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Mechanical behavior and failure analysis of recycled polymers by use of miniature punch specimens

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ABSTRACT: In today's society, recycling is a priority, and using recycled materials to obtain new 100% reusable materials is an important aspect of some manufacturing sectors. Clearly, the mechanical properties of these new materials can vary considerably from the original material, so it is necessary to carry out a complete mechanical characterization to know its behavior for both standard specimens and final components. One of the common drawbacks is the inability to extract standard size specimens from a final component with a reduced size. In this paper, the use of the small punch test is proposed as a means of solving this problem. At present, this test is used for estimating mechanical properties in those cases when there is not a sufficient amount of material to perform standard tests. The main purpose of this paper is to analyse the feasibility of using miniature punch specimens for the mechanical characterization of recycled polymers. The results are compared with those obtained from standard uniaxial tensile specimens, and a corresponding correlation between the two tests is established. © 2015 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2016**, *133*, 42911.

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INTRODUCTION

Nowadays, recycling plays a fundamental role in sustainability and respect for the environment. For this reason, the use of recycled materials to obtain new 100% reusable materials is a priority in some manufacturing sectors. Clearly, the mechanical properties of these new materials can vary considerably from the original nonrecycled materials, so it is necessary to carry out a complete mechanical characterization in order to know their behavior both for standard specimens and for final components. For standard characterization, it is usual to perform tests according to ASTM D638-14[1] or to EN ISO 521-1,[2] standard test methods for tensile properties of plastics, or to ASTM D882-12,[3] the standard test method for tensile properties of thin plastic sheeting. These test methods are designed to produce tensile property data for the control and specification of plastic materials. These data are also useful for qualitative characterization as well as for research and development.

One common drawback is the inability to extract standard size specimens from a small final component. To overcome this problem, there are now several alternative tests for miniature specimens. One is the small punch test (SPT), successfully used for mechanical property estimation in those cases where there is not sufficient amount of material to perform standard tests. This test was developed in the nuclear field in the eighties[4] and has since been used successfully on numerous occasions on different materials including metallic,[5–10] polymeric,[11] and even biological.[12] The experimental setup can be consulted in the CEN Code of Practice for small punch testing.[13] This Code of Practice was developed to provide guidance in the experimental conditions for the small punch test, and it is suitable for obtaining accurate and reproducible results.

The main objective of this paper is to analyse the feasibility of using miniature punch specimens for the mechanical characterization of recycled polymers. The results are compared with those obtained from standard uniaxial tensile specimens and a corresponding correlation between the two tests is established.

EXPERIMENTAL

Different compositions of BASELL X9077 polypropylene with varying percentages of recycled polypropylene were selected. The recycled polypropylene comes from defective pieces which have been injected with nonrecycled material (BASELL X9077). These defectives pieces are then ground up and later mixed with non-recycled material to obtain recycled material. Two types of specimens, standard tensile type 5A,[2] and rectangular (60×10×4 mm), were obtained by injection. The following percentages of recycled polypropylene were used: 0, 10, 20, 30, 40, and 50%.

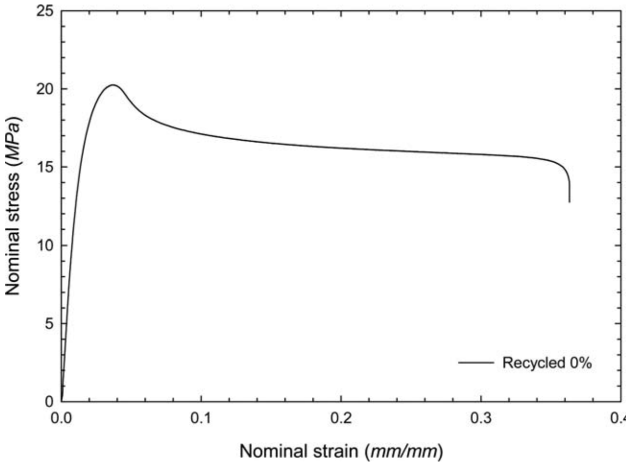

Figure 1. Nominal stress–strain curve.

