Surface and interface studies at IUC-DAEF, Indore

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The article lists the facilities available at IUC-DAEF, Indore Centre for study of surfaces and interfaces and gives examples of results obtained on these facilities.

THE Inter University Consortium for DAE Facilities (IUC-DAEF) is setting up a photoelectron spectroscopy beamline for studies of surfaces in the incident photon energy range from 10 to 200 eV. In addition, it has established an ESCA machine with depth profiling, a commercial AFM/STM operating in air, an X-ray reflectometer, an X-ray diffractometer and a conversion electron Mössbauer spectrometer (CEMS) for surface and interface studies. An ultra high vacuum (4×10^{-10} torr) system for growing multilayers on cooled or heated substrates with a control of a fraction of an Angstrom has been designed and built recently[1] and many multilayers have been grown. A locally built ion beam sputter deposition system has also been commissioned. Some examples of experiments on surfaces, interfaces and thin films, undertaken on these facilities by university and IUC scientists will be described here.

Films

Ag on glass (Mangalore University and IUC)

Silver of ~100 Å on glass substrate produces island films. DC resistance measurements show that the resistance of these films in air increases with aging. In order to ensure that this increase is connected with the growth dynamics and not with oxidation of the films, *in situ* DC resistance measurements were made in the vacuum chamber with oxygen partial pressure < 10^{-11} torr. Figure 1 shows the variation of resistance with time. A percolation transition was reproducibly observed under certain conditions for the first time[2] and the resistance dropped by about 40 times from ~ 2 MΩ to ~ 50 KΩ. Increased fluctuation is observed just before transition. These measurements have provided a clear evidence of mobility of islands. AC conductance of films which have undergone this transition is very different from films of comparable resistance directly grown on the same substrate, indicating the difference in the nature of connectivity of the islands in the two cases[3].

Ag and Pt on Si (Pune University and IUC)

Silver (and platinum) films with thickness varying from 100 to 1000 Å were also grown on Si(111) (and Si(100)) substrates with a view to studying the surface morphology on different length scales using specular and diffuse X-ray reflectivity and AFM at IUC and a home-built STM at Pune University. Figure 2 shows X-ray specular reflectivity curves for platinum films along with fits: the insert shows the increase in the derived roughness by a factor of three from 6 Å to ~ 20 Å as the thickness increases from 100 to 1200 Å (substrate roughness ~ 6 Å). The trend is the same for silver films. The observations from AFM, STM and the reflectometer were quantitatively analysed in terms of height–height correlations in the light of dynamical scaling approach[4]. The scaling exponents α and β (ref. 5) for roughness and growth, respectively, as extracted from the three types of measurements are generally consistent with each other.

