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Friction force microscopy characterization of semiconductor heterostructures

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Abstract

Measurement of frictional forces in a scanning force microscopy has been applied to perform compositional characterization of semiconductor heterostructures. Semiconductor interfaces as well as multiquantum wells are resolved with 3 nm of spatial resolution. The chemical sensitivity of this method is studied by imaging a step graded $In_xGa_{1-x}As$ sample. Changes of 10% in indium (or gallium) composition are detected. These results point out the potential of friction force microscopy for simultaneous topography and compositional characterization of semiconductor materials.

Keywords: Gallium; Indium; Multiquantum wells; Semiconductors

1. Introduction

Since its invention [1], the scanning force microscopy (SFM) has demonstrated its ability to probe the topography of a wide variety of surfaces with atomic and nanometer resolution [2–5]. In conventional SFM (contact mode), a sharp tip attached to a cantilever beam scans in a raster fashion the sample while tip and sample are in mechanical contact. The topography of sample surface is reproduced by keeping constant the normal force between tip and sample. Recently, the measurement of the lateral forces between the tip and the sample during the scanning has produced a new scanning probe method, called friction force microscopy (FFM). This has allowed the study and understanding of tribological phenomena of the surfaces at nanometric scale, such friction, wear and lubrication [6–10]. FFM studies of silicon surfaces used for magnetic recording [11], studies of the lubricant properties of thin organic layers films [8,12] are some of the latest examples of the applications of this technique.

Frictional forces could also be used to perform chemical maps of surfaces at nanometer level. The frictional response can be related to the chemical composition of the surface [13]. This has been applied to imaging with chemical contrast the spatial arrangements of different chemical species in organic films, such as composite Langmuir–Blodgett films and patterned self assembled monolayers [14,15]. Furthermore, recent works have used specific interactions between the tip and the sample to map chemical components. One promising approach changes the pH of an electrolyte solution to distinguish chemical species by their different isoelectronic point [16]. Another approach uses chemically functionalized tips to map specific functional groups by molecular recognition [17].

In this work, we propose the use of FFM to obtain chemical maps of semiconductor heterostructures with nanometer resolution. Frictional force images of semiconductor interfaces, multiquantum wells and compositional step graded structures are presented. The lateral resolution as well as chemical sensitivity are discussed.

2. Experimental details

Fig. 1 shows the standard scheme used to measure the frictional force between tip and sample [18,19]. The tip-sample force produces the cantilever deflection. This is measured by changes in the position of a laser spot reflected from the cantilever and collected in a segmented quartered photodiode. The signal difference between upper and lower sectors is zero in the absence of tip-sample interaction. When the tip is in contact with the sample the repulsive force gives a positive signal difference between upper and lower sectors (Fig.

