

## Fabrication of independent nickel microstructures with anodizing of aluminum, laser irradiation, and electrodeposition

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**Abstract:** Independent microstructures made of Ni metal were fabricated by five sequential processes: porous anodic oxide film formation, pore sealing, laser irradiation, Ni electroplating, and removal of the aluminum substrate and anodic oxide films. Aluminum plates and rods were anodized in an oxalic acid solution to form porous type anodic oxide films, and then immersed in boiling distilled water for pore sealing. The anodized and pore-sealed specimens were irradiated with a pulsed neodymium-doped yttrium aluminum garnet (Nd-YAG) laser beam in a Ni plating solution to remove anodic oxide film locally by rotating and moving up / down with an XYZ $\theta$ -stage. Nickel was deposited at the area where film had been removed by cathodic polarization in the solution before removing the aluminum substrate and anodic oxide films in NaOH solutions. Cylindrical or plain network structures were fabricated successfully.

**Key words:** anodize; laser irradiation; nickel metal; electrodeposition

### 1 Introduction

Localized metal deposition on materials such as metals and semiconductors is very important for microfabrication techniques [1,2]. The fabrication processes of micrometer- and nanometer-scale metal structures mostly comprise the formation of a resist pattern, localized metal deposition, and etching of the substrate, and these processes are based on photolithography with ultraviolet light in the semiconductor industry or X-rays in LIGA (Lithographie, Galvanoformung und Abformung) processes [3-6]. Since photolithography involves many steps with toxic reagents, and it is seldom applied to materials with three-dimensional structures, such as stepped and curved surfaces, it is necessary to develop a new technique for fabricating three-dimensional structures without photolithography.

For direct patterning without photolithography on flat surfaces, many new methods have been reported with simple and more flexible processes to fabricate microstructures [7-13]. Three-dimensional microstructure fabrication has also been attempted for microelectronics and micromechanical components [14-20].

Recent investigation has demonstrated the possibility of fabrication of a fine metal pattern on insulating boards with maskless laser irradiation and localized

metal deposition [21-26]. In this technique, an aluminum specimen covered with anodic oxide film is irradiated with a pulsed neodymium-doped yttrium aluminum garnet (Nd-YAG) laser in an electro- or electro-less plating solution to remove the oxide film by laser ablation of the aluminum substrate. Then selective metal deposition is achieved at the area where film has been removed by the electro- or electro-less plating. Finally, the specimen is attached to an epoxy resin board before dissolving the aluminum substrate and the oxide film in alkaline solution. Fine pattern coils with 5  $\mu\text{m}$  wide Ni lines can be formed on the insulating board with the above processes. Fabrication of 30  $\mu\text{m}$  wide semicircular grooves has also been achieved using successive anodizing, laser irradiation, re-anodizing at the laser-irradiated area, and stripping of the oxide film [27]. In this investigation, the independent microstructures are fabricated by anodizing aluminum, laser irradiation, Ni electroplating and aluminum substrate and oxide film dissolution.

### 2 Experimental

#### 2.1 Specimens and pretreatment

Three types of aluminum were used as specimens in this investigation: i) 99.99% (mass fraction) aluminum plate, ii) 99.5% aluminum tube (1.6 mm inside diameter, 2.0 mm outside diameter), iii) 99.8% aluminum square rod (2.0 mm $\times$ 2.0 mm) after degreasing

and electroplishing. The pretreated specimens were anodized in 0.16 mol/L  $\text{H}_2\text{C}_2\text{O}_4$  solution at 293 K for 30 min with a constant current density of  $100 \text{ A}\cdot\text{m}^{-2}$  to form porous type oxide films, and then boiled in doubly distilled water for 15 min to seal the pores in the porous oxide film.

### 2.2 Fabrication of independent Ni structures with laser irradiation, electroplating and removal of the metal substrate

The anodized specimen was immersed in 0.31 mol/L  $\text{NiSO}_4$  / 0.40 mol/L  $\text{H}_3\text{BO}_3$  solution in a cell at 293 K with stirring and set at a 5 mm defocused position of the laser beam passed through a beam splitter, an iris diaphragm, a convex lens, and a quartz window. After setting, the specimens were irradiated with 1.0-10.0 mW of a pulsed Nd-YAG laser to remove the anodic oxide film from the aluminum substrate. During the laser irradiation, the specimen was moved at 40-100  $\mu\text{m/s}$  with a XYZ-stage, and rotated at 2.5-10.0 ( $^\circ$ )/s with a  $\theta$ -stage to remove the oxide film continuously from the aluminum substrate.

