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IN SITU CHARACTERIZATION OF ATOMIC LAYER DEPOSITION IN MESOPOROUS THIN FILMS BY GRAZING INCIDENCE SMALL ANGLE X-RAY SCATTERING

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Mesoporous materials are of interest to a broad range of applications such as the development of optical, electronic and sensor devices, and catalytic surfaces. For many of these applications, the interior surface of the porous material needs to be functionalized. Atomic layer deposition (ALD) has become an attractive technique for tailoring porous materials because of its ability to produce conformal coatings. We present a synchrotron-based *in situ* characterization of TiO_2 ALD in mesoporous titania thin films by means of x-ray fluorescence (XRF) and grazing incidence small angle x-ray scattering (GISAXS). The experiments were carried out in a thin film growth facility installed at the NSLS at Brookhaven National Laboratory. The XRF technique was used to determine the amount of Ti atoms deposited, while the average electron density of the mesoporous thin film was probed with GISAXS via the so-called Yoneda peak, i.e. an enhancement of the scattered intensity at the exit angle which equals the critical angle of the scattering medium. The GISAXS patterns also provided information on the ordering and spacing of the pores. This work demonstrates that both XRF and GISAXS are suitable for *in situ* monitoring the filling of porous thin films by ALD.

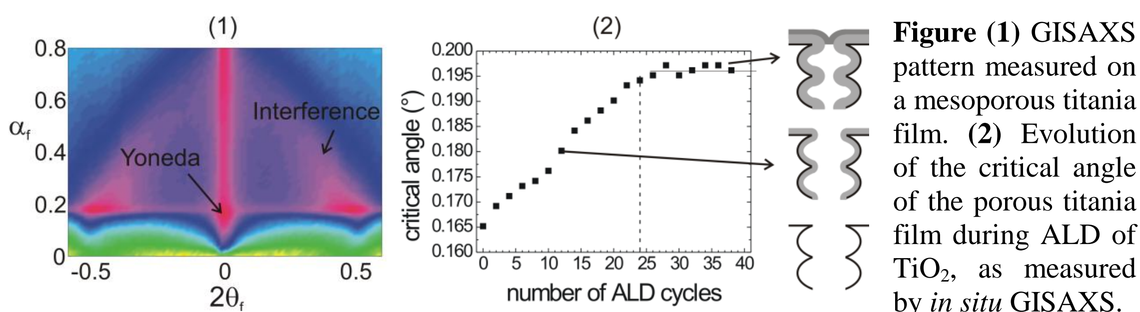

Figure (1) GISAXS pattern measured on a mesoporous titania film. **(2)** Evolution of the critical angle of the porous titania film during ALD of TiO_2, as measured by *in situ* GISAXS.