Local conductance measurement of few-layer graphene on SiC substrate using an integrated nanogap probe

M. Nagase, H. Hibino, H. Kageshima and H. Yamaguchi

NTT Basic Research Labs., Nippon Telegraph and Telephone Corp., 3-1, Morinosatowakamiya, Atsugi, Kanagawa 243-0198, Japan

nagase@aecl.ntt.co.jp

Abstract. We report the first measurement of spatially resolved in-plane conductance of few-layer (one- or two-layer) graphene grown on a SiC substrate, measured using an integrated nanogap probe. The morphology and number of layers of the thermally grown graphene were confirmed by in-situ observation using low energy electron microscopy (LEEM). The gap current (conductance) images were measured using an integrated nanogap probe with a 30-nm-gap on a conventional SPM system in vacuum. Island shapes with a typical width of 30 nm were clearly observed in the conductance image. Single- and double-layer graphene islands could be clearly distinguished, because the conductance of double-layer graphene is about four times that of single-layer graphene. The layer number of few-layer graphene has been successfully estimated from the electrical transport measurement using the integrated nanogap probe.

1. Introduction
Few-layer graphene has recently attracted much attention as a new electronic material with interesting electronic transport properties, such as field effects and quantum hall effects [1, 2]. There have been many theoretical discussions about how the electronic structure of graphene depends on its microscopic geometry [3]. However, it is very difficult to measure the electrical properties of the nano-order graphene assumed by theorists. A fabrication technique that offers atomically precise control of graphene shape has not been established [4]. We try another method based on scanning probe microscopy (SPM) to measure the electronic transport properties of nano-order graphene instead of the conventional method, which involves the fabrication of a nanodevice. In the method, a local conductance of few-layer graphene on SiC substrate is measured using an integrated nanogap probe consisting of two Pt electrodes separated by a nano-order gap fabricated by focused ion beam milling of a Si cantilever. This integrated nanogap probe on a SPM system enables us to measure in-plane conductance with nanometer resolution without lithographic techniques [5, 6]. Our goal in this work was to measure the local conductance of few-layer graphene with nanometer spatial resolution. In this paper, we report, for the first time, spatially resolved in-plane conductance of one- or two-layer nanographene grown on a SiC substrate.

2. Experimental setup
We used nominally flat 6H-SiC(0001) wafers (n-type, N-doped, 0.02-0.2 Ωcm) as starting materials. The samples were chemically cleaned and introduced into the UHV chamber of a low energy electron
microscopy (LEEM) apparatus through the load lock. The base pressure of the LEEM was less than \(7 \times 10^{-9} \text{ Pa}\). The samples were annealed by the electron-beam bombardment from the back side. The graphene coverage was monitored by LEEM [7] and was controlled to be relatively low for the formation of nano-order islands. The holder temperature measured by thermocouple was 1093°C, which was lower than a real sample temperature. The coverage of single-layer graphene was 24% and that of double-layer graphene was 2% for the sample described in this paper.

The sample surface after thermal treatment was observed using a nanogap probe and a Si point probe on a conventional SPM system (E-sweep/SPI400: SII-NT). Figure 1(a) shows a schematic of our nanogap probe. A Pt electrode on the tip of Si cantilever (3 N/m) was divided into two parts by using FIB milling [6]. The gap distance between the electrodes was 30 nm. The nanogap probe was mounted on the modified cantilever holder of the SPM system. Since the probe was mounted with a tilt angle, the top edges of the probe electrodes made contact with the sample surface. The X-direction in images is the longitudinal direction of the cantilever as shown in Fig. 1(a). Conductance measurements were performed in a vacuum \((3 \times 10^{-4} \text{ Pa})\) at -137°C [Fig. 1(b)]. The estimated load force for the conductance imaging and measurements was about 700 nN. The gap current was measured with the current amplifier of the SPM system. The gap voltage was applied using a source meter (Keithly 6430). The applied voltage for the nanogap was 20 mV for all current images in this paper.