After the standard tensile type 5A specimen testing, it is possible to extract the elastoplastic parameters such that the tendency of each parameter, depending on the amount of recycling, can be found. Two of the most important elastoplastic parameters during component validation by numerical simulation are Young's modulus (E) and yield strength (σ_y). Additionally, three more parameters have also been extracted: strain at yield strength (ε_y), ultimate stress (σ_b) and ultimate strain (ε_b).

The small punch test basically consists of deforming a miniature specimen (10×10×0.5 mm), whose edges are firmly gripped by a die, using a high strength punch. During the test, values of applied load and punch displacement are collected, after proper treatment of the stored data a load–displacement curve is obtained. Each of the zones in which the SPT load–displacement curve can be divided is influenced by different material parameters.[14] However, the material properties cannot be extracted directly from the SPT load–displacement curve but rather associated with certain values extracted from this curve such as the yield load, maximum load and displacement at maximum load.

In order to analyse the feasibility of using miniature punch specimens for the mechanical characterization of recycled polymers and to establish a corresponding correlation between the two tests (SPT-tensile test), SPT specimens were extracted from the injected rectangular specimens, all of which were polished to a thickness of 0.5 mm. The tests were conducted at room temperature using a punch diameter of d_p=2.5 mm, the punch drop rate was v=0.5 $mm/$min and the hole in the lower die had a diameter of D_d=4 mm and a fillet radius of r=0.5 mm. For each SPT specimen, a load–displacement curve was obtained. Due to the influence on the results of the specimen thickness, which in some cases had not been perfectly achieved, the SPT load-displacement curves had to be corrected to a reference thickness (t=0.5 mm) to be able to compare them.[15] For polymeric materials, the shape of these curves can be very different depending on the type of polymer tested, so it is necessary to carefully analyse the shape to verify which parameters can be extracted.[11]

One of this parameters, the yield load (P_y), can be correlated directly with the yield strength. In other research, one can find different ways to extract P_y from the load–displacement curve. The best correlation results are obtained with two of these: the "two tangents method", proposed by Mao and Takahashi,[16] which consists of extending the linear sections of the first stages of the curve and obtaining their cutoff point, and the "$t/10$ offset method", which consists of drawing a parallel line to the elastic slope of the load–displacement curve for the corresponding $t/10$ displacement value and determining its cutoff point.[11] In this paper, the second method has been used, because the first was especially developed for metallic materials or others with similar behavior.

RESULTS AND DISCUSSION

For tensile tests, Figure 1 shows a typical curve for the material used. It should be noted that for all percentages of recycled material the shape of the curve is the same. Table I shows the mean values of the elastoplastic parameters, which have served to establish the corresponding correlation between the uniaxial tensile test and the small punch test.

In Figure 2, several SEM images corresponding to the failure area of the tensile specimens can be seen. These images were obtained with a JEOL JSM-6460LV scanning electron microscope. For all percentages of recycled polypropylene, two distinct zones are observed: both a ductile (DF) and a brittle (BF) failure zone. This brittle zone becomes more irregular as the

Table I. Mean Tensile Parameter Values

Recycled (%)	Young's modulus (MPa)	Yield strength (MPa)	Strain at yield strength (mm/mm)	Ultimate stress (MPa)	Ultimate strain (mm/mm)
0	1299.9 ± 11.8	20.9 ± 0.17	0.040 ± 0.001	21.9 ± 1.6	0.354 ± 0.079
10	1290.7 ± 44.5	20.1 ± 0.17	0.038 ± 0.001	23.3 ± 3.1	0.454 ± 0.086
20	1313.1 ± 56.6	20.2 ± 0.04	0.040 ± 0.001	27.8 ± 1.5	0.630 ± 0.061
30	1283.5 ± 1.7	20.2 ± 0.07	0.042 ± 0.001	27.7 ± 0.4	0.612 ± 0.001
40	1298.8 ± 6.2	20.1 ± 0.03	0.042 ± 0.001	25.1 ± 0.5	0.515 ± 0.017
50	1305.0 ± 64.6	20.3 ± 0.35	0.038 ± 0.006	25.1 ± 1.1	0.493 ± 0.062