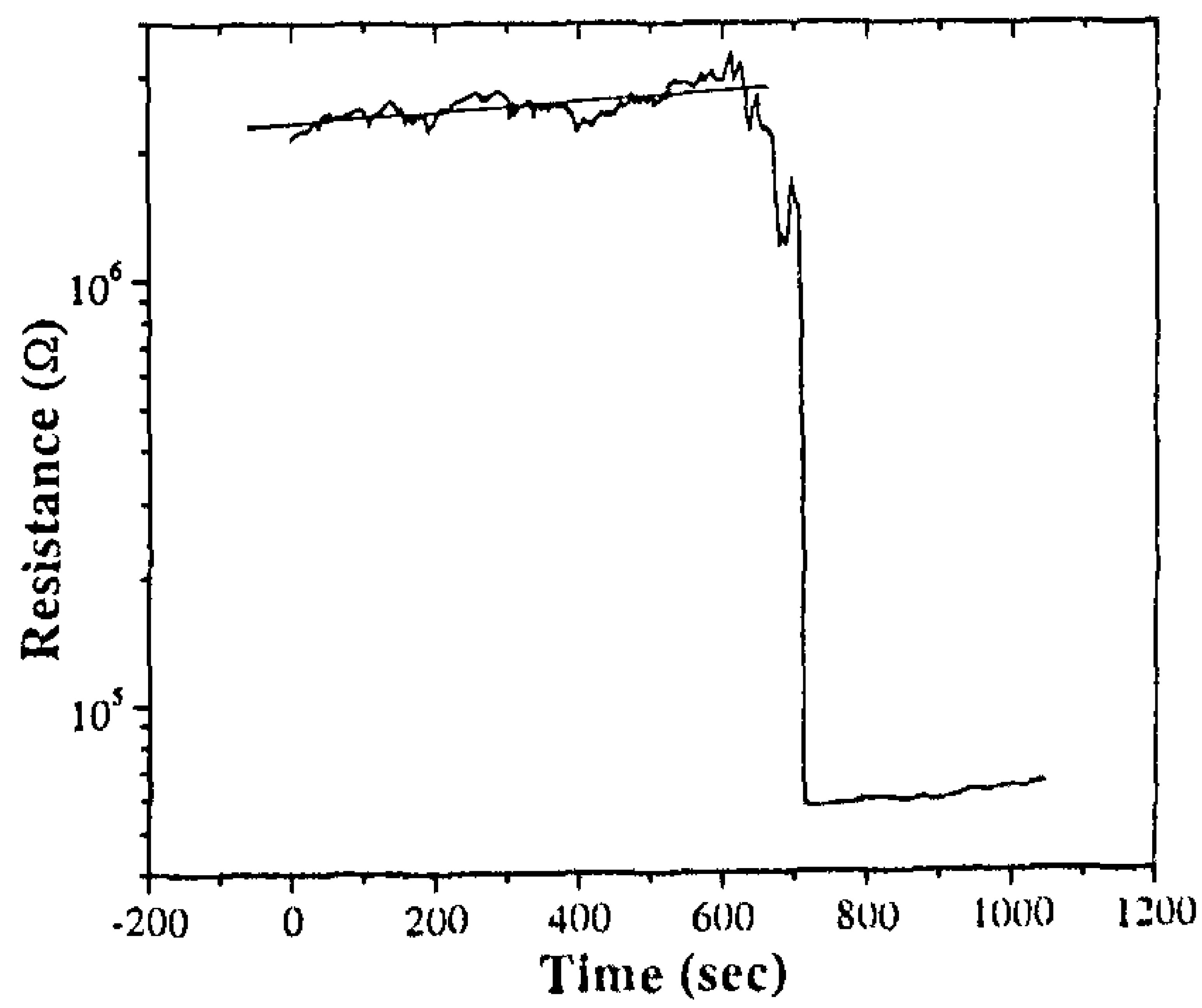

Figure 1. Resistance vs time of ~100 Å Ag film on glass.

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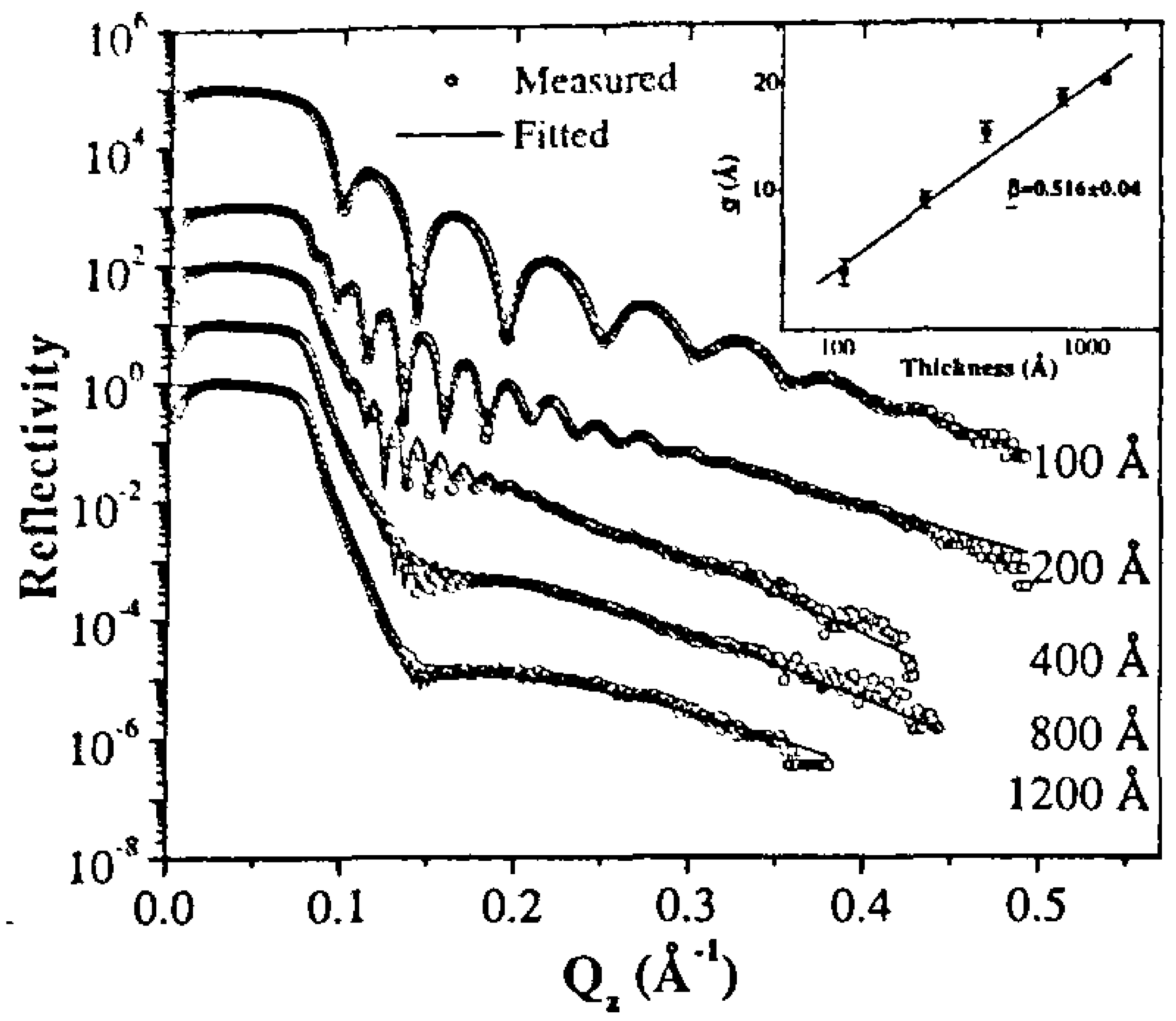

Figure 2. Specular reflection of X-rays from Pt films on Si(100) (shifted for clarity).

