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Development and Application of "Soft" Probe for Various Types of Electric Measurements

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A simple probe that is applicable as an electric contact to mechanically weak materials such as nm-thick films and 2D films (graphene, MoS_2, and so forth) without destroying the specimen has been developed. The concept of the development of the probe is based on the repulsive region used in atomic force microscopy technique but without any precise feedback. The robust electric contact with the probe has been demonstrated by some examples of electric measurements including that of a 5-layer graphene film on sapphire.

1. Needs for "Soft" Probe

For any types of electric measurements such as I-V or C-V curves and biased photoemission measurements, good electric contact with specimens is important. When specimens are not mechanically strong enough to stand against a conventional probe, which is usually a needle-like tungsten, special sample preparation for measurements is necessary, which sometimes contaminates specimen, making impossible to use the specimen for further other measurements.

To solve this problem, we have developed a "soft" probe, which can make a good electric contact with nm-thick film, 2D materials such as graphene and fragile materials without damaging the specimens.

2. Principle of "Soft" Probe Operation

To obtain a good electric contact, electron orbitals of the two materials in contact should overlap. The degree of the electron overlap can be monitored by a force curve in atomic force microscopy (AFM) as shown in Fig. 1. In the repulsive region in Fig.1, robust electric contact is realized and good for electric measurements without damages.

A typical force in the repulsive region is calculated using a force curve on Cu measured with a cantilever with a force constant of 3 [N/m]. Elastic deformation

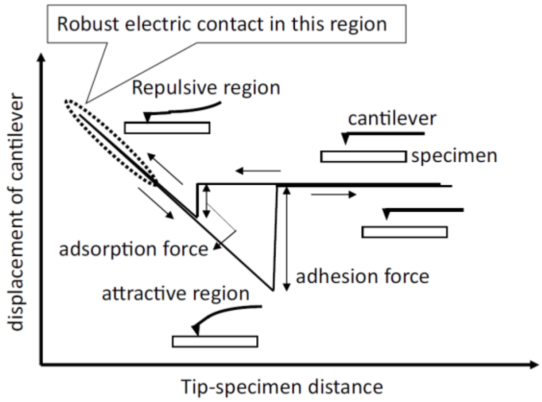

Fig. 1 Condition for electric contact in AFM force curve.

was observed at approximately 5 nm displacement. By taking into account the estimated radius of the contact area of the cantilever, the pressure is $\{3\ [N/m] \cdot 5\ [nm]\}/\{\pi \cdot 10^2\ [nm^2]\} \sim 5 \cdot 10^7$ [Pa].

In order to realize this pressure with a larger contact probe (a radius of the contact area of 100 μm), the force to be applied is $5 \cdot 10^7\ [Pa] \cdot \{\pi \cdot (100\ \mu m)^2\} \sim 1.5$ [N]. Using a commercially available spring plate with a thickness of 0.5 mm, the displacement with a force of 1.5 [N] is approximately 1 mm, which can be easily detected by eyes. In practice, an Au ball formed on the top of a hair pin shaped W wire with an appropriate spring constant is fabricated as a probe. When other materials than Au are favorable as a contact material such as Pt, other materials are coated on the Au ball.

3. Example of Electric Measurements

The example of an electric measurement using soft probes with a conventional digital tester has been demonstrated on 5-layer graphene transferred to sapphire [1]. Two soft probes were pushed toward the graphene with a contact distance of approximately 1mm apart as shown in Fig 2. The resistivity measured by a conventional two probe method was 3 kΩ, giving the resistivity of approximate $8\cdot10^{-6}$ [Ωm]. The resistivity of graphite (= infinitely thick graphene) is reported as $3–60\cdot10^{-5}$ [2], $7.837\cdot10^{-6}$ [3], $2.50–5.00\cdot10^{-6}$ (parallel to the basal plane) [4], and $4.0\cdot10^{-7}$ [Ωm] [5], while the resistivity of $8.8\cdot10^{-5}$ [Ωm] is reported for 2–10 layer-reduced graphene oxide [5]. The possible lowest resistivity of graphene estimated from the atomic vibration is $1.0\cdot10^{-8}$ [Ωm] [6]. Therefore, the measured value using the probe appears reasonable.

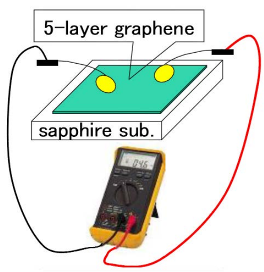

Fig. 2 Schematic illustration of resistance measurement on graphene using the developed probes.

4. Control of Contact Area

For measurements such as capacitance and current density, we need to know the electrical contact area. Therefore, soft probes whose electrical contact area is controlled have been developed.

4.1 Contact-area-controlled Probe

The contact area is controlled by patterning a circle at the top of hemisphere of Au (or Pt-coated Au) ball on the top of a probe with laser lithography. Fig. 3 shows the results of lithography on Pt-coated Au ball.

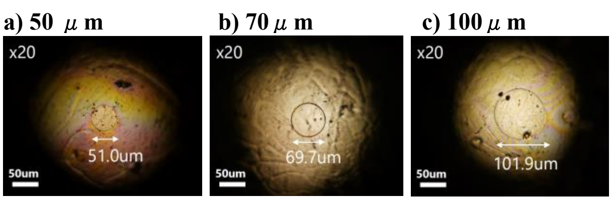

Fig. 3 The contact-area-controlled probes with different sizes. Outside of the circles is covered with insulating resist film and the probe material is exposed bare inside.

4.2 Example of Measurements

Contact-area-controlled probes have been applied for the measurement of ReRAM materials (NiO film). The specimen is columnar NiO film as shown in the upper side of Fig. 4. When voltage is applied between the top and the bottom of NiO film, the film shows resistive switching behavior [7]. There are different types of grain boundaries in the columnar structure in Fig. 4, one of which, very low resistance (VL)-mode boundary is highly conductive (destroying switching behavior). When conventional Pt deposited electrode film is used for electric contact, the film fills the unevenness of NiO surface and touches the VL-mode boundary, killing switching function due to seriously large leakage current (thick arrow in the right of Fig. 4). In contrast, Pt-coated soft probe does not touch the VL-mode boundary and gives switching function through the current pass (white arrow in the left of Fig. 4).

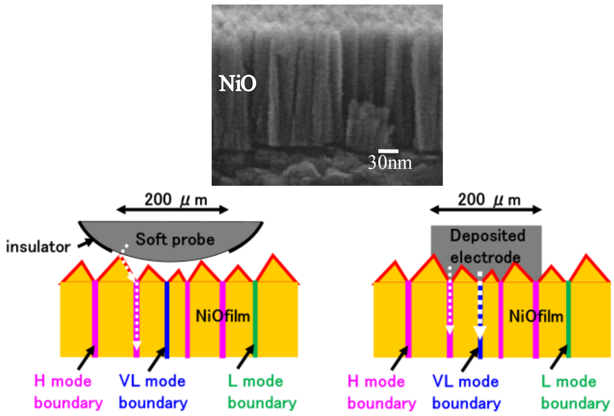

Fig. 4 Photo of NiO ReRAM film (top) and schematically shown electric contact of NiO film with soft probe (bottom left) and deposited film (bottom right).

5. References

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