Optimization of SR Exposure and Etching Techniques for Microneedle Structure with Deep-hole

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Abstract

In this paper, the method for fabrication of PMMA (polymethylmethacrylate) through-hole microneedle is discussed. Typically, the fabrication of hole by Synchrotron Radiation (SR) exposure was possible up to the aspect ratio of depth and hole-diameter of 20 where the required microneedle hole-diameter and depth was 30-µm and 800-µm depth respectively. Therefore, the process to enhance the higher aspect ratio and smooth surface of the microneedle was investigated. The optimum X-ray dose of 0.02 A.h to a static expose area, 50ºC temperature of GG-developer and 10 hours of developing time have been determined by a number of collected data. With these optimum parameters, the targeted length of microneedle at 400 µm, through-hole at 700 µm and the chip thickness at 300 µm were achieved.

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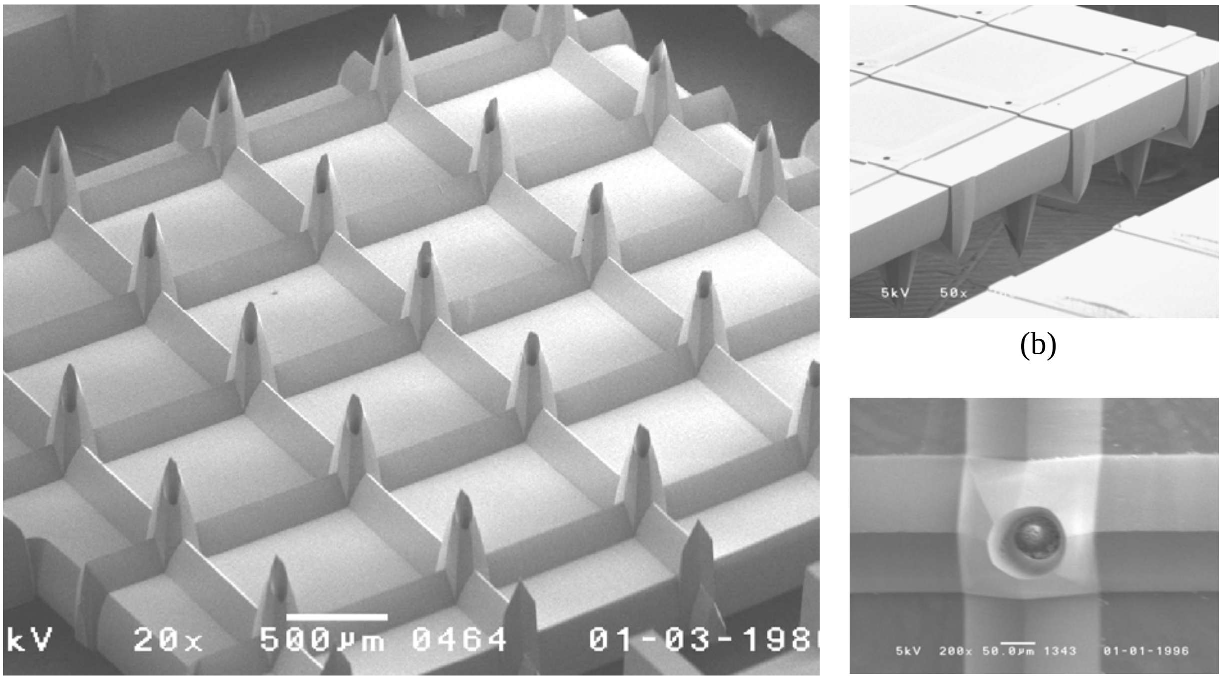
1. Introduction

Deep-hole microfabrication process becomes the main difficulty for the fabrication of some microstructures e.g., through-hole microneedle by Synhrotron Radiation (SR) lithography. Although SR lithography has been recognized as the most efficient method for fabrication of high-aspect-ratio microstructure, the deposition radiation dose is also based upon the size of hole, beam intensity and SR wavelength. A few reports have been mentioned about using a filter for a fabrication [1], expose SR from both surfaces of the resist [2], or bonding multilayer substrate [3] which the high precision alignment is necessary. However, the description of several influences during the fabrication process has not yet been available.

In the previous work, we have succeeded in fabrication of through-hole mironeedle where the thickness of chip was left to only 200 µm and it was happened to be very difficult to handle since the PMMA after processing has been changed in the properties [4], for example, PMMA after being reacted by GG developer appears to be fragile as silicon. Therefore, we attempted to leave the substrate as thick as possible. Figure 1 illustrates the former microneedle array fabricated by PCT (Plane-pattern to Cross-section Transfer) technique [5] and alignment X-ray lithography [6] which the thickness of the chip was 400 µm while the hole fabrication was not possible with the common X-ray dose and developing time obtained from the plot in Fig. 2. Although the back side image shows that through holes were fabricated, some non-developed part was observed in the middle of the hole as shown in Fig. 1(c).



(a) (b) (c)

Fig. 1 Images of microneedle fabricated by PCT and Alignment exposures, (a) the microneedle array, (b) back side of the array and (c) incomplete hole observed.

2. Influences in X-ray lithography

The X-ray dose, developing time and temperatures have been considered as parameters influencing the hole fabrication. Since the X-ray lithography has to be performed twice, the deposited X-ray dose from a PCT exposure and from an alignment exposure requires the adjustment. The PMMA needle structure was once revealed by developing process whilst the structure was etched again after the alignment exposure of the hole without any method for protection. Consequently, it is necessary to develop the PMMA microneedle shortly after PCT exposure in order to use the developed structure for an alignment. The rest of structure will be left for a completely revealed after the alignment exposure.

