

11-Surfaces, Interfaces and Thin Films

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PS-11.01.35 THE APPLICATION OF STM AND AFM IN MINERALOGICAL STUDIES IN CHINA By Libing Liao*, Zhesheng Ma and Nicheng Shi, X-Ray Lab., China University of geosciences, Beijing 100083, China.

STM and AFM have rapidly and widely been used in various study fields since they were invented by Binnig et al. in 1982 and 1985. Up to now, all the study results by STM and AFM have proved that they are really powerful devices for material surface structure studies. Their applications in mineralogical studies were started by Zheng et al. in 1988. In China, as far as we know, our Lab. is the first group and also the only group that studied mineral surfaces with STM and AFM. We began to put STM and AFM in mineralogical studies in 1988 and 1991 respectively. Till now, seven minerals in all have been studied (five for STM and two for AFM). They are galena, molybdenite, hematite, stannite, pyrite, calcite and coal. For galena, both S and Pb atoms of its {100} surface were observed in air under positive Vbias. For stannite, we are the first to image its surface by STM and atomic resolution images of its surface were obtained. For pyrite, some new phenomena were observed which can not be explained satisfactorily by pyrite known band data. Therefore, it wait for further studies. Calcite and coal surfaces were studied by AFM. Some interesting results have already been obtained.

11.02 - Thin Film Structures

MS-11.02.01 THE STUDY OF CHEMICAL INTERFACES AND SURFACES USING NEUTRON REFLECTIVITY. By J. Penfold, Isis Science Division, Rutherford Appleton Laboratory, U.K.

The specular reflection of neutrons is now well established as a technique for the study of surfaces and interfaces. In combination with isotopic labelling it has been shown to be a powerful technique for the study of problems in Surface Chemistry. Examples of its application to the determination of adsorbed amounts and structure of surfactants, polymers, proteins and their mixtures at the air-liquid and liquid-solid interfaces will be described. Particular emphasis will be placed on the study of surfactants and mixed surfactants adsorbed at the air-liquid interface.

MS-11.02.02 SPECULAR AND DIFFUSE SCATTERING STUDIES OF INTERFACES By M.K. Sanyal, Solid State Physics Division, Bhabha Atomic Research Centre, Bombay 400 085, India.

The availability of intense synchrotron X-ray sources has resulted in a rapid growth of new techniques to investigate interfacial structures of thin films. In this talk we shall discuss the use of specular and diffuse scattering studies to understand the interfacial structures in single layer and multilayer thin films. It has been shown (Sanyal et al., Phys. Rev. Lett., 1991, 66, 628) that capillary wave fluctuations lead to remarkable long-range algebraic decay of the density correlations at liquid-vapour interfaces as a consequence of

dimensionality; this in turn produces power law tails in the diffuse scattering. We shall discuss our results of the analysis of polymer surfaces done using similar approach. We present a new method (Sanyal et al., Euro-Phys. Lett., In Press) of obtaining a model independent electron density profile for a thin film using X-ray reflectivity measurements carried out at energies close to, and away from a X-ray absorption edge of the substrate. We shall illustrate this method using both simulated data and data obtained from a synchrotron experiment carried out on Langmuir Blodgett films. We shall also show that the analysis of small angle specular reflectivity and diffuse scattering data for multilayer films (Sanyal et al. Mat. Res. Soc. Symp., 1992, 237, 393) can provide us information regarding the interfacial roughness and its conformality.

MS-11.02.03 SELF-ASSEMBLED MONOLAYERS ON CRYSTAL SURFACES STUDIED BY SURFACE X-RAY SCATTERING.* By Keng S. Liang, Exxon Corporate Research, Annandale, New Jersey 08801, USA.

