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X-ray micromachining SU-8 resist for a terahertz photonic filter

Bor-Yuan Shew[1], Han-Chieh Li[2], Ci-Ling Pan[3] and Cheng-Hao Ko[4]

[1] Device Technology Group, National Synchrotron Radiation Research Center, Hsinchu 300, Taiwan, Republic of China
[2] Department of Mechanical Engineering, National Chiao-Tung University, Hsinchu 300, Taiwan, Republic of China
[3] Departments of Electro-Optical Engineering, National Chiao-Tung University, Hsinchu 300, Taiwan, Republic of China
[4] Graduate Schools of Electro-Optical Engineering, Yuan Ze University, Chung-Li 320, Taiwan, Republic of China

E-mail: yuan@nsrrc.org.tw

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Abstract
Deep x-ray lithography (DXL) was used to fabricate two-dimensional photonic crystals (PCs) for a terahertz high-pass filter. Instead of the conventional poly(methyl methacrylate) resist, high-sensitivity SU-8 resist was used to pattern the ultra-deep (1 mm), and high aspect ratio (>30) PC structures. In this study, an x-ray mask with thick Si membrane was used in the DXL process owing to the high-sensitivity nature of SU-8. The robust mask structure can significantly improve the fabrication yield of the conventional x-ray mask with a very thin (<2 μm) membrane. Preliminary results demonstrate that SU-8 has poor pattern definition after DXL process, probably due to its high sensitivity. This phenomenon can be eliminated by dissolving oxygen into the resist to 'quench' the excessive photochemical reaction. Thanks to the high adhesion property of SU-8, the sticking problem of the high aspect ratio crystals is easily solved with natural drying by reducing the unbalanced capillary force. After optimizing all the processes, the proposed DXL SU-8 technique successively fabricated the terahertz crystals with high efficiency, high precision (<1 μm) and high surface quality ($R_a \sim 12$ nm). The resist pillars were then deposited with a 300 nm thick gold layer for subsequent terahertz measurement. The measurement results show that the PC structure acts as a high-pass filter in the terahertz range, agreeing with the simulation results.

1. Introduction

Photonic crystals (PCs) are periodic structures in which the refractive index modulation gives rise to stop bands for electromagnetic waves within a certain frequency range. By introducing a defect into the PCs, photons with definite wavelengths can locally be trapped inside the defect volume. These effects lead to some novel applications in quantum optics such as low-cost waveguide, high efficiency microwave antennas, high-Q resonators and zero-threshold lasers [1, 2]. Structures of PCs also provide a promising tool for controlling and manipulating the EM waves in integrated optical circuits. Therefore, interest is growing in developing PCs-based devices with novel functionalities.

The terahertz frequency roughly extends from 100 GHz ($\lambda = 3$ mm) to 3 THz ($\lambda = 100\ \mu$m). Recent interests in terahertz radiation stems from its ability to penetrate deep into many organic materials without causing the damage associated with ionizing radiation such as x-rays. Additionally, because terahertz radiation is readily absorbed by water, it can be used to distinguish between materials based on their water content—for example, fat versus lean meat. These properties lend themselves to applications in process and quality control, biomedical imaging, terrestrial and astronomical remote sensing. Terahertz radiation can also help scientists understand the complex dynamics involved in condensed-matter physics and processes such as molecular recognition and protein folding [3, 4].

Fabrication of the PC structure is always a critical issue. A rough estimate of the lattice size is given by the wavelength of the light divided by the refractive index of the crystal material. Since the wavelengths of the terahertz rays are about hundred micrometres, the lattice size for terahertz PCs is about a couple of tens of micrometres. However, an additional important factor that determines the properties of two-dimensional PCs is light confinement in the third (thickness) dimension. Therefore, terahertz PCs must be an ultra-deep (∼1 mm) and high aspect ratio microstructure, which is difficult to fabricate using conventional micromachining techniques. Wu *et al* [5] adopted a stereo lithography technique by polymerizing a UV curable resin layer-by-layer to fabricate such a high aspect ratio PCs structure. A thin gold layer (∼300 nm thick) was then deposited on the polymer structure as the metallic PCs. However, obvious noise signals arise due to the deformation of the PCs. Kiriakidis and Katsarakis [6] used the deep x-ray lithography (DXL) technique to fabricate three-dimensional PC structures. Highly intense and collimated synchrotron x-rays are used as the light source, making them an effective means of fabricating high aspect ratio microstructures. In the DXL process, poly(methylmethacrylate) (PMMA) is always used as the resist material because it can provide excellent lithographic quality [7]. However, PMMA has poor sensitivity to x-ray irradiation. Exposure time over 10 h is always required for patterning an millimetre-thick microstructure. Additionally, PMMA has poor resistance to stress corrosion, leading to a fragile structure and poor adhesion with the substrate.

