PREPARATION AND MICROSTRUCTURAL INVESTIGATION OF CARBON FIBERS/CARBON NANOFIBERS REINFORCED CARBON COMPOSITES BY CHEMICAL VAPOR INFILTRATION

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Introduction

Carbon-carbon composites consisting of carbon fibers(CF) in pyrolytic carbon (PyC) matrix have beeing attracted particular interests due to their high strength and light weight[1]. The interface between carbon fibers and the surrounding PyC matrix plays great roles in the oxidation and mechanical properties of carbon-carbon composites[2,3]. Though carbon nanofibers (CNFs) are not as perfect in structure or as good in properties as carbon nanotubes, they should be a good alternative to carbon nanotubes as an additive in composites due to their industrial production[4,5]. When CNFs were grown on carbon fibers, it would be a good way to improve the interface properties of carbon fibers reinforced composites and then enhance the properties.of the obtained composites. Therefore, in the present work, CNFs were grown on carbon fibers of the felts by catalytic chemical vapor deposition with nickel nanoparticle as catalysts in order to strengthsen the carbon fiber/PyC interface of carbon-carbon composites.

Experimental

The used PAN-based carbon fiber felts had a size of 20×10×60 mm^3. Ni-Al-Cu alloy nanoparticles were prepared on the carbon fibers of the felts by homogeneous precipitation method followed by hydrogen reduction at proper temperature. Afterwards, natural gas was used as carbon source and hydrogen was used as dilute gas to grow carbon nanofibers on the carbon fibers by catalytic chemical vapor deposition at 650~750ºC in a vertical graphite furnace for 45 minutes and then cooled down in the furnance. Then the felts were washed in diluted hydrochloric acid for 15 minutes to dissolve the alloy catalyst followed by washing with de-ioned water for three times and drying at 100 ºC for about 2 hours. The felts were heated to about 1120ºC in the same vertical graphite furnace for densificarion by isothermal chemical vapor infiltration with natural gas as carbon source and hydrogen as dilute. After 50 hours' densification, the samples were cooled down in the furnace. Then the specific density and thermal conductivity of the as-obtained carbon-carbon composites was mesured. The size of the sample for conductivity test is Φ10×4mm. The freshly fracutured surfaces were examined by field-emission scanning electron microscope (FESEM) without evaporating conductive layers prior to the investigation.

Results and Discussion

Most of the carbon fibers in the needle-punched carbon fiber felts was grown with CNFs on by catalytic chemical vapor deposition for 45min, which was shown in Fig.1. CNFs in length of microns grew on the carbon fibers. The CNFs were crawled along the carbon surface or grown nearly perpendicular to the carbon fiber.

