ESRF	**Experiment title:** 1) Grazing angle diffraction using the effect of total internal reflection at high photon energies (Block allocation of beamtime with Si-504)	**Experiment number**: MI-339
Beamline: ID 15A	**Date of experiment**: From: 20.10.1999 to: 2.11.1999	**Date of report**: 24.2.1999
Shifts: 18	**Local contact(s)**: Veijo Honkimaki	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Harald Reichert, Oliver Klein, Johann Trenkler

Max-Planck-Institut fuer Metallforschung
Heisenbergstrasse 1
D-70569 Stuttgart
Germany

Report:

Most commonly, interfaces and surfaces are studied in surface scattering geometries (Grazing Angle Diffraction (GAD) and Crystal Truncation Rod Diffraction (CTRD)) at typical photon energies around 10keV. These techniques are not applicable in the case of deeply buried solid-liquid interfaces for several reasons: Conventional x-ray scattering geometries are unable to separate the scattering signals from a structurally modified thin liquid layer at an interface from the large background of bulk-like liquid scattering signals. Another problem is the strong absorption of the incoming and scattered beams within the solid and the liquid producing very weak scattering signals on top of a large background from the penetrated solid.

In order to achieve the necessary interface sensitivity the geometry of GAD has been modified in such a way that total internal reflection occurs at the solid-liquid interface. This produces evanescent waves in the liquid closest to the interface. The evanescent wave within the thin liquid interface layer is then subject to scattering at the density inhomogenities within the liquid. In this project we have successfully installed and tested this scattering geometry at the high energy beamline ID15 taking advantage of the high brilliance of the source.

In our experiment a monochromatic and highly collimated high energy x-ray beam impinges on a flat and mirrorlike interface. The beam is passing almost normal to the wall of the solid with negligable refraction effects and gets subsequently reflected at the interface. For this scenario the electron density of the solid must be smaller than the electron density of the liquid. By chosing the incidence angle α_i smaller than the critical angle for total internal reflection α_c an evanescent wavefield is transmitted into the liquid (see Figures) .

In order to minimize bulk liqid scattering as well as the parasitic background contributions from the solid, the beam height of the incident x-ray beam has been limited to the projected height of the interface which is typically 10µm depending on the thickness of the sample and the wavelength used. In order to meet this requirement we have installed tungsten collimators in the incoming beam producing an effective beamsize of 10µm with a vertical divergence of 0.3µrad at the sample position. The cylindrical Si crystal (diameter : 20mm) is almost transparent at an x-ray energy of E=71.5keV.

The necessary diffraction equipment has been supplied by the Max-Planck-Institute (Stuttgart). Since structure factor measurements at thin solid-liquid interfaces are intensity limited even at modern synchrotron radiation sources, it is favorable to use high Z liquids like Pb in order to maximize the scattering signals from the liquid structure factor. The first experiments have successfully been carried out at the interface Pb(liq.)-Si(100) with a photon energy of E=71.5keV. Fig.1 shows the reflectivity as a function of the incident angle α_i from such an interface exhibiting a roughness of 5Å. The measured critical angle of total internal reflection corresponds to the calculated electron density difference between Pb and Si.