SU-8 is a negative-tone, chemically amplified resist that has been widely used in MEMS applications [8, 9]. Moreover, the epoxy-based SU-8 resist has excellent chemical stability, adhesive strength and stress corrosion resistance. SU-8 is normally exposed to a UV light source (i-line, 365 nm); however, Fresnel diffraction raises the difficulty in patterning highly precise, millimetre-thick SU-8 resists just using UV lithography. Bogdanov and Peredkov [10] examined the lithographic behaviour of SU-8 under x-ray irradiation. They found that the sensitivity of SU-8 was approximately 100 times higher than that of the PMMA resist. Many researchers have successively x-ray patterned SU-8 resists with a resist thickness of below 600 μm [11, 12], however, no previous report was found using the DXL SU-8 technique to fabricate such a high aspect ratio PC structure. In this work, a 1 mm thick SU-8 resist was prepared and patterned using synchrotron radiated x-rays. The lithographic processes is systematically studied and optimized to yield fine terahertz PCs. The feasibility of using the DXL SU-8 technique to fabricate PC structures is evaluated via practical terahertz measurement.

2. Design of a terahertz photonic filter

This study designs a PC-based high-pass filter and uses it for evaluating the quality of the DXL SU-8 technology. The device comprises 17 × 25 pillar arrays with a lattice constant (a) of 170 μm. The crystal diameter and depth are 30 μm and 1000 μm, respectively. Meanwhile, the photonic band structure of the gold PCs is simulated with BandSOLVER (Rsoft Design Group, Inc., USA). As shown in figure 1, the PC has a wide band gap at a/λ of about 0.05–0.36, corresponding to a frequency range of approximately 0.08–0.66 THz. FDTD simulation results also indicate that the terahertz-wave at this frequency range will be totally reflected by the array of gold PCs. This means that the PC array act like a high-pass filter. This photonic device will be fabricated based on deep x-ray micromachining in the work. Once the fabrication platform is established, the geometry and dimension of the PCs can be easily modified to give different functionalities.

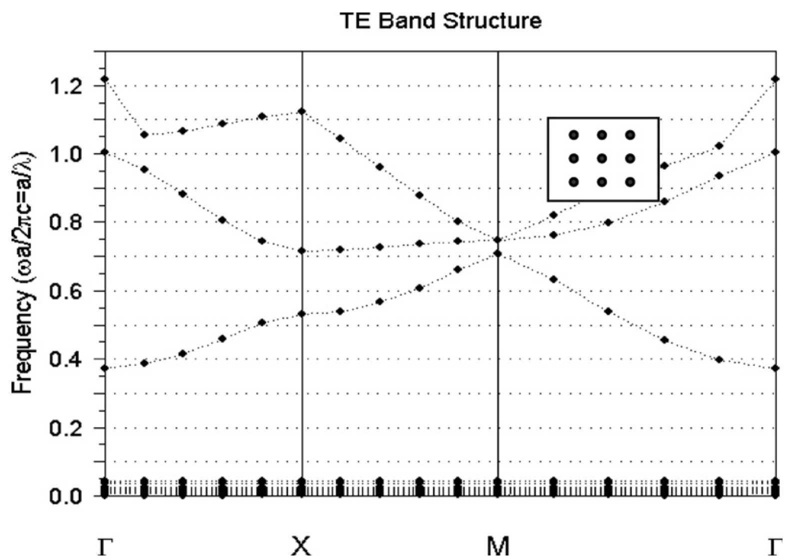

Figure 1. Photonic band-gap structure of the two-dimensional PCs. The crystal arrays have a lattice constant of 170 μm and a diameter of 30 μm.