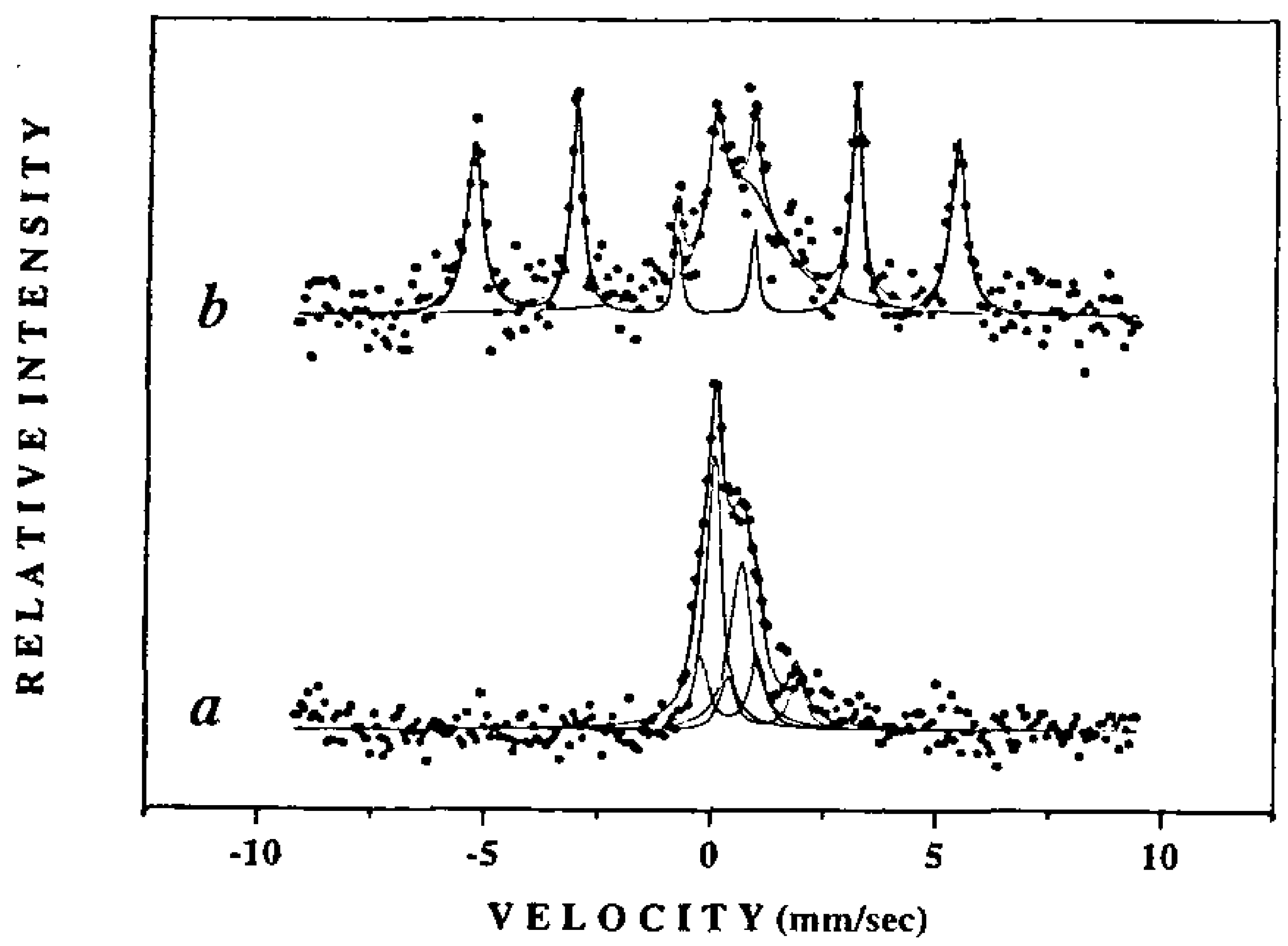

Figure 3. CEMS of iron nitride film in as-deposited (a) state showing state of Fe on top ~ 30 Å. Upper figure shows the effect of annealing (b) at 400°C giving α-Fe precipitates.

FeN on glass (IUC)

An ion beam sputtering set-up developed in-house has been used to deposit films of amorphous iron nitride, with a view to exploring the possibility of using it as a material for preparing $^{56}Fe/^{57}Fe$ multilayer nuclear Bragg monochromator[6]. Our grazing incidence X-ray diffraction confirmed the amorphous nature of the 1300 Å thick nitride film deposited on float glass substrate. Mössbauer measurement (Figure 3) shows that the film is nonmagnetic at room temperature – a desired characteristic for nuclear Bragg monochromator. X-ray photoelectron spectroscopy (XPS) has been used to get the surface stoichiometry of the film as $FeN_{0.7}$. This agrees with the composition estimated by comparing the CEMS spectrum with those of various nitrides of iron given in the literature[7]. The surface roughness of the film was also estimated from AFM and X-ray reflectivity measurements to be 6 Å and 8 Å, respectively when compared with the roughness of 6–7 Å for the substrate. Thus ion beam sputtered amorphous Fe nitride seems to be a good candidate for making nuclear Bragg monochromators.

Multilayers

Multilayers are of interest from several points of view, both basic and applied. Supermirrors for neutrons and X-rays, giant magnetoresistive multilayers, magnetic storage devices, superconducting/ferromagnetic multilayers and Langmuir–Blodgett (LB) multilayers are some examples which have been examined.

Supermirrors for neutrons and X-rays (IUC)

Reflectivity of $(34\ Ti/24Ni)_{10}$ multilayer on float glass was measured using CuKα X-rays[8]. Bragg reflections were discernible up to the fourth order indicating reasonably good quality of interface. Both Ni and Ti are amorphous (from XRD). In this case, though the roughness of Ti-on-Ni interface (~7.5 Å) and that of Ni-on-Ti (11 Å) are not equal, a single set of roughness parameter describes the data well. As against this $(22.5Fe/90.5Ni)_{10}$ multilayer whose reflectivity is shown in Figure 4 has crystalline components and the interfacial roughness increases cumulatively with a cumulative roughness parameter $\sigma_s = 4.1$ Å. It is again interesting to note that while, as with single films, the FeNi multilayer with crystalline layers requires cumulative roughness for its description the FeTi multilayer with amorphous layers does not.

