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Abstract

Fabrication and microscopic characterization of nanosheets exfoliated from artificially-synthesis mica

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Two-dimensional (2D) materials with atomic or molecular thickness have attracted extensive interest in material research communities because of their exotic properties for novel functional materials and devices. As represented by graphene, the method of mechanical exfoliation of cleavable 2D materials has opened a new era in 2D nanomaterials. Innovation using such 2D nanomaterials crucially depends on the scalability of their thickness and width, as well as the degree of purity and interaction with the substrate, which should be thoroughly investigated.

In this study, we successfully fabricated wide (several hundredths of micrometers) single layer (1 nm) to 20-layer nanosheets of pure artificially synthesized phlogopite on Si substrates. We then characterized their thickness and width by atomic force microscopy (AFM) and scanning Auger electron spectroscopy/microscopy (SAM), including SAM analysis of their chemical composition. In addition, by measuring the current–voltage (I–V) curves of metal–nanosheet–metal contacts by conductive AFM, we show the potential of phlogopite as a thin insulating 2D material with discrete thickness for electron tunneling devices. The nanoscale characterizations of mica nanosheets with focused electron beam has been investigated.

Phlogopite is a member of the mica family of silicate (phyllosilicate) minerals. It is a single crystalline oxide with a band gap energy of ~7 eV, perfectly cleavable along the (001) plane, and fascinating because of its high resistance to heat, water, and chemical agents. The phlogopite used in this study was purely artificially synthesized, and its quality was sufficiently high to be used for electronics applications. Thus, a method to control its discrete thickness by units of 1 nm and placing it on a substrate is of great importance to exploit phlogopite as an insulating 2D nanomaterial.

First, we propose a conventional approach using a polyurethane hand roller to exfoliate one- or few-layer 2D nanosheets with wide area from 2D cleavable crystals. In comparison with other exfoliation methods for 2D materials, including transfer processes to a substrate, our method easily and reliably provides clean and much wider 2D nanosheets with one- and few-layer thickness on the substrate.

Second, we propose a method to determine the thickness of mica nanosheets in the range from one to five layers on a Si substrate using SAM by taking the intensity ratios of the Auger electron spectroscopy (AES) peaks of Mg and O as the compositional elements of the mica with respect to the peaks of the elements of the substrate, such as Si and the metal (Au and Ir in the present study). We derived thickness calibration curves based on the AES peak intensity ratios experimentally obtained for mica nanosheets with different thicknesses. The curves showed the AES intensity attenuation through the mica nanosheets with the inelastic mean free path, which is often used in photoelectron spectroscopy. Although SAM analysis of insulating materials tends to be avoided owing to terrible charging effects, we clearly demonstrate that SAM analysis is a powerful method for investigating insulating 2D nanomaterials.

Third, the electronic properties of the mica nanosheets were examined by conductive atomic force microscopy to measure their current–voltage (*I*–*V*) curves, exhibiting the characteristics of metal-insulator-metal contact having a tunneling barrier which is decreased with decreasing thickness. This would allow us to conduct the SAM analysis by avoiding the terrible charging effect on the insulating mica.

Finally, focused electron-beam induced etching of mica layers has been investigated as a selective nanoscale etching technique. In order to gain an understanding of the process, the effects of beam current, beam energy, and scan time on the process were examined as of controlling parameters. Experimental results indicated that the etching process is governed by the electron-stimulated desorption of mica elements from the mica compound.

This study demonstrated that the elemental and structural analysis of even insulating thin 2D layered materials can be plainly conducted by the SAM, the information of which is fundamental and critical to realize nanoscale electronic devices, composed of conducting and insulating 2D layered materials.

Keywords: Mica nanosheets, atomic force microscopy, scanning Auger electron microscopy/spectroscopy, current-voltage characterizations, focused electron beam