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Development of a Surface Magneto-Transport Measurement System with Multi-Probes and the *In situ* Measurement of Bi Nanofilms Prepared on Si(111)7×7

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We have developed an independently-driven double probe-stage system that enables *in situ* magneto-transport measurements on surfaces and ultrathin films, prepared in ultrahigh vacuum. The measurements can be made at temperature down to 7.6 K and under magnetic field up to 7 T. The demonstration of Bi(001) crystal nanofilms on the Si(111)7×7 surface is presented.

1. Introduction

Nowadays, there has been growing interests in materials of the nanometer/atomic-scale that are synthesized by self-organizations on solid surfaces, typically in ultrahigh vacuum (UHV).[1–5] Transport measurements on such fine structures have inevitably required the surface-sensitive probes and, recently, so-called the micro-four-point probe (Micro-FPP) method has been developed.[1–3] This is the four-point probe method with the probe-spacing of micrometers so that the probing current flows only near the surface region. The measurements have been performed using monolithic Micro-FPP or independently driven four-tip scanning tunneling microscope (STM) probes.[3,6,7] Conductance of various nanofilms and surface superstructures on semiconductor substrates has been reported by the technique.[1,4,5] On the other hand, the surface transport researches have lacked measurements under magnetic field that provide information, for example, on the carrier-type and the carrier-density (the Hall effect) or on the phases of the conducting electrons (the weak localization effect). Only a few attempts[8,9] have been made on such *in situ* measurements, mainly due to the experimental difficulty.

In the present article, we report development of our independently-driven double probe-stage system for *in situ* magneto-transport measurements for surface systems. The measurement can be performed at low temperature down to 7.6 K, with magnetic field up to 7 T under UHV condition below 1×10^{-8} Pa. Various types of probes, i.e., a monolithic Micro-FPP[3] or an STM tip, can be mounted on the probe-stage. We demonstrated magneto-transport measurement on a metal nanofilm on a semiconductor substrate with a Micro-FPP. We chose a Bi film as a sample since it was reported to show large magneto-resistance (MR). MR of the film has been argued to depend critically on the film preparation process.[10–13] Recently, a Bi(001) crystal film as thin as 2 nm thickness was fabricated on Si(111) surface by the MBE growth in UHV.[14] Taking an advantage of the UHV compatibleness of our system, we measured MR of Bi(001) nanofilms of 2.3–780 nm thickness (6–2000 BL, 1 BL = 0.39 nm).

2. Instrumentations

Figure 1(a) shows a photograph of our custom-made experimental UHV station (Unisoku), being composed of a main chamber, a preparation chamber, and a load-lock chamber. The load-lock chamber is to introduce samples and probes from air. The preparation chamber is to make cleaning and MBE growth of samples, followed by the crystal characterization with reflection high energy electron diffraction (RHEED). The main chamber is for *in situ* transport measurements of samples, transferred in UHV from the preparation chamber. Figure 1(b) is a schematic drawing of the main chamber. A solenoid-type magnet of a NiTi superconducting coil (Cryomagnetics) is placed in a liquid helium cryostat that also cools the double probe-stage system, Fig. 2(a). Magnetic field can be applied up to 7 T at the sample position (a center of the magnet). The stage system, including the sample and two piezo-actuator units, is entirely surrounded with twofold radiation shields and movable shutters. The sample can be cooled down to 7.6 K. The probes and samples can be replaced with a transfer rod from the preparation chamber by elevating the stage up and down, as indicated in Fig. 1(b). Pressure in the main chamber is kept less than 1×10^{-8} Pa during measurements.

Figure 2(a) is a photograph of the independently-driven double probe-stage system. The two stages are capable to set various probes such as a Micro-FPP on the stage #1 and an STM tip on the stage #2, as shown in Fig. 2(a). The system is an assembly of actuator units of stacked piezoceramics supported by sapphire plates. By applying voltages, the probe actuator units control three-dimensional motions of the probes, while the sample actuator units regulate the sample rotation. These manipulations allow the probes to approach an arbitrary position on a sample and the operation is observed through a view port with an optical microscope. After placing a probe close to a sample surface, an electrical contact is made by two-steps:[6] 1) an automatic approach of the probe until a tunneling contact with the surface and 2) a minimum manual approach until the ohmic contact. An STM tip is used, at the moment, as an electrode of the current or the voltage probe for conductivity measurements.[1,7] A single tip and a double tip STM observations[15–17] under magnetic field have been considered as the future plan.

