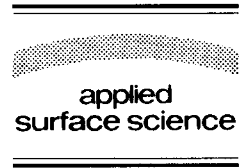
ELSEVIER

Applied Surface Science 123/124 (1998) 339–342



Characterization of semiconductor heterostructures and quantum dots by friction force microscopy

Javier Tamayo, Ricardo García *

Instituto de Microelectrónica de Madrid, CNM, CSIC, Parque Tecnológico de Madrid, Isaac Newton 8, Tres Cantos, 28760 Madrid, Spain

Abstract

The capability of friction force microscopy to obtain compositional maps of semiconductor structures grown by molecular beam epitaxy was studied. Experiments on InP/InGaAs multiquantum wells determined a compositional spatial resolution of 3 nm. Additionally, variations in indium concentration smaller than 10% were detected in $In_xGa_{1-x}As$ structures. Friction maps of InAs and InSb quantum dots on InP(001) are also presented. The experimental results show that the frictional force is sensitive to the presence or absence of a wetting monolayer. Based on these experiments, the potential of friction force microscopy to develop a spatially resolved spectroscopy is discussed. © 1998 Elsevier Science B.V.

PACS: 61.16.Ch; 81.05.Ea; 81.40.Pq

Keywords: Friction force microscopy; Semiconductor heterostructures; Compositional maps

1. Introduction

Since its invention [1], the scanning force microscope (SFM) has demonstrated its ability and versatility to image the topography of a wide variety of surfaces with atomic and molecular resolution [2,3]. A desirable goal to be achieved by the SFM is to determine the chemistry of the surfaces with atomic and molecular resolution. The measurement of frictional forces that appear between the tip and the sample during the scanning opens interesting possibilities [4]. It was demonstrated that the frictional force is sensitive to the chemical composition of the surface probed by the tip. Thus, compositional contrast in mixed organic films, C_{60} islands on NaCl and GeS, were obtained, to name a few cases [5–10]. In a friction force microscope, topography and compositional contrast can be obtained simultaneously and independently.

These results open the possibility of using friction to develop a spatially resolved spectroscopy [11]. To accomplish this it is necessary to determine the spatial resolution, the chemical sensitivity and the depth of the sample that contributes significantly to the frictional force. A theoretical model that provides the interfacial properties involved in energy dissipation at atomic and nanometer scales is also required.

In this work, we explore the capability of friction force microscopy (FFM) to obtain compositional maps of semiconductor surfaces [9,10,12]. In this way, we have used InP/InGaAs multiquantum wells and quantum dots (QD) formed after deposition of InAs and InSb on InP(001). A spatial resolution of 3 nm is obtained in InP/InGaAs multiquantum wells.

* Corresponding author. E-mail: rgarcia@imm.cnm.csic.es.

0169-4332/98/$19.00 © 1998 Elsevier Science B.V. All rights reserved.
PII S0169-4332(97)00467-4

It is also demonstrated that FFM is sensitive to the presence or absence of wetting monolayer in QD systems [13]. Finally, the capabilities and present difficulties of friction force microscopy to obtain true chemical maps will be discussed.

2. Experiments and discussion

The semiconductor structures were grown under ultra high vacuum by molecular beam epitaxy. They were mounted in a special SFM cell that allows environmental control of the relative humidity as well as micropositioning of the tip on epitaxial layers. We have used a commercially available SFM (Nanoscope III, Digital Instruments). The experiments have been performed with sharpened Si_3N_4 cantilevers with nominal radii below 20 nm (Olympus, Japan). Absolute values of frictional force are calculated by a method similar to the one used in Ref. [11].

To determine the ability of FFM to map chemical variations a test sample of $InP/In_{0.53}Ga_{0.47}As$ multi-quantum wells of 10, 5, 4, 3 and 2 nm thickness was grown.

Fig. 1 shows topographic and friction cross-sections of a region with 3, 4 and 5 nm structures. The topographic cross-section shows a biatomic step, however the location of InP and InGaAs regions can not be inferred. On the contrary, the frictional profile clearly differentiates InP and InGaAs regions. Previous experiments on InP/InGaAs epitaxies allow us to associate higher friction regions with InP.

