Application of combi-electrochemistry to the synthesis and characterization of electroactive materials for batteries and fuel cells

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In the first part of this work [1] we will explain the application of combinatorial methods to investigate the effects of Mn^{2+} and H_2SO_4 concentrations and anodic current density on the electrochemical synthesis and characterization of electrolytic manganese dioxide (EMD). The combinatorial method involved the rapid electrochemical deposition of EMD, from electrolytes with various Mn^{2+} and H_2SO_4 concentrations and at various anodic current densities onto individual Ti electrodes, in an overall array consisting of 64 electrodes [2]. These electrodes were then characterized in terms of average plating voltage, open circuit voltage and chronoamperometric simultaneous discharge (Fig.1) in 9M KOH. The applicability and benefit of the method was demonstrated by the characterization results, which suggested that the best performing EMD could be prepared using 32.50 gL^{-1} Mn^{2+}, 38.75 gL^{-1} H_2SO_4 and 62.5 Am^{-2} anodic current density. These values correspond to existing knowledge regarding the synthesis and electrochemical performance of EMD [3], and provide us with a starting point for future experimentation. An added benefit of the method apparent from this work was the considerable time saved during experimentation.

In the second part of this work, we will discuss the high throughput synthesis and electrochemical characterization of fuel-cell materials for the electrocatalytic oxygen-reduction and methanol oxidation. Automated synthesis techniques such as PVD or rapid electroplating are employed for the parallel generation of catalyst libraries onto an array of individually addressable electrodes with a broad range of composition space. The catalysts are subsequently subject to a hierarchy of screening levels in order to focus the search towards the most active composition: Primary high-throughput screening involves the simultaneous evaluation of the current-voltage-time behavior of each individual electrode material. The technique allows for the rapid identification of compositions most active for the electrocatalytic reaction, and has been applied to ternary and higher order compositions (Fig.2). In addition, important trends in the composition-activity relation become obvious which direct subsequent synthesis strategies towards activity optima. Different analytical characterization techniques are used directly on the electrode array to evaluate compositional and structural information for the most promising catalysts. The most active materials were scaled up by conventional synthesis techniques, subject to secondary screens such as RDE or tested directly in a fuel cell. We will show and discuss our results.

References:
1. M. Devenney, S. W. Donne and A. Gorer, JECS, 2003, in press
2. R. C. Haushalter, L. Matsiev, M. Devenney, E. Danielson and C. J. Warren; U.S. Patent 6187164 (2001).
3. R. P. Williams, R. Fredlein, G. Lawrance, D. Swinkels and C. Ward, Progress in Batteries and Battery Materials 13 (1993) 102.

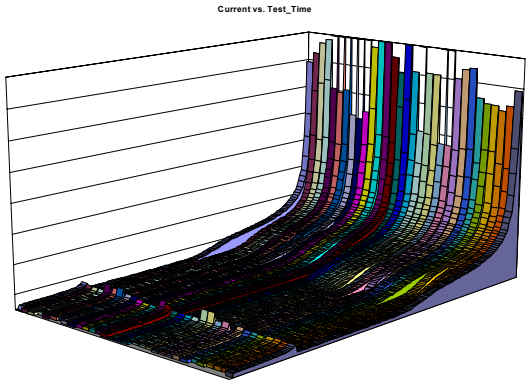

Fig.1. Characterization of an array of 64 EMD electrodes.

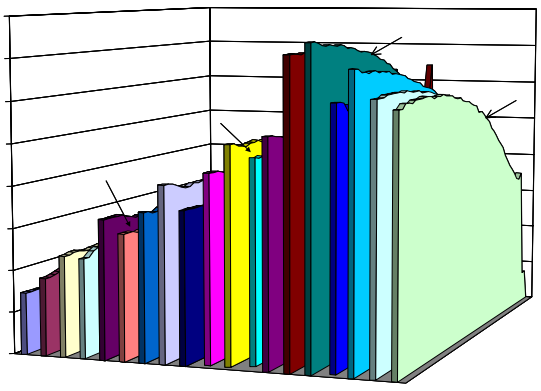

Fig.2. Characterization of an array of catalysts for methanol oxidation.