

## 11-Surfaces, Interfaces and Thin Films

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**PS-11.02.13 X-RAY REFLECTIVITY STUDY OF POLYMERIC THIN FILMS.** By Lichen Wang and Earle Ryba, Department of Materials Science and Engineering, The Pennsylvania State University, University Park, PA 16802

Recently, neutron and X-ray reflectivity have emerged as powerful tools for the investigation of the surface, interface behavior of polymeric materials. We have used the X-ray reflectivity technique to study various types of polymeric thin films. The density profile for a very thin polyethylene film on a gold substrate, determined through non-linear model fitting of the reflectivity data, will be presented. The density of the film is significantly higher near the substrate, where the substrate surface appears to have induced an enhanced crystallinity. Near the air/polyethylene interface, the density is slightly higher than that in the bulk of the film. The crystallinity near this interface appears to be greatly affected by the surface tension. It is interesting that the roughness near both of these interfaces derived from the model fitting of the reflectivity curve is smaller than what we expect. Support from the Eastman Kodak Company is gratefully acknowledged.

**PS-11.02.14 STRUCTURAL STUDIES OF MEMBRANES AND SURFACE LAYERS USING VARIABLE PERIOD X-RAY STANDING WAVES.** By J. Wang and M. Caffrey\*, Department of Chemistry, The Ohio State University, U.S.A.; M. J. Bedzyk, Department of Materials Science and Engineering, Northwestern University and Material Science Division, Argonne National Laboratory, U.S.A.; T. Penner, Corporate Research Laboratories, Eastman Kodak Company, U.S.A..

The ability to obtain structural information with subångström resolution on Langmuir-Blodgett (LB) model membranes using long period x-ray standing waves (XSW) has been demonstrated previously (Bedzyk *et al.*, Science, 1988, 241, 1788-1791; Phys. Rev. Lett., 1989, 62, 1376-1379). In the present study, we wished to determine 1) if the variable period XSW generated by an x-ray mirror during total external reflection could be used to locate precisely and accurately a heavy atom layer positioned several hundred ångströms or higher above the mirror surface which would prevail in a host of biologically relevant model systems, and 2) if the thermally induced phase transitions occurring in LB films are sensitive to the number and identity of the lamellae in the membrane stack. The sample series examined in addressing the first question consisted of an octadecyl thiol coated gold mirror on top of which a variable number (0, 2, 8, 14) of  $\omega$ -tricosenoic acid ( $\omega$ TA) bilayers followed by a single, upper bilayer of zinc arachidate was deposited by the LB technique. With 14 bilayers of  $\omega$ TA, zinc-to-gold surface

separations were close to 1,000 Å. The XSW measurements showed that the zinc  $K_{\alpha}$  fluorescence yield profiles from the sample set are in excellent agreement with the calculated XSW electric field distribution (Wang *et al.*, Nature, 1991, 354, 377-380). Further, the numerical fitting of the data reveals that ångström precision can be achieved in determining both zinc atom layer mean position and width above the gold mirror surface. The two samples chosen for the thermal stability study of LB films were identical to those described above and incorporated 0 and 2 bilayers of  $\omega$ TA, respectively. Variable period XSW measurements provided precise positional information on the zinc layer mean position and width and were used to track the collapse of the heavy atom layer during thermotropic phase transitions. The two samples showed quite disparate pretransitional rearrangements, transition temperatures and apparent cooperativity and final, high temperature zinc distribution. Further, the temperature-induced change observed was not reversed upon cooling to and subsequent storage at room temperature. The results of these experiments demonstrate clearly that the XSW field is well defined at close to a thousand ångströms above the mirror surface and that the XSW method is well suited for determining the position of layered heavy atoms with a precision of ångströms while the heavy atom layer is up to 1,000 Å or even higher above the mirror surface. Additionally, the quality of these data shows the enormous potential of XSW as structural probes in membranes and in thin film related phenomena.

**PS-11.02.15  
STRUCTURE OF MOLYBDENUM SULPHIDE THIN FILMS**

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Thin films of  $\text{MoS}_2$  (150 nm - 600 nm in thickness) were produced by HF-magnetron sputtering onto silicon wafers. The structure of the films has been investigated as a function of temperature using X-ray diffraction and transmission electron microscopy. Fig. 1 shows the obtained diffraction patterns as a function of temperature. The X-ray patterns indicate that there is a continuous development of the structure rather than transition from amorphous to crystalline state.