The spatial resolution obtained above was 3 nm. This is determined by the contact area between the tip and the sample. The contact area depends on the curvature radius of the tip, the load applied, the elastic moduli and surface energies. Sharp tips, low loads and environments with low adhesion forces are needed to improve the spatial resolution. Calculations indicate that with tips of radius of 5 nm reproducible subnanometer spatial resolution could be achieved [9].

The presence of frictional forces does not imply damage or wear of the sample. Repeated imaging of the same area shows no structural changes. In 1929, Tomlinson proposed a model that provides dissipation energy with only elastic interactions [14]. Subsequent models based on this demonstrated that energy dissipation is possible without plastic deformations by stick and slip processes [15]. In the range of the applied forces, the dissipated energy per atom is smaller than 0.1 eV, i.e. about one order the magnitude smaller than the cohesive energy between atoms of III–V semiconductors compounds.

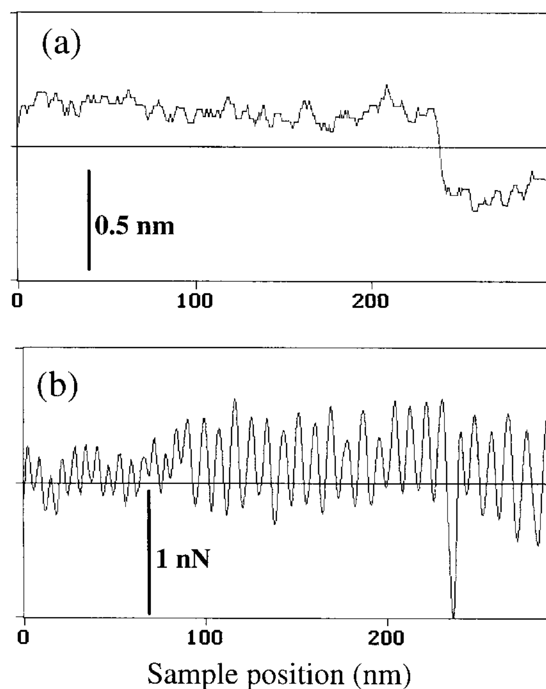


Fig. 1. Topography (a) and frictional cross-sections (b) of InP/InGaAs multiquantum wells. The sample presents thicknesses of 3, 4 and 5 nm (from left to right). The total normal force applied is 4.6 nN. (a) A biatomic step is produced during the cleavage. (b) The friction profile differentiates higher friction regions (InP) from lower friction regions (InGaAs).

We have also studied the chemical sensitivity of FFM. A step graded $In_xGa_{1-x}As$ structure was grown where the relative concentration of indium (x) was changed in steps of 10%. The results showed that indium composition variations smaller than 10% can be detected [12].

Friction force microscopy has been also applied to determine the presence or absence of a wetting layer in quantum dot systems. We have examined 3.5 ML of InAs on InP(001) and 2 ML of InSb on InP(001). No capping layer was grown on the quantum dots.

Fig. 2 shows a topography and friction image of QD's formed after deposition of 3.5 ML of InAs on