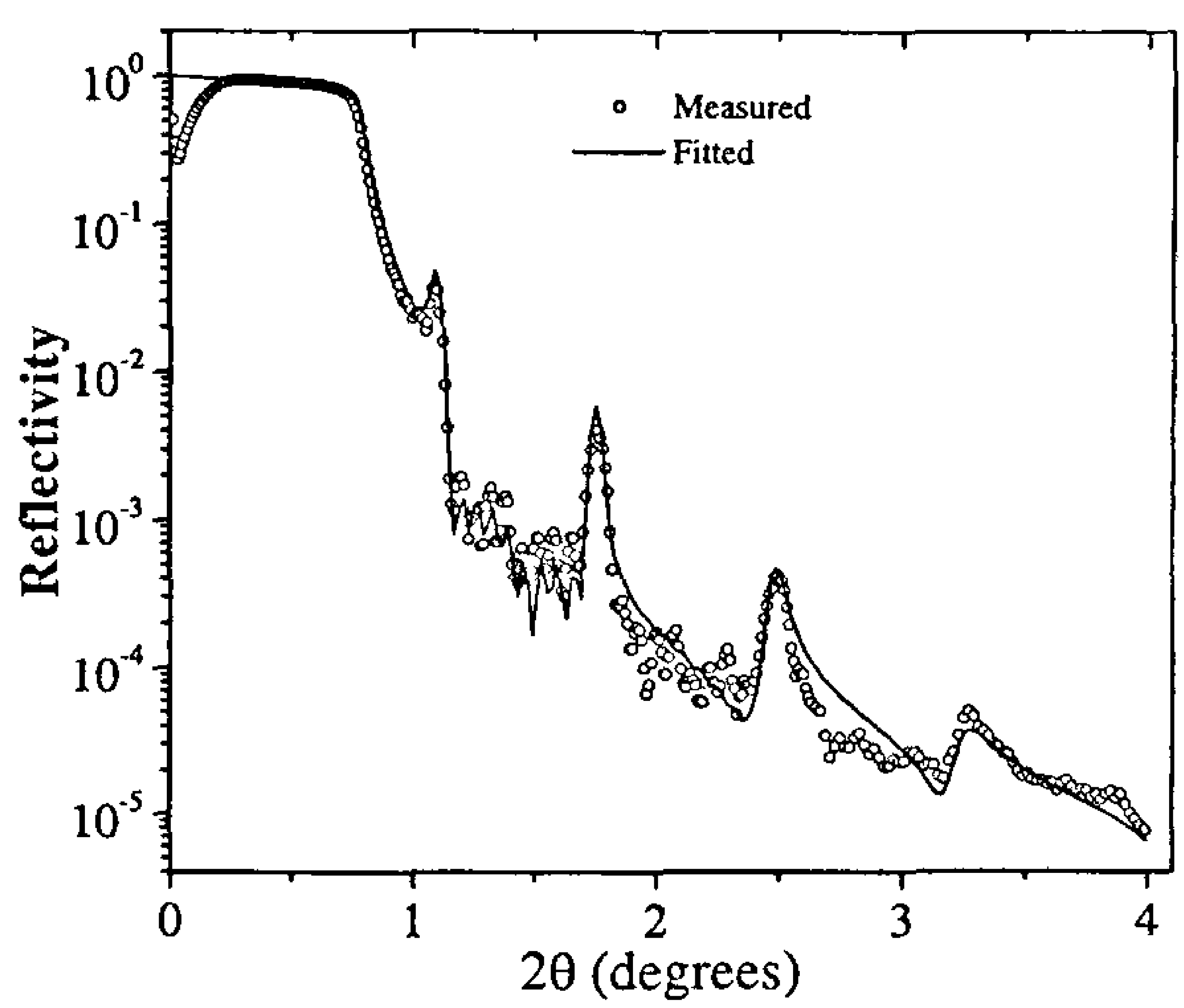

Figure 4. Reflection of X-rays from FeNi multilayers, showing Bragg reflections.

