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Improved carbon nanotube growth inside an anodic aluminum oxide template using microwave radiation

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ABSTRACT

In this study, we achieved superfast growth of carbon nanotubes (CNTs) in an anodic aluminum oxide (AAO) template by applying microwave (MW) radiation. This is a simple and direct approach for growing CNTs using a MW oven. The CNTs were synthesized using MW radiation at a frequency of 2.45 GHz and power was applied at various levels of 900, 600, and 450 W. We used graphite and ferrocene in equal portions as precursors. The optimum conditions for the growth of CNTs inside a MW oven were a time period of 5 s and power of 450 W. In order to grow uniform CNTs, an AAO template was applied with the CNTs synthesized under optimum conditions. The morphology of the synthesized CNTs was investigated by scanning electron microscopy analysis. The average diameters of the CNTs obtained without the template were 22–27 nm, whereas the diameters of the CNTs prepared inside the AAO template were about 4–6 nm.

1. Introduction

Carbon nanotubes (CNTs) have unique structures and properties, such as well-defined hollow interiors, inert surface properties, and resistance to acid and base environments, which make them excellent candidates for use as supports in catalytic reactions [1–7], energy storage, and conversion media [8–11]. The special structural characteristics of CNTs greatly influence the performance of the supported particles [12–15]. Properties such as the inner diameter, wall thickness, length, crystallinity, electronic structure, size of the CNT hollow core, and electron density distinguish these materials from other species of carbonaceous materials [14,16]. In addition, CNTs have outstanding mechanical, chemical, and electronic properties, which make them among the most fascinating materials in the field of nanotechnology. The use of CNTs as fillers in composites for improving the electrical conductivity or mechanical properties requires a facile and direct approach for producing bulk quantities [6]. Extensive studies have investigated various CNT synthesis techniques, but arc discharge [17], laser ablation [18], and chemical vapor deposition [19] are the main methods employed. These widely used CNT synthesis methods have been successful despite the high production costs. These methods require feed stock gases, vacuum, high temperature chamber, inert atmosphere, and a long process time [6].

Microwaves (MW) are electromagnetic waves in the frequency range of 0.3–300 GHz [20]. It is important to understand the MW absorption properties of raw materials. MW-assisted heating is a simple, time-saving, and low-cost method that consumes less energy than the other techniques mentioned above, where this method has been used for organic [21] and inorganic synthesis [22], the preparation of catalysts [23], and in mineral treatment processes [24]. Applying direct heating via MW radiation is an early step in CNT synthesis processes. Recently, CNTs were synthesized with MW heating for 3–5 min using conducting polymers as precursors [20]. Liu et al. [25] reported a "pop-tube" technique for CNT growth using a MW oven at room temperature for 15–30 s. Nie et al. [26] synthesized CNTs by applying radiation to ferrocene for 15–20 s with single C-fibers in a MW oven. Takagaki et al. [27] used a specially designed MW oven in order to grow CNTs, where they used complicated multi-step processes before applying MW radiation to the precursor materials. MW radiation was applied in a nitrogen gas atmosphere for 5 min. Some other studies also used as MW oven as a heat source for CNT growth but they were conducted in a vacuum or inert atmosphere and the heating duration was tens of minutes [20,27–30].

The template carbonization technique is a suitable method for synthesizing precisely controlled CNTs with the desired diameter and open caps. The template serves as a structural framework within which the carbon material can be generated by carbonization from precursors. The template carbonization method involves the carbonization of an organic gas or polymer in the nano-space of an inorganic template and releasing

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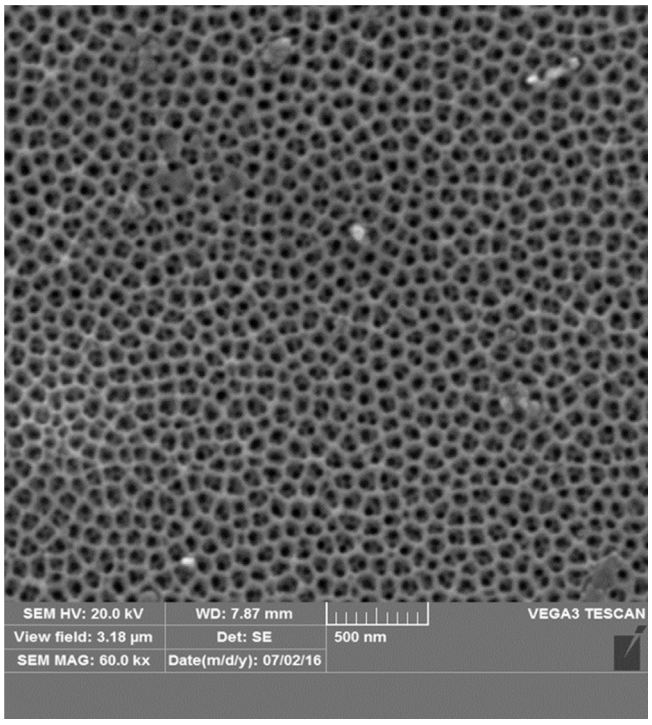

