Effect of heating rate on the properties of a graphite/phenolic-based composite

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Abstract. Graphite/phenolic-based composite was fabricated through hot compression molding followed by heat-treatment process. The length, density, flexural strength and electrical conductivity of composite are analyzed to determine the influences of heating rate on the physical, mechanical and electrical properties of composite. It is found that the shrinking in length of composites heat-treated at low heating rate was larger than that at high heating rate, however the changes in density showed the opposite trend. The flexural strength, hardness and electrical conductivity of the heat-treated composites generally decreased with increasing heating rate. Comprehensive comparison of mechanical, electrical performance and manufacturing cost at different heating rates obtained from the above work, the optimum heating rate is 2 ℃/min. The corresponding values of flexural strength, hardness and electrical conductivity of composite are 69 Mpa, 55 and 6.44×10^3 S/m, respectively.

Introduction

Conductive polymer-base composites are attractive materials for electrical applications such as electrical switches, electronic brushes, electrical contacts and electrodes, where high mechanical strength, good wear resistance and high thermal and electrical conductivity are needed [1-3]. The design and manufacture of composites with high electrical conductivity have been a research focus in recent years [4]. In order to improve the electrical conductivity, heat-treatment of material using heat-treatment process would be a effective way. Many works have been done to investigate heat-treatment of various polymer-based composites. Kuo [5] studied the effect of heat-treatment rate in a wide range (1, 100 and 1000 ℃/min) on the properties of a PAN/phenolic-based carbon/carbon (C/C) composite, and found that the composite processed at a higher heat-treatment rate had a higher porosity level, more large pores and a more graphitic structure than that processed at a lower heat-treatment rate. Lee [6] compared two different impregnating methods to investigate the influence of heat-treatment rate on the tribological properties of carbon/carbon (C/C) composites, and observed that the specimens with the higher heat-treatment rate exhibited the lower bulk density and infiltration efficiency, but the higher porosity and friction coefficient. Thus far, little has been reported on the research of the heat-treatment of graphite/phenolic-based composite.

In the present paper, the graphite/phenolic-based composite was fabricated through hot compression molding followed by heat-treatment process. Meanwhile, the effect of heating rate on the properties of a graphite/phenolic-based composite were investigated.

Experimental

Materials. The polymer used in this work is a phenolic resin, which is a mixture of 60 wt% phenolic resin and 40 wt% ethanol. Graphite (average particle size 4 m) with flake shape was used for base material.

Sample preparation. The mixture of phenolic resin and graphite powder was mixed in a mechanical mixer for 10 min at 3000 rpm speed, and then spread in a metal mould. The mould was placed on flat plate sulfuration bed and heated to 170 ℃. A pressure of 20 MPa was applied and held for 1.5 h to fabricate the composite. After the mould was cooled at room temperature, the sample was pulled off from the mould. The samples were then heat-treated in a furnace in a nitrogen atmosphere. The final heat-treatment temperature was 800 ℃. Then the heat-treated specimens were allowed to cool in the furnace itself.

Characterization techniques. The samples were cut into specimens 3 × 6 × 50 mm3 in size for resistivity measurement. The resistivity was measured at ambient temperature using a DC low ohm meter (TH2512B). The average resistivity of each sample was obtained from five repeated measurements at different locations on the sample, and was then converted to electrical conductivity. A three-point flexural test was performed to measure the flexural strength of the composites using a universal testing machine (MTS, C45). Five specimens of size 60 × 10 × 5 mm3 were tested for each set of samples and the mean values were reported. Morphological observation was performed on a Hitachi S4800 field-emitting scanning electron microscope, prior to which the surfaces of composite samples were vacuum coated with a thin gold layer to avoid charging.