Various types of probe arrangements for our transport measurement system are presented in Fig. 2(b). Figures 2(c)–2(e) show photographs of the probes, (c) macroscopic multi-point probe (Macro-MPP), (d) Micro-FPP, and (e) STM tip, on our transferrable probe holders. Macro-MPP has six point probes with ∼1 mm spacing for Hall (transverse) and longitudinal resistance measurements for a sample with its width of millimeters and length of centimeters. Each probe (Kita Manufacturing) of Macro-

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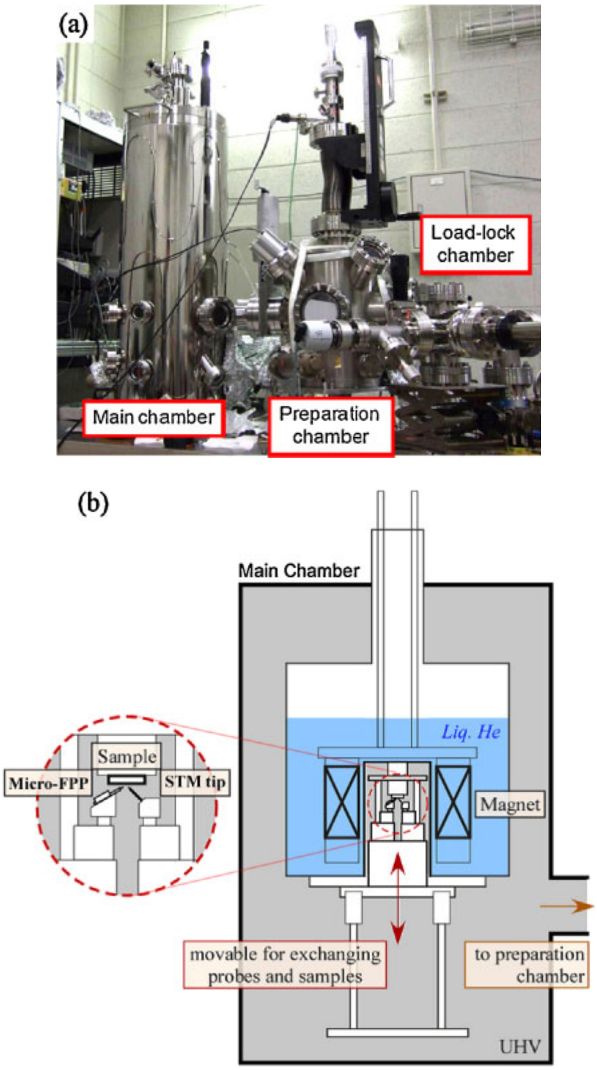

Fig. 1. (Color online) (a) A photograph of UHV chambers (main, preparation, and load-lock) for our magnetic surface transport measurement. (b) A schematic drawing of cross-section of the main chamber.

