

# Surface physics at Saha Institute

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We report in this article our present activities on the study of various aspects of surface and interface physics carried out at Surface Physics Division of Saha Institute of Nuclear Physics. These include growing of materials and their characterization using techniques available at the institute. For growing materials, we have a magnetron sputtering unit, Langmuir–Blodgett trough and polymer spin coater. The analytical tools are grazing incidence X-ray reflectivity, scanning probe microscopy and secondary ion mass spectrometry. A set-up to carry out bombardment-induced light emission spectroscopy has been developed to understand the atomic excitation processes in sputtering as well as to explore its potentiality as a surface analytical tool. We have presented some of the recent results obtained in our laboratory.

## 1. Introduction

Ultra-thin single and multi-layered films play an important role in various fields of modern-day science and technology. Novel devices are being extensively fabricated using thin film technology and the role of the structure and composition at the surfaces and interfaces of the materials governs the performance of such devices. Because of high surface-to-volume ratios, the study of surfaces and interfaces is crucial in the understanding of many technologically important processes as well as the physics of low-dimensions. The present paper describes the surface analytical facilities at Saha Institute of Nuclear Physics and highlights some of the important work being carried out in this direction.

The presently available thin film growth facilities include DC/RF magnetron sputter coating unit, Langmuir–Blodgett (LB) trough and spin coating unit for the synthesis of single and multi-layered metallic/non-metallic, metal–organic and polymer films, respectively. Grazing Incidence X-ray Scattering (GIXS), Scanning Probe Microscopy (SPM) and Secondary Ion Mass Spectrometry (SIMS) techniques are used for the characterization and subsequent analysis of the grown films. Recently, we have developed a facility called Bombardment-induced Light Emission Spectroscopy (BLES), which is capable

of performing surface characterizations, in addition to fundamental studies on atomic excitations in sputtering processes under heavy ion bombardment.

## 2. Growth, morphology and structure of thin films

Characterization of low-dimensional structure becomes important to understand the underlying growth mechanism. This understanding helps us in fabricating new desired structures, which manifest novel physical properties for device applications. In this section, we briefly describe the growth techniques followed by characterization and results. Metal–organic multilayer films are grown using LB trough. The trough contains water on which one generally spreads monolayers of amphiphiles and then multilayer films are deposited on a substrate by repeated dipping<sup>1</sup>. The film deposited using this technique is called a LB film. By embedding polymers between the interfaces of amphiphilic layers, one can mimic biological systems. Polymer films of various thicknesses are grown using conventional spin coating by controlling the revolution speed of the spin coater disc in combination with the dilution of the polymer in solvent. Metallic single and multilayer films are grown using magnetron sputtering unit which contains two dc and one rf targets at present. One can sequentially deposit a thin multilayer stack by sputtering of the targets. Smooth interfaces can be formed if the substrate on which the film is grown is rotated across the sputtering target instead of keeping it stationary. This can also control the thickness much more accurately. Recently, we have developed a new technique of growing nanostructures having wire-like forms using Atomic Force Microscope. These nanostructures are grown by modification of an ultra thin film due to tip–surface interaction<sup>2</sup>.

Figure 1 shows a typical specular GIXS data of a LB (9 monolayer of cadmium arachidate) film<sup>1</sup> and a metallic (Pt/Cu)<sub>9</sub> multilayer system<sup>3</sup>. In this figure, bilayer Bragg peaks (marked by arrows) of both these multilayer systems are clearly observed. The gap between the peaks of two successive small oscillations shown between the marked arrows gives the total thickness of the stack. The structure of the film across the depth can be

