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### Fabrication of highly ordered porous nickel oxide anode materials and their electrochemical characteristics in lithium storage



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#### ABSTRACT

The structure and electrochemical properties of silicon microchannel plates (MCP)-supported NiO nanocomposites (NiO/Si-MCP) synthesized by silicon micromachining, electroless plating, and thermal annealing are investigated as anodes in lithium ion batteries. Galvanostatic charge and discharge results indicate that the NiO/Si-MCP is capable of delivering a higher capacity than the bare nickel-oxide film. At a 1 C current, the NiO/Si-MCP nanocomposite film shows an enormous first discharge capacity of about 3190 mA g<sup>-1</sup> and charge capacity of 1977 mA g<sup>-1</sup>. After 15 cycles, the NiO/Si-MCP nanocomposite retains a reversible capacity of 1531 mA g<sup>-1</sup> with 63.7% of the capacity maintained in the 2nd cycle. The lithium storage capacity is maintained at ~880 mA h g<sup>-1</sup> after 50 discharge/charge cycles and it is much larger than that of NiO and its composites. The enhanced electrochemical performance of the highly ordered three-dimensional materials is attributed to the synergistic effects offered by the silicon microchannel plates in the nickel oxide film subsequently facilitating electrolyte penetration, diffusion, and migration. The structure is promising anode materials in lithium-ion batteries.

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#### 1. Introduction

High-energy capacity lithium-ion batteries have attracted much interest due to their high gravimetric and volumetric charge capacity and excellent cycling properties suitable for electronics devices and electric-driven vehicles [1,2]. Much research is being varied out to develop new anode materials with improved electrochemical properties, and the ultimate objective is to develop economical and efficient lithium ion batteries. Graphite-based materials are considered leading candidates as anode materials because they offer large capacity, high electronic conductivity, and low working voltage with respect to lithium [3,4]. However, the reversible capacity of graphite anodes in Li-ion batteries is only 372 mA h g<sup>-1</sup> and incompatibility with microelectronics processing remains an obstacle to meet the power demand for future monolithic integration with Si-based microelectronic devices [5]. Si has the highest theoretical energy capacity (4200 mA h  $g^{-1}$ ) and low Li-insertion potential, but the large volume change (>30%) during Li-insertion and extraction results rapid capacity fading [6,7]. Several strategies have been proposed to accommodate the large volume change of Si. Recently, three-dimensional porous electrodes have attracted great interest due to their large surface area and short ion diffusion distance, increased contact areas, short Li diffusion path, and excellent accommodation of the strain incurred during Li insertion/ extraction [8,9].

Nanosized transition metal oxides (such as CoO, CuO, NiO, SnO, and SnO<sub>2</sub>) have recently been investigated as candidates in reversible anode materials because they have a higher capacity of lithium storage than traditional graphite anode [10–15], no intercalation/ de-intercalation sites in their crystal structures, and non-alloying with lithium during charge and discharge [10]. In particular, nickel oxide has a high theoretical capacity of 718 mA h g<sup>-1</sup> which is about 2 times than that of graphite and its density of 6.81 g cm<sup>-3</sup> is larger than that of graphite (2.268 g cm<sup>-3</sup>) enabling simple machining technology and lower cost. Various methods to synthesize nickel oxide as anode materials in Li-ion batteries have been reported, for instance, chemical liquid precipitation, sol–gel technique, hydrothermal, template, pulsed-laser ablation, etc. [16–20].

Highly ordered three-dimensional porous structures can improve the reversible capacity and rate capability of a NiO electrode and ordered meso-porous nickel oxide has been fabricated. NiO nanotubes with a specific capacitance have been synthesized using a precursor method [21]. Zhao et al. [22] have prepared an ordered mesoporous NiO film by heating an HI-e Ni(OH)<sub>2</sub> film

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Fig. 1. SEM images of the microstructure of the NiO/Si-MCP morphology: (a) Top-view and (b) cross-sectional image.

electrodeposited from the Brij 56-Ni(NO<sub>3</sub>)<sub>2</sub> liquid crystal template. NiO/Ni [23] nanocomposites have been prepared as electrode materials for Li-ion batteries and show excellent initial discharge specific capacities. However, the synthesis is tedious and the template needs to be removed after the synthesis. It is thus advantageous to combine microelectronics and silicon technologies to produce integrated anode materials for Li-ion batteries. In this paper, we report the synthesis of nickel oxide nanomaterials on a silicon microchannel plate (MCP) backbone as the anode in Li-ion batteries. The electrode has unique advantages such as threedimensional nanostructure, high specific surface area, low current density, polarization on unit surface area compared to a planar electrode, short Li<sup>+</sup> diffusion length, easy accessibility, and sufficient space to buffer the volume change of active materials during lithium insertion-extraction. The electrochemical properties of the NiO/Si-MCP nanocomposites are investigated by cyclic voltammetry (CV), galvanostatic charge/discharge. The results suggest that this ordered macro-porous structure is promising in Li-ion batteries.

#### 2. Experimental details

P-type  $\langle 100\rangle$  silicon wafers with resistivity of 2–8  $\Omega$  cm and thickness of 525  $\mu m$  were cleaned using the standard microelectronics process and the process to produce the MCP has been described [24,25]. In brief, a 300 nm thick SiO<sub>2</sub> layer was thermally grown to provide a mask on the wafer and 3.0  $\mu m \times 3.0$   $\mu m$  square regions aligned on the front side were defined by a standard photolithographic process. The windows were opened by a buffered hydrofluoric acid solution and nucleation centers for anodizing was formed at 85 °C in a tetramethylammonium hydroxide solution for a few minutes, followed by photo-assisted electrochemical etching resulting in the formation of three-dimensional ordered macroporous silicon.

The silicon MCP was cut into rectangular chips with dimensions of about  $1 \text{ cm} \times 1 \text{ cm}$  and nickel oxy-hydroxide was produced on the sidewall of the Si-MCP by electroplating. The plating solution consisted of sodium acetate, nickel sulfate, and sodium sulfate. NiSO<sub>4</sub> served as the Ni ion source, sodium acetate as



Fig. 2. (a) Typical XRD spectrum and (b) EDS spectrum of the NiO/Si-MCP composite anode.

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