## Vanadium Oxide Nanoroll Electrode Arrays for Secondary Lithium Batteries

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The development of microelectromechanical systems (MEMS) has created the need to have new micropower sources with small areal footprints. Due to limitations in the amount of loading and diffusion kinetics, thin film batteries are not able to provide sufficient capacity for $mm^2$ footprint areas. In contrast, 3D electrode designs provide a way to obtain high energy and power per unit area. The present work addresses the central issue in 3D design: fabrication of electrode arrays.

Figure 1 shows the process flow for electrode array fabrication. We have previously reported the method of fabricating silicon molds containing arrays of holes.[1] The individual holes are 70 μm wide by 200 μm deep and the areal footprint of the electrode array is 10 $mm^2$.

Vanadium oxide nanorolls (VONR) were used as the active electrode material in these studies. These materials were prepared by a ligand-assisted templating method and ion-exchanged with $Na^+$ prior to use. A mixture of VONR with PVDF (10 wt%) and carbon black (10 wt%) was dispersed in a solvent such as propylene carbonate (0.01g/mL) by sonication and stirring. The mold was filled with the electrode material by centrifuging, taking care to first remove trapped air by sonication. The filled mold was then dried in an oven at 200°C to remove the solvent and promote binding of the electrode particles. Silver epoxy, serving as the current collector, was spread over the back-side of the mold and electrically connected each electrode post. The mold was released by immersion in a tetraethylammonium hydroxide (TEAOH) aqueous solution at 80°C for several hours. The TEAOH solution was deoxygenated by bubbling nitrogen gas to prevent the VONR from oxidizing. The released array was washed with D.I water and dried at 120°C under vacuum for 2 hours before testing.

Electrode arrays composed of approximately 900 VONR posts/10$mm^2$ were fabricated (Figure 2) with an aspect ratio ~3. The array structure is mechanically stable with no signs of collapsed or missing posts. The electrical and electrochemical properties of the arrays have been characterized. Cyclic voltammetry and galvanostatic charge/discharge tests establish that the VONR arrays exhibit reversible lithium intercalation/de-intercalation reactions whose discharge characteristics resemble those of standard electrodes (Figure 3). The array electrodes exhibit capacities on the order of 0.15 mAh for a footprint area of 10 $mm^2$.

Current research efforts are directed at increasing the aspect ratio of the posts to 10:1 and reducing the diameter of individual posts. Methods for interdigitating 3D anode/cathode structures will be presented.

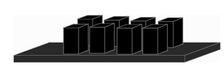
[1] G. Baure, C-W. Kwon, F. Chamram, C-J. Kim and B. Dunn, in *Micropower and Microdevices*, E. J. Brandon, A. Ryan, J. Harb and U. Ulrich, Editors, PV 2002-25, P. 36, The Electrochemical Society Proceedings Series, Pennington, NJ (2003).

Figure 1: Process Flow for Fabrication of Electrode Arrays


Step 1. Si mold micromachining

Step 2. Fill the mold with electrode materials

Step 3. Add silver epoxy



Step 4. Mold releasing using TEAOH

Figure 2: SEM of VONR Array

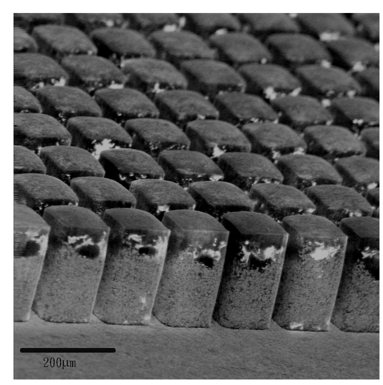



Figure 3: Discharge curves of the VONR array at 20μA rate