

Spectroscopic ellipsometry for the determination of thickness and porosity of mesoporous metal oxide films

René Sachse^a, Andreas Hertwig^b, Vasile-Dan Hodoroaba^b, Ralph Kraehnert^a

^a Technical University Berlin, Berlin, Germany

^b Federal Institute for Materials Research and Testing, Berlin, Germany

Thin mesoporous metal oxide films are versatile and attractive candidates for several energy applications like photovoltaics, electrolysis or batteries. Due to their high surface area and ordered pore structure, mesoporous metal oxides demonstrate higher activities. The performance of the porous films is affected by properties like size and shape of the mesopores as well as the crystallinity of the framework. The exact determination and metrological evaluation of the complex morphology of thin mesoporous films requires a new analytical approach employing combined data from different analytical techniques.

In this contribution we present an evaluation procedure based on spectroscopic ellipsometry (SE) to analyze thin mesoporous iridium oxide films. Mesoporous IrO₂ films were prepared via dip-coating from a solution containing a triblock-copolymer and an iridium precursor in ethanol.^[1] Deposited films were calcined in air at temperatures between 300 and 600 °C. Their morphology was studied with SEM and electron probe microanalysis (EPMA)^[2] and correlated to SE using the Bruggeman effective medium approach (BEMA)^[3]. Figure 1a shows a top-view SEM image of mesoporous IrO₂ film calcined at 375 °C. The image reveals that the films exhibit a well-ordered mesopore structure. Figure 1b is a parity plot of film thicknesses determined by cross-section SEM versus SE of IrO₂ film samples prepared at different calcination temperatures. The porosity from the SE model is in good agreement to the porosity values obtained by EPMA (Fig. 1c).

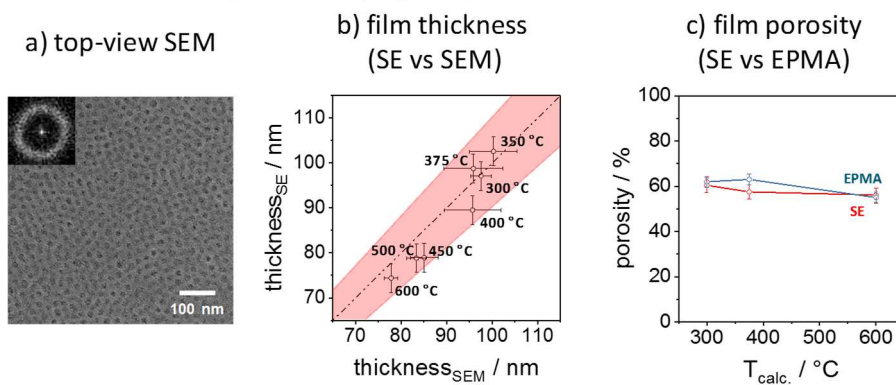


Figure 1: Characterization of mesoporous IrO_x thin films via scanning electron microscopy (SEM), spectroscopic ellipsometry (SE) and electron probe microanalysis (EPMA).

The contribution will assess in detail the novel approach to analyses the morphology and porosity of thin metal oxide films. Moreover, the facility of a multi-sample analysis, the sensitivity analysis as well as the mapping of samples will be discussed.

References

- [1] E. Ortel et al. *Chemistry of Materials* **2011** 23 (13), 3201-3209.
- [2] E. Ortel et al. *Analytical Chemistry* **2016** 88 (14), 7083-7090.
- [3] D.-M. Rosu et al. *Applied Surface Science* **2017** 421, 487-493.