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High resolution x-ray micromachining using SU-8 resist

Bor-Yuan Shew[1], **Jui-Tang Hung**[2], **Tai-Yuan Huang**[1], **Kun-Pei Liu**[3] **and Chang-Pin Chou**[3]

[1] Microstructure Group, National Synchrotron Radiation Research Center, 101, Hsin-Ann Rd, 30077, Hsinchu SBI Park, Taiwan
[2] Department of Mechanical Engineering, Yuan-Ze University, Chungli, Taiwan
[3] Department of Mechanical Engineering, National Chiao-Tung University, Hsinchu, Taiwan

E-mail: yuan@nsrrc.gov.tw

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Abstract
This paper investigates the feasibility of using SU-8 as a high contrast x-ray resist. The SU-8 resist was irradiated with various x-ray doses and then developed for a fixed time. The developing rate was then measured and plotted versus absorbed x-ray dosage. The results revealed that SU-8 exhibited very high lithographic contrast other than elevated sensitivity. Therefore, a very thin mask absorber is required to switch the negative resist 'ON' or 'OFF'. Preliminary results showed that 1 μm wide, 17 μm thick SU-8 resists could be successfully patterned using an absorber of submicron thickness. Once the absorber thickness is effectively reduced to less than 1 μm, a high-resolution x-ray mask can be patterned simply by conventional UV lithography. An extra soft x-ray beamline was not required to increase the absorber's thickness in certain cases. The process of fabricating the membrane x-ray mask could thus be considerably simplified. X-ray micromachining may be an easy way to pattern high-resolution and high-aspect-ratio microstructures for optical and photonic applications.

1. Introduction

In deep x-ray lithography (DXL), high intensity synchrotron radiating (SR) x-rays are used as the light source to transfer the mask pattern onto the resist. The short wavelength and high penetration power of the x-ray are such that DXL is a powerful tool for generating submicron resolution and high-aspect-ratio (>50) microstructures [1–3]. When the feature size of the microstructure is reduced to $\sim$1 μm (the scale of the optical wavelength), it will interact strongly with the light and even changes its behavior. These effects have been extensively used in waveguides, gratings, Fresnel zone plates and photonic crystals [4]. Moreover, optical polymer technology has considerably advanced over the last decade. Many silica optical devices can now be replaced with low-cost polymer materials. Unlike silicon-based technologies, DXL can easily be combined with electroforming and molding techniques (the LIGA process) to mass-produce polymer microstructures. This technique has many potential applications in fabricating polymer micro-optics [3, 5, 6].

PMMA is the most popular resist in the conventional DXL process, because it can provide high-resolution capability (<1 μm) and excellent sidewall quality (R_a <30 nm). However, PMMA has poor stress corrosion resistance, resulting in low lithographic yield, especially for small and freestanding resist structures. Furthermore, PMMA has very low lithographic sensitivity and contrast. The x-ray mask is then charged to provide sufficient contrast of exposure intensity between mask/unmask areas. Therefore, the mask membrane must be thin (<2 μm) and transparent to x-rays; in contrast, the mask absorber must be sufficiently thick (>3 μm) to block x-ray irradiation [1, 2]. Such a mask structure is very fragile and difficult to fabricate.

Some problems also arise in patterning a thick absorber. As illustrated in figure 1, e-beam writing and gold plating

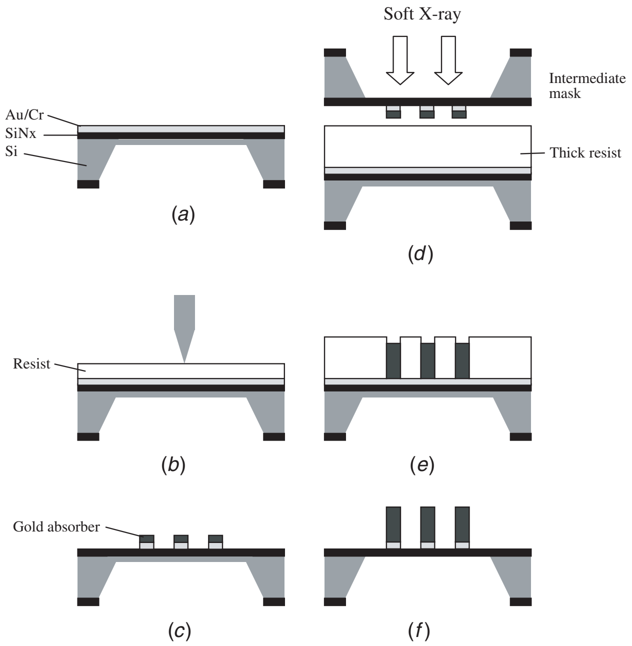

Figure 1. Conventional processes of fabrication of a membrane x-ray mask: (*a*) back-side wet etching; (*b*) e-beam writing; (*c*) plating gold absorber; (*d*) soft x-ray lithography; (*e*) plating thick absorber; (*f*) strip resist and etch the back wall Si layer.

