared by twin screw extrusion in the *Centre Català del Plàstic.* Nanocomposites with different MMT concentrations were prepared in a three-step melt-extrusion process using a Collin ZK-35 co-rotating twin-screw extruder (L/D = 36; D = 25 mm). The first step consisted in the preparation of a masterbatch containing 10 wt% of clay. In the second step this masterbatch was reprocessed in order to achieve a homogeneous mixture. In a third step the masterbatch was diluted with the polymer matrix to the desired concentrations. In all stages, the twin-screw rotation speed was set at 80 rpm. In the case of EVOH the temperatures were between 160 °C in the feed section and 220 °C at the extrusion die and in the case of PLA the temperatures were between 150 and 175 °C. EVOH was prepared with nominal clay content of 1 (EVOH_1%) and 2.5 (EVOH_2.5%) wt% and PLA contained 0.5 (PLA_0.5%) and 2.5 (PLA_2.5%) wt% of MMT. Sample preparation is described in detail in Refs. [20,21].

Films of PLA were subjected to a de-aging thermal treatment [16], consisting of heating to 60 °C for 20 min and quenching by immersion in an ice-water bath for 5 min. These samples were coded with a T in order to indicate the thermal treatment (PLA_0T, PLA_0.5T and PLA_2.5T).

3. Tensile and small punch tests

The mechanical characterization of the different polymers was carried out using tensile and SPT tests. While some experiments were conducted in different laboratories, special care is taken to ensure that the same conditions apply in all case studies.

In the case of polypropylene, analyzed at the University of Burgos, 5A type standard tensile specimens and rectangular samples (60 × 10 × 4 mm), both obtained by an injection process, were supplied by the manufacturer [19]. SPT specimens (10 × 10 × 0.5 mm) were extracted from the rectangular samples, all of which were machined and polished to a thickness of 0.5 mm.

The rest of the polymers were manufactured in the Centre Catalá del Plástic. In these cases, the tensile specimens had a standard type IV geometry and were extracted along the longitudinal direction of the films. Nominal SPT specimens with the thickness of the film were also machined.

All the mechanical tests were performed at room temperature. Tensile experiments were carried out according to the ASTM D-638