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Influence of NaOH Concentration on Transfer Process of Graphene

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Abstract
The process of transferring a monolayer of graphene using two different concentrations of sodium hydroxide (NaOH) solution unto a silicon dioxide (SiO2) coated Si chip using electrochemistry was performed. The transfer process is crucial for the delamination of a continuous graphene monolayer film from copper foil. After examining and inspecting the integrity of the graphene monolayer, it was observed that the lower concentration to NaOH led to slower rate of hydrogen bubble generation; this condition was found to be less destructive and yielded a graphene film with fewer visible tears.

Keywords
graphene, transfer, monolayer

Disciplines
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Influence of NaOH concentration on transfer process of graphene

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The process of transferring a monolayer of graphene using two different concentrations of sodium hydroxide (NaOH) solution unto a silicon dioxide (SiO2) coated Si chip using electrochemistry was performed. The transfer process is crucial for the delamination of a continuous graphene monolayer film from copper foil. After examining and inspecting the integrity of the graphene monolayer, it was observed that the lower concentration to NaOH led to slower rate of hydrogen bubble generation; this condition was found to be less destructive and yielded a graphene film with fewer visible tears.

Key Words: graphene, transfer, monolayer

I. Introduction

Graphene is a monolayer of sp2 carbon atoms arranged into an ordered honeycomb-like lattice, a basic building block for carbon-based materials1. What makes graphene so interesting is that it is an excellent thermal and electrical conductor. It is a semi-metal, a zero-gap semiconductor, that exhibits an ambipolar electric field effect such that charge carriers can be tuned continuously between electrons and holes. The very high carrier mobility and low resistivity that graphene exhibits at room temperature makes it a promising material for applications including transistors, and chemical and biochemical sensing2. When transferring the graphene through the wet chemical process known as electrochemical delamination, the concentration of NaOH solution plays a large role in the success of the graphene transfer process. Wang et al. performed the electrochemical delamination of graphene on copper with an aqueous solution of K2S2O83.

II. Graphene transfer

The graphene in this study was transferred onto a 24x24mm Si chip coated with 128 nm of SiO2. On the chips are metal lines, 100nm thick Au over 10nm thick Ti, that serve as the contacts for the graphene once it is successfully transferred. To attach the graphene to the receiving substrate the following processes were performed:

- The chip was cleaned through O2 plasma at 50W and 1.5 Torr for 2 minutes using a Technic Series 800 Reactive Ion Etcher(RIE).
- The monolayer of graphene grown on copper foil, provided by Groltex Inc, was placed on a glass slide and spin-coated with poly(methyl methacrylate)(PMMA) at 2000RPM for 45 seconds. Note that the ridges and bends on the foil must be minimized; a second glass slide and soft paper is used to press on the copper film to reduce them. The PMMA resist acts a protective layer and as scaffold that keeps the graphene film from folding on itself or tearing after it delaminates from the foil. Figure 1 is a photograph of 3 pieces of graphene prepared this way.
- The foil was then soft baked with a hot plate for 3 min at 150°C
- An aqueous solution of NaOH at 0.05M was employed as electrolyte in the electrochemistry process. A power supply was used to cathodically polarize the foil to the solution. The graphene/Cu electrode was cathodically polarized at 18 V, and hydrogen bubbles emerge at the graphene/Cu interfaces due to the reduction of water:

\[ 2H_2O + 2e^- \rightarrow H_2(g) + 2OH^- (aq) \]

The formation of the H2 bubbles aid in giving a gen-
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FIG. 2. (a) An image of the Copper foil and separated graphene film in NaOH solution. The graphene, coated with PMMA is seen in the area noted by the dashed line region. Note the bubbles of $\text{H}_2$ under the film. (b) Glassware for solution and rinsing and a copper film coated with PMMA and taped to a glass slide. (c) A closeup of some of the equipment set up for the graphene transfer process, including the power supply.

ttle and persistent force to separate the film from the foil. A secondary function of the bubbles is that it serves as the support that helps keep the graphene afloat in the solution and during the subsequent rinses. Figure 2a shows a graphene film released from the Cu foil. Figures 2b and c show the apparatus.

- Using a glass micropipette, gently coax the graphene film onto a transparent plastic sheet from the solution, into two subsequent DI water dishes and finally onto the substrate of choice.

- The film must be dried so it sticks properly to the surface. Let the film air dry for 15min to 20 min and then spin dry using a spin coater at a speed of 5000RPM for 2min.

- Remove the PMMA still coated on the film by immersing in acetone for 10min, and then immersing in IPA for 3min.

- Finally, soft bake with hot plate at 150°C for 3min.

- Inspect using optical microscope.

III. Results and Discussion

The processes involved during the graphene transfer requires a lot of dexterity and practice in order to get continuous graphene sheets. In earlier experiments the concentration of the NaOH was 0.5$M$, and while the process is successful, the fast formation of $\text{H}_2$ bubbles caused many tears and breaks in the film, making the gathering of electrical characterization data difficult. Lowering the concentration of the solution from 0.5$M$ to 0.05$M$ gave rise to $\text{H}_2$ bubbles at a much slower rate and size therefore being much less destructive to the graphene film causing any breaks in the film. Figure 3 show microscope images of graphene deposited on substrates, separated by the 0.5$M$ solution. Note the hide density of tearing.

Figure 4 shows images of graphene separated with the more dilute 0.05$M$ solution. Fewer tears are seen.

Fig. 3. A graphene device that employed the NaOH concentration of 0.5$M$ with graphene tears. Because of the tears caused many electrodes did not function and therefore yield of graphene devices were low. The whiter regions pointed by the arrows represent the tears.

Having as few tears in the graphene as possible is necessary for our purposes, as the experiment calls for a
FIG. 4. A graphene device that employed the NaOH concentration of 0.05M. The darker regions show where the film is, and the whiter regions corresponds to a break in the film. Although breaks are present, they are smaller in size and thus increase our probability of continuous film.

IV. Summary

This experiment examined the wet chemical process of electrochemical delamination to separate a monolayer film of graphene from its copper foil growth substrate. In order to achieve the best removal process, two different concentrations of NaOH were studied for graphene release and it was found that the lower concentration (0.05M) yielded a higher quality film. A reduction in the concentration by 10x led to a slower formation of H$_2$ bubbles that in turn led to a less destructive graphene/Cu foil separation process.

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