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Petersen, Christian Leth; Grey, Francois; Shiraki, I.; Hasegawa, S.

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Microfour-point probe for studying electronic transport through surface states

C. L. Petersen and F. Grey[a]
Mikroelektronik Centret, Technical University of Denmark, Bldg. 345 East, DK-2800 Lyngby, Denmark

I. Shiraki and S. Hasegawa
Department of Physics, School of Science, University of Tokyo, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-0033, Japan

Microfour-point probes integrated on silicon chips have been fabricated with probe spacings in the range 4–60 μm. They provide a simple robust device for electrical transport measurements at surfaces, bridging the gap between conventional macroscopic four-point probes and scanning tunneling microscopy. Measurements on Si(111) surfaces in ultrahigh vacuum reveal that the Si(111)–$\sqrt{3}\times\sqrt{3}$–Ag structure induced by a monolayer of Ag atoms has a four-point resistance two orders of magnitude lower than that of the Si(111)–7×7 clean surface. We attribute this remarkable difference to direct transport through surface states, which is not observed on the macroscopic scale, presumably due to scattering at atomic steps. © *2000 American Institute of Physics.*
[S0003-6951(00)02749-2]

Clean facets of many crystalline materials exhibit reconstructions of the outer atomic layers, which result in a new two-dimensional band structure at the surface. While the dispersion of these bands can be measured by spectroscopic techniques, the characterization of charge transport in these surface states remains a challenge.[1,2]

Four-point probes with electrode spacings in the millimeter range are not well suited for the study of surface state conductivity, as transport through these states is interrupted by steps and defects on the surface. It is possible to investigate surface state transport indirectly by scanning tunneling microscopy (STM) by studying the scattering of electrons at surface defects.[3–6] Direct transport measurements, however, require a multiprobe technique.

The microfour-point probes described in this letter were made using silicon-based microfabrication technology following a fabrication procedure similar to that for atomic force microscope probes. The probes consist of four sharpened silicon oxide cantilevers, coated with Ti, extending from a silicon support chip.[7] The cantilevers are very flexible, so that contacting a surface is straightforward. Electrode spacings of 8 and 20 μm were used in the experiments described here.

The microscopic probes were integrated in a customized ultrahigh vacuum (UHV) scanning electron microscopy (SEM) system. Microslides moved the four-point probe into contact with the sample surface while the approach was monitored in the UHVSEM, which included an Ag evaporation cell to produce the Si(111)–$\sqrt{3}\times\sqrt{3}$–Ag reconstruction. The microfour-point probe was connected to a custom dc measurement system (current source and a high-precision instrumentation amplifier). A current I applied over the outer electrodes produces a voltage drop V over the inner probes, the four-point resistance defined as $R = V/I$.

The samples were 20×3 mm^2 n-type Si(111) with a nominal resistivity of 10–100 Ω cm. The sample surfaces were patterned to generate large terraces during flashing. The patterning was done using a laser etching facility.[8] To make large step-free terraces, grids of micron-sized small holes were etched with the laser, similar to the patterns used by Ogino.[9] Grid spacings of 5, 10, 15, and 20 μm were used. The samples were heated resistively at 1250 °C in intervals of 10–60 s, while the chamber pressure was kept below 5 $\times10^{-9}$ Torr. The flashing procedure created step bunching due to electromigration. After a total of 3000 s of flashing, the step bunches were roughly aligned to the position of the original hole grid.

The two surface reconstructions investigated in this study were the Si(111)–7×7 and the Si(111)–$\sqrt{3}\times\sqrt{3}$–Ag. Both have a surface state at the Fermi energy, and should thus be conducting. However, the dispersion of the half-filled state on the Si(111)–7×7 surface is very low,[10] and recent STM measurements indicate that this surface has a low conductance.[6] The Si(111)–$\sqrt{3}\times\sqrt{3}$–Ag surface, on the other hand, has a band with strong dispersion at the Fermi level.[2] Macroscopic four-point measurements showed a 10% difference in conductance between the two reconstructions.[11]

The position of single atomic steps was revealed by deposition of around 0.1 monolayers of Ag, which decorates step edges. It was thus confirmed that atomically flat terraces were created between the step bunches. The microscopic four-point probe was then brought into contact with the sample, as shown in Fig. 1(b). The total probe width is larger than the width of the terrace, so the electrodes are positioned on neighboring terraces.

