Microbeam in situ grazing incidence small-angle X-ray scattering (µGISAXS) investigation of the growth mechanism of metal nanostructures of metals on a flat surface during sputter deposition.

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Sputter deposition is one of the most widely used methods to deposit nanostructured thin films in science and technology [1]. It is used for polymer-metal-nanocomposites production, to introduce contacts in photovoltaic devices using [2] or to tailor ultra-high density magnetic structures using radio-frequency (RF) sputter deposition [3]. In previous studies, we introduced the so-called stop-sputter deposition method in combination with grazing incidence small-angle X-ray scattering (GISAXS) at DESY [2,5,6]. This method enables to follow in detail the selective deposition of metals on polymers and to investigate the layer's nano-and meso-structures to obtain the relevant growth laws and correlations [5,6].

Here, we report on novel stop-sputter-experiments at the Micro- and Nanofocus X-ray Scattering (MiNaXS) beamline P03 [4] of PETRA III, DESY. We combined a high-flux, low divergence microfocus X-ray beam with a specially designed, automated RF sputter deposition chamber in a novel way. We used two setups: a DC magnetron source (sputter rate 4nm/min) and an Au-sputter rate of 0.5nm/min (RF power P=50W, Argon (Ar)-pressure $2x10^{-2}$mbar), which is comparable to the used setup described elsewhere [5,6]. The substrate was acid-cleaned silicon (Si) [Ref], the size of the beam was (38x20) μm^2 and the incident angle of the beam was α_i=0.45°. During the stop-sputter experiment, the Au-deposition times were 10s (incremental Au mass deposit of Δd=0.083nm) up to a total Au-layer thickness d<2.5nm and 30s afterwards. All acquisition times were fixed to t_{acq}=10s. Fig. 1 shows a first evaluation of the µGISAXS data using the software package DPDAK [7].

In Fig. 1a), the evolution of the intensity near the Au Yoneda peak is presented as a function of deposited Au. Clearly, the leveling off - when maximum coverage is reached - is around d=6nm, i.e. at the nominal percolation threshold of Au on flat surfaces. In Fig. 0.1b), we present the evolution of the lateral nanostructuring as a function of deposited Au. D denotes the most prominent in-plane length, i.e. the distance of the cluster formed by self-assembly on the Si surface at RT. While around d=6nm no profound change is visible, a first cross-over point is already seen around d=0.6nm: The slope of the curve clearly changes.

As a second example, we present first results of sputter deposited Au on a blend of diblock copolymers film of P(S-*b*-MMA) and P(EO-*b*-MMA) using a magnetron source at a rate of 4nm/min. Fig. 2 shows an atomic force microscopy image (AFM) of the diblock film covered with 11nm of Au. Clearly, the morphology of the Au film is strongly correlated with the underlying polymer template.

In summary, we have presented a first stop-sputter experiment using a model system (Au on Si), combining successfully RF sputter depositon and in-situ µGISAXS at MiNaXS. This type of experiment gives in future access to assess the metallic film nanostructure at low coverages. Due to the high flux in the microbeam, further investigations will focus on the influence of deposition parameters (rate, sample temperature, sputter pressure) on the structural build-up as well co-sputter deposition of hybrid materials.