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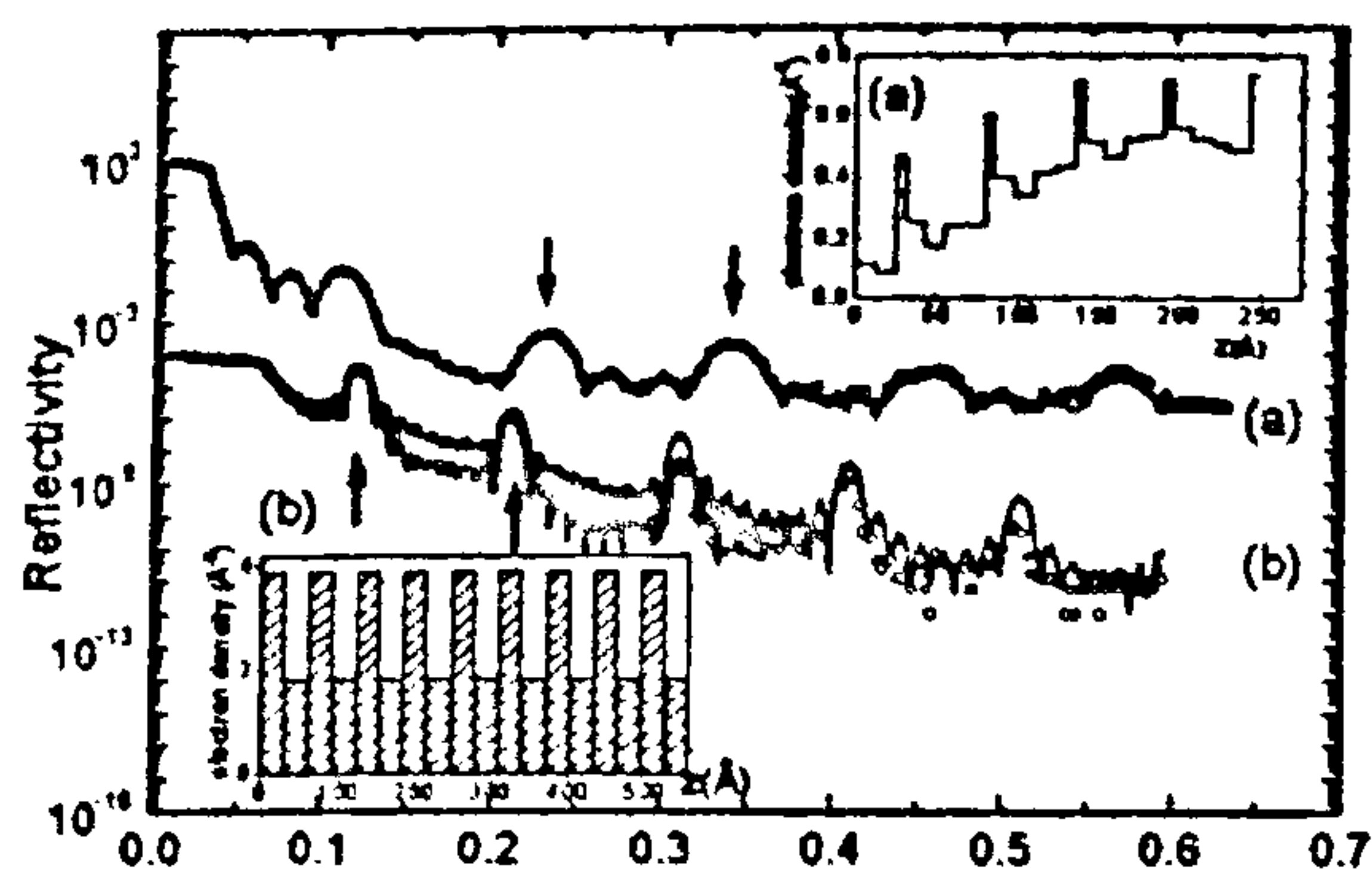


Figure 1. X-ray reflectivity for the (a) LB (9 monolayer of cadmium arachidate) film and (b)  $(\text{Cu/Pt})_9$  multilayer system; data for  $(\text{Cu/Pt})_9$  has been shifted down for clarity. Multilayer Bragg peaks are marked by the arrows. Solid lines are the fit for the LB film and for  $(\text{Cu/Pt})_9$  multilayer. The respective EDPs are shown in the insets.

