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TECHNICAL PAPER

Fabrication of antiscatter grids and collimators for X-ray and gamma-ray imaging by lithography and electroforming

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Abstract Creatv MicroTech has developed unique fabrication techniques to make high precision, high-aspect-ratio metal microstructures to custom specifications. A lithography based fabrication method permits precise fabrication of various microstructures. Collimators and antiscatter grids with continuous, smooth, thin, parallel or focused septa have been fabricated using deep X-ray and optical lithography, combined with metal electroforming. Microfabrication of high-aspect-ratio structures, especially of relatively large areas, presents many challenges: specialized mask design and X-ray mask fabrication; resist preparation, optimal exposure parameters, post-exposure processing, electroforming, polishing, and final assembly. Here, we present microstructures of various designs that we fabricated and describe the challenges that had to be overcome.

1 Introduction

Creatv MicroTech Inc. (Creatv) has developed fabrication processes to make high quality, high-aspect-ratio metal microstructures to meet custom specifications. The fabrication method is based on lithography that allows precise fabrication of various microstructures. To fabricate the metal parts, we use different resist materials, X-ray or UV-lithography as appropriate, and electroforming or electroless plating of a variety of metals (e.g., Cu, Ni, Pb, Ag, and Au). The experience gained over several years allows us to move from custom design to a completed part in a relatively short period of time.

The collimator and anti-scatter grid are crucial elements in determining the sensitivity, resolution, and contrast of images in nuclear medicine and X-ray imaging. In order to maximize the transmission of primary radiation, septa walls must be as thin as possible and be parallel to the primary radiation. In order to eliminate the transmission of scattered radiation, the grid must be tall enough and made from suitable absorbing metal. To fabricate a metal grid structure, the resist mold consisting of channels is fabricated first. Microfabrication of deep channels, especially in a relatively large resist area, is very challenging and seldom reported. In this paper, we present a number of strategies to fabricate high-aspect-ratio grids and collimators of various designs.

2 Method

Lithography and electroforming is a unique method that allows manufacturing of microstructures with dimensions from several microns to several millimeters with high precision on a large area. The main steps of the optical lithography method are:

- Design and fabricate the optical mask on chromium-on-glass substrate.
- Transfer the pattern from the optical mask to a thick resist layer attached on conductive substrate.
- Develop the resist, such that the residual resist layer serves as a mold for metal electroforming.

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- Electroform the part using the developed resist/substrate.
- Lap and polish the electroformed part, after which the remaining resist mold is removed, resulting in a freestanding metal layer.
- Stacking, if necessary, of several layers to achieve the desired height is performed using precise alignment pins.

For X-ray lithography, an additional step of X-ray mask fabrication is required. We use both optical and X-ray lithography for the fabrication of custom microstructures. Both a positive polymethylmethacrylate (PMMA, Goodfellow, CQ-grade) and a negative SU-8 (MicroChem) tone photoresists have been used. X-ray exposures were performed at the wiggler beamline at the Center for Advanced Microstructures and Devices at Louisiana State University in Baton Rouge. Optical exposures are performed at the Center for Nanoscale Materials of Argonne National Laboratory using a Karl Suss MA 6 mask aligner, and at Creatv MicroTech using a custom UV-point source.

2.1 X-ray masks fabrication

X-ray masks of a good contrast are necessary to achieve precise pattern transfer. Hard X-ray masks for deep X-ray lithography at synchrotron radiation sources require a gold absorber of 20–100 μm thickness on a low-Z substrate. The thicker the gold absorber, the better mask contrast and the more accurate dimensional transfer of the mask pattern into the resist can be achieved. We use rigid graphite membrane as a substrate for mask fabrication. The properties of rigid graphite—low atomic number, strength, rigidity, good thermal and electrical conductivity, and low cost, make it attractive substrate for mask fabrication. The fabrication of graphite-based masks was first reported in 1998 (Coane et al. 1998), the authors used graphite substrate coated with a metal plating base. We simplified the process, and use bare graphite, for which the surface porosity provides very good adhesion of the resist (Makarova et al. 2003). We fabricated X-ray masks by UV lithography using photoresist SU-8 25 and 0.5-mm thick rigid graphite (Goodfellow) membrane.