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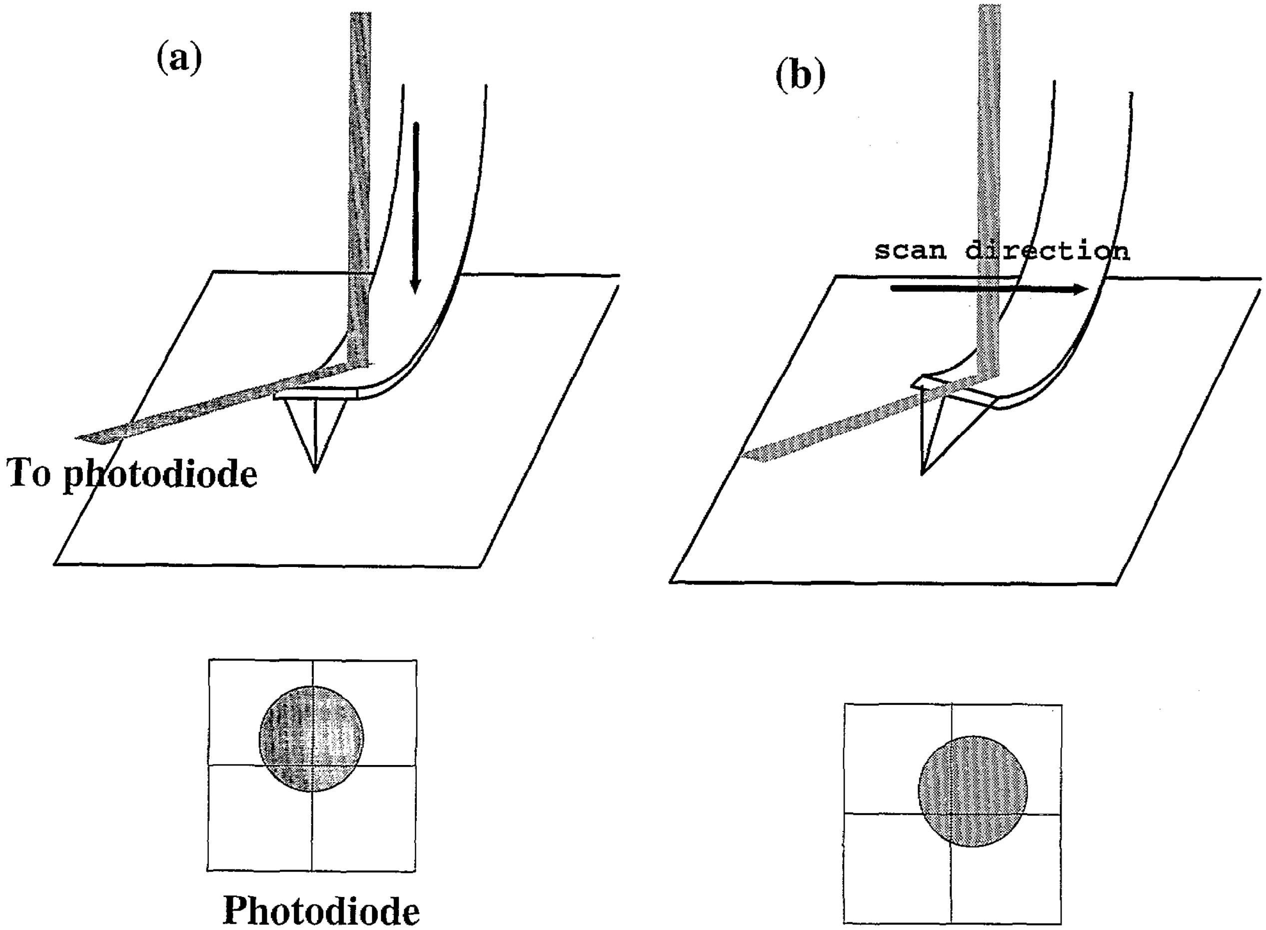

Fig. 1. Scheme of the detection of the normal and lateral forces between the cantilever and the sample. When there is mechanical contact between the tip and the sample but not relative movement (a) and during imaging (b).

1(a)). During imaging, a frictional force opposite to the cantilever movement causes its torsion, providing a signal difference between the left and right sectors (Fig. 1(b)).

The deflection and torsion of the cantilever lead to a displacement of the laser spot in orthogonal directions. Then, topographic and frictional images can be acquired simultaneous and independently with an adequate experimental set up. In our configuration, the plane defined by the incident and reflected laser beam coincides with the plane formed by the cantilever long axis and the photodiode, preventing any cross talking between normal and lateral forces.

In this work, InP/InSb interfaces, GaInSb/AlInSb multiquantum wells and a step graded $In_xGa_{1-x}As$ structure have been studied. The semiconductor structures were grown under ultra high vacuum by molecular beam epitaxy. After that, the samples were cleaved so as to expose the (110) face for examination. They were mounted in a special cell that allows environmental control of the relative humidity as well as micropositioning of the tip on epitaxial layers. Electronics and software came from Nanoscope III (Digital instruments, Santa Barbara, CA). The experiments have been performed with beam-shaped Si_3N_4 cantilevers with sharpened tips. The nominal curvature radius is about 10 nm and bending elastic constant 0.05 N m^{-1} (Olympus, Japan). The torsional constant is 158 N m^{-1} and unlike V-shaped cantilevers, the beam-shaped cantilevers have torsional constants independent of load and lateral force [20].