are always used to generate a fine mask pattern. The plated gold absorber is never thick enough for DXL applications since the penetrating depth of the electron beam is limited to 2 μm. Accordingly, the thickness of the absorber must be extended using soft x-ray lithography via this 'intermediate' mask (figure 1(*d*)) [1]. These complex processes of fabricating an x-ray mask always represent a big challenge in the DXL process.

Resist contrast is normally defined as the slope of the change of developing rate to the absorbed dosage. If the resist itself has high contrast, the required thickness of the mask absorber can be reduced in consequence. Many researchers have tried to find a better x-ray resist; however, no remarkable progress has been reported [7].

SU-8 is a negative-tone, chemically amplified UV resist, which is widely used as a thick resist material in the UV-LIGA process [8, 9]. Since SU-8 is very transparent to i-lines (365 nm), the patterned resist sidewall can be very vertical, even when the resist is as thick as 100 μm. The epoxy-based SU-8 resist also exhibits excellent chemical stability, adhesive strength and resistance against stress corrosion. However, the Fresnel diffraction effect raises difficulty in patterning submicron, high-aspect-ratio SU-8 resists using only a conventional UV light source.

Actually, the SU-8 resist is also sensitive to e-beam and x-ray irradiation. In 2000, Bogdanov and Peredkov examined the lithographic behavior of SU-8 exposed to x-rays [10]. They found that its sensitivity was around 70 times higher than that of PMMA; consequently, the exposure throughput was significantly improved. Several researchers have also recently worked on the DXL SU-8 resist, but most of them have focused on the lithographic sensitivity and precision in thick film (>100 μm) applications [11–13]. In this study, the x-ray lithographic contrast of the SU-8 resist is examined. The feasibility of using SU-8 as a high resolution x-ray resist will also be evaluated.

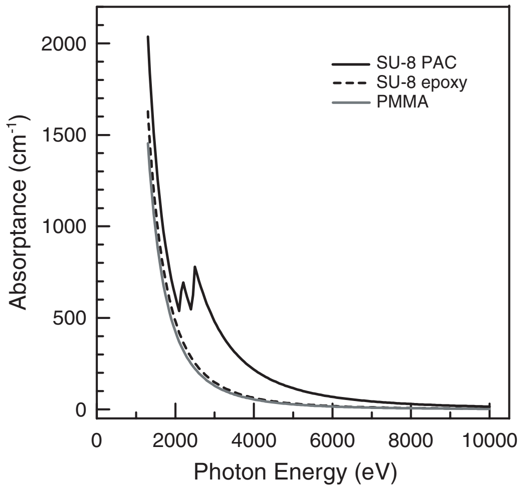

Figure 2. Absorptance of the chemical components in the SU-8 resist.

2. Dose simulation

Under x-ray irradiation, the distribution of doses in the resist is a function of the SR spectrum, beam line optics and the resist material. The deposited dose must be calculated to help in selecting optimal exposure parameters. SU-8 is chemically more complex than PMMA resist: it consists of EPON SU-8 resin, solvent (gamma butyrolacton) and photon active compound (PAC, mixed triarylsulfonium/hexafluoroantimaonate salt)[4]. The dose simulation capability of such a multi-component resist must be established before x-ray lithography experiments can be conducted.

In this work, SHADOW[5] freeware is used to calculate the dose deposited in the resist material. This work is based on the parameters of the SR source and the micromachining beamline at NSRRC (National Synchrotron Radiation Research Center). Since most of the solvent is removed after soft baking, the residuals are ignored to simplify dose simulation. As shown in figure 2, SU-8 epoxy and PMMA exhibit similar absorption behavior because they consist of the same elements (C, H, O). PAC exhibits stronger x-ray absorbance because it contains sulfur and phosphorus atoms as well as C, H and F elements. Although the PAC content in the SU-8 resist is relatively low, a slightly higher dose is deposited in SU-8 than in PMMA when the resist thickness is less than 400 μm (figure 3).