determined from the electron density profile (EDP) obtained through analysis of the GIXS data<sup>1,4-7</sup>. Inset of Figure 1 shows such EDPs for the above two systems. From the transverse GIXS data shown in Figure 2 one can obtain the lateral in-plane correlation of the surface at each interface. In the LB film system one obtains the lateral correlation of self-affine nature. Other types of lateral correlation have been observed as well<sup>1,4</sup>. We have also proposed the growth mechanism of LB film from the analysis of both specular and transverse GIXS data<sup>1</sup>, that the growth occurs via 1D deposition followed by a 2D adsorption/desorption process. Recently, we have studied the melting of LB films using GIXS and SPM<sup>6</sup>. We have studied the growth of polystyrene films and have observed the in-depth layer formation, each layer having thickness typically of the order of the radius of gyration of the polymer chain. This layer formation was attributed to the confinement effect, i.e. the polymer film is confined by the substrate/polymer interface and the polymer/air interface<sup>7</sup>. While growing multilayers with two different materials, say A and B, deposited sequentially, it was interesting to observe that an alloy formation at the interface is possible when A is deposited on B but no such formation occurred if B is deposited on A. For example, in case of a Si/Ge multilayer a Si-Ge alloy was found to form at the interfaces where Ge was deposited on Si, but not when Si was deposited on Ge<sup>8</sup>.

Recently we have also observed that nanofabrication of metallic systems can be induced by tip-surface interaction and the induced structures have a wire-like form<sup>2</sup>. Figure 3 shows one such typical induced structure on a thin silver film. We have observed that beyond a film thickness of 50 Å such fabrication becomes difficult and the film shows elliptical-shaped grains<sup>9</sup>.

