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Fabricating needle arrays with a gray-scale x-ray mask

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A gray-scale mask for x-ray lithography, made using microelectromechanical systems technology, offers a new approach to manufacturing needle arrays.

Microscopic needles long enough to penetrate skin but short enough to avoid touching nerves provide a method for painless injections. Research into a process for mass-producing microneedle arrays for drug delivery is leaping forward as the needles become a commodity. A painless needle can be less than 150μm long for injecting cosmetics, but for delivering medicine it must be 500μm–1mm, so that it can reach the subcutaneous tissue or even the underlying muscle. Short needles are easily created by silicon (Si) dry etching. Fabricating longer ones, however, is quite difficult. To solve this problem, we used x-ray lithography, which can handle a resist thicker than 1mm, combined with a gray-scale mask.

A microstructure fabrication method called LIGA (from the German: 'Lithographie,' 'Galvanik,' and 'Abformung')[1] already incorporates binary x-ray lithography. In a typical LIGA process, a resist master is produced by x-ray lithography, and a metallic mold is replicated from the master by electroforming. Then the pattern can be mass-produced using a molding technique. Because of its laserlike directional property, a synchrotron source is used to produce x-rays. LIGA has been successfully employed to make pillar structures with vertical sidewalls. However, because x-rays cannot be easily focused or reflected, it has not been successful in building 3D structures with sloped sidewalls. We proposed that the slopes needed to make pointed needles could be achieved by incorporating a gray-scale x-ray mask into the LIGA process.

A conventional binary x-ray mask consists of a membrane with a pattern made of an x-ray-absorbing heavy element such as gold. The x-rays penetrate the membrane but are blocked completely by the absorber. Fabrication of the mask involves photolithography and electroplating to create 4μm-high vertical absorber sidewalls. We developed a gray-scale mask with inclined-sidewall absorber sections so that we could create a resist structure by x-ray lithography that results in microstructures with sloping side walls. We used this mask with 3D LIGA to fabricate a needle array.[2]

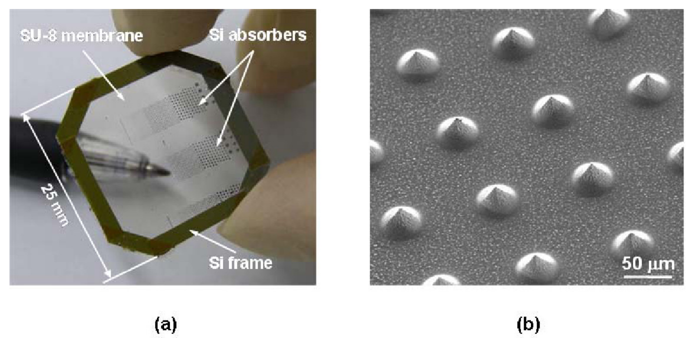

Figure 1. (a) Photograph of x-ray gray-scale mask with silicon (Si) absorbers on an x-ray-transparent membrane. (b) Scanning electron microscope (SEM) image of Si cones before backside etching of the substrate.

The gray-scale mask required several steps and employed microelectromechanical systems (MEMS) technology. Figure 1(a) shows an x-ray gray-scale mask with a Si absorber on an SU-8 membrane. A photoresist was first spin-coated onto an active Si layer atop a Si-on-insulator wafer, then irradiated and patterned through a mask by ultraviolet light. This was followed by a tapered-trench-etching step to form an array of Si cones, as shown in Figure 1(b). The structure was then coated with SU-8 resist followed by dry etching of the Si and silicon dioxide layers, resulting in a mask that contained 30μm-tall absorber structures 50μm in diameter.

Once the mask was made, we used it to create an array of microneedles. The process is shown in Figure 2. X-rays passed through the gray-scale mask and irradiated a 1mm-thick methylmethacrylate (PMMA) sheet. After development, platinum (Pt) was deposited on the PMMA to serve as a seed layer. The sheet was then immersed in a nickel (Ni)-plating bath and a voltage was applied between the Pt seed layer and an electrode board to

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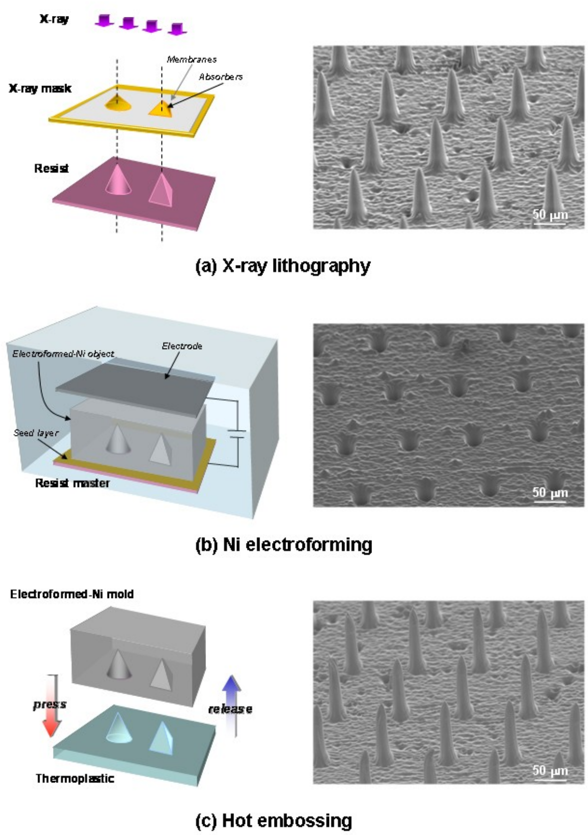

Figure 2. *Process flow of 3D LIGA with an x-ray gray-scale mask and the middle production structure in each process. (a) X-ray lithography and the PMMA resist structure, (b) electroforming and the surface of nickel (Ni) mold, and (c) hot embossing of polycarbonate, and the final printed pattern.*

electroform Ni onto the PMMA structure. The electroformed-Ni object was formed into a metallic mold. This mold was then used for hot embossing on a 0.5mm-thick polycarbonate sheet.

To be more specific about our process: The x-ray lithography experiments were performed on beamline BL-4 at the TERAS SR facility of the National Institute of Advanced Industrial Science and Technology. The dose energy of x-rays was 833mA·h. The exposed PMMA sheet was developed at room temperature over a period of 3 days. A sulfamic acid Ni was used in the Ni electroforming process. The polycarbonate sheet was imprinted using a heating temperature of 180°C, a cooling temperature of 130°C, a contact force of 2kN, and a 30min contact time. These values produced a needle array with features 50μm in diameter and 135μm long.

There are other methods of generating needle arrays using LIGA. However, these techniques require highly skilled rotation and scanning of the x-ray mask.[3] In contrast, our approach of using LIGA with the x-ray gray-scale mask consolidates the 3D and mask fabrication processes. Hence, producing 3D microstructures using an x-ray gray-scale mask becomes as simple as conventional x-ray lithography. Lengthening the exposure time results in longer needles. However, the replication process is not well suited to demolding the taller structures. For this reason, we plan to develop a molding technique more appropriate for high-aspect-ratio patterns.

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