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Fabrication of LIGA Masks for Microfluidic Analytical Systems

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Abstract—Preliminary results of fabrication and testing of LIGA mask samples for deep x-ray lithography in the spectral range of 3.5–13.5 keV are presented. The mask fabrication method is based on direct mask patterning with minimum element sizes of ≥10 μm by a synchrotron radiation x-ray microbeam. Such a method does not require an intermediate mask, which significantly simplifies fabrication and reduces the laboriousness and cost of LIGA masks.

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INTRODUCTION

Currently, the world of science is going through a technological revolution directed toward application of small and ultrasmall devices to study functions of biological macromolecules, genomes, cells, and cellular structures, as well as in clinical diagnostics and biochemical studies. This line of inquiry is based on the use of micro/nanofluidic systems (MNFS).

Micro/nanofluidic bioanalytical systems are integrated devices including modern high-technology and science intensive elements of electronics, micromechanics, nanotechnology, optics, hydraulics, gene and cellular engineering, and bioinformatics. The importance of developments in the field of micro/nanofluidic technology is emphasized in a series of thematic publications, e.g., [1].

The basic element of micro/nanofluidic systems is a glass or polymer plate with a multilevel system of channels, microreactors, valves, and pumps, operating with micro- or nanovolumes of liquid. An important advantage of the MNFS is the possibility of operating with individual cells at various stages of their development and with cellular systems. The MNFS allows the microscale implementation of the most important transport methods of modern analytical chemistry, flow–injection analysis, the use of advantages of laminar flows, optimization of the volume-to-surface ratio in microreactors, high-efficiency capillary electrophoresis. Radical miniaturization of sizes of experimental devices, achievable using micro/nanofluidic technologies, offers a transition to qualitatively new, low-cost, and high-efficiency methods for solving a wide range of fundamental and applied problems of molecular and cellular biology, biotechnology, and biomedicine. Operation of MNFS with micro- and nanovolumes of liquids allows a drastic (by orders of magnitude) decrease in the number of analyzed biological objects; accordingly, consumption of expensive reagents and cost of analyses are lowered. In this study, we consider a method for fabricating polymeric MNFSs with channel sizes of ~40×40 μm.

Deep x-ray lithography in resistive layers from tens of micrometers to one millimeter or thicker is the first stage of the LIGA technology aimed at mass fabrication of a wide range of microproducts. X-ray irradiation of such thick resist layers with sufficient uniformity of the absorbed dose over depth requires rather hard radiation with a photon energy of ≥10 keV. This imposes specific requirements on x-ray LIGA masks through which thick resist layers are exposed. To provide a sufficient contrast, topological patterns of such masks should be formed from a relatively thick x-ray absorbing material (usually 10–30 μm of gold or other element with a high atomic number). At the same, x-ray transparent substrates of such masks can be made relatively thick to provide the necessary strength.

In the conventional method for fabricating the LIGA mask, as a rule, an intermediate x-ray mask with a thin x-ray absorbing pattern (~1 μm of a heavy material) is fabricated by electron lithography on a thin x-ray transparent substrate (silicon, diamond) several micrometers thick [2–4]. Upon exposure through such a mask to a relatively soft x-ray region of the spectrum (1–2 keV),

