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Experimental and Morphological Investigations of Fracture Behavior of PBT/TPEE

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Abstract

Short-fiber-reinforced polymers are widely used in industry. They are light-weight, have excellent mechanical properties and can be processed via injection molding. This allows the mass production of high-quality components with high geometric accuracy. Their superior electrical isolation properties make them a good choice for electrical housings in the automotive sector. Due to the importance and precise nature of applications, in which such products are employed, many studies have investigated the properties of these materials. Polybutylenterephthalat (PBT) with thermoplastic polyester elastomer (TPEE), an impact-enhancing additive, is a typical example. Still, there is a lack of knowledge regarding the effect of TPEE on mechanical and fracture behaviors of short-fiber-reinforced PBT and the effect of its microstructure on the dynamic performance. To study the characteristics of modified short-fiber-reinforced PBT and to assess the effect of the filament, two types of polymers - standard PBT-GF10 and PBT-GF10 blended with 10\% TPEE - were compared. Morphological investigation of fracture surfaces produced in tensile tests at different loading rates was undertaken with scanning electron microscopy (SEM). Further two-dimensional image analysis was completed with the image processing software ImageJ. The morphological analysis showed that TPE-E generally affected the microstructure of the material. Micrographs of fracture surfaces demonstrated a decrease in the size of area of ductility with increasing loading rate. These results will support the development and design of optimized parts and their processing methods.

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1. Introduction

To avoid synthesizing new materials and investing into expensive polymerization equipment, polymer blending has become more attractive over the years. Blending of two polymers provides a simple and cost-effective method to obtain new materials with enhanced properties without losing the material’s original advantages.

Poly-butylene terephthalate (PBT), a linear aromatic polyester with high-performance semi-crystalline resin, is one of the most versatile engineering thermoplastics. It has high mechanical strength, excellent processing characteristics and outstanding electrical properties, making it applicable for a broad range of products. To increase the strength, modulus and toughness of the matrix, short glass fibers are added to PBT. This composite, short-fiber-reinforced PBT (SFR PBT), is widely used in industry, and its characteristics and behavior are well studied by the academic community. But SFR PBT has a few limitations. One of most important disadvantages is its low impact strength.

Thermoplastic polyester elastomer (TPEE), a new member in the thermoplastic-elastomer (TPE) family, has recently attracted much attention. It was proven to be one of the TPEs, suitable for blending with PBT. Its hard segments are crystalline polyesters like these of PBT (Kalfoglou et al., 1977). Research was recently focusing on the effect of TPEE on impact strength of SFR PBT. For instance, Verma et al. (2008) observed significant improvement in the impact toughness of PBT/TPE blends.

But the reported work concerning the effect of TPEE on the microstructure and fracture behavior of SFR PBT is still very limited. Generally, indications of ductility or brittleness on a fracture surface may give a good idea of the mode of fracture and the material’s mechanical properties. For instance, the ductile area, observed on fracture surfaces of PA66-GF35 specimens failed under fatigue loading, was reported to be larger than that for specimens tested under static loading (Horst and Spoormaker, 1997). Other studies showed the effect of TPE on tensile and impact strength of PBT; with increasing TPE content impact strength increased, and tensile strength decreased as reported by Verma et al. (2008). The effect of loading rate on the microstructure of impact-modified PBT and the impact on various mechanical properties has not been considered to date.

In this paper, specimens of PBT-GF10 (10% glass fibers) and PBT-GF10 blended with 10% TPEE are tested to failure in tension at different loading rates. The morphology of fracture surfaces is studied for different loading rates to assess the effect of TPEE on the microstructure and damage mechanism of PBT-GF10. Material performance of these materials will be discussed in detail in further publications.

2. Experimental details

2.1. Materials

Two materials were studied:

- a commercially available short-fiber-reinforced PBT containing 10 wt. % of glass fibers (designation: PBT-GF10);
- a commercially available short-fiber-reinforced PBT with 10 wt. % of glass fibers and 10%vol. TPEE (designation: PBT-GF10 TPEE)

Fig. 1. ISO 527 dogbone specimen. (all dimensions are in mm)
2.2. Specimen

All the used samples were standard ISO 527 dogbone specimens (Fig. 1) produced with injection molding. As a result, the fibers were distributed randomly in the matrix, mainly oriented along the flow direction in the mold, which was the longitudinal direction of the tensile tested specimen (i.e. along the loading direction).