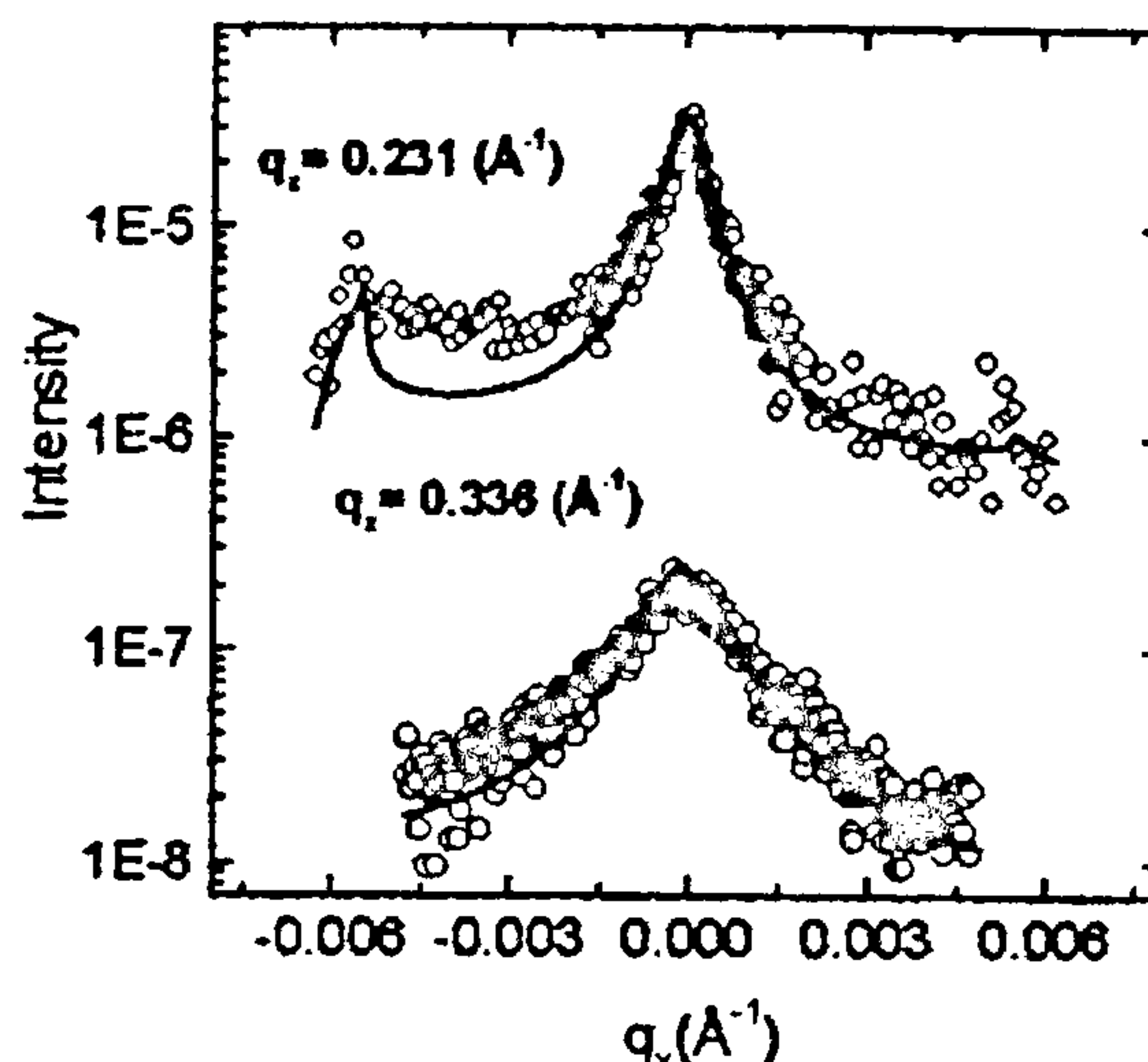


Figure 2. Transverse X-ray reflectivity data of LB film for 2nd and 3rd Bragg peaks. The intensity profiles show Lorentzian nature indicating self-affine interfaces.

