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RESEARCH PAPER

The effect of a chemical vapor deposited carbon film from acetylene on the properties of graphitized PAN-based carbon fibers

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Abstract: Polyacrylonitrile (PAN)-based carbon fibers (CFs) were coated with carbon film by chemical vapor deposition using acetylene as precursor. The morphologies of the as-received and modified CFs were observed using SEM and AFM, and their mechanical properties, crystalline parameters, and electrical conductivity were measured after graphitization. It was found that the graphitized CFs that were prepared showed excellent mechanical properties (2 GPa for strength and 270 GPa for modulus) and good electrical conductivity (5×10^{-4} Ωcm) compared with the unmodified ones.

Key Words: carbon fiber; CVD; graphitization

1 Introduction

Carbon fibers (CFs) exhibit a very wide range of thermal, electrical, and mechanical properties and are widely used in many important fields. Various techniques are needed to develop CFs with high tensile strength and high modulus. Polyacrylonitrile (PAN) fibers are the most suitable precursors for producing high-performance CFs [1–3]. Heat treatment of PAN fiber under tensile load is beneficial to improve fiber quality [4]. Bahl and coworkers pretreated precursors with CuCl to produce high-performance CFs [5,6]. Raskovic and Morinkovic investigated stabilization of PAN fibers by oxidation with SO_2, oxygen, or air and observed that sulfur was incorporated into the chemical structure of the fiber, which improved the mechanical properties of CFs [7]. Ko et al. modified PAN precursors with cobalt chloride to increase mechanical properties of CFs and carbon films [8,9].

In the present study, the effect of modification of CF by chemical vapor deposition (CVD) of acetylene on properties and microstructure of the resultant graphitized CFs was discussed. Before this study was carried out, the authors of this study assumed that the modification of CFs could improve mechanical properties and electrical conductivity of the resultant CFs more than that by any conventional process. In this study, PAN-based CFs were coated with carbon film by CVD of acetylene to modify their surface. The resultant CFs were graphitized to prepare high-performance CFs with excellent mechanical property and good electrical conductivity. The microstructures and properties of the graphite CFs were analyzed by X-ray diffraction (XRD), Raman spectra analysis, scanning electron microscopy (SEM), and atomic force microscopy (AFM).

2 Experimental

2.1 Raw material

In this study, PAN-based CFs (T700SC, supplied by Toray Ltd., Japan) were used. A single tow contains 12,000 monofilaments.

2.2 Carbon film coating on the surface of CFs

T700SC bundles were coated with hydrocarbon in a constantly driven thermal chemical vapor deposition (CVD) process using acetylene as a precursor [10]. Before the experiment was started, the as-received CFs were unsized with organic solvent to remove sizing agents. The resultant CFs was mounted on a sample holder and inserted into a furnace, which was subsequently heated to synthesis temperature and then purged with N_2 for 5 min. Precursor gas acetylene was blown to the furnace at a rate of 50 cm^3/min to modify CFs by CVD, and the furnace was maintained in an isothermal state at 950℃ for 10, 15, 20, 25, and 40 min to yield sam-

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ples, which were labeled as A, B, C, D, and E, respectively. After this stage, the modified CFs were heat treated up to 2700℃ for graphitization.

2.3 Observation of morphology of CFs

The surface morphologies of the modified PAN carbon fibers were observed using SEM (S300, Hitachi, Japan) and AFM (Thermo Microscopes Auto Probe Research). With respect to AFM observation, a noncontact mode was employed with a scanning frequency of 2 Hz and a scanning set point of –0.41 to –0.5 μm.

2.4 Crystalline parameters of CFs

Crystalline parameters of carbon fibers were measured using XRD (MXP-3, MAC Science Ltd., Japan) with Cu Kα radiation. The degree of graphitization of CFs was analyzed using Raman spectrometer (Renishaw instrument with a Raman imaging microscope system 2000) with an argon ion laser of 514.5nm line as the incident radiation.