3. X-ray lithographic behavior of SU-8 resist

After the dose was simulated, the SU-8 resists (150 mm thick) were exposed to various x-ray doses, post-baked and then developed for a fixed time (2 and 5 min) to examine their lithographic behavior. An aluminum filter (50 mm thick), which can absorb most low-energy photons, was used to increase the uniformity of the x-ray dose in the depth direction.

[4] http://www.microchem.com/products/su_eight.htm.
[5] http://shadow.xraylith.wisc.edu/shadow.

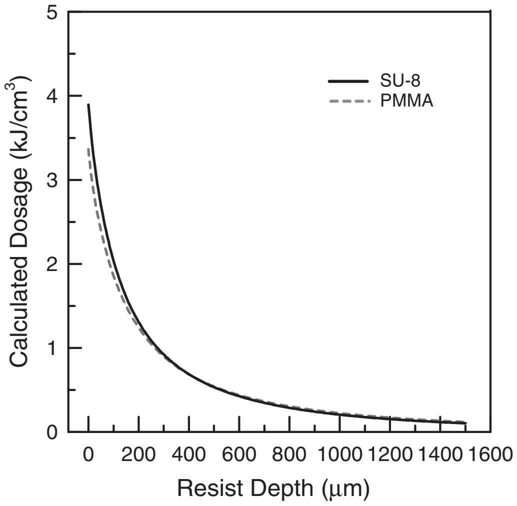

Figure 3. Simulated dosage distribution in both PMMA and SU-8 resists under an exposure expenditure of 1000 mA min cm^{-1}. The dose was calculated based on the parameters of the Taiwan light source and micromachining beamline (with 10 μm Al filter) at NSRRC.

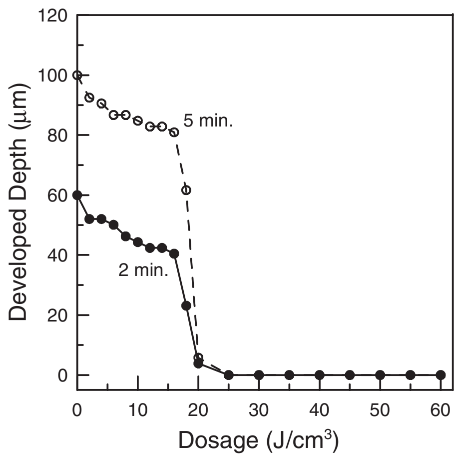

Figure 4. Developed depth versus x-ray dosage of SU-8 resist after different developing times. The original thickness of the SU-8 resist is 150 μm.

The developed thickness was then measured and plotted against dose, as shown in figure 4. The results indicate that the developed thickness decreases as the x-ray dose increases. No thickness was lost when the dose exceeded 25 J cm^{-3}. This result is similar to that reported by Singleton *et al* [12]. The SU-8 resist is much more sensitive to x-ray irradiation than the PMMA resist, which has a sensitivity of about 4000 J cm^{-3}.

AFM (atomic force microscopy) was used to determine the quality of the sidewall surface of the developed resist microstructure. Six measurements were taken along the depth direction; each scanning area was 1 μm^2. Figure 5 presents typical sidewall topography of the lithographic resist. The average roughness (R_a) was 12.2 nm and the standard deviation of the measurements was 1.3 nm, so the quality was as good as that of the surface of the x-ray lithographed PMMA resist.

As shown in figure 4, the SU-8 resist was also found to exhibit very high x-ray lithographic contrast in this study. When the dose exceeded its sensitivity (25 J cm^{-3}), no dissolution was observed; but when the dose was slightly

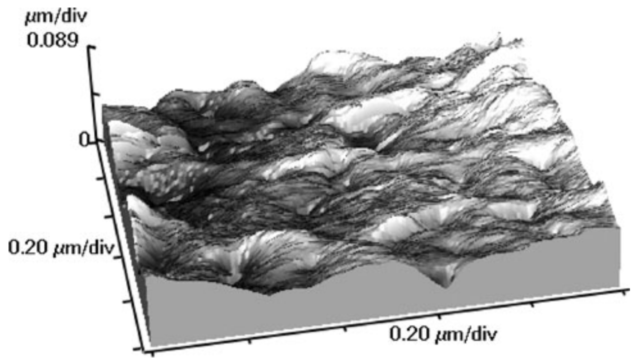

Figure 5. Typical AFM sidewall topography of the developed SU-8 resists. The average roughness is about 12.2 nm and the standard deviation of the measurement is 1.3 nm.