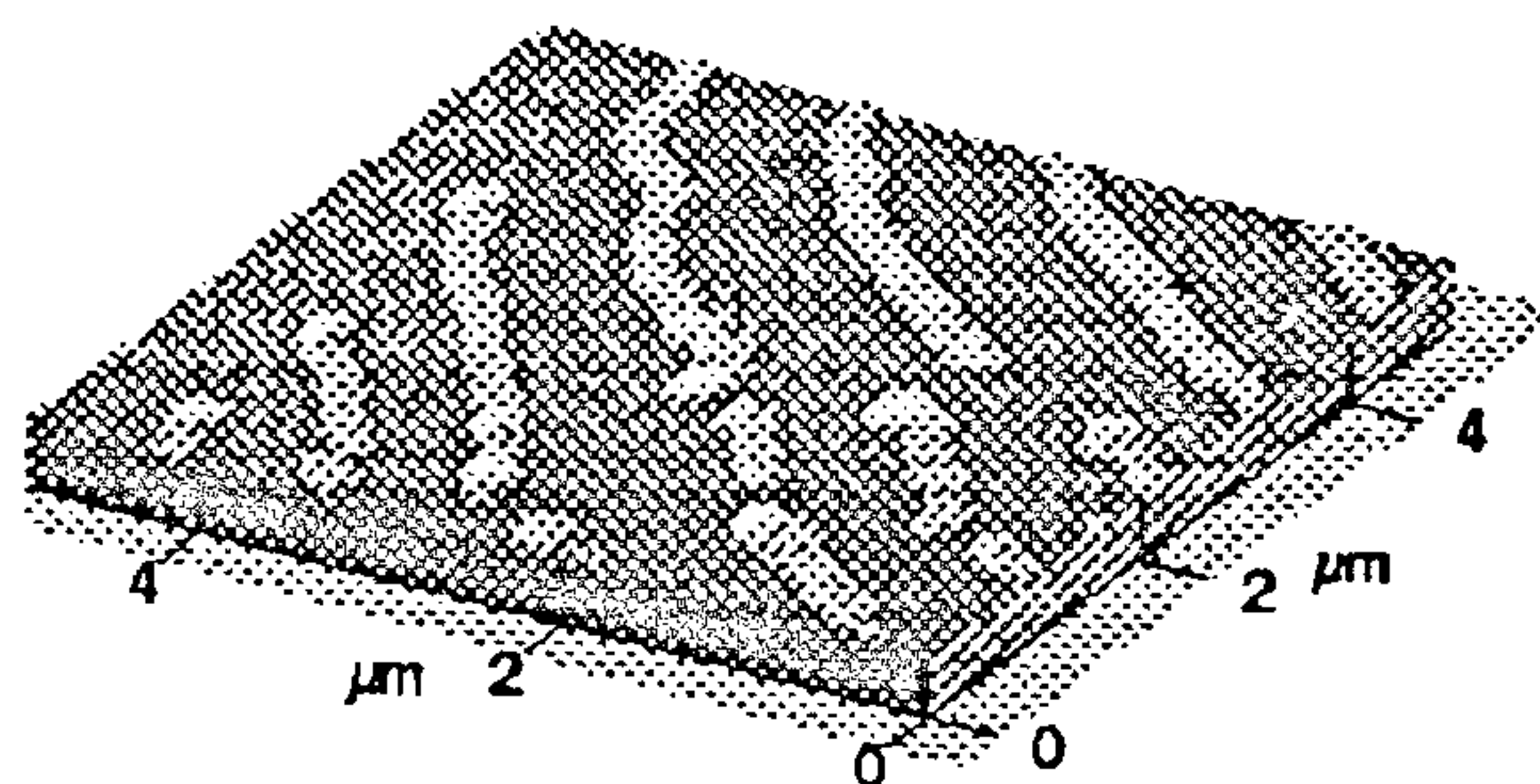


Figure 3. AFM image showing tip-induced wire-like structures of thin silver film grown on Si(001) substrate.

Interfacial diffusion is a commonly occurring phenomenon in thin films. The process being totally a system-dependent one, no universal theory exists that can predict the in-depth compositional variation in films of different materials. SIMS as an extremely powerful analytical probe in detecting an elemental concentration (down to ~ ppb) in the host matrix is being applied for the stoichiometric analysis of the grown films of interest<sup>10</sup>. We have studied the interdiffusion phenomenon for an MOCVD-grown 16-bilayered AlAs/AlGaAs structure<sup>11</sup> and have obtained an asymmetry in Al diffusion through the interfaces between AlGaAs grown on AlAs and AlAs grown on AlGaAs. Such an asymmetry was corroborated by the pronounced difference in interfacial roughness occurring between these two different materials obtained through GIXS measurements. We have recently made SIMS and high angle X-ray diffraction studies<sup>12</sup> of a double quantum well (InP/InGaAs/InP/InGaAs/InP) structure (Figure 4) and



have confirmed the diffusion of P into the quantum wells. These two measurements along with a simulation program yielded the stoichiometry of the composition at any depth throughout the stack.

We have recently installed a SIMS machine (Hiden Analytical Limited, UK) in our laboratory for both static and dynamic SIMS applications. It employs a 45° sector-field electrostatic energy analyser and a high-sensitivity quadrupole mass spectrometer capable of handling masses up to 100 amu. The multiport UHV chamber incorporates an additional Cs ion gun needed for negative SIMS analysis. The secondary ions passing through the instrument have simple curved trajectories with virtually 100% transmission within the pass band. Recently we have performed a dynamic SIMS measurement on a 400 Å-thick spin-coated polystyrene film grown on Si substrate. The sample was bombarded with 5 keV Cs<sup>+</sup> ions rastered over a broad area on the surface and the secondary negative species were detected from