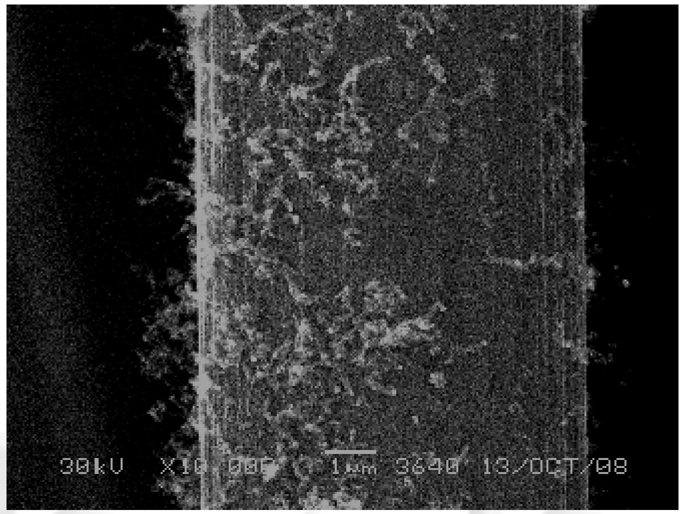

Fig.1 SEM image of the carbon fibers with carbon nanofibers

After desification of the felts by chemical vapor infiltration for 50h, carbon fibers/carbon nanofibers reinforced carbon matrix (C/CNF/C) composies were obtained. The density of the obtained C/CNF/C composites was up to 1.75 g/cm^3. The fractured morphorlogies examined by FESEM were shown in Fig.2 and Fig.3. Some carbon nanofibers established bridges between carbon fiber surface and the surrounded pyrolytic carbon matrix, which should be helpful to the interface performance. Most interestingly, some straight carbon nanofibers in-situ grew during chemical vapor infiltration of pyrolytic carbon as shown in Fig.2 and Fig.3. It should be noted that the temperature for chemical vapor infiltration of pyrolytic carbon was 1120±5ºC, which was much higher than that for growth of CNFs by chemical vapor deposition with natural gas as carbon source, i.e. 650~750 ºC in the present work. Moreover, after dissolving in diluted hydrochloric acid and washing with de-ioned water, the felts with CNFs grown on the carbon fibers should consisted of very little of catalysts. However, in the case of higher temperature and lack of catalysts, it is true that depostion of pyrolytic carbon and growth of carbon nanofibers could take place at the same time during chemical vapor infiltration. With CNFs grown in the pyrolytic carbon matrix during chemical vapor infiltration, the properties of the obtained carbon-carbon composites would be better than that without CNFs in the carbon matrix. But at present, the mechanism of infiltration of pyrolytic carbon and growth of CNFs was still not clear.

The thermal conductivity of the as-obtained C/CNF/C composites was shown in Table 1. It showed that with CNFs grown on the carbon fibers and in the carbon matirx, the thermal conductivity was much improved.

Table 1. Thermal Conductivity of C/CNF/C Composites

Sample	P（g/cm^3）	$\lambda_{//}$(w/m·k)	$\lambda_{\perp}$(w/m·k)
C/CNF/C	1.75	96.42	53.48

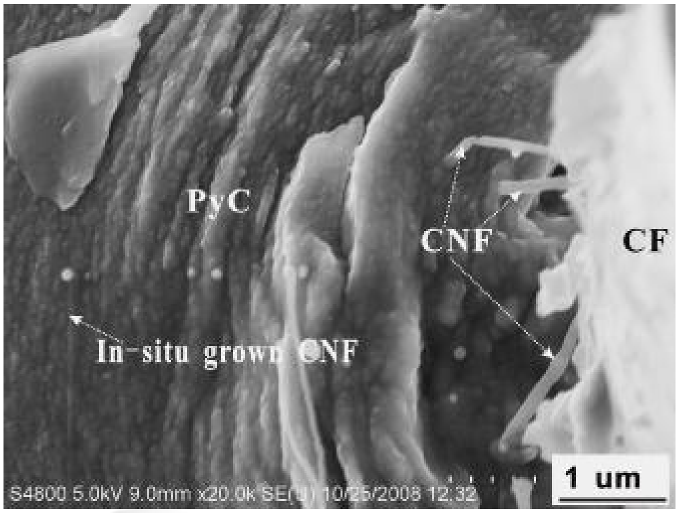

Fig.2 CNF bridge-combinations between carbon fiber and the surrounded pyrolytic carbon matrix

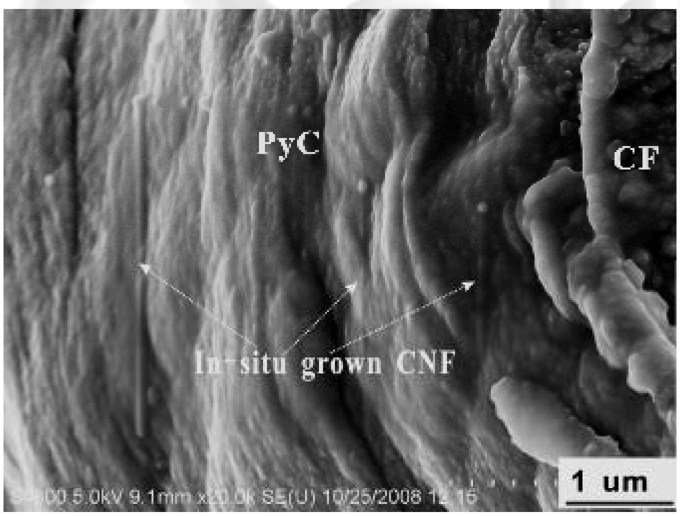

Fig.3 In-situ grown CNFs in pyrolytic carbon during chemical vapor infiltration processing

Conclusions

CNFs were grown on carbon fibers of needle-punched felts by catalytic chemical vapor deposition in a vertical graphite furnace with Ni-Al-Cu alloy nanoparticles as catalyts and natural gas as the carbon source. The CNFs established bridge-combinations between carbon fiber surface and the surrounded pyrolytic carbon matrix, which led to higher thermcal conductivity. Some straight carbon nanofibers in-situ grew during chemical vapor infiltration of pyrolytic carbon.

Acknowledgment

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