![Figure 1](image.png)

**Figure 1.** (a) Schematic of the integrated nanogap probe on a sample surface. A Pt electrode is separated by nanogap fabricated by FIB milling. (b) Experimental setup: integrated nanogap probe on a SPM system.

### 3. Conductance measurement of few-layer graphene

A sample surface before thermal treatment was covered with step-trace structures defined by the offset angle of the sample surface. Surface atoms on the SiC substrate were partially desorbed by the high-temperature treatment in UHV. Figure 2(a) shows the sample surface morphology after high-temperature treatment as observed using the Si point probe. The dark area in trace was created by the desorption of surface atoms on the SiC substrate. Since a desorption rate of silicon is higher than that of carbon, the SiC surface after heating is carbonized. At the first step of graphene growth by thermal treatment, graphene tended to grown on the step edge and around the hole. The morphology and number of layers of graphene nanoislands (nanographene) were confirmed by in-situ observation using LEEM and controlled by the thermal treatment conditions [7]. We used a sample with relatively low graphene coverage in this experiment. Nano-order graphene islands were formed on the SiC substrate as shown in the LEEM image of Fig. 2(d).

Figure 2(b) shows the topographic image measured using the gap probe. Because the width of the electrodes (~1 µm) of the gap probe is relatively large compared with the structures on the carbonized SiC surface, the detailed structures are not visible. Despite the lack of detail in the topographic image, the nano-order islands are observed in the gap current image as shown in Fig. 2(c). Figure 2(e) is a magnified image of region in the box in (c). Figure 2(e) shows an outline sketch of the conductive
island in Fig. 2(d). The island shape and size coincide with the LEEM result shown in Fig. 2(d). Graphene islands are observed as dark contrasts in the LEEM image. Typical island width was about 30 nm. The nanographene island area ranged from 600 to 10000 nm². The coverage of the conductive area measured from the gap current image, which is about 15 %, is smaller than that measured from the LEEM image. The conductance of an island that is smaller than the gap distance of the probe can not be measured. This is one of reasons why the conductance area measured from the nanogap probe image is smaller than that measured from the LEEM image.

The gap current is almost constant for each island. The average current of each outlined nanographene island shown in Fig. 2(e) was measured. Figure 3 shows a histogram of the average currents of the islands.
The nanographene islands are clearly divided into two groups depending on the average current. The conductance of the larger-current group (average current = 5.2 nA) was about four times that of the smaller-current one (average current = 1.4 nA). The total area of the smaller-current group was about ten times that of the larger one. This area ratio is almost the same as the area ratio of the single- to double-layer graphene estimated from the LEEM image. These results strongly suggest that the smaller-current group consists single-layer graphene islands and that the larger-current one consists the double-layer graphene islands.

The conductance of the graphene islands measured by the integrated nanogap probe digitally depends on the layer number. However the value of the resistivity of the nanographene could not be estimated quantitatively from the present data, because contact resistance between graphene and Pt electrode could not be estimated correctly. A size dependence of the conductance of the single-layer nanographene similar to be that of the reported paper [4] is observed and will be discussed elsewhere after detailed analysis.

The field effect of graphene is a very attractive subject [1, 2, 4, 8]. Unfortunately, the electrode of the integrated nanogap probe made contact with the surrounding region of nanographene, where the withstand voltage is too low for field effect measurement. Additional surface treatment will be needed for applying electrical field for graphene. Furthermore, miniaturization of the electrode of the probe will be effective for the field-effect measurement of nanographene.

4. Conclusion

Conductance image of nanographene was successfully obtained using an integrated nanogap probe with a 30-nm gap. The typical width of nanographene in the conductance image is about 30 nm. From the conductance measurement, single- and double-layer nanographene could be clearly distinguished. The results indicated that the integrated nanogap probe is a useful tool for nano-order electrical transport (in-plane) measurements.

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