Microstructure and fracture properties of glass fiber-reinforced polyamide 66 with high volume fraction of glass fibers

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Abstract
This paper presents the experimental results for the fracture properties of injection-molded glass fiber/polyamide 66 composites. A weighed amount of glass fibers was mixed with polyamide 66 using a twin extruder and the amount of glass fibers was varied from 17 to 41vol%. Two types of single-edge notched bend (SENB) specimens were used to evaluate the fracture properties of the composites. The C specimens were cut from the injection-molded plates in three different directions: parallel to the flow direction (C-0 specimens), normal to the flow direction (C-90 specimens), and 45°to the flow direction (C-45 specimens). The injection molded specimens (M specimens) were also prepared using a metal mold. Three point bending tests were carried out on the SENB specimens. The effects of the specimen orientation, fiber volume fraction and loading rate (cross-head speed) on the fracture properties were investigated. In addition, the crack extension behavior of the composites was examined by an optical microscope using dyed specimens and the microstructures of the fracture surfaces of the composites were observed by a scanning electron microscope (SEM).

1. Introduction

Because of their light weight and attractive mechanical properties, glass fiber/polyamide 66 composites are potential materials for fastening products. However, very limited information has been reported for injection-molded glass fiber/polyamide 66 composites with a high volume fraction of glass fibers, which are potentially suitable for fastening products.

Various approaches and methods have been used to study the fracture behavior of different types of molded composite materials. It is commonly known that molded composite materials have layers with different fiber orientations which affect their melt flow behavior. Sankaran and Mallick investigated the effect of injection molding conditions on the mechanical behavior of 18 vol% glass fiber/polyamide 66 composites including layers with different fiber orientations [1]. Horst and Spoormaker also used a similar approach to investigate 17 vol% glass fiber/ polyamide 6 composites [2]. Friedrich evaluated the stress intensity factor of the various polymer composites [3]. Sanada and Shindo estimated J integral values of woven glass-epoxy laminates under three point bending at cryogenic temperatures[4]. Moreover, Lhymn and Shultz calculated the strain energy using the fiber fracture energy, fiber debonding

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energy, fiber pull-out energy and matrix fracture energy [5]. On the other hand, He et al. carried out fracture toughness tests on double-edge notched tension specimens and investigated the effect of the ligament length and fiber orientation on the fracture properties of 10 vol% glass fiber/polyethylene composites [6]. Mutou et al. performed the fracture toughness tests on single-edge notched bend (SENB) specimens and investigated the effect of the loading rate and fiber orientation on the fracture behavior of 15vol% glass fiber/polyamide 66 composites [7]. However, the investigation of injection-molded polymer composites with a high volume fraction of glass fibers has been very limited.

The aim of this work is to investigate the fracture properties of injection-molded glass fiber/polyamide 66 composites with various fiber volume fractions and fiber orientations. Three-point bending tests were carried out on SENB specimens. In addition, the microstructure of the fracture surfaces of the composites was examined by an optical microscope and a scanning electron microscope (SEM).

2. Experimental procedure

2.1. Materials and specimens

The polyamide 66 (Leona 1300S) used as a matrix material in this study was a commercial product supplied in pellet form by Asahi Kasei Chemicals Company, Japan. The chopped glass fibers (CS3J459) used as a reinforcing material, which had 11μm diameter and 3mm length, were also a commercial product supplied by Nittobo Company, Japan. The densities of the polyamide 66 and glass fibers were 1.2 and 2.6g/cm^3, respectively. A weighed amount of glass fibers was mixed with polyamide 66 using a twin extruder and the amount of glass fibers was varied from 17 to 41 vol%.

The geometry of the SENB specimens is illustrated in Figure 1. The geometry was in accordance with the specifications outlined in ASTM E 1820-01 [8]. The notch was made using a cutter with a chevron shape. Specimens were prepared by cutting injection-molded plates in three different directions: parallel to the flow direction (C-0 specimens), normal to the flow direction (C-90 specimens), and 45° to the flow direction (C-45 specimens). The specimen-cutting directions of the injection-moded plates are shown in Figure 2. The arrows in Figure 2 show the flow directions of the melted composites. Each injection-molded plate had a gate of 1mm thickness between the runner part and the product part. Generally, a gate is used to ensure easy separation of a product from a runner when the die is opened. The gate results in microstructure layers with different fiber orientations because it has a significant effect on the melt viscosity of the composites which affects the fiber orientation. Injection-molded specimens (M specimens) cut from injection-molded tensile specimens were also used. The layout of the M specimen is shown in Figure 3. The arrows in Figure 3 show the flow directions of the melted composites. The dumbbell-shaped die did not have a gate. Table 1 gives the conditions of injection molding.