Measurements on the Si(111)–7×7 and Si(111)–$\sqrt{3}\times\sqrt{3}$–Ag surfaces are shown in Fig. 2. These measurements were performed with a probe with 20 μm electrode spacing in a region with 15 μm spaced step bunches. The four-point probe resistance was 24 kΩ on the 7×7 surface and 280 Ω on the $\sqrt{3}\times\sqrt{3}$ surface. The measured values were reproduced
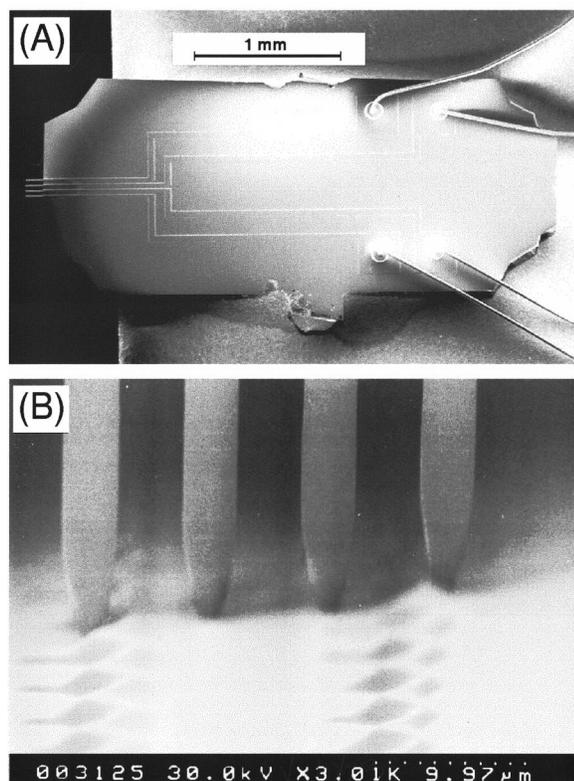

FIG. 1. (a) SEM image of a microscopic four-point probe. The microcanti-levers are connected electrically to bonding pads on a silicon substrate. Thin gold wires are bonded from these pads to the external measurement cir-cuitry. (b) UHVSEM image showing a microfour-point probe with an elec-trode spacing of 8 μm positioned with the inner two electrodes on a single terrace on a Si(111)–7×7 surface.

with two different probes on two different (111) substrates before and after Ag deposition. The difference between the results for the 7×7 and $\sqrt{3} \times \sqrt{3}$ reconstructions, nearly two orders of magnitude in four-point resistance, is remarkably large compared with the 10% difference measured with mac-roscopic probes.[11]
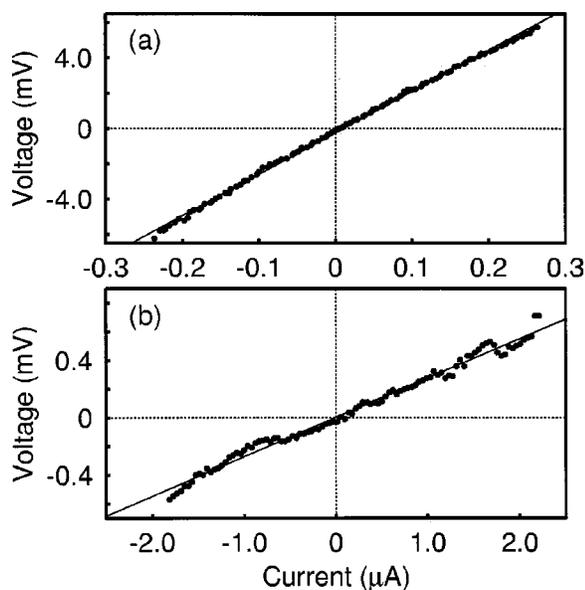

FIG. 2. Linear $I-V$ curves obtained with a microfour-point probe with 20 μm electrode spacing on the Si(111)–7×7 surface (a) and the Si(111)–$\sqrt{3}$ ×$\sqrt{3}$–Ag surface (b) on the same sample.

We suggest two possible explanations. One is that a larger fraction of the current runs in the space charge region for the microscopic probe and thus the effect of the space charge layer is more significant in the microscopic case. This is mainly a geometrical effect. The other possible explana-tion is that a larger fraction of the current runs in the surface states.

Concerning the space charge layer, we note that the clean Si(111) surface has the Fermi level pinned at 0.65 eV above the valence-band maximum.[12] For an n-type 100 Ω cm silicon crystal, the bulk Fermi level is approximately 0.75 eV above the valence-band maximum.[13] The band bending be-neath the 7×7 surface is thus only about 0.1 eV, and so the space charge layer region has a conductance close to that of the bulk. Under these conditions, the sample can be approxi-mated as a semi-infinite system with a uniform homogenous conductance. In this limit, the bulk resistivity ρ can be cal-culated from the four-point probe resistance R as $\rho = 2\pi s R$, where s is the electrode spacing of the probe.[14] A resistivity value of 295 Ω cm is deduced, which is compa-rable with the nominal bulk resistivity of the sample, and hence consistent with the semi-infinite approximation.

