

Combi-Electrochemistry: Exploring the Phase Diagram of New Materials for Advanced Batteries

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Combinatorial techniques have been applied for the first time in the synthesis of anode materials for advanced Li-ion batteries. $\text{Mo}_{1-x}\text{Sn}_x$ is chosen as the system of study. A range of stoichiometries ($0 < x < 0.6$) was deposited by DC magnetron sputtering onto a copper substrate. Appropriate mask slots, through which linear and constant deposition profiles are achieved, were machined and placed opposite Mo and Sn targets separately. We have performed Thermo-gravimetric, Electron Dispersive Spectroscopy, X-ray Diffraction, ^{119}Sn Mössbauer and Electrochemical studies to thoroughly characterize the structure, composition, defect oxygen concentration and electrochemistry of this material. Conclusions are drawn as to how electrochemical activity is induced as Sn is substituted for Mo in the BCC Mo structure.

In June 1995, X-D. Xiang and co-workers¹ established a combinatorial approach to Materials Discovery that "combined thin film deposition and physical masking techniques for the parallel synthesis of spatially addressable libraries of solid-state materials." Combinatorial Materials Science (CMS) offers the researcher with a tool to accelerate the processes by which novel materials are discovered. The CMS technique is perfectly suited to the search for novel electrode materials for use in advanced Li-ion batteries.

Films of $\text{Mo}_{1-x}\text{Sn}_x$ were deposited using DC magnetron sputtering. Opposite the appropriate target, mask slots (Fig. 1) which produce linear and constant

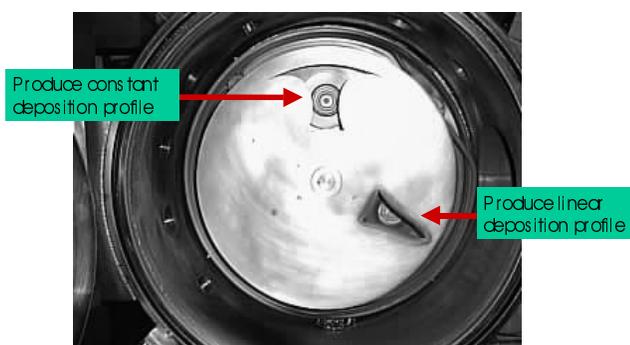


Figure 1. Experimental arrangement of the slots through which sputtered Mo and Sn fall onto the copper substrate (at left).

deposition profiles are placed. In order to deposit the range of compositions corresponding to $0 < x < 0.50$, Mo and Sn were sputtered onto a rotating substrate (5 rpm) in very thin (about 5 Å/pass) layers to ensure intimate mixing. The 2"-Mo target was placed opposite the constant deposition profile slot and the 2"-Sn target was placed opposite the linear profile slot.

EDS analysis demonstrate that the tin content spans the range $0 < x < 0.6$, consistent with that predicted by measurements of the sputter deposition rate (Fig. 2). Thus we have successfully produced the desired composition range.

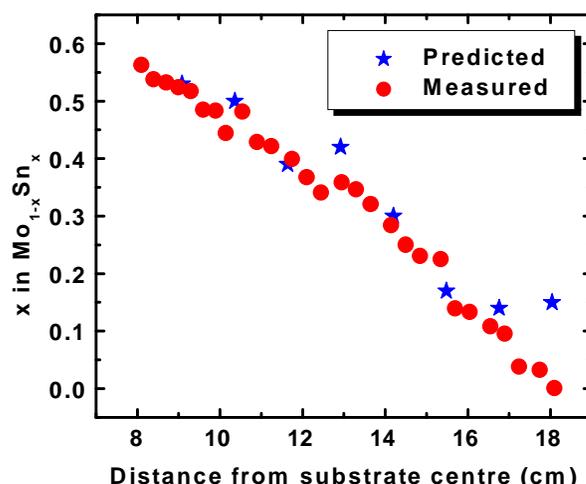


Figure 2. Composition of the films as determined by EDS, presented as a function of the distance from the substrate centre.

Figure 3 shows XRD of the films in a selected range of angles, with corresponding measurements of the peak position and width of the (110) Mo peak. The films show all the Bragg peaks of the Mo BCC structure and no Sn peaks.

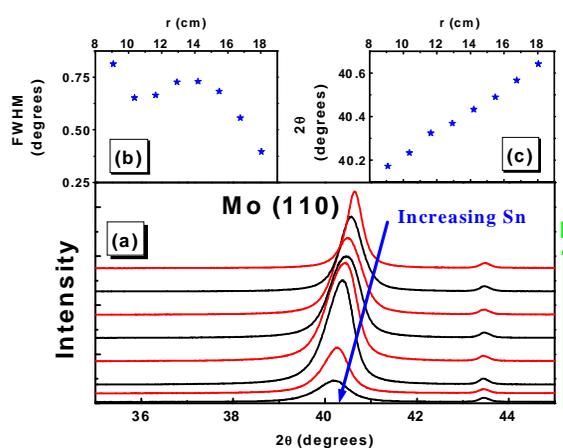


Figure 3. (a) XRD patterns, (b) FWHM and (c) scattering angle in the range of the (110) Mo peak.

With increasing Sn content, the Mo(110) peak position decreases, implying a lattice expansion with Sn incorporation in solid solution.

The electrochemical behaviour of the materials will be reported at this meeting and the onset of electrochemical activity in the films with Sn content will be discussed.

We acknowledge the financial support of NSERC, 3M and 3M Canada.

1. X. -D. Xiang et al. *Science* **268**, 1738-1740 (1995)