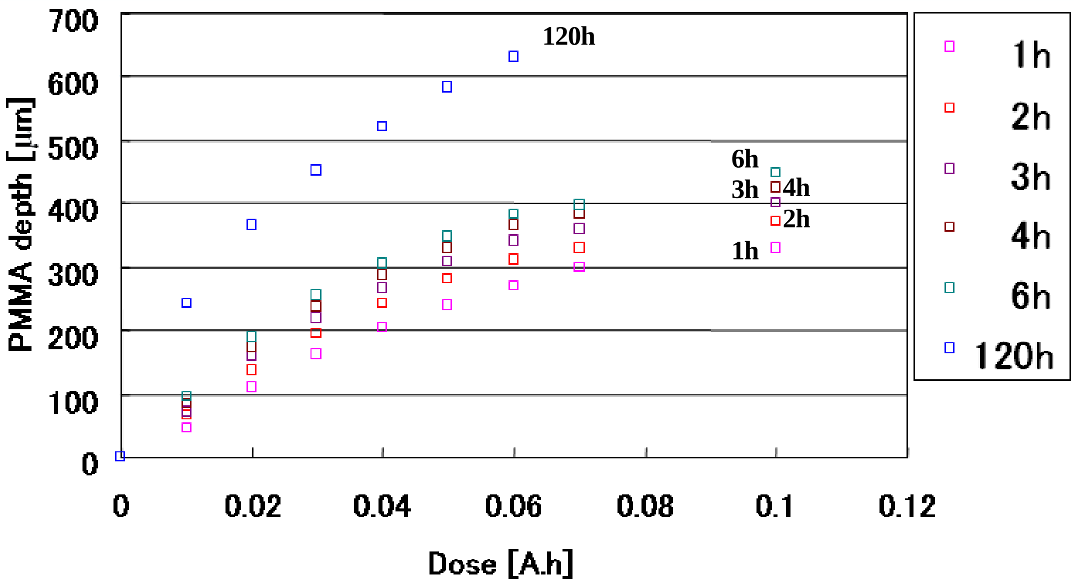
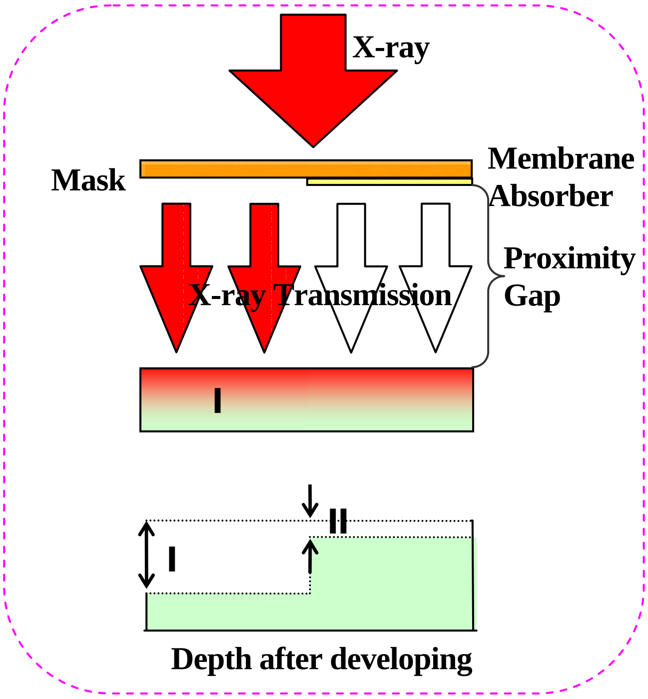


Fig. 2 General data plot used for PMMA exposured by SR from AURORA light source at BL-13 with developing temperature of 37ºC.

It is noted that the higher X-ray dose, the shorter developing time. We need to adjust the dose for PCT exposure with the alignment exposure, a long developing time will be required to reveal the hole. Moreover, in the hole-exposure, the dose which is over the absorption limit of the absorber will be taken into account. This means the amount of dose absorbed by Au will be exceeded and goes through the PMMA. Figure 3 shows the schematic diagram of X-ray transmission through mask's membrane and absorber. The different shade of each arrow at X-ray transmission area indicated the light intensity which goes through the mask. The lighter shape refers to lower intensity. The shade color at the PMMA means the deposited X-ray energy. The sample problem of over limit of the absorber is illustrated in Fig. 4.

The two main influences apparently observed from Fig. 4 are the proximity gap which affects the size of hole-diameter and over-transmission of X-ray through the absorber which affects the undesired expose area to be unavoidably exposed. The first influence is considered difficult to reduce since the gap should not be too narrow in order to protect the X-ray mask from scratching by PMMA especially when performing the PCT process which the resist was moved during the exposure. Moreover, according to the data from Fig. 2, to reach the depth of 400 µm by developing temperature of 37ºC, the developing time has to be more than 6 hours for the case of maximum dose (0.06 A.h) while more than 120 hours was consumed to develop the structure exposed by the dose 0.02 A.h.



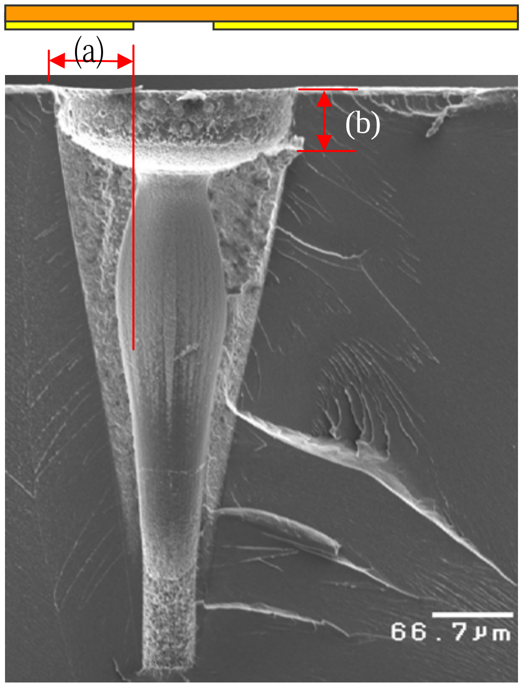


Fig. 3 Schematic of X-ray transmission through the mask

Fig. 4 Effects of (a) proximity gap and (b) X-ray transmission through absorber

3. Experimental Results

The problem could be solved by rising the developing temperature in order to reduce the X-ray dose. Figure 5 shows a plot of PMMA depth against the developing time for various developing temperature.