UV-exposure was done using Carl Suss MA-6 mask aligner at the wavelength of 365 nm. The exposure time was optimized to achieve vertical wall slope, and was found to be 23 s (lamp intensity at 365 nm is 25 mW/cm^2) for the resist layer of about 90 μm thick. We use mask fabrication process described in Divan et al. 2004 as a guide, and process parameters were optimized for each particular design in order to control critical dimensions (CDs) with accuracy of ±1 μm, and to avoid adhesion failure. SU-8 resist is very sensitive to process parameters, especially to the post-exposure bake (PEB), which can vary for different pattern design such as open field or densely packed structures, long and narrow lines, etc. SU-8 internal stress due to a strong crosslinking reaction after resist exposure is one of the main problems in SU-8 resist processing. This problem causes pattern distortion, resists cracking and adhesion failure, and is more pronounced in the connected structures of large overall area. To minimize the stress, we use (1) stress barrier in the mask design, proposed by several authors (Chang and Kim 2000; Lin et al. 2002; Divan et al. 2004), (2) 30 min sample relaxation after exposure, and (3) conduct PEB with a very low heating and cooling rate. PEB is essential for achieving a good adhesion of the structure to the substrate, but the longer the bake, the larger the CDs in SU-8 will be. To compromise, we adjust the dimensions on the optical mask, usually CDs on the mask to be about 2–3 μm smaller than the target CD of the final structure.

Gold electroforming is done using Neutronex 309 (Cookson Electronics) plating solution at 45°C with the current density of 1 mA/cm^2. The process provides electroforming of uniform low stress gold deposit. The SU-8 resist has low X-ray absorption and good radiation stability (Simon et al. 2006); this allows leaving resist in place without losing the mask contrast. A photograph and an optical microscope image of one of the fabricated masks are shown in Fig. 1. The grid area is 60 × 60 mm, septa width is 25 μm, periodicity is 300 μm, and the thickness of gold absorber is 75 μm.

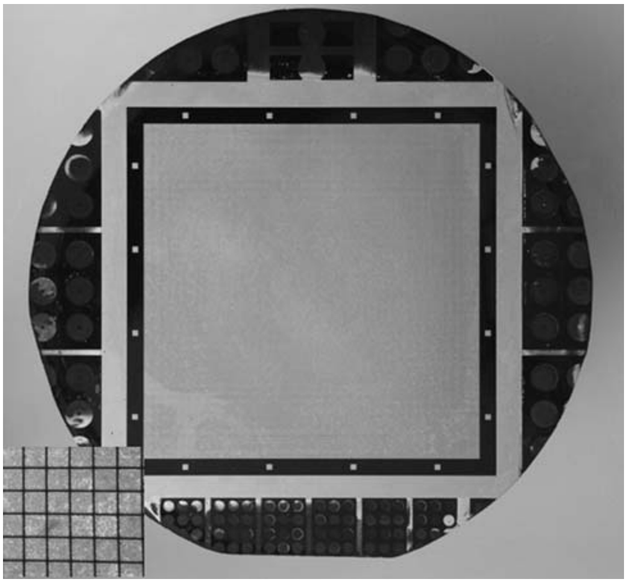

Fig. 1 Photograph and close-up image (insert) of the mask for grid fabrication

2.2 Resist and substrate preparation: PMMA versus SU-8

We use both a positive polymethylmethacrylate (PMMA, Goodfellow, CQ-grade), and a negative SU-8 (MicroChem) tone photoresists for the mold fabrication for metal electroforming.