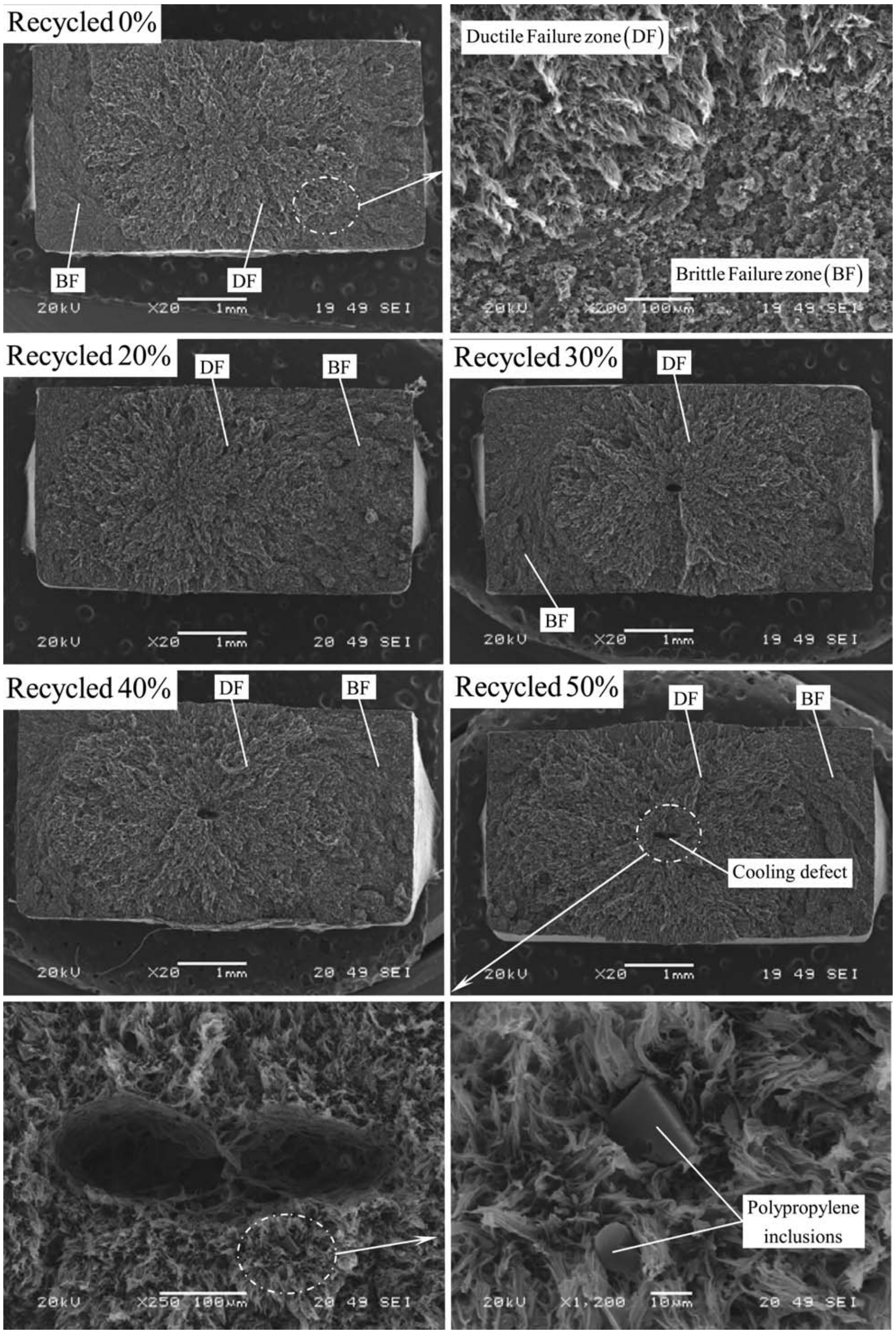

Figure 2. Tensile specimens.

percentage of recycled material is increased since areas with "plateaus" appear. It was also detected in some specimens that from 30% or higher of recycled material, a central tunnel-shaped defect appears, possibly formed during cooling of the injected specimen. This fact, which may depend on the quality of the recycled material and processing conditions, requires special attention because it could be a weak point in the injected component and cause premature failure. In addition to that, for the 50% recycled specimens, polypropylene irregular inclusions were found in the central area of the specimen which were not well fused with the material matrix.

