EFFECT OF THE CNT GROWTH TEMPERATURE ON THE TENSILE STRENGTH OF CARBON FIBER

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ABSTRACT

Carbon fiber reinforced plastics (CFRPs) are expected to be used for the structural parts of automobiles and aircrafts due to their mechanical properties, including high specific stiffness and high specific strength. It was reported that grafting of carbon nanotubes (CNTs) on the carbon fiber can improve the fiber matrix interfacial property, which plays an important role in the mechanical behavior of CFRP. On the other hand, grafting CNTs using the chemical vapor deposition (CVD) method sometimes weakens the tensile strengths of carbon fibers. In particular, when the growth temperature is relatively high, the fiber degradation is remarkable. In this study, CNTs were grafted on carbon fibers at growth temperatures from 550 to 700°C using Ni as the catalyst, and their tensile strengths were measured by single fiber tensile tests. The results indicated that the CVD processes, especially the higher temperature heat-treatment, accelerate the diffusion of catalysts into carbon fibers, and degrade the fiber strength. When the growth temperature was relatively low (550–600°C), there was no degradation of tensile strength for CNT grafted carbon fibers.

Keywords: CFRP, Carbon Nanotube (CNT), Chemical Vapor Deposition (CVD), CNT grafted carbon fiber, single fiber tensile test, tensile strength

1 INTRODUCTION

In recent year, Carbon fiber reinforced plastics (CFRPs) are expected to be used for the structural parts of automobiles and aircrafts due to their mechanical properties including high stiffness, high strength and low density. The mechanical behavior of composites depends not only on the properties of the reinforcing fibers, but also on the characteristics of the interface between the fiber and matrix. The fiber matrix interface is responsible for stress transfer from the matrix to the carbon fibers. It was reported that grafting of carbon nanotubes (CNTs) on the carbon fiber can improve the fiber matrix interfacial shear strength (IFSS) due to the increase of the surface area and mechanical interlocking by CNTs [1], [2].

CNTs are graphene sheets in the shape of a tube whose diameter is on the nanometer order. With their small size and remarkable mechanical, electrical and thermal properties, CNTs have been considered as promising materials to use in advanced composites [3]. Most CNTs are produced by carbon-arc discharge, laser ablation, and chemical vapor deposition (CVD). Of these, CVD provides a typical method to graft CNTs directly onto carbon fibers [3]–[5]. On the other hand, grafting CNTs using the CVD method sometimes weakens the strength of carbon fibers. In the previous studies, CNTs were grafted onto carbon fiber at over 700°C and the tensile strength of CNT grafted carbon fiber was decreased [2], [6], [7].

In this paper, we focused on the effect of growth temperature on fiber degradation. CNTs were grafted on carbon fibers at growth temperatures from 550°C to 700°C using Ni as the catalyst and ethanol as the carbon source. The tensile strength of Ni plated and CNT grafted carbon fibers were measured by single fiber tensile tests.

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2 MATERIALS AND EXPERIMENTAL PROCEDURES

2.1 Materials

Spread PAN-based carbon fibers for 24K (Nippon Tokushu Fabric) were used in this study. Ni was carried onto the surface of carbon fibers by electrolytic Ni plating method. The components of plating bath were Nickel sulfate hexahydrate (240 g/L), Nickel chloride hexahydrate (45 g/L) and Boracic acid (30 g/L). The fiber bundles were connected on one end side to the electrode and dipped in the plating bath. The plating time is set for 15s and the current density is set for 0.13mA/mm^2. The morphology of the surface of carbon fibers was examined by scanning electron microscope (SEM, JSM-6390LT, JEOL) and field emission scanning electron microscope (FE-SEM, JSM-7500FD, JEOL). To evaluate the effect of the heat treatment on the reaction of Ni to carbon fibers, Ni plated carbon fibers were heat-treated at 600°C and 700°C for 30 min. Before and after the heat treatment, the size of Ni particles in 1 μm^2 was measured by SEM.