- PMMA process is well established, reliable and allows fabrication of very high-aspect-ratio structures with high fidelity (Khan Malek et al. 1998, 2003; Pantenburg et al. 1998). We have fabricated grid structures up to 3 mm tall (aspect ratio over 100) using deep X-ray lithography and copper electroforming (Makarova et al. 2002, 2003, 2004a, b, c). The use of PMMA requires X-rays from the synchrotron. Unfortunately, beam lines are not readily available for many researchers, and relatively long exposure times make the process expensive.
- SU-8 negative resist developed by IBM with its high sensitivity to X-rays and excellent resolution is a new cheaper alternative to PMMA. The use of SU-8 instead of PMMA significantly reduces the cost of the fabrication process because of much shorter X-ray exposure time. UV-lithography of SU-8 is another lower cost alternative that simplifies the fabrication process, since there is no need for an X-ray mask, and exposure can be performed at the laboratory UV-source. Due to SU-8's low optical absorption in the near-UV range, relatively good exposure dose uniformity over the entire resist thickness can be achieved. Many research efforts have been done to obtain very-high-aspect ratio microstructures (Lorenz et al. 1998; Chang and Kim 2000; Ling et al. 2000; Lin et al. 2002; Chan-Park et al. 2004; Williams and Wang 2004; Makarova et al. 2004a,b, c; Becnel et al. 2005; Lawes 2005; Reznikova et al. 2005). However, SU-8's sensitivity to the process parameters and difficulties of its removal from the final metal structure limit its practical use for many grid and collimator applications.

When choosing substrate for mold fabrication, one also should take into account what electroforming process will be performed. PMMA swells at elevated temperatures, which leads to dimensional errors during electroforming (Ruzzu and Matthis 2002; Goods et al. 2003), and in some cases difficulty to electroplate the entire structure. For example, we were not able to electroplate gold into 25-μm wide and 0.5-mm deep channels in PMMA, because of the swelling, while we had no problem with gold electroforming into SU-8 molds of the same dimensions.

The substrate preparation was done on commercially available 1-mm thick rigid graphite sheets (Goodfellow). PMMA sheet of various thicknesses (from 1 to 3 mm) was solvent bonded to rigid graphite; the details of substrate preparation can be found elsewhere (Makarova et al. 2003). To achieve a thick layer of SU-8 resist in a single coating we use a resist self-planarization method (Lin et al. 2002). SU-8 50 (MicroChem) was loaded by weight onto the substrate placed on the leveled hot plate and allowed planarize. More details can be found in (Makarova et al. 2004a, b, c). The soft bake was done at 95°C, the time of baking was chosen to achieve a residual solvent content about 4% (Reznikova et al. 2005). To avoid stress accumulation, the substrate was cooled down to room temperature at a slow rate of 10°C/h.

2.3 Resist exposure and development

PMMA. CAMD wiggler beamline with photon energies up to 40 keV is used for the X-ray lithography. A DEX 3 scanner with efficient cooling for the mask and substrate is used for pattern transfer. Process optimization of bottom dose and top/bottom dose ratio was performed using the mask shown in Fig. 1. To obtain 25 μm wide and up to 2 mm deep trenches in PMMA, the minimal bottom dose of 3.8 KJ/cm^3 and a dose ratio of less than 2.5:1 were found to be optimal (Goettert et al. 2007). The major problem in developing such structures is that edges of the structure develop faster than the central portion. We use magnetically stirred GG-developing system at room temperature. Due to overdeveloping the edges and under developing of the center part, defects such as wider or shifted septa or missed septa are introduced into the microstructures. To address this problem, we vary the bottom dose and use megasonic assisted development in order to achieve well-developed structures over the entire area without adhesion failure. We successfully have developed and copper electroplated over entire area up to 1.5 mm thick PMMA structures. A scanning electron microscope (SEM) image of developed channels in PMMA (1), and a photograph of freestanding copper grid obtained by copper electroforming into PMMA mold (2) are shown in Fig. 2. A 2 mm thick PMMA was developed in the most areas using megasonic assistance, but adhesion failure occurred in few rows located close to the sample edges (Goettert et al. 2007).

