ESRF	**Experiment title:** Structural investigation of nanoparticle-decorated organic semiconductor matrices using soft deposition techniques.	**Experiment number**: SC-3514
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Report:

Overview

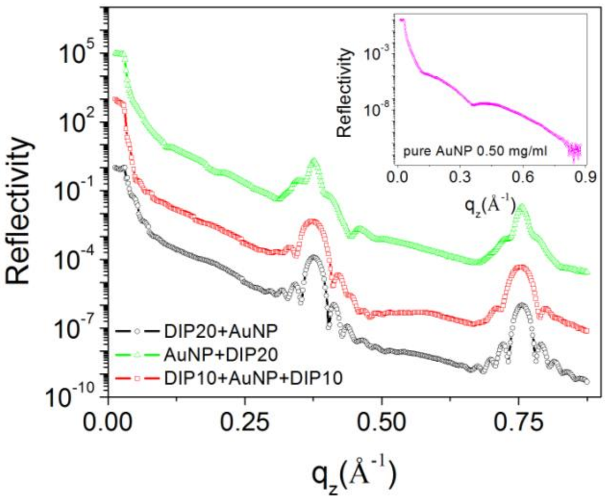
As stated in the proposal, we have performed structural characterization of hybrid structures synthesized by incorporation of metal nanoparticles (MNPs) in organic semiconductors (OSCs) using “soft” (i.e. non-invasive) deposition techniques. The aim was to have a detailed knowledge of the structure and morphology of such hybrid systems, particularly the out-of-plane and in-plane ordering of the incorporated MNPs within the matrix, using X-ray reflectivity (XRR), grazing incidence small angle X-ray scattering (GISAXS) and grazing incidence X-ray diffraction (GIXD). XRR, GIXD and GISAXS were carried out on ex-situ samples enclosed in a sample cell which provides a nitrogen atmosphere. All measurements were performed at constant energy (11 keV), below the Au absorption edge using a focused beam and Pilatus detectors. Finally we checked for the data reproducibility and radiation damage. Detailed quantitative analysis is in progress, so we can only present some preliminary findings.

Quality of measurement and data

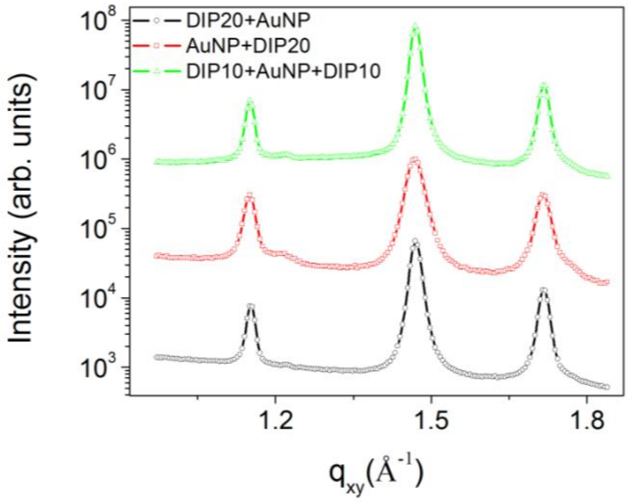
The ID03 beamline is very suitable because of its surface/interface scattering and diffraction expertise and resolution. The data sets are of high-quality with high signal-to-noise ratio inspite of the fact that the scattering signals from thin organic films (~ 50 nm) and MNPs (5-20 nm) are not very strong. The beam was very stable during the time alloted to us and we managed to succesfully measure most of our 20 samples. While the XRR and GIXD data show promising results the GISAXS data did not show any scattering features. This could be due to the roughness of the samples as well as air scattering.

Figure 1 shows the XRR profiles of the samples with layers of MNP (AuNPs for this experiment) embedded in different configurations in the organic semiconductor diindenoperylene (DIP) matrix. For the 1st series of samples, 20 nm of DIP was grown on silicon substrate and then the AuNPs (with three different concentration of AuNPs deposited per sample series) were deposited on top. We present only representative data for one of the samples per series. For the 2nd series of samples the AuNP layer was deposited first and then 20 nm of DIP deposited on top of it and for the 3rd series of sample, the AuNP was sandwiched in between two 10 nm layers of DIP. Inset of Figure 1 shows the XRR of pure AuNP layer for comparison. It is clear that the presence of

the AuNP in the DIP matrix appreciably effects the out-of-plane structure of the DIP host matrix. For the 1st sample, the XRR resembles the XRR of pure DIP. However the XRR for the 2nd sample where the DIP was grown on top of the AuNP layer, shows a broader Bragg peak and fewer Laue oscillations than the 1st sample. The XRR for the 3rd sample shows a superposition of two Bragg peaks arising from the two DIP layers that sandwich the AuNP layer. It does not feature any Laue oscillations unlike the 1st and the 2nd samples. The XRR features have a similar trend for the samples prepared with different concentrations of the AuNPs.



<u>Figure1:</u> XRR measurement of AuNP embedded in DIP host matrices with different layering strategy. Inset shows the XRR of pure AuNP layer for comparison.



<u>Figure2:</u> GIXD measurement of AuNP embedded in DIP host matrices with different layering strategy. The change in the peak width signifies differences in the in-plane crystallite size.

The GIXD measurements of the hybrid samples (see Figure 2) demonstrate that the in-plane crystalline size changes depending on the layering strategy of the AuNPs within the host DIP matrix. All the GIXD peaks observed belong to the DIP thin film phase and although we scanned for higher q_{xy} values there was no detectable Au peak. The DIP peaks in the GIXD of the 2nd sample (DIP grown on top of AuNP layer) are broader than the ones in the 1st and the 3rd samples indicating smaller in-plane crystallite size of the DIP molecules.

Status and progress of evaluation

The XRR and GIXD data have to be analysed to compare structural features and the effect of incorporation of the AuNPs in the host matrix. This would help to correlate the structural features of such hybrid materials to their opto-electronic properties. Complementary measurements like AFM, Raman scattering and near-field scanning optical microscopey (NSOM) are currently being performed additionally.

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