3. Resist preparation

SU-8 resists (2050) with a thickness of 1 mm were prepared on a silicon substrate. Spin coating was not used; instead, resist with a constant weight was poured directly onto the substrate to produce a thick resist layer. The resist was then soft baked on a hotplate. Since the SU-8 resist has very low viscosity at elevated temperature, the resist spreads all over the substrate to a uniform thickness.

SU-8 resist is always suggested to be soft-baked at 95˚C; however, it takes over 10 h to bake millimetre-thick SU-8 resist. Since the photon active compound (PAC) of the resist degrades at temperatures above 130˚C, the thick resist was baked at 120˚C to accelerate the soft-baking process in this work. Figure 2 illustrates the weight loss of SU-8 resist with baking times. The results show that the weight loss initially increases and then saturates with baking time; meanwhile, the weight loss (drying) is significantly faster at high baking temperature. Subsequent experiments indicated that lithographic quality is good when the weight loss is controlled at 21–22%, so that the optimal baking times are about 5 h and 20 h at the two baking temperatures. The baking rate thus can be increased fourfold when the baking temperature is raised from 95˚C to 120˚C.

4. Deep x-ray lithography of SU-8 resist

4.1. Sensitivity

To examine the DXL behaviour of the SU-8 resists, they were irradiated with various x-ray doses, post-baked and then developed for 2 min; the development rates were then measured and plotted against dose, as shown in figure 3. X-ray exposure was performed at the micromachining beamline of the NSRRC. The electron energy of the synchrotron source is 1.5 GeV, and the characteristic photon energy is 2.3 keV. An aluminium filter (50 μm thick), which can absorb most low-energy photons, was used to increase the uniformity of the x-ray dose in the depth direction. According to figure 3, the development rate decreases drastically as the x-ray dose exceeds 15 J cm^{-3}. No thickness loss was observed when the dose exceeded 25 J cm^{-3}. This result resembles that reported by Singleton *et al* [12]. Consequently, the SU-8 resist is much more sensitive to x-ray irradiation than the PMMA resist, which has a sensitivity of around 4000 J cm^{-3}.

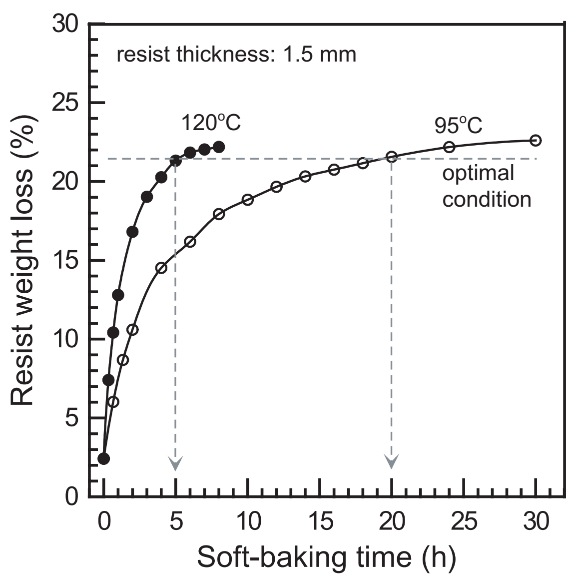

Figure 2. Weight loss of the SU-8 resists baked at various soft-baking times and temperatures.

4.2. X-ray mask

Since SU-8 has higher sensitivity than PMMA, this resist can be patterned effectively using even a 'robust' x-ray mask with a thick membrane. This fact markedly improves the fabrication yield of the conventional x-ray mask in which a very thin (<2 μm) nitride film is used as the membrane. Figure 4 shows the calculated expenditure with various silicon back-wall (membrane) thicknesses both for PMMA and SU-8 resists with a thickness of 1 mm. The simulation is performed when their minimum doses (sensitivity) are achieved. Simulated