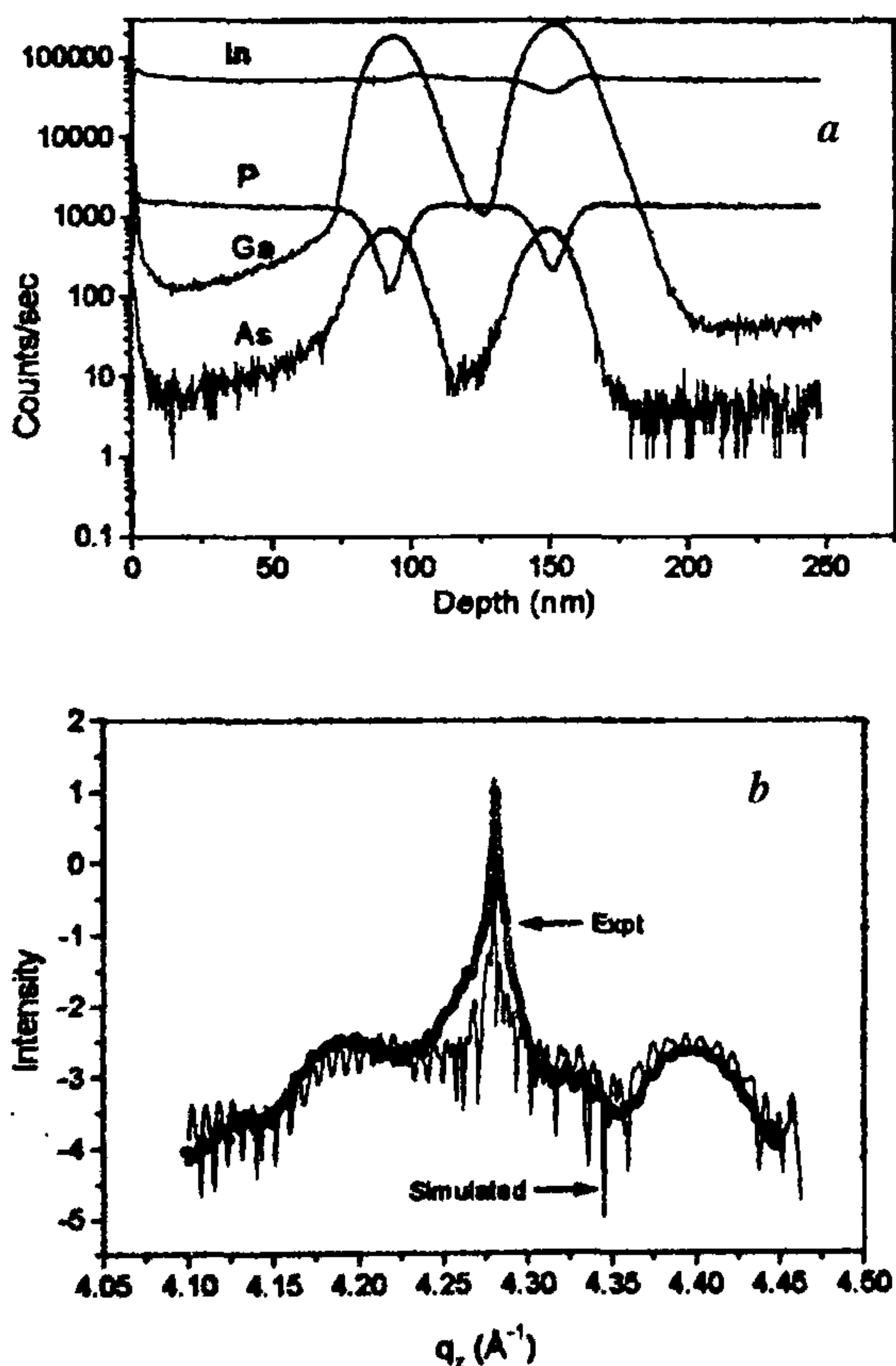


Figure 4. (a) SIMS depth profile and (b) high angle X-ray diffraction curve along with the simulated profile of the double quantum well (InP/InGaAs/InP/InGaAs/InP).

