EFFECT OF SURFACE TREATMENT WITH ISOPTHALIC ACID ON THE DISPERSION STABILITY OF MULTI-WALLED CARBON NANOTUBES

[1,2]Ok-Kyung Park, [3]Jin Sun Lee, [1,3]Joong Hee Lee, [2]Junkyung Kim, [2]Sung Ho Lee, [2]Bon-Cheol Ku

[1]Departments of BIN Fusion Technology, Chonbuk National University Jeonbuk, 561-756, Korea

[2]Institute of Advanced Composite Materials, Korea Institute of Science and Technology, Jeonbuk, 565-902, Korea

[3]Department of Hydrogen and Fuel Cell Engineering, Chonbuk National University, Jeonbuk,561-756, Korea

Introduction

Polyacrylonitrile (PAN) is used as carbon fiber (CF) precursors due to its high tensile strength. Multi-walled carbon nanotubes (MWCNTs) have attracted considerable interest as effective nanoscale reinforcements of properties such as tensile strength and modulus of carbon fiber [1]. However, the main limitations of MWCNTs for the reinforcement of carbon fiber are their poor dispersion in a polymer matrix and weak interfacial interaction with the matrix. The aggregation tendency of MWCNTs in the composites, leads to decrease the mechanical properties of the resulting carbon fiber. Therefore, the dispersion of MWCNTs in solvent is the biggest challenge for the fabrication of high performance carbon fiber. MWCNTs were treated with isophthalic aicd (IPA) to enhance the dispersion stability of the MWCNTs. The isophthalic acid group was covalently functionalized on the surface of MWCNT by solvent-free functionalization method. Hence, this study examined the effect of surface treatment with IPA on the dispersion stability of MWCNTs

Experimental

0.5g of MWCNTs was dispersed in a 1:3 mixture of concentrated HNO_3 and H_2SO_4 (200 mL) and sonicated for 30 min. The mixture was then poured into a flask. The oxidation process was carried out at 60ºC for 2 hr. The oxidized MWCNTs (O-MWCNTs) were washed with excess deionized (DI) water until the pH was approximately 7 and finally washed with ethanol. Then 0.3g of O-MWCNTs was immersed in a 100 mL H_2SO_4 at room temperature and sonicated for 30 min. The mixture was then poured into a flask. After that, 1.8g of amino isophtalic acid and 0.69 g sodium nitrite was quickly added via syringe. The mixture suspension was vigorous stirred at 60ºC for 1hr. After cooling to room temperature, the suspension was diluted and washed with DMF and finally washed with ethanol to remove excess DMF. The product was dried at 60ºC for 24 hrs in a vacuum oven. The resulting MWCNTs are designed as IPA-O-MWCNTs.

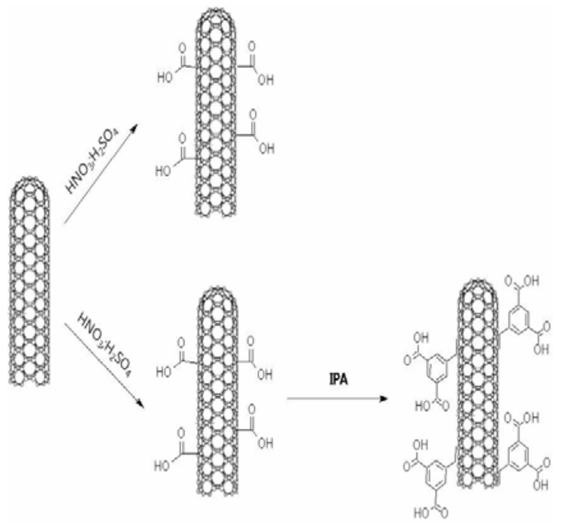

Fig.1. Synthetic scheme of O-MWCNTs and IPA-O-MWCNTs

Results and Discussion

Fig. 2 shows the FT-IR spectra of the raw MWCNTs and surface treated MWCNTs (O-MWCNTs and IPA-O-MWCNTs). The peaks at around 1630, 1730 (C=O) and 1100 (C-O) cm^{-1} correspond to the stretching mode of the carboxylic acid (-COOH) group. The carbonyl (C=O) stretching appeared at $1731cm^{-1}$ (1723 in precursor

IPA salt) and the band at around 1630cm^{-1} is assigned to the C=O stretching mode in quinine groups. The IPA treated O-CNTs (IPA-O-CNTs) shows a peak at 664 cm^{-1} due to the strong O-C-O δ bands [2]. And another new peak at 1246cm^{-1} may be assigned to the characteristic C-O stretch, indicating the formation of the phenolic oxygen groups [2]. This suggests that surface treated MWCNTs introduce isophthalic acid groups on the MWCNTs surface.