2.2 Growth of CNTs on carbon fibers

CNTs were directly grafted onto carbon fibers by CVD system (MPCVD-70, Microphase) at 550˚C, 600˚C, 650˚C and 700˚C with a flow of ethanol at a flow rate of 2mL/min as the carbon source to grow CNTs. The spread carbon fiber bundles on a ceramic tray were placed into a quartz tube. After purging with Ar gas at the flow rate of 200mL/min, the temperature of quartz tube was raised to the growth temperature and ethanol gas was injected for 30 mins. After exposure to ethanol gases, the tube and carbon fibers were cooled down to room temperature under flowing Ar.

2.3 Single fiber tensile test

The tensile strengths of as-received carbon fiber, heat-treated carbon fibers, heat-treated Ni plated carbon fibers and CNT grafted carbon fibers were measured by single fiber tensile test. A single filament was separated from the spread fiber bundles and attached to the film with a gauge length of 15mm, as show in Fig. 1. Single fiber tensile tests were conducted by micro-material testing machine (MMT, MMT-11N, Shimadzu) with a 2.5N load cell and displaced at an extension rate of 1.67×10^{-5} m/s (1mm/min). The diameter of the carbon fiber was measured by SEM after each tensile test. Ten single fibers were tested for each condition.

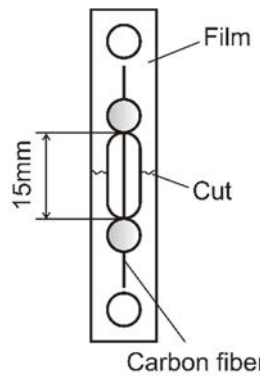


Figure 1: Schematic drawing of tensile test specimen.

3 RESULTS AND DISCUSSIONS

3.1 Effect of heat-treatment on Ni particles

Fig. 2 shows the surface of Ni plated carbon fiber. Ni exists on the carbon fiber surface in a state of particle. Ni plated carbon fibers heat-treated at 600˚C and 700˚C are shown in Fig. 3 and measured Ni particle size is shown in Fig. 4. While Ni particles size of heat-treated Ni plated carbon fibers at 600°C is same as that of untreated one, that at 700°C has smaller Ni particle size. This indicates that the diffusing of Ni particles into carbon fibers occurred over 600°C.

3.2 Results of single fiber tensile strength

Fig. 5 shows CNT grafted carbon fibers at growth temperature of 550°C, 600°C, 650°C and 700°C. While the carbon fiber at growth temperature of 550˚C was coated with short CNTs, longer CNTs, about micro-meter order, are grafted on the carbon fibers over 600˚C for growth temperature.

The results of single fiber tensile tests for heat-treated carbon fibers, heat-treated Ni plated carbon fibers and CNT grafted carbon fibers are respectively represented in Figs. 6, 7 and 8. Carbon fibers without Ni catalyst did not show any decrease in tensile strength under all conditions. On the other hand, the degradation of the tensile strength can be seen for the 650°C and 700°C conditions in both Figs 7 and 8. At 650°C, the tensile strength of Ni plated fiber and CNT grafted fiber showed 4.0 GPa and 4.6 GPa, decreases of 21% and 10% as compared to as-received fiber (5.1 MPa), respectively. At 700°C, the tensile strength of Ni plated fiber and CNT grafted fiber were 48% and 45% lower than that of as-received fiber. As shown in Fig. 4, the diffusion of Ni particles into carbon fibers is indicated to occur over 600°C for heat-treatment. This significant degradation of tensile strength is considered to come from the diffusing of Ni into carbon fiber. The strength of CNT grafted carbon fibers is strongly influenced by the interaction of Ni particle with the carbon fiber.

