



Research Letters

Fabrication of microcellular metal foams with sphere template electrodeposition

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Abstract

Microcellular metal foam (MMF) is a new class of metallic material with many potential applications such as solar energy storage. In this study we present a sphere template electrodeposition process for bulk MMF fabrication. To overcome ion diffusion limitation and uneven metal growth problems we explore the benefit of a pulse reverse electrodeposition technique. An experimental study was conducted on the effect of pulse voltage and frequency. It is shown that metal deposition uniformity can be significantly improved with high pulse frequency and amplitude. This study suggests that bulk MMFs could be fabricated with a microsphere template electrodeposition process.

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Microcellular metal foam (MMF) is a new class of metallic material with pore size in the range of 1–100 μm . Compared with conventional metal foams, MMFs are characterized by significantly smaller pore size and much larger surface area, in addition to desirable properties of both metal and porous material including low density and high thermal and electrical conductivities [1,2]. Metal foams can be close celled or open celled. Open-celled metal foams allow fluids to flow through and have attracted tremendous attention due to their clean energy applications, such as lithium-ion battery [3–5], super capacitor [6–8], and solar energy storage [9,10]. In these applications open-celled metal foams are used as scaffolds to load with active electrochemical or thermal material for high efficiency operation. It has been shown that the pore size of these foams is highly important to system performance [12]. As the pore size reduces, the volume of the loaded

active material become smaller, thus the distance for charge or heat transfer from the center of active material to the conducting scaffold becomes shorter. Therefore, a faster charging and discharging rate of the energy system can be achieved.

Open-celled metal foams have been fabricated by metal casting, electroplating, and electroless-plating [1,12–14]. These methods depend on availability of open-celled polymer foam templates, most of which have pore sizes on the level of several hundred micrometers. Recently close-packed polymer sphere templates have been used to fabricate open-celled metal foams through electrodeposition [15–19]. Well-defined micro- and even nano-cellular porous structure has been fabricated; however, the thickness of foam that has been achieved is only a few micrometers.

In this study we aim to fabricate MMFs for large-scale energy storage applications where bulk MMFs are needed. The sphere template electrodeposition process is used for better pore size control. When desired foam thickness increases, the electrodeposition process is limited by slow

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ion diffusion in the sphere templates. More importantly, the process suffers from uneven metal deposition due to potential template imperfection. To overcome these problems, we explore a pulse reverse electrodeposition technique that has been used to improve quality of surface electroplating processes [20–23]. We examine the benefit of this technique for MMF fabrication and study effects of electrical parameters including pulse voltage and frequency. The results demonstrate that bulk MMFs could be fabricated with pulse reverse electrodeposition of polymer microsphere templates for large-scale energy storage applications.

1. Experimental setup and procedure

A schematic of the experimental setup is shown in Figure 1. A DC power source (PK precision 1672) was superposed onto an amplified square wave AC signal (BK Precision 4012A and PYLE PTA1000) to provide the electrical power for electrodeposition. A separate DC source was needed in this case to maintain a constant average DC voltage, such that the average amount of metal deposition could be kept constant. The electrolyte solution consisted of nickel sulfate hexahydrate ($\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, 99% purity, Sigma Aldrich, Inc.), Boric acid (H_3BO_3 , 99.5% purity, Fisher Scientific, Inc.), and deionized water. The concentrations of $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ and H_3BO_3 were 0.3 g/mL and 0.03 g/mL, respectively. A small piece of nickel ($50 \times 15 \times 2$ mm, 99.9% purity) was used as the anode.

The templates used in this study were made with ethylene acrylic acid (EAA) microspheres (1–20 μm in diameter, Kobo, Inc.). EAA spheres were dispersed in deionized water to form a 2% (g/ml) suspension, which was then fed into a plastic mold with several layers of filter paper (2.5 μm pore size, Fisher Scientific, Inc.) at the bottom. The mold had an inner diameter of 26 mm. By controlling the suspension volume, templates were obtained at 3 mm thick. The templates were first air dried for 24 h at room temperature, and then sintered at 110 $^\circ\text{C}$ for 7 min in a vacuum oven (MTI EQ-DZF-6020). A conductive carbon glue layer was applied on one side of the template as the cathode.

The experiments were conducted with two levels of AC amplitude and frequency, respectively, following a full factorial experimental design. The amplitude levels were 2.6

and 4.9 V, and the frequency levels were 50 and 200 Hz. In all the cases, the average DC level was kept at 1.8 V. Duty cycle of the AC signal was kept at 50%. Two sets of experiments were conducted, with electrodeposition time of 12 and 20 h, respectively. After the deposition process, cross sections of the samples were observed under an optical microscope. Nickel deposited areas could be seen in gray, while undeposited EAA region in white. The area between the starting surface and nickel growth front was defined as the deposition layer, where nickel coverage varied from case to case. The fraction of nickel coverage over the entire surface area in the deposition layer was defined as deposition uniformity and used as a response variable for data analysis. The deposition uniformity was measured with image processing software Image J based on the brightness of each pixel.

Electrodeposited samples were treated at 450 $^\circ\text{C}$ for 1 h in a muffle furnace to remove EAA templates. The remaining nickel oxide foam was reduced at 650 $^\circ\text{C}$ for 6 h with a mixture of gas flow containing H_2 at 0.020 SLPM and Ar at 0.180 SLPM. The obtained nickel foams were characterized with scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDS).

2. Results and discussion

Figure 2 shows cross sectional images of four deposited samples obtained from a set of experiments with the deposition time of 12 h. Nickel growth fronts are indicated with dashed lines. It can be seen that nickel coverage in the deposited region is not uniform, with darker areas showing nickel presence. Both deposition voltage and frequency play a significant role in the deposition layer thickness and uniformity. On average, samples with a higher AC voltage and a higher frequency have a more uniform deposition layer. The thickness of deposition layer is inversely related to the uniformity of nickel deposition layer, since the total volume of nickel deposition is constant, determined by the average 1.8 VDC voltage.

The results on both growth thickness and uniformity for samples deposited for 12 and 20 h are summarized in Table 1. The samples deposited for 20 h show a consistent trend with those for 12 h. The difference between these two groups of samples is the deposition thickness, which appears to scale well with the deposition time. It is worth noting here that the thicknesses of the microcellular nickel foams are all greater than several hundreds of micrometers. The thickness can be further improved by increasing the electrodeposition time and the average DC voltage level.

Figure 3 shows SEM and EDS results from a nickel foam after removal of EAA via thermal treatment and reduction of oxidized nickel. The foam consists of around 90 atomic% nickel, the remaining being oxygen, carbon, and sodium, indicating a successful nickel foam recovery. Open celled MMF structure are obtained, with larger voids resulted from non-uniform deposition of nickel in the template.

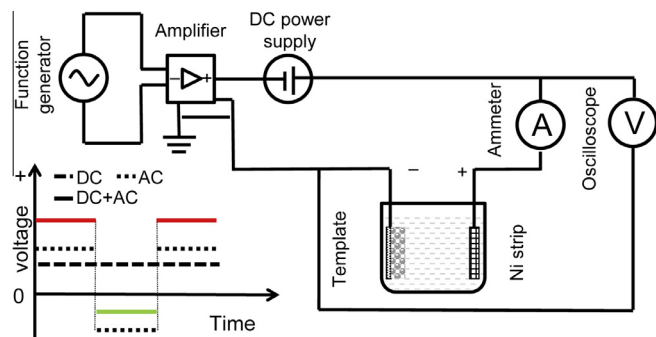


Figure 1. A schematic of the sphere-template electrodeposition process.

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