Characterization of ALD coatings in nanoporous thin films by ellipsometric porosimetry

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During the last decade, ellipsometric porosimetry (EP) has successfully been applied to determine the total porosity and pore size distribution of porous thin films, especially of porous low-k films.[1] EP uses spectroscopic ellipsometry (SE) to measure the change in optical properties and thickness of the porous film during adsorption and desorption of a solvent vapor. When the optical properties of the liquid adsorptive are known, the measured optical properties can directly be related to the volume fraction of the liquid in the film. As such, adsorption and desorption isotherms (adsorbed volume as a function of the pressure relative to the equilibrium pressure of the solvent vapor) can be obtained by EP. The porosity and pore size distribution of the porous film are then calculated from these isotherms.

Due to its conformal nature, ALD is an attractive technique for tailoring porous materials: both the pore size and the composition of the pore wall material can be tuned. In this work, EP is introduced as a promising technique to evaluate ALD layers deposited in nanoporous thin films.[2] By using appropriate models for the optical properties of the ALD coated porous film, the EP technique can provide information about the effect of the ALD coating on the porosity and the pore size of the porous film and, consequently, about the layer thickness of the ALD coating (Fig. 2). This will be illustrated by EP data obtained during ALD of TiO_2 and Al_2O_3 in mesoporous and microporous silica thin films. It will further be demonstrated that the EP technique is compatible with ALD and can therefore, in principle, directly be implemented onto the ALD growth chamber. Fig 1 illustrates how we adapted our ALD chamber equipped with *in situ* SE for EP measurements by connecting a container with toluene (the adsorptive), a pumping system and a dedicated pressure gauge to our system. These modifications allow us to study the evolution of the porosity and the pore size with progressing ALD growth in porous films without exposing the sample to air.

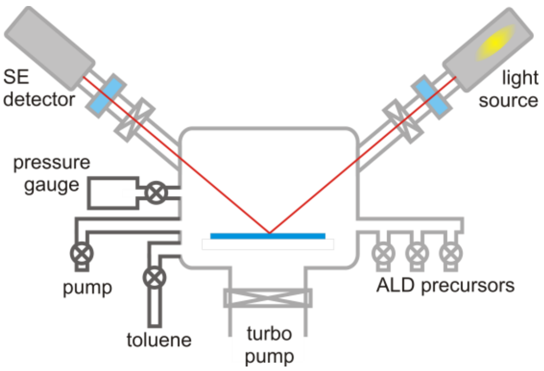

Fig. 1: Schematic showing the ALD chamber adapted for in situ EP.

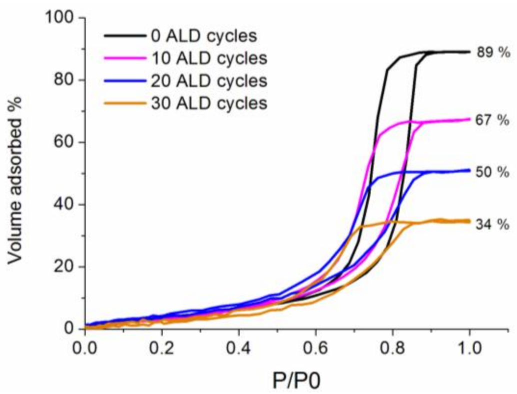

Fig. 2: Isotherms recorded by EP during ALD in a mesoporous silica film with an initial pore size of 15nm. The initial and remaining porosities are indicated.

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