Self-assembled monolayers (SAM's) formed on solid surfaces by the spontaneous chemisorption of long chain functionalized hydrocarbons have attracted much interest recently. We report our studies of the structures and phases of n-alkyl thiols SAM's on crystal surfaces of Au(111), Au(100), and Ag(111) using surface X-ray scattering techniques with synchrotron radiation. The samples were investigated both under UHV conditions on pre-deposited films and in-situ in an electrochemical cell. In this talk, we will discuss: (1) the reconstruction of the metal surface as the result of thiol adsorption, and the consequent effects of the reconstruction, (2) the global (n,T) phase diagram of $\text{CH}_3(\text{CH}_2)_{n-1}\text{SH}$ on a Au(111) surface¹ which reflects the relative importance of hydrocarbon and interface interactions in these system, and (3) the modification of the electrolyte/SAM interfaces using different head groups and counter ions in solution². The implications of our results for future molecular design of SAM's will be discussed.

* The work is performed in collaboration with P. Fenter, J. Li, P. Eisenberger, and G. Scoles.

¹ P. Fenter, P. Eisenberger, and K. S. Liang, to appear in Phys. Rev. Lett.

² J. Li, K. S. Liang, and G. Scoles, to be published.

MS-11.02.04 MICROROUGHNESS OF POLISHED SILICON SURFACES AND MAGNETIC MULTILAYER INTERFACES EVALUATED FROM X-RAY GLANCING-ANGLE REFLECTIVITY DATA, By M. Nakanishi*, A. Yu. Nikulin, O. Sakata and H. Hashizume, Res. Lab. of Engineering Materials, Tokyo Inst. of Technology, Nagatsuta, Yokohama 227, Japan.

We have determined microroughness of mechanochemically polished silicon (100) surfaces from X-ray glancing-angle reflectivity data. Effects of macroscopic surface corrugations were eliminated by measuring reflectivities as a function of X-ray scattering angle 2ϕ on a triple-axis reflectometer with Ge(111) monochromator and analyzer crystals. Three silicon samples polished using standard but different strengths of mechanical and chemical factors showed equally smooth surfaces under a WYCO optical phase-shift interferometer with Gaussian rms roughness values (σ) of 3.5~4.1 Å. X-ray Fresnel reflectivity data revealed distinct decay features of the scattering profiles for these samples. Profile fits evaluated σ values at 11~12 Å under oxide layers of a density close to 2.0 g/cm³. The disagreed σ values could be explained by the different lateral resolutions (nanometers versus micrometers) of the X-ray and optical methods. Nonlinear least-squares profile fits also indicated a stoichiometry not far from SiO₂ for the overlayer oxide.

The same technique was applied to (Ni, Fe) multilayers for magnetic disk applications. The samples, prepared on a glass substrate by a sputtering technique, include two magnetic layers with slightly different alloy compositions on the both side of a Ta isolation layer. X-ray reflectivity data determined the thickness of each layer (80~200 Å) to 3% accuracy with σ values at the (Ni, Fe)-Ta interfaces at 14~15 Å. A 30 Å-thick oxide layer was found on top of the first magnetic layer.

MS-11.02.05 GROWTH AND STRUCTURE OF SINGLE CRYSTAL COPPER/NICKEL SUPERLATTICES. By K. Sakaue*, M. Niboshi, T. Nagahara, N. Sano and H. Terauchi, School of Science, Kwansai-Gakuin University, Nishinomiya 662, JAPAN; A. Yoshihara, RISM, Tohoku University, Sendai 980, JAPAN.

In recent years, there is a great deal of attention due to the specific features arising from metallic superlattices (SL's). Anomalous elastic feature, called supermodulus effect, was found on copper/nickel SL's in the vicinity of the modulation wavelength of 17Å (T. Tsakalacos *et al.*, J. Appl. Phys., 1983, 54, 734.). In 1991, Kumar *et al.* (S.Kumar, R.Bhadra, A.Fartash and M.Grimsditch, Phys. Rev., 1991, B44 5905.) and Davis *et al.* (B.M.Davis, D.N.Seidman, A.Moreau, J.B.Ketterson, J.Mattson and M.Grimsditch, Phys.Rev., 1991, B43 9304.) reported skeptical results on the supermodulus effect in Cu/Ni systems.