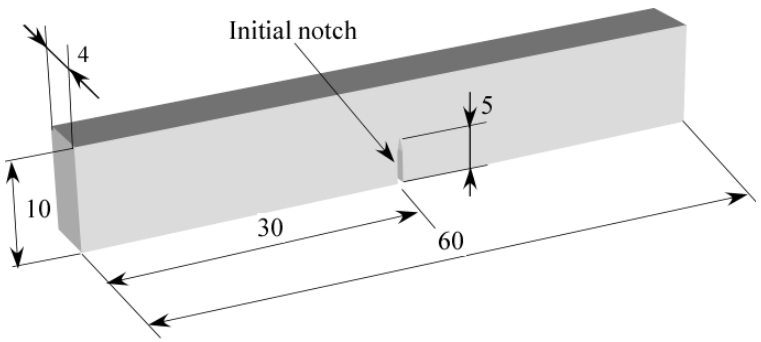

Figure 1. Geometry of specimens used for fracture toughness tests (dimensions in mm).

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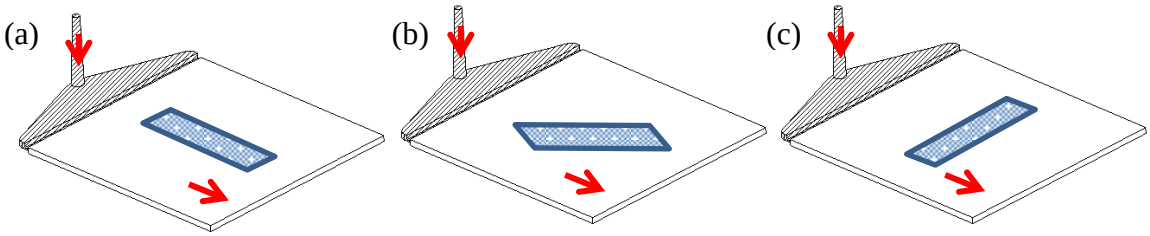

Figure 2. Directions of specimens cut from the injection-molded plates: (a) parallel to the flow direction (C-0 specimens); (b) 45° to the flow direction (C-45 specimens); (c) normal to the flow direction (C-90 specimens).

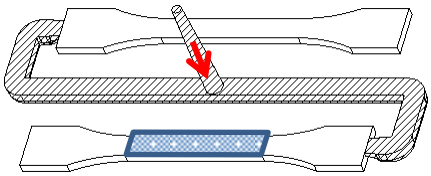

Figure 3. Layout of M specimen cut from the tensile specimens.

Table 1. Conditions of injection molding.

Molding Machine	FNX140 (clamp capacity 140t)
Desiccating condition	80℃/12h (in a vacuum)
Melt Temperature	280-300℃
Mold Temperature	120-140℃
Injection Speed	40mm/s
Holding Pressure	50MPa

2.2. Test methods

Fracture toughness tests were performed at room temperature using a universal testing machine (Instron 55R-4206) in accordance with ASTM E1820-01 [8] at a cross-head speeds of 0.4 and 2.0mm/min. During the fracture toughness tests, the applied load and load-line displacement were continuously recorded with the load-line displacement of the specimens defined on the basis of the cross-head displacement. The strain energy at the maximum load were evaluated using load versus load-line displacement curves. The elastic and plastic parts of the area were separated by considering the strain energy at the maximum load as shown in Figure 4.

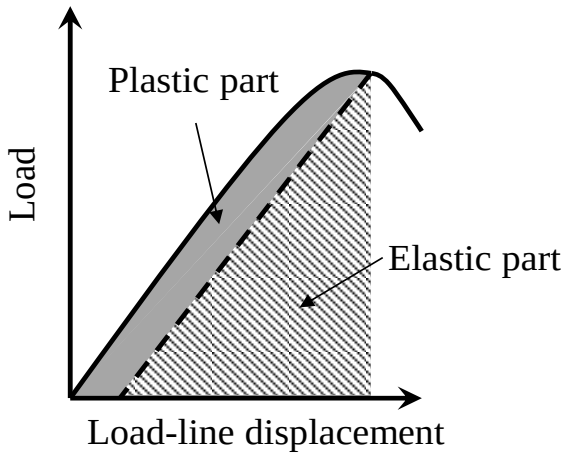

Figure 4. Definition of the area separated into elastic and plastic parts

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Optical microscope and SEM examinations were carried out on the fracture surfaces of the specimens subjected to fracture toughness tests. In the optical microscope examination, the specimens were dyed with an acid dyestuff (Erionyl Red A-2BF) by dipping in water 100℃ for 30min. Before the SEM examination, the specimens were sputtered with a platinum coating using a Hitachi E102 Ion Sputter surface-coating machine and were viewed with a Hitachi S-3400N SEM.