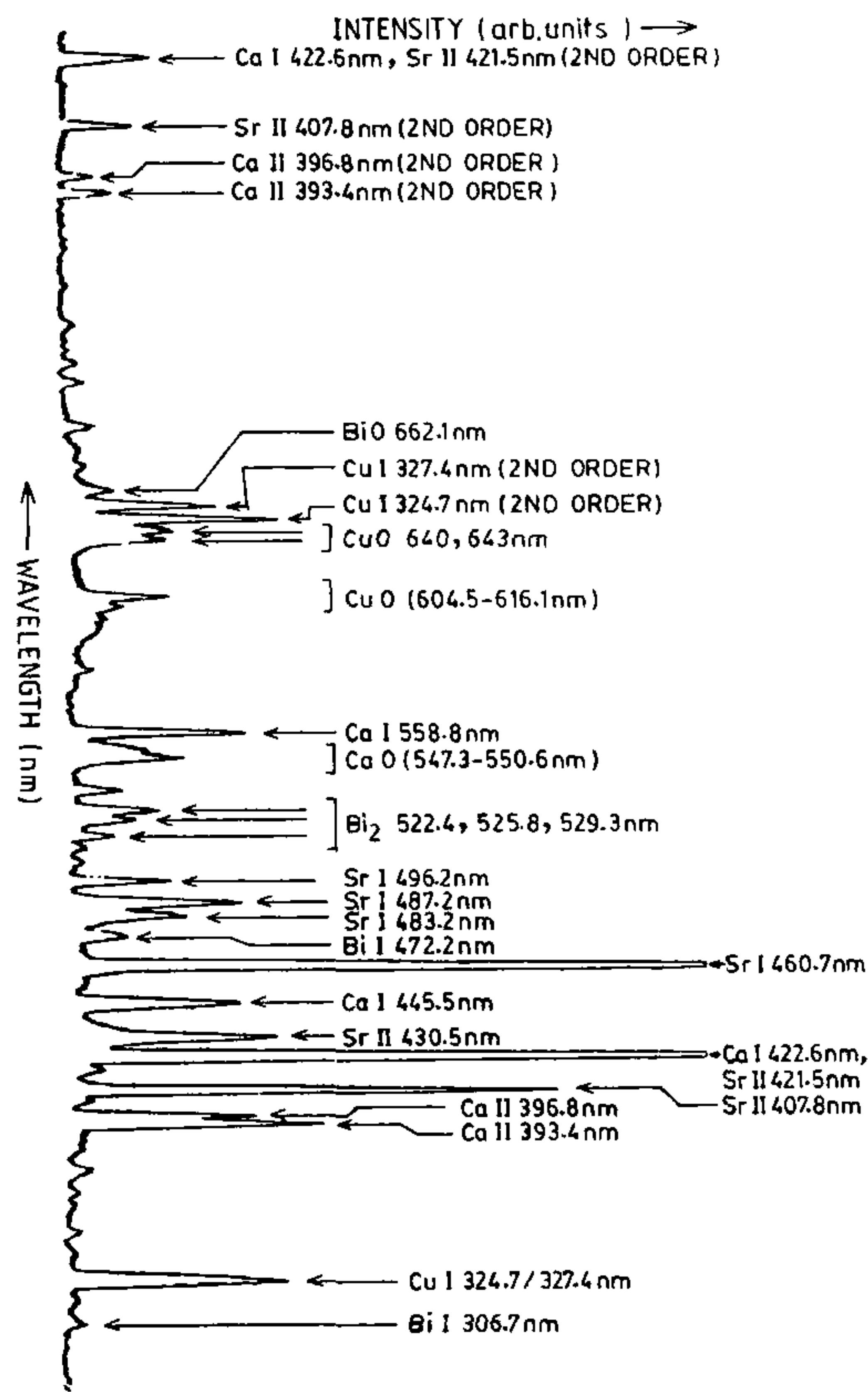


Figure 5. Typical optical spectrum emitted during 6 keV Ar<sup>+</sup> ion bombardment on Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8</sub>.

the central acquisition area much smaller than the rastered area. It was found that the polystyrene/Si interface was quite prominent, where all the individual secondary species characteristic of the polystyrene had a drastic fall, beyond which Si intensity increased sharply followed by its saturation.

### 3. Spectrochemical analysis using ion beams

Ion beam sputtering of solids gives rise to the ejection of target atoms, molecules and ions in excited electronic states, which subsequently decay by photon emission<sup>13</sup>. Figure 5 shows a typical optical spectrum emitted during 6 keV Ar<sup>+</sup> bombardment of Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8</sub> recorded between 200 and 800 nm wavelengths. The emitted spectral lines are the characteristics of the elements present in the sample. One of the postulates in light emission processes is that the excited atom yield is correlated with the local band structure of the surface from which sputtered atoms were initially emitted. We, however, have observed that this may not be always valid at least in the case of sputtered Al atoms from