For small punch tests, representative curves of different percentages of recycled polypropylene can be compared in Figure 3(a). Figure 3(b) shows a typical SPT load–displacement curve of the polypropylene used in which the parameters that were extracted for each miniature specimen are indicated. These are the following: the yield load (P_y), maximum load (P_{max}), displacement at

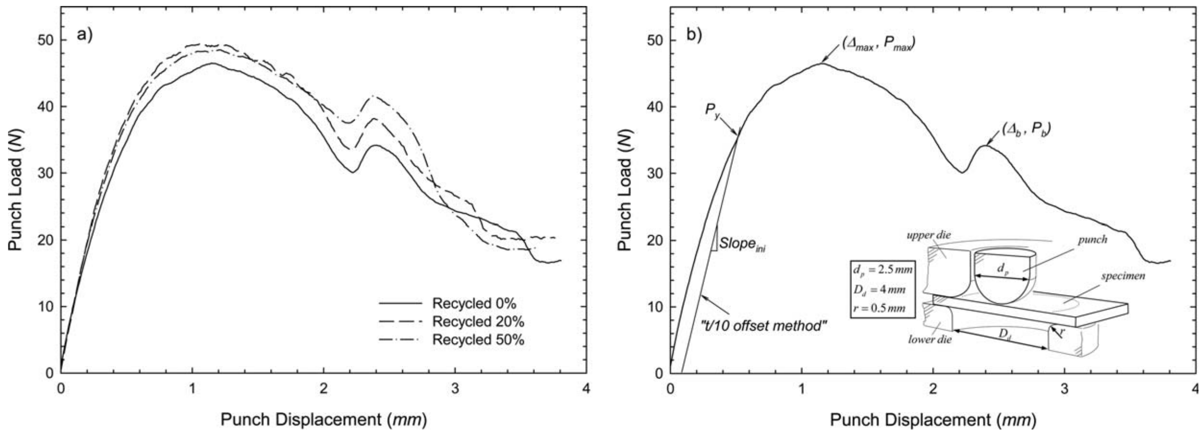

Figure 3. Representative SPT curves and SPT parameters.