SU-8. To fabricate SU-8 molds, mostly we use UV-lithography, because it is lower cost and more widely available in the laboratory than X-ray lithography. UV-lithography of up to 1-mm thick SU-8 resist was done using Creatv's custom UV exposure system. The exposure system uses an electric discharge high-pressure mercury lamp, with a distance between the electrodes of about 0.1 mm. To estimate the limitations in feature size produced by UV-lithography in 1-mm thick resist we did

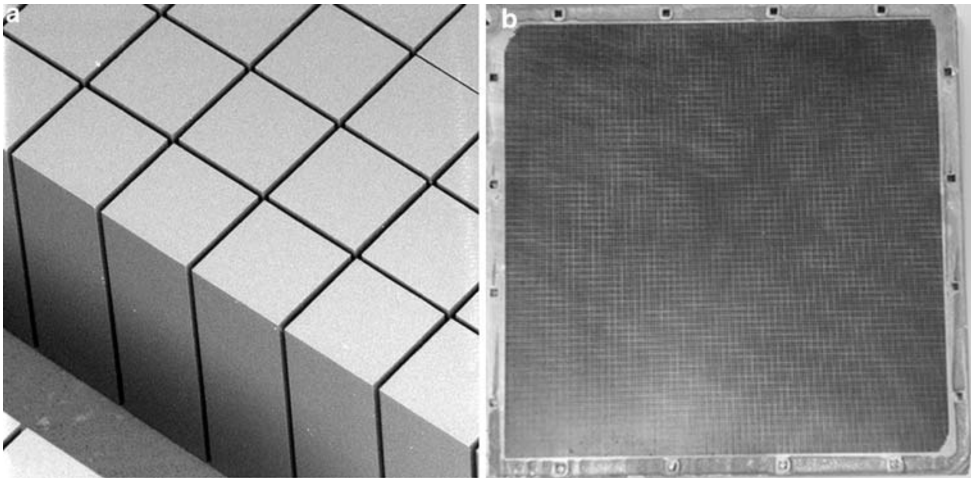

Fig. 2 SEM image of developed 25-µm wide and 1.5-mm deep trenches in PMMA (**a**), and a photograph of the electroplated grid (**b**)

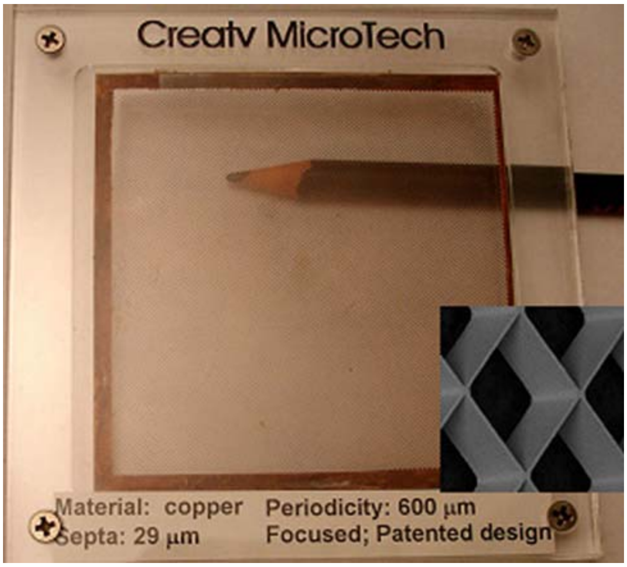

Fig. 3 Photograph and SEM image (insert) of focused to the point freestanding copper grid of patented design with 29-µm wide and 600-µm tall septa, and periodicity of 550 µm made by UV-lithography

simple calculations. Diffractive smearing at 1 mm distance behind the mask for the light with wavelength of 0.4 µm limits minimal CD by about 20 µm [$\sqrt{(1000.2^2 - 1{,}000^2)} \approx 20$]. Comparable smearing caused by the light source size in the ray optics approximation will correspond to 6 mm size of the light source placed at the distance of 300 mm from the mask ($20/1{,}000 \times 300 = 6$). Small size of our UV-source provides good contrast between irradiated and shadow region of the resist over entire resist thickness. For high resist thickness such "point" UV-source is more suitable than the MA-6 mask aligner, which has a light diffuser in its optical scheme.

