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An experimental approach that assesses in-situ micro-scale damage mechanisms and fracture toughness in thermoplastic laminates under out-of-plane loading

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ABSTRACT

Studying the response of laminated composites under out-of-plane loading routinely involves mechanical tests, such as quasi-static indentation or impact. The phenomenology during these tests is so complex that it is difficult to identify different material properties related to each failure mechanism (damage mode). We aim at providing an experimental approach, which is practical and fast, for assessing the in-situ micro-scale damage mechanism and extracting the fracture toughness in thermoplastic laminates under out-of-plane loading. To this end, we developed a dedicated, micro-scale, three-point bending (micro-3PB) test fitted inside a scanning electron microscope (SEM). In a single experiment, we were able: (i) to assess the initiation of a transverse crack, the transverse crack-to-delamination transition, delamination growth, development of shear-induced microcracks during delamination, and fibrillation, and (ii) to evaluate the effective fracture toughness during transverse cracking and delamination under a representative out-of-plane loading. We used this approach to rank two types of glass fiber-reinforced polypropylene cross-ply laminates, i.e., based on either homopolymer PP (ductile matrix) and copolymer PP (less-ductile matrix), according to their relative fracture parameters. We also performed short edge notch bending (SENB), double cantilever beam (DCB) and end-notch flexure (ENF) to obtain the standard fracture toughness values. We found that the relative fracture toughness values obtained by SENB, DCB and ENF are comparable with that of micro-3PB results. Furthermore, ENF results showed that the delamination process during micro-3PB is dominated by Mode-II fracture.

1. Introduction

Continuous fiber-reinforced composites based on thermoset matrices have been conventionally used to build lightweight and durable load-bearing structures, such as pipelines, boat hulls, aircraft wings, and fuselages. Recently, thermoplastic-based composites have been increasingly used to produce load-bearing components due to their shorter production cycles, recyclability, improved environmental resistance, and ductility. Thermoplastic composites with improved ductility are particularly promising for impact-prone automotive structures, such as beam stickers, internal door supports, and roofs. These automotive applications call for a detailed understanding of the composite's response under out-of-plane loading.

Multidirectional laminates consisting of plies with various fiber orientations exhibit a complex damage phenomenology under out-of-plane loading. Damage typically starts in the plies that are transversely loaded in-the-plane, beginning with fiber/matrix debonding or microcracking in the bulk matrix and then growing to form transverse cracks [1–3]. Transverse cracks then initiate delamination between or within plies until final failure [4–7]. A key step in impact damage is the transition from transverse cracking to delamination. Delamination results from a concentration of stress at the interface due to out-of-plane shearing of existing transverse cracks. Most models have failed to represent the coupling between these two mechanisms correctly. Only models that explicitly introduce cracked surfaces in the micromechanical description [8] or that introduce non-local coupling between plies and interfaces at the meso-scale [9] have successfully captured this coupling. Even so, feeding these models with well-identified material parameters remains challenging.

Observation of damage and crack progression during out-of-plane testing of laminates can be challenging. Testing of plates under quasi-static indentation or impact does not allow the detailed damage

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mechanism to be monitored during the loading process. Several studies used a simplified test configuration based on a small beam-type specimen under 3- or 4-point bending, in which the fiber and matrix phases could be clearly observed under a scanning electron microscope (SEM) [10–14]. The specimen configuration enabled the tracking of damage and fracture at the fiber/matrix level and the validation of numerical models. Hobbiebrunken et al. [10] studied transverse cracks in a cross-ply of carbon/epoxy. From the numerical model, they determined that transverse cracks are essentially governed by the interfacial normal strength of the fiber/matrix. Likewise, Canal et al. [11] conducted similar tests on a glass/epoxy beam with notches. They used a finite element simulation of the transverse crack based on the embedded cell method, which revealed that the fracture toughness mainly depended on fiber/matrix interface properties rather than the bulk matrix properties.

Despite progress in this field, how micro-scale test results should be compared with standard fracture test results remains unclear. Certainly, the absolute fracture toughness values obtained by both strategies are expected to be different because the loading configurations and morphologies are different. Yet, an approach to compare the relative values of these strategies need s to be further investigated. Additionally, most micro-scale studies were conducted on thermoset-based composites, in which the effect of ductility and viscosity may not be comparable with that in thermoplastic-based composites. Furthermore, characterization of microscopic damage and fracture toughness of thermoplastic-based composites (e.g., on carbon fiber-reinforced PEEK [15–21] and on glass fiber-reinforced polypropylene [22–27]) was only conducted under post-mortem conditions, i.e. observing the fractured surfaces of the failed samples. In-situ observations of microscopic damage mechanisms involving multiple types of damage in thermoplastic composites have not been revealed in details.

In this work, we developed a three-point bending test setup for facilitating in-situ micro-scale observation with SEM. The setup allows the transverse cracks and delamination to be isolated and observed at micro-scale where damage features and fracture toughness of respective mechanisms of degradation can be estimated. We used the setup to study the damage behavior of continuous glass fiber-reinforced polypropylene composites made from either homopolymer or copolymer polypropylene. To better understand what should be the proper interpretation of the fracture toughness obtained by our three-point bending test setup, we carefully compared the relative quantities obtained in our tests with those of standard fracture tests, namely the single edge notch bending (SENB) test for intralaminar Mode-I fracture toughness, and the double cantilever beam (DCB) and end-notched flexure (ENF) tests for Mode-I and Mode-II interlaminar fracture toughness, respectively.

