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Effects of Ni catalyst on growth velocity and morphology of vapor growth SiC nano-fibers

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Abstract. Using Ni granules as a catalyzer, composite felts reinforced by both SiC nano-fibers (SiC-NFs) and carbon fibers were prepared at 1273K. SiC-NFs were deposited on the surface of the carbon fibers in-situ by chemical vapor growth. The microstructure and morphology of the fibers after being electroplated and deposited were characterized by scanning electron microscopy and transmission electron microscopy. The phase of the fibers after being deposited was characterized by X-ray diffractometry. The results show that the SiC-NFs produced by chemical vapor growth are single crystals of \( \beta \)-SiC. It was found that Ni catalyst had important effects on the growth of SiC-NFs. Smaller nano-granules were more active as catalyzers, resulting SiC-NFs appeared more spindle-like and had a more homogeneous dispersion. The weight change of the samples before and after deposition shows that a longer electroplating times results in SiC-NFs having a faster growth velocity.

1. Introduction

Since the discovery of carbon nanotubes (CNTs) by Iijima in 1991 [1], researchers have been very actively trying to gain controls on length, diameter, etc. [2-3]. CNTs and carbon nano-fibers (CNFs) are interesting candidates for realizing novel composite materials due to their almost one-dimensional shape coupled with the exceptional mechanical, thermal and electrical properties [4-5]. The transition elements are promising candidates for use as catalyzers for preparing CNTs and CNFs [6-10]. The method of preparing CNTs and CNFs is also used for preparing SiC nano-fibers (SiC-NFs) [11]. SiC-NFs or SiC nanoparticles have some special properties such as high strength, high modulus, high corrosion and oxidation resistance and so on. So, experiments on SiC-NFs deposited on the surface of carbon fibers in-situ was perfectly developed in carbon fiber (CF) reinforced carbon composites (C/C composites) and CF reinforced carbon composites (C/SiC composites) [12]. The configuration and distribution of the catalyzer plays a key role in the process of vapor growth. During our research, we applied electroplating Ni granules on the surface of the CFs to afford a random distribution of the Ni catalyzer. Then, SiC-NFs were grown on the surface of the carbon fibers in-situ by chemical vapor growth. By the method of chemical vapor growth, it is possible to achieve a large scale synthesis of these composite felts reinforced by both SiC-NFs and CFs. The resulting composite felt material is the subject of intensive research, and appears to be one of the most promising candidates for the future.
preparation of a reinforced framework for C/C and C/SiC composites. The effect of different electroplating times on the morphology and growth velocity of the SiC-NFs is discussed in this work.

2. Materials and methods

2.1. Raw materials
Carbon fibers were produced by Toyor Co.; Japan (T700, 12K). Needled integer felts (0.4 g/cm³) were produced by the Tianniao Company, Yixing city, Jiangsu Province. Ni₂SO₄ was produced by Qinxian Chemical Co.; Ltd in Shanghai. The electroplating equipment produced by Antaixin Electronic Co.; Ltd in Shenzhen comprises a direct current power supply with a symmetrical output, the type of which is PMR155.

2.2. Electroplating Ni on carbon felts to form catalyst nano granules
The felt was cut into spindle samples of 6×6×50mm in the same direction, soaked in acetone for 24 hours, washed in deionized water repeatedly, and then dried. Such samples were placed into an electroplating solution (Ni₂SO₄, 10 wt %), and then electroplated for 2.5, 5, 7.5 and 10min. The electronic current intensity was 100mA. Then, the samples were soaked in deionized water, washed, dried and weighed. The microstructure and surface morphology of the carbon fibers after being electroplated with Ni were observed by scanning electron microscopy (SEM, TSM-6360LA, and Japan).

2.3. Growth of SiC-NFs by chemical vapor growth
These samples were first electroplated with Ni for different periods of time, and then SiC was deposited on the electroplated samples in a chemical vapor growth furnace at 1273K for 2, 4, 6, 8 and 10h respectively. The resulting samples were cooled and then removed from the furnace and weighed. Methyltrichlorosilane (MTS, 95%) was used as the source of carborundum, Argon (99.99%) was used as the carrier gas, and hydrogen (99.99%) was employed in the chemical vapor growth process in a dilute form. The pressure inside the furnace was 200–400pa. After growth of the SiC-NFs, SEM was used to characterize the morphology of the carbon fiber integer felt. Transmission electron microscopy (TEM, JEOL 2010F, Japan) was used to characterize the diameter, morphology and type of crystal formed. Composite felts first plated with Ni for 10 min and then coated with SiC by vapor growth for 10h were used as samples for powder X-ray diffractometry (XRD, PANalytical Company in Holland) to ascertain the phase of the composite felts.

3. Results and discussion
Figure 1 shows the SEM morphology of the CFs after electroplating with Ni for different times and Figure 2 shows the CFs after coating with SiC-NFs by chemical vapor growth. As shown in figure 1 (a) and (b), the electroplated Ni features are smaller than those in figure 1 (c). The nano-fiber shown in figure 2 (a) and (b) is more spindle-like than that in figure 2 (c) and also disperses much more homogeneously.