3. Results and discussion

Figure 5 shows load versus load-line displacement curves of the M, C-0, and C-90 specimens with 41vol% glass fibers. The load versus load-line displacement curves for the C-0 and C-90 specimens were similer, despite the different cutting directions. The maximum load of the M specimens was largest in this test. This is attributed to the greater number of glass fibers transverse to the loading direction.

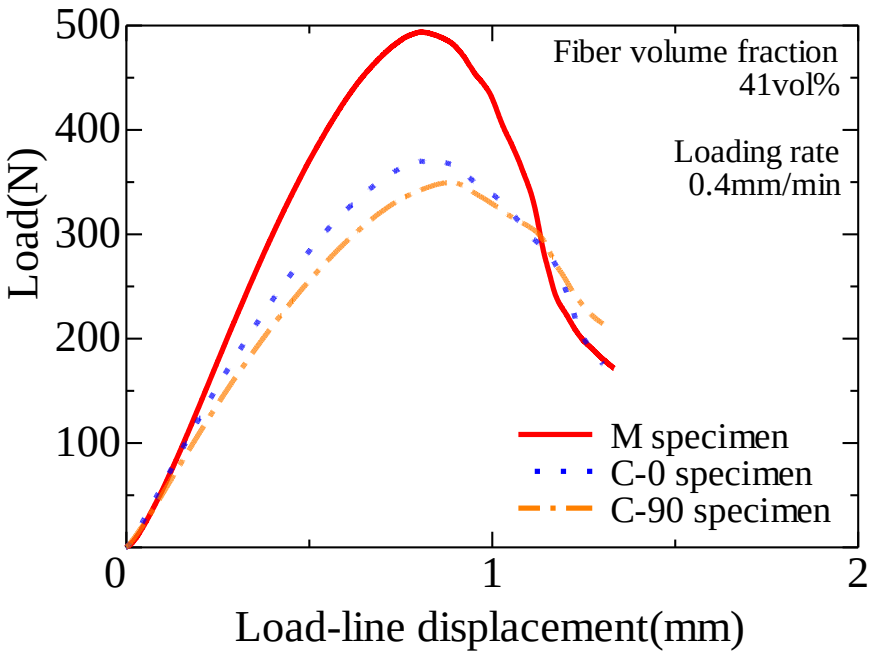

Figure 5. Load vs load-line displacement curves of the M, C-0, and C-90 specimens

Figure 6 shows the strain energy at the maximum load in the three point bending test of the SENB specimens for the various specimen cutting directions and loading rates. The strain energy of the SENB specimens at the maximum load was similar for each cutting direction. However, when the loading rate was increased from 0.4 to 2.0mm/min, the elastic part of the strain energy increased and the plastic part decreased.

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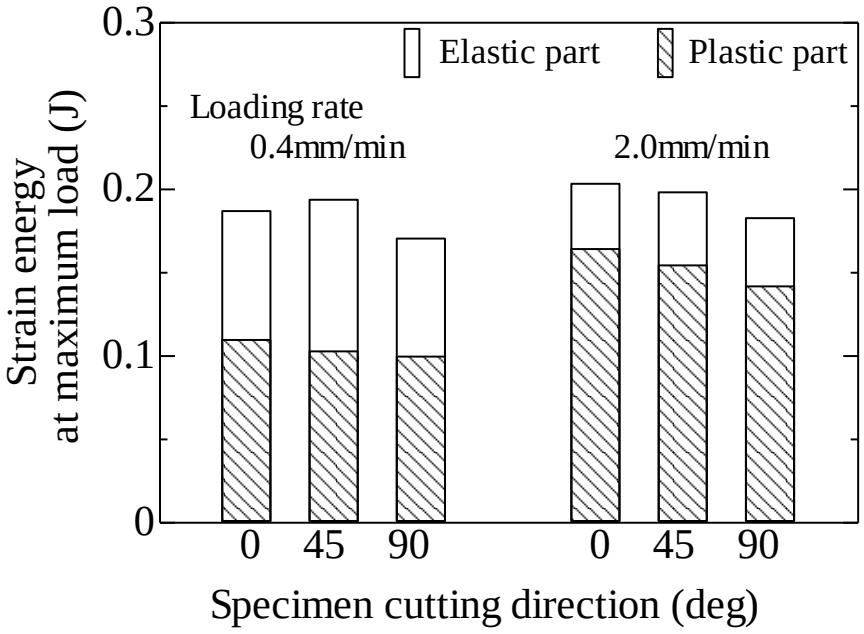
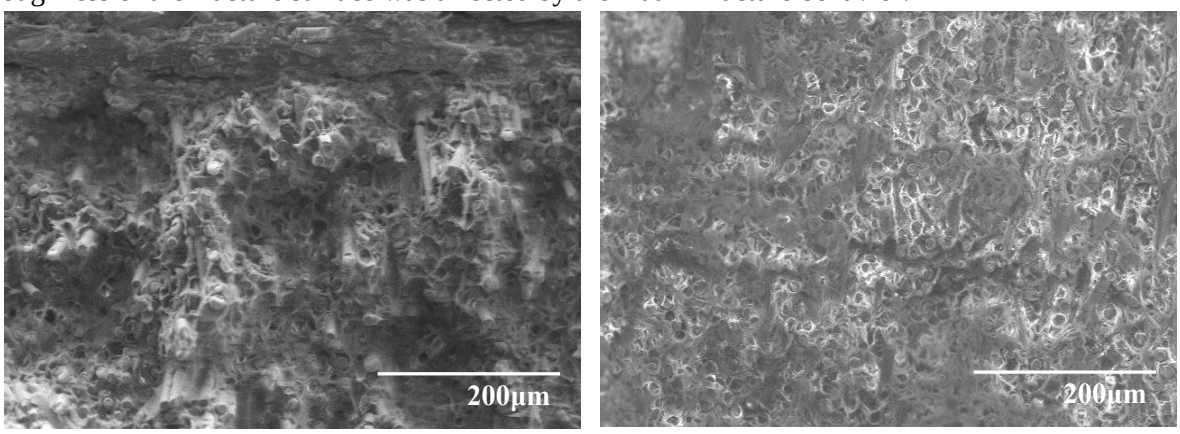