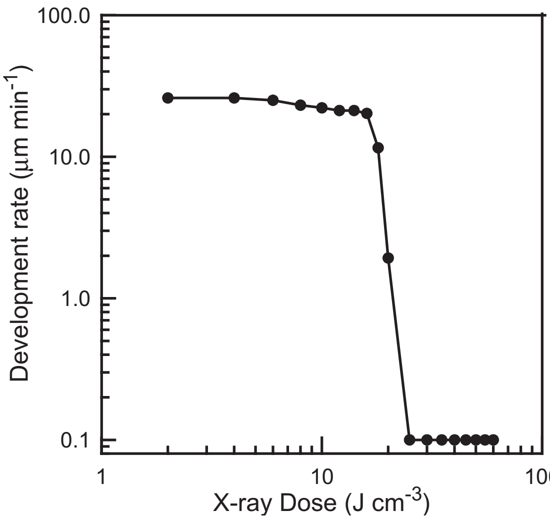

Figure 3. Development rate of the SU-8 resist with various x-ray doses.

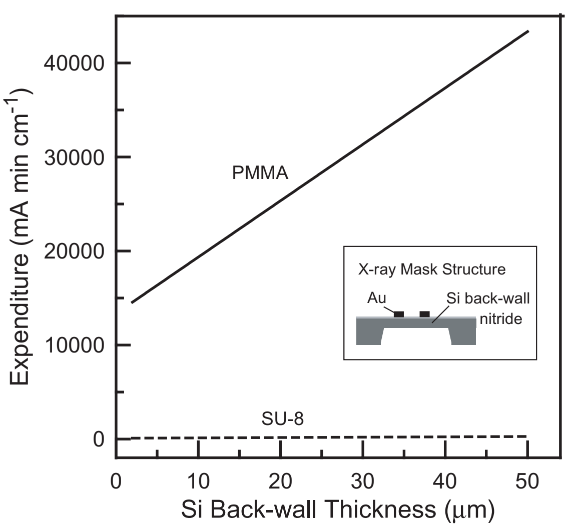

Figure 4. Expenditure both for PMMA and SU-8 resist (1 mm thick) with various mask back-wall thicknesses.

results show that the expenditure of PMMA is considerably higher than that of SU-8, and increases noticeably with Si back-wall thickness. In contrast, the expenditures of SU-8 remain low even Si membrane is thick. For example, when using an x-ray mask with a 20 μm thick Si membrane, the expenditures for PMMA and SU-8 are 24 300 and 150 mA min cm^{-1}, corresponding to exposure times of around 20 h and 10 min, respectively. The DXL SU-8 process thus not only saves x-ray exposure time, but also simplifies the fabrication process of the corresponding x-ray mask.

4.3. Oxygen quench effect

An x-ray mask with wet-etched Si membrane (20 μm thick) and electroplated gold absorbers (15 μm thick) was used for patterning the SU-8 resist. A graphite foil was placed between the x-ray mask and the resist to absorb the x-ray induced fluorescence from the mask. The resists were then exposed to achieve a minimum x-ray dosage of 25 J cm^{-3}, post-exposure baked (65˚C, 1 h), ultrasonically developed for 30 min and then blown dry for subsequent inspection.

Preliminary results showed that lithographed SU-8 resists exhibit very poor pattern definition, as illustrated in figure 5(*a*). A gel-like resist, which should be removed during development, is observed in the masked area even when the absorber thickness is increased. The x-ray induced fluorescence inside the resist is believed to cause the excessive cross-linking reaction of the high-sensitivity resist [13]. In our previous study, this effect could be 'quenched' by holding the resist in oxygen atmosphere sufficiently long before soft baking. As shown in figure 5(*b*), the DXL SU-8 resists reveal better pattern definition after oxygen atmosphere treatment. The possible mechanism of the oxygen quench effect is discussed elsewhere [13].

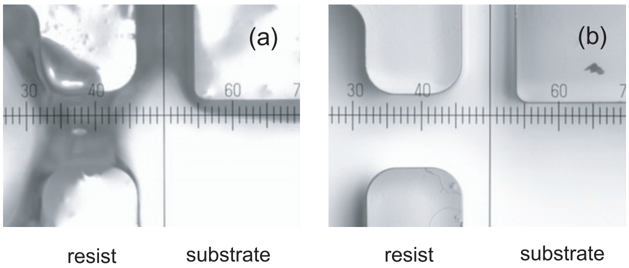

Figure 5. Top view of the x-ray lithographed SU-8 resists (*a*) without and (*b*) with oxygen treatment before soft baking.