3. Results and discussion

In Fig. 2, topographic and friction images obtained simultaneously on the same region of an InP/InSb interface are presented. The topographic image shows several multiatomic steps produced during cleavage (Fig. 2(a)). The position of the interface boundary can not be inferred from this image. However the frictional image shows a clear change in contrast between InP and InSb regions that allows the location of the boundary (Fig. 2(b)). Higher frictional force corresponds to the InP region (brighter in the image). The image was taken with an applied normal force of 5 nN, providing frictional force of 4.2 and 1.7 nN for InP and InSb regions, respectively.

Although steep topographic features can contribute to lateral force signals, this can always be separated from the contributions of chemical origin. This is easily visualized by comparing the forward and backward scan lines obtained on InP/InSb interface (Fig. 3). Fig. 3(a) shows a topographic scan line, the topographic features are independent of scanning direction. In Fig. 3(b), the forward and backward lateral force scanlines constitute a hysteresis cycle. The steps shown on Fig. 3(a) produce depressions in the lateral force whose sign does not change with the scanning direction. In the forward direction, a slight decrease of friction force happens when the tip scans down the steps. However, in the backward direction, a remarkable increase of friction force appears when the tip climbs up the multi-atomic steps. This hysteresis of the friction force can be related with the activation barriers for diffusion across step edges [18,21].

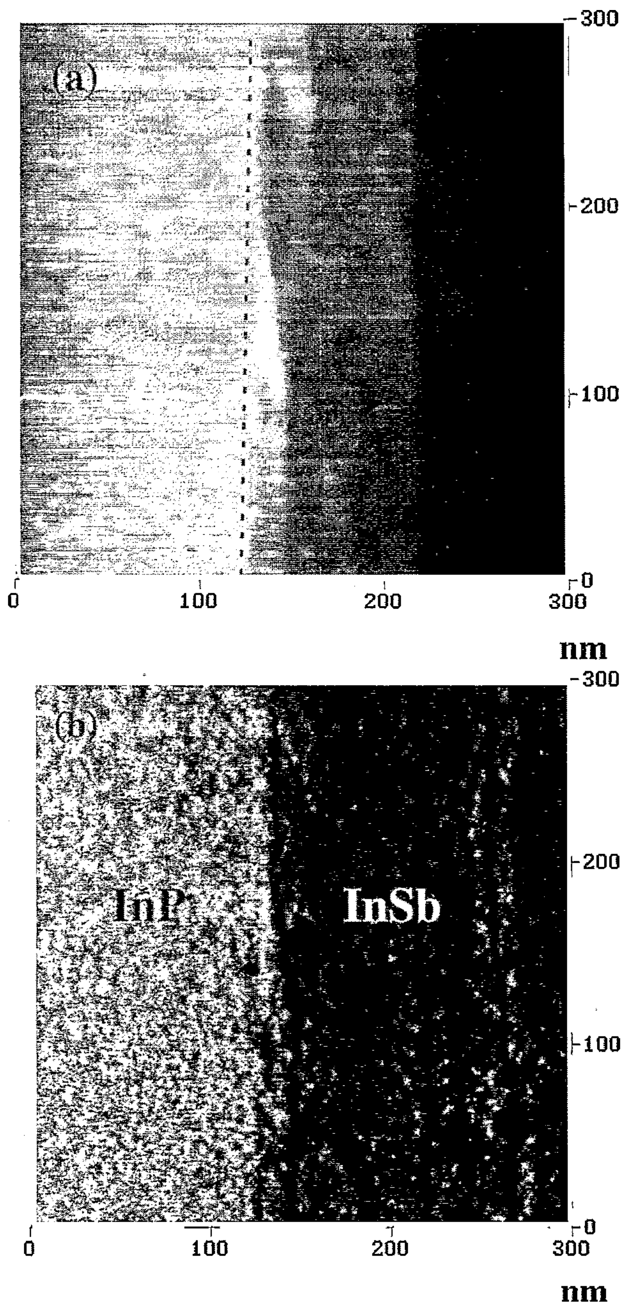

Fig. 2. Topographic and friction image of InP/InSb interface, taken simultaneously with a total force of 5 nN (adhesion force of 3.5 nN). (a) Topographic image. Brighter gray levels correspond to higher heights. Several steps were produced as a result of the cleavage. The three main steps have heights of 0.98, 1.3, 2.2 nm. The position of the boundary inferred by FFM is indicated as a dashed line. (b) FFM image. Bright regions (higher friction force) correspond to InP. Relative humidity of 40%.

