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Synchrotron Studies of X-ray Reflectivity from Surfaces**Prof. P. S. Pershan, Harvard University****November 2, 1990**

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PROGRESS

During the previous report period we observed that although thin liquid Hg films will wet metallic substrates such as copper, with which it forms stable intermetallic compounds, it appears that there is a progressive build up in the concentration of substrate atoms (e.g., Cu) that leads to a time dependent surface which is not suitable for study. On the other hand, there is good evidence that wetting does occur at interfaces between liquid and solid metals when the surfaces are free from oxides, even if they are mutually insoluble and even if they do not form intermetallic compounds.^(1;2;3;4;5;6) During this report period we completed the design of a UHV chamber which will be suitable for study of materials, such as Ga, Sn and In,^(7;8;9;10) that have vapor pressures in the range of 10^{-11} torr at temperatures of 708 C, 800 C and 644 C respectively. Their melting temperatures are respectively 29.9 C, 232 C and 156 C; however, alloys such as Ga-In 24%wt In and Ga-Sn 84% Sn have melting temperatures at 15.7 C and 20.3 C. For these materials standard UHV surface techniques can be employed and the design of the chamber allows for AES analysis. All of the essential components of the vacuum system have been ordered and our first design was submitted to both the Perkin-Elmer and the Electrofusion companies for bids. We selected Electrofusion on the basis of their experience with construction of custom chambers of comparable complexity. The first set of engineering drawings that were prepared by them in August had a major flaw in that their design had the seal of the beryllium x-ray window crossing a weld. They are currently reworking these drawings; however, they have quoted us delivery date of the vacuum tested chamber in early 1991.

While waiting for the main chamber we have constructed an inexpensive "test chamber" using a glass vacuum bowl with a specially designed, but inexpensive, stainless steel top on which were mounted electrodes capable of sustaining a dc plasma discharge. This chamber is currently being used to gain experience with the manner in which Ga-In alloys wet various substrates. In addition, the design of this chamber is particularly simple and once we have developed the requisite experience, it should be possible to construct a similar chamber, except that it will have a stainless steel body and a Be-foil x-ray window, that can be used to study liquid Hg.

MASTER

The pressure in the test chamber was easily brought down to $\sim 10^{-8}$ to 10^{-9} torr using one of the spare turbomolecular pumps, available in the laboratory, and a plasma was sustained with Ar pressures of the order of 10-500 mtorr and a dc voltage between 400 and 1000V. Experiments, with varying degrees of success were carried out on a Ga-In eutectic alloy placed on a Ta substrate (i.e., the cathode of the discharge) and a stainless steel anode; and also with the same alloy and Ta substrate but with a Ta anode replacing the stainless steel one. In the second case there was evidence for some wetting immediately following the start of the discharge; however, after about 20 minutes the surface of the liquid Ga-In eutectic became visibly contaminated. Ta is known to have a very low sputtering rate and we believe that the surface became hot before there was very much cleaning. Following this we repeated the experiment with a Ni cathode, which has a sputtering yield that is 2 to 3 times larger than that of Ta, and, using a repeated cycle of alternate heating and cooling. In this case there was a significant improvement in both the spontaneous spreading of the alloy and the contact angle; however, the wetting was still not complete. We believe that this is due to a combination of the fact that our method of delivering the sample to the substrate allowed a relatively high degree of contamination, and secondly, that the substrate becomes too hot during the discharge. We have tested the latter hypothesis by measuring the temperature of the substrate. In fact the liquid drop starts to spread on the substrate immediately following the initiation of the discharge; however, it stops as the substrate temperature rises to $\sim 150^{\circ}\text{C}$. After a few minutes of cooling with the discharge turned off, spreading begins immediately following restart of the discharge. We are currently exploring various ways to cool the substrate..

We have also continued to collaborate with colleagues of the IDT, in the development of the instrumentation for beam line X25 at the NSLS. In the past year Dr. Lee and Mr. Kawamoto have spent approximately 4 weeks on this aspect of the project. In addition, as part of their training for synchrotron measurements, each of them has participated, in a support capacity, in experiments on the surfaces of non-metallic liquids. This aspect of their training is now complete and we have confidence in their ability to carry out independent measurements using the Harvard(MRL)/BNL(Physics) liquid surface reflectometer on beam line X22b.

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