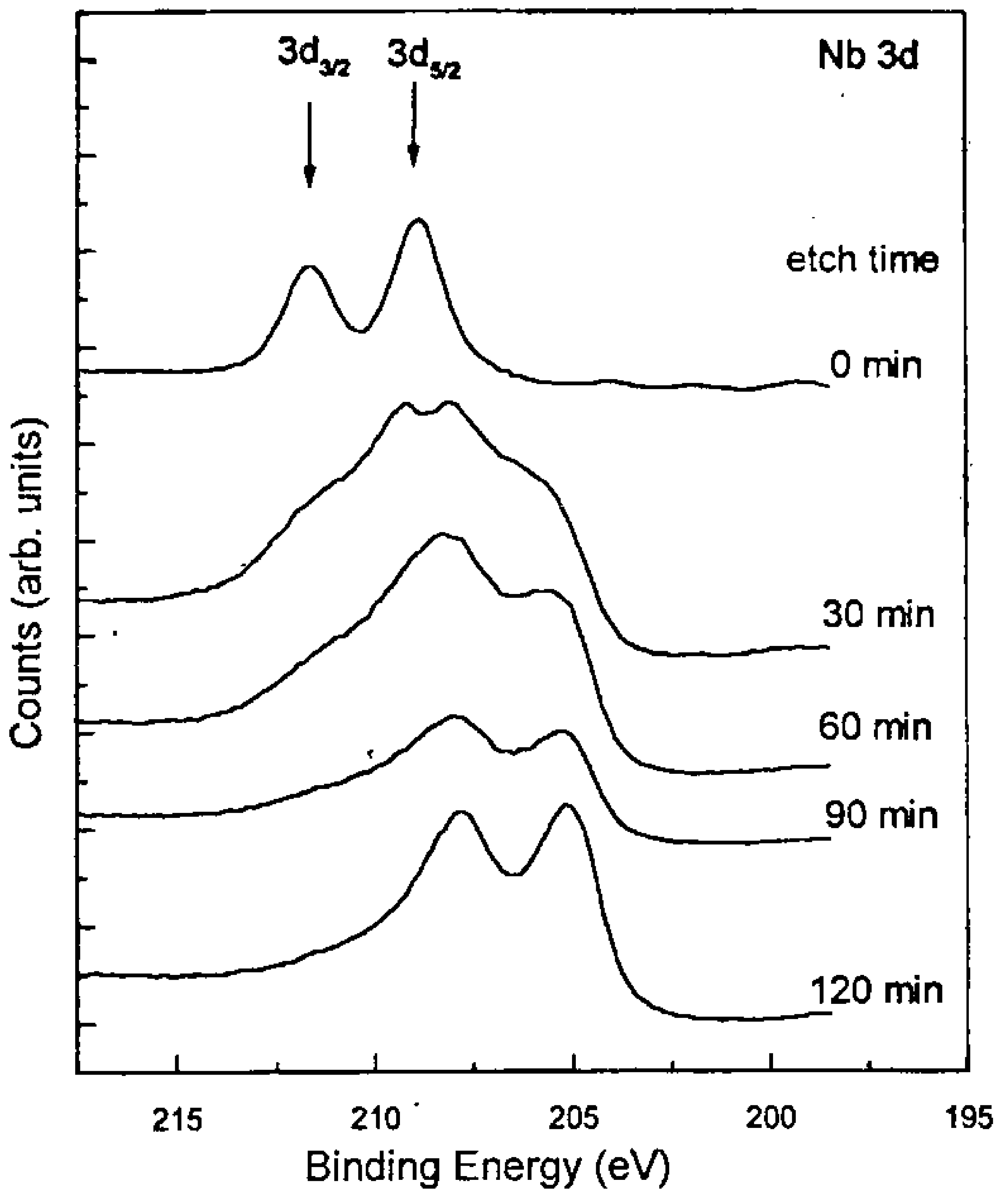

Figure 5. ESCA as a function sputtering time (i.e. depth) for Nb/C multilayers.

ESCA with depth profiling is another technique which can be effectively used for studying interface. Some preliminary results of this type have been obtained on Nb/C multilayer with top Nb layer. Figure 5 shows the photoelectron spectra as a function of etching time. At $t = 0$ one observes only the NbO peaks. With ion etching, pure Nb peaks along with NbC peaks become visible. The bottom spectrum essentially shows pure Nb with NbC.

Magnetic multilayers Fe/Cr and Fe/Tb (IUC and Indore University)

Magnetic multilayers form another class of interesting systems which have found application in magnetic storage devices. Two systems in this class which have been studied in some detail at IUC are Fe/Cr which exhibits a giant magnetoresistance (GMR)[9] and Fe/Tb which exhibits a perpendicular magnetic anisotropy (PMA)[10]. In both the types of multilayers, the magnetic properties are known to depend sensitively on the state of the interfaces[11]. A set of Fe/Cr multilayers simultaneously deposited on float glass substrates having different surface roughness and characterized using X-ray reflectivity and Mössbauer spectroscopy measurements, shows that the interface roughness varies from specimen to specimen without appreciable change in the thickness of the interdiffused layer at the interface. XRD and AFM measurements show that the morphological features like grain size, grain texture, etc. are also similar in all the specimens. Magnetoresistance measurements on these specimens have been able to separate the effects of interface roughness from those of interdiffusion and morphological changes in the films. The results show that increasing interface roughness causes a decrease in the magnetoresistance (Figure 6). On the contrary, Fe/Tb multilayers produced in a similar manner and which exhibit a large PMA, as determined from Mössbauer measurements, do not show any significant dependence on interface roughness.

Superconductor/ferromagnetic multilayer Nb/Fe (IUC and CAT)

Another interesting multilayer is the superconductor/ferromagnetic combination of Nb/Fe. Two types of Nb/Fe multilayer structures, each having fixed Nb layer thickness of 400 Å and Fe thickness of 6 Å and 24 Å, respectively, were prepared on Si(110) substrates. Till now this is the thinnest layer (6 Å) that we have prepared. The magnetic state of Fe was investigated by a SQUID magnetometer at the Centre for Advanced Technology (CAT). Figure 7 shows the temperature depend-