The crystal structure of hexagonal  $\text{MoS}_2$  (space group  $P6_3/mmc$ ) is characterized by a stacking of Mo and S layers. The initial state of the films is characterized by a random stacking of S-Mo-S layers forming two-dimensional lattice in the a-b plane. The extension in c-direction of the stacks is around 2 nanometres. Thermal treatment leads to an increase in grain size to around 20 nm at 900°C.

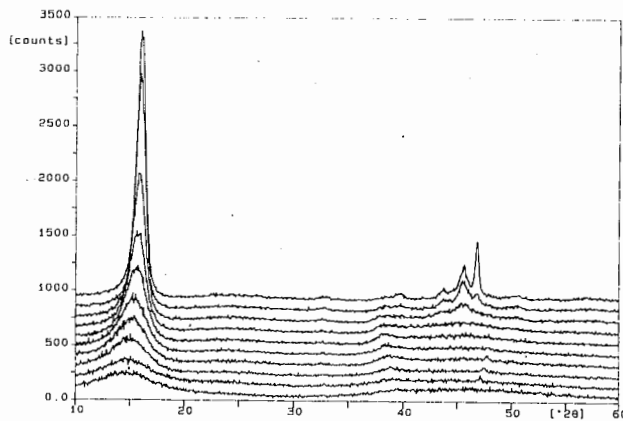


Fig. 1: Diffraction pattern of a 450 nm thick  $\text{MoS}_2$  film at  $T = 35$  (lower), 100, 200, 300, 400, 500, 600, 700, 800, 900°C (upper)

Parallel to the crystal growth a reduction of the fraction of stacking faults is observed indicated by the decrease of the c-lattice parameter and by the appearance of (hkl)-reflections. Differences in the initial film thickness influence the structural state of the untreated material but they disappear during heat treatment.

**PS-11.02.16 THE STRUCTURE AND MAGNETIC ANISOTROPY OF Pt/Co MULTILAYERS** By Zhi-hong Jiang<sup>1)</sup>, Chang-lin Kuo<sup>2)</sup>, De-fang Shen<sup>1)</sup>, Rong-fa Guo<sup>2)</sup>, Tian-shen Shi<sup>1)</sup>. 1) Shanghai Institute of Metallurgy, Chinese Academy of Sciences, Shanghai, P.R.China. 2) Shanghai Institute of Ceramics, Chinese Academy of Sciences, Shanghai, P.R.China

Recently, Pt/Co multilayer has stimulated the interests of many researchers due to its potential as a high-density magneto-optical storage material. Generally, such a candidate material must have a large Faraday or Kerr effect at short wavelength, a perpendicular anisotropy and a relatively large coercivity at room temperature. Pt/Co multilayers happen to satisfy all these conditions.

In our experiments, Pt/Co multilayers were prepared using dc magnetron sputtering on silicon substrates. The structures were determined by X-ray diffraction and the magnetic properties were measured by Kerr angle hysteresis loops. In table 1 are listed the sample parameters and their magnetic properties (omitted).

The low angle X-ray diffraction pattern, as shown in Fig.1, clearly confirmed the existence of multilayer structure of sample 1. The bilayer thickness deduced from Bragg formula agreed well with the sample parameter.

A simple simulation of high angle x-ray diffraction pattern was made. At any circumstances, the  $n=-1$  satellite peak was higher than the  $n=+1$  satellite peak, which is in contradiction to the real pattern (shown in Fig.2). We contribute this phenomena to the appearance of PtCo alloy at the interfaces, whose (111) peak overlaps with the Bragg peak of multilayers and (200) peak is at the same position of  $n=+1$  satellite peak (C.-J. Lin and G.L.Görman, Appl. Phys. Lett., 1992, 61(13), 1600). In sample

2, when Co layer was much thicker, the multilayer diffraction overwhelmed the PtCo alloy diffraction and the relative heights of these two peaks reversed.

