

## Electrochemical synthesis of oxide films

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Several methods have been described in the literature to synthesize oxide materials. Among them, electrochemical procedures have been used to produce valve metals oxides such as  $\text{ZrO}_2$ ,  $\text{TiO}_2$ ,  $\text{Nb}_2\text{O}_5$ , and also  $\text{Al}_2\text{O}_3$ . In this case, a metallic substrate is polarized and the metallic oxide film forms over the electrode. One important advantage is that the material is formed under room temperature, and, depending on the experimental conditions, in its crystalline form. Besides, the material is formed on locally extreme energy conditions but using smooth general experimental values, because the process occurs in the electrode/solution interface. As consequence, different phenomena which occurs at elevated temperature can also be observed at room conditions, such as crystallization, doping, and phase transformation. Besides, it is also possible to control the material morphology, obtaining films with homogenous distribution of nanopores or nanotubes. The controlled nanopore distribution in  $\text{Al}_2\text{O}_3$  films have been described since Masuda landmark work and can be used for many purposes. In our lab, we have used such material to build a filtration element for water containing pathogenic bacteria. The pore diameter, in this case, must be lower than 200 nm (0.001 the thickness of a human hair wire), and, as consequence, the water flux is low. Then, to increase it we have used  $\text{Al}_2\text{O}_3$  with wider pore diameter, but with deposited silver particles inside the pores. In both cases, we have obtained water free from any pathogenic agent. In a second work, we have synthesized partially stabilized cubic/tetragonal  $\text{ZrO}_2$  at room temperature. It is well described in the literature that, at room temperature,  $\text{ZrO}_2$  is in the monoclinic phase, which is converted to tetragonal around 1000 °C. Decreasing the temperature, there is a back transition to monoclinic phase which also leads to a mechanical failure of the material due to differences in the molar volume of both phases. As high temperatures are generally necessary for the material processing, to inhibit the back transition, it is added doping elements, such as Mg or Ca, to stabilize the tetragonal (or cubic) phases at room temperature. In any case, the high temperature processing is still necessary. It is well described in the literature, that during the galvanostatic electrochemical synthesis of valve metal oxides, occurs also a secondary phenomena called electrolytic breakdown. In the present case, during the electrochemical synthesis procedure, we have dissolved the doping elements in the solution. In the electrolytic breakdown region of the oxide growth, the oxide are locally dissolved and built again. During this process, the dopants are incorporated into the film leading to the tetragonal phase formation which is stable at room temperature.

In conclusion, electrochemical synthesis of metallic oxides open new promising route to nanostructured materials design.