Fabrication of Planar-type Nano Structures on thin Graphite layer using Focused Ion Beam 3-D Etching Technique

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Key words: Nonlinear characteristics, Planar-type structures, focused ion beam, anisotropic.

1. Introduction

In this paper, we are reporting about the fabrication of planar-type nano-structures (along *ab*-plane and *c* direction) on thin graphite layer by using focused ion beam (FIB) 3-D etching technique. We have fabricated several in-plane areas of planar-type nanostructures/patterns on thin graphite layer (thickness ~ 500 nm) using FIB. Those in-plane area sizes were 6 μm x 6 μm, 6 μm x 4 μm and 6 μm x 2 μm. The *c*-axis stack with the height of several nanometers was also fabricated. The electrical transport characteristics were studied for these fabricated structures. We have observed nonlinear (curve-like) anisotropic transport behavior from the current *(I)* - voltage *(V)* characteristics which have shown a clear transition from an ohmic behavior at 300 K to curve-like nonlinear characteristics below 110 K for *ab*-plane and *c*-axis stack. A clear nonlinear characteristics has been observed at 25 K. The curves observed in resistance *(R)* - temperature *(T)* and *I-V* characteristics of the *ab*-plane and *c*-axis stack strongly resemble this transition behavior. These results show the superiority of graphite-nanostructures for futuristic graphite-based nonlinear electronic devices.

Graphite is a three dimensional (3-D) material which has a sheet-like layered structure where the carbon atoms all lie in a plane and are only weakly bonded to the adjacent graphite sheets [1]. It is normally a basic material for all above carbon allotropes. Recently, the research on graphite materials such as two-dimensional graphene (single atomic layer of carbon), zero dimensional fullerenes (C60) and carbon nanotubes have attracted much attention by their unique properties for micro and nano-electronic applications. Particularly, graphene becomes an active replacement material for silicon which is being used heavily in semiconductor industries nowadays [2]. Each sheet has hexagonal lattice of carbon bonded by strong σ bonding (sp^2) in the *ab*-plane. The perpendicular π-orbital electrons along the *c*-axis are responsible for *ab*-plane conductivity [3]. In this paper, we report a detailed fabrication technique for planar-type nano-structures on thin graphite layer and their nonlinear characteristics observed below 110 K for *ab*-plane and *c*-axis stack fabricated by using focused ion beam. In general, non-linear electronic devices such as diodes, bipolar junction transistors (BJT's), and field effect transistors (FET's) are described in terms of their nonlinear *I-V* curves.

Recently, these devices have been developed with respect to low noise, low power, and high electron mobility transistor applications [4]. Their electronic transport properties present remarkable scientific and technological potential.

The studies on bulk graphite have been investigated for many years, however there has been no work reported on the fabrication of planar-type nanostructures on thin graphite layer using focused ion beam. As well as the observation of nonlinear characteristics have not been ever reported elsewhere. Thus, our research has focused primarily on the fabrication of nano-structures and their nonlinear *I-V* characteristic studies.

2. Experimental Methods

In this study, we used thin graphite crystallites extracted from highly ordered pyrolytic graphite (HOPG) using the mechanical exfoliation technique, as this method had been shown to form perfect crystallites [5]. Fig. 1. shows the SEM image of exfoliated graphite layer on Si/SiO_2 substrates. We have fabricated planar-type nanostructures or pattern (along *ab*-plane and *c*-axis) on thin graphite layer (thickness ~ 500 nm) using focused ion beam (FIB) 3-D etching technique. Those in-plane area sizes were 6 μm x 6 μm, 6 μm x 5 μm and 6 μm x 2 μm. These in-plane areas were etched by the tilting the sample stage by 30º anticlockwise with respect to ion beam and milling along *ab*-plane (ref. Fig.2a.) The *c*-axis stack with height of several nanometers was fabricated (Fig.3) by rotating the sample stage by an angle of 180º and then tilted by 60º anticlockwise with respect to ion beam and milled along the *c*-axis (ref. Fig.2b). The schematic diagram for this FIB fabrication is shown in Fig.2. The fabricated *c*-axis stack size was W = 2 μm, L= 1 μm, H = 200 nm which is shown in Fig. 3. The schematic picture of stack arrangement in graphite layer is shown as inset in Fig. 2.

The electrical transport characteristics were performed for both *ab*-plane and *c*-axis stack structures using four-probe contact measurement by using closed-cycle refrigerator system. These nano-fabrication etching details were reported in detail by Kim S J *et al* [6].

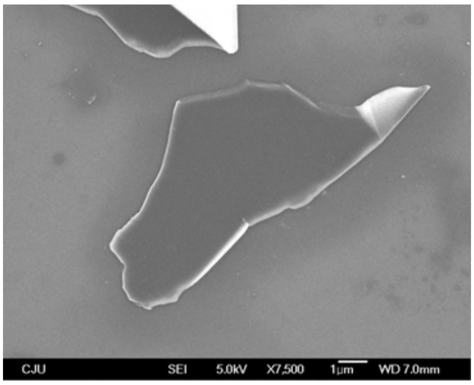

Fig. 1. SEM image of a thin graphite flake on Si/SiO2 substrate exfoliated from bulk graphite.

3. Results and Discussion

In Fig. 3. (upper inset) represents the resistance (R) – temperature (T) characteristics of *c*-axis stack which shows semiconducting behavior till 50 K and then metallic behavior below 50 K. This is well agreed with previous theory reported by Matsubara *et al* [7]. Below 50 K, the impurity-assisted interlayer hopping conduction combined with scattering of carriers can be responsible for the mettaliclike behavior.