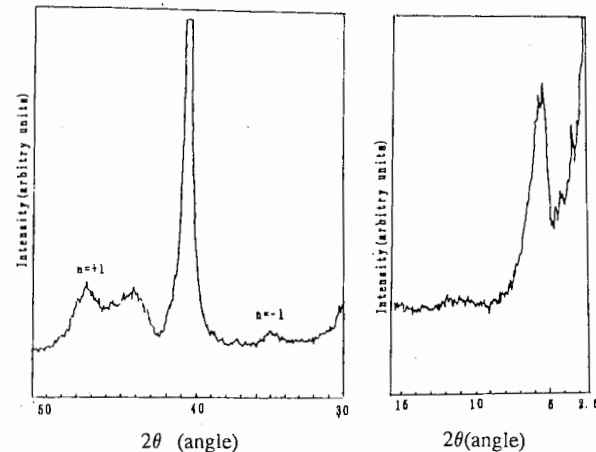


Fig.1 Low angle X-ray diffraction pattern of sample one

Fig.2 High angle X-ray diffraction pattern of sample one

In conclusion, we have found the PtCo alloy diffraction at Pt/Co multilayers. And the appearance of PtCo alloy did not destroy the perpendicular magnetic anisotropy which is believed to be mainly induced by interface anisotropy. The influence of interface atoms on the anisotropy can be further studied.

**PS-11.02.17 CROSS-HATCHED SURFACE MORPHOLOGY IN InGaAs/GaAs SUPERLATTICES.** By S. F. Cui\*, Z. H. Mai, G. M. Wang, W. Feng, L. S. Wu, C. R. Li, J. H. Li, D. Y. Dai and J. M. Zhou, Institute of Physics, Chinese Academy of Sciences, Beijing 100080, China

The cross-hatched morphology (CHM) has been commonly noticed in strained III-V semiconductor films (K. H. Chang et al, J. Appl. Phys., 1990, 67, 4093-4098). The CHM has been observed by means of Nomarski interference, x-ray topography, TEM, cathodoluminescence and photoluminescence but few results about the CHM were presented for InGaAs/GaAs strained-layer superlattices (SLSs).

In the present letter we report the x-ray topographic examinations of InGaAs/GaAs SLSs. The specimens used in the experiments were grown by molecular beam epitaxy (MBE) on GaAs substrates. The nominal structure of the SLS samples were 150 periods of 70 Å  $\text{In}_{0.04}\text{Ga}_{0.96}\text{As}$  and 250 Å GaAs with about a 1 μm GaAs buffer layer and a 3 μm capping layer, respectively. In order to eliminate the influence of the capping layer on the reflection topographs a sample (denoted A) was etched to remove its capping layer. The synchrotron radiation experiments on sample A were performed at 4W1A beam line of the Beijing Synchrotron Radiation Facilities (BSRF).

The actual structure of the specimens were determined by the simulations of experimentally measured rocking curves based on the dynamic diffraction theory for deformed crystals (Z. H. Mai, S. F. Cui and C. G. He, Phys. Rev. B, 1990, 41, 9930-9934). Percentage relaxations of the two component layers of SLS were particularly found from the simulations.

224 reflection topographs were taken at the zeroth order peak of the SLS and the substrate peak, respectively. They were characterized as orthogonal striation parallel to the [110] and  $[\bar{1}\bar{1}0]$  directions, respectively. X-ray topographs were also taken under anomalous transmission conditions using  $\text{Cu K}\alpha_1$  radiation ( $\mu t=17$ , where  $\mu$  is the linear absorption constant and  $t$  the sample thickness). In 220 or 220 anomalous transmission topographs shown in Fig. 1 the striations parallel to [110] or  $[\bar{1}\bar{1}0]$  disappeared, respectively. According to the invisibility criteria of dislocations, both the striations parallel to [110] or  $[\bar{1}\bar{1}0]$  were edge-type dislocations with Burger's vectors in  $[\bar{1}\bar{1}0]$  or [110] direction in the (001) growth plane.

It is interesting to see that in addition to the striations in the (001) growth plane other striations (see regions C of Fig. 1) which obeyed the same extinction law were observed on the cleavage planes of sample A. The stereoscopic topographs provide us a evidence that the misfit dislocations distribute over the SLS and