Results and discussion

To study the effect of heating rate on the properties of material, four different rates, 1 ℃/min, 2 ℃/min, 5 ℃/min and 10 ℃/min were selected to heat-treating composites, the final heat-treatment temperature was fixed at 800 ℃. The changes in length and density were measured after heat-treatment as listed in Table 1. Comparison of the data in Table 1 revealed that the changes in length of samples heat-treated at low heating rate were larger than that at high heating rate, however the changes in density showed the opposite trend. The greater the length contraction, the more compact structures. The sample heat-treated at 1 ℃/min and 2 ℃/min possessed the more compact structure which was associated with the properties of composite.

Table 1 Changes in length and density of samples after heat-treatment.

Heating rate (℃/min)	Length (%)	Density (%)
1	-0.49	-0.58
2	-0.53	-0.60
5	-0.42	-0.69
10	-0.30	-0.71

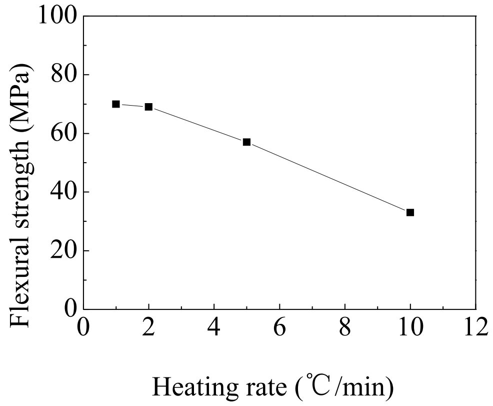

Fig. 1 Flexural strength of composite with heating rate.

The relationship between the flexural strength and heating rate of composites was shown in Fig. 1. It can be seen from Fig. 1 that the flexural strength of composites generally decreased with the increase of heating rate. Heating rate increasing from 2 ℃/min to 10 ℃/min caused a decrease in flexural strength by about 52%. The sample heat-treated at 2 ℃/min had the flexural strength of 69 MPa. However, the flexural strength of composite treated at 1 ℃/min showed a very little change in comparison to that of composite at 2 ℃/min. Nam [7] reported that the large heating rate could generate a large temperature gradient, resulting in nonuniform carbonization inside composite. As a result, a large pressure is built up within the composite which, in turn, could cause a localized delamination and/or a general damage to the matrix structure. Data from this work also indicate that a lower heating rate favors the mechanical properties of graphite/phenolic resin-based comosite. Fig. 2 showed the SEM micrographs taken from the fractured surfaces of composites heat-treated at 2 ℃/min and 10 ℃/min. Clearly, the sample heat-treated at 2 ℃/min exhibited a compact structure, however there were many microcracks inside the sample heat-treated at 10 ℃/min. This provided a further evidence of the flexural strength decreasing as heating rate increasing.

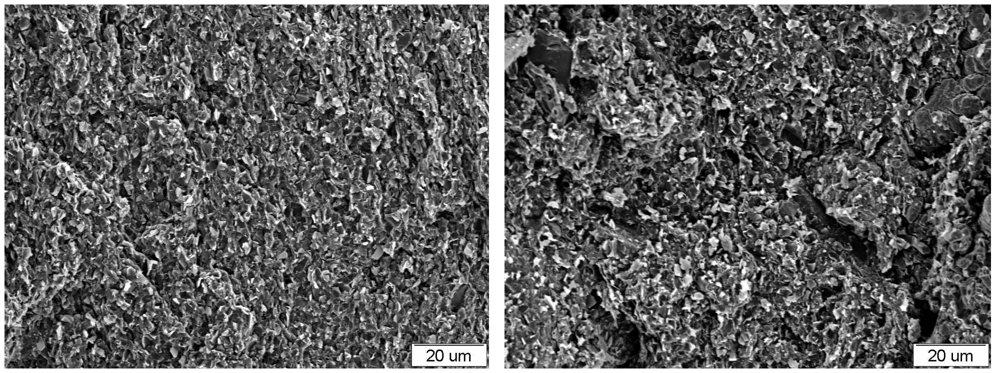

Fig. 2 SEM images of the fracture surface of composites heat-treated at (a) 2 ℃/min and (b) 10 ℃/min.

