	Experiment title: Octadecyltrichlorosilane coated silicon dioxide and in-situ measurements of the coating process	**Experiment number**: SI-2354
Beamline: ID10B	**Date of experiment**: from: 09.09.11 to: 12.09.11	**Date of report**: 14.08.12
Shifts: 9	**Local contact(s)**: Oleg Konovalov	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Hans-Georg Steinrück*, Universität Erlangen Nürnberg

Andreas Magerl*, Universität Erlangen Nürnberg

Report:

The aim of the proposed experiment was to investigate via specular X-ray reflectivity in-situ the growth process of octadecyltrichlorosilane and triacontyltrichlorosilane (TTS, $C_{30}H_{61}Cl_3Si$) (OTS, $C_{17}H_{38}Cl_3Si$) from solution on different metal oxides. Due to limited beamtime (9 shifts) we focused on the coating process of OTS on the native amorphous silicon dioxide of a silicon wafer and (001)-oriented quartz single crystals during the allocated beamtime. Prior to the experiment an in-situ coating cell was designed and put into operation. In preperation reference samples were synthesized at our home laboratory. These were OTS as well as TTS on amorphous SiO_2 and on several single crystalline substrates (SiO_2 (001), Al_2O_3 (001), TiO_2 (100)). Reflectvities of these samples were measured at ID10B during the allocated beamtime. Referring to this reflectivity data, all substrates have high quality coatings with OTS/TTS. This is further supported by contact angle measurements (all θ_c>100°). These results will be followed up by investigating the growth of OTS and TTS on these metal oxides in-situ during future beamtime allocations.

As further reference scans for the in-situ measurements, clean and fully coated amorphous SiO_2 and quartz substrates were measured against the utilized solvent heptane, showing a distinct minimum in the reflectivity at about q=0.38$Å^{-1}$ for the coated samples. In-situ measurements in highly dilute solutions of OTS in heptane (0.005 - 0.0005 mM) were performed on amorphous SiO_2 and quartz substrates, taking a snapshot of the coating state every 30 minutes via a reflectivity measurement. We observed changes in the reflectivity curves already with the first snapshot. Experiments were stopped once the profiles did not change for several subsequent snapshots. Comparing the data with the reference scans it has to be stated that a full monolayer coverage was not reached even after five attempts for a succesful coating . The reason for this is unclear as the utilized concentrations were tested in the same cell at home yielding a full monolayer. One interpretation may be that the high humidity present during the experiment may have caused the molecules to cross-link with each other already in solution. As a result of this they are no longer available to form a monolayer coating. Similar experiments carried out at the high resolution diffraction beamline P08 at DESY resulted in full monolayers.

Measurements were carried out at 20.925 keV using a line detector.

Unfortunately a modelling of the reflectivity data for all samples is not possible due to q-scale issues. This becomes readily visible from the positions of the extrema of the reflectivity curve which are shifted to lower q values as compared to measurements of the same sample at our lab source and at P08 DESY. These shifts increase with increasing q, indicating some sort of stretching of the q-scale. This is exemplary indicated in fig. 1. Extensive post-beamtime discussions with the beamline scientist as well as ideas of rescaling the q-scale via comparing minimas did not solve this problem. In our opinion the problem may relate either to the beam energy, the distance between sample and detector, or the line size of the detector.

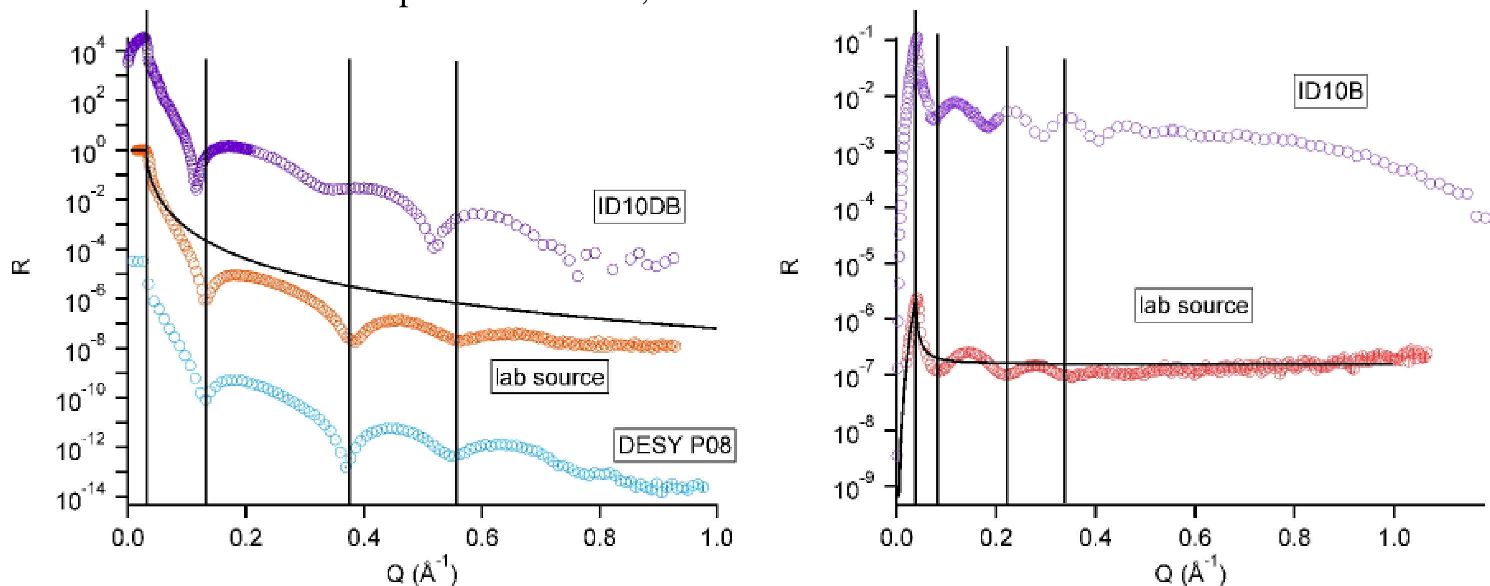

Fig. 1: Profiles are shifted for clarityon the ordinate. left: OTS on amorphous SiO_2, solid line represents the Frensel reflectivity for an ideally flat silicon wafer; Crucial features of the Reflectivity profiles are similar for lab source and P08, whereas the spectra are shifted to lower q values for ID10B. right: TTS on crystalline TiO_2, R is divided by q^4 for clarity, thesolid line represents Frensel reflectivity of an ideally flat TiO_2 wafer. Thextrema are shifted to lower q values for ID10 as compared to data from the lab source.