Nanogap electrodes on Si cantilever for local conductance measurement

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Abstract. We developed a new nanotool of scanning probe microscopy (SPM) for local conductance measurement. Two Pt electrodes with a nanogap fabricated by focused ion beam (FIB) milling are integrated on a Si cantilever with Al electrodes. The minimum gap fabricated on the cantilever is 20 nm. Local conductance measurements of conductive materials, a carbon film and a gold film, were performed using the nanogap probe on a conventional SPM system. Ohmic contacts between the gap electrodes and the conductive samples were established. The resolution of the conductance image is almost the same as the gap distance of the electrodes. A single gold grain was successfully imaged with sub-100-nm resolution.

1. Introduction
Recent progress in the miniaturization of electronic devices has achieved very small devices with nanometer dimensions. For the further development and improvement of these electronic devices, a new measurement technology for the nanometer region, nanometrology, is now urgently required. Micro- and nano-order electrical conductivity measurement is one of the most important issues in nanometrology.

Conventional SPM local conductance measurements provide a powerful one but their use is limited because only the conductance between the probe and the substrate surface can be measured. A multi-probe system is required to measure the transport characteristics of conductive materials. There are two types of approaches to realize a multi-probe system based on SPM technology. One is a multi-scanner system in which each scanner controls a single probe [1]. This type of multi-probe system requires another microscope to accurately confirm the contact position of each probe. Furthermore, the total system is large and complex. The other type is a multi-probe system integrated on a single scanner. This type of multi-probe system requires an integrated probe fabricated by micro- [2] or nanolithography [3-5] and a little modification of a conventional SPM system.

We have previously developed various types of multi-probes integrated on a Si cantilever [3-5] and demonstrated that the integrated multi-nanoprobe can be used for conductance measurements of a wide variety of materials [5]. However, the distance between the nanoprobes of the previous studies is large and not adaptable for local conductance measurement in the nanometer region. We therefore tried to improve a fabrication technique of a split electrode probe using focus ion beam (FIB) milling [3], and successfully fabricated the electrode with a sub-100-nm gap. In this paper, we report on the fabrication and the first SPM-based in-plane conductance measurements for a carbon film and a gold film with sub-100-nm resolution.
2. Fabrication of nanogap electrode

The width of the groove fabricated by FIB milling is at its maximum at the surface and is larger than the beam diameter. In the normal configuration of gap electrode fabrication, an ion beam is irradiated from the surface of a metal layer of the electrode [3]. In this case, it is difficult to make a gap electrode with sub-100-nm distance, because the effective diameter including beam tail is typically larger than 100 nm. On the other hand, the width of the bottom of the groove is smaller than the beam diameter. If the metal layer of the electrode is on a membrane, it is possible to produce a groove from the opposite side of the electrode surface, as illustrated in Fig. 1(a). The width of the gap at the electrode surface is expected to be smaller than the beam diameter of the focused ion beam (Fig. 1(b)). Using this method, a platinum (Pt) electrode on the tip of a SPM cantilever was divided into two electrodes using FIB milling.

Figure 2 (a) is a microphotograph of a Si cantilever for a nanogap probe before FIB milling. A platinum electrode on the tip of a Si cantilever is connected to four aluminum (Al) electrodes. Figures 2 (b)-(c) show scanning electron microscopy (SEM) micrographs of the fabricated probes. In the first step, the tip of the Pt electrode on an insulator layer is narrowed to about 2µm by FIB milling. Then the Pt electrode is divided into two parts by the nanogap, using FIB milling from the backside as illustrated in Fig. 1. The width of the both electrodes is about 1µm.

Figure 2 (a) A Si cantilever with four Al electrodes and a Pt electrode before FIB milling. (b) A nanogap probe fabricated by FIB milling. (c) A magnified image of nanogap with 44 nm-gap. (d) 20 nm-gap
A magnified image (Fig. 2(c)) of the tip of the probe shows that the gap between the electrodes is 44 nm. Figure 2(d) shows the other gap probe with a 20-nm-gap fabricated in the different condition. The gaps of the probes are smaller than the effective diameter of the ion beam, which is about 100 nm.

3. Local conductance measurement

The fundamental characteristics of a nanogap probe fabricated by FIB milling were investigated using conductive samples, a sputtered carbon and gold films. The first sample, a nanocarbon is a carbon film deposited by electron cyclotron resonance (ECR) plasma sputtering [6]. The ECR nanocarbon film consists nano-size domains (typically <10 nm) of diamond-like-carbon and graphite [6] and has a very flat surface [7]. In this experiment, 50-nm-thick nanocarbon was deposited on a Si substrate covered with 100-nm-thick SiO₂. A 100-nm-thick gold film was deposited by conventional sputtering on a Si substrate with thin Ti film. The typical grain size of polycrystalline gold is 100 nm.