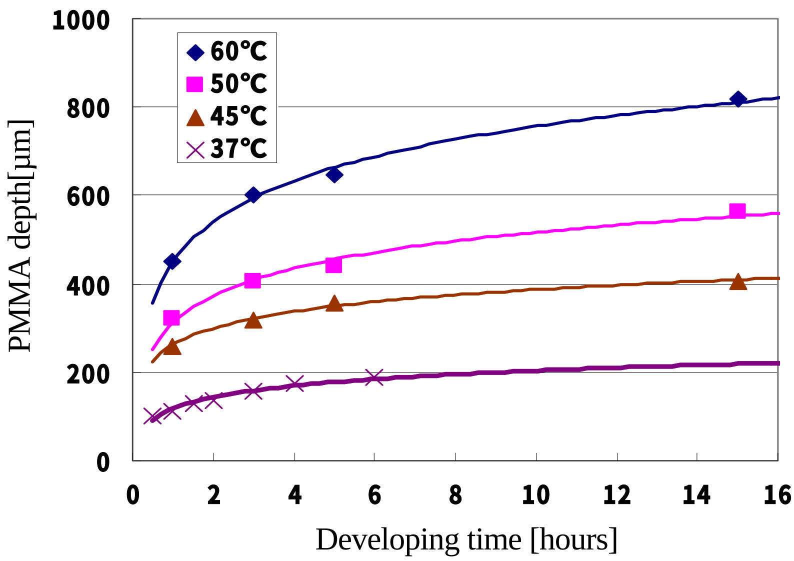


Fig. 5 Data plot of PMMA depth revealed by various developing temperatures against the time (the X-ray dose amount of 0.025 A.h)

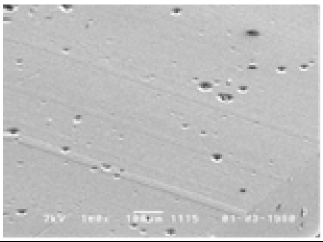
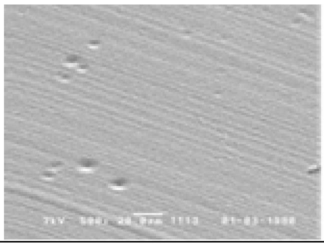
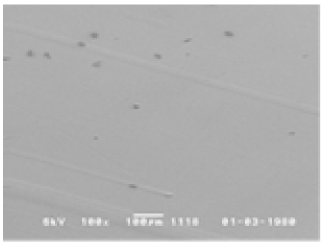
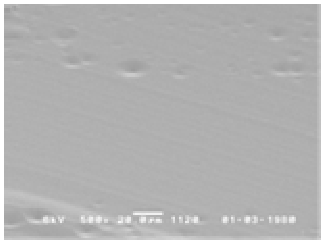
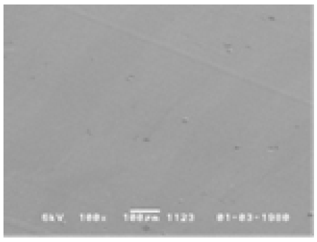
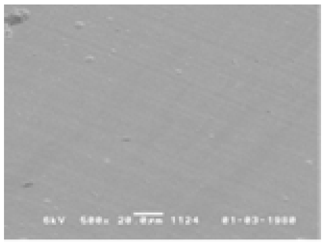
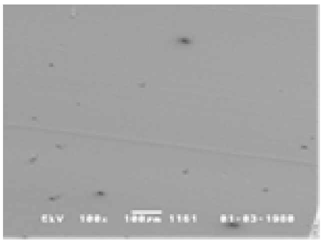
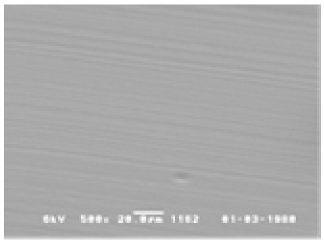
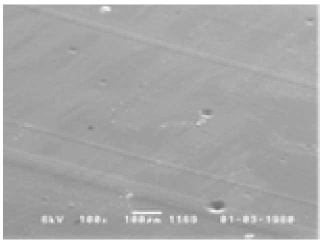
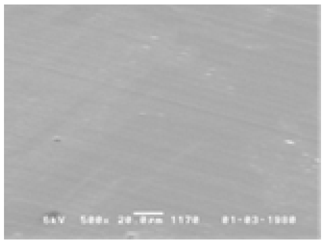
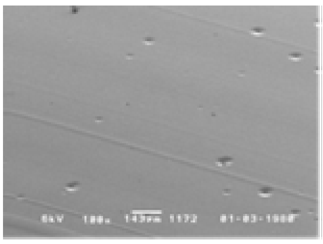
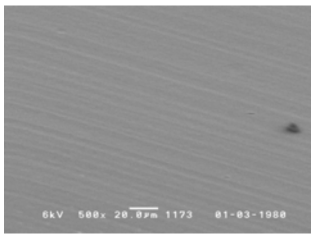
Temp.[ºC]	SEM 100 Magnitude	SEM 500 Magnitude
37		
40		
45		
50		
55		
60		

Fig. 6 SEM images of PMMA surfaces after developing

The surface quality of PMMA after developing was also observed. Figure 6 shows the SEM images PMMA surfaces developed by 37ºC, 40ºC, 45 ºC, 50ºC, 55 ºC and 60ºC. As a result, the higher developing temperature, the smoother surface of PMMA was achieved.

However, at the temperature over 50 ºC caused a swollen surface as shown in Fig. 7. Therefore, the developing temperature at 50ºC was selected for the further process.