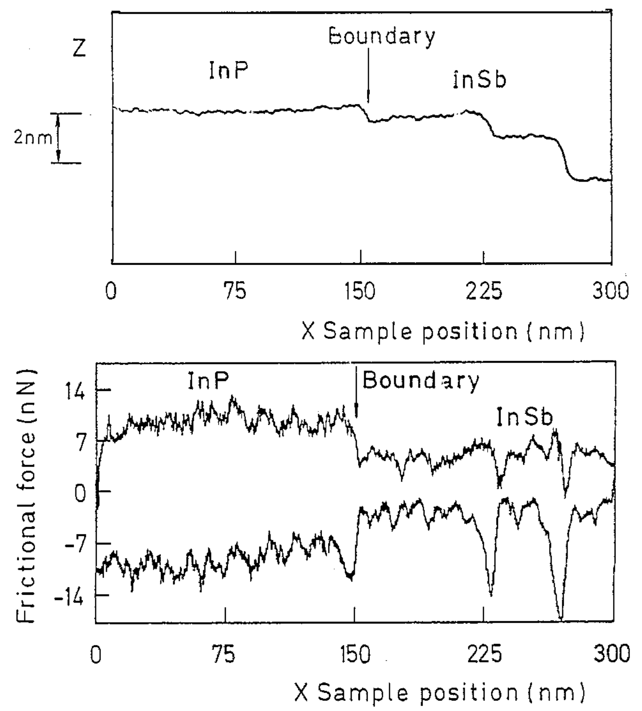

Fig. 3. Forward and backward scan line of InP/InSb interface. (a) Topographic scan line. The topographic lines overlap. (b) Lateral force scan lines.

On the contrary, frictional contributions are always opposite to cantilever's motion. From the hysteresis cycle can be estimated a ratio of frictional forces between InP (left) and InSb (right) of 2.6. This value was roughly independent of the load in the range between 0 and 40 nN.

Frictional forces imply energy dissipation. This is illustrated by recording the variation of the lateral force during forward and backward tip-sample displacement (Fig. 3(b)). The area enclosed by this friction loop represents the energy dissipated per cycle. It is important to realize that energy dissipation may not imply modification of the sample surface. We do not observe structural changes of the surface after repeated scanning. The loads applied in these experiments have been

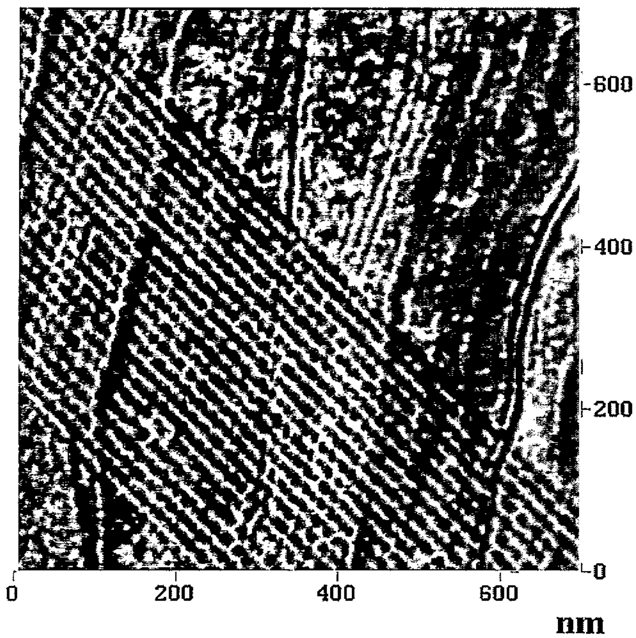

Fig. 4. Friction image of ten quantum wells (parallel straight lines) of GaInSb/AlInSb. The AlInSb provide higher friction (darker regions). The contour of multiatomic steps (irregular lines) is also seen in the image.

kept below 10 nN. With these values, we estimate an upper limit for the energy dissipated per atom of about 0.14 eV. This value is smaller than the cohesive energy III–V semiconductors per bond, 1.5–2 eV. Therefore, the images have been taken in the regime of friction without wear. This shows the non destructive character of FFM to characterize semiconductor structures.

