



# Architecting graphene nanowalls on diamond powder surface



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## ARTICLE INFO

### Article history:

Received 10 October 2014

Received in revised form 2 December 2014

Accepted 4 December 2014

Available online 12 December 2014

### Keywords:

A. Nano-structures

A. Metal-matrix composites (MMCs)

B. Thermal properties

D. Electron microscopy

## ABSTRACT

Graphene nanowalls (GNWs) have been synthesized on diamond particle surface by a simple heat-treatment process in vacuum induction furnace. The thickness of the as-grown GNWs is in the range of 20–40 nm, and most of the nanowalls are smooth and flat. The growth of GNWs is affected by the type of catalyst and treatment temperature, and the mixed catalytic effect of Fe and Ni is better than Fe or Ni respectively. High-density GNWs was grown on the diamond particle entire surface, when the heat treatment process is performed at 1473 K. Al/diamond composites with high thermal conductivity of 423 W/(m K) was obtained, which was achieved by the formation of large number of GNWs on the diamond particle surface. Systematic analyses reveal that the growth models such as classic precipitation upon cooling (PUC) model are not applicable to explain this kind of GNW's growth mechanism. Hereby, an extended PUC model is proposed to interpret the GNW's growth process on diamond surface.

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## 1. Introduction

Two dimensional carbon nanostructures such as GNWs and carbon nanowalls have attracted much attention due to its potential application in different fields of science and technology owing to its exceptional high thermal conductivity and electrical conduction velocity [1–5]. Diamond features the extraordinary properties of high thermal conductivity, good resistance to corrosion and electrical insulator. Covering diamond with GNWs is considered to be a very promising way for fabricating super-high thermal conductivity materials and excellent electrochemical electrode [6].

In the last several years, large amount of experiment studies have been performed on two-dimensional GNWs [7–10], and a great progress has been obtained up to now. The main reported methods for preparation of GNWs so far are various kinds of chemical vapor deposition (CVD), in which carbon source gas such as CH<sub>4</sub> is used and hydrogen dilution is necessary in the synthesis of GNWs. Pejman et al. reported a microwave plasma method for the preparation and deposition of carbon nanowalls in a traditional microwave oven [11]. Wu et al. reported that they synthesize carbon nanowalls by microwave plasma CVD [12]. Dikonimos et al. grown carbon nanowalls in a hot filament CVD reactor on Si

substrate [13]. Marcel et al. fabricated carbon nanowalls by atmospheric direct-current plasma enhanced CVD method [14]. Shiji et al. reported the fabrication of carbon nanowalls using radio-frequency plasma-enhanced CVD [15].

In this paper, we present a simple method to grow GNWs on diamond powder surface in vacuum induction furnace without the decomposition of source gases. The effects of processing on the resulting morphology, along with other characterization techniques have been used to understand the graphene structure. Given its simplicity nature, it can be performed in most laboratories by using a conventional vacuum carbon tube furnace, without the precise control of the reaction process.

## 2. Experimental

The growth of GNWs on diamond surface was carried out in vacuum induction furnace. Diamond particles and the mixture of iron and nickel particles were used as solid carbon source and catalyst respectively. The mixture of diamond, iron, and nickel powders was used as raw material which consists of 200 μm diamond particles, reduced iron powders in size of 20–30 μm and nickel powders with particle size in the range of 1–5 μm. The mass fraction of the mixture was Fe-20 wt% and Ni-20 wt%. The mixed raw powder materials were first set in a graphite crucible and then placed in the vacuum induction furnace that was pumped to a base pressure of about 10<sup>-3</sup> Pa. The powders mixture was then heat up to 1473 K and kept for 2 h.

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After cooling down, the powders was rinsing in dilute hydrochloric for 4 h to remove the redundant iron and nickel powders. And then the treated diamond powder was cleaned in alcohol, dried and kept in desiccators. Subsequently, diamond powders with GNWs were mixed with a designated amount of 54 vol% Al powder to prepare Al/diamond composites. These composites powders were heated to 993 K within 72 min and held for 1 h in vacuum sintering furnace [16].

Various analysis technique including SEM, TEM, Raman spectra and XPS were employed to investigate the as-grown GNWs on diamond surface. For TEM specimen preparation, GNWs/diamond particles were dispersed in ethanol and ultrasonically vibrated for 10 min. As the ultrasonic collision among diamond, a few of GNWs broke and suspended in the ethanol, and these suspending GNWs were collected for TEM analysis. Raman spectra were collected with a Renishaw Raman microscope (532 nm laser excitation wavelength). Thermal diffusivity of the composite samples was measured by a laser flash apparatus (LFA457, NETZSCH, Germany) with an international standard. The specific heat ( $C_p$ ) of the specimen was measured using a differential scanning calorimeter with reference materials of single-crystal alumina under argon gas. The thermal conductivity could be derived from the equation: Thermal conductivity  $\lambda = \alpha \times C_p \times \rho$ .

