

## Fracture Toughness of Carbon Nanofiber Reinforced Polylactic Acid at Room and Elevated Temperatures

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### Summary

Poly(lactic acid) (PLA) is a biodegradable plastic made from lactic acid, and can be produced by renewable raw materials. The mechanical properties of PLA are, however, not sufficient for structural materials. In our study, carbon nanofiber reinforced PLA was fabricated to overcome the deficiency of PLA and the mechanical properties were measured at room and elevated temperatures. Vapor grown carbon fiber (VGCF) was used for reinforcement. Three point bending specimens were fabricated by using injection molding, and then bending stiffness, bending strength and fracture toughness were measured for amorphous and crystallized specimens. As a result, it is shown that the bending stiffness increases with increasing the weight fraction of VGCF, but the addition of VGCF does not affect the bending strength and fracture toughness.

### Introduction

Poly(lactic acid) (PLA) is a biodegradable plastic made from lactic acid, and can be produced by renewable raw materials. In addition, PLA has better mechanical properties and commercial availability than those of other biodegradable plastics. The mechanical properties of PLA are, however, not sufficient for structural materials. Polymers filled with carbon nanofibers are widely fabricated to enhance the mechanical and other properties [1, 2]. For example, PC [3], PS [4], PP, PA, PEEK [5], PI [6], UP [7] and Epoxy [8] based carbon-nanotube composites have been investigated. Authors [9] have reported bending strength, stiffness and heat deflection temperature of carbon nanofiber reinforced amorphous PLA at room temperature. Enomoto et al. [10] reported crystallization behavior of PLA with VGCF.

In our study, mechanical properties of carbon nanofiber reinforced PLA at room and elevated temperatures were investigated. Vapor grown carbon fiber (VGCF) was used for reinforcement, which has 150 nm in diameter and 10  $\mu$ m in length. No surface treatment of VGCF was conducted. The weight fraction of VGCF ranged from 1wt% to 10wt%. Three point bending specimens were fabricated by using injection molding, and then bending stiffness, bending strength and fracture toughness were measured for amorphous and crystallized specimens. As a result, it is shown that the bending stiffness increases with increasing the weight fraction of

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### Specimens

For matrix, polylactic acid (PLA) (Lacty #9000, Shimadzu Corporation [11]) was used. The melting point is approximately 160°C. For reinforcement, Vapor grown carbon fiber (VGCF, Showa Denko K.K.) was used. The diameter is approximately 150nm and the length is approximately 10 $\mu$ m. No surface treatment was conducted. A twin screw extrusion machine (KZQ15TW-30MG-NH-700, Technovel Corporation) was used to compound VGCF into PLA, and then the compound was pelletized. Before injection molding, the pellets were dried for 5 hr at 100°C.

Three point bending specimens were molded by using a small injection molding machine whose injection pressure was applied by hand (Shinko Sellbic Co., Ltd.). The mold temperature was 45°C. The specimen size was 60 mm long, 10 mm wide and 3 mm thick. In order to investigate the effect of the weight fraction of VGCF on mechanical properties, specimens with 0wt%, 1wt%, 5wt% and 10wt% of VGCF were prepared. In addition, to examine the effect of crystallinity, annealed specimens at 130 °C for 3hr were also prepared. The crystallinity is supposed to be 60% [10].

Injection-specimens are shown in Fig.1. As-casted neat PLA specimens were transparent, which means amorphous. All composite specimens looked black because of carbon nanofibers.

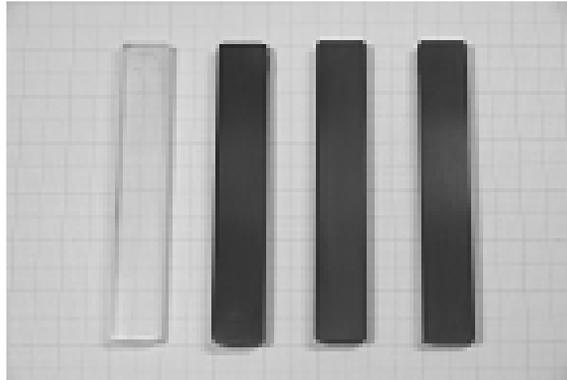


Figure 1: Inject-molded specimens (Neat PLA, 1wt%, 5wt%, 10wt%)

### Three Point Bending Test

Three point bending tests in air at 20°C and 50°C were conducted based on JIS K 7171. The cross-head speed was 1mm/min. Figure 2 shows the bending stiffness and Fig. 3 shows the bending strength. ▲ represents the results of amorphous spec-

imens at 20°C,  $\blacklozenge$  represents the results of annealed specimens at 20°C,  $\blacktriangle$  represents the results of amorphous specimens at 50°C,  $\blacklozenge$  represents the results of annealed specimens at 50°C.

The bending stiffness of all specimens increased with increasing the weight fraction of VGCF. The degree of increase is, however, approximately 5% of estimated values by using the shear-lag theory. This implies that the stress transition through interface between matrix and fiber is imperfect. In addition, stress transition between carbon layers in VGCF may not be enough to enhance the stiffness. The bending stiffness of annealed specimens is higher than that of amorphous specimens at the same ambient temperature because crystallization increases Young's modulus of PLA. The bending strength at 20°C slightly decreased as increasing the weight fraction of VGCF. The bending strength at 50°C, however, was almost constant regardless of the weight fraction of VGCF.