Good contact between the mask and resist is essential for good lithographic quality. Thick resist layer always has some surface imperfections. To suppress light reflection and scattering at the resist and mask surfaces, we use glycerol to fill the gap between mask and resist surfaces as was suggested in (Chuang et al. 2002; Yang and Wang 2005).

For the thick resist layers, the use of broadband light source tends to result in over-exposure at the top of the resist layer and under exposure at the bottom of the resist layer because SU-8 shows higher absorption at shorter wavelengths (Reznikova et al. 2005). To improve the dose distribution over entire resist thickness and obtain better lithographic results we use Hoya UV-34 filter to eliminate short wavelengths as was suggested by Lee et al. (2003).

Using our "point" UV-source, we can fabricate point-focused structures, where the focal distance is controlled by the distance between the UV source and the mask/substrate assembly. A freestanding point-focused copper antiscatter grid of patented design 70 × 70 mm in size with 29-µm wide septa walls and 600-µm periodicity is shown in Fig. 3. SU-8 was removed using a molten salt. The use of two-dimensional freestanding antiscatter grids with septa walls oriented toward the focal point of mammography system results in contrast improvement and significantly better image quality in comparison with the conventional one-dimensional antiscatter grids.

The PEB is a very important step in SU-8 processing, and should be optimized for almost every pattern design. For example, grid structures of thin septa and large periodicity should be baked at the temperature below glass transition of unexposed SU-8. Glass transition temperature for unexposed SU-8 film is about 50°C (http://memscyclopedia.org/su8.html). At the high-temperature bake, the unexposed part of the resist is fluid, while the exposed part is partially hardened and easy to deform. An example of a PEB-deformed grid is shown in Fig. 4. On the other hand, too little PEB leads to adhesion failure after sample development. The 160-µm tall, 50-µm wide, and

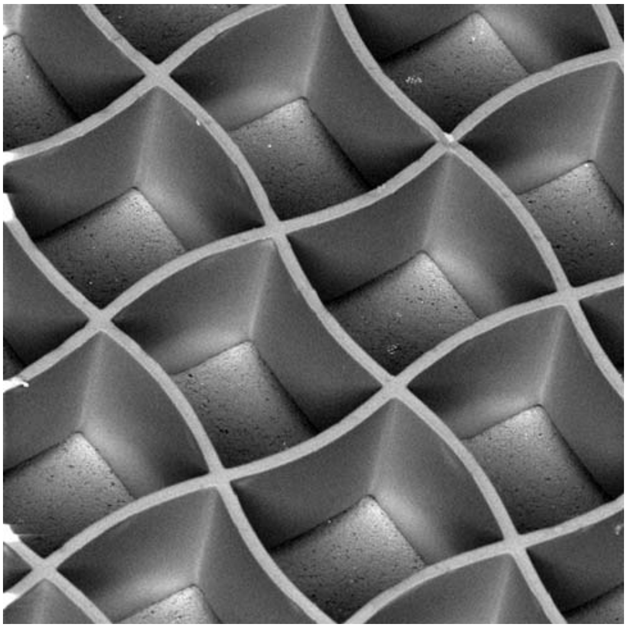

Fig. 4 SEM image of deformed SU-8 grid, septa 25-μm wide and 400-μm tall, periodicity 400 μm developed after PEB done at 95°C. Optimization of the SU-8 process parameters eliminated the distortion