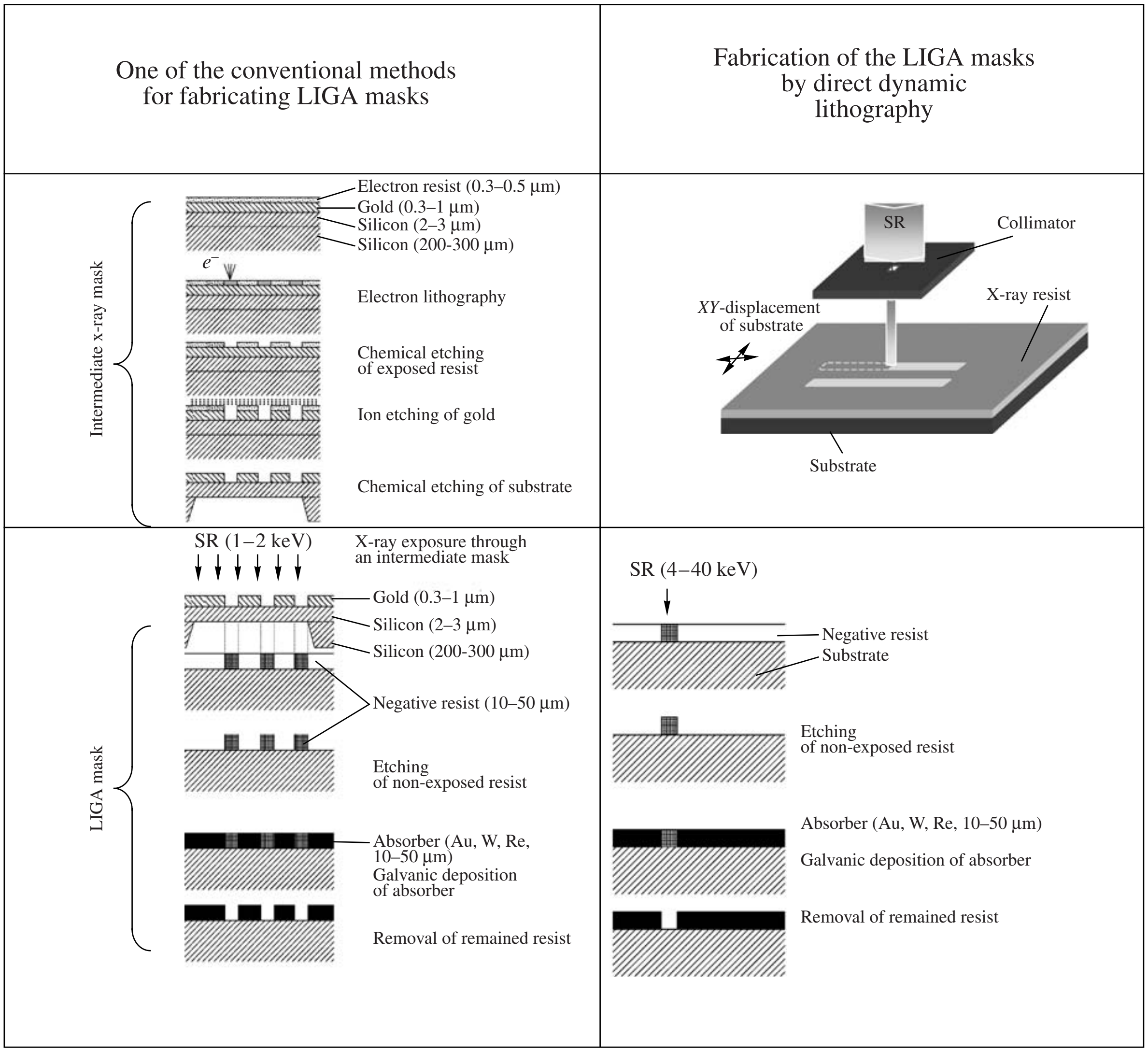

Fig. 1. Main stages of work cycles of x-ray mask fabrication.

a pattern is formed in a rather thick resist layer (10–30 μm), serving as a LIGA mask base. Such a process is multistage and laborious; fabricated LIGA mask are expensive, which significantly constrains widespread adoption of the LIGA technology for mass fabrication of various microproducts and microdevices in a variety of applications.

At the Institute of Nuclear Physics (Novosibirsk), the technology of direct fabrication of LIGA masks (without an intermediate mask) for subsequent fabrication of deep (to 1 mm) microstructures with minimum transverse sizes of elements (≥10 μm) was developed [5]. As a rule, it is impossible to fabricate such microstructures with a high aspect ratio by other methods.

PRINCIPLES OF LIGA MASK FABRICATION AND USED MATERIALS

For comparison, Fig. 1 schematically shows the technological stages of LIGA mask fabrication by the conventional method and direct x-ray lithography proposed by the authors. To remove the intermediate mask from the LIGA mask fabrication process, direct formation of the topological pattern of the mask was performed by controlled deep exposure of thick (tens of micrometers) resistive layers deposited onto a relatively thick x-ray transparent substrate to the x-ray synchrotron radiation (SR) microbeam. After removing the non-irradiated resist and galvanic deposition of the x-ray absorbing layer, the LIGA mask can be obtained.

The basic experimental setup is the LIGA station of the VEPP-3 storage ring (Institute of Nuclear Physics, Novosibirsk) [6]. The SR spectral region at the LIGA station is controlled by the parameters of the radiation source, i.e., the VEPP-3 storage ring ($E = 2$ GeV, $H_{rad} = 2$ kgf), and beryllium foils with a total thickness of 500 μm, placed in the SR extraction channel for vacuum safety. Additionally, to protect resistive layers against damage between technological processes, exposed samples were closed by an aluminum foil 10 μm thick. Under

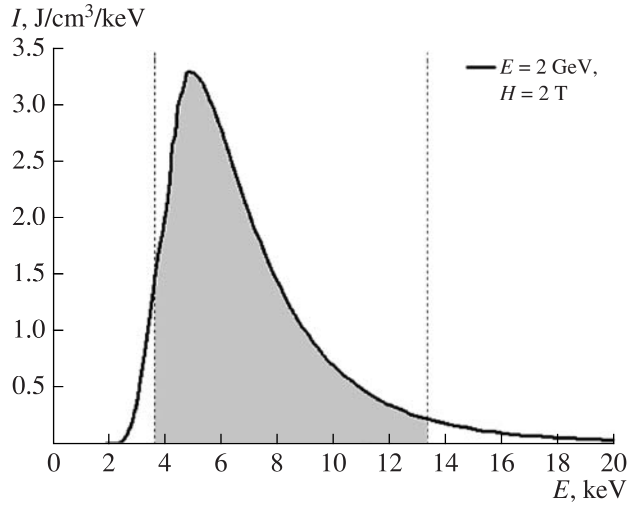

Fig. 2. Spectrum of radiation absorbed in the SU-8 resist under chosen conditions.

