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Characterization of SiC nanowires prepared on C/C composite without catalyst by CVD

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Abstract: SiC nanowires were prepared on C/C composite surface without catalyst by chemical vapor deposition (CVD) using CH_3SiCl_3 as precursor. SEM images of the CVD-product reveal that some long nanowires have grown to tens of micrometers with some gathered as a ball. Some short nanowires agglomerate like chestnut shell with many thorns accompanied by some deposited nano-particles. XRD, Raman-spectrum and FTIR patterns indicate that the product is a typical β-SiC. TEM images show that the nanowires have a wide diameter range from 10 to 100 nm, and some thin nanowires are bonded to the thick one by amorphous CVD-SiC. A SiC branch generates from an amorphous section of a thick one with an angle of 70° between them, which is consistent with the [111] axis stacking angle of the crystal. SAED and fast Fourier transform (FFT) patterns reveal that the nanowires can grow along with different axes, and the bamboo-nodes section is full of stacking faults and twin crystal. The twisted SiC lattice planes reveal that the screw dislocation growth is the main mechanism for the CVD-SiC nanowires.

Key words: SiC nanowires; C/C composite; chemical vapor deposition; growth mechanism; characterization

1 Introduction

Carbon fiber reinforced carbon matrix composite (C/C composite) has been widely used as thermal structural material, for example, brake material, seal ring, rocket nozzle and throat insert, which is benefited from low density, low coefficient of thermal (COF), high special strength and thermal conductivity [1]. But one obvious defect, easily being oxidized in air above 450 °C, has limited the application of C/C composite. Therefore, oxidation-resistance is a key technology for C/C composite to ensure the properties in harsh surrounding [2].

After decades of research, SiC has become a kind of good material to prepare oxidation- and ablation-resistant coating for C/C composite [3,4]. But thermal residual stress, caused by different COFs between C/C composite and SiC, would induce micro-crack and be a fatal defect for the coating to deteriorate the property when facing severe temperature changes [5].

SiC nanowires, with high elastic modulus and decent chemical stability, have been widely used to reinforce ceramic-matrix composite [6,7]. LI et al [8] prepared the nanowires to reinforce SiC coating for C/C composite using Ni or Co as catalyst and proved good effect. Therefore, it is worth preparing SiC nanowires at first, which could improve the bonding between C/C composite and the coating [9].

SiC nanowires can be gotten by chemical vapor deposition (CVD), hydrothermal reaction, in-situ reaction, precursor pyrolysis and so on. For example, QING et al [10] prepared SiC nanowires at low temperature using silica sol, glucose, urea, PAM and nitric acid as raw materials, but the oxygenated chemicals would react with carbon material and influence the property of C/C composite. EICK and YOUNGBLOOD [11] produced the SiC nanowires from the spinning of poly (carbomethylsilane) with pyrolysis to ceramic, LI et al [12] also got it by using the off-gas from polycarbosilane-derived SiC. It is an interesting and good challenge for the coating-preparation on C/C composite. LIU et al [13] prepared the nanowires by deposited silicon on carbon nanotube and then annealed it at 1200 °C. KIM et al [14] synthesized the nanowires by a solid−vapor reaction between carbon nanowires and SiO vapor using Ni−Fe, Fe−Al and Ni−Fe−Al as catalysts. XU et al [15] and LIU et al [16] also got the nanowires by using CH_3SiCl_3 as raw materials and Ni as catalyst. Those technologies have produced the

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nanowires with good properties, but the catalyst is difficult to be removed thoroughly and the coating might be impure. Thus, it is necessary to prepare the nanowires on C/C composite surface without such residual metal-impurity.

Therefore, in this work, we prepared SiC nanowires on C/C composite surface without catalyst, and analyzed their nano-structure and CVD mechanism.

2 Experimental

C/C composite with the density of 1.83 g/cm^3 was provided by Hunan Boyun New Material Company Limited, China. It was machined to dimensions of 30 mm × 30 mm × 5 mm firstly, followed by being polished with 3000# SiC abrasive paper, cleaned in alcohol using ultrasonic cleaner and then dried prior to the test.

The preparation of SiC nanowires was conducted in a home-made isotherm and isopiestic CVD furnace. CH_3SiCl_3 was used as a precursor with H_2 as carrier- and dilution-gas. CVD temperature was selected in the range of 1000−1100 °C under pressures of 0.5−1.0 kPa in the deposited area. The deposited time was chosen as 1−3 h.

