Title	周波数変調原子間力顕微鏡を用いた力学的散逸エネルギー測定による表面電子特性の研究
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Abstract

In the last three decades, a variety of surface structures of exotic materials have been profoundly investigated on a nanoscale by atomic force microscopy (AFM) using a tiny cantilever. Therein the force acting between a sample and an atomically sharpened tip on the cantilever was measured from the bending of the cantilever. While the bending was kept constant and the tip was scanned over the surface, the surface topography was depicted apparently. However, it was pointed out that damages of the tip and the sample surface often took place while scanning in contact of the tip to the sample. Toward no damage observation with high force sensitivity at an atomic level, frequency modulation (FM)-AFM was invented, in which the cantilever was oscillated at its resonance frequency (f) and the shift of the frequency (Δf) due to the change of the force was measured precisely and kept constant during scanning. Under this control, the weak force interactions were successfully measured, leading to depiction of the atom-resolved images. In the FM-AFM, the tip was able to be brought closer to the surface less than 1 nm in a stable manner. This means that we can survey the electrical and mechanical properties of the sample through the tip in close proximity to the sample surface. Based on the FM-AFM, various mechanical and electrical properties of samples have been unveiled.

Among the atom-level analysis by the FM-AFM, it is noticeable that the conservative and non-conservative (dissipative) force interactions between the tip and the sample can be separately measured. Through the non-conservative interactions, mechanical energy stored in the oscillating cantilever dissipates. One of the dissipative interactions is Joule heat induced by displacement current through the gap between a sample and the oscillating tip under an electric potential difference; the gap is simply modeled by a capacitor of two different materials of a tip and a sample. When there are resistive parts in the tip–sample system, the displacement current makes Joule heat, which decreases the mechanical oscillation energy stored in the oscillating cantilever. In the present work, the change in energy dissipation is measured by FM-AFM with a conductive tip for a mica nanosheet with varying thicknesses of nanometers placed on a conductive substrate. The mica nanosheet acts as an ultra-thin dielectric layer, which changes the capacitance between the tip and the sample. The characteristics over the conductive substrate is measured and compared with mica nanosheet varying for thin to thick layers. From the dissipation data that depends linearly on electrostatic interaction, the resistance generating the Joule heat is estimated.

In the dissertation contents, for the test sample, mica nanosheets are used, which can be regarded as a 2D single-crystal insulating material. Its minimum thickness is 1 nm. The mica nanosheets have attracted much interest owing to its high dielectric constant, excess thermal stability, and high resistivity used for quantum tunneling barriers. Phlogopite, a member of the mica family, is artificially synthesized with high purity. The perfectly cleavable basal (001) plane of phlogopite is suitable to prepare ultra-thin nanosheets. Our developed

mechanical exfoliation technique using a polyurethane hand roller is used to exfoliate and affix the nanosheet on the conductive substrate; here an iridium (Ir)-coated Si (Ir/Si) substrate is used, on which it is found for the nanosheets to be mechanically stable. The dimensions of the mica nanosheets on the substrate are measured by the FM-AFM, and the usefulness of the hand roller technique is demonstrated. For the tip, a commercially available platinum–iridium (Pt – Ir) tip on a Si cantilever is used, resulting in high-reproducible results.

The topographic and energy dissipation images of the sample surface are simultaneously observed, and the changes in the resonance frequency shift (Δf) and dissipation (D_{dis}) with respect to the sample bias voltage at our preferred set point are measured over the mica nanosheets, for example, 4-layer (4 nm), 8-layer (8 nm), 11-layer (11 nm) and 15-layer (15 nm), and no-mica (bare) regions on the Ir/Si substrate. From the Δf – distance curves, the numerically converted attractive forces using Sadar method where stable scanning was performed at target Δf measured with a magnitude of ~1nN over the sample surfaces. From the dissipation mapping, we observed faint contrast for thicker mica similar in dimension to that of topographic contrast but almost nothing for the thinner mica sheet.

When we performed the spectroscopic measurement, we observed almost equal dissipation for bare Ir/Si and thin mica nanosheet (4-layer) measured separately, but higher in dissipation for the thicker mica sheets (11- and 15-layer) following sharp ordinary parabolic behavior and a tendency to increase with respect to its thicknesses. By fitting our parabolic curve, we measured the contact potential value (CPD) for the corresponding surfaces which later been used to extract pure electrostatic (Δf_{ele}) force interaction. From the dissipation (D_J) and Δf_{ele} curves which are linear in characteristics for all layers, the surface resistance for Joule dissipation was measured and summarized as the order in GΩ. We explained such a high resistance value for the metal and thin nanosheet is possibly due to surface charge scattering and dielectric loss under tip oscillation. The present work possibly paves the way for nanoscale mechanical and electrical characterization based on measurement of the energy dissipation via non-contact probing in the FM-AFM.

Keywords:

Frequency modulated atomic force microscopy (FM-AFM), 2. Phlogopite mica, 3. Polyurethane hand roller, 4. Charge scattering, 5. Energy dissipation.