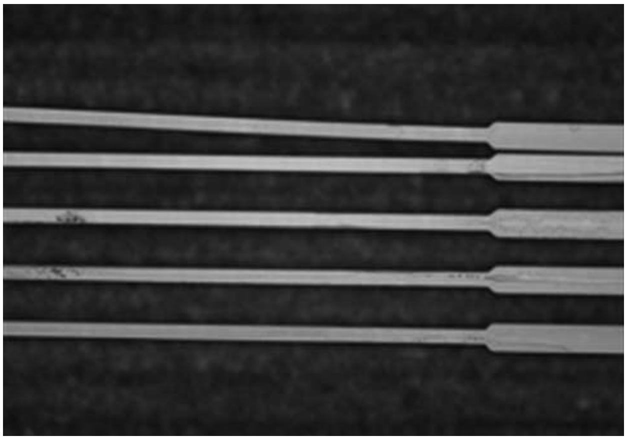

Fig. 5 Optical microscope image of 50-μm wide, 160-μm tall and 10-mm long displaced SU-8 lines developed after PEB done at 65°C. Optimization of SU-8 process parameters eliminated displacement of SU-8 lines

10-mm long SU-8 lines shown in Fig. 5 were parallel to each other after the PEB performed at the temperature of 65°C. Adhesion failure occurred after the sample was removed from the SU-8 developer, as surface tension forces bent the line that was not adhering well to the substrate.

2.4 Electroforming

Copper sulfate, nickel sulfamate, lead solderon and gold Neutonex plating processes are well established at Creatv. These plating processes can produce low stress metal deposits of high (up to 3 mm) thickness. We have succeeded in electroforming over 500-μm thick gold into an SU-8 mold. The process produces a uniform fine grain gold deposit, as shown in the SEM image of the gold slits Fig. 6. For some applications also make use of electroless nickel and silver plating over copper.

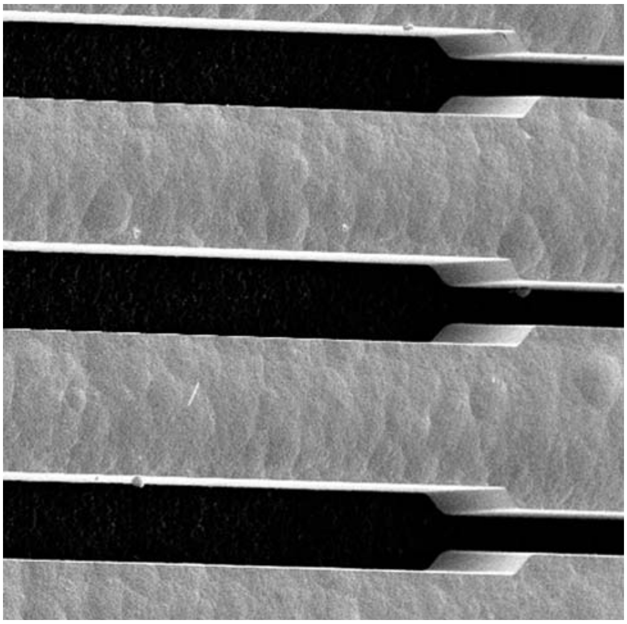

Fig. 6 A SEM image of 50-μm wide and 10-mm long slits in 150-μm thick gold

2.5 Polishing and resist removal

After electroforming is complete, the metal microstructure is carefully detached from the graphite substrate, and the upper surface planarized using machine or hand polishing. Up to 3 μm increase in line width was noted after this polishing step, due to burring. The burr is limited to the top surface of the structure, very thin, and thus can be neglected in most applications. After polishing the PMMA can be removed by soaking in acetone, and the SU-8 can be removed using a piranha solution from gold microstructures and molten salt from copper and nickel structures. Nano Remover PG (N-methyl pyrrolidone) recommended by MicroChem is not suitable for the SU-8 removal from the grid structures with thin septa. Swelling of the SU-8 resist in PG remover leads to grid distortion.