less than 25 J cm^{-3}, the resist developed rapidly. The slope of the line relating the change in developed thickness to the absorbed dose is quite steep. Therefore, only a thin absorber is required to switch the SU-8 resist 'ON' or 'OFF'. This can be illustrated by a specific case. Figure 6 plots the dissolution rates of PMMA [14] and SU-8 resists against x-ray dose. If the thicknesses of the resist and the gold absorber are 10 μm and 0.5 μm, respectively, then when the PMMA reaches its sensitivity (4 kJ cm^{-3}), the maximum dose under the mask is 1.8 kJ cm^{-3}. The corresponding dissolution ratio is only around eight, as shown in figure 6(*a*). Clearly, the selectivity of dissolution between the mask/unmask areas cannot give a well-defined resist microstructure. In contrast, when the dose of SU-8 reaches the sensitivity threshold (25 J cm^{-3}), the maximum dose under the mask is about 12 J cm^{-3}. As illustrated in figure 6(*b*), the corresponding dissolution ratio of SU-8 is much higher than that of PMMA. This result indicated that a thin mask absorber is required to pattern SU-8 resist by x-ray lithography. This fact greatly simplifies the fabrication of the high-resolution x-ray mask.

4. Mask design and fabrication

When the absorber is relatively thin, RIE (reactive ion etching) is the preferred means of transferring a high-resolution mask pattern. Especially, tungsten metal can be etched simply using CF_4/O_2 plasma without a toxic chloride atmosphere. Figure 7 is a proposed process for fabricating the membrane x-ray mask based on the RIE technique. First, silicon nitride is deposited by LPCVD on both sides of the silicon wafer (figure 7(*a*)). The residual stress is carefully controlled as a low tensile stress to ensure that a flat and reliable membrane is produced. The substrate is then wet-etched to open a window with a thin back wall for mechanical support (figure 7(*b*)). Subsequently, the tungsten absorber is sputtered on the top of the substrate with a thin adhesion layer (figure 7(*c*)).

Chromium is commonly used as the etching mask in the RIE process. The Cr mask pattern is generated by resist lithography and thin film deposition (lift-off process). However, the lift-off debris always sticks to the substrate, and is very difficult to remove completely. If the absorber is sufficiently thin, the resist can be used directly as the RIE mask. Figure 8 is the etching profile of tungsten/resist layers using a specific RIE recipe. The results indicate that the etching

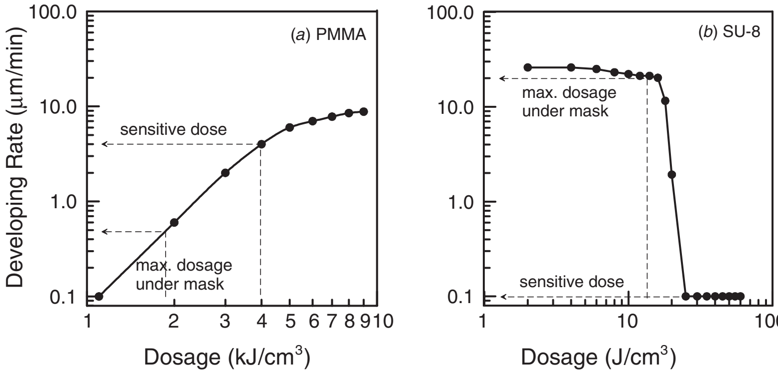

Figure 6. Developing rate of (*a*) PMMA and (*b*) SU-8 resist under various x-ray dosage. The dashed lines indicate the developing rate at its sensitive dose and at the maximum dose under the mask. The x-ray mask has a 1 μm thick S_3N_4 membrane and 0.5 μm thick Au absorber in this case.

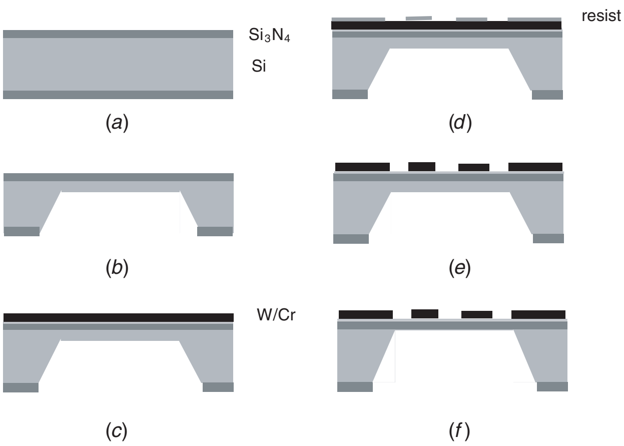

Figure 7. Fabrication processes of a membrane x-ray mask using the photoresist as the RIE mask to pattern a thin tungsten absorber: (*a*) LPCVD nitride membrane; (*b*) backside wet etching with a thin back wall; (*c*) sputtering Cr/W layer; (*d*) resist UV lithography; (*e*) RIE W absorber and strip resist; (*f*) etch residual back wall.