Dispersion stability of surface treated MWCNTs and untreated MWCNTs were investigated by measuring the transmittance of visible light at 630 nm using UV/vis spectrometry. The transmittance as a function of settling time was recorded and is shown in Fig 3. For the purpose of normalization, we define the initial transmittance as 0%, which represents the case where no sedimentation occurred at time 0 min. The initial increase in transmittance was attributed to the rearrangement of particles. The initial transmittance of O-MWCNTs and IPA-O-MWCNTs dramatically increased during 2 hrs. After that transmittance slowly increases and finally reached to 8.6% and 5.1%, respectively. These results observed that the IPA-O-MWCNTs showed much better dispersion stability than that of O-MWCNTs. This is due to the fact that the defect side walls of the MWCNTs carry more dissociated COO- groups after oxidation with the acid mixture, which can stabilize the nanotubes via an electrostatic stabilization mechanism. This suggests that the dispersion stability initially increases with increasing concentration of carboxylic acid groups on the surface of the MWCNTs.

Conclusion

This research has focused on the effect of surface treatment with isopthalic acid on the dispersion stability of MWCNTs. The results indicated that the IPA was covalently attached on the surface of MWCNTs. Incorporation of IPA groups has strong influence on the dispersion stability. The dispersion stability and mechanical properties of functionalized MWCNT reinforced PAN composites will be investigated and reported.

Acknowledgement

This work was supported by a grant from KIST institutional program and the fundamental R & D Program for Core Technology of Materials funded by the Ministry of Knowledge Economy, Republic of Korea. This work was also financial supported by World Class University (WCU) program (R31-2008-20029) funded by the Ministry of Education, Science and Technology.

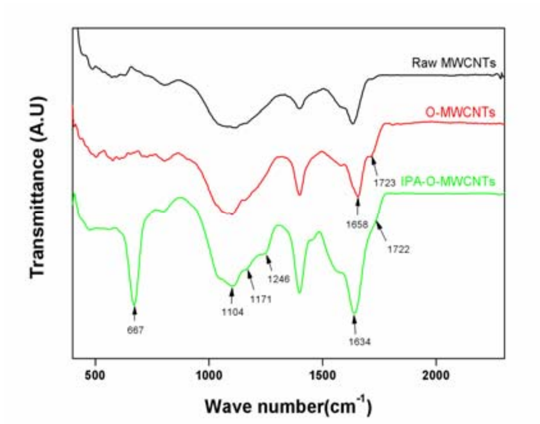

Fig 2. FT-IR data of raw and surface treated MWCNTs (O-MWCNTs and IPA-O-MWCNTs).

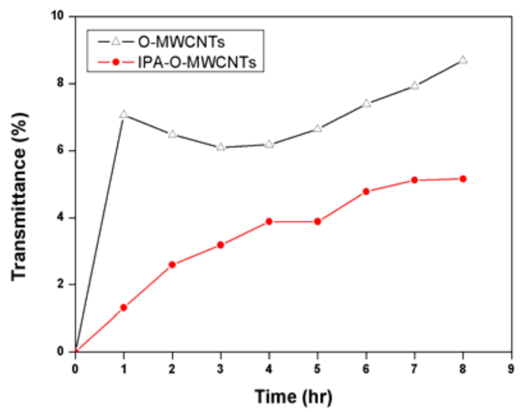

Fig 3. The UV/Vis curves of O-MWCNTs and IPA-O-MWCNTs

References

1. Chae, HG., Choi ,YH., Minus, ML., Kumar ,S. Carbon nanotube reinforced small diameter polyacrylonitrile based carbon fiber. *Composites Science and Technology* 69 (2009) 406-413.

2. Yang, S-Y., Long, L-S., Huang. R-B., Zheng, L-S., Ng, SW. Hydroxylation of isophthalic acid in the hydrothermal reaction of CuII-isophthalate system – Syntheses and crystal structures of two coordination polymers. *Inorganica Chimica Acta* 358(2005) 1882–1886