

**Experiment title:**

Adsorbed Polyelectrolyte Layer at a Solid/Water Interface

Experiment number:

SC 314

Beamline:**Date of experiment:**

from: 24th June to: 29th June

Date of report:

15Aug

Shifts:**Local contact(s):**

Francois Rieutord, Oleg Konalov

*Received at ESRF:***26 AOUT 1998****Names and affiliations of applicants** (* indicates experimentalists):

Thomas A. Waigh*, O.Theodoly*, C.E.Williams, R.Ober*, College de France
J.Daillant*, SPEC, Saclay

Report:

Following on from previous experiments, a series of practical improvements were implemented of general use in liquid/solid reflectivity experiments:

- a) The planarity of the Silicon wafers was checked before use with a lab based rotating anode X-ray source and inferior sample were discarded.
- b) Thicker Silicon wafers (10mm) were used to reduce problems of curvature during pumping. The liquid cell was redesigned to facilitate the positioning of the new thicker samples.
- c) A detailed investigation was made in to problems of beam damage of the silane surfaces Figure 1 indicates a series of normal electron density maps as a function of the time for the irradiation of silane in air. It can be observed that the rugosity of the surfaces rapidly increases after 0.5 hour as the silane is stripped off the surface. Immersed the sample in water slow this damage, but it was still found necessary to reduce the exposure time on the sample to less than half an hour to circumvent these problems. Furthermore the whole liquid cell was placed on a motorized horizontal stage to allow a series of spectra to be obtain from the same sample without entering the hutch. Thus a series of low exposure spectra could be averaged and compared to study their reproducibility.

Results

The thickness of adsorbed polyelectrolyte layers was examined as a function of bulk ionic strength, 0M, 0.01M, 0.1 and 0.2M CsCl. Figure 2 shows electron density profiles derived from Patterson functions[1] for 42 and 55% charged CsPSS in pure water. When salt was added it was discovered that there was little effect on the globular structure of the lower charged PSS (42%) whereas the higher charged samples collapsed on to the surface. The electron profiles of the 42 and 55% charged polymers appear to indicate that a mushroom and pancake structure exists for the two charge contents respectively[2]. This conclusion is inferred from the difference in the size of the depletion region near to the surface. Off-specular scans were also taken and indicate a change in rugosity on the addition of salt. Addition of TEE, a solvent known to preferentially solvate the hydrophobic regions of PSS and destroy globular structures, was also studied. It was found to increase the thickness of the adsorbed layer of 35% charged CsPSS (3.5 to 3.9nm), but to bring about desorption of the 55% charged CsPSS.

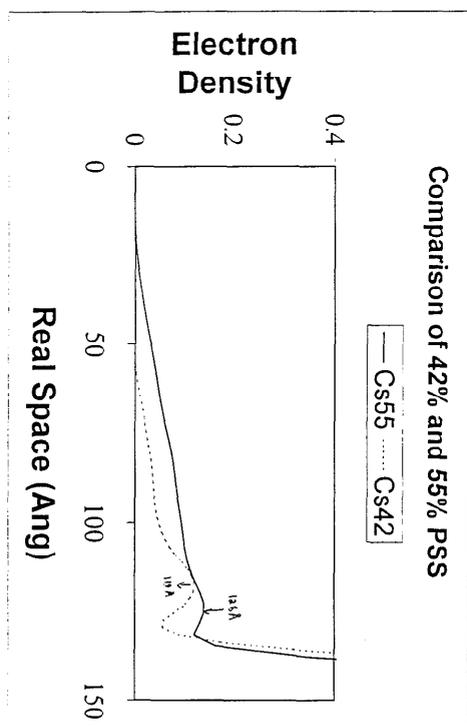


FIGURE 2

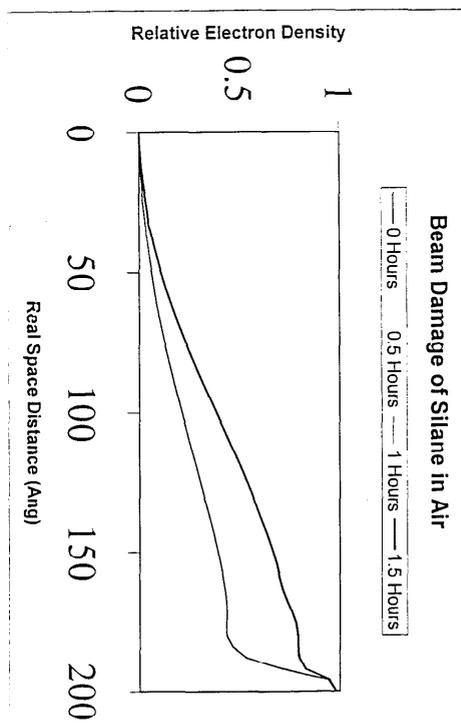


FIGURE 1

References

- (1) J.S.Pederson, *J.Appl. Cryst.*, 25, 129-134, 1992
- (2) E.Zhulina, C.Singh, A.C.Balazs, *J.Chem.Phys.*, 108, 3, 1175-1183, 1998