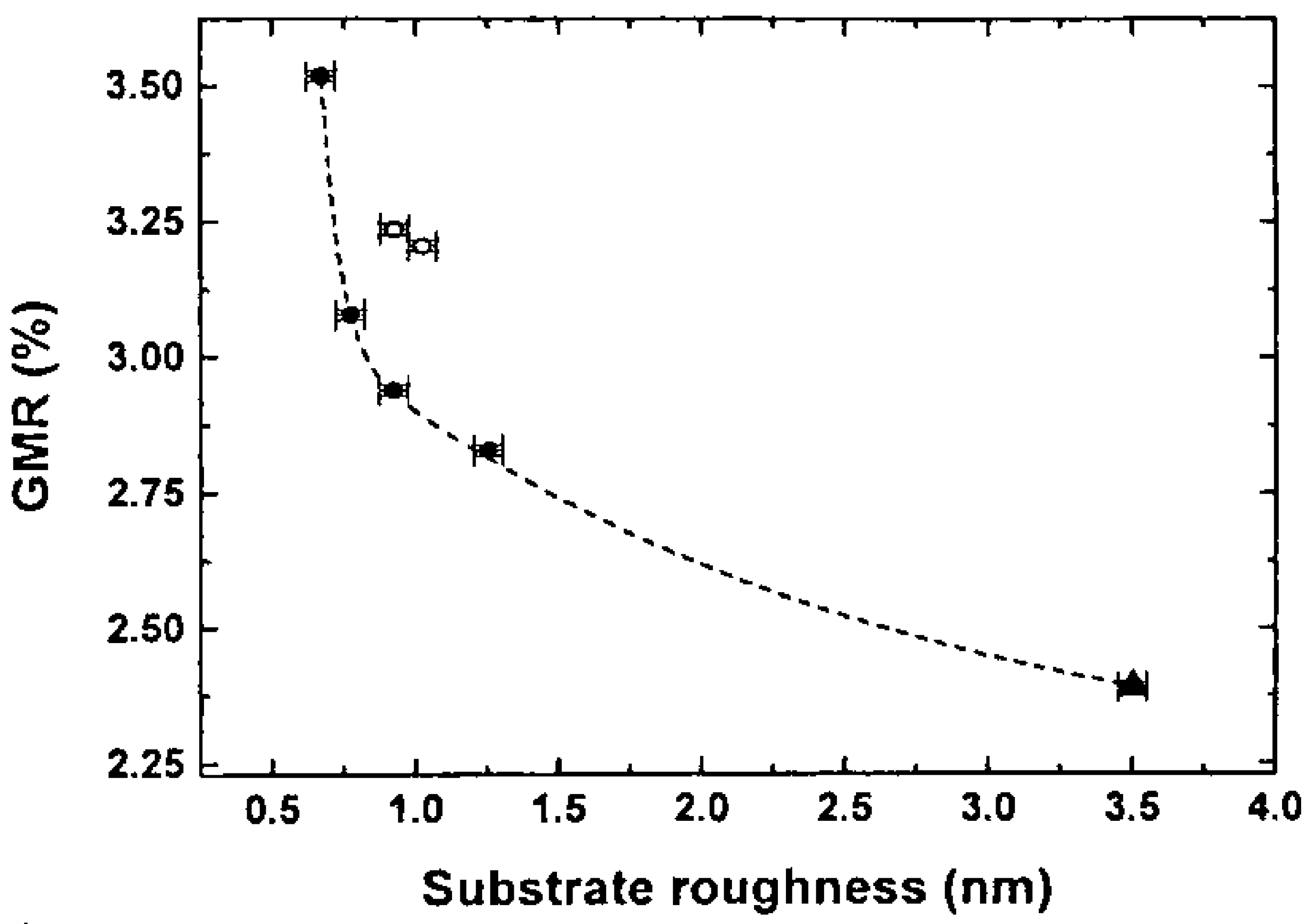

Figure 6. Variation of giant magnetoresistance in $[30Fe/12Cr]_{20}$ multilayers as a function of the roughness of the float glass substrate.

