Direct Observation of Evolution in Graphene Layers by Electrical Properties

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Abstract
Graphene demonstrated potential for practical applications due to its remarkable electronic property. In this work, we observed the morphology of graphene with widths down to 20 nanometer in various electronic effects via in-situ transmission electron microscope (TEM). The hourglass-like shape graphene was instant broke up to 750μA, which showed that graphene exhibit an impressive breakdown current density. The current carrying capacity was in the order of 4.66x10¹⁸ A/cm², which was several orders higher than copper. The current-carrying capacity was found to relate with the resistivity of graphene. In addition, we also observed that the atomic resolution of combined multilayer graphene change volume via surface diffusion and joule heating. These results point out that the breakdown mechanism is approximated to Joule heating.

1. Introduction
Graphene is a unique material with variety applications in electronic¹⁻³, biological⁶⁻⁶ and optoelectronic⁵⁻⁸ due to their interesting properties such as high intrinsic mobility⁹, high current carrying capacity, ballistic transport, width-dependent bandgap¹⁰ and high thermal conductivity. Recently, it has been reported that graphene exhibited an impressive breakdown current densities on the order of 10⁶ A/cm². Nowadays, The graphene electronic devices grown via chemical vapor deposition (CVD) on polycrystalline Cu foil shows great promise, which has proved in wafer scale fabrication, and in monolithic integration with CMOS.

An energy band gap can be build via quantum confinement and edge effects by patterning graphene in the form of nanostrictions, nanoribbons and quantum dots, which could avoided lacking an energy band gap in bulk graphene. These development techniques not only supplied measuring the electronic properties but also exhibited the structure images with subnanometer resolution.

To this end, we fabricate graphene nanoconstrictions by focused ion beam (FIB) nanosculpting, which exhibited it’s widths down to 20 nanometer. The electronic properties were measured via in-situ transmission electron microscope (TEM). In situ TEM has been proven to be a powerful tool for atomic-level observation, including phase/shape transformation, molten nanofluidic migration and electromigration. In this work, we clearly demonstrated the evolution of the graphene structure by breakdown effect that current carrying capacity is 4.66x10¹⁸ A/cm². In addition, we also observed that graphene flakes combined and volume of cluster changed by surface diffusion and joule heating. This phenomena allowed us to prevent the other materials took graphene as a transmission path from the thermal caused failure. Besides, the high current capacity density in the order of 4.66x10¹⁸ A/cm² is several orders higher than copper, which has attracted intense interest for a wide range of electronics applications.

2. General Instructions
This experiments were based on suspended graphene film on in-situ Si₃N₄ membranes. Details of the device fabrication processes can be referred to figure 1. The schematic diagram illustrated the preparative in-situ samples shown in figures 1 (a-b). The photolithography was used to define Ti/Au source and drain electrodes with a separation space by FIB milling to cut a 1.4μm x 80μm slit in the Si₃N₄ membrane in the region between the electrodes. Figure 1 (c) shows CVD grown graphene layer was transferred to the chip surface. Our clean graphene film is suspended across two free-standing Ti/Au electrodes. The hourglass-like pattern was used electronic beam to sculpt the shape as shown in figure 1 (d). Beam irradiation was used to remove carbon atoms from the graphene edges and to reduce the film width. The graphene film with a small freely suspended region over a slit in the Si₃N₄ membrane, which was in good electrical contact with the Au electrodes as shown in figure 1 (e). TEM image of an hourglass-like graphene film is shown in figure 1(f).

Fig. 1. The process of preparing the specimens for in-situ TEM observation. (a) The in-situ sample schematic. (b) The observation slit via FIB. (c) The graphene was transferred to sample, which was suspended across two free-standing Ti/Au electrodes. (d) The hourglass-like pattern was used Ga+ ion beam to sculpt the shape. (e) Finally, sculpting process the hourglass-like was observed. (f) TEM image of an as-fabricated graphene film connected to Ti/Au electrode. The scale bar is 500 nm.
We carried out a study of the single layer graphene with G-mode peak to sculpt the hourglass-like pattern as shown in figure 2(a). The original length (along the current pathway) and width were 500 nm and 20 nm, respectively. Firstly, the bias voltage was ramped to 3.6 V. The I-V curve is smooth when V < 3V. However, as the pronounced small current drops at 3.25V, the graphene begins to creak and expands, where the graphene length was increased from 500 to 520 nm as shown in figure 2(b). When applied voltage approached to 3.6 V, the suspended graphene exhibit an impressive breakdown current, which is 4.66x10^{10} A/cm^2. In addition, the maximum power exceeded 1.2 mW, which calculated that the suspended graphene film could reach a temperature as high as 2000 °C. Therefore, we expected that the breakdown mechanism was associated with joule heating. The graphene failed and breaking at center completely at the same time the current drops to zero, as shown in figure 2(c).

The necking and creaking phenomena occurred at the center of the graphene film between the Au/Ti electrodes where the rising temperature is highest. The creases initiate from the outside edges simultaneously toward the center through current induced sublimation of carbon atoms from the crack edges. When the crease approached the center, the graphene formed a narrow constriction, which had been reported in previous research. The graphene length was expanded end to 620 nm via joule heating effect, as shown in figure 2(d).

3. Conclusions

In conclusions, we observed the morphology evolution of graphene with widths 20 nm in various electronic effects via in-situ transmission electron microscope (TEM). The hourglass-like shape graphene was instant broke up to 75uA, which shows that graphene exhibit an impressive breakdown current density. The current carrying capacity is in the order of 4.66x10^{10} A/cm^2.

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