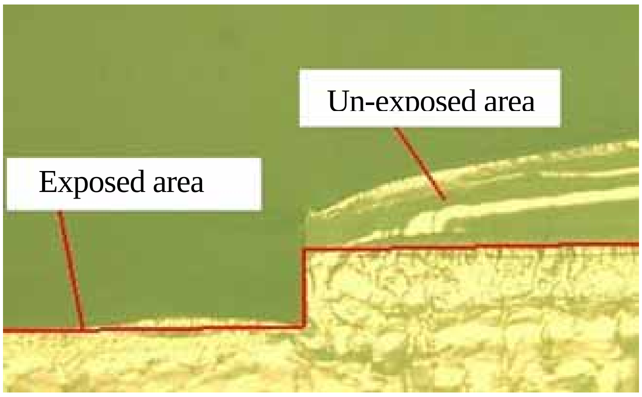


Fig. 7 Swollen surface of PMMA after developing by temperature at 55ºC

The plot in Fig. 5 was used to confirm that PMMA 400 µm can be achieved by the temperature of 50ºC with the developing time of 10 hours. This data is kept for the fabrication of microneedle using PCT exposure while the dose for alignment exposure has to be adjusted for the 700 µm through-hole fabrication revealed by the same developing time. The X-ray dose for hole fabrication can be calculated consequently.

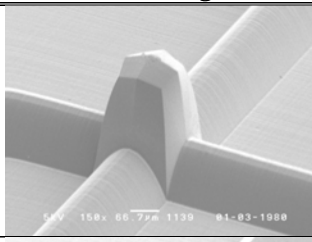
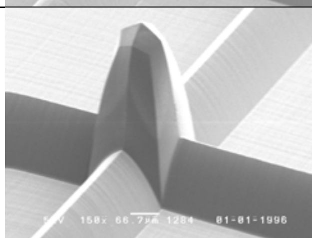
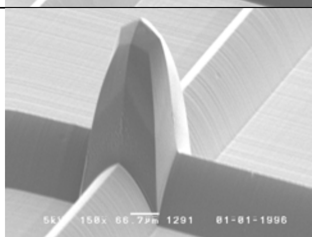
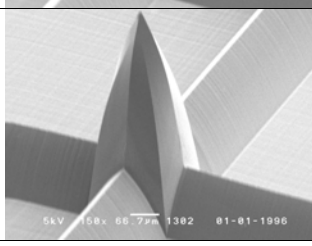
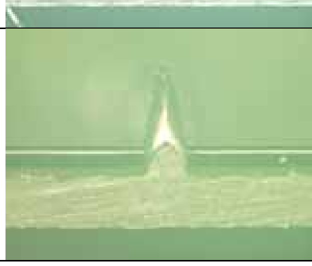
		SEM Images	Digital Microscope Images
Dose amount [A.h/line]	0.01		
	0.015		
	0.02		
	0.025		

Fig. 8. SEM images and digital microscope images of microneedle exposed by various doses.

The reason the dose of 0.025 A.h was from the data in Fig. 8 where the microneedle was exposed with dose of 0.01, 0.015, 0.02, and 0.025 A.h. The developing time was 10 hours. The microneedle structure was completed by the dose of 0.025 A.h.

The dose data for an alignment exposure was observed by a number of experiment and given in Fig. 9.

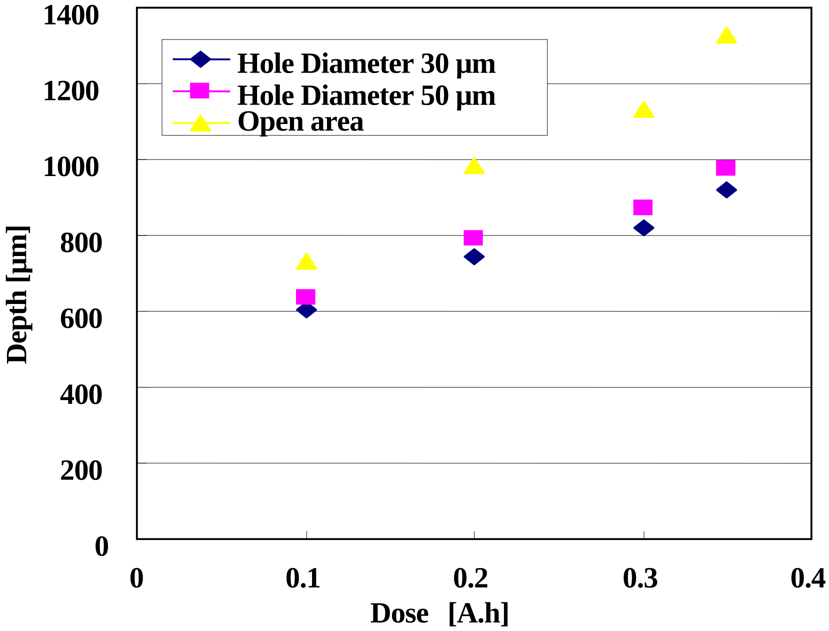


Fig. 9 Dose data for the hole-exposure for 10 hours development at 50 ºC

The final result of microneedle fabrication with through-hole was achieved as shown in Fig. 10. The microneedle with length of 400 µm, 30 µm hole-diameter and 300 µm of the chip thickness was succeeded. The improvement also shows that the aspect ratio of microneedle hole was enhanced from 20 to 23.