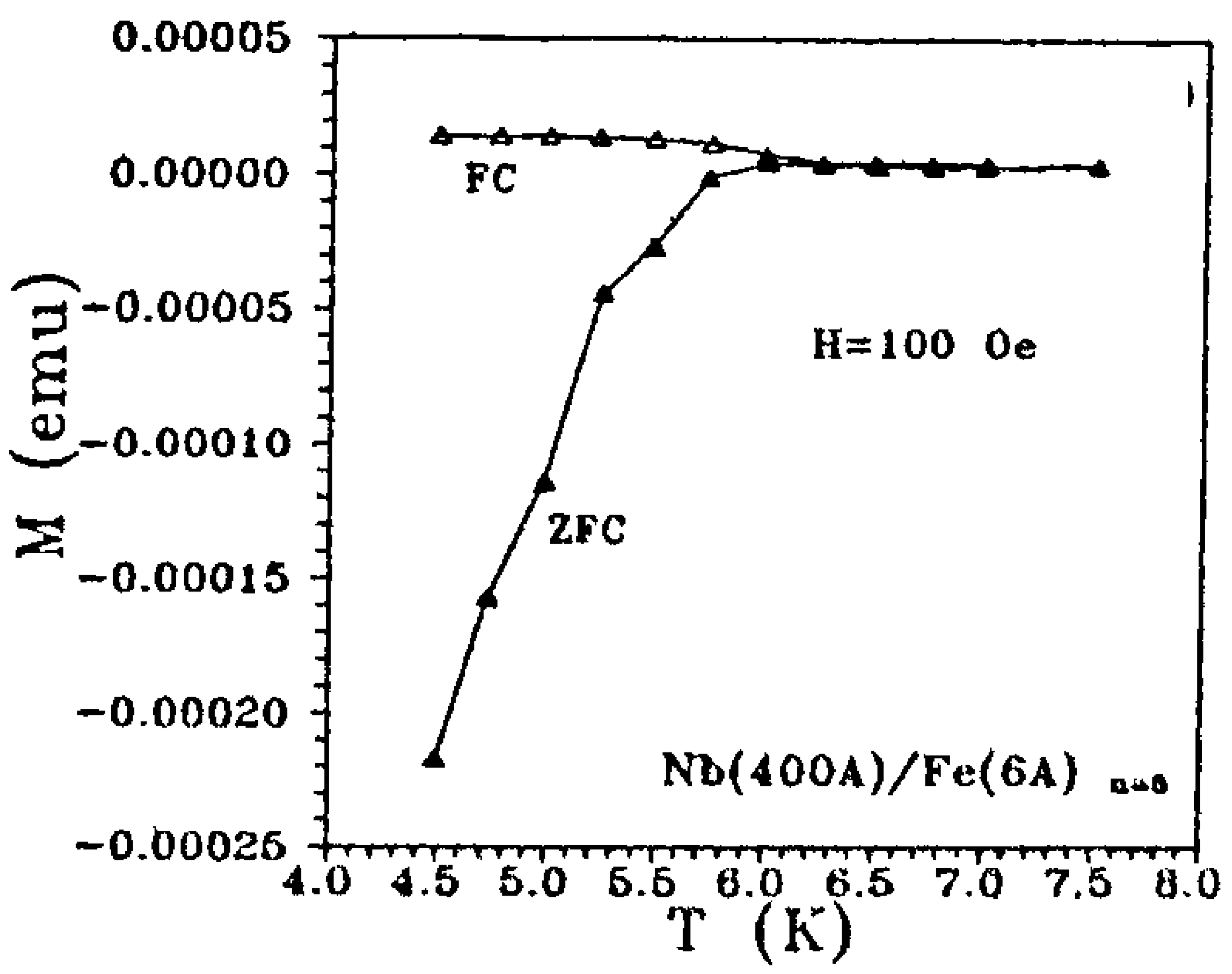

Figure 7. Magnetization of $(400\ Nb6Fe)_5$ multilayer with a Nb top layer.

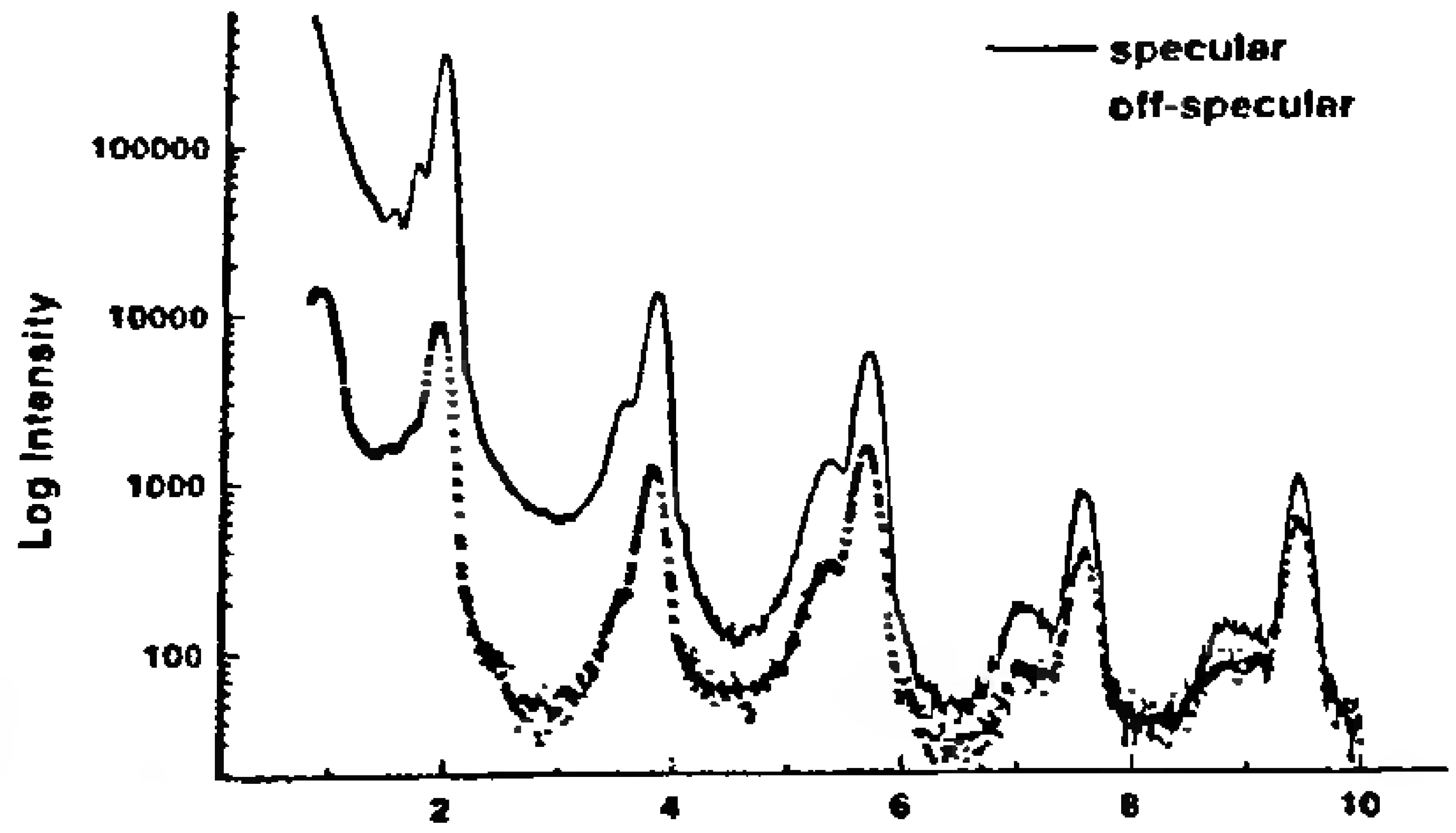

Figure 8. X-ray reflectivity of representative zinc arachidate LB film.

