Nanostructured Inorganic Thin Films prepared by Modified SILAR-method

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Successive ionic layer adsorption and reaction (SILAR) represents a promising wet chemical approach for preparation of chalcogenide and oxide thin films. In its simplest form, a substrate is alternately introduced in solutions containing respectively the cations and the anions of the target material. One disadvantage with conventional SILAR is that heat treatment between successive layer depositions is not provided.

We present here an automated set-up for parallel processing of 24 samples, including heating, drop-removal and online measurement of conductivity and light transmission. An extensive range of sulfide-, oxide-, phosphate- and carbonate thin films have been prepared by our modified SILAR method. The films were deposited on substrates of glass by using an automated high-throughput set-up developed in-house for the purpose. A commercial robotic arm is used to dip 24 substrates in parallel in well plates containing different solutions followed by heating in inert or reactive gas (Fig1). Typically, one 24-well plate is prepared with six different salt solutions in rows of four wells, containing the target materials cations. Another 24-well plate is prepared with solutions of four different compounds containing the anions for the target materials. Additional well-plates are filled with distilled water for washing. The set-up includes in-house made instrumentation for on-line measurement of conductivity and transmittance. X-ray diffraction (XRD), SEM, AFM and XPS techniques have been employed to investigate the structure and surface morphology of as-deposited films, for example comparing films prepared with different heat treatments during the deposition sequence. The method is particularly suitable for the preparation of films with thickness ranging from a few nm to 1 micrometer, the thickness depending both on dipping cycles and solution concentrations. Specifically, we have been searching for new transparent conductive materials, and it was found that for sulfides, in a narrow compositional range around Zn/Cu = 10, semi-transparent films with sheet resistivity lower than 100 Ohms/sq were obtained for samples heated to around 200 ºC (Figs. 2a and 2b). This amounts to about 100 times higher conductivity than for materials of similar composition previously reported. When we prepared samples with the same chemical composition without heat treatment, the sheet resistance was above 200 MOhms/sq. SEM-analysis indicated that the films were not uniform in early stages of the deposition-process. Elongated “islands” could be observed, which grows with increasing number of dipping cycles. However, after 90 dipping-cycles, the SEM analysis shows continuous and homogeneous films.

In conclusion, we have shown that the modified SILAR-method can be used to prepare thin films with improved properties, by the introduction of on-line heating in the course of the process.

Figures

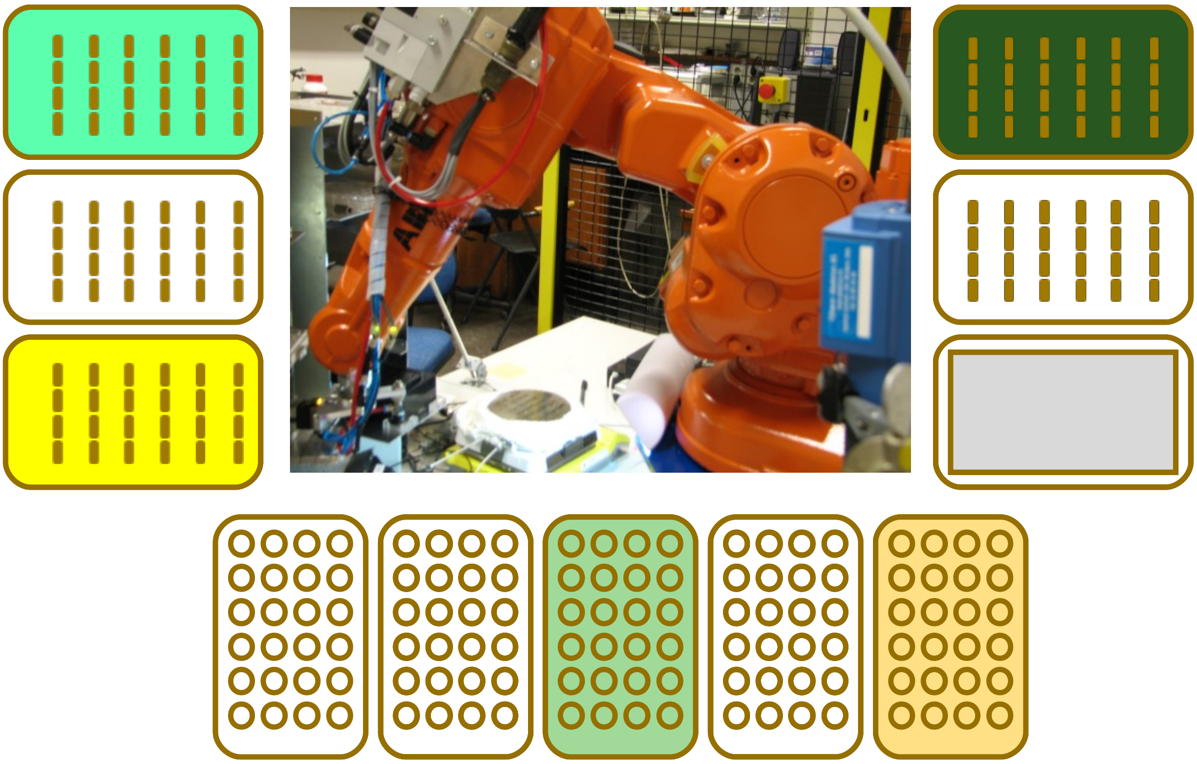

Figure 1. Robotic system used for SILAR. 24-well plates and 24-well format stations for treatment and characterizations shown schematically.

Figure 2. a) Highly conductive sulfide samples produced by modified SILAR. b) Sampleholder with 24 glasslides being withdrawn from heating station.