Single crystal (111)- and (001)-Cu/Ni SL were successfully grown by molecular beam epitaxy. In-plane orientation was characterized by the grazing incidence X-ray measurement as well as by the *in-situ* reflection high energy electron diffraction (RHEED). Total thickness of the superlattices was kept 4000Å for the Brillouin scattering measurement. The periodic structure was verified by the X-ray satellite peak even from the modulation wavelength (Å) of 10Å, implying that the superlattice gave the distinct modulation profile. The streak RHEED pattern observed during superlattice growth indicates the fairly smooth interface/surface.

Brillouin scattering measurement of the single crystal SL showed large increase in the surface phonon velocity propagating along [110] direction on the [111] SL and little increase in that along [100] on the [001] SL.

We will discuss the epitaxy, crystallinity and perfection of the SL from the X-ray measurement and transmission electron microscopy.

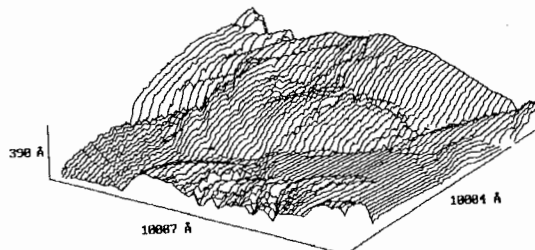
PS-11.02.06

FRactal ANALYSIS OF YBa₂Cu₃O₇ THIN FILM SURFACE USING STM TOPOGRAPHS By M. V. H. Rao, B.K. Mathur and K.L. Chopra, Department of Physics, Indian Institute of Technology, Kharagpur 721 302, India.

Points, segments and plains have fractal dimensions(FD) 0, 1 and 2 respectively. But in between those objects with integer dimension, lies complex, irregular objects whose FD can be thought of as a measure of their irregularity. The FD of an irregular surface can lie between 2 and 3 depending on how much volume it fills. Thus FD can be used to compare the complexity of surfaces, and therein lies its importance for applications. In material science, FD is directly related to surface roughness. Other modes of surface roughness characterization require a large number of parameters for qualitative and quantitative description where as a FD can give us a simple, one-parameter definition of surface roughness.

Surfaces of thin films formed from the vapor phase arise due to such nucleation and growth processes as transport, diffusion, condensation and coalescence and so can be modeled as fractals. Since the areal picture or the three dimensional image of the surface can be obtained by using Scanning Tunneling Microscopy, it has become possible to extend fractal analysis to characterize thin film surfaces. The evaluation of FD of the surface S utilizes a profile P, which is a section of S by a plane, whose FD is evaluated using box-counting method. The FD_{surface} is given by 1 + FD_{profile}. The box-counting method involves counting the number of boxes N(L) through which the profile P passes for a given box length L. By varying L in a sequence, N(L) is counted for each case. The slope of the line obtained by plotting ln(N(L)) against ln(profile length/L) gives the FD of the profile.

Fractal characterization of YBa₂Cu₃O₇ (YBCO) films has been carried out and an attempt has been made to correlate their physical properties with their FDs. These films are prepared by rf magnetron sputter deposition on MgO at room temperature from a single target and subsequent annealing at a high temperature(850-980°C) in oxygen ambient. During our investigation, a 1µm x 1µm STM topograph of YBCO thin film surface was recorded as 256 x 256 data points. A topograph of YBCO film annealed at 980°C is shown in the diagram below.



The FD of the YBCO thin film surface ($T_{\text{anneal}} = 850^\circ\text{C}$) calculated by box-counting method is around 2.03 ± 0.005 . It is observed that the FD value decreases as the film goes from non superconducting ($T_{\text{anneal}} < 900^\circ\text{C}$) to superconducting phase ($T_{\text{anneal}} > 900^\circ\text{C}$). After the formation of superconducting phase, FD value increased with annealing temperature along with increase in size of the grains which are having many growth steps.