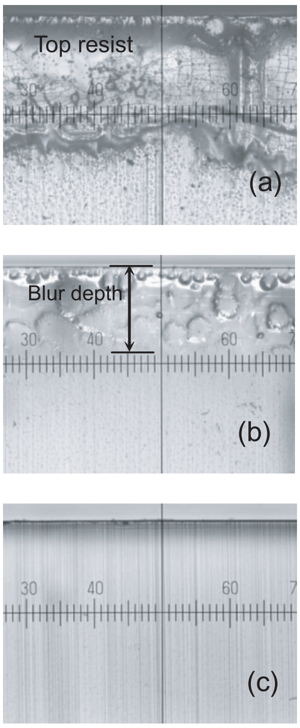

Figure 6. Side view of the x-ray lithographed SU-8 resists with various top dosages of about 350 (*a*), 225 (*b*) and 200 J cm^{-3} (*c*). The bottom dose of the SU-8 resist is 25 J cm^{-3}.

4.4. Top/bottom dose ratio

Following oxygen treatment, the sidewall still reveals poor surface quality at the top of the resist (figure 6(*a*)). The top dosage may be too high to induce excessive photochemical reaction, but no reference data could be found regarding the maximum dosage for the SU-8 resist.

The dose distribution in the resist can be easily controlled by inserting the filter to absorb low-energy photons of the synchrotron radiation. Figure 7 illustrates that increasing Al filter thickness can effectively improve the uniformity of the dose distribution in the resist. Accordingly, the top dosage of the SU-8 resist was controlled using Al filters of varying thicknesses. The depth of the blur sidewall was then measured and plotted against the top/bottom dose ratio to find the optimal condition. As illustrated in figures 6 and 8, the blur depth decreases with dose ratio (or top dose). No blur sidewall was observed when the dose ratio was less than 7.8, or when the top dose is below 200 J cm^{-3}. Atomic force microscopy (AFM) was used to measure the surface roughness of the resist sidewall after the DXL process. Six measurements were performed along the depth direction; each scanning area was 1 μm^2. The measurement results show that the average roughness (R_a)

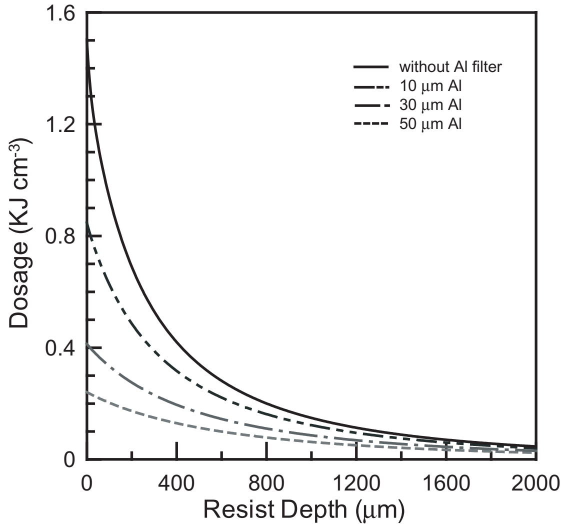

Figure 7. Dosage distribution in SU-8 resist with aluminium filter of various thicknesses.

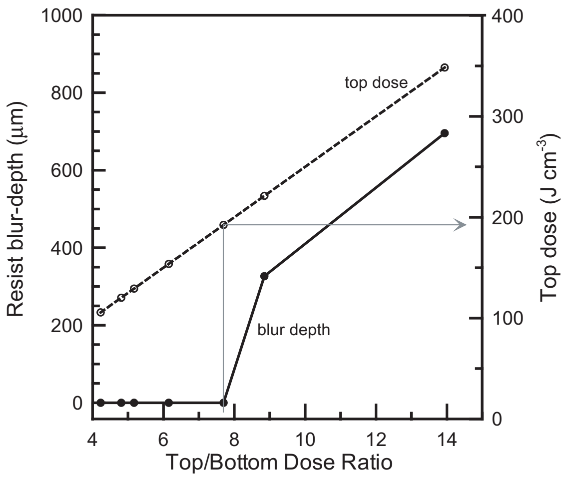

Figure 8. Sidewall blur-depth of the x-ray lithographed SU-8 resist at various top/bottom dose ratios. The bottom dose of the SU-8 resist is 25 J cm^{-3}.