The hardness of composites after heat-treatment was shown in Fig. 3. The hardness of composite showed a very little change when heating rate ranged from 1 ℃/min to 5 ℃/min. If increasing heating rate to 10 ℃/min, the hardness exibitted a sharply drop. The hardness of the composite heat-treated at 10 ℃/min decreased by about 42% compared to that of the composite heat-treated at 5 ℃/min. The higher heating rate leads to the presence of significant amounts of microcracks in the matrix as shown in Fig. 2, accounting for the lower hardness of composite heat-treated at 10 ℃/min.

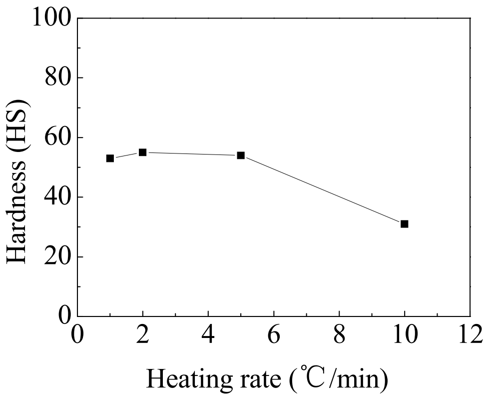

Fig. 3 Hardness of composite with heating

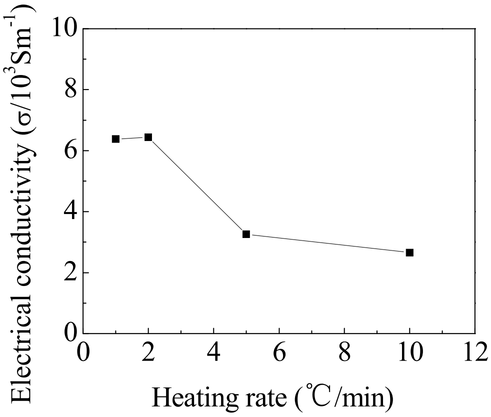

Fig. 4 Electrical conductivity of composite with heating rate.

The electrical conductivity of heat-treated composite is largely influenced by heating rate. As anticipated from Fig. 4, the electrical conductivities of composites heat-treated at 1 ℃/min and 2 ℃/min possessed almost same values. Over 2 ℃/min, the electrical conductivity decreased with increasing heating rate. Comparison of the data in Fig. 4 revealed that heating rate increasing from 2 ℃/min to 5 ℃/min caused a considerable decrease in electrical conductivity by about 49%. High heating rate caused a lot of microcracks and pores in the composites, which hindered the conductive channel and resulted in the decrease of electrical conductivity. In addition, the phenolic resin exhibit a transition from a polymer-like insulator to a carbon-like conductor, which enhanced the electrical conductivity of composites. However, the high heating rate generated damages to the carbonized matrix structure and caused the decrease in electrical conductivity.

Comprehensive comparison of mechanical, electrical performance and manufacturing cost at different heating rate obtained from the above work, the optimum heating rate is 2 ℃/min. The corresponding values of flexural strength, hardness and electrical conductivity of composite are 69 Mpa, 55 and 6.44×10^3 S/m, respectively.

Conclusion

Graphite/phenolic-based composite was fabricated through hot compression molding followed by heat-treatment process. The effects of heating rate on the properties of composites were investigated experimentally. The shrinking in length of composites heat-treated at low heating rate was larger than that at high heating rate, which means the sample heat-treated at 1 ℃/min and 2 ℃/min possessing the more compact structure. The flexural strength, hardness and electrical conductivity of heat-treated composites generally decreased with increasing heating rate. The values of flexural strength, hardness and electrical conductivity of composite heat-treated at 2 ℃/min are 69 Mpa, 55 and 6.44×10^3 S/m, respectively.

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