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Note

Studies on Nanoscaled Ni-P Plating of Carbon Fiber Surfaces in a Composite System

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The carbon fibers are frequently given in a surface treatment, the exact nature of which is a trade secret. These treatments increase the surface active sites and then improve bonding between fibers and resin matrix [1, 2]. Metal plating has been used to produce metal matrix [3] or polymer matrix [4, 5]. In the case of electroless plating, carbon fiber surfaces are metallized when exposed to plating solutions coating molten salt in the presence of a reductant. The nickel process is based on a redox reaction in which the reducing agent is oxidized and Ni^{2+} ions are reduced on the substrate surfaces. Once the first layer of nickel is deposited, it is acting as a catalyst for the process. If the reducing agent is sodium hypophosphite, the obtained deposit can be a nickel-phosphorus (Ni-P) alloy. And chemical reaction consists of two kinds of simultaneous reactions: the one, the cathodic reaction of Ni^{2+}, H^+, and $H_2PO_2^-$ or the deposition of Ni-P alloy and the production of hydrogen [11-14], others, the anodic oxidation of $H_2PO_2^-$. It is generally accepted that a microcrystalline, amorphous or a co-existence of these two phase can be obtained depending on phosphorus contents [6].

The objective of this study is to evaluate the influences of the Ni-P deposition on the fiber surface properties and ductile properties of composites with epoxy resin.

For the present investigation, the reinforcement materials used were the continuous PAN-based carbon fibers (12K, TZ-307) manufactured by Taekwang of Korea. Epoxy resin, YD-128, (supplied from Kangnam Chem. Co. of Korea) was used as the polymeric matrix. Nickel sulfate ($NiSO_4$) and nickel chloride ($NiCl_2$), and sodium hypophosphite (NaH_2PO_2) were, respectively, used as the molten salts and reducing agent in the nano-scaled electroless plating systems for the carbon fiber surface treatment. A wide-angle X-ray diffraction (XRD) patterns of these samples was obtained with a Rigaku Model D/MAX-III B diffractometer equipped with a rotation anode and CuK_α radiation (λ=0.15418 nm) as the source for measuring the interlayer spacing (d_{002}). The surfaces of carbon fibers were analyzed using a VG Scientific LAB MK-II X-ray photoelectron spectrometer (XPS). The spectra were collected using a Mg K_α X-ray source (1253.6 eV). The impact properties of CFRP produced by Ni-P deposited carbon fibers studied were carried out by means of a falling weight impact tester (Rosand Precision Instrument) according to the ASTM D256.

The wide-angle XRD patterns of the carbon fibers studied are shown in Fig. 1. The location and broadness of this peak indicate that carbon fibers have a coke like character with disordered carbonaceous interlayer. Also, it seems that the carbon basal planes of the carbon structure are barely changed by nanoscaled nickel-plating, otherwise Ni-P compounds are newly introduced on surfaces of carbon fibers by plating, as seen in Fig. 1. The diffraction patterns around 2θ=32.5 and 45^o present the nickel phosphide (NiP_2) and the regular Ni-P amorphous alloy, indicating a typical amorphous character, on surfaces of nickel-plated carbon fibers, respectively [7]. It is indicated that two phases, such as microcrystalline and amorphous, co-exist on surfaces of nickel-plated carbon fibers and affect to the mechanical interfacial properties of the composite systems.

XPS spectra for the non-treated and Ni-P deposited carbon fibers are shown in Fig. 2. As anticipated, the non-treated

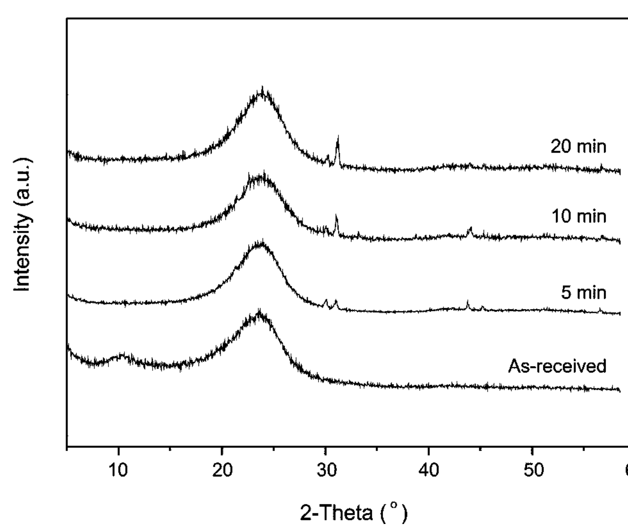

Fig. 1. XRD patterns of the Ni-P deposited carbon fibers with plating time.