The morphology and structure of the CVD-product were detected using an FEI-Nova Nano 230 SEM, a JEM2100F TEM and a D/max2550 XRD, respectively. Infrared spectrum of the product was detected using an AVATAR−360−FTIR Fourier transform infrared spectrometer with a resolution of 2 cm^{-1} and a scanning range of 500−4000 nm^{-1}. Raman spectrum was detected using an ALMEGA confocal micro-Raman spectrometer with wave length of 32.81 nm.

3 Results and discussion

Figure 1 presents FESEM images of the product on C/C composite surface. As shown in Fig. 1(a), some SiC nanowires gather into a ball, some straight or curved nanowires are distributed on the surface randomly. Similar to this study, many researches using catalyst also produced such randomly-deposited SiC nanowires [12−15]. This indicates that similar nanowires can be obtained with or without catalyst. As shown in Fig. 1(b), the short aggregated nanowires, prepared using a modified CVD technique, look like some tightly accumulated Chinese chestnut shell with many thorns. As shown in Fig. 1(c), the short nanowires form an irregular multi-dimensional network. Furthermore, many nano-particles are found on the surface of nanowires, which indicates that the nanowires are favorable deposited sites for the following CVD matter. At the

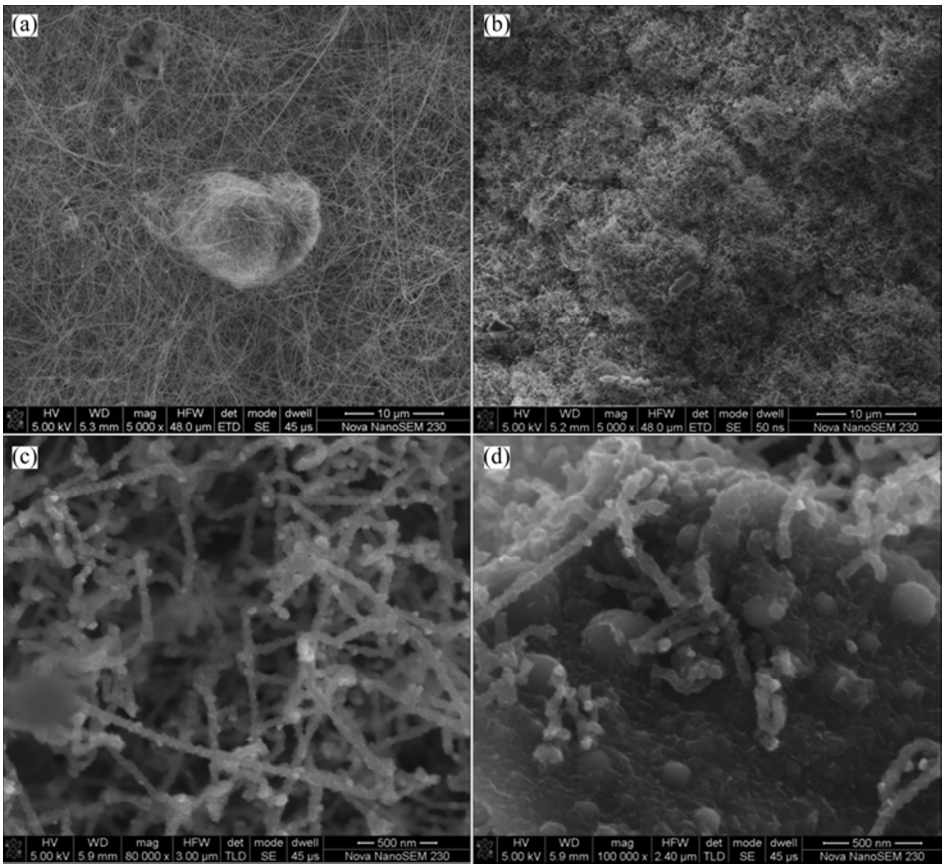

Fig. 1 FESEM images of SiC nanowires on C/C composite surface: (a) Long SiC nanowires; (b) Short SiC nanowires; (c) Irregular multi-dimensional SiC nanowire network; (d) Randomly-deposited SiC nanowires and hemispheric particles