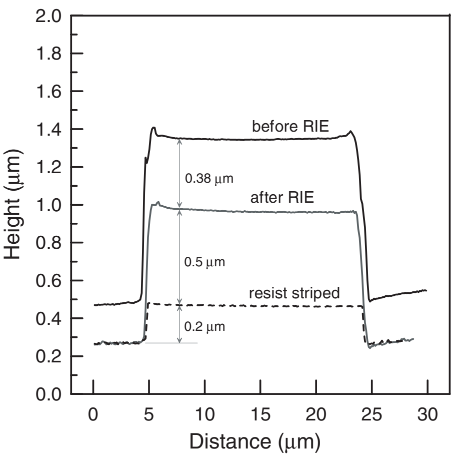

Figure 8. AFM measurements reveal the RIE profile of the resist/W layer at different process steps. The results show the etched depths of the resist and the tungsten layer are 0.38 μm and 0.2 μm after a given RIE process.

selectivity between tungsten and resist is approximately 1:1.9. That means a 1 μm thick resist can be used as the RIE mask to pattern the W absorber with a thickness of about 0.5 μm. As illustrated in figures 7(*d*) and (*e*), a positive resist is then used as the RIE mask to pattern the tungsten absorber in this study. The first RIE results show a seriously non-uniform etching profile, as illustrated in figure 9(*a*), because of the irregular distribution of the electric field, the magnetic field, the etching gases or the reaction products (called the microloading effect). In this work, 'L9' Taguchi experiments were performed to optimize the RIE parameters. The gas composition and RF power were found to be the dominant factors that controlled the etching uniformity. After the RIE recipe was fine-tuned, a quite uniform etching profile, as shown in figure 9(*b*), was obtained.

After the tungsten absorber was patterned, the resist and the Cr interlayer were chemically removed (figure 7(*e*)). Finally, the residual Si back wall was wet-etched to reveal the nitride membrane (figure 7(*f*)). Figure 10 presents a photograph of a membrane x-ray mask made by the fabrication processes described above. The diameters of the wafer and the window are 10 cm and 4 cm, respectively. The thicknesses of the membrane and the absorber were 1.5 μm and 0.8 μm, respectively. Since the fabrication process was relatively simple, the process yield of the membrane mask could be very high.

5. X-ray lithography with SU-8

The membrane mask with a thin absorber (0.8 μm) was used to pattern the resist to examine the feasibility of using SU-8 as a high-contrast x-ray resist. All the specimens were prepared using standard SU-8 resist processes. The resists were then irradiated by SR x-rays with photon energies of 800–1800 eV. After post-exposure baking, the SU-8 resists were developed for 3 min and were then inspected using a scanning electron microscope.

Figure 11 shows the cross section of the developed SU-8 resists with a line-width of 1 μm and a thickness of 17 μm. The well-defined microstructure indicates that SU-8 could be x-ray patterned using only a thin (0.8 μm) mask absorber. In fact, the thickness of the absorber can be reduced further. However, once the absorber thickness is reduced below 1 μm, the high-resolution x-ray mask pattern can be easily transferred by conventional UV lithography. An extra soft x-ray beamline is not required to increase the thickness of the absorber in some cases. The fabrication of the x-ray mask, and then the DXL process is greatly simplified.