Since the four-point probe resistance falls two orders of magnitude after deposition of a monolayer of Ag, we can conclude that the surface channel effectively shorts out the bulk channel in this case. The Fermi level for the $\sqrt{3} \times \sqrt{3}$ surface is pinned at a position 0.1 eV above the valence-band maximum,[2] and thus the band bending is in this case 0.65 eV. The electrode spacing is considerably larger than the space charge layer thickness, as estimated from the nominal bulk resistivity. Therefore, we can approximate the space charge layer as an infinite two-dimensional sheet, and the surface sheet resistivity R_s can then be extracted for the $\sqrt{3} \times \sqrt{3}$ surface as $R_s = R \pi / \ln 2$. This yields a sheet resistance of 1.2×10^3 Ω, or a corresponding conductance of 8.0×10^{-4} Ω^{-1}. For comparison, macroscopic four-point mea-surements by Hasegawa *et al.* show a difference in conduc-tance between the $\sqrt{3} \times \sqrt{3}$ and the 7×7 surfaces of about 1.2×10^{-4} Ω^{-1}.[2] In Fig. 3, the microscopic and macro-scopic results are displayed together with a curve depicting the theoretical variation of the space charge layer conduc-tance σ_{sc} as a function of band bending, calculated from bulk carrier mobilities μ_n, μ_p and the excess carrier densi-ties ΔN, ΔP in the space charge layer[15]

$$\sigma_{sc} = e\mu_n \Delta N + e\mu_p \Delta P.$$

The surface conductance of the $\sqrt{3} \times \sqrt{3}$ surface as measured with the microscopic four-point probe is nearly one order of magnitude higher than expected from band bending alone. We therefore conclude that the extra conductance measured with the microfour-point probe on the $\sqrt{3} \times \sqrt{3}$ surface is due to conduction directly through the surface states.

We suggest two possible explanations for obtaining this result on the microscopic scale. One is that the scattering in the surface state channel is reduced due to the smaller num-ber of atomic steps between the voltage probes. The other explanation is improved electrical contact to the surface states using the flexible microcantilever electrodes. Tests show that the electrodes leave only minimal damage over a contact area of ~100×100 nm².
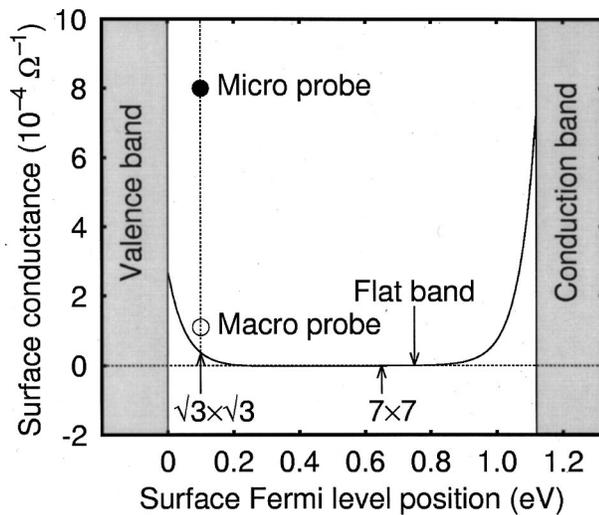
FIG. 3. The solid line shows the calculated space charge layer conductance as a function of the Fermi level position. The closed circle represents the measurement with the microfour-point probe, and the open circle represents a previous measurement performed with a macroscopic four-point probe (Ref. 2).

An estimate of the conductivity of the surface states can be deduced from the difference in conductance between the microprobe measurement and theoretical prediction for band bending alone, $7.6 \times 10^{-4} \ \Omega^{-1}$, and the typical thickness of a reconstructed surface, 1 nm. This yields a resistivity of $1.3 \times 10^{-4} \ \Omega \, \text{cm}$, comparable with the resistivities of bulk metals such as bismuth. The $\sqrt{3} \times \sqrt{3}$ structure has no relationship to metallic silver.[16] Therefore, we emphasize that this metallic conductivity is not due to a thin metallic film, but is an intrinsic property of a surface reconstruction.

A more detailed theoretical analysis of this result will require modeling four-point probe measurements in realistic systems (not semi-infinite or planar),[17] and including effects of step structure and finite contact area. In addition, comparison with theory will require a complete mapping of the band structures of these surfaces.

The technique presented here can clearly be extended to study other surface reconstructions,[17] each having potentially unique electronic transport properties. The microfour-point probe is thus a useful addition to local probe techniques for applications in surface science and technology.

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