Electron beam processing of 6H-SiC substrate to obtain graphene-like carbon films

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Abstract. We report on growth of graphene-like carbon films on 6H-SiC {0001} substrate by electron-beam. The processing was carried out on a specialized electron beam system with the Pierce electron gun. The D, G, and 2D peaks as well as D/G (0.2-0.9) and 2D/G (0.7-0.9) ratios are detected on processed samples by Raman spectroscopy. The prominent bands D, G, and 2D are located at 1350, 1584, and 2707 cm$^{-1}$, respectively. Atomic force microscopy showed that the average roughness lies in the range from 5 to 30 nm, and ten point height – from 40 to 200 nm. The results demonstrate that the electron-beam technique is appropriate to form graphene-like structures directly on 6H-SiC substrates and could be used for electronic device fabrication.

1. Introduction

The ever-growing development of technologies brings demands for discoveries of new physical and chemical phenomena, design and fabrication of multifunctional materials, and manufacturing of next-generation electronic devices [1-5]. Over the past few decades, carbon materials have been attracting a growing research interest for variety of applications [5-8]. Graphene has tremendous perspectives due to its unique properties including high electron mobility with low electronic scattering that make it a very promising candidate for next generation nanoscale electronics and photonics devices [1,9-12]. Therefore, achieving quality synthesis of graphene, graphene-like carbon films, and related materials is in demand.

Several outstanding advancements in graphene fabrication technology have been explored including research grade mechanical exfoliation of graphite [2], chemical vapour deposition [13], thermal decomposition of SiC [11], and chemical methods [14]. Among the various methods of its obtaining the most interesting is the synthesis directly on silicon carbide substrates [5], e.g. thermal decomposition [9,10,15]. However, this high-temperature treatment prevents commercial use of SiC substrates for large-scale production of graphene-based electronic devices.

Recently, various approaches using irradiation technique or particle beam (laser, ion and electron beams) were extensively paid attention to due to high precision, efficiency, mature control method, and fast speed [5,16-18]. A method for converting the area of 6H-SiC substrates into homogeneous epitaxial graphene by axial electron beam irradiation is proposed [5,16]. 6H-SiC has a hexagonal structure and
represents an ideal pattern for graphene growth. One of the main advantages over other methods is SiC decomposition, i.e. graphene layers are formed on a semiconductor substrate, so no transfer is required to process the device. In localized interaction, scattering, excitation, and ionization between the primary electrons and the SiC surface lead to SiC bond rupture, and the excess electron energy is dissipated as heat, which leads to selective sublimation of the Si, contributing to the formation of epitaxial graphene. Using I-shaped electron beams \cite{17,18} instead of axial ones to produce graphene can have obvious advantages, such as suitability for large-scale graphene fabrication.

2. Experimental

Semi-insulating 6H-SiC wafer was used as a substrate. The substrates were cleaned in acetone and isopropyl alcohol prior to placing into the chamber. Then electron beam processing (EBP) was carried out on a specialized electron beam vacuum system with the Pierce electron gun \cite{17}. The chamber was evacuated to the pressure of 0.3-0.5 mTorr. The emission current was fixed for and finely tuned between 50 and 180 mA. Since the beam energy and irradiation time are precisely controllable, the corresponding temperature ranged from 1680 to 2000 K \cite{18,19}. The as-grown layers and initial substrate surfaces on the reference sample were characterized by Raman spectroscopy and atomic force microscopy.

3. Results and discussion

A series of {0001} 6H-SiC samples were electron beam processed and nanostructured carbon films were available on the sample surfaces. Raman spectra of some samples are shown in Figure 1.

The D, G, and 2D peaks as well as D/G (0.2-0.9) and 2D/G (0.7-0.9) ratios are detected by Raman spectroscopy. The prominent bands D, G, and 2D are located at 1350, 1584, and 2707 cm\(^{-1}\), respectively. The full-width-at-half-maximum (FWHM) of 2D peaks is in the range of 61-81 cm\(^{-1}\). It is widely reported that the FWHM of the 2D band for the tri- and tetra-layers graphene grown on SiC is ~ 60 and 85 cm\(^{-1}\), respectively, whereas for the single and bilayer epitaxial graphene, it is found to be 30 and 45 cm\(^{-1}\), respectively \cite{20-22}. The dominant G band intensity indicates good quality of graphene. The results of Raman spectroscopy of all samples are presented in Table 1.

The surface morphology of the 6H-SiC samples processed by electron beam was effectively examined by AFM with semi-contact mode (Fig. 2). These images show several types of morphology. The average roughness lies in the range from 5 to 30 nm, and ten point height – from 40 to 200 nm (Fig. 2a and 2b, respectively). Figure 2c demonstrates terraces of 300-1100 nm in width with step height of 150-750 nm.

![Figure 1. Raman spectra of reference (black line) and electron beam processed (coloured lines) 6H-SiC samples.](image-url)
Figure 2. AFM images of electron beam processed 6H-SiC samples. Scan area: (a), (b) 3×3 µm², (c) 1×1 µm².

Table 1. The results of Raman spectroscopy.

| Sample | FWHM D (cm⁻¹) | FWHM G (cm⁻¹) | FWHM 2D (cm⁻¹) | D/G | 2D/G |
|--------|----------------|----------------|-----------------|-----|------|
| 1      | 48             | 72             | 81              | 0.7 | 0.9  |
| 2      | 11             | 56             | 76              | 0.2 | 0.7  |
| 3      | 22             | 50             | 62              | 0.4 | 0.8  |
| 4      | 15             | 43             | 61              | 0.4 | 0.7  |

Comparison of the obtained results with known Raman spectra and atomic force microscopy data [9,10,16] allows us to assume that nanostructured carbon films, from monolayer to multilayer graphene and turbostratic graphite with various degrees of defects, are formed under the conditions of electron beam processing.

4. Conclusion
Distinct nanostructured carbon films have been synthesized on {0001} 6H-SiC by electron beam processing. The observed FWHM of the 2D band at 60 and 85 cm⁻¹ on Raman spectra, as well as the average roughness in the range from 5 to 30 nm obtained by AFM, allows us to assume that nanostructured carbon films, from monolayer to multilayer graphene and turbostratic graphite with various degrees of defects, are formed under the conditions of electron beam processing. It is expected that further optimization of processing conditions will allow producing the required number of layers and quality of graphene.
The results could be utilized to optimize the processing conditions in order to obtain single-layer and double-layer graphene films with good electrical properties. The preparation of graphene on silicon carbide substrates by electron beam processing should be a promising technique in carbon electronic device fabrication.

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