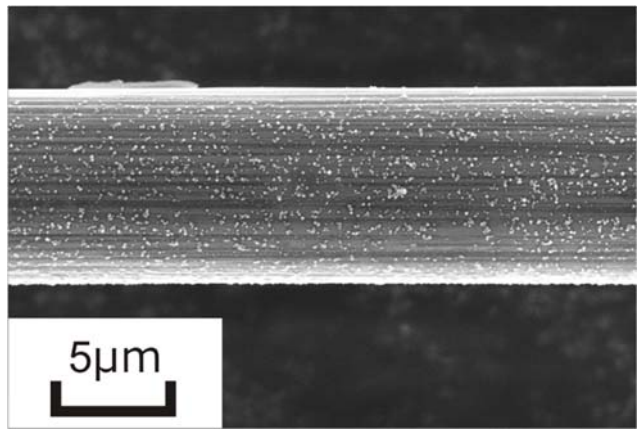
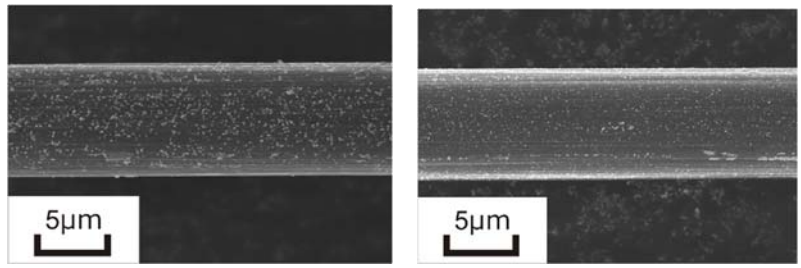


Figure 2: SEM images of the Ni plated carbon fiber.



(a) 600˚C (b) 700˚C

Figure 3: SEM images of Ni plated carbon fibers heat-treated at 600°C and 700°C.

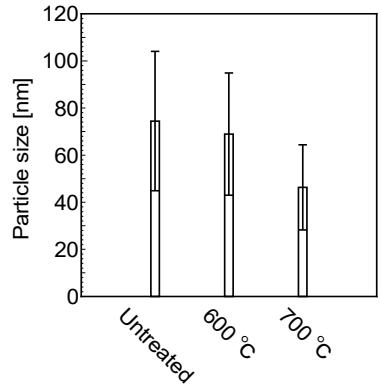
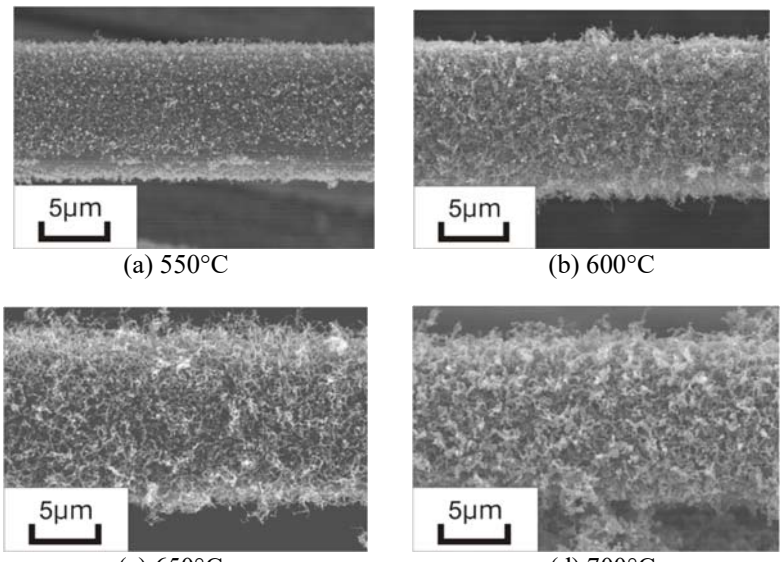


Figure 4: Ni particle size on the surface of carbon fibers heat-treated at 600°C and 700°C.



(a) 550°C (b) 600°C

(c) 650°C (d) 700°C

Figure 5: CNT grafted carbon fibers at growth temperature of 550°C, 600°C, 650°C and 700°C.

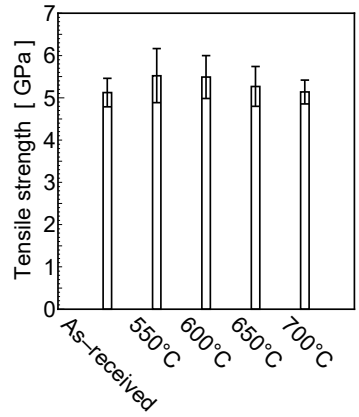


Figure 6: Tensile strengths of heat-treated carbon fibers at different temperatures.