$\mu$ -size  $\text{Al}_2\text{O}_3$  grains embedded in Si matrix<sup>14</sup>. A certain fraction ( $\approx 10\%$ ) of the excited Al atoms is found to interact strongly with the Si surface in the immediate vicinity of each  $\text{Al}_2\text{O}_3$  particle. A general consensus is that the photon emission by bombarding a pure sample of metal with an oxygen beam or bombarding in the presence of oxygen is equivalent to that from an inert gas ion bombarded metal oxide surface. It means that there will be no transient regime in the time dependence of spectral lines due to sudden interruption of the incident ion beam in either case. However, we have found that beam-off transients exist due to  $\text{O}_2^+$  ion bombardment of a Cu (110) single crystal target, the results of which are similar to that due to  $\text{Ar}^+$  bombardment except an enhancement of photon emission by a factor of 15 in the case of  $\text{O}_2^+$  bombardment<sup>15</sup>. Finally, we have shown that bombardment-induced light emission phenomenon may also be utilized to study preferential oxidation of elements in a compound target, eg.  $\text{Fe}_{52}\text{Ni}_{48}$ , if any<sup>16</sup>.

#### 4. Conclusion

For studying the growth process of low dimensional systems, GIXR and SPM which are complementary to each other have been found to be very useful. In X-ray reflectivity one obtains a statistically averaged information over a large area whereas SPM gives a very local information. The detailed structure of the film and interface can be obtained using these two techniques. Recently it has been observed that SPM is not only useful for imaging the surface but is also useful in modifying the surface topography. SIMS on the other hand, is very useful in obtaining the chemical profile as a function of depth and gives an additional information in the analysis of the multilayer structure as a function of depth. Bombardment-induced light emission spectroscopy has also

the capability of surface chemical analysis, but only qualitatively mainly because of the lack of proper theoretical formulation of the excited atom yield.

1. Basu, J. K., Hazra, S. and Sanyal, M. K., *Phys. Rev. Lett.*, 1999, **82**, 4675.
2. Pal, S., Mukherjee, S., Banerjee, S., Kundu, S. and Sanyal, M. K., (to be published).
3. Kundu, S., Pal, S., Banerjee, S. and Sanyal, M. K., (to be published).
4. Basu, J. K. and Sanyal, M. K., *Phys. Rev. Lett.*, 1997, **79**, 4617.
5. Sanyal, M. K., Hazra, S., Basu, J. K. and Datta, A., *Phys. Rev. B*, 1998, **58**, R4258.
6. Basu, J. K., Sanyal, M. K., Banerjee, S. and Mukherjee, M., *Physica B*, 2000, **283**, 6.
7. Sanyal, M. K., Basu, J. K., Datta, A. and Banerjee, S., *Europhys. Lett.*, 1996, **36**, 265.
8. Banerjee, S., Sanyal, M. K., Datta, A., Kanakaraju, S. and Mohan, S., *Phys. Rev. B*, 1996, **54**, 16377.
9. Kundu, S., Hazra, S., Banerjee, S., Sanyal, M. K., Mandal, S. K., Chaudhuri, S. and Pal, A. K., *J. Phys. D, Appl. Phys.*, 1998, **31**, L73.
10. Chakraborty, P., *Pramana*, 1998, **50**, 617.
11. Sanyal, M. K., Datta, A., Srivastava, A. K., Arora, B. M., Banerjee, S., Chakraborty, P., Caccavale, F., Sakata, O. and Hashizume, H., *Appl. Surf. Sci.*, 1998, **133**, 98.
12. Sarkar, S., Chakraborty, P., Sanyal, M. K., Caccavale, F. and Arora, B. M., (to be published).
13. Ghose, D. and Hippler, R., in *Accelerator-based Atomic Physics Techniques and Applications* (eds Shafroth, S. M. and Austin, J. C.), American Institute of Physics, Woodbury, New York, 1997, pp. 649–676.
14. Ghose, D., Agarwal, P. and Bhattacharyya, S. R., to be published.
15. Ghose, D. and Bhattacharyya, S. R., *Phys. Lett. A*, 1997, **227**, 133.
16. Agarwal, P., Bhattacharyya, S. R. and Ghose, D., *Jpn. J. Appl. Phys.*, 2000, **39**, in press.

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