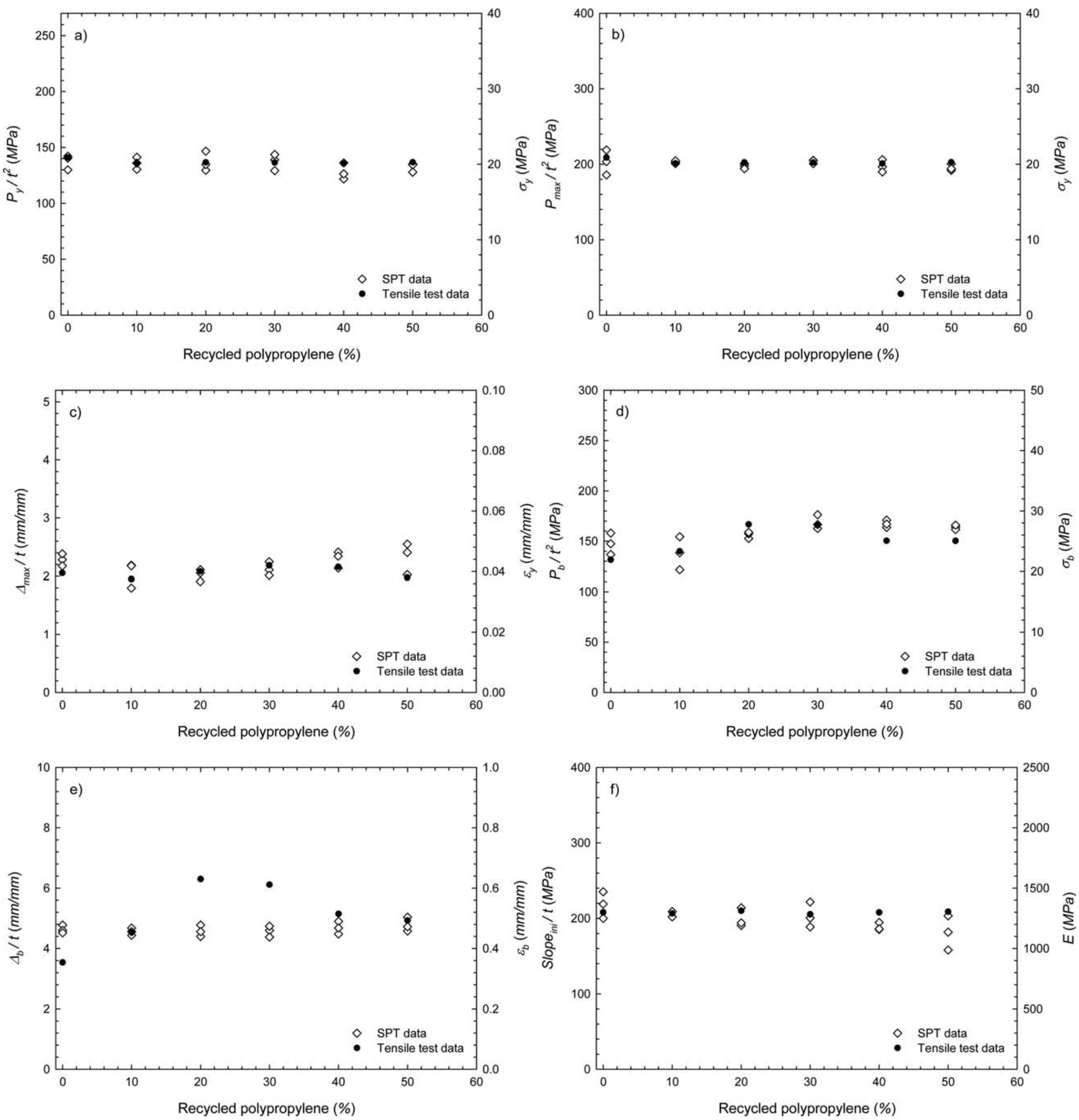

Figure 4. Parameter tendencies depending on the percentage of recycled material.

