Conventional Exfoliation Method using a Hand Roller for Artificially-synthesized Mica Nanosheets with a Single Layer and Multi-layers in Wide-area and their Characterization

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Mechanical exfoliation of two-dimensional (2D) materials with adhesive tapes, followed by transfer them onto a substrate, has often been used to prepare single- or few-layer thick 2D samples. Although mechanical exfoliation techniques have led to understanding of novel physical phenomena of 2D materials, the exfoliation techniques fall short in terms of scalability. Here, we present a conventional approach to exfoliate single-layer or few-layer 2D nanosheets with wide area from 2D cleavable crystals using a polyurethane hand roller with a diameter of 20 mm (Fig. 1). The sample was single-crystal oxide of artificially-synthesized phlogopite ($KMg_3AlSi_3O_{10}F_2$), one of mica family. Prior to transfer of the exfoliated nanosheets on Si substrates with/without 10-nm Au deposition via DC magnetron sputtering, the substrates were cleaned in an ozone cleaner with ultraviolet light irradiation to removed absorbates, and heated to extend the contact area at the interface between the mica nanosheet and the substrate by evaporation of water layer adsorbed on the substrate.

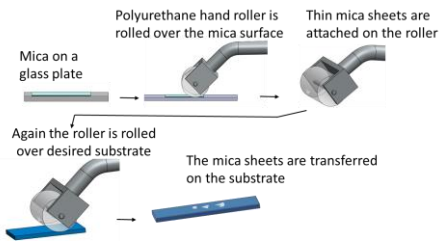

Figure 1: Illustrative outline of mechanical exfoliation and affixing processes of mica nanosheets on the substrate. First, a polyurethane hand roller is rolled over the mica. Subsequently, the exfoliated mica nanosheets on the surface of the roller are transferred and affixed to the substrate by rolling the roller over the substrate.

Figure 2(a) shows a typical atomic force microscopy (AFM) image of the mica nanosheet on the Si substrate. The thickness of the center part in Fig. 2(a) was approximately 1 nm over ~10 µm width, corresponding to a cleavable single layer of the mica. We analyzed the mica nanosheets with various thicknesses on Si substrate using scanning Auger electron microscopy/spectroscopy (SAM) in regard to the change of peak-to-peak intensities of the Auger electron spectroscopy (AES). The AES spectra showed distinct changes in the intensities according to the change of the number of layers of mica nanosheets (Fig. 2(b) and (c)). We propose the thickness evaluation method of mica nanosheets in the range of 1 to 5 layers based on the SAM analysis only. It is noted that SAM analysis can be applied to the mica nanosheets, even which electrical conductivity is poor. We also measured the current–voltage characteristics on the mica nanosheets using conductive AFM, and evaluated the change in barrier height with thickness, the detail of which will be discussed according to a model of metal-insulator-metal junction.

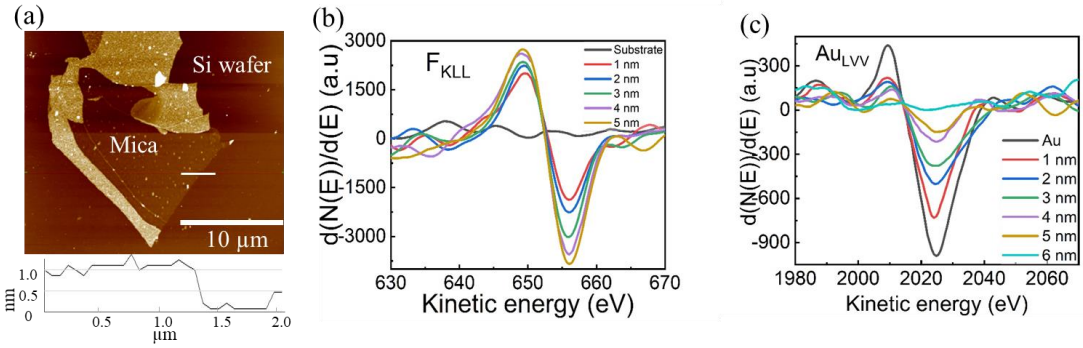

Figure 2: (a) AFM image of a mica nanosheet having a 1 nm thickness (single layer) at the center, with 3-layer and 6 layer regions on the Si substrate. The cross-sectional profile along the white line is shown below (a). (b) AES spectra of F_{KLL}, one of the mica elements, and (c) Au_{LVV}, from the mica nanosheets with different thicknesses on a 10-nm Au deposited Si substrate. The peak-to-peck intensity of AES signals for elements of mica increased with increasing number of mica layers, whereas the intensity of Au from the substrate attenuated with increasing number of mica layers.