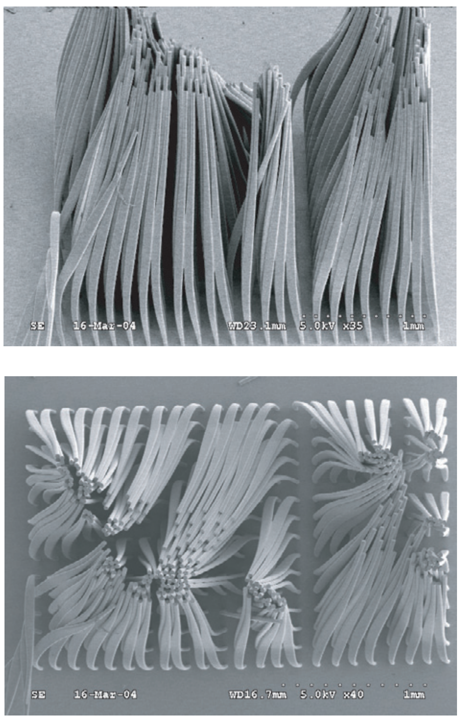

Figure 9. Collapse of the high aspect ratio SU-8 resists after development.

was only 12.2 nm and that the standard deviation of the measurements was 1.3 nm. The quality is as good as that of the DXL PMMA process.

4.5. Resist sticking

Based on the above lithography processes, an SU-8 resist (1 mm thick) was exposed through an x-ray mask to fabricate the PC structure. The resists were then post-exposure baked (65˚C, 1 h), developed for about 2 h, rinsed with IPA, and finally dried on a hot plate. However, the resist pillars stick together and collapse toward the centre of the array as shown in figure 9.

Microstructure sticking is caused mainly by capillary force, which is commonly observed in the micromachining process. Supercritical drying is the ultimate method for solving the sticking problem; however, it requires a pressurized chamber and complex equipment. This work eliminated the sticking phenomenon simply by reducing the unbalanced capillary force during the drying process as illustrated in figure 10. Compared with hot plate drying, the unbalance force is relatively low under air-drying; as a consequence, the resist pillars have more chance to overcome the sticking problem. Experimental results show that sticking-free, high aspect ratio SU-8 pillars were obtained after the natural drying process (figure 11). From the figures, it can be seen that the resist pillars are quite perpendicular to the substrate and the dimension deviation is less than 1 μm along the whole of the depth. Additionally, the yield of the DXL SU-8 process is near 100%, indicating excellent adhesion strength between the SU-8 resist and the silicon substrate. This phenomenon differs markedly from the behaviour of thick SU-8 resists, which always detached from the substrate after UV lithography.

The adhesion properties of the resists were evaluated by the pull-off test in this study. A thin SU-8 resist was prepared on the silicon substrate, irradiated with appropriate doses of UV and x-ray and then cut into small blocks. Kapton taps are uniformly adhered to the resists and then gently torn from the resists. The percentage of the residual resist block is then used as an index of adhesion strength. As shown in figure 12, the adhesion strength of x-ray irradiated resist is obviously superior to that of UV irradiated resist even if the UV dosage is higher. The improvement might be attributed to the enhanced diffusion process at the resist/substrate interface under high energy x-ray irradiation.

5. Terahertz measurement

Before terahertz measurement, a 300 nm thick gold layer was sputtered on the SU-8 PC microstructure. Since the gold thickness exceeds the skin depth of the terahertz radiation in Au (about 80 nm at 1 THz), the whole PCs can be treated as metallic without considering the contribution of the embedded polymer.