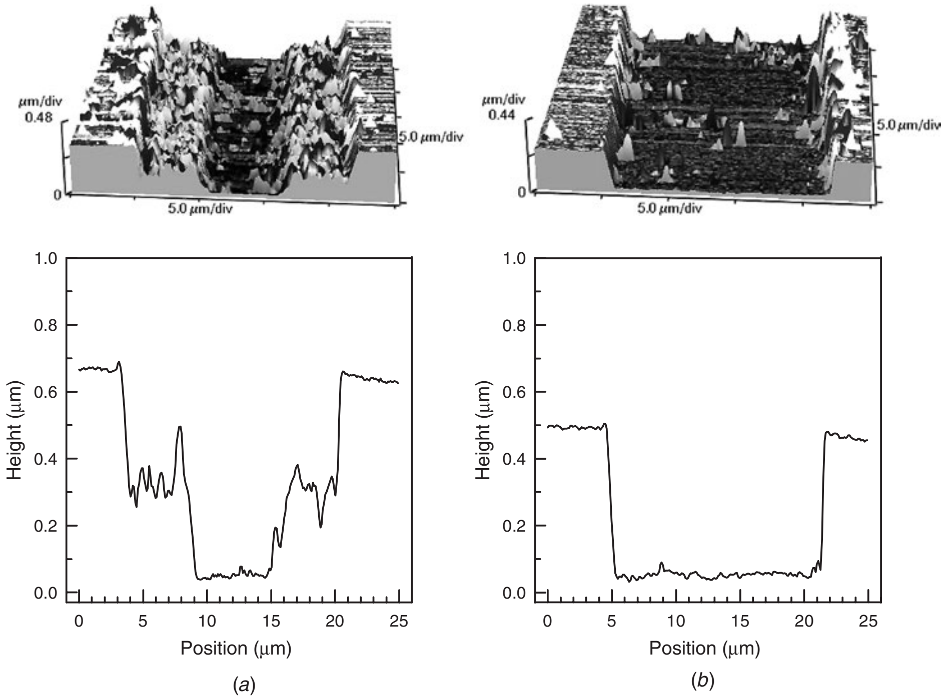

Figure 9. Cross-section of the RIE tungsten absorber (*a*) before and (*b*) after process optimization.

Figure 10. Photograph of a membrane x-ray mask with 1.5 μm thick S_3N_4 membrane and 0.8 μm thick tungsten absorber. The diameter of the mask window is 4 cm.

The sample in figure 11 was broken to reveal its cross section. Although the fine resist structures slightly deform, they still stand alone without collapse. It indicates that the epoxy-based SU-8 resist has excellent mechanical properties, chemical resistance and adhesive strength. Consequently, SU-8 resist is expected to give a much higher lithographic yield than conventional PMMA resist. Actually, the SU-8 resist is so strong that stripping the resist is always a problem. However, in most optical applications, the thickness of the microstructure is usually less than 100 μm; the SU-8 resist can be easily plasma etched in a reasonable time.

6. Summary

After x-ray irradiation, the lithographic behavior of the SU-8 resist was investigated in this study. The quality of the side-wall surface of structured SU-8 resist was found to be as good as that of PMMA. The results also show that SU-8 exhibits very high lithography contrast as well as high sensitivity.

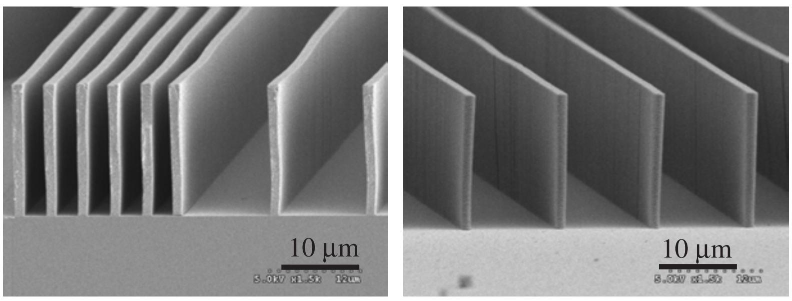

Figure 11. Cross section of the developed SU-8 resists. The resist was patterned via a membrane x-ray mask with 0.8 μm thick absorber. The line-width and the thickness of the resist structure are 1 μm and 17 μm, respectively. The resist structure slightly deforms when breaking the specimen.

Therefore, the resist can be x-ray machined using a relatively thin mask absorber. In this work, a 17 μm thick SU-8 resist was successfully patterned using only a thin (0.8 μm) tungsten absorber. The minimum thickness of the absorber remains to be studied in future work. However, once the absorber thickness is effectively reduced, the fabrication of the x-ray mask becomes much simpler. A high-resolution x-ray mask can be easily patterned by conventional UV lithography. The RIE technique is a promising method for patterning a fine mask absorber. Moreover, an extra soft x-ray beamline is not required to increase the thickness of the absorber in certain cases. High-quality x-ray micromachining techniques will become more accessible for innovative optical and photonic applications.

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