After laser irradiation, electroplating was immediately carried out in the  $\text{Ni}^{2+}$  solution-containing cell used for laser irradiation at 293 K for 15 min under cathodic polarization at  $-1.2 \text{ V}$  to deposit a Ni metal layer at the laser irradiated area. Finally, the specimen was immersed in 1.0-3.0 mol/L NaOH solution at room temperature to lift off the Ni metal layer by dissolution of the aluminum substrate and the oxide film. Details of the procedures of laser irradiation and Ni deposition have been described elsewhere [26,27].

### 3 Results and discussion

Figure 1 shows FE-SEM images of three-dimensional Ni microstructures that were obtained by anodizing, laser irradiation, Ni electroplating, and removal of the aluminum substrate and oxide films. Commercial aluminum tubes were used for fabricating all the structures in figures 1 except for that in figure 1(c), and the structure in figure 1(c) was fabricated using a square aluminum rod. Figure 1(a) shows a Ni micro-spring with 2 mm spring-diameter, 40  $\mu\text{m}$  metal line width, and 50  $\mu\text{m}$  gap intervals. The coil pitch of the micro-spring appears to be slightly irregular, not due to the irregular movement of the specimen in the local film-removal during laser irradiation, but due to deformation of the spring after removal of the aluminum substrate and the oxide film. Figure 1(b) shows a cylindrical Ni network microstructure with 2 mm diameter and 40  $\mu\text{m}$  metal line width. The network structure consists of rings connected by 8 pillars with 40  $\mu\text{m}$  gaps between the rings. The line intervals of the microstructure are extremely regular, because the pillars prevent deformation after removal of the metal substrate and oxide films. Figure 1(c) shows a prismatic Ni network microstructure of a prismatic spiral supported by 4 pillars. Nickel micro-rings with / without openings in the walls are shown in figures 1(d) and (e). The diameter of the micro-rings is 2 mm and the wall thickness is about 10  $\mu\text{m}$ . Figure 1(f) shows a Ni micro-pulley or micro-bellows with 2 mm diameter. The gap between the top and the bottom of the convex / concave structure on the wall is 80  $\mu\text{m}$ ,

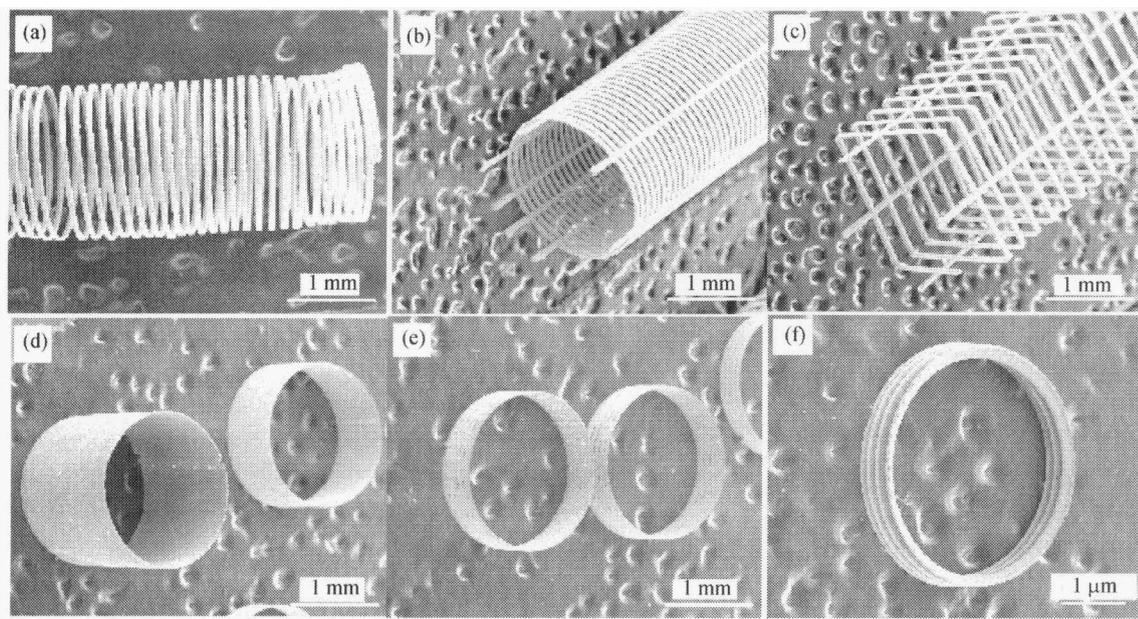
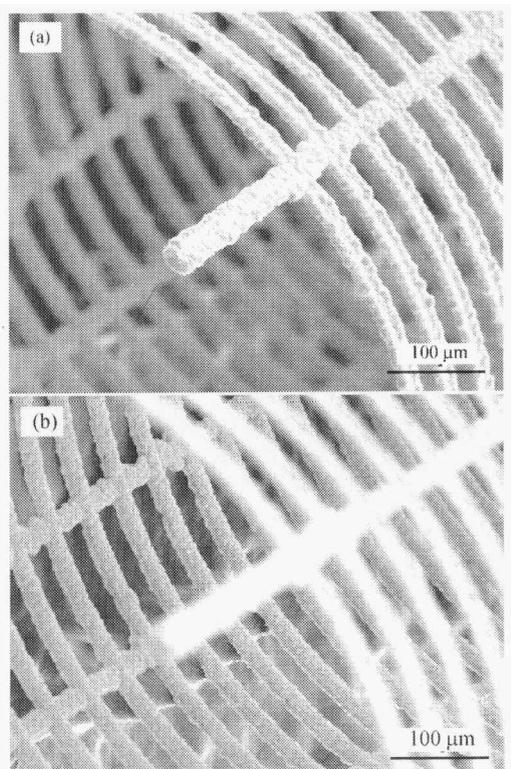


