Kelvin probe force microscopy of beveled semiconductors

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For the first time, we present the results of Kelvin probe force microscope studies on beveled samples. The ease of sample preparation and simplicity of the measurement make this technique a good candidate for the rapid characterization of semiconductor multilayers. The GaSb/InAs and Ge/SiGe/Si samples presented demonstrate both the utility and the limits of the technique. Beveling has allowed us to easily image quantum wells of 7.5 nm thickness, which is well beyond the resolution available on cleaved samples. Bevel and sample roughness are shown to be the most critical parameters in obtaining good results. © 2002 American Vacuum Society.

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I. INTRODUCTION

The past ten years have seen the swift development of the Kelvin probe force microscopy (KFM) technique.1–3 The ability of KFM to resolve the local potential with a spatial resolution of approximately 50 nm and potential resolution on the order of 0.1 mV has allowed several interesting applications. Mizutani, Arakawa, and Kishimoto measured the potential through cross-sectioned GaAs metal-semiconductor field effect transistors and AlGaAs/InGaAs high electron mobility transistors while they were under bias,4,5 Chavez-Pirson et al. have studied the potential distribution in GaAs/AlGaAs n-i-p-i structures,6 and Robin et al. have studied the potential distribution in an InP/InGaAsP laser diode.7

However, investigation of many modern devices requires a resolution better than 50 nm. Devices including lasers, solar cells and transistors often include quantum wells with well widths as narrow as several angstroms.

As Jacobs et al. demonstrated, the measured KFM potential does not exactly match the surface potential below the probe tip, but rather it is a weighted average of all the potentials on the surface with the derivative of probe-surface capacitances versus distance for each point on the surface as the weighting factors8

\[
\Phi_{\text{meas}} = \sum_{i=1}^{n} \left( C'_{ii} \Phi_i \right) \left/ \sum_{i=1}^{n} C'_{ii} \right.,
\]

where \( \Phi_{\text{meas}} \) is the KFM signal, \( \Phi_i \) is the local surface potential at point \( i \) on the surface, and \( C'_{ii} \) is the derivative of the capacitance versus distance between the tip and point \( i \) on the surface.

Therefore increasing the capacitive coupling between the probe tip and the area directly beneath it, while simultaneously minimizing the stray capacitances, is one method of improving resolution. There are many examples of this technique being applied through the use of high-aspect ratio probes.8

We propose sample beveling as a technique to improve spatial resolution. Some pioneering work on beveling as a way to improve resolution has been done by F. Giannazzo et al.9 for scanning capacitance microscopy (SCM). A bevel exposes a cross section of the sample as a surface and provides a magnification equal to \( \sin^{-1} \theta \), where \( \theta \) is the bevel angle. The effect of this magnification is to present regions where the potential is changing over a much larger surface area. Referring back to Eq. (1), the capacitive coupling is not changed by the bevel, but the potential \( \Phi_i \) changes more slowly versus distance. The slow variation in potential reduces the effect of the stray capacitance—as the potential being coupled by the stray capacitance is similar to that under the probe tip, the error introduced in the KFM signal is small.

Beveling is widely used in other semiconductor characterization techniques, particularly spreading resistance profiling (SRP),10 and the equipment needed to prepare bevels is widespread and inexpensive. With our beveling equipment it is possible to make bevels with magnifications of \( 10 \times, 20 \times, 50 \times, 100 \times, 200 \times, 400 \times , \) and \( 800 \times \). When beveled at a magnification of \( 800 \times \), a 10 nm quantum well appears as a broad stripe 8 \( \mu m \) wide.

The signal measured by KFM is a true surface signal. This is in contrast to techniques such as Raman spectroscopy, secondary ion mass spectroscopy (SIMS), SCM, and SRP, which also often use beveled samples, where the signal is generated from a volume of the sample. The sensitivity of KFM to surface conditions allows KFM to be scaled to extremely high spatial resolution with beveling as the presence of a single layer of atoms on the surface can be detected.11

The surface sensitivity of KFM also makes sample preparation particularly important.

II. EXPERIMENT AND DISCUSSION

In this article, we present KFM studies of two beveled samples in two different material systems, which demonstrate the utility as well as the limits of this technique. All
images were acquired using a Digital Instruments Nanoscope III controlled by a Dimension 3100 controller. The probes were Nanosensors PointProbe electrostatic force microscopy tips with a PtIr5 metallic coating and typical tip radius of approximately 25 nm. The first sample consists of a 20 nm InAs quantum well surrounded by GaSb cladding layers. The second sample is a 7.5 nm Ge quantum well surrounded by Si$_{0.33}$Ge$_{0.67}$ cladding layers. In this sample, a Si capping layer is also present.

