



# Large-area metal foams with highly ordered sub-micrometer-scale pores for potential applications in energy areas

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## ABSTRACT

Nanoporous metallic foams with an exceptionally high specific surface area can be a perfect solution for advanced energy applications. There have been an increasing number of recent efforts to achieve nanoporous metallic foams, but the latest research has paid much attention to the processing and characterization of noble nanoporous metallic foams (Pt and Au) through the conventional dealloying technique. This study proposes a new and innovative method of processing non-noble nanoporous (sub-micrometer-scale) metallic foams: a technique that combines the conventional electroless plating and three-dimensional proximity-field nanopatterning. Copper and nickel foams with sub-micrometer-scale pores are processed and characterized in this study.

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## 1. Introduction

Thus far, the development of nanoporous structures with high specific surface area for use in energy or functional applications has been traditionally confined to nanoporous organic or inorganic materials. The fabrication of nanoporous metallic materials is considered difficult, probably because of the challenges associated with the fabrication of nanoporous metallic materials. Indeed, at the nanoscale they may suffer from poor stability, and poor oxidation and corrosion resistance. Despite these difficulties, sustained research efforts are being devoted to utilize the promising potentials of nanoporous metals in advanced functional applications, such as high-efficiency heat-exchanger substrates, catalysts, sensors, actuators, and microfluidic flow controllers [1–4]. More interestingly, nanoporous metallic electrodes can allow very efficient and rapid electrochemical reactions, owing to their high specific surface area and uniform distribution of pores [5]. For the same reason, they are also considered excellent substrate materials for catalysts [6]. Furthermore, nanoporous metallic structures are considered to exhibit better mechanical properties and long-term operational reliability than their polymer or ceramic counterparts. In particular, nanoporous metallic structures exhibit excellent specific strength and fracture

toughness, high thermal and electrical conductivities, and relatively high melting temperature.

Herein, we propose a novel method for fabricating nanoporous Cu and Ni foams with precisely controlled pores by a modified electroless plating technique using a proximity-field nanopatterned (PnP) polymer template. Unlike the conventional dealloying methods, the technique proposed in this study enables highly ordered distribution of the submicron pores in both Ni and Cu foams. Furthermore, the PnP technique allows the fabrication of large-area nanoporous polymer template, which is beneficial for the industrial-scale production of submicron-scale Ni and Cu foams. In addition, the PnP technique facilitates the direct fabrication of nanoporous Ni and Cu foams of several tens of microns in thickness, without requiring any additional material machining or shaping process. This is especially important from practical perspective, as the metallic foams must be prepared in the form of a thin film of thickness from tens to hundreds of microns in order to be used as electrodes in energy areas such as batteries, die-sensitized solar cells, or fuel cells, as schematically illustrated in Fig. 1.

## 2. Material and methods

In the typical process, a photopolymer (SU-8, Microchem, Newton, MA) layer of 10 μm in thickness was spin-coated onto a glass substrate. The photopolymer-coated glass substrate was subsequently heated at a temperature of 60–90 °C, to evaporate

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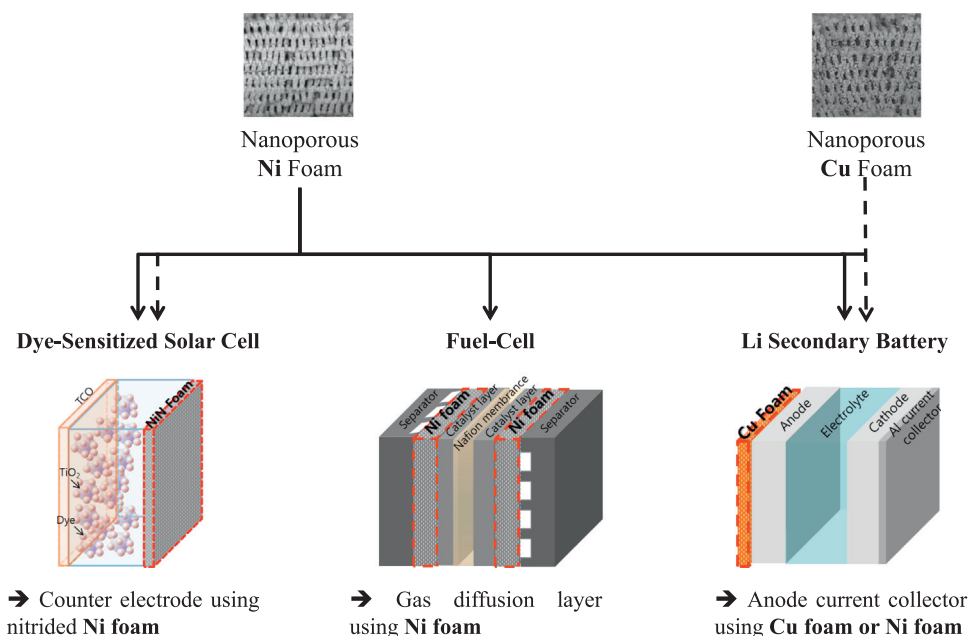


Fig. 1. Schematic illustration of potential applications in energy areas for Cu and Ni foams fabricated in this study.