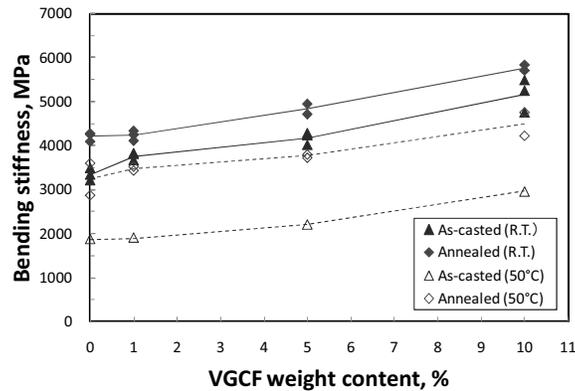


Figure 2: Bending stiffness

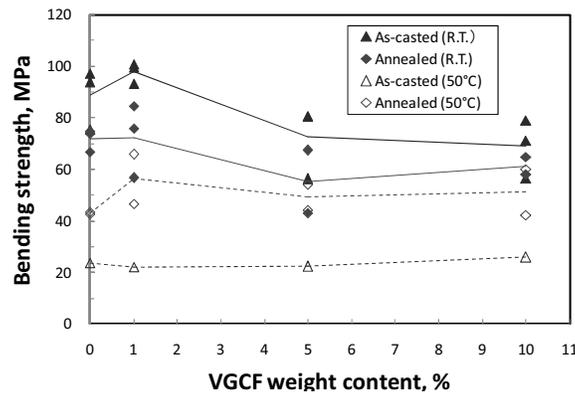


Figure 3: Bending strength

### Fracture Toughness Test

Fracture toughness in air at 20°C and 50°C was measured based on ASTM D 5045. The cross-head speed was 1mm/min. The specimen configuration is shown in Fig.4. Since several specimens did not satisfy the plain-strain criteria,  $K_Q$  was used as fracture toughness.

Figure 5 shows the results. Since amorphous specimens at 50°C had no crack propagation and showed only plastic deformation, the fracture toughness could not be measured. The figure shows that the effect of VGCF addition on fracture toughness is small.

Figure 6 shows the typical fracture surface. Pull-out of VGCF is clearly seen. As we mentioned, the efficiency of modulus enhancement was low, and both the bending strength and the fracture toughness were not affected by the addition of VGCF. These results imply that the interface strength and the friction force between VGCF and PLA is significantly low.

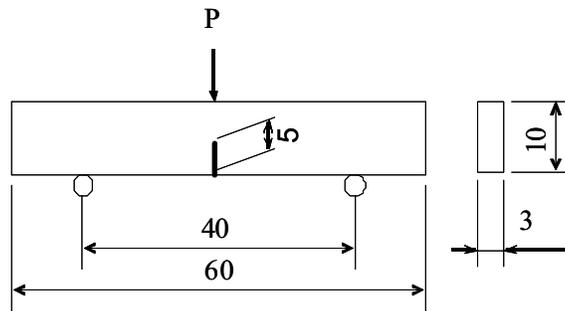


Figure 4: Fracture toughness specimen

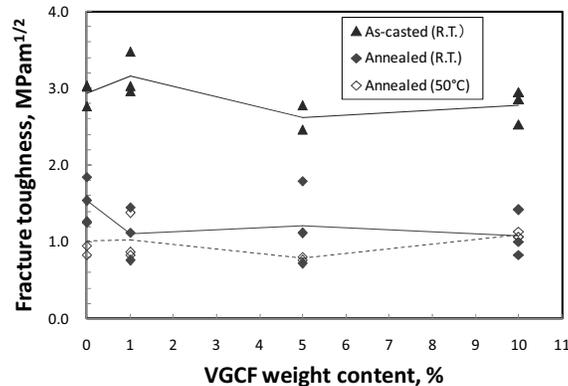


Figure 5: Fracture toughness

### Conclusions

VGCF/PLA composite specimens were fabricated and the bending stiffness,

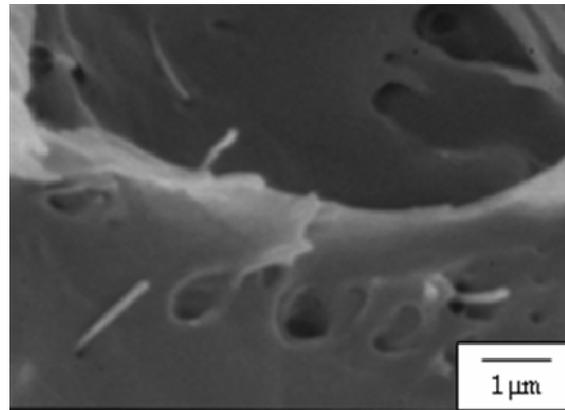


Figure 6: Fracture surface of amorphous composite specimen (10wt%)

bending strength and fracture toughness were measured at room and elevated temperature. As a result, it is shown that the bending stiffness increases with increasing the weight fraction of VGCF, but the addition of VGCF does not affect the bending strength and fracture toughness at least up to 10wt% of VGCF.

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