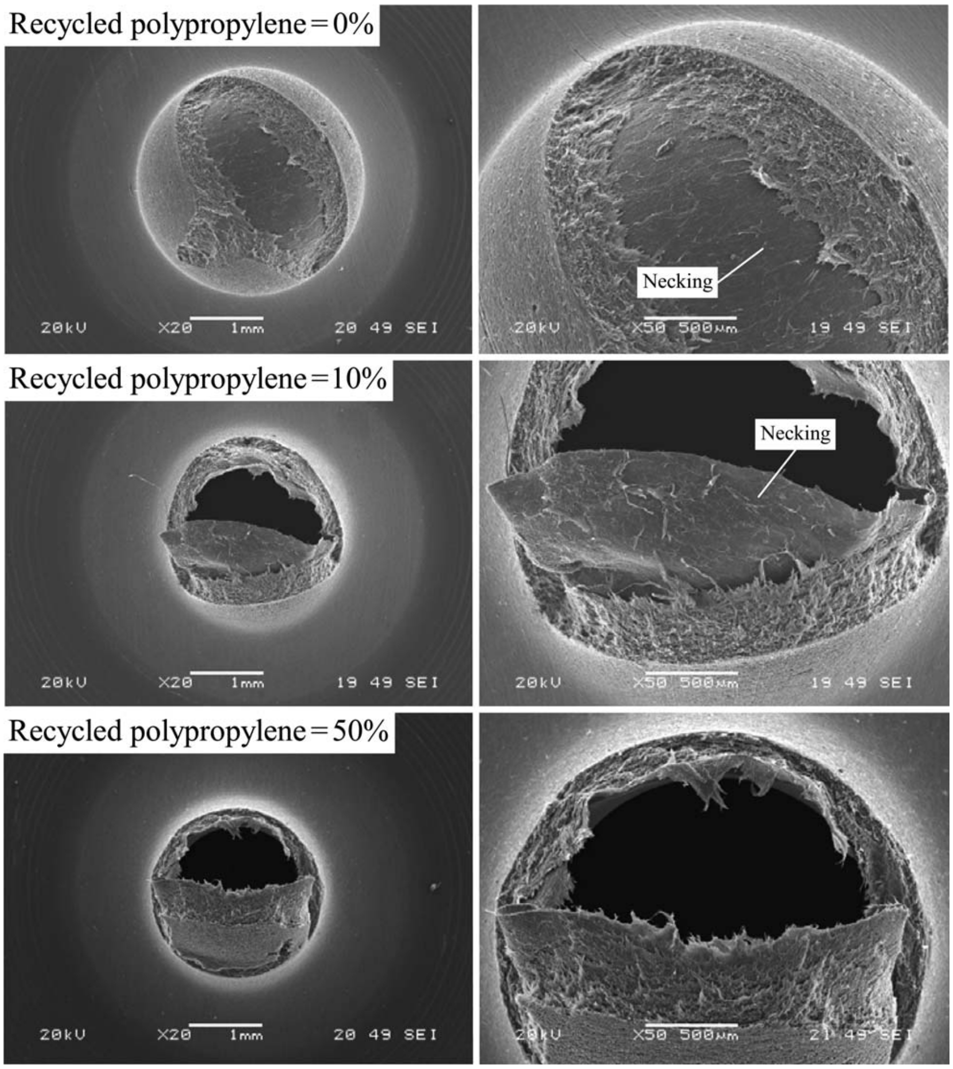

Figure 5. Small punch test specimens.

maximum load (Δ_{max}), rupture load (P_b), displacement at rupture load (Δ_b), and the initial zone slope (Slope_{ini}). These parameters, conveniently normalised using the specimen reference thickness (t), could be related to their corresponding parameters for the tensile test using a dimensionless coefficient (α, β, φ, γ, η, and λ) in each case by means of the following equations:

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

$$\sigma_y=\beta\cdot\frac{P_{max}}{t^2} \quad (2)$$

$$\varepsilon_y=\varphi\cdot\frac{\Delta_{max}}{t} \quad (3)$$

$$\sigma_b=\gamma\cdot\frac{P_b}{t^2} \quad (4)$$

$$\varepsilon_b=\eta\cdot\frac{\Delta_b}{t} \quad (5)$$

$$E=\lambda\cdot\frac{\text{Slope}_{ini}}{t} \quad (6)$$

For correlations (4) and (5), the data of ultimate stress and ultimate strain have been used respectively, because it was observed that SPT specimen failure occurs when the second load peak was reached.

Once results of both tests (tensile test and SPT) are available, the parameter tendency for the different specimens can be analysed depending on the percentage of recycled material, with Figure 4 showing these tendencies. In each of the graphs, a standard SPT parameter with its equivalent tensile test parameter is shown. For example, in Figure 4(a) the tendency between P_y/t^2 and σ_y, depending on the percentage of recycled polypropylene, can be compared. For all pairs of the compared parameters, a suitable correspondence between the two tests can be observed. Only in Figure 4(e), which corresponds to the pair of parameters $\Delta_b/t-\varepsilon_b$ and for recycled percentages of 0, 20, and 30%, there is a noticeable divergence between the two tests.

As indicated above, the parameters in both tests can be related to each other using expressions (1) to (6), in which the corresponding dimensionless coefficient must be calculated for each one. The question that logically arises at this point of the

