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Research Letter

# Fabrication of bulk graded microcellular nickel foams using combined electroless and electroplating of polymer sphere template

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#### Abstract

Graded microcellular nickel foams are a special category of nickel foams having regions with varying pore sizes. Potential applications for these functionally graded foams include heat-exchangers, energy absorption, and filtration. In this study, a new technique of fabricating bulk graded microcellular nickel foams employing a combined electroless and electro-deposition on a polymer template is presented. The polymer template is fabricated using a vacuum extraction technique. Electroless plating ensures initial nickel deposition on the polymer template and subsequent electroplating is used to control the density of the nickel deposits. The resulting foams have average porosity of around 94%.

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# 1. Introduction

Microcellular nickel foams have gained significant attention in the recent past because of their lightweight structure combined with good mechanical and electrical properties that result in numerous scientific and industrial applications [1–4]. The applications include filtration, vibration absorption, thermal management, batteries, mobile electronics and also as a substrate for growing graphene [5–10]. Functionally graded microcellular nickel foams are a special category which have pores of varying size across the thickness with potential applications such as heat exchangers, energy absorption, flow diffusion and filtration [11–13]. The majority of current techniques to fabricate microcellular metal foams use a polymer template because it offers direct control over resulting pore size and porosity [6,14]. The polymer template technique has been adapted to fabricate regular and hierarchal foams also with pore size in the micron and nanometer range. However the current approaches result in foams with non-uniform pore size distribution and are only a few microns thick. [11,15–22].

In this study we aim to fabricate a functionally graded bulk microcellular nickel foam with alternating layers of  $40 \ \mu\text{m}-20 \ \mu\text{m}-40 \ \mu\text{m}$  sized uniform pores with thickness on the order of a few millimeters which to the author's best knowledge has not been reported before. Initially a polymer sphere template is fabricated from highly uniform polymer spheres using a vacuum extraction technique. Subsequently, metal deposition on the polymer template is accomplished via combined electro- and electroless plating to achieve faster deposition rates. The resulting nickel foams have average porosity of 94%.

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# 2. Methodology of fabrication

#### 2.1. Materials

The fabrication of polymer template with uniform sized features is crucial because of its replication on the resulting nickel foams. The main requisite was availability of uniformly sized thermoplastic polymer microspheres with a low cost that would facilitate feasibility of this process on a bulk scale. Polystyrene spheres, with the commercial name DYNOSEEDS<sup>™</sup> TS (MicroBeads<sup>™</sup>, Norway), were chosen to fabricate the graded polymer templates. Two batches of uniform shaped and monosized polymer spheres having an average diameter of 20 µm and 40 µm respectively were procured.

# 2.2. Fabrication of polymer templates

The polymer sphere template was fabricated as per the schematic shown in Fig. 1. Suspensions of 20 and 40 µm Dynoseeds TS polystyrene spheres (0.04 g/ml) in deionized water were stirred separately at 800 rpm for 45 min (IKA RW-20) for effective dispersion of spheres. The polymer sphere suspensions were deposited into a metal preset mold placed above three layers of filter paper (20 µm pore size, Whatman<sup>™</sup>) in the upper section of a vacuum flask as shown in Fig. 1(b). Initially 10 ml of the 40 µm suspension was deposited followed by 10 ml each of the 20 µm and 40 µm sphere suspensions respectively. Water was extracted from the solution using a rotary vacuum pump (Model 6912, FJC Inc.). After the water was extracted completely, the mold was removed from the vacuum flask and a ram (forming interference fit with the mold) was used to eject the polymer template from the mold as shown in Fig. 1(c). The template thus obtained was left at room temperature for 12 h for the residual water to evaporate. The dry template was then heated in a vacuum oven (DZF 6020, MTI Corp.) at 150 °C for different time durations of either 13, 15 or 17 min for the spheres to attach to each other and form a stable template (sintering process) for the nickel deposition process.

### 2.3. Electroless and electro-plating

Electroless plating is a chemical based technique for depositing metal and the major challenge associated with it is generating the flow of the solution into the small pore sized polymer template. In order to overcome this issue, ethanol based plating solutions were used as detailed in Sundarram et al. [23]. Electroless plating deposits only a thin layer of metal on the polymer template. Hence, electroplating was used to deposit additional nickel on the polymer template. A nickel based conductive glue was applied on one surface of the template and a piece of metal wire was attached to it which acts as the cathode for plating. Cement glue was applied atop the nickel based glue, to ensure unidirectional nickel growth through the polymer template. This enabled the nickel deposition to start on the nickel based glue layer and eventually grow through the interstitial gaps in the polymer template. A strip of pure nickel (0.12 cm thick) was used as the anode. The two electrodes were immersed in a standard electroplating solution consisting of 20 g nickel sulfate hexa hydrate (NiSO<sub>4</sub>·6H<sub>2</sub>O) (Sigma Aldrich), 2 g boric acid (H<sub>3</sub>BO<sub>3</sub>) (Sigma Aldrich) and 50 ml deionized water. A voltage of 5 V with an average current of 10 mA was applied for a duration of 20 h with the electroplating solution being stirred occasionally.

# 2.4. Polymer burnout

The nickel deposited graded polymer template was heated in a muffle furnace (KSL 1100X, MTI Corp.) to decompose the polymer spheres resulting in a graded nickel foam. The sample was heated to 400  $^{\circ}$ C (decomposition temperature of polystyrene) in air for one hour and allowed to naturally cool down inside the furnace.

# 3. Characterization

The cross section and surfaces of the polymer template and nickel foam were observed using a Topcon ABT-60 scanning electron microscope (SEM). Image processing software Evex NanoAnalysis and Image J were used to

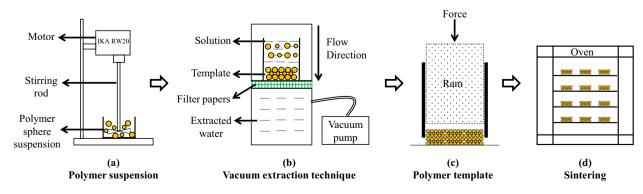


Fig. 1. Schematic of graded polymer template fabrication process.

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