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Fabrication of graphene and graphite thin films from organic coating

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ABSTRACT

Monolayer graphene and graphite thin films were fabricated on SiO_2/Si substrates by organic coating and post annealing. Pure nickel (Ni) was deposited on the substrate surface as the catalyst. Then the samples were dipped in the Orange II organic solution. Raman spectroscopy measurements suggested that the Orange II organic solution resolved on the Ni surface and formed graphene and graphite thin layers during the heating process.

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1. Introduction

Atomically thin carbon film is one of the hottest materials in various fields. Graphene is a single layer of graphite that has excellent physical properties [1–5]. It is a two-dimensional honeycomb lattice material bonded with a single layer of sp^2-hybridized carbon atoms. This material is known as the mother of carbon nanotube (CNT) and fullerenes. The first report of monolayer graphene was given by Nobselov et al. [1], the Nobel Prize work of 2010. It is claimed that graphene has large carrier mobility of about 200,000 $cm^2 V^{-1} s^{-1}$ at room temperature (RT), which is one hundred times as that of Si. Studies on the device applications of graphene are in progress [6–8]. Graphene has high visible optical transmittance, and is expected to replace ITO as a transparent electrode [7]. Furthermore, graphene is a pure carbon material with abundance of resources, low environment impact, and safety in use. However the stable supplying method of graphene has not been fixed yet. There have been reported several method to growth graphene, such as a scotch tape method (mechanical exfoliation) [1], chemical vapor deposition (CVD) [8–10], epitaxial growth [11]. Although the production by CVD is the present mainstream, long growth time and poor crystallinity are still problems. In this paper, we report a simple process of organic solution coating to form graphene and thin graphite films. Our results may provide a way to produce graphene layer with low coat.

2. Experimental

Si(100) wafer with 250–300 nm thick oxide layer on its surface was used as the starting material. After chemically cleaning of the Si wafer, pure nickel film was deposited on the oxide layer as the catalyst [12,13]. The background pressure of the deposition chamber was about 10^{-4} Pa. Afterwards, the Si wafer was dipped in an Orange II: acetone saturated solution. The Orange II (Acid Orange 7, $C_{16}H_{11}N_2NaO_4S$) was coated on the Ni/SiO_2/Si surface by the dipping, as shown in Fig. 1. Then, the sample was annealed in vacuum at temperatures from 450 °C to 1050 °C by an IR lamp. The annealing time was changed from 2 min to 5 min. The fabricated graphite thin films were investigated by Raman spectroscopy (NRS-3200, JASCO). The Raman spectroscopy was used to check of graphite and/or graphene layers. Two Raman peaks were known for graphite materials, which were called as D-band and G-band. An intense G-band indicates a high crystallinity of the graphite layer. The G/D intensity ratio is a factor of characterizing the graphite crystallization. Furthermore, an intense 2D-band at about 2700 cm^{-1} indicates the formation of single layer graphene [14–16]. A field-emission scanning electron microscope (FE-SEM, Carl Zeiss) and an atomic force microscope (AFM, SII) were used to evaluate the graphene surface.

3. Results and discussion

Fig. 2 shows the Raman spectra of the samples annealed at difference temperatures. The thickness of deposited Ni used in these samples was 100 nm. For the annealing temperature of 450 °C the D-band and G-band peaks at wavenumbers of 1350 cm^{-1} and

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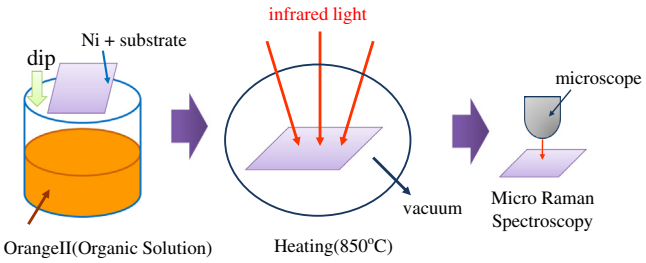

Fig. 1. Fabrication process of organic coating samples.

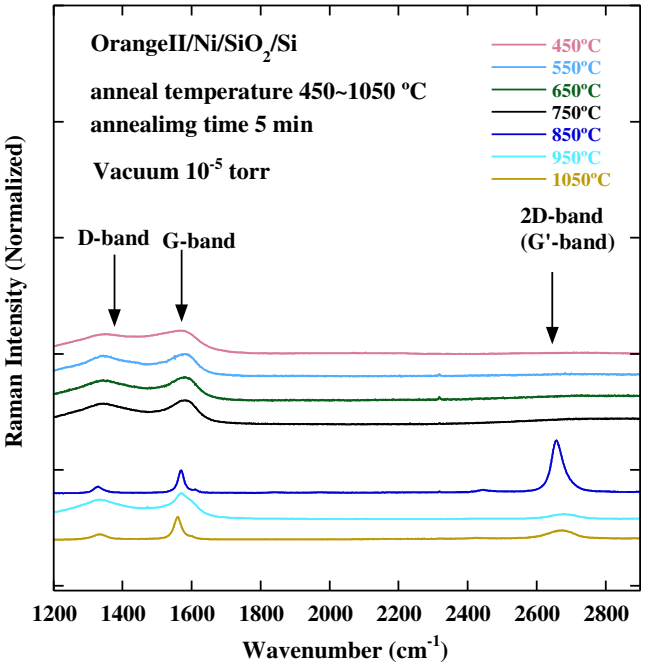

Fig. 2. Raman spectra of samples at different annealing temperatures.

