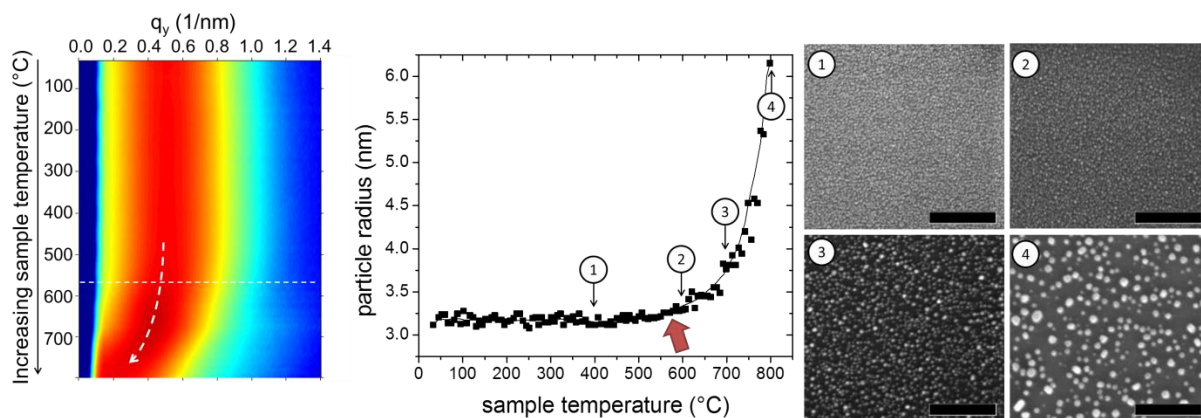


**Fig. 1:** Synchrotron-based *in situ* characterization of  $O_2$ - vs.  $N_2$  plasma-based ALD of Pt nanoparticles. For each process, three 2D GISAXS patterns are shown, corresponding to Pt loadings of *ca.* 60, 120 and 190 atoms/nm<sup>2</sup>, respectively. For the  $O_2$ -based ALD process, the main scattering peak shifts to lower  $q_y$ -values with increasing Pt loading, indicative of an increase in center-to-center distance between the Pt nanoparticles (and hence decrease in nanoparticle coverage). For the  $N_2$  plasma-based process, the position of the main scattering peak remains constant, pointing to a constant particle center-to-center distance. The sketches below the 2D GISAXS patterns are schematic representations of the nanoparticle morphology, i.e. shape, size and coverage, derived from the GISAXS data analysis. The results reveal a different nanoparticle growth behavior for the two ALD processes.



**Fig. 2:** Synchrotron-based *in situ* characterization of the thermal stability of ALD-grown Pt nanoparticles. Left: *In situ* data recorded during thermal annealing of Pt nanoparticles under 18%  $O_2$  in He: horizontal line profiles, taken along  $q_y$  at the Si Yoneda  $q_z$ -position, as a function of temperature. For temperatures above *ca.* 600°C, the position of the main scattering feature shifts to lower  $q_y$ -values due to coarsening of the nanoparticles. Middle: Evolution in particle radius during annealing, as obtained from the GISAXS analysis. The onset temperature for particle coarsening is indicated with the red arrow. The numbers correspond to the SEM images of *ex situ* samples on the right. Right: SEM images with 250 nm scale bar.