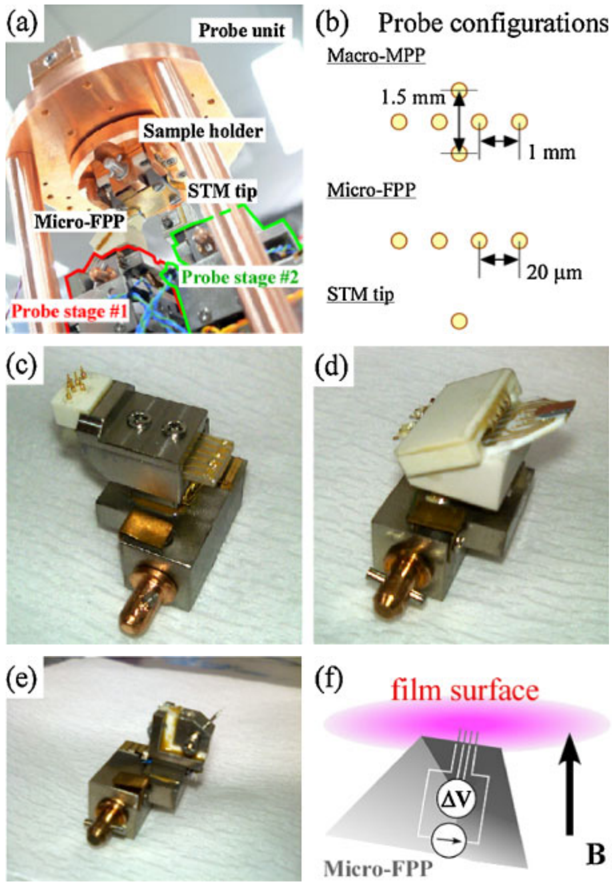

Fig. 2. (Color online) (a) A photograph of the independently-driven double probe stages, equipped with a micro-four-point probe (Micro-FPP) and a STM tip. (b) Schematic drawings of the probe configurations with typical probe-spacing values. Photographs of (c) a macroscopic-multi-point probe (Macro-MPP), (d) a Micro-FPP, and (e) a STM tip on the probe holders. (f) Illustration of magneto-trasnport measurement with a Micro-FPP (see text).

MPP is made of BeCu, coated with Au, and it is connected with a spring to make stable electrical contacts. Micro-FPP (CAPRES A/S) has four point probes, fabricated by etching Si cantilever with lithography, followed by Au coating.[3] Point probes of Micro-FPP are placed linearly and the probe-spacing are in 10 μm. Different probe-spacings can be used by replacing Micro-FPP. An STM tip can be used to make either tunneling contact or a point Ohmic contact. A combination of these probes on the two probe stage allows various types of transport measurements. In the present paper, we demonstrate magneto-transport measurement with the Micro-FPP of 20 μm probe-spacing, as shown in Fig. 2(d).

3. Results and Discussion

Figure 2(f) shows schematic drawing of the transport measurement with Micro-FPP. The outer two probes flow electric current (I), while the inner two probes measure the voltage difference (ΔV). Since the Micro-FPP probe is much smaller than a size of the sample for our measurements, the surface can be treated infinitely large and homogeneous. Magnetic field, $\boldsymbol{B}$, is applied to the sample along the surface normal direction. The arrangement corresponds to a problem to solve the potential and the electric field in two dimensional plane perpendicular to $\boldsymbol{B}$. For a linear probe configuration with the equidistant probe-spacing, the measured resistance R is related with magneto-conductivity, σ, as

$$R = \frac{\Delta V}{I} = \frac{\ln 2}{\pi}\frac{1}{\sigma}. \quad (1)$$

It is noted that σ corresponds to the diagonal component of the two-dimensional conductivity (sheet conductivity).

We demonstrate here the basic performance of our magnetic surface transport measurement system by measuring MR of Bi crystal nanofilms. The atomically flat Bi(001) films are prepared on the Si(111)7×7 surface (n-type, 1–10 Ω cm) by Bi deposition at room temperature, followed by post-annealing at 350 K.[5,14,18,19] The high crystallinity of the film was confirmed by RHEED. The film thickness was calibrated with ordered surface phase[20] of $\beta-\sqrt{3}\times\sqrt{3}$-Bi/Si(111) and with thickness of the Bi allotropic transformation.[14,18] Figure 3(a) shows σ of 6 BL Bi(001) films as a function of temperature T ($\boldsymbol{B}=0$). The conductivity has increased by cooling, indicating metallic behavior. Since a Si substrate is semiconductor and the Bi nanofilm is also semiconducting due to quantum size effect, the metallicity in the electron transport is most likely