1550 cm^{-1}, respectively, were broad, which indicate the amorphous carbon formation on the sample surface due to the thermal decomposition of the Orange II. Azo groups of Orange II decomposed at temperatures of 450 °C or lower. These peaks became sharper with the increased annealing temperature. At annealing

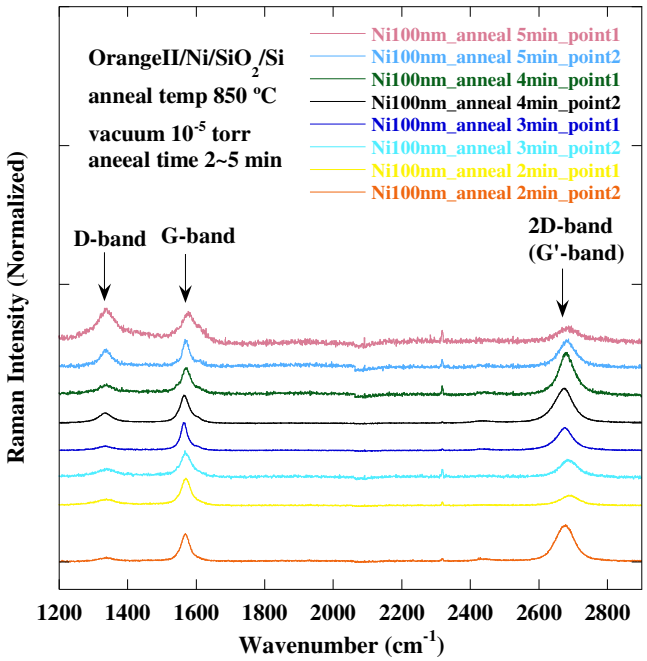

Fig. 4. Raman spectra samples of different annealing time.

temperature of 850 °C, an intense and sharp peak at the 2D-band position occurred, indicating the graphene formation. The growth process of graphene in this work was considered as follows: the Orange II decomposed at low temperature; then the carbon atoms diffused into melted Ni; afterwards, the graphene formed at the cooling process. At higher annealing temperature than 850 °C, the 2D-band became weaker and/or disappeared. This might be due to the graphite formation. In Fig. 3, we show the AFM image of a 850 °C annealed sample. A commercial graphite (highly ordered pyrolytic graphite, HOGP) sample was used as a comparison. The totally similar images were obtained. The measuring area was on the top of polycrystalline Ni film, the formation of graphene was strongly suggested. Since the ratio of 2D/G exceeds 2, a single layer graphene was obtained. More carbon out diffusion from Ni might cause the increase of layer thickness, resulting in graphite and/or amorphous carbon formation, which can be seen in the case of high temperature annealing. Fig. 4 shows the Raman spectra of samples prepared by changing the annealing time from 2 min to

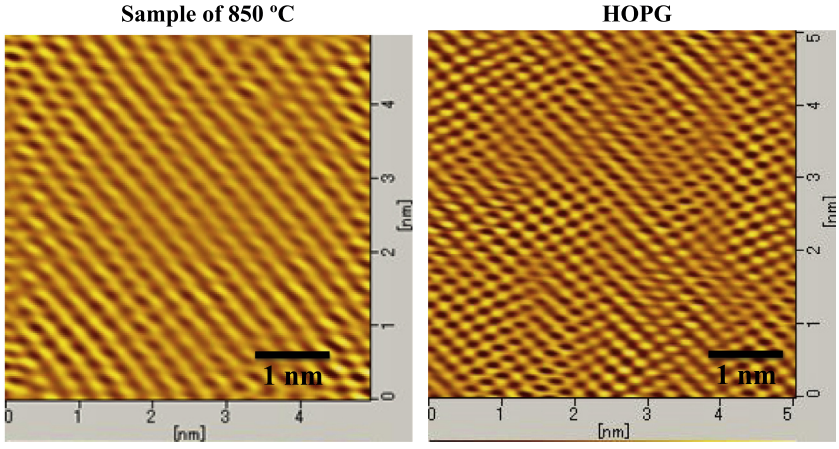

Fig. 3. AFM atomic images of graphene and HOPG.

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