

The dependence of the oriented growth of carbon filaments on the intensity of a magnetic field

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Abstract

Carbon filaments have been obtained by the thermal decomposition of mesitylene in the presence of a strong magnetic field. The shape dependence of the pristine carbon filaments on increasing the magnetic field is demonstrated. The proposed mechanism for filament growth involves the formation of a colloid-like system during dehydrogenation and dissociation of mesitylene. The imposed orientation of graphitic clusters (basic structural units) in the strong magnetic-field forces those located at the curved surfaces forming the tips of the filaments to expose their reactive edges, facilitating the observed preferential growth of filaments.

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1. Introduction

The magnetic properties of nanotextured carbon particles are attracting growing interest [1–4]. It is well known that small amounts of carbon are capable of destroying the strong exchange interactions between iron atoms in stainless steel. Another example is the graphite crystal ability to rotate in weak magnetic fields. This phenomenon is due to its large diamagnetic anisotropy: graphite is known as the material with the largest diamagnetic anisotropy [2]. At $B \sim 10^{-2}$ T, the magnetic-field induced anisotropy energy can balance the thermal activation energy at 300 K and rotate a graphite crystal to align the hexagonal layers in parallel with the field [5,6]. Strong paramagnetism has been reported in disordered carbon [7] with susceptibilities of $3 \times 10^{-7} \text{ m}^3 \text{ kg}^{-1}$ ($\sim 3 \times 10^{-5} \text{ emu g}^{-1}$) [2], and there

is growing experiment evidence supporting the existence of defect-related ferromagnetism in pure carbon [8,9].

Magnetic-field induced growth of filaments has been achieved for ferromagnetic nanowires (Fe_3O_4) [10]. Magnetic fields could therefore have some effect on the growth of carbon materials. Directional growth of carbon rods or nanotubes requires the use of catalytic particles or templates to direct carbon growth, otherwise spherical or unshaped carbon particles are obtained. For example, pure carbon spheres [11–15], beads [16], and onions [17], have been synthesized by very different processes such as a chemical vapor deposition process [11,12] from methane– H_2 mixtures, a hydrothermal method [13], carbonization under pressure [15,16], or by a catalytic process, i.e., from camphor vapors using ferrocene as the catalyst precursor [18].

To the best of our knowledge, the first report on magnetic-field induced growth for pure carbon particles was reported in an earlier communication on the growth of car-

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bon ‘sausages’ [19], when early results of this research were published. The current manuscript demonstrates the importance of the increase in the intensity of the magnetic field in shaping the structure of the products, and proposes a mechanism to explain the process. The preferential growth of elongated carbon particles or carbon filaments is induced by strong magnetic fields during the thermal decomposition of 1,3,5 trimethyl benzene (mesitylene), heated up to 700 °C in a closed cell at autogenic pressure, when magnetic fields varying from 1 to 10 T are applied. This synthesis route yields perfectly spherical carbon particles when carried out in the absence of a magnetic field. Preferential growth occurs at the surfaces where reactive edges are exposed due to the orientation of graphitic clusters (BSU), imposed by the strong magnetic field. The preferential growth along the field direction causes the increase in aspect ratio during growth of the carbonaceous particles, yielding the observed carbon filaments.

The obtained carbon filaments were characterized by XRD, TEM, EDX, SEM, EPR, and Raman spectroscopy. The presence of localized spins as the origin of paramagnetism is indicated by ESR measurements. X-ray and Raman measurements indicate a nanotexture in the carbon filaments that can be described as a three-dimensional disordered network of nanographitic basic structural units. The disordered regions between nanographites are rich in lattice edges, where non-bonding unpaired electrons can be the origin of the paramagnetic effects.

2. Experimental

2.1. Synthesis of carbon filaments under a magnetic field

The oriented growth of solid carbon is induced by applying a magnetic field during synthesis, using a simple chemical route developed to synthesize carbon spherules described elsewhere [19]. The synthesis was carried out at a zero magnetic field and under static magnetic fields of 1, 3, 5 and 10 T, generated using a helium-free superconducting magnet [19]. A ceramic oven containing the Swagelok filled with mesitylene was placed between the magnetic poles. The Swagelok was placed at the point in the bore of the magnet where the magnetic field is maximal, and the gradient of the magnetic field is the smallest. The closed cell was heated at 700 °C for 3 h. The reaction took place at the autogenic pressure of the precursor. The cell was cooled to room temperature, opened at room temperature and a dark black powder was collected. The yield of the product was 76% in weight according to mesitylene (C_9H_{12}) or 85% according to carbon wt.%.

2.2. Characterization

The X-ray diffraction patterns of the product were measured with a Bruker AXS D* Advance Powder X-ray diffractometer (using $CuK\alpha = 1.5418$ radiation). The carbon product morphology was observed by transmission

and scanning electron microscopy. High-resolution scanning electron microscope (HR-SEM) images were obtained using an Hitachi S4100 and the elemental composition of the material was analyzed by energy-dispersive X-ray analysis. Transmission electron microscopy was carried out on a Zeiss EM 912 OMEGA at an accelerating voltage of 120 kV. One drop of sample suspension in ethanol was placed on a carbon-coated 400 mesh copper grid and dried under air. The surface area measurements were performed by a Micromeritics (Gemini 2375) surface area analyzer. Thermogravimetric analysis was carried out under a stream of nitrogen, at a heating rate of 3 °C/min using a Mettler TGA/STDA 851. Differential scanning calorimetric (DSC) analysis of the sample was carried out up to a temperature of 550 °C in a crimped aluminum crucible, using a Mettler DSC-301 under a stream of nitrogen, at a heating rate of 3 °C/min. Electron paramagnetic resonance (EPR) spectra, a powerful tool for studying the microstructure and the electronic properties of carbon materials, were recorded on a Bruker EPR spectrometer (ER083 CS) operating at X-band ($\nu = 9.77$ GHz), with 100 kHz magnetic-field modulation. The Raman spectra were recorded in a standard Renishaw spectrometer, using the 514 nm line of an Ar laser as the excitation source.

3. Results and discussion

Fig. 1 compares carbon spherules obtained at a zero magnetic field (Fig. 1a) with carbon filaments obtained upon applying a magnetic field. The carbon spherules have a narrow size distribution, with their diameter in the range of 2.4–2.5 μm . They show a smooth surface and are solid, not hollow, as was shown previously by TEM measurements of their microtomed cross-sections [20]. These spherules were used as a substrate for the sonochemical deposition of air-stable iron on their surface [21]. Scanning electron microscope (SEM) images in Fig. 1b–e show the typical morphology of the carbon filaments at different magnetic fields. At 1 T the carbon product is composed of spherules, spheroids and some filaments (Fig. 1b). At 3 T the change in shape is very clear; instead of spherules, we observed elongated sausage-like particles, about 4 μm long and with a circular cross-section of ~ 1 μm diameter in the plane perpendicular to the long axis (Fig. 1c). These carbon ‘sausages’ or filaments obtained at an applied field of 3 T have a similar volume to the spherules and spheroids obtained at 1 T. At 5 T, the length of the filaments and the diameter in the circular section grow to ~ 15 and 2–3 μm , respectively (Fig. 1d). Further growth to a length of ~ 20 –30 μm and a diameter of 3–4 μm , is observed at 10 T (Fig. 1e). Therefore, the aspect ratios, defined as the length of the long axis divided by the diameter of the circular cross-section, increase with the magnetic field. At lower fields below 3 T, they range from 1 to more than 4. At the same time, the average volume of individual particles remains practically unchanged. At higher fields of 5 and 10 T, the aspect ratios are around 6 and the length

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