2.3. Tensile testing

Specimens were conditioned at 50% relative humidity at room temperature for 4 weeks. Tensile tests were performed on a Zwick Z010 machine equipped with a 10 kN load cell. Specimens of these two materials were tested up to failure at four different loading rates: 2, 20, 200 and 400 mm/min.

2.4. Morphological analysis - SEM observations and image analysis

Morphological analysis was completed using Phenom XL scanning electron microscope with 5-15kV accelerating voltage. To prevent specimens from charging, a charge-reduction mode, which is presented as low vacuum mode, was activated. This mode produced best results with respect to noise reduction and micrographs quality. Another microscope, JSM-7500F scanning electron microscope with 5-15kV accelerating voltage was also used. For investigations with this microscope, specimens were sputter-coated with a 10-15 nm thick layer of gold-palladium to provide an efficient charge transfer. The aim of employing two different microscopes and different methods was to ensure that there were no image quality losses due to the charging problem. All fractographs were processed using the image processing software ImageJ to calculate the areas of ductile regions and single conic structures.

3. Results and discussion

In SEM analysis, the parts of surfaces of both materials could be clearly classified into two major types: ductile and brittle; Fig. 2 shows the fracture surface obtained in a tensile test completed at a loading rate of 2 mm/min.

3.1. Ductile area

As can be seen in Fig. 2, a part of each surface had a rough structure characterized with stretched ligaments of the matrix, a clear indication of a ductile-failure mode of the matrix. Such a structure can be a result of formation of micro cracks at a fiber-matrix interface and their coalescence (such ductile regions are surrounded with a yellow line in the fractographs shown in Fig. 2). The analysis of all micrographs at different loading rates demonstrated that there was only one region of ductile area on each fracture surface. Referring to the fact that the ductile area is the region of crack initiation and its stable growth, a conclusion can be drawn that for both materials the crack was formed at a single region inside the matrix. Furthermore, it was noticed that location of ductile areas was unpredictable on the fracture surfaces, at all loading rates: ductile area was located at the edge of the fracture surface for some specimens and randomly in the middle for other specimens.

Fig. 2. Ductile area surrounded by yellow line on fracture surface of specimens after tensile test at 2 mm/min: (a) PBT-GF10; (b) PBT-GF10 TPEE
3.2. Brittle area

The second major region on the fractures of tested specimens included considerable fiber pull-outs but a relatively smooth surface pattern on the matrix, as seen in Fig. 3. This is a clear manifestation of a brittle failure mode of the matrix. Still, the difference of these regions in both materials can be clearly noticed. PBT-GF10 in Fig. 3a shows a smoother and more planar texture than the brittle area observed for PBT-GF10 TPEE. This texture is similar to that reported by Schaaf et al. (2014) for unstable fatigue-crack propagation in PBT-GF30 and was described as micro-brittle. The PBT-GF10 TPEE surface has indications of microductile behavior on the brittle fracture zone, as seen in Fig. 3b.

3.3. Fiber pull-out and fiber-matrix bonding

The differences in fiber-matrix interfacial adherence could be also identified in Fig. 3. The matrix film on the fibers in PBT-GF10 was fairly thick (Fig. 3a). Fibers were covered with a layer of matrix, implying sufficiently high fiber-matrix bonding, and fiber pull-out was the dominant failure mechanism. The failure mainly occurred in the matrix and particularly in close vicinity of fiber-matrix interface, resulting in fiber pull-outs. On the other hand, in PBT-GF10 TPEE, the matrix layer on the fibers was very thin. Most of the fibers were white in the micrographs (Fig. 3b), pointing to a very weak fiber-matrix bonding. Since clear indications for fiber failure for both materials could not be found, it can be concluded that it was not the cause of the failure. Matrix failure was the dominant mechanism in both cases despite the differences in the interfacial matrix-fiber adhesion.