Figure 6. Effect of specimen cutting direction and loading rate on the strain energy at the maximum load for the SENB specimens with 41vol% glass fibers.

Figure 7 shows SEM images of the fracture surfaces of the C-0 specimens with 41 vol% glass fibers after the fracture toughness test with the two loading rates. Hardly any holes generated by the pulling out of fibers were observed whereas fiber breakage was observed on the fracture surface of each specimen. Moreover, the fracture surface was rougher for the loading rate of 0.4 mm/min. Thus, the roughness of the fracture surface was affected by the matrix fracture behavior.



(a) (b)

Figure 7. SEM images of fracture surfaces of C-0 specimens with 41 vol% glass fibers:
(a) 0.4mm/min loading rate; (b) 2.0mm/min loading rate.

Figure 8 shows the crack propagation on the fracture surface of a C-0 specimen with 41 vol% glass fibers after the fracture toughness test. Crack occurred in the region with the darker color. The amount of crack propagation in the core layer with the fiber orientation parallel to the crack propagation direction was less than that of the skin layer with the fiber orientation normal to the crack propagation direction. This may be due to the large plastic deformation of the matrix in the core layer.

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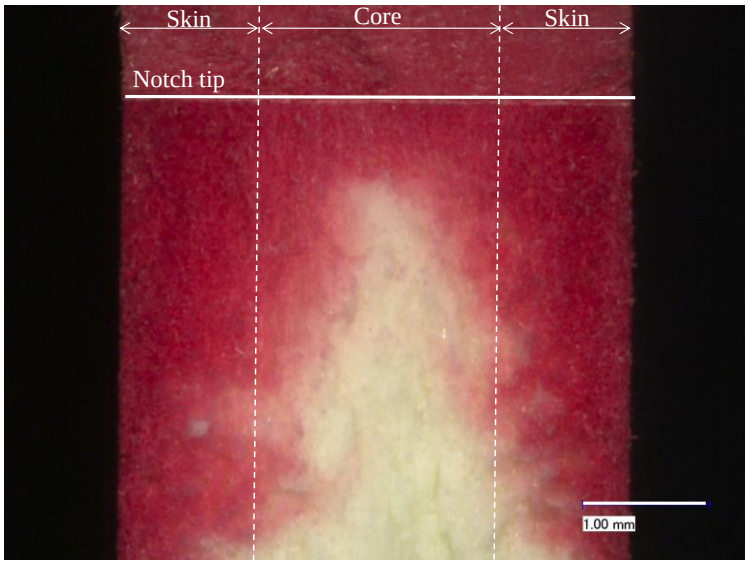

Figure 8. Photograph of the fracture surface of a C-0 specimen with 41 vol% glass fibers.

3. Conclusions

The fracture properties of glass fiber/polyamide 66 composites with various fiber orientations and fiber volume fractions were investigated. The fracture toughness tests were carried out on SENB specimens under loading rates of 0.4 and 2.0 mm/min. The results can be summarized as follows.

- The strain energy at the maximum load was similar for each fibers orientation.
- When the loading rate was increased from 0.4 to 2.0mm/min, the elastic part of the strain energy increased and the plastic part decreased.
- When the loading rate was increased from 0.4 to 2.0mm/min, the roughness of the fracture surface decreased.

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