Report:

The aim of our proposed experiment was to investigate the in-depth electron density distribution of self-assembled monolayers (SAMs) deposited from solution and evaporated semi-conducting small molecules on atomic layer deposited (ALD) AlOx and sapphire via X-ray reflectivity (XRR). A possible crystalline arrangement was examined via grazing-incidence diffraction (GID). Our group utilizes SAMs as dielectric layers in organic thin-film transistors (OTFTs) and as bi-functional SAMs including an insulating and a semiconducting part within a molecule for the use in SAM field-effect transistors (SAMFET).
Prior to the experiment at the ESRF, all SAMs were deposited in the laboratory of the OMD-group in Erlangen either on 100 Å ALD AlO_x on Si or on sapphire substrates with an orientation of (0001) and (1-102). The substrates were examined via AFM for surface roughness. The presence of the SAM was examined with static contact angle (SCA) measurements which showed no deviation from expected values.
At BM28 firstly XRR was performed on the following SAMs:
HO-C_{11}-phosphonic acid (PA), Mixed C_{16}-PA/NH_3^+-C_{12}-PA, Cl^--NH_3^+-C_{12}-PA, NH_2-C_3-PA, Br^--Imidazole-C_{12}-PA, mixed F_{15}-C_{18}-PA/C_{60}-C_6-PA (at different ratios), mixed C_{14}-PA/C_{60}-C_6-PA + 3.5 nm evaporated C_{13}-BTBT (benzothienothiophene), BTBT-C_{12}-PA, BTBT-C_{11}-PA, mixed C_{10}-PA/C_{60}-C_{18}-PA, mixed C_{14}-PA/C_{60}-C_{18}-PA, mixed C_{16}-PA/C_{60}-C_{18}-PA, mixed C_{18}-PA/C_{60}-C_{18}-PA, mixed F_{15}-C_{18}-PA/C_{60}-C_{18}-PA, mixed F_{21}-C_{12}-PA/C_{60}-C_{18}-PA.
The point detector set-up required the mount of the detector perpendicular to the reflected beam at low angles (between 0° and 0.5°) where the reflected beam was deflected to 90° before hitting the detector without any further absorber. Above 0.5° the detector was placed in reflected beam direction without the deflector. Unfortunately below 0.5° severe radiation damage on the samples was discovered which led to non-reproducible reflectivity data. XRR measurements had to be repeated after the first 3 shifts. Consequently the position of the sample was changed above 0.5°. We obtained i.a. the following results on the distribution of C_{60} moieties in different SAM combinations after fitting the data.

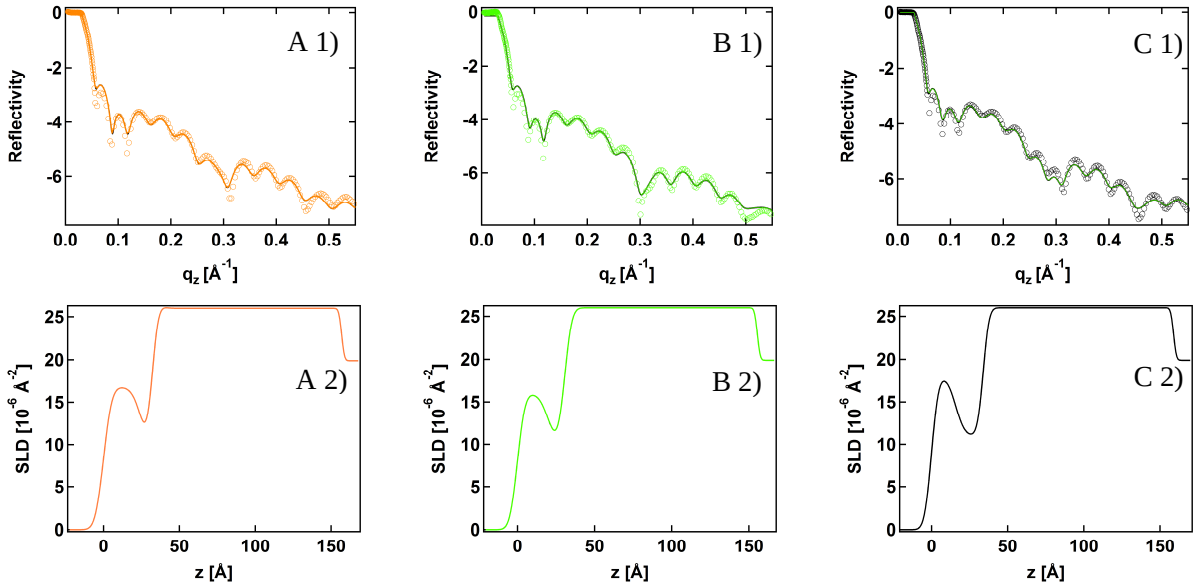

Fig. 1: Reflectivity profiles and corresponding fits of A) pure $C_{60}C_{18}$-PA SAM B) mixed C_{10}-PA/$C_{60}C_{18}$-PA SAM and C) mixed C_{14}-PA/C_{60}-C_{18}-PA SAM on ALD AlO_x substrates.

The mixed C_{14}-PA/C_{60}-C_{18}-PA SAM (Fig. 1 c) exhibits the most confined C_{60} peak with the lowest SLD value in the proximity of the ALD surface suggesting a narrow fullerene distribution in-depth compared to other comnbinations.
GID measurements with a point detector as well as with the MARCCD area detector were not successful for our systems. Due to the loosely crystalline structure of our SAMs on sapphire and ALD surfaces counting time had to be increased which led to radiation damage. During the recent Beamtime at ID10 the experiments were repeated and crystalline structures could be resolved (see report for MA-1869) due to higher flux at ID10.