The GaSb/InAs sample was prepared by traditional diamond beveling of the type used to prepare samples for SRP analysis. Polycrystalline diamond with a maximum diameter of 0.1 mm was used as the grinding material. A bevel angle of 4' was chosen to produce a magnification of 800×. After beveling the surface was wiped clean with a sequence of Kemet CO-42, a proprietary solvent with properties similar to trichloroethylene and methanol.

In Fig. 1(a), there is very clear contrast in the surface potential between the InAs quantum well and the GaSb cladding layers. The 20 nm QW appears as a broad stripe of 16 μm after beveling to a magnification of 800×. The oscillation along both edges of the QW is due to the bevel roughness. (b) A cross section of (a) showing the KFM potential perpendicular to the QW. (c) A schematic image of the troughs and peaks generated by the diamond bevel exposing both materials A and B to the surface at the heterointerfaces.

Fig. 2. Peak-to-peak roughness of a bevel produced by diamond polishing is 10 nm (top curve). By substituting Syton for diamond, the bevel roughness can be greatly reduced (bottom curve).

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Fig. 1. (a) KFM image of an InAs quantum well (QW) surrounded by GaSb cladding layers. The 20 nm QW appears as a broad stripe of 16 μm after beveling to a magnification of 800×. The oscillation along both edges of the QW is due to the bevel roughness. (b) A cross section of (a) showing the KFM potential perpendicular to the QW. (c) A schematic image of the troughs and peaks generated by the diamond bevel exposing both materials A and B to the surface at the heterointerfaces.

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In Fig. 1(a), there is very clear contrast in the surface potential between the InAs quantum well and the GaSb cladding layers. The 20 nm quantum well can be easily seen as a broad dark stripe of 16 μm (20 nm×800). A cross section perpendicular to the quantum well is given in Fig. 1(b), illustrating the quite abrupt transition between the two materials. The beveling/KFM technique provides a quick and inexpensive method for accurate calibration of semiconductor layer thicknesses. Total turnaround time for this measurement including sample preparation was less than 30 min.

The ability to image thin layers, on beveled samples, is not limited by the probe dimensions (though a high-aspect ratio probe should still be used) but rather by the material quality of the sample and the beveling technique used.

Surface roughness due to the bevel [see Fig. 1(c)] can lead to inaccuracy in the KFM measurement. The troughs of the bevel expose the Material “A” while the peaks of the bevel expose Material “B.” This distorts thin layers and exposes them to the surface with the same characteristic oscillation as the bevel. Standard mechanical beveling using 0.1 μm polycrystalline diamond results in a peak-to-peak roughness of approximately 10 nm. Figure 2 (top curve) shows the
peak-to-peak roughness of the GaSb/InAs sample after beveling and this oscillation is clearly manifested in the edges of the quantum well in Fig. 1.

To image thinner layer structures successfully, beveling techniques producing less surface roughness are needed. We have adapted the traditional diamond polishing setup to use Syton, a common agent in chemical-mechanical polishing in an effort to reduce the surface roughness.\textsuperscript{12} Though Syton is intended for polishing of silicon, we have found that it also gives good results for germanium and alloys of all compositions of SiGe. Typical peak-to-peak roughness is less than 1 nm.\textsuperscript{12} This improvement in surface roughness can be clearly seen in Fig. 2 (bottom curve). Using this technique, the Ge/SiGe/Si sample was prepared with a bevel angle of 34° to give a magnification of 100×.

In Fig. 3(a), the 7.5 nm Ge quantum well is clearly visible as the dark stripe in the center of the image. In Fig. 3(b), the cross section perpendicular to the quantum well is given, showing the transition between the two materials and the influence of the material roughness in more detail.

Here the quality of the KFM image is not limited by the surface roughness of the bevel, which is less than 1 nm, but rather by the peak-to-peak surface roughness of the as-grown sample, which is 15 nm [see Fig. 3(c)]. This large roughness is due to the strain mismatch between the SiGe layers and the Si substrate.\textsuperscript{13} A flat bevel plane will cut through the corrugated quantum well in several places, resulting in an aliasing effect where one quantum well will actually appear as several parallel stripes. This aliasing is apparent in Fig. 3(a).

Bulk and surface properties both contribute to the magnitude of the measured KFM signal. As our KFM studies were done under ambient conditions, the measured KFM signal cannot be used to reveal bulk properties, such as alloy composition or concentration of the dopants. However, it should be feasible to perform KFM measurements in beveled samples under UHV conditions where surface effects can be accurately quantified. The extracted bulk potential, a very useful property in its own right, could then be related to the alloy composition and the concentration of dopants.

III. CONCLUSIONS

In conclusion, we have combined sample beveling with Kelvin probe force microscopy and for the first time have effectively imaged quantum wells as thin as 7.5 nm.

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