In the measurement bench, the terahertz ray (T-ray) was generated from a biased antenna irradiated with a Ti–sapphire laser. The T-ray was focused on the PCs by a parabolic mirror, and the transparent T-ray irradiated on the ZnTe crystal. Owing to the electro-optical effect, the refraction index of the ZnTe material changes with T-ray intensity; this change is simultaneously monitored by a detection light split from the Ti–sapphire laser. Consequently, the transmitted intensity through the PCs structure can be measured in the ranges of terahertz frequency.

As indicated in the figure 13, the terahertz measurement result indicates that the transmission of the PCs array is relatively low at a frequency below 1.2 THz (or a/λ below 0.6); the PCs array functions as a high-pass filter that agrees with the simulation results. However, the T-ray transmits the PCs at a frequency above 1.2 THz, which exceeds the calculated frequency of about 0.66 THz (or a/λ above 0.36) as discussed in section 1. The deviation might result from the strong absorption of water at the frequencies of 0.5, 0.7, 0.9

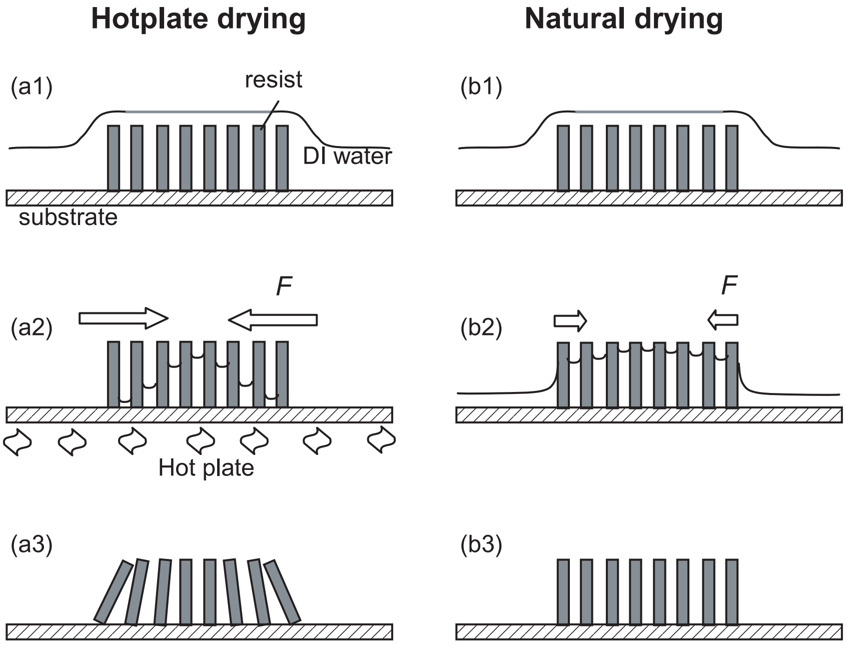

Figure 10. Schematic diagrams illustrating the unbalanced capillary force under (*a*) hot plate and (*b*) natural drying.

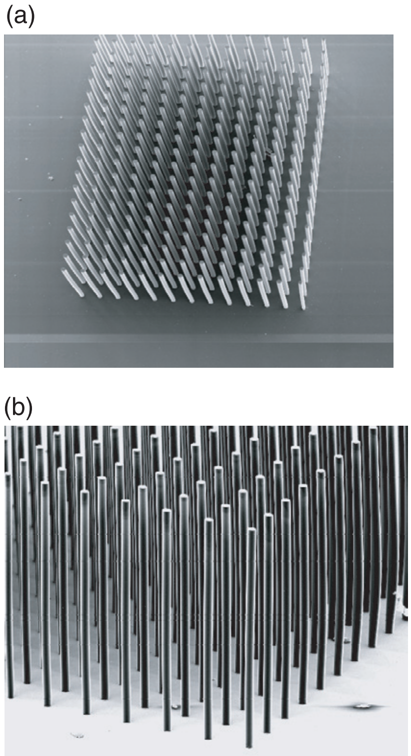

Figure 11. SEM micrographs of the SU-8 PCs fabricated by DXL. The thickness and the diameter of the pillar crystal are 1000 μm and 30 μm, respectively.