The nanogap probe is mounted on the modified cantilever holder of a conventional SPM system (E-sweep/SPI4000: SII-NT). Since the probe is mounted with the tilt angle, the top edges of the probe electrodes contact with the sample surface. The X-direction in images is the longitudinal direction of the cantilever as shown in Fig. 2(a). Conductance measurements were performed in a vacuum (<1E-3Pa) at room temperature.

3.1. Contact characteristics between nanogap probe and flat sample

Figure 3(a) shows local conductance characteristics between the electrodes of the nanogap probes shown in Fig. 2(c). A linear relationship between the gap current, \( I_{\text{Gap}} \), and voltage, \( V_{\#1\#2} \), indicates that ohmic contact is established between the nanogap electrodes and the sample. Figure 3(b) shows height (Z) dependences of a gap current and a contact force (force curve) measured simultaneously. The height of arbitrary zero (Z=0) is defined by the height of a contact point in the force curve. The value of the gap current suddenly changes at the contact point. These results show that the contact heights of two electrodes of the nanogap probe are strictly the same in nano-order. Since the surface of the nanocarbon film is very flat, no position dependence of the electrical contact height is observed.

Since the nanogap probe is mounted on the scanning mechanism of a SPM system, the conductance image can be measured using the nanogap probe itself. Figure 4 shows (a) a topographic and (b) a conductance image of the nanocarbon film. The contact force is about 500 nN. The voltage of the gap (\( V_{\#1\#2} \)) is 30 mV. The surface roughness measured from Fig. 4(a) is 0.1 nm in root-mean-square. Because of the extreme flatness of the nanocarbon film, no texture is observed in the topography (Fig.
4(a)). On the other hand, textures with the size of 100-nm-range are observed in the conductance image (Fig. 4(b)). When the sample surface is flat and the probe electrodes establish good contact with the sample, the current between the gap electrodes mainly flows under the gap region in the sample. This explains how we can observe nano-textures in the conductance image whose size for Y-direction is smaller than the width of the electrodes (about 1µm).

This is the first report of a local conductance image with nano-order resolution using a multi-nanoprobe integrated on an SPM cantilever. Figure 4(b) suggests that the nanocarbon film contains nano size domains whose conductivity is higher than the surrounding area. The details of the local conductance characteristics of the nanocarbon film will be presented elsewhere.

3.2. Conductance image of single grain of poly-crystalline film
The gap distance of the fabricated probe was smaller than the typical grain size of polycrystalline metal, i.e. gold. The nanogap probe was used for conductance measurement of a single gold grain. Figure 5 presents images of a sputtered gold film, (a) shows the topography measured using a conventional Si probe. (b) A conductance image measured using the nanogap probe shown in Fig. 2(c). (c) A schematic of the single grain on the sample surface and the nanogap probe.
conventional Si probe and (b) is a conductance image measured using the nanogap probe shown in Fig. 2(c). The contact force is about 500 nN. The voltage of the gap ($V_{gap}$) is 8 mV. The clear island contrast with 50-nm diameter shown in the inset of Fig. 5(b) represents the single grain of the gold film.

Figure 5(c) is a schematic of the nanogap probe and the single grain on the sample surface. When the gap is located at the top of a gold grain, the conductance of the single grain can be measured as shown in the inset of Fig. 5(b). The resolution for the X-direction is defined by the edge shape of the probe and the resolution for the Y-direction is defined by the gap of the electrodes. In this case, the effective resolution for the Y-direction is evaluated from the profile of the conductance images as about 50 nm. As described in the previous section, the gap distance between the electrodes of Fig. 2(c) is 44 nm. The conductance image of the gold single grain is observed as circle because the resolutions for X-direction and Y-direction are almost the same. Since the gold surface is rough, the images of the single grain can be observed in only limited regions. The large width of the two electrodes creates an artifact in the conductance image. Narrower electrodes are required to reduce the artifact of the probe shape.

The nanogap probe fabricated using FIB milling enables us to measure the electrical conductance with nano-order resolution. The specifications of the nanogap probe at this point are not yet sufficient to measure the local conductance of nanomaterials with high accuracy and high resolution. In future work, the distance of the gap and the width of the electrodes will be miniaturized to below 10 nm [8] and 100 nm, respectively by tuning FIB milling conditions.

4. Summary
A nanogap probe with a sub-100 nm gap was successfully fabricated by FIB milling on a Si cantilever with Al electrodes. Ohmic contact was established between the nanogap probe and ECR sputtered carbon film. Nano-order texture was observed in the conductance images of both carbon film and gold film measured using the nanogap probe. Local conductance of the flat nanocarbon film is modified by the nano-size domain in the film. A single grain in the gold film was observed in the conductance image with sub-100-nm resolution.

Local conductance measurement of polycrystalline films, such as poly-silicon and organic semiconductor films, is one of the major targets of the nanogap probe. The other target is an investigation of electric transport in isolated nanostructures, such as quantum dots and single molecules on an insulator substrate.

The nanogap electrode is a basic component of the tools for nanotechnology. Nanogap electrodes on a substrate have been used for many studies on mesoscopic transport. Now we can use the nanogap in the SPM world.

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