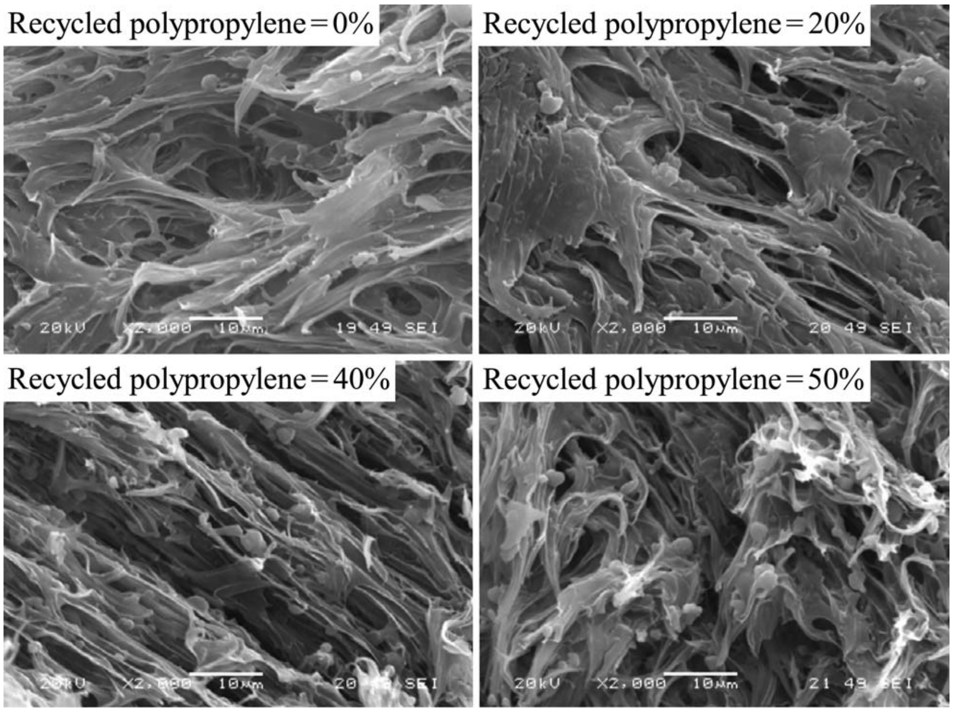

Figure 6. Microstructure of the fracture zone.

research is whether the coefficient for each expression has a single constant value or one that varies according to the percentage of recycled material. It can quickly be seen that a single tendency does not appear for all coefficient values. In three of them (α, β and φ), the value hardly varies as the percentage of recycled material increases. Two of them reach their maximum for intermediate values of the percentage of recycled, specifically γ for 10–20% recycled material and η for 20–30% recycled material. The last one (λ) increases progressively with increasing recycled material. Accordingly, for those coefficients that do not remain constant, it is necessary to know the percentage of recycled material in the component to be analysed if the mechanical properties are to be estimated using the small punch test. These coefficient variations are directly related to how the recycled material affects the mechanical properties of the injected component. So the SPT can be a useful tool to estimate the behavior of any part of an injected component when standard specimens cannot be extracted.

Finally, the failure mode of miniature specimens, based on the percentage of recycled material, has been analysed (Figure 5). Two distinct failure modes have been found. The first predominates in the specimens with low percentages of recycled material (0–20%), and the second is characteristic of the high percentages (30–50%). On the one hand, the first failure mode (necking failure) occurs in a manner analogous to that observed in Nakazima specimens[17] and also causes a small necking in the upper layer of the specimen that is in contact with the punch. On the other hand, the second failure mode (circumferential failure) occurs circumferentially and is typical for ductile materials in small punch tests.[5]

For all percentages of recycled material analysed, a ductile failure similar to that observed in the ductile failure zone (DF) of tensile specimens (Figure 2) can be observed. Figure 6 shows the microstructure of the fracture zone for various percentages of recycled material, in which no significant differences can be observed. In all of them, due to high strain, the polypropylene chains are stretched and lined up, creating microvoids between them.

CONCLUSIONS

Since the feasibility of using miniature punch specimens for the mechanical characterization of recycled polymers has been shown, it could be said that the main objective of this paper has been reached. It has been demonstrated that by using the small punch test, the behavior variation of the material according to the percentage of recycled material can be discerned. Not only that, from the results obtained in the tensile and small punch tests, the corresponding correlation for each typical parameter in each test has been established, so the variation that these parameters undergo can be easily observed as a function of the percentage of recycled material. Concerning the dimensionless coefficients, different tendencies in some of them have been found based on the percentage of recycled polypropylene. For those coefficients that do not remain constant, knowledge of the percentage of recycled material in the component to be analysed is obviously necessary when using the small punch test to estimate mechanical properties, however for those coefficients that remain constant, knowing those percentages is not necessary. Obviously, these correlations and conclusions cannot be extended directly to other polymers, and more research is needed to know the behavior of other recycled polymers, but this paper represents a first step in the determination of the mechanical behavior of recycled polymers from miniature specimens when there is not enough material to perform standard tests.

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