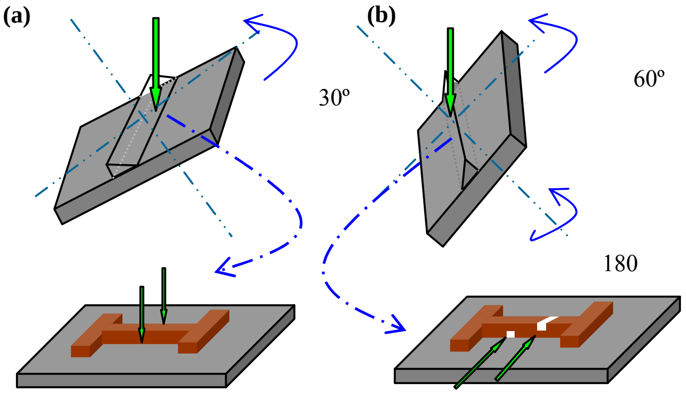

Fig. 2. The schematic diagram of FIB process for *ab*-plane and c-axis etching. (a) Initially, sample stage tilted for 30º for *ab*-plane etching. (b) The sample stage rotated by an angle of 180º and also tilted by 60º anticlockwise with respect to ion beam and milled along the *c*-axis.

Above 50 K, thermal excitation of carriers plays a major role for semiconductorlike temperature dependent behavior. Most noticeably, we observed an linear-ohmic behavior at 300 K and the same has been turned into nonlinear curve-like characteristics below 110 K from *I-V* characteristics.

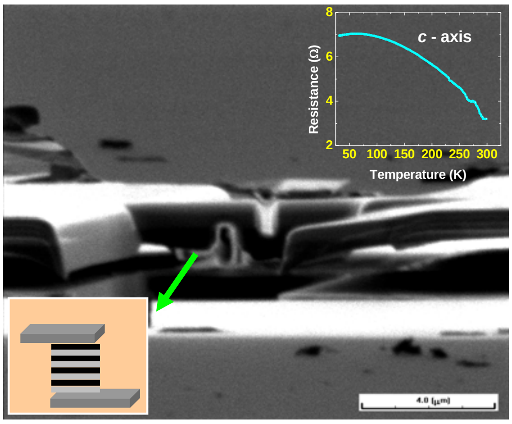

Fig. 3. The FIB image of *c*-axis stack fabricated on graphite layer. The stack size was W = 2 μm, L = 1 μm, H = 200 nm. Inset (left bottom) shows the schematic diagram of stack arrangement along the *c*-axis. Inset (top right) shows the *R-T* characteristics of *c*-axis stack.

However the fabricated in-plane structures exhibit a typical metallic behavior similar to the characteristics observed for bare graphite flake. Interestingly, we have noticed a kink in the *R-T* curves between the temperatures 40 to 60 K for in-plane fabricated structures. The temperature where the kink-structure noticed, is varied with respect to the sample resistivity. This small upturn in resistance at low temperature is not yet understood but it is either due to electrode contact or contact of electrode to the graphite flake. Also, we ensured that there is no substrate-induced effect behind this kink formation.

In Fig. 4. we show the *I-V* characteristics of the 6 μm x 2 μm size planar-type structure. Similar to *c*-axis stack, the fabricated in-plane structures also shown a linear ohmic behavior at 300 K which turns into nonlinear behavior below 110 K.

A clear nonlinear characteristic has been observed at 25 K. We propose that below 110 K, there is a rapid decrease in effective charge carriers which could stimulate the ohmic behavior to nonlinear characteristics. Most noticeably, a symmetry in the *I-V* curves has been observed.

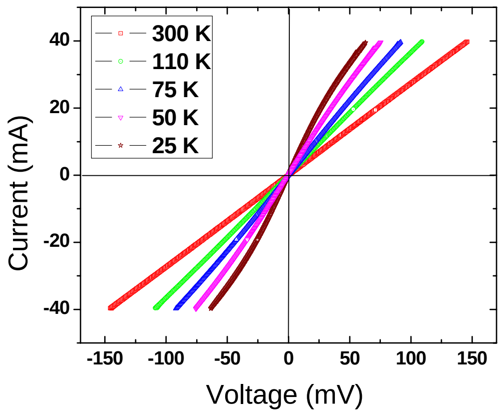

Fig. 4. The *I-V* characteristics of 6 μm x 2 μm size planar-type structure shows linear-ohmic behavior at 300 K and the same is turned into nonlinear curve-like characteristics at 25 K.

As the *ab*-plane and *c*-axis stack show their respective metallic and semiconducting transport characteristics, the transition from linear to nonlinear behavior in their *I-V* curves clearly indicates their respective behavioral directionality. We also observed similar characteristics as well for all other fabricated planar-type structures of sizes 6 μm x 6 μm and 6 μm x 4 μm.

The *ab*-plane and *c*-axis stack transport results were compared from which we declare that the *c*-axis stacks behave as a high barrier to charge carrier tunneling. This is because of the high resistance generated by weakly bonded adjacent layers in the stack [1]. The detailed size-dependent nonlinear characteristics will be discussed further. Our observation of this linear-to-nonlinear transport behavior opens the road to a new generation of graphite-based nonlinear electronic devices.

ACKNOWLEDGEMENT

We gratefully thank Prof. H.– J. Lee, POSTEC, Korea for supplying graphite material for our research and his valuable suggestions.

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