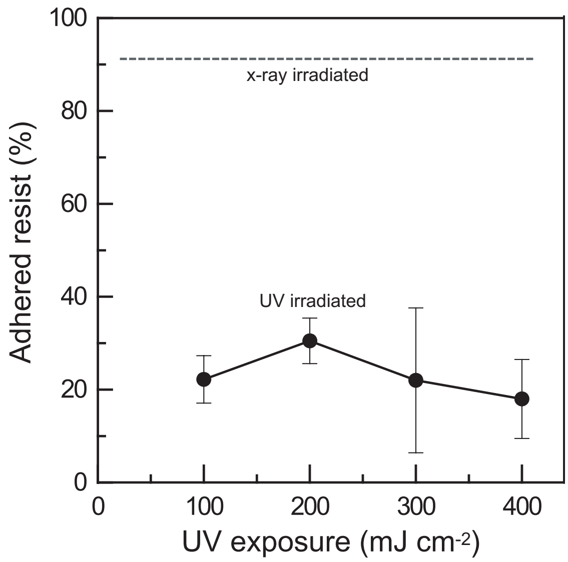

Figure 12. Adhesion properties of the SU-8 resist after UV and x-ray irradiation.

and 1.2 THz as observed by Zhang and Grischkowsky [14]. This noise might be eliminated by performing terahertz measurement in a humidity-controlled environment in the future.

In summary, the DXL SU-8 technique appears to be a promising way for fabricating terahertz photonic device. Attempts are underway to fabricate a tunable PCs array deposited with ferromagnetic materials by changing external magnetic field. Many novel photonic devices also can be realized using the technical platform reported in this work.

6. Summary

This study used high-sensitivity SU-8 resist as the x-ray resist for patterning the ultra-deep (1 mm), and high aspect ratio (>30) terahertz PC structure. Using SU-8 offers several advantages. First, the exposure time can be reduced to approximately 1/100 compared with PMMA. Second, the corresponding x-ray mask is much easier to make because the

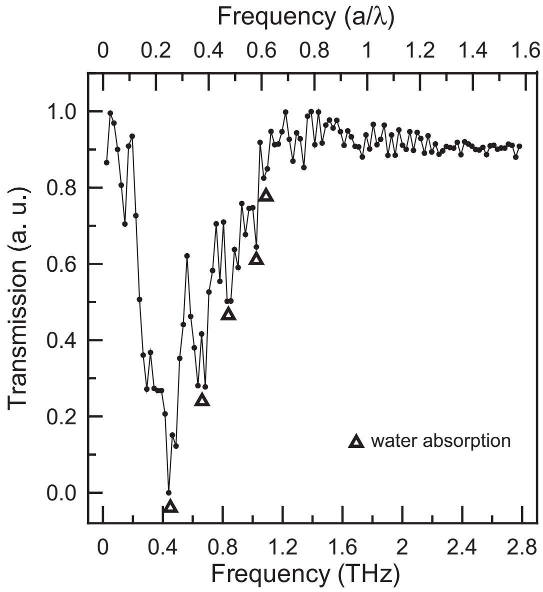

Figure 13. Transmission of the SU-8 photonic filter in the terahertz range. A gold layer (300 nm thick) is sputtered on the SU-8 crystals.

mask can have a more robust structure. Third, the adhesion strength and stress corrosion resistance of SU-8 exceed that of PMMA, leading to high fabrication yield. However, using SU-8 resist also raise difficulties in providing high-quality x-ray lithography. The structured thick resist is poorly defined and reveal blur patterns. The x-ray induced fluorescence is believed to cause the excessive cross-linking reaction of the high-sensitivity resist. This phenomenon can be eliminated by dissolving oxygen into the resist to 'quench' the excessive photochemical reaction.

After optimizing all the processes, the proposed DXL SU-8 technique successively fabricates the terahertz crystals with high efficiency, high precision ($<1\ \mu$m) and high surface quality ($R_a \sim 12$ nm). The terahertz measurement results show that Au-coated SU-8 photonic crystals act as a high-pass filter in the terahertz range, in agreement with the simulation results. Although the measurement bench must be optimized to reduce the deviation from the theoretical calculation, DXL SU-8 appears to be a promising technology for fabricating high-quality terahertz PCs. In the future, other PC-based devices with various functionalities can be devised based on this technical platform for innovative applications.

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