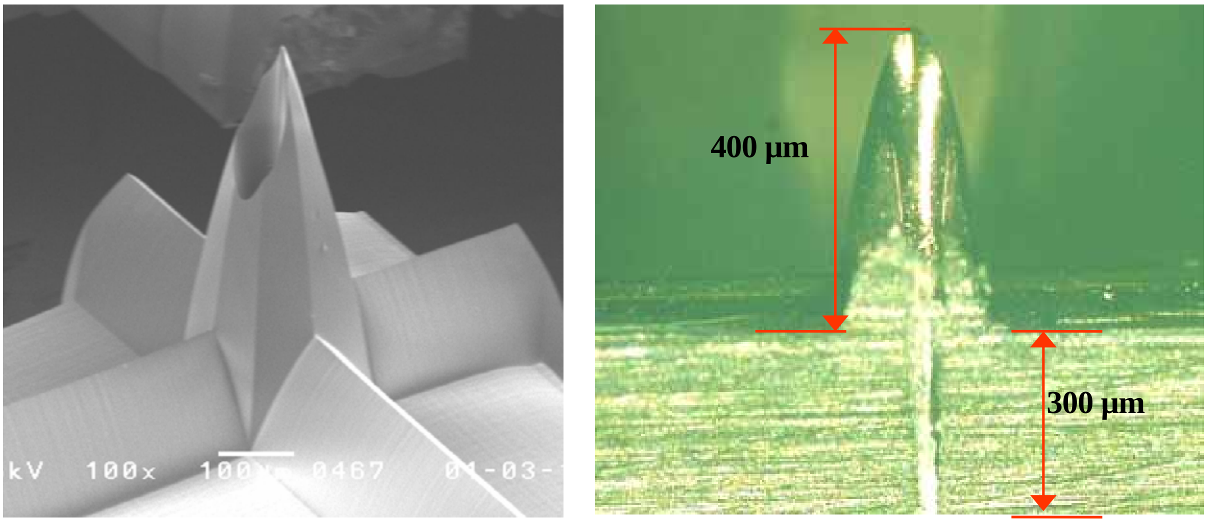


Fig. 10 Microneedle with desired configuration and completely through hole

4. Conclusion

Microneedle arrays are important in Bio-medical applications, especially drug delivery systems. We have reported that the optimal configurations of microneedle shapes and the location of hole were necessary for the penetration into the skin. However, a deep though-hole of microneedle is one of the key successful in a flow of drugs delivered to the body. There are a number of influences which cause the difficulties in fabrication of through hole. The influences affected during the exposure and development. Since up to present, the description of several influences during the fabrication process of microneedle hole has not yet been available, we attempted to develop a useful method and avoid those influences. In this work, we have succeeded in fabrication the microneedle with through hole as desired shape.

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3D PTFE Structures Fabricated by PCT (Plane Pattern to Cross Section Transfer) technique

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Abstract

Microfabrication technique using Synchrotron Radiation (SR) ablation for three dimensional PTFE(polytetrafluoroethylene) structure is reported in this work. We have investigated the necessarily fundamental data for the fabricated PTFE structure e.g. the processed depth and the etching rate. Moreover, the exposure energy distribution was given to the surface of polytetrafluoroethylene by using the X-ray lithography with PCT (Plane Pattern to Cross Section Transfer) technique to fabricate the three dimensional microstructures. To establish three dimensional configurations of a highly accurate PTFE processing technology, the research on parameters is expected to suppress the deformation of micrstructure by chemical development effects as occurred in PMMA(Polymethylemethacrylate). The application of PTFE microstructure can be either in μ-TAS (Micro Total Analysis System) for Bio-medical applications or a master structure for a metal mold fabrication.

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1. Introduction

Polytetrafluoroethylene (PTFE), a material in fluoroplastics group is remarkable as its excellent material characteristics e.g. high-voltage insulation, chemical and creep resistance, thermal stability, etc. Centering on the nonstick frying pan, the filter, etc., PTFE is used in broad fields, such as household articles, OA equipment, a semiconductor, and automobile. As for PTFE, the innovations developed with its outstanding characteristics have been reported continuously for more than 60 years. However, microfabrication of PTFE is considered as a tough technology. Due to the excellent chemical resistance property, it is impossible to fabricate wet etching with chemicals (acidic, alkali) used in a number of microfabrication. Among the methods of PTFE fabrication, using synchrotron radiation ablation, an innovative technique has been developed for a predictable 3-D microstructure.

In 1996, the technique of PTFE direct etching by Synchrotron Radiation (SR) light was firstly reported [1]. The technique was a completely dry etching which the use of wet etching chemicals was not required, unlike in X-ray lithography. Using this technology, it is possible to fabricate 2.5-dimentional microstructures which has high aspect ratio of over 50. However, to apply the microfabrication of PTFE to <u>M</u>icro <u>E</u>lectro <u>M</u>echanical <u>S</u>ystem (MEMS), the method for fabrication of three-dimension microstructure with a complex form such as sloped side-wall or free-form surface is demanding. Therefore, we have investigated a method for fabricating microstructures with an controllable side-wall by exposed energy distribution to the surface of PTFE while the resist stage is moving (dynamic exposure).

2. Fabrication and Experimental Conditions

A number of experiments were carried out using beam line number 15 (BL-15) at the superconductivity compact synchrotron radiation (SR) source "AURORA", at the SR center, Ritsumeikan University, Japan. The vacuum atmosphere at 10^{-5} Torr was provided and a substrate heater was equipped in the chamber of BL-15. Generally, in the PTFE fabrication process using SR ablation, it is noted that the etching rate becomes greater when PTFE is etched in a vacuum chamber and heated during the ablation [2]. In addition, a stage system with high flexibility is in the chamber of BL-15, and fine drive control is also possible by PC control respectively. The outline figure of Bl-15 is shown in Fig.1.

The properties of SR at AURORA are, wavelength of 0.15 nm range to visible light range, applied electron energy and the maximum storage current in the experiment were 575 MeV and 300 mA, respectively. The light from AURORA penetrates two 200μm Be windows, and uses within the chamber the light which has a 015 to 0.95nm wavelength domain.

The X-ray mask consists of a Polyimide membrane with a thickness of 50 μm and an Au absorber with a thickness of 3.5 μm. PTFE used the thing of 1mm thickness of Yodogawa Hu-tech Co.,Ltd. A processing mechanism is shown in Fig. 2. If PTFE is exposured by SR light, PTFE will cause a photochemical reaction and main chain of PTFE is decomposed. And it changes to fluorocarbon gas and the exposed part is etched. If the temperature of PTFE is high at this time, the secession rate of fluorocarbon gas can be promoted. In order to prevent polluting a mask and Be window by fluorocarbon gas, a 25μm polyimide film was attached before each. An outline figure is shown in Fig. 3.

