In-situ observation of facet formation in M-plane $\alpha$-Al$_2$O$_3$ during high-temperature annealing

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The crystallographic M-plane has the highest surface energy of all crystallographic planes present in $\alpha$-Al$_2$O$_3$. At high temperatures, a surface cut parallel to the M-plane will therefore reconstruct into facets composed of the lower energy S-plane and R-plane surfaces. These enclose angles of $17^\circ$ and $32^\circ$ respectively, with the initial surface. Thus, facettet $\alpha$-Al$_2$O$_3$ substrates can be produced by high-temperature annealing of M-plane $\alpha$-Al$_2$O$_3$ wafers in air [1].

In order to adapt the facetted surface morphology for further purposes of the substrate, it is highly desirable to be able to control height and width of the facets via the annealing conditions. At the beamline BW4 (DORIS III) we have therefore conducted an in-situ Grazing Incidence Small Angle X-ray Scattering (GISAXS) experiment during high-temperature annealing of M-plane $\alpha$-Al$_2$O$_3$ wafers.

For this experiment, a tube furnace was mounted on a double tilt stage and the samples were centred inside the tube using a customized alumina holder. A MAR165 CCD camera was used as an area detector. GISAXS patterns were recorded at room temperature, during heating up to maximum temperatures of 1325°C to 1420°C, and during annealing at maximum temperature for 12h to 24h. The samples were oriented such that the facets form parallel to the incoming beam. We observed the formation of a periodic lateral structure with increasing period length, beginning at about 700°C. The symmetric scattering patterns correspond to symmetric islands nucleating and growing on the surface. The scattering patterns become asymmetric when these islands develop into asymmetric facets. The angles with respect to the $q_z$ axis of two distinct features in the scattering patterns (indicated by dotted lines) relate directly to the facet tilt angles with respect to the initial surface normal [2]. These angles are increasing continuously during annealing, finally reaching values of $(19\pm2)^\circ$ and $(33\pm2)^\circ$. Thus, the facet angles are determined by the duration of annealing and adaptable nanofacetted substrates can be prepared as versatile basis for the fabrication of highly ordered arrays of nanostructures [3].

References