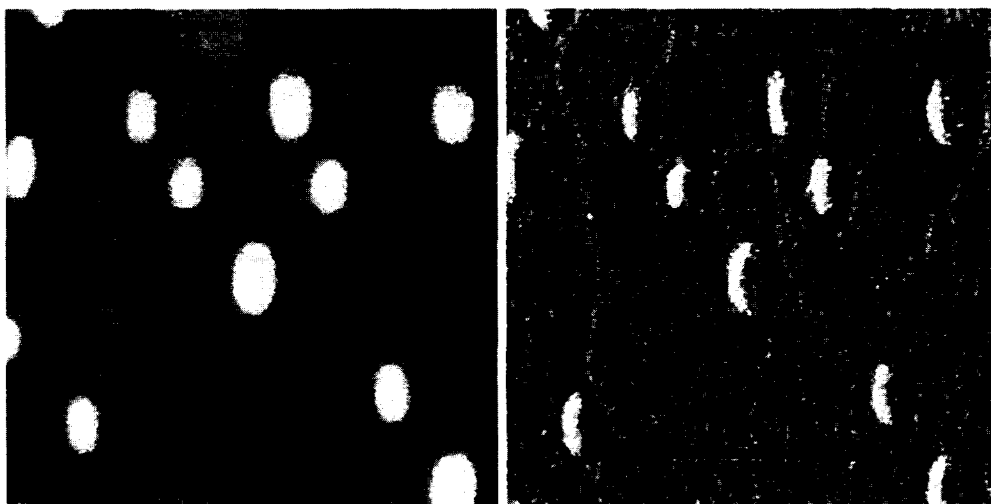


Fig. 2. (a) Topographic image of 3.5 ML of InAs on InP(001). The image shows several monoatomic terraces in the regions between dots. The total normal force is 5.8 nN. (b) Friction force image. The friction force on the dot's plateau is the same as on the substrate, 2.5 nN. The variations of the friction force in the dots' edges come from abrupt topographic changes.

InP. The topographic image shows the dots as truncated pyramids. The average lateral dimensions are 55×45 nm^2 and the average height is 4.8 nm. The lateral dimensions are slightly overestimated by the finite tip's size. The same frictional force was measured on top of the dot than on the substrate. This suggests an uniform chemical composition across the surface. We conclude that a wetting layer of InAs covers the InP.

This result is confirmed by transmission electron microscopy analysis [16]. There, a wetting layer of 1.4–1.5 ML was estimated for InAs coverages larger than 1.8 ML. This indicates that about 1.5 ML InAs provide the same frictional force as 16 ML.

We have also examined a QD system formed when 2 ML of InSb are deposited on InP. The topographic image is similar to Fig. 2. The lateral dimensions of the dots were 72×51 nm^2 and the height was 18 nm (mean values). Fig. 3 shows topographic and lateral force cross-sections of a representative dot. The lateral force cross-sections (forward and backward scans) form a hysteresis loop. Topography and friction contribute to the lateral force profile. Topography produces peaks in the lateral force when the tip runs into the dot's edges. The friction contribution is associated with the physical and chemical properties of the sample. The sign of the topographic contribution is the same for forward and backward scan lines. However, the sign of frictional forces is always opposite to the direction of tip motion. The area enclosed by the friction loop represents the dissipated energy in each cycle.

A frictional force of 7.0 nN was measured in the region between dots, while 2.5 nN was measured on the top of the dot. We associated this remarkable change of friction with a different chemical composition of the substrate and the dot. This suggests that less than 1 ML of InSb has been formed on the InP surface. Photoluminescence experiments confirm this result, indicating a wetting layer thickness about 0.7 ML [13]. The surface composition of the substrate is probably an $In_xSb_{1-x}P$ alloy.

These results show the potential of FFM for compositional mapping of semiconductor structures. However, to develop a spatially resolved spectroscopy based on friction, a more quantitative study is necessary [13]. This requires a theoretical model that relates the measured frictional force with the tip–sample contact area, interatomic potential and probably with the phonon spectra of the interface.

Experiments with different tips provide different frictional forces because the contact area depends on geometry of the tip. We have also found a dependence of frictional forces on the relative humidity.

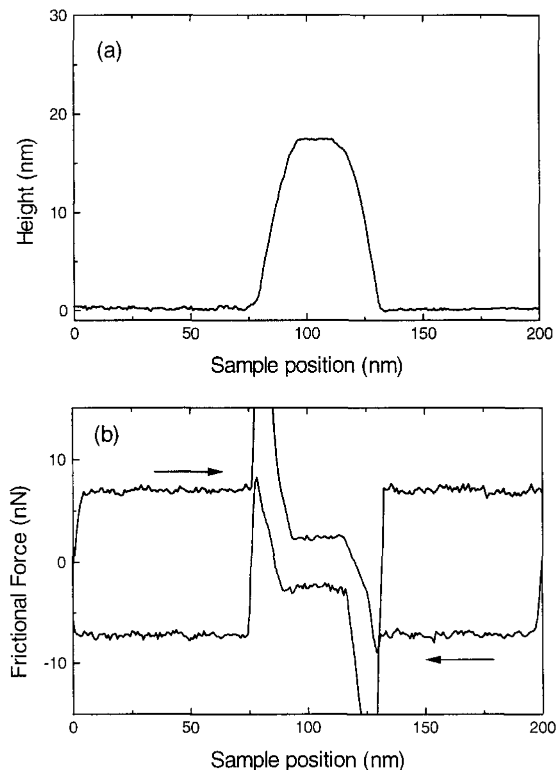


Fig. 3. Topography (a) and lateral force cross-sections (b) of InSb dot formed after 2 ML deposition on InP(001). The effective normal force is 7.2 nN. A friction force of 7 nN is measured on the substrate, roughly three times the value on dot's plateau. The arrows indicate the scanning direction.