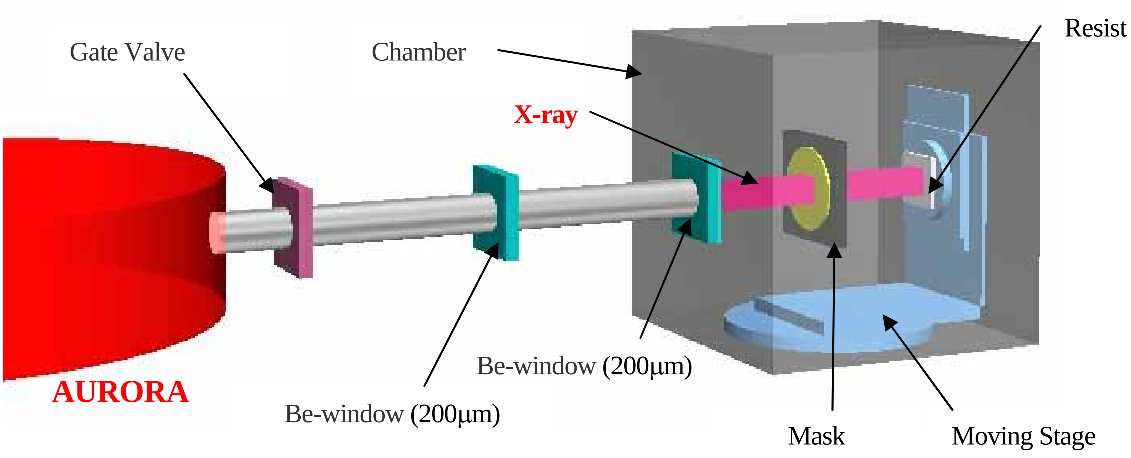

Fig. 1 The schematic of BL-15 and the stage system

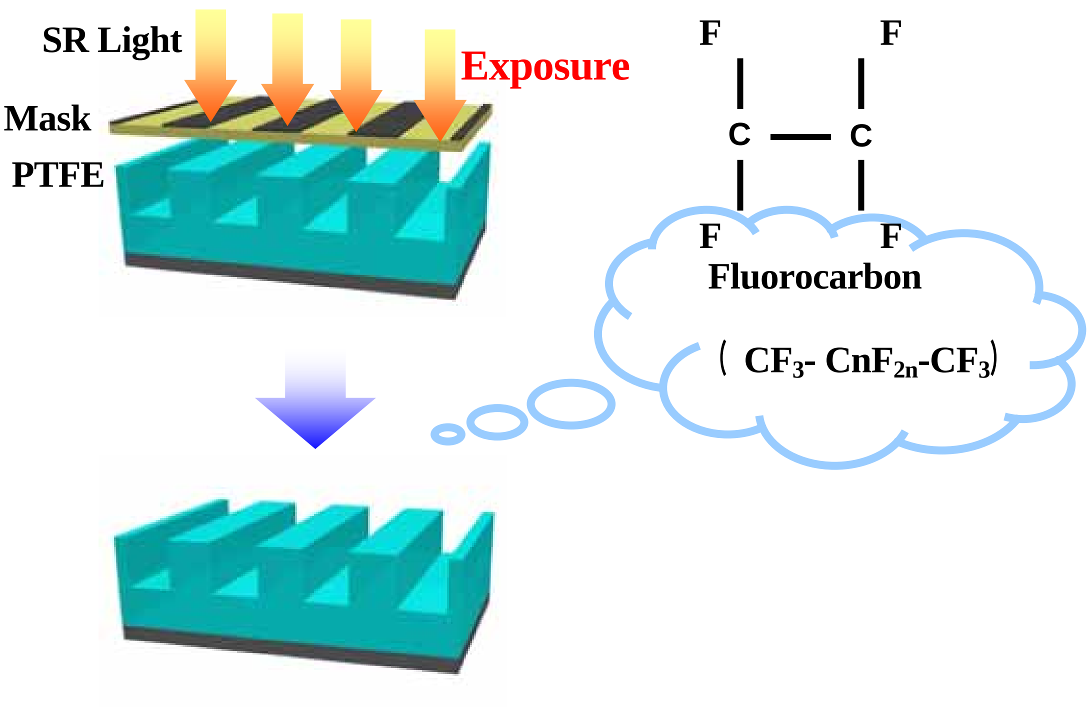

Fig. 2 PTFE etching mechanism

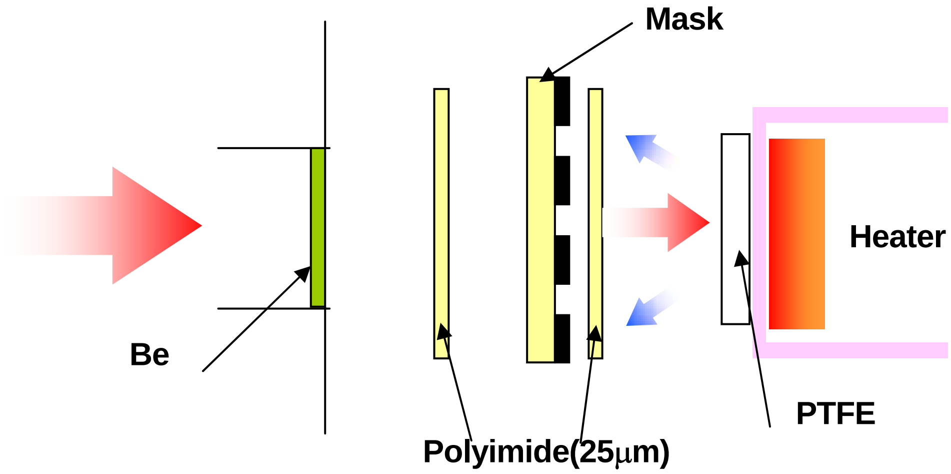

Fig. 3 The schematic of experimental system

The Plane-pattern to Cross-section Transfer (PCT) technique was used as an exposure method controlled by giving exposure energy distribution [3]. Since PCT technique has successfully provided an arbitrary PMMA three-dimensional structure, we have recently begun to adapt PCT for exposing PTFE while the stage was moving. Fig.4 introduces the PCT technique by a group of triangular masks. In the case of PCT when PTFE is exposed, resist is scanning. During an exposure, 2-D configuration of a mask pattern is transferring to a 3-D structure whose cross-section shape is similar to that of the mask pattern. In addition, more complex shape can be fabricated by exposing it again by rotating resist by 90 degrees after the exposure first.

