Simultaneous Investigation of Nanomechanical Cantilever Sensors by Phase Shifting Interferometry and µ-focused x-rays

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Nanomechanical cantilever sensors (Figure 1C) are a powerful tool for measuring small forces in thin films[1]. To do so cantilevers a single sidedly coated with material of interest. From the resulting bending of the cantilever the stress inside the coating can be determine. Due to lack of a solid support, obtaining structural information of the coating layer is not possible with most standard methods, e.g. scanning probe microscopy. X-ray scattering methods on the other hand do not require a solid support and are therefore an ideal method to obtain structural information. µ-Focus options like available at BW4, provide a small enough beam size to individually address single cantilevers[2].

To directly relate stress and structural changes, simultaneous measurements of the curvature [3] and x-ray scattering is performed. Therefore a phase shifting interferometer (PSI) is installed in BW4. (Figure 1A). The use of a hexapod allows us to freely define the pivot point, enabling us to precisely align each individual cantilever (Figure 1D).

As a proof of principle experiment a cantilever coated with poly(methyl methacrylate) brushes was investigated. The sample cell was heated from room temperature to 50°C by jumps of 10°C. X-ray reflectivity measurements were performed after each jump in temperature. At room temperature and at the final temperature of 50°C a GISAXS image was recorded. Throughout the x-ray measurements the curvature of the cantilevers was recorded.

As can be seen in Figure 2 the polymer-coated cantilever has a negative curvature value which increasing at each temperature jump. The uncoated reference cantilever on the other hand stays constant during the entire experiment.

![Figure 1: (A) Experimental setup consisting of the Phase Shifting Interferometer mounted on top of a hexapod for alignment.(B) Sample cell with nanomechanical cantilever sensor. (C) SEM image of the NCS. (D) Profile obtained for alignment.](Image)

![Figure 2: Curvature values vs time recorded during x-ray scattering measurements](Image)
Since no reflectivity option is available at BW4 the 2D MARCCD detector was utilized. From the obtained x-ray reflectivity curves the film thickness could be calculated at all temperatures. The resulting film thickness of $d = 51$ nm is in good agreement with the expected value calculated from the molecular weight of the polymer. The obtained GISAXS image (Figure 3) show a yoneda and specular (blocked by beamstop) as expected for a polymerfilm on top of a silica surfaces.

We conclude that with the presented setup simultaneous investigation of nanomechanical cantilever sensors by phase shifting interferometry and $\mu$-focused x-rays is feasible. The conducted x-ray reflectivity experiments allow us to determine the film thickness of the coating, while the GISAXS images yield additional structural information in the lateral direction. By comparing this to the results of the bending experiments the setup allows a direct correlation of stress and structure.

![Figure 3: (Left) 2D-XRR images. (Middle) Obtained XRR curves at different temperatures. (Right) GISAXS images of the PMMA brush coating on top of the cantilever](image)

**References**