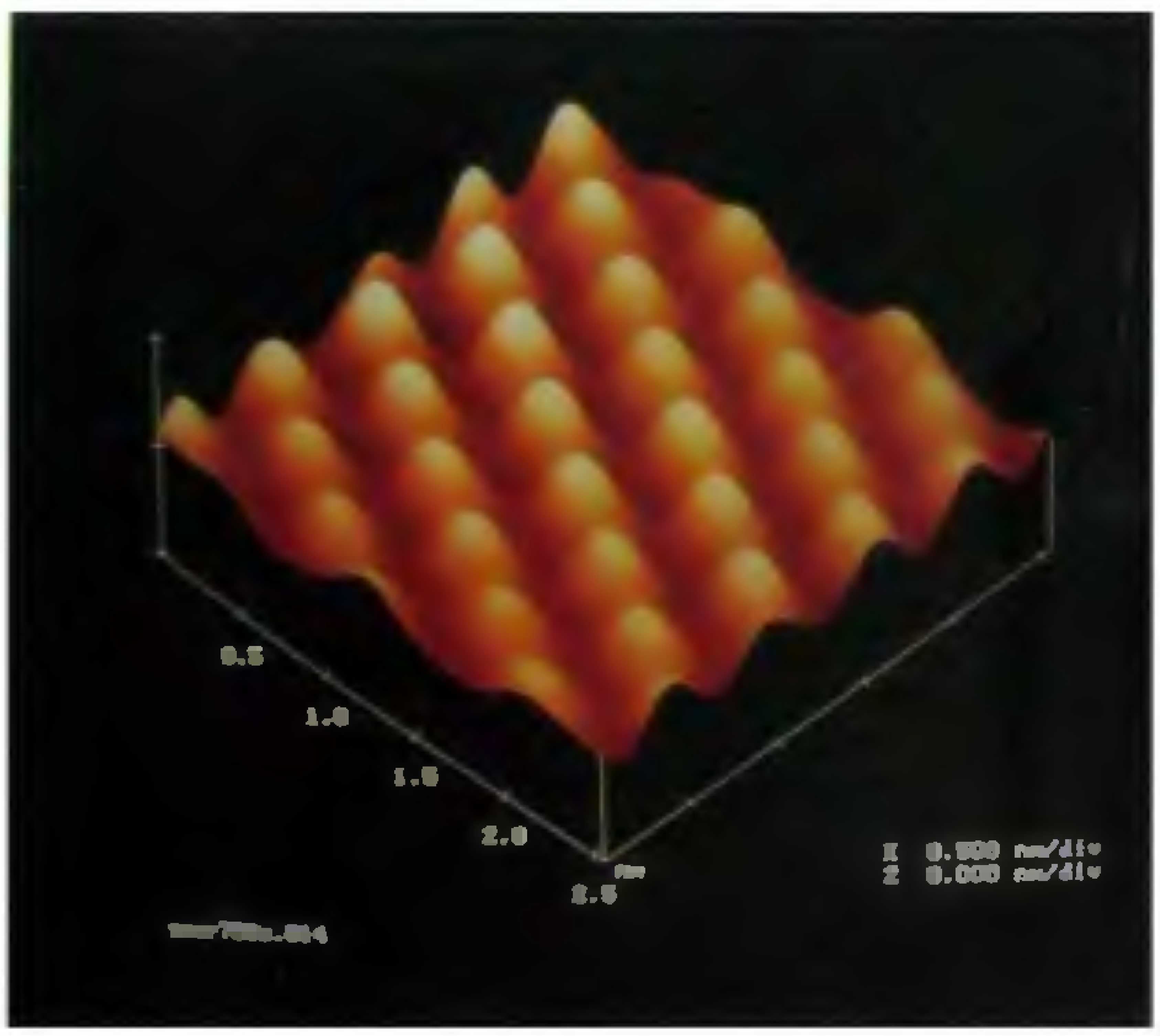

Figure 9. AFM images of ZnA at Angstrom resolution.

ence of observed magnetization on the 6 Å Fe film both in zero field cooled and field (100 Oe) cooled conditions[12]. Compared to a superconducting T_c of ~ 9.3 K for pure Nb, the multilayer with 6 Å Fe shows T_c of 6.25 K. However in the FC condition the magnetic moment in the superconducting state is positive and resembles the typical paramagnetic Meissner effect-like behaviour observed in some hard type-II superconductors including HTSCs. In contrast, the multilayer with 24 Å Fe does not show superconductivity down to 4.5 K.

LB films: Cd and Zn arachidates (IIT, Mumbai and IUC)

Work on LB multilayers of cadmium arachidate (CdA) and zinc arachidate (ZnA) using X-ray reflectivity and AFM has been carried out[13]. Figure 8 gives the X-ray specular as well as off-specular X-ray reflectivity patterns of LB multilayers of 25 monolayers of ZnA deposited on quartz substrate: the films get deposited with two distinct periodicities of 55 Å and 47 Å, which correspond to two different tilt angles. Relative fractions of the two types of periodic structures vary with the pH of the solution. The off-specular X-ray reflectivity curves in all the specimens closely follow the specular ones, suggesting that roughness of the interfaces in a multilayer is highly conformal. There is also an important qualitative difference in the nature of rocking curves for the two types of multilayers. AFM measurements with atomic resolution give information about the in-plane packing of the molecules (Figure 9). It is found that in-plane structure in ZnA multilayer differ significantly from that in CdA: while in the case of CdA, the in-plane structure is centred rectangle (herring bone) and in the case of ZnA, it is hexagonal.

Summary

GIXRD, reflectivity, AFM, ESCA with depth profiling and CEMS facilities at IUC, Indore have been used to study surface morphology, composition, structure and magnetic nature of films and multilayers grown inhouse using e-beam evaporation and ion beam sputtering facilities. A variety of applications are given as examples. These facilities are available for scientists from any research institution with interesting problems.

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