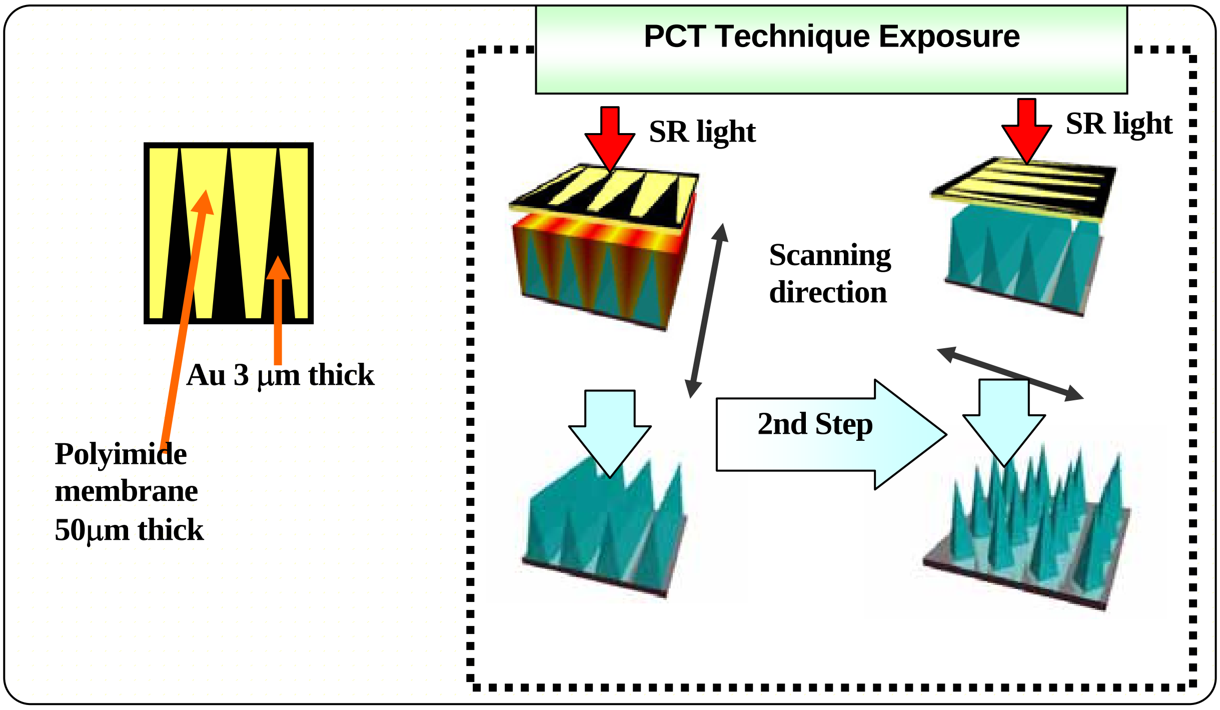

Fig.4 PCT technique exposure

3. Results

3.1 Fundamental data about PTFE

The fundamental data of PTFE was investigated. PTFE was exposed by SR light using the mask pattern (1500 μm x 30 mm) shown in Fig. 5. Figure 6 shows the relation between the beam current and the processing depth [exposure time 30 minutes when changing PTFE temperature with 110°C, 140°C, 170°C and 200°C, respectively. Fig. 7 shows the relation between the beam current and the processing depth [PTFE temperature with 200°C when changing exposure time, 10min, 15min and 30min, respectively]. Fig. 8 shows the relation between the dosage, and the processing depth.