2.6 Assembly

The desirable heights of grids and collimators have been achieved by stacking the appropriate number of metal

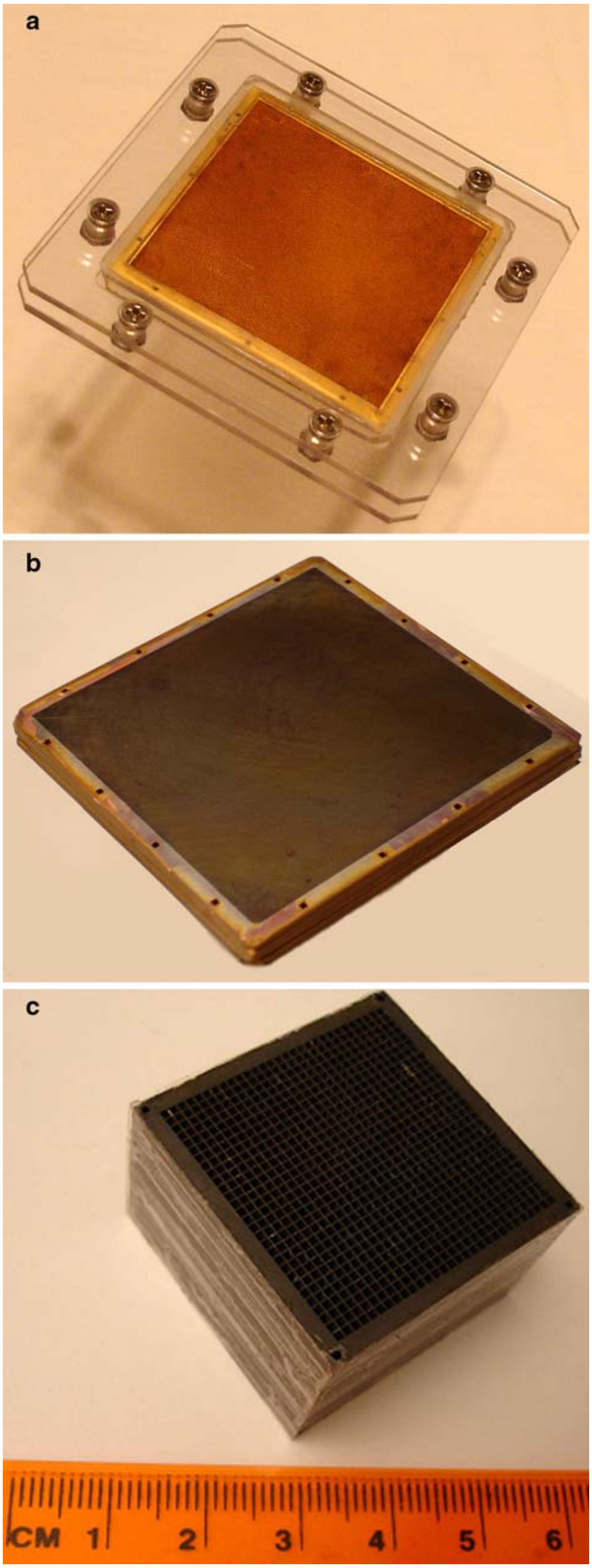

Fig. 7 Photographs of gold (**a**), copper (**b**), and lead (**c**) collimators fabricated using X-ray lithography and metal electroforming

layers using high precision alignment pins. Alignment holes for the assembly are lithographically made and can be seen in Figs. 1 and 2b. Examples of the fabricated collimators are shown in Fig. 7. Figure 7a shows a free-standing gold collimator 40 × 40 mm in size with 25-μm wide septa and of about 2-mm tall. This collimator was made by stacking five gold layers fabricated with SU-8 resist and X-ray lithography. Collimators capable of higher resolution and optimized for greater sensitivity can significantly improve the imaging of γ-cameras used in planar scintigraphy of small animals in single-photon-emission computed tomography. A copper collimator 6 mm tall, 64 × 64 mm in size, with 25-μm wide septa made by assembly of five copper layers is shown in Fig. 7b. Copper electroforming was performed in PMMA molds. Collimators for high-energy γ-cameras should be at least several millimeters tall and made of lead. A photograph of a 20-mm tall lead collimator with septa width of 130 μm is shown in Fig. 7c. The molds were up to 3 mm tall and made of PMMA.

3 Conclusion

General rules for microfabrication of metal parts using lithography and electroforming were established. Collimators and antiscatter grids of various designs have been fabricated with high degree of precision.

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