Figure 1 FE-SEM images of the three-dimensional Ni microstructures fabricated via anodizing, laser irradiation, electroplating, and aluminum substrate and oxide film dissolving, (a) micro-spring; (b) cylindrical network microstructure; (c) prismatic network microstructure; (d) micro-ring; (e) micro-ring with openings; and (f) micro-bellows.

produced by changing the laser power periodically during the laser irradiation.

**Figure 2** shows high magnification FE-SEM images of the Ni cylindrical microstructure (figure 1(b)), focusing on the outer surface (figure 2(a)) and the inner surface (figure 2(b)). The microstructure is composed of Ni grains of 10-20  $\mu\text{m}$ , and the outer surface is more irregular than the inner surface. The outer surface of each circle and pillar has a concave center part, while the inner surface is convex. This is due to the concave shape of the grooves produced by laser irradiation, and to the relatively low rate of Ni deposition at the center part of the groove.



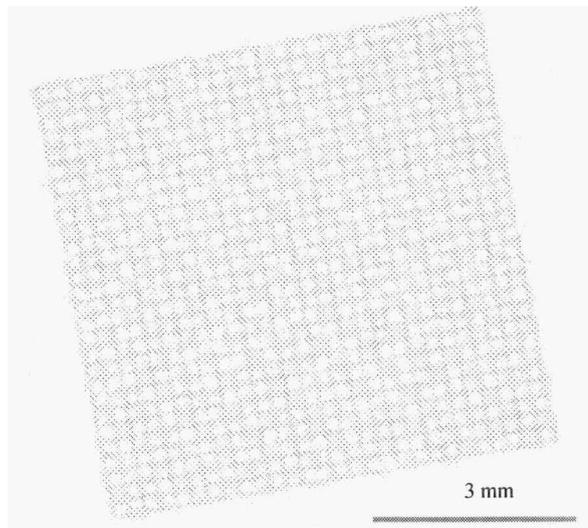
**Figure 2** High magnification FE-SEM images of the Ni cylindrical microstructure, focusing the outer surface (a) and the inner surface (b).

Figures 1 and 2 show that the film-removal by laser irradiation and Ni deposition at the area where film is removed is regular and uniform and results in network and ring structures without defects, and that the substrate and non-laser-irradiated oxides can be removed completely in NaOH solution without deformation of the structure of the nickel remaining undissolved.

**Figure 3** shows a nickel micromesh with 50  $\mu\text{m}$  wire width and 200  $\mu\text{m}$  gap, fabricated using a aluminum plate specimen. Here, a uniform network structure without defect can be obtained.

In summary, it must be emphasized that most of the independent microstructures obtained in the present investigation are difficult to fabricate by photolitho-

graphy or LIGA techniques, and that the more reliable less difficult technique described here can be applied to many fields, including MEMS, bio-system, and micro-reactors.



**Figure 3** Nickel micromesh with 50  $\mu\text{m}$  wire width and 200  $\mu\text{m}$  gap.

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