These results emphasize the need of an experimental method for in situ determination of tip–sample contact area and the use of tips with well defined geometry [17,18].

3. Conclusion

In this work, friction force microscopy was applied to obtain chemical maps of semiconductors at nanometer level. A compositional lateral resolution of 3 nm was obtained in InP/InGaAs interfaces. Also, variations smaller than 10% of indium concentration were detected in $In_xGa_{1-x}As$ structures. Measurements on quantum dots of InAs and InSb on InP demonstrated that the frictional force depends mainly on the properties of the outer monolayer of the sample. As a consequence, friction force microscopy is suitable for studies of wetting layer formation.

Acknowledgements

We are grateful to F. Briones, L. Gonzãlez, Y. Gonzãlez and T. Utzmeier for sample preparation. This work has been supported by Direcciõn de Investigaciõn Científica y Tecnica of Spain (DGICYT) (PB94-0016).

References

[1] G. Binnig, C.F. Quate, Ch. Gerber, Phys. Rev. Lett. 56 (1986) 930.
[2] Y. Sugurawam, M. Otha, H. Ueyama, S. Morita, Science 270 (1995) 1646.
[3] F.A. Schabert, C. Henn, A. Engel, Science 268 (1995) 92.
[4] C.M. Mate, G.M. McClelland, R. Erlandson, Phys. Rev. Lett. 59 (1987) 1945.
[5] R.M. Overney, E. Meyer, J. Frommer, D. Brodbeck, R. Lüthi, L. Howald, H.-J. Güntherodt, M. Fujihira, H. Takano, Y. Goth, Nature 359 (1992) 133.
[6] C.D. Frisbie, L.F. Rozsnyai, A. Noy, M.S. Wrighton, C.M. Lieber, Science 265 (1994) 2071.
[7] R. Lüthi, H. Haefke, E. Meyer, L. Howald, H.-P. Lang, G. Gerth, H.-J. Güntherodt, Z. Phys. B 95 (1994) 1.
[8] U.D. Schwarz, W. Allers, G. Gensterblum, R. Wiesendanger, Phys. Rev. B 52 (1995) 14976.
[9] J. Tamayo, L. González, Y. González, R. García, Appl. Phys. Lett. 68 (1996) 2297.
[10] J. Tamayo, R. García, Mater. Sci. Eng. 42 (1996) 122.
[11] E. Meyer, R. Lüthi, L. Howald, M. Bammerlin, M. Guggisberg, H.-J. Güntherodt, L. Scandella, J. Gobrecht, A. Schumaker, in: R. Prins, B.N.J. Persson, E. Tossati (Eds.), Physics of Sliding Friction, Kluwer, Dordrecht, 1996, p. 349.
[12] R. García, J. Tamayo, L. González, Y. González, in: B. Bhushan (Ed.), Micro/Nanotribology, Kluwer, Dordrecht, 1997, p. 275.
[13] J. Tamayo, R. García, T. Utzmeier, F. Briones, Phys. Rev. B 55 (1997) R13436.
[14] G.A. Tomlinson, Phil. Mag. Ser. 7 (1929) 905.
[15] I.L. Singer, J. Vac. Sci. Technol. A 12 (1994) 2605.
[16] J. Groenen, A. Mlayah, R. Carles, A. Ponchet, A. Le Corre, S. Salaün, Appl. Phys. Lett. 69 (1996) 943.
[17] U.D. Schwarz, O. Zwöner, P. Köster, R. Wiesendanger, in: B. Bhushan (Ed.), Micro/Nanotribology, Kluwer, Dordrecht, 1997.
[18] R.W. Carpick, D.F. Ogletree, M. Salmeron, Appl. Phys. Lett. 70 (1997) 1548.