### 3. Results and discussion

#### 3.1. Microstructure of GNWs/diamond particles

Fig. 1a and b are typical SEM images taken from diamond surface revealing the GNWs stood on diamond powder surface. The thickness of these two-dimensional carbon sheets is in the range of 20–40 nm. Almost all of the nanowalls are smooth, vertically and uniformly distributed on diamond surface, which is different from the crumpled and folded sheets reported in the literatures [17,18]. The final thickness of the graphitic layers depends on the treatment temperature, the species of the catalytic metal and different diamond orientation. Our results using Fe/Ni on the different diamond orientation proves that a lower catalytic reactivity

on a {100} diamond surface. Also the result proves that the catalytic effect of Fe and Ni with Ni/Fe ratio of 1 is much larger than catalytic reactivity of using Fe or Ni as catalyst respectively. And when the heat-treatment temperature is higher than 1473 K, the graphene layer turns out to be spherical nanoparticles, which has been referred to in the literature [19].

Fig. 1c and d shows a representative high-resolution TEM and TEM images of a graphene sample. Fig. 1d indicates that the graphene sheet with a size scale of several hundred nanometers is flat, smooth, which is consistent with SEM results. Further information on the nature of the graphene sheet on the diamond particle surface is provided by HRTEM in Fig. 1c. The number of graphene layers is in the range of 2–10 layers on different locations of the sample.

The Raman spectra of the GNWs grown on diamond are shown in Fig. 2. The peak at  $1352\text{ cm}^{-1}$  and  $1582\text{ cm}^{-1}$  of the Raman spectra of GNWs are assigned to typical D band and G band of GNWs respectively [20–22]. The D band is usually thought to be associated with the dispersive, defect-induced vibrations. The G band originated from the in-plane vibrational mode, and points to the existence of  $\text{sp}^2$  crystalline domains. The intensity ratio of the D band to the G ( $I_D/I_G$ ) is related to the in-plane crystallite size of graphene [23]. The ratio of around 0.6 for GNWs grown on diamond particles as shown in Fig. 2 indicates that the average crystallite size in the GNWs is 20–30 nm. This is consistent with reported domain sizes for standing multiple-layer GNWs [24,25]. The G band peak is accompanied by a shoulder peak at  $1618\text{ cm}^{-1}$  (D' band), which is associated with finite-size graphite crystals and graphene edges. The 2D can also be observed at  $2695\text{ cm}^{-1}$ , which is widely used in literatures to estimate number of layers in the graphene samples. It is noted that there is a diamond peak at  $1332\text{ cm}^{-1}$  beside the D band.

The corresponding XPS studies have been employed to reveal the nature of the carbon and oxygen bonds present in the GNWs samples (Fig. 3). As revealed in Fig. 3a, the C1s peak can be perfectly deconvoluted into four peaks by Gaussian fitting. The main peak centered at a binding energy of about 284.6 eV is assigned to graphitic carbon, and the secondary intense peak centered at around 285.3 eV corresponds to  $\text{sp}^3$  hybridization [26]. The  $\text{sp}^3$

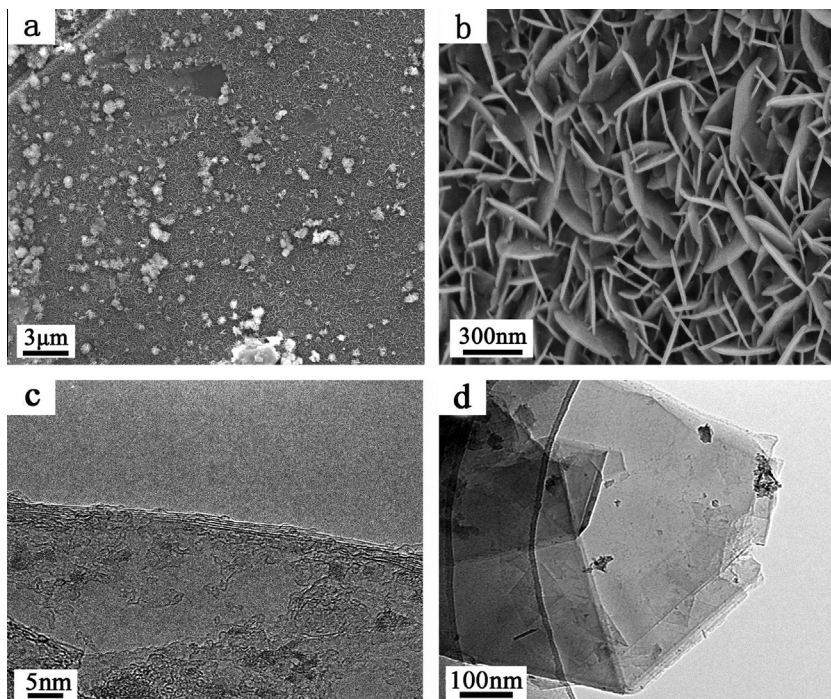


Fig. 1. (a, b): SEM images of GNWs grown on diamond surface. (c, d): HRTEM and TEM images of GNWs grown on diamond surface.

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