Fig. 1. SEM image of the AAO template prepared in this study.

the deposited carbon from the template [31]. Two-step anodization can be employed to obtain an anodic aluminum oxide (AAO) film from aluminum metal in an acidic medium. The uniform AAO film has straight nano-size channels with tailored length and diameter [32,33]. The porous AAO template has parallel and straight channels and a highly uniform distribution of cylindrical pores arranged in a hexagonal array. Porous AAO films are ideal for templating in CNT synthesis. The other advantages of AAO films are their mechanical and thermal stability, even at a high carbonization temperature of 900 °C in an inert gas atmosphere [34].

In this study, we synthesized CNTs inside a MW oven at room temperature and an air atmosphere as an inexpensive method. In order to grow uniform CNTs, a porous AAO template was applied to the optimally prepared CNTs. We used two alternative methods for synthesis with and without the porous AAO template, and using ironic catalysts (ferrocene and/or iron nitrate).

The novel feature of our method is the synthesis of CNTs in a MW oven with an AAO template, which facilitates the well-organized growth of CNTs in a short time period.

2. Experiments

2.1. Preparation of the porous AAO template

An aluminum plate (99.5%) was degreased in acetone and rinsed in ethanol solution, before drying in the air atmosphere. In order to obtain a mirror finish, the aluminum sheet was electropolished in a mixture of perchloric acid/ethanol solution [35], where we used the two-step anodization method. In the first anodization step, a voltage of 42 V was applied to a 0.3 M oxalic acid solution at 17 °C for 3 h. Subsequently, the AAO film was chemically etched in a mixture of phosphoric acid and chromic acid solution at 60 °C for 2 h. The second anodization step was performed under the same conditions as the first, which resulted in the formation of a highly ordered AAO template with a pore diameter and depth of 58 nm and 1 µm, respectively. a scanning electron microscopy (SEM) image of the AAO template is shown in Fig. 1.

2.2. CNT synthesis using two methods

2.2.1. Synthesis of CNTs without a template in a MW oven

Precursor materials comprising graphite rods extracted from common pencil (HB2 grade), and ferrocene (batch S6170678-Merck) were mixed in equal ratios (50:50 mg, 1:1) [20]. The mixed powder was then placed in an isolated alumina crucible and subjected to thermal MW radiation at a frequency of 2.45 GHz and various power levels (900, 600, and 450 W) for different time periods. Finally, the optimum conditions were determined. The conditions employed for the synthesis of two samples are described below.

Sample1. Equal ratios of 50 mg graphite and ferrocene were mixed well. The mixture was then placed in a closed-lid crucible and irradiated with MW radiation in three steps at power levels of 900, 600, and 600 W for 10, 5, and 5 s, respectively. We determined the most suitable time and power level for the synthesis of the CNTs by trial and error. Fig. 2a shows an SEM image of the CNTs synthesized in this process.

Sample 2. The same precursors were irradiated in one step at 450 W in an open-lid crucible for 5 s and the CNTs obtained in this process are shown in Fig. 2b. We found that the optimum conditions for the synthesis

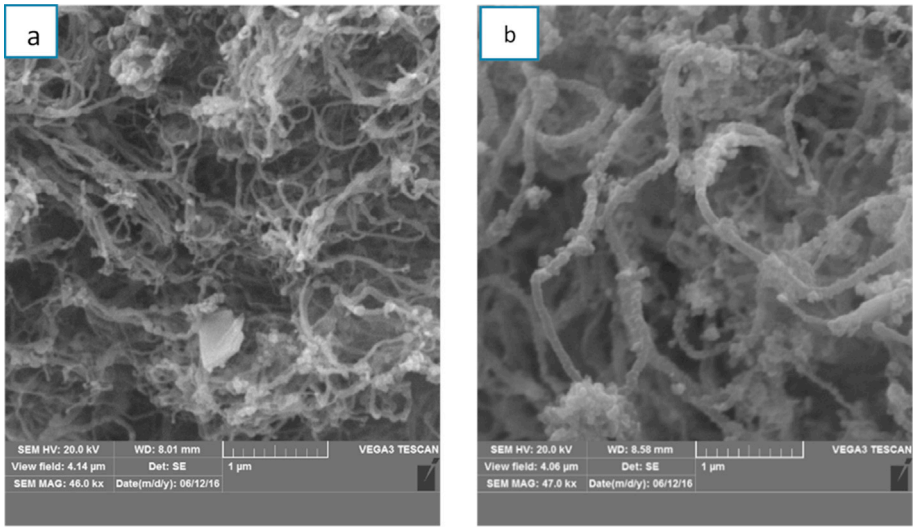

Fig. 2. SEM images of the synthesized CNTs in: (a) sample 1 and (b) sample 2.