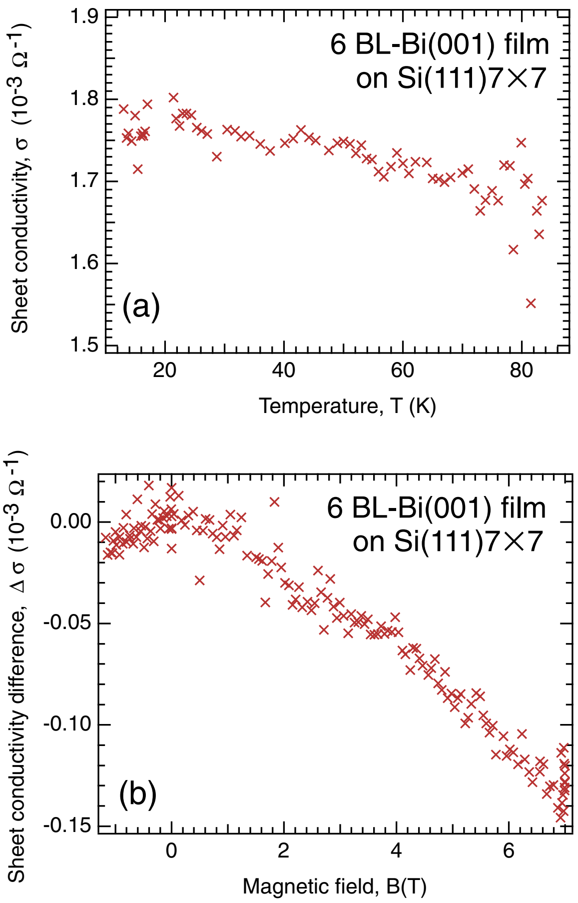

Fig. 3. (Color online) The sheet conductivity, σ, and the sheet conductivity difference, $\Delta\sigma$, of 6 BL Bi(001) films as a function of (a) temperature T ($B = 0$) and (b) magnetic field B ($T = 7.6$ K).

attributed to the metallic surface states of the Bi(001) films.[19] The previous photoemission spectroscopy research reported the surface-state bands of the large Fermi surfaces.[19] The dominancy of surface is due to the large surface/volume ratio in a thin film and such large contribution of surface-states of the Bi(001) nanofilm in electron transport has been also reported by the previous surface conductivity measurements with Micro-FPP.[5] Figure 3(b) shows $\Delta\sigma = \sigma(B) - \sigma(B = 0)$ of 6 BL Bi(001) films as a function of $\boldsymbol{B}$ ($T = 7.6$ K). $\Delta\sigma$ decreases monotonically with magnetic field. This is consistent to classical magneto-transport picture that MR has increased due to bended electron trajectory by the Lorentz force.[8] Absence of any observable localization effect in the present experiment may relate to forbidden back scattering in the Rashba system of the Bi(001) surface states.[19,21]

Figure 4 summarizes the measured MR ratio of Bi films prepared by the present MBE process at various thickness, obtained by

$$\text{MR ratio} = \frac{R(B = 5T) - R(B = 0)}{R(B = 0)}, \quad (2)$$

where $R(B)$ is magneto-resistance. For comparisons, the MR ratio of Bi films measured with Macro-MPP and those prepared by other procedures of MBE,[12] electrodeposition,[10] sputtering,[11] and solidification/annealing[13] in the literatures are also plotted in the figure. Although the

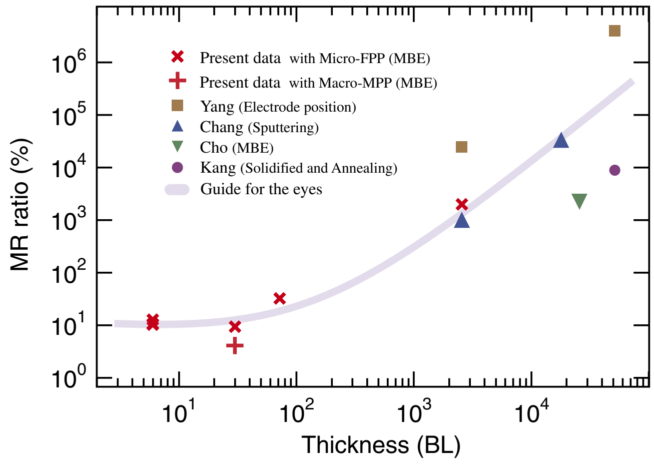

Fig. 4. (Color online) Thickness dependence of the MR ratio of Bi(001) films prepared by MBE (the present reseach with Micro-FPP and Macro-MPP), MBE, electrodeposition, sputtering, and solidification/annealing.

previous values scatter, one notices a systematic tendency that the MR ratio decreases for the thinner films. Then, the MR ratio becomes constant below 30 BL. Such independence of the MR ratio with film thickness indicates contribution of the (metallic) surface-states in magneto-transport. This is consistent to the metallic transport behavior, shown in Fig. 3(a) and to the previous conductivity experiment.[5] We infer a MR ratio of the surface layer is ~10%. It is noted that the surface layer is destroyed in air and the surface effect has been ignored in the previous transport researches.

4. Conclusions

In summary, we have developed an independently-driven double probe-stage system that enables *in situ* magneto-transport measurements on surfaces and ultrathin films, prepared in UHV. With the Micro-FPP method, we have made the measurements on the Bi(001) nanofilms, prepared on Si(111)7×7, at temperature down to 7.6 K and under magnetic field up to 7 T. The system has demonstrated to detect magnetoresistance of even the surface layer.

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