As an example of the potential of FFM to map chemical variations, GaInSb/AlInSb multiquantum wells have been imaged (Fig. 4). Alternating regions of 10 nm width are clearly separated. The AlInSb components provide higher friction (darker regions).

The lateral resolution is determined by the contact area between the tip and the sample. The contact area depends on the tip radius, Young's modulus of the tip and the sample, the applied load and the adhesion energy between the tip and the sample [22]. Sharp tips, low loads and low surface energies are required to optimize the lateral resolution. In a previous work [23], a standard sample of InP/InGaAs multiquantum wells was grown to determine experimentally the compositional lateral resolutions. There, a spatial compositional resolution of 3 nm was demonstrated. Theoretical calculations (J. Tamayo and R. García, unpublished results), indicates that this spatial resolution could be improved by using sharper tips and under environmental conditions that give very small adhesion forces.

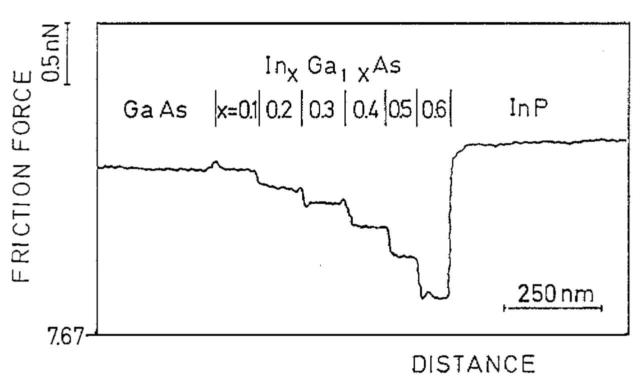

Fig. 5. FFM cross-section of a step graded $In_xGa_{1-x}As$ structure. The indium and gallium composition is changed in steps of 10% from GaAs to $In_{0.6}Ga_{0.4}As$. The structure is completed with an InP capping layer. The profile is an average of 300 FFM scan lines.

In Fig. 5, a FFM profile of a $In_xGa_{1-x}As$ structure on GaAs (100) is shown. The indium concentration (x) was varied from 0 to 60% in 10% steps. The cross-section shows five levels associated with regions with indium concentration between 10 and 60%. The frictional force decreases as the indium concentration increases with respect to gallium. However, the transition between GaAs and $In_{0.1}Ga_{0.9}As$ is not detected. It seems that sensitivity to compositional variation increases with the amount of indium. From Fig. 5, it is inferred that for indium compositions higher than 30%, compositional changes smaller than 5% could be detected.

To improve the chemical sensitivity, cantilevers with smaller torsional elastic constants are desirable (these are about three orders of magnitude higher than normal elastic constants). The signal-noise ratio is also improved, when higher normal forces are applied. The frictional force is roughly proportional to the normal force (Amonton's law). However, high normal forces decrease the lateral resolution. A compromise between lateral resolution and chemical sensitivity is required.

4. Conclusions

It has been showed that frictional forces allow us to obtain chemical maps of semiconductor heterostructures. Compositional lateral resolutions of 3 nm have been demonstrated. Also, the friction force microscopy has a high chemical sensitivity. Measurements of frictional forces in $In_xGa_{1-x}As$ samples demonstrate that variations smaller than 10% of indium composition can be detected.

Although, further experiments are needed to establish the precise relationship between frictional forces and indium (or gallium) composition, these results provide the first attempt to quantify chemical composition with nanometer resolution by friction force microscopy.

This work shows the ability of friction force microscopy to assess the quality and to characterize defects of the semiconductor structures. The technique presents several advantages as its sensitivity, its high compositional lateral resolution, general applicability and non destructive character. Among possible applications can be mentioned the characterization of the interface boundaries and the measurement of thickness and composition in complex structures as buffer layers.

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