2. Experimental details

2.1. Materials and manufacture of laminates

We utilized a polypropylene (PP) matrix reinforced with continuous E-glass fibers. To show that our approach is able to rank two material grades with respect to their resilience to out-of-plane loading, we used two PP matrix systems, i.e. homopolymer PP and impact-modified PP (copolymer) designated as “GF-PP” and “GF-IPP”, respectively. We reported results on an investigation of GF-IPP earlier [28]. The materials were provided by SABIC in the form of unidirectional tape (0.25 mm thick and 110 mm wide). The fiber volume fractions of GF-PP and GF-IPP were 45% and 41%, respectively. We used a static hot press (Pinette Emidecau Industries 15T) to manufacture GF-PP and GF-IPP laminates. We stacked layers of tape, and inserted them into a custom-made metallic mold (aluminum mold for GF-PP and steel mold for GF-IPP). The consolidation cycle for GF-PP was as follows: under a constant pressure of 6.8 bar, the laminate was heated to 230 °C and dwelled there for 20 min; the temperature was then reduced to room temperature at a rate of 22 °C/min. The consolidation cycle for GF-IPP was similar to that of GF-PP but with constant pressure and dwelling temperature of 7.5 bar and 210 °C, respectively. The dimension of the GF-PP plate was 250 × 110 mm^2, while that of GF-IPP was 275 × 110 mm^2.

2.2. Standard fracture test methods

We evaluated Mode-I intralaminar fracture toughness of glass/polypropylene laminates using the single-edge-notch bending (SENB) test following ASTM D-5045 Standard [29]. Here, SENB intralaminar fracture toughness is measured for a transverse crack growing in the thickness direction, to be distinguished from intralaminar fracture toughness based on the transverse crack growing along the fiber direction [30]. The sample lay-up was $[0]_{30}$ and the dimension was 36 × 7.5 × 4 mm^3. A notch of 3.75 mm was introduced in the middle of the sample using a diamond cutting saw. Then, a razor blade was manually tapped into the notch to create an initial crack of 0.5 mm. We verified that these procedures were giving a consistent dimension among samples. We performed the SENB test using a custom-made three-point bending (3PB) setup installed in the micro tensile/compression machine (Kammrath & Weiss) with a 5-kN load-cell. The loading speed was 1.2 mm/min. We measured the energy derived from the integration of force-displacement curve and considered it as the Mode-I fracture toughness. The integration process was made up to a critical point, which is the maximum force if it falls within 1.00–1.05 C (C is the sample's elastic compliance). Otherwise, the critical load is taken as the load value precisely at 1.05 C. We also included a correction for specimen compression and system compliance as well as pin indentation at the loading point [29].

Mode-I interlaminar fracture toughness of glass/polypropylene was evaluated using the double cantilever beam (DCB) test following ASTM D-5528 Standard [31]. We prepared $[0]_{16}$ samples (240 × 20 × 4 mm^3) containing an initial delamination of 60 mm length made by inserting a non-adhesive tape (70 micron thick) into the specimen midplane. A pair of loading blocks were bonded to one end of the specimen and was connected to the load cell. We used an Instron 5882 (500 N load cell) to perform the DCB test with a loading speed of 5 mm/min. Before testing, we used a razor blade to slightly open the delamination up to 10 mm, and then used Instron's load cell to fully open the delamination up to 60 mm. Once the initial delamination has been fully opened, the mechanical loading was applied and the force-displacement curve was recorded. The delamination growth on the specimen edge (containing scales) was captured using PCO SensiCam camera with a frame rate of 0.165 Hz and 0.330 Hz for GF-PP and GF-IPP, respectively. We used higher frame rate for GF-IPP because the delamination growth in GF-IPP was relatively faster than that in GF-PP. Mode-I fracture toughness (G_c) was calculated using a Modified Beam Theory (MBT) described in [31]. We calculated G_c-initiation at the onset where the delamination started to propagate (pop-in) and was observable in the optical images. G_c-propagation was calculated as an average G_c value in the plateau regime of the R-curve (when G_c value has been stabilized) over a range of delamination length. The range of delamination length was 80–230 mm and 120–225 mm for GF-IPP and GF-PP, respectively. The stable delamination growth in the GF-PP takes longer time than that in GF-IPP due to a more extensive fiber bridging.

Mode-II interlaminar fracture toughness was evaluated using the end-notched flexure (ENF) test based on ASTM D-7905 Standard [32]. The specimen specification used in ENF test was exactly the same as the one used in DCB test: $[0]_{16}$ lay-up, 240 × 20 × 4 mm^3, initial delamination of 60 mm via a non-adhesive tape. The ENF test was conducted using a three-point bending (3PB) setup in an Instron 5882 (10 kN load cell) at the loading speed of 0.5 mm/min. Mode-II fracture toughness was calculated using Compliance Calibration (CC) method where the maximum load was considered as a critical load [32]. In CC method, we first positioned one of the 3PB supporting pins at three distances sequentially, i.e. 20 mm, 40 mm and 30 mm, while we recorded the force-displacement curves. We first positioned one of the supporting pins 20-