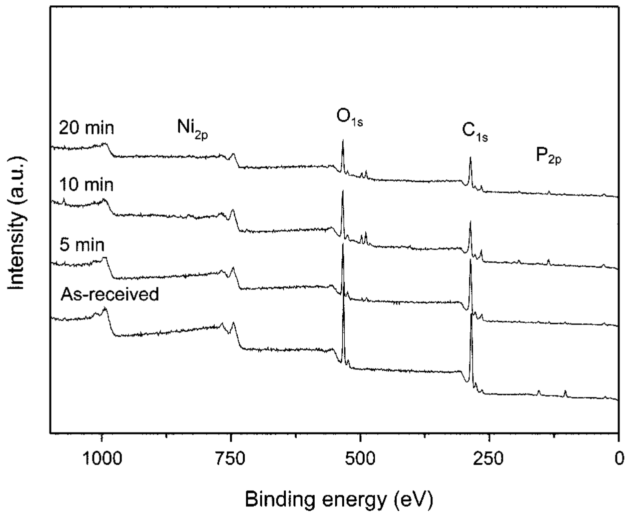

Fig. 2. XPS spectra of the Ni-P deposited carbon fibers with plating time.

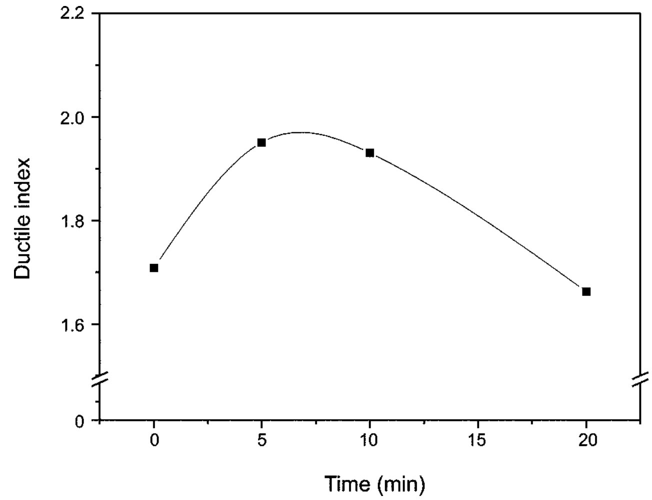

Fig. 3. Evolution of the ductility index as a function of the electroless nickel-plating time.

carbon fibers show a C_{1s} peak and a substantial O_{1s} peak at 284.6 and 532.8 eV, respectively. The O_{1s} peak is probably due to surface carbonyl or carboxyl groups of carbon fibers in intrinsic nature. Otherwise, for the Ni-P deposited carbon fibers, carbon, oxygen, nickel (B.E.=857.6 eV), and phosphorus (B.E.=135 eV) peaks are observable in XPS spectra. The O_{1s} peak of nickel plated carbon fibers is probably due to carbon fiber surfaces of NiO, C=O or -OH, O-C-O, P_2O_5, and -C-O groups [8].

As the experimental XPS results, it is found that the surface composition of carbon fibers changed substantially after the electroless nickel plating. The carbon content of nickel-plated fibers decreases when the fibers are plated with metallic nickel, whereas the oxygen, nickel, and phosphorus of coated fibers are higher than those of non-treated. These active groups on the carbon fiber surfaces, which are produced with nickel plating, can help to change the polarity and the functionality of fiber surfaces [9].

Meanwhile, the load history during impact test is considered by the sum of two regions; initiation and propagation of the fracture. The ratio of propagation energy to initiation one can be considered as ductility index, *DI*, for evaluating the total absorbed energy of a material [10, 11]. The results of the *DI* of the CFRP produced by Ni-P deposited carbon fibers are shown in Fig. 3. As a result, the plating time leads to an increase in increasing the *DI* to 10 min. These results indicate that the ductility of the nickel plated carbon fiber-reinforced plastic composites are much higher than that of non-treated materials. It can be due to the deposited Ni-P alloy. When the composites that have been reinforced by the fibers with brittle coating is under load, the cracks in the matrix or in the coating propagate to the interface between the fibers and the coating, and then the directions of the crack propagation is greatly influenced on the stress field of the crack tip and the mechanical properties of fiber and coating. If ductile layer exists between fibers and matrix, then when the crack propagates to the ductile layer, it increases the crack propagation path and adsorption energy and decreases stress concentration on interfaces between fibers and matrix. Thus the fiber cannot (or little) be damaged. Therefore, the coating with a Ni-P layer can change the propagation direction of crack, blunt the stress field at the crack tip and provide fibers protection, and the aim of fibers protecting can be realized [12].

Acknowledgements

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