![Figure 1](image1.png)

**Figure 1.** SEM images of carbon fibers after electroplating with Ni for different times. (a) after electroplating Ni for 5min, (b) after electroplating with Ni for 7.5min and (c) after electroplating with Ni for 10min.
Figure 2. SEM images of carbon fibers after coating with SiC-NFs by chemical vapor growth for 2h. (a) after chemical vapor growth for 2h with plating Ni for 5 min, (b) with Ni for 7.5min and (c) with Ni for 10min.

Figure 3 shows the XRD patterns of carbon felts after coating with SiC by chemical vapor growth for 6h with plating Ni for 10min. In the diffraction graph, the diffraction peaks of C, β-SiC and Ni were observed. The weight content of β-SiC was found to have reached 10 percent as determined by phase analysis of XRD.

Figure 4 shows the TEM morphology of the SiC-NFs and diffraction spot of a selected section on the sample surface. As shown in figure 4(a) and (b), the SiC-NFs are unbent, have a single crystal diffraction spot and Ni catalyst nanogranules were found at the point of the arrow in Figure 4 (c).

3.1. Preparation of SiC-NF/carbon fiber composite felts
As shown in figure 1 (a) and (b), the Ni-nanogranules that are produced have diameters of several nanometers. But the Ni granules in figure 1 (c) are bigger than those of figure 1 (a) and (b), and have
diameters in the range of hundreds of nanometers, and the whole surface of the CFs is almost covered with Ni granules. In this respect, by keeping the current density at 100mA, the size, morphology and the amount of plating Ni on the surface of the CFs would be controlled by adjusting the plating time. As shown in figure 2, after plating the surface of the CFs with Ni, the nanofibers were deposited on the surface of the CFs in-situ. 

In figure 2 (a) and (b), the carbon fibers are bridged by chemical vapor growth SiC-NFs, making each of them not be a single fiber as reinforcement phase in composite materials. In addition, the traditional carbon felts, whether planar, two-dimensional or three-dimensional, invariably cause anisotropy after compositing. As described above, the effect of bridging and growing random nanofiber composite felts will improve this anisotropy. Moreover, it is also expected to improve the properties of the fiber-reinforced composites such as strength, modulus, corrosion and oxidation resistance, electric and heat conduction performance, etc.

3.2. Phase analysis of SiC-NF/CF composites

As shown in figure 3, the observed phases of the composite felts after chemical vapor growth include C, β-SiC and Ni. The C diffraction peak is broad; this corresponds with the fact that the carbon felts contain polycrystalline graphite. The β-SiC diffraction peak is sharp and is characteristic of the different 2θ angle accorded with the JCPDS CARD (No.29-1129), where the crystal content is close. From above, the β-SiC nanofibers are grown on the CF surface in-situ in the carbon felts after first plating with Ni and then coating with SiC using chemical vapor growth.

Figure 4(b) shows that the as-produced SiC-NFs are single crystals and have a single crystal diffraction spot. The β-SiC fibers behave as an intensifier for the composite material resulting in properties of high specific strength, high specific modulus, high-temperature stability and high corrosion and resistance durability [13]. Moreover, the β-SiC nanofibers can improve the compatibility between the carbon fibers and the matrix material (pitch, resin or metal) [12]. Thereby, the β-SiC nanofibers improve the properties of the resulting composite material prepared with C/C or C/SiC composites [14, 15].

3.3. Effect of plating time on growth velocity and morphology of SiC-NFs

The weight gain of as-received samples and the different-timed electroplating Ni samples are converted into weight increase percent. Figure 5 was obtained by taking the electroplating time coordinate as the X axis and the weight increase percent as the Y axis. In figure 5, comparing with the non-electroplated Ni samples, electroplating with Ni can improve the deposition velocity of SiC. The longer of electroplating times are, the more SiC-NFs produced by chemical vapor growth are. But if the electroplating time surpasses 5 min, the corresponding weight increase is negligible. Moreover, by keeping the same electroplating time, and prolonging the vapor growth time, more SiC-NFs are observed on the CF surface and the weight increases accordingly. Therefore, carbon felts electroplated with Ni and coated with SiC by chemical vapor growth, the density velocity in chemical vapor infiltration (CVI) C/C and C/SiC composite materials is found to increase.

Figure 5. The relation between electroplating time and weight increase percent: 1-CVD for 2h; 2- CVD for 4h; 3- CVD for 6h; 4- CVD for 8h; 5-CVD for 10h.
4. Conclusions
The SiC-NF/CF composite felts are prepared by the methods of chemical vapor growth SiC-NFs in situ. The results show the SiC-NFs are single crystals of $\beta$-SiC. The catalyzer of Ni granules have a great effect on the growth of SiC-NFs. Smaller and well-distributed Ni granules result in more spindle-like SiC-NFs with a better distribution. Longer electroplating times result in more Ni nanogranules growing on the surface of the CFs, and a faster growth velocity of SiC-NFs.

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