

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 ω -tricosenoic acid (ω TA) bilayers followed by a single, upper bilayer of zinc arachidate was deposited by the LB technique. With 14 bilayers of ω TA, zinc-to-gold surface

separations were close to 1,000 Å. The XSW measurements showed that the zinc K_{α} 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 ω 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 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 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.