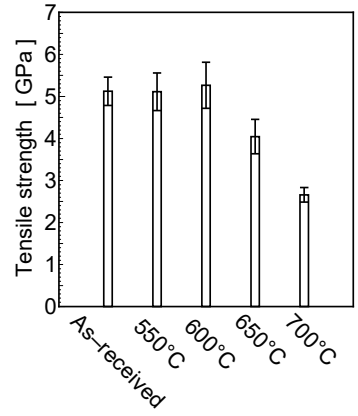


Figure 7: Tensile strengths of Ni plated carbon fibers heat-treated at different temperatures.

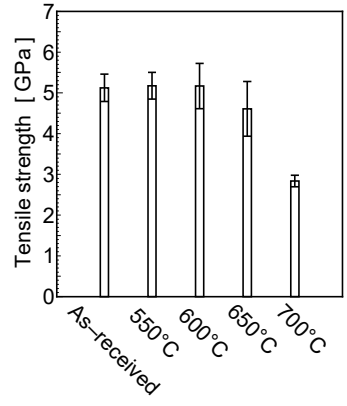


Figure 8: Tensile strengths of CNT grafted carbon fibers produced with the CVD process at different growth temperatures.

Weibull plots of tensile strength for Ni plated carbon fibers and CNT grafted carbon fibers are shown in Figs. 9 and 10. Several studies reported that the growth of CNTs on carbon fiber improves the tensile strength of carbon fiber as a result of increasing of cross-links between carbon crystals and repairing the damages of fiber surface by the growth of CNTs [7], [8]. At 650°C, some of damages caused by diffusing of Ni particles are considered to be repaired by growth of CNT.

4 CONCLUSIONS

CNTs were grafted on carbon fibers at growth temperatures from 550˚C to 700˚C using Ni as the catalyst and ethanol as the carbon source. The tensile strength of Ni plated and CNT grafted carbon fibers were measured by single fiber tensile tests. The investigation yielded the following conclusions:

The size of Ni particles on the surface of carbon fibers was decreased by the heat treatment at 700°C. This indicated that the heat-treatment accelerates the diffusion of Ni particles into carbon fibers.

There was no degradation of tensile strength for CNT grafted carbon fibers at the temperature of 550°C and 600°C. The tensile strength of Ni plated and CNT grafted carbon fibers heat-treated at over 650°C was decreased by the fiber degradation due to the diffusion of catalysts into fibers.

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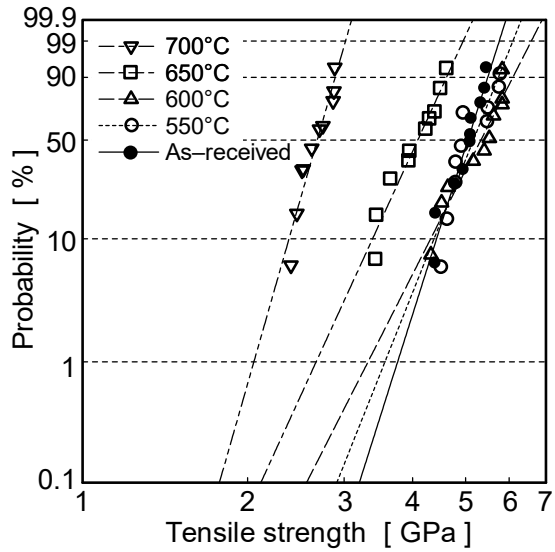

Figure 9: Weibull plots for as-received carbon fibers and Ni plated carbon fibers heat-treated at different temperatures.

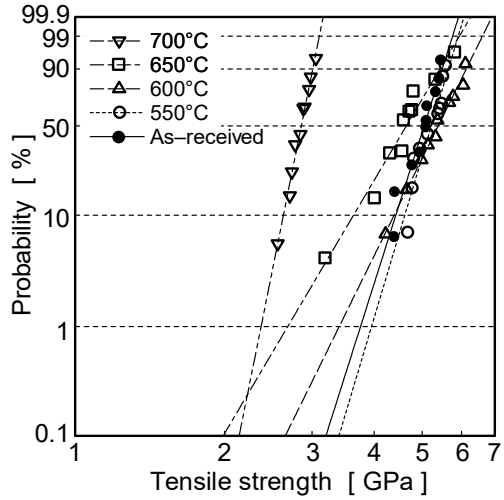

Figure 10: Weibull plots for as-received carbon fibers and CNT grafted carbon fibers produced with the CVD process at different growth temperatures.

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