

In situ GISAXS during atomic layer deposition

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Atomic layer deposition (ALD) is a self-limited growth method that is characterized by alternating exposure of the growing film to chemical precursors, resulting in the sequential deposition of (sub)monolayers [1]. The key advantages of ALD are the atomic-level thickness control and the excellent conformality, even on complex 3D nanostructures. In recent years, there has been a significant increase in ALD-related research. However several fundamental questions remain unanswered. Firstly, the initial nucleation of the film is a key issue. In many ALD processes, the surface reactions during the initial cycles are clearly different from the behavior during growth on top of an existing film. Secondly, although ALD is in principle ideally suited for conformal coating of high aspect ratio features, one can wonder about its limits.

In this work, all depositions and measurements were performed in the UHV film growth facility, adapted for ALD, installed at beamline X21 of the National Synchrotron Light Source at Brookhaven National Laboratory. We utilized in situ GISAXS to monitor the evolution of film roughness during the initial stages of HfO₂ growth on planar Si and Ge substrates [2]. Figure 1 shows the difference roughness evolution, due to differences in starting surfaces. In addition, GISAXS was utilized to monitor the decrease in internal surface area upon ALD in a porous titania thin film [3]. Figure 2 shows that after ca. 40 cycles the pores of the film are no longer accessible.

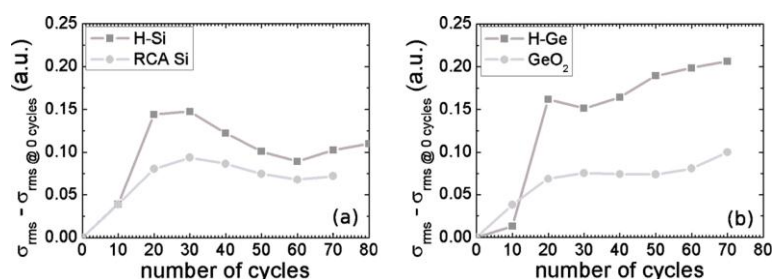


Figure 1: Roughness evolution based on the integrated GISAXS pattern on (a) H-terminated Si and RCA cleaned Si and on (b) H-terminated Ge and GeO₂.

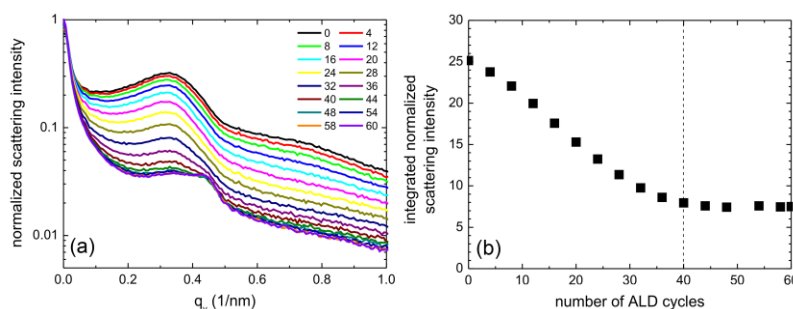


Figure 2: In situ GISAXS during ALD of TiO₂ on a mesoporous titania film. (a) Evolution of the GISAXS signal as measured every 4 ALD cycles ($q_z \sim 1 \text{ nm}^{-1}$). (b) Integrated intensity showing that after ca. 40 cycles the pores of the film are no longer accessible.

References

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- [3] J. Dendooven, J. Phys. Chem. C, 115, 6605–6610 (2011)