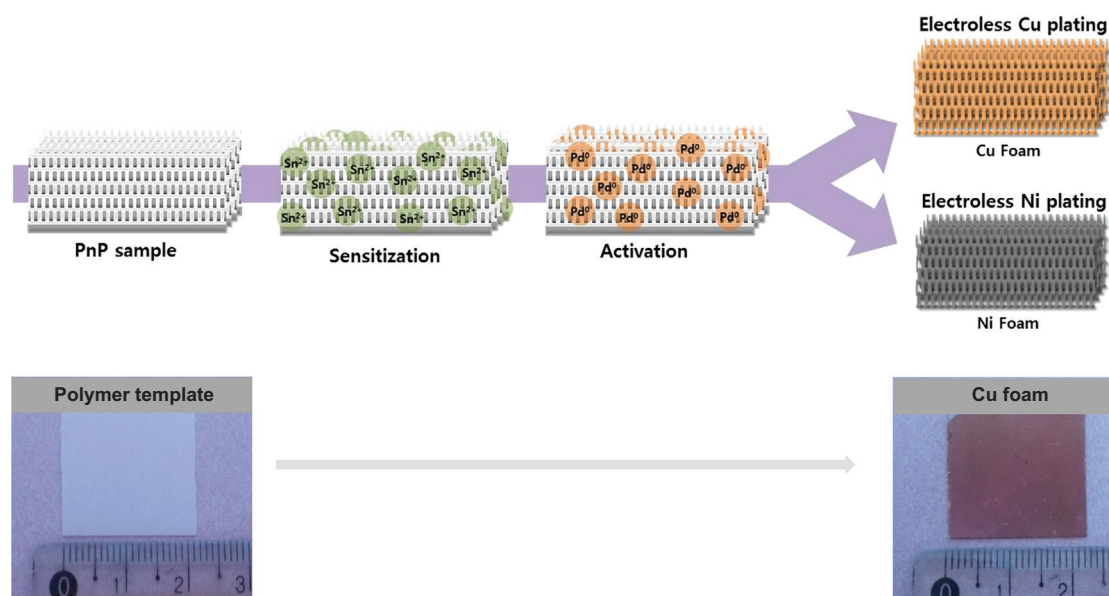


Fig. 2. Schematic illustration of the steps involved in the novel electroless plating method used for the fabrication of Cu and Ni foams with submicron pores. Shown in the micrographs are the pristine polymer template and the polymer template electroless-plated with Cu.

the solvent used in the process. Following that, a conformal transparent phase mask was placed on the polymer template and irradiated using an Nd:YAG microchip laser (wavelength: 355 nm, 500 mW) for selective exposure. A selective curing reaction of the polymer was carried out at a temperature of 50–60 °C. The polymer was then developed using a propylene glycol methyl ether acetate (PGMEA) solvent for 30 min. More details of this process are described elsewhere [6,7]. Prior to electroless plating of the metal, the non-conductive polymer template thus obtained needs to be ‘activated’ by an appropriate pre-treatment process (Fig. 2). The surface of the polymer template was made catalytically active to the metal by dipping the template in a pre-treatment solution composed of dilute tin chloride ( $\text{SnCl}_2$ ) and palladium chloride ( $\text{PdCl}_2$ ) solutions. In the pre-treatment process, the polymer template was dipped in 10.0 g/L of tin chloride

( $\text{SnCl}_2 \cdot \text{H}_2\text{O}$ ) and 40.0 ml/L of hydrochloric acid (HCl, 35%) at 30 °C for 3 min. Following that, the polymer template was dipped in 2.0 g/L of palladium chloride ( $\text{PdCl}_2$ ) and 16.3 ml/L of hydrochloric acid (HCl, 35%) at 40 °C for 5 min. The dipping of the polymer template in the pre-treatment solutions was carefully carried out under ultrasonic conditions. The electroless solution bath used for Cu plating was composed of 6.4 g/L of Cu sulfate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ), 70.0 g/L of ethylenediaminetetraacetic acid ( $\text{C}_{10}\text{H}_{16}\text{N}_2\text{O}_8$ ), 18.0 g/L of glyoxylic acid ( $\text{CHOCOOH}$ ), and 0.5 g/L of polyethylene glycol. The pre-treated nanoporous polymer template was immersed in a Cu plating bath (pH ~12.5) at a temperature of 70 °C. Similarly, the electroless solution bath for Ni plating was composed of 21.3 g/L of Ni sulfate ( $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ ), 25.3 g/L of sodium hypophosphite monohydrate ( $\text{NaPO}_2\text{H}_2$ ), 32.9 g/L of lactic acid ( $\text{C}_3\text{H}_6\text{O}_3$ ), and 2.2 g/L of propionic acid

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