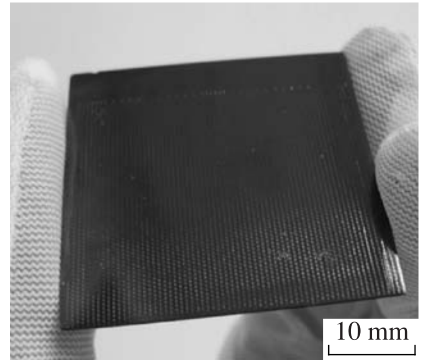

Fig. 3. Glass carbon plate with the test pattern of SU-8 resist.

these conditions, for polymeric resists such as plexiglas or SU-8, the spectrum of absorbed radiation is in the range of 3.5–13.5 keV (Fig. 2). In this case, the nonuniformity of the depth distribution of the absorbed dose in the resist in a layer to 100 μm thick does not exceed 30%.

Commercially available negative resist SU-8 was used as a resist, which allows deposition of layers from 1 to 200 μm and thicker for a single cycle. Such layers feature good mechanical, chemical, and thermal stability [5, 7]. As a substrate material for fabricating LIGA masks was used SU-900 glass carbon produced by the Research Institute of Structural Graphite Materials, Moscow [5].

RESULTS AND DISCUSSION

Ground glass carbon plates 500–700 μm thick were used in a series of test experiments performed. Before depositing the resist, substrates were treated in sulfuric acid followed by drying at a temperature of 200°C [5]. Additional deposition of conducting layers for electroplating was not performed. Figure 3 shows the glass carbon substrate with a test cellular microstructure formed by the SU-8 resist.

Samples with a deposited resist 20–40 μm thick were irradiated using their transverse displacement with respect to a collimated x-ray SR microbeam 40 × 40 μm in size [5]. The samples were moved by a two-coordinate scanner with a velocity of 50 μm/s and an accuracy of 1 μm. During multipass patterning, the needle-like x-ray beam formed a latent image of the required pattern microstructure immediately in the thick resist layer. Typical doses absorbed in unit volume during sample exposure were 40–60 J/cm^3.

X-ray absorbing gold coating 14 μm thick was galvanically deposited onto glass carbon substrate areas stripped of the resist by etching. The contrast of such a pattern for plexiglas exposure in the above spectral region was estimated as 35.

Thus, x-ray mask samples with the prescribed topology were obtained. The mask structure represents reaction chambers connected by channels 40 μm wide (Fig. 4). Using the masks fabricated at the LIGA station, more than thirty MNFS samples for biological studies were produced. MNFSs with channels 30–40 μm deep and 40 μm wide in plexiglas plates are shown in Fig. 5. The fabricated MNFS samples were developed for biological experiments with liquid microflows and individual *E. coli* cells at the Institute of Cytology and Genetics, Novosibirsk [8]. No changes in the masks after multiple uses under the SR beam were indicated.

The presented method makes it possible to obtain LIGA masks with good x-ray contrast, mechanical strength, and radiation resistance. However, some limitations should be noted. The formation of complex large-area patterns in the resist by the SR microbeam requires significant time. Moreover, fluorescence caused by hard photons with energies above 10 keV and a rather long range of secondary electrons can result in undesirable exposure of non-irradiated areas, which restricts the minimum possible size of elements to about 5 μm. Nevertheless, many practical problems

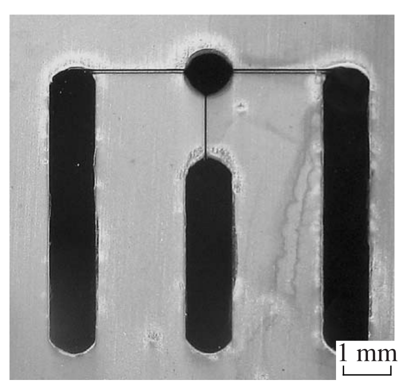

Fig. 4. Micrograph of the gold-plated MNFS x-ray mask.

Fig. 5. Samples of polymeric MNFSs fabricated using a finished mask.

require fabrication of microstructures tens of micrometers in size at a depth of tens and hundreds of micrometers. In such cases, the proposed method for fabricating LIGA masks could be optimal.

CONCLUSIONS

The feasibility of the complete work cycle for fabricating LIGA masks on glass carbon substrates using direct x-ray lithography was demonstrated. Test samples of LIGA masks with gold coating for deep x-ray lithography were fabricated.

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