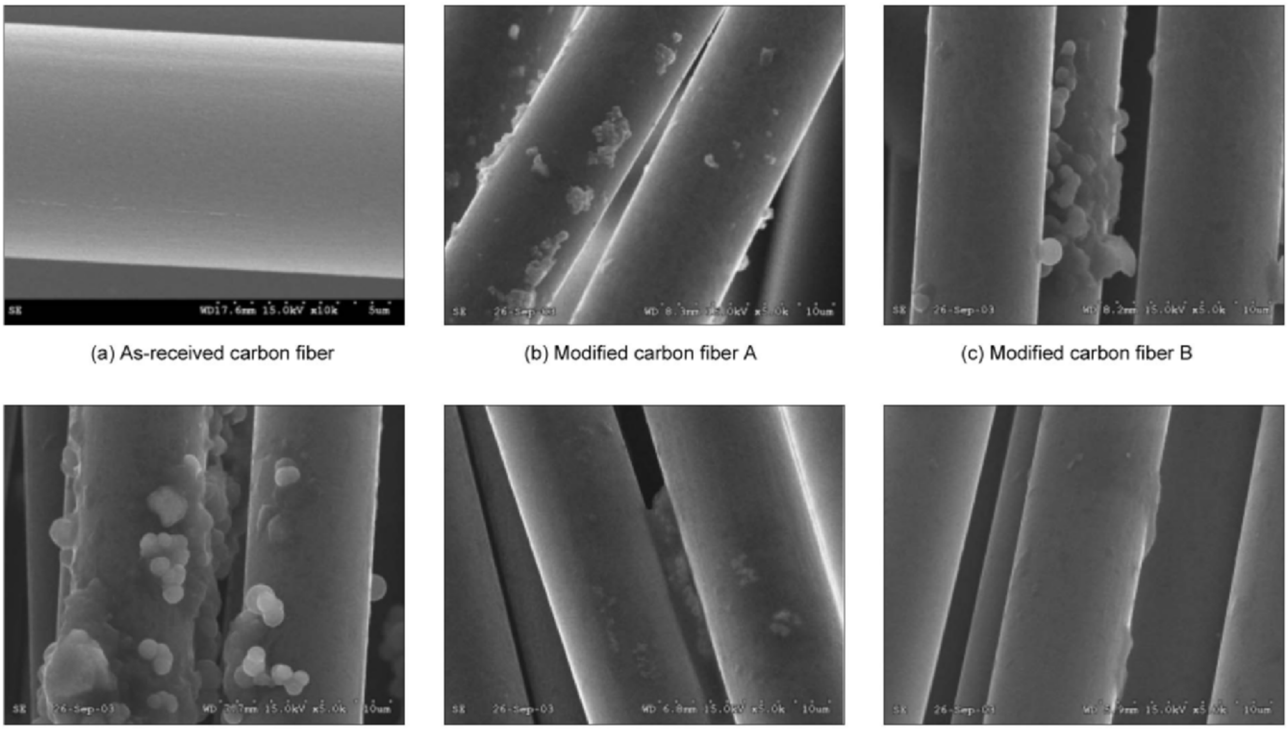
2.5 Testing of mechanical and electrical properties

Mechanical properties of CFs were tested by a single filament force testing machine (CY-6040A8, CY Ltd., Taiwan), in which load cell of 1000 g and cartridge spacing of 25 mm were used. The electrical conductivity of carbon fibers was measured using a table electric instrument with a test spacing of 10 mm.

3 Results and discussion

3.1 Analysis of surface structure

The surface of the as-received carbon fibers is quite smooth as shown in Fig.1(a). The surfaces of modified carbon fibers vary according to the CVD times. At the first stage of 10 min, there is only little deposition of carbon film on the surface of carbon fibers as shown in Fig.1(b), with irregular shape and small size of 0.01–4μm^2. With CVD times of 15 and 20 min, there is remarkable deposition of carbon film on the surface of carbon fibers (Fig.1(c) and (d)). The deposition exhibits spherical shape, and there is more accumulation on the surface of carbon fibers. Excess deposition appears when the CVD time is 25 min, which leads to cohesion of some carbon fibers, as shown in Fig.1(e). In the stage, it was assumed that carbon spheres on the surface of carbon fibers are overgrown and destroyed, thereby causing cohesion of carbon fibers. Fig.1(f) shows smooth surface appears on carbon fibers when the deposition time is 40 min.



(a) As-received carbon fiber (b) Modified carbon fiber A (c) Modified carbon fiber B

(d) Modified carbon fiber C (e) Modified carbon fiber D (f) Modified carbon fiber E

Fig.1 SEM images of (a) as-received carbon fibers and (b–f) their modified forms produced using CVD process with different retention times