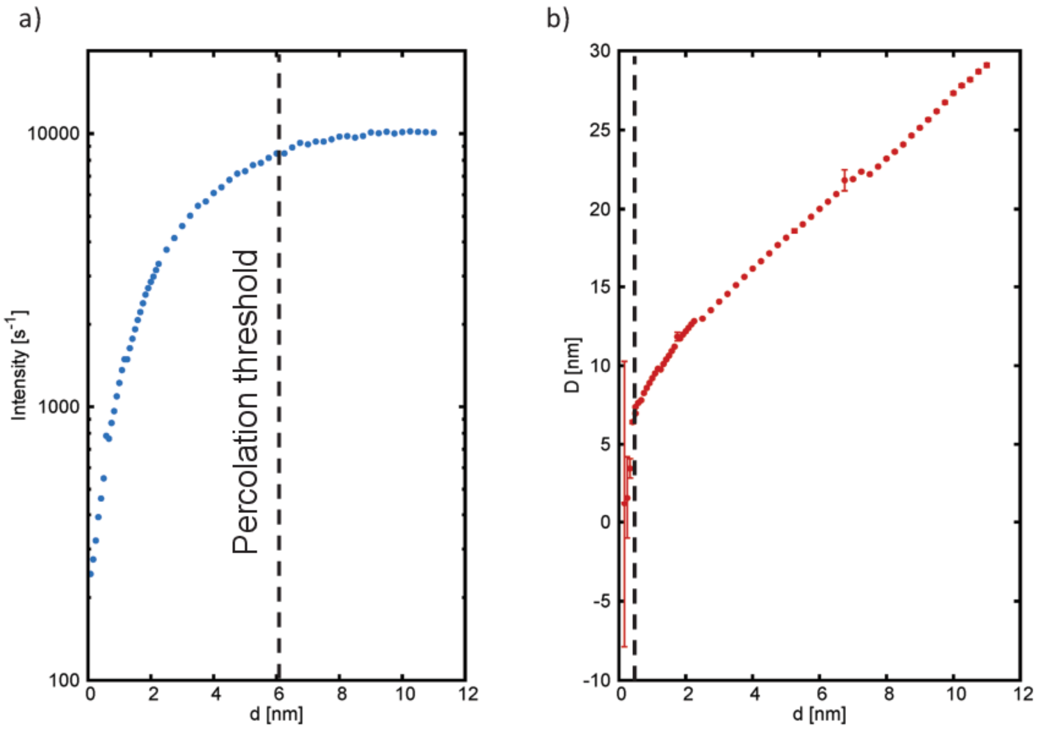

Figure 1: a) Intensity evolution at α_f =0.41° near the gold (Au)-Yoneda peak as a function of sputter deposited Au mass thickness d. The dashed lines marks the nominal percolation threshold. b) Evolution of the most-prominent in-plane lenght D as a function of d. The dashed lines show a first transition point at around d=0.6nm

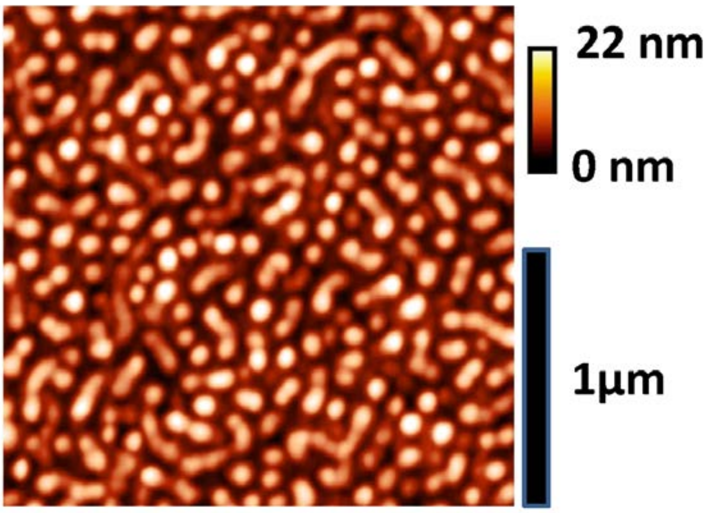

Figure 2: AFM topography image of 11nm Au on top a diblock copolymer film.

References

[1] F. Faupel, et al., Adv. Eng. Mater. **12**, 1177 (2010)
[2] G. Kaune et al., ACS Appl.Mater. Interfaces **1**, 353 (2009)
[3] H. Greve, et al., Appl. Phys. Lett. **88**, 123103 (2006)
[4] S. V. Roth et al.,, J. Phys. Cond. Matter **23**, 254208 (2011)
[5] E. Metwalli et al., Langmuir **24**, 4265 (2008)
[6] A. Buffet et al., Langmuir 27, 343-346 (2011)
[7] http://www.desy.de/ beneckeg/dpdak