

Diagnostic in TCOs CVD processes by IR pyrometry

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The control of deposition processes requires *in situ* analysis techniques and real time monitoring to determine the growth kinetics and more generally to detect any events occurring during the growth of the film. We show in this work, as representative example, that IR pyrometry, which is commonly used for the temperature measurement for instance in CVD processes, can be used as surface diagnostic tool to provide fruitful informations during the growth under atmospheric pressure of TiO₂ films on various opaque substrates.

Significant variations of the pyrometric signal were observed during the deposition of TiO₂ thin films by MOCVD due to interferences in the transparent growing film resulting from multi-reflections at the interfaces and scattering induced by the surface roughness. Such oscillations are known to complicate the temperature measurement in high temperature processes [1]. Here, we demonstrate that modeling of the time dependence of the IR pyrometric signal allows simultaneously the determination of the layer thickness, the growth rate, surface roughness and refractive index of the thin films under the growth conditions (Fig. 1).

Previously this optical diagnostic technique was used for *in situ* control of the nucleation of diamond thin films [2] and, more recently, we have successfully used it to analyze the oxidation of very thin Cr-based barriers [3]. The present results confirm that IR pyrometry can be used to analyze the growth of various transparent thin films such as TCOs layers grown on opaque substrates. Furthermore, it is a sensitive, simple and low-cost technique well adapted to control CVD processes operating under atmospheric pressure since low pressure is not required for the real time analysis. Perspectives and capabilities of the method are discussed.

[1] G. Llauro, D. Hernandez, F. Sibieude, J.M. Gineste, R. Verges and D. Antoine, *Appl. Surf. Sci.*, **135**, 91 (1998).

[2] S. Barrat, P. Pigeat, I. Diedueze, E. Bauer-Grosse and B. Weber, *Thin Solid Films*, **263**, 127 (1995).

[3] C. Gasquères, F. Maury and F. Ossola, *Chem. Vap. Deposition*, **9**, 34-39 (2003).

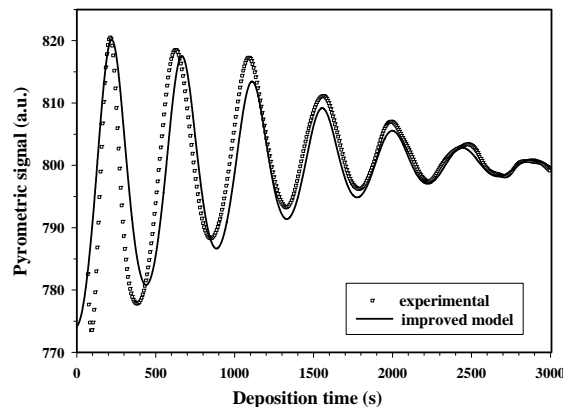


Fig. 1: Experimental (dotted line) and theoretical variations (full line) of the pyrometric signal with the deposition time for a TiO₂ film grown out at 500 °C on steel. The parameters provided by the model are: $G = 0.92$ nm/s; $n_{\text{film}} = 1.97$; $k_{\text{film}} = 0.0013$; $\sigma_{\text{film}}(\text{nm}) = 0.035 \cdot \text{time}(\text{s}) + \sigma_{\text{subst}}(40 \text{ nm})$; thickness = 2760 nm (SEM thickness = 2800 nm).