3.4. Effect of loading rate on microstructural behavior

To focus more on the ductile zone, or the area of stable crack initiation, the fractographs were analyzed using the software ImageJ. Specimens of two materials were analyzed for each loading rate and a fraction of the ductile area of each fracture type was calculated. The existence of transition areas between the ductile and brittle zones on the fracture surface introduced a calculation error of the ductile areas. The maximum error for all micrographs was 2% of the calculated area, and it did not affect the trends identified in this study. The analysis showed that for both materials the area of ductile behavior decreased with increasing loading rates; this trend is obvious in Fig. 4. The highest value was found at loading rate of 2 mm/min, decreasing to a minimum at 400 mm/min. For all loading rates it was noticed that the average fraction of ductile area for PBT-GF10 TPEE was greater than that of PBT-GF10. At 2 mm/min, a ductile area recorded for PBT-GF10 TPEE was 12 percentage points larger. Generally, in PBT-GF10 the ductile area was extremely small at 200 and 400 mm/min: the ductile area demonstrated a 1.8-fold increase at 2 mm/min, 1.7 at 20 mm/min, being in presence of TPEE in the PBT matrix 16 and 18 times larger at 200 and 400 mm/min, respectively.
3.5. Basic features of ductile area

Under higher magnification (Fig. 5a), conic-shaped structures formed as a result of matrix failure at bases of pulled or broken glass fibers are apparent. When the fiber started to pull out, the stress intensity around it increased. The volume occupied by the fiber acted like a cylindrical void, as the fiber was not attached to the matrix. The area around the conic structures is characterized by a fibrilar structure (Fig. 5b); Such a phenomenon was interpreted as indication of macro-crack propagation by Klimkeit et al. (2011).

Such structures were reported frequently in the literature. Horst and Spoormaker (1997) reported that this phenomena was observed only in ductile-to-brittle transition areas for glass-fiber reinforced polyamide specimens in fatigue and tension. Selvadurai (1995) and Selvadurai and Ten Busschen (1995) investigated matrix-fracture initiation at a fiber fracture in a single-fiber fragmentation test; they noticed conical and penny-shaped cracks or a combination of these two. To understand this phenomenon and its effect on formation of the ductile area, conic-structure areas where measured with ImageJ. (Fig. 5b) illustrates the method used to measure each single conic structure for all specimens loaded at all loading rates.

![Image](image_url)
Table 1 Median size of conic structures for PBT-GF10 TPEE

<table>
<thead>
<tr>
<th>Loading rate (mm/min)</th>
<th>Conic-structure size in (µm² x 10⁻³)</th>
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<tbody>
<tr>
<td>2</td>
<td>4.0</td>
</tr>
<tr>
<td>20</td>
<td>3.8</td>
</tr>
<tr>
<td>200</td>
<td>2.6</td>
</tr>
<tr>
<td>400</td>
<td>2.1</td>
</tr>
</tbody>
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When presented as cumulative distribution function, the results for sizes of conic structures demonstrate clear differences and pronounced trends. The normal standard distribution analysis shows that the probability of formation of larger conic structures decreases with increasing loading rates. Table 1 presents the mean size of a conic structure at different loading rates for PBT-GF10 TPEE.

4. Conclusions

Morphological analysis of fractures obtained from tensile tests of PBT-GF10 and PBT-GF10 TPEE specimens at different loading rates was implemented. The matrix material of PBT-GF10 TPEE behaved in a more ductile manner than PBT GF10. The investigation showed that the ductile area for both materials decreased with increasing loading rates; TPEE blended material had a bigger ductile area at all rates. This is a clear indication of a change in microstructure due to matrix composition. The size and shape of ductile areas show that fracture behavior was a result of blending with TPEE. Interfacial adhesion between the matrix and fibers were affected negatively by TPEE. The fractographs demonstrated that fiber pull-out was a dominant failure mechanism. Further analysis of the ductile area showed a higher probability of creation of larger conic structures at lower loading rates.

The results confirm that formation of the ductile area is a relatively slow process when compared to the rate at which the brittle area forms. When a stable crack growth dominates, the crack propagates much more rapidly through the specimen in a brittle manner, not allowing the matrix material to deform plastically and, therefore, leading to final failure of the specimen. These facts are supported by the decreasing trend of the ductile area at higher loading rates. PBT-GF10 TPEE showed that, even at higher loading rates, a measurable ductile area was available; this may explain the good impact properties of this material. The weak matrix-fiber bonding and the modified microstructure could affect tensile properties negatively; further investigations of this will be presented in elsewhere.

References


