Process for Detaching Suspended Graphene Structures from Silicon Carbide

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Abstract:

Epitaxial graphene was grown on the silicon-terminated face of silicon carbide (SiC). A novel approach was developed to produce suspended graphene structures for transmission electron microscope (TEM) analysis. TEM analysis allows for structural and chemical characterization of graphene structures at the atomic level. TEM analysis, however, is constrained to ultra-thin materials because the microscope's electron beam must travel through the material before collection by the detector. Consequently, TEM analysis of suspended graphene structures requires detachment from the SiC substrate. This paper details the success of the fabrication process, which marks the first time graphene structures have been detached from the SiC substrate by means other than mechanical exfoliation.

Introduction:

Graphene consists of an atomically-thin planar sheet of sp^2-bonded carbon atoms arranged in a hexagonal lattice. Graphite can be envisioned as a large number of graphene sheets stacked atop one another. Since graphene's isolation in 2004, it has become one of the most widely researched materials [1,2]. At room temperature, graphene claims the highest known carrier mobility, thermal conductivity, and in-plane strength [3-5]. These promising electrical, thermal, and structural properties may lead to applications in high speed and flexible electronic devices.

Of the existing graphene fabrication processes, epitaxial growth from SiC is particularly promising. The semi-conducting character of SiC allows for seamless integration with existing Si-based electronics [6]. In addition, graphene growth from SiC is a well characterized and mature process potentially suitable for large-scale graphene production [1].

The aim of the research was two-fold: to experimentally verify the novel fabrication process' ability to detach suspended graphene structures from the SiC substrate, and to analyze the suspended graphene chemically and structurally using transmission electron microscopy.

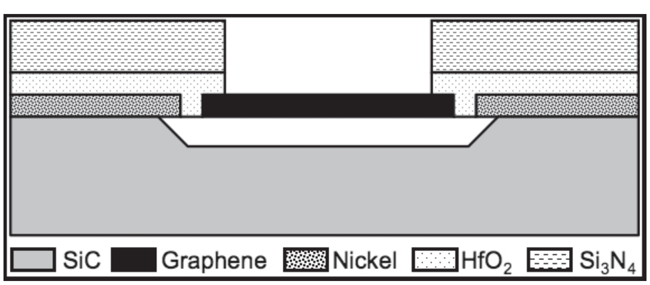

Figure 1: Cross-sectional view of all layers involved in the fabrication process.

Experimental Procedure:

Figures 1 and 2 complement the following description.

Graphene was grown epitaxially on the Si face of chemically-mechanically polished n-type 4H SiC. Raman spectroscopy and atomic force microscopy were employed to verify the presence of quality graphene. The graphene was patterned using a reactive ion oxygen (RIE) etch. A sacrificial nickel (Ni) layer was deposited around the patterned graphene using a photolithographic lift off process. The entire sample was then blanketed with 2 nm of electron beam evaporated hafnium, 15 nm of atomic layer deposition hafnium oxide (HfO), and 500 nm of plasma-enhanced chemical vapor deposition low stress silicon nitride (SiN).

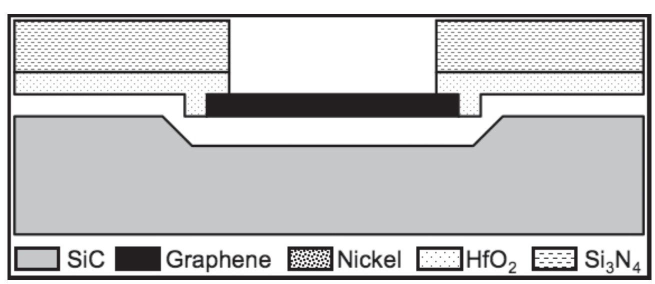

Figure 2: Cross-sectional view after dissolution of nickel layer. Notice the SiC substrate is entirely detached from the graphene and the graphene support structure.

SiN was chosen for its rigidity and would serve as the support structure after detachment of the graphene structures from the SiC substrate. HfO was employed to prevent the plasma-enhanced deposition of SiN from destroying the graphene structures. To enhance the uniformity of the HfO deposition, a seed layer of electron beam evaporated Hf was deposited and allowed to oxidize. This ensured a continuous protective coating of HfO over the graphene structures. The concept stemmed from literature reporting the use of seed layer aluminum for enhanced aluminum oxide growth [7].

The SiN atop the patterned graphene areas was partially etched away using a CF_4 RIE. Subsequent immersion in buffered oxide etch 6:1 removed the HfO layer. The graphene was then suspended by removing the underlying SiC via a newly developed photoelectrochemical (PEC) etch process [8]. Successful lift off required the SiC undercut to reach the Ni layer (see Figure 1). Finally, Ni etchant was employed to dissolve the sacrificial Ni layer. The graphene—now wholly supported by the low stress SiN—was lifted off the SiC substrate and transported to a TEM grid for analysis. Preliminary imaging was conducted using scanning electron microscopy (SEM).

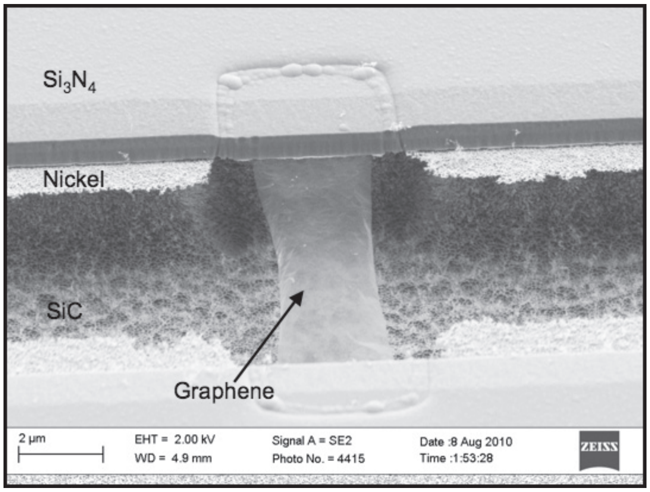

Figure 3: SEM of sample after PEC etch of SiC. [8]

Results and Discussion:

Figure 3 depicts a suspended graphene structure after a PEC etching of the SiC [8]. The transparency of the graphene structure suggests that it is a few monolayers. The SiN support structure is only attached to the SiC substrate via the sacrificial Ni layer. The PEC etch appears to have adequately undercut the SiC, so the nickel layer was dissolved.

Figure 4 depicts the SiN support structure lifted off the SiC substrate with the suspended graphene structure intact. Notice the wrinkles in the graphene, especially in the center and fringe areas. This undesirable effect is the result of a compromise in the fabrication process. A low stress SiN support structure was required to prevent the support structure from folding onto itself after detachment. This low stress constraint prevented the support structure from providing the tensile stress necessary to hold the graphene taut.

The research demonstrated the ability of the fabrication process to detach suspended graphene structures from the SiC substrate. The graphene structures were successfully transported to a TEM grid for analysis. The research marks the first time graphene structures have been detached from the SiC substrate by means other than mechanical exfoliation.

Future Work:

Suspended graphene structures will be analyzed at the atomic level using TEM. TEM analysis will be employed to confirm the hexagonal lattice structure and carbon composition of the suspended graphene structures. TEM analysis will provide further information about the grain structure and overall quality of the silicon-face epitaxial growth process. Lastly, TEM analysis may provide new insights into silicon-face graphene growth.

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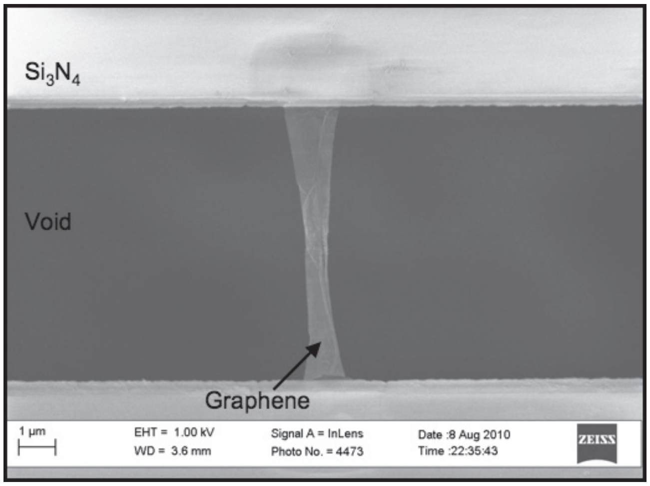

Figure 4: SEM of sample after dissolution of nickel layer and transport to transmission electron microscope grid.