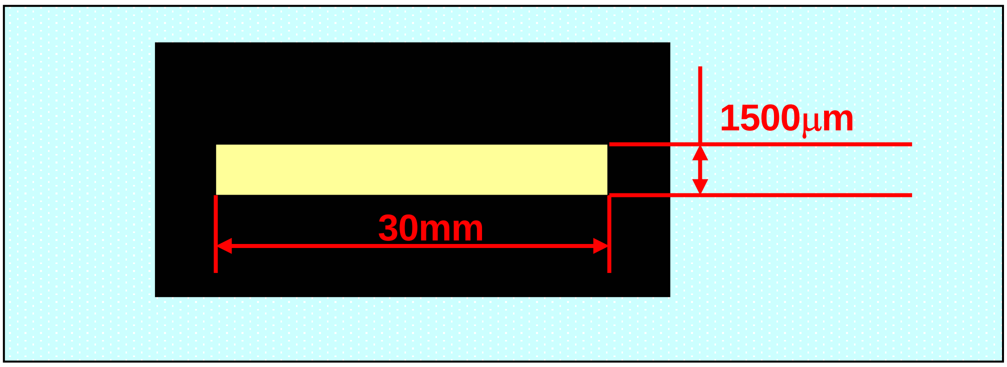

Fig.5 mask pattern and PTFE surface

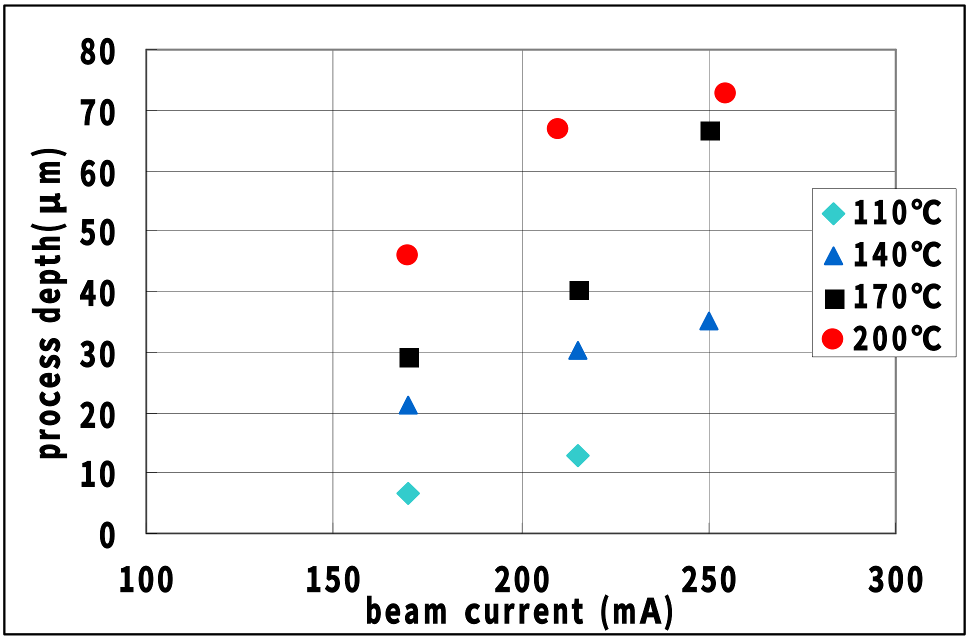

Fig.6 beam current VS the processing depth (30min)

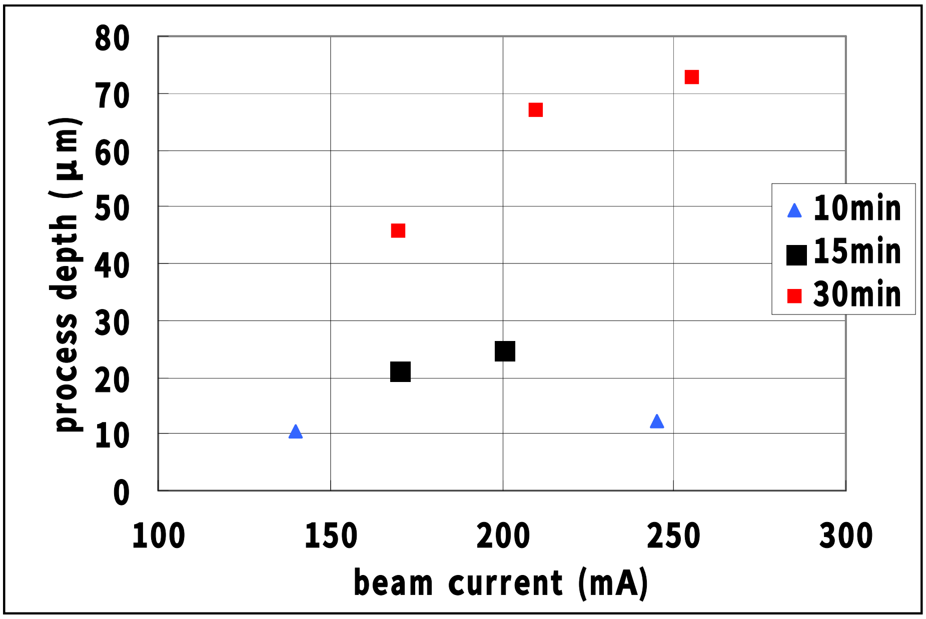

Fig.7 beam current VS the processing depth (200°C)

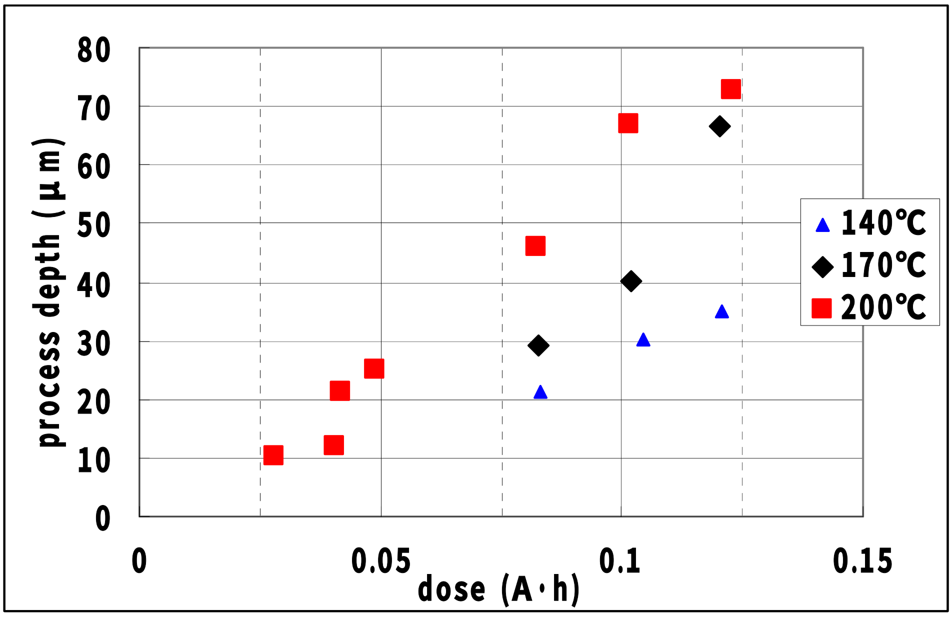

Fig.8 dosage VS processing depth

3.2 3-D fabrication results

The SEM photos of the microstructure exposed by using a triangular mask pattern at the PTFE temperature of 140 °C is shown in Fig. 9. Moreover, the SEM photos of the structure exposed by using a same triangular mask pattern at the PTFE temperature of 200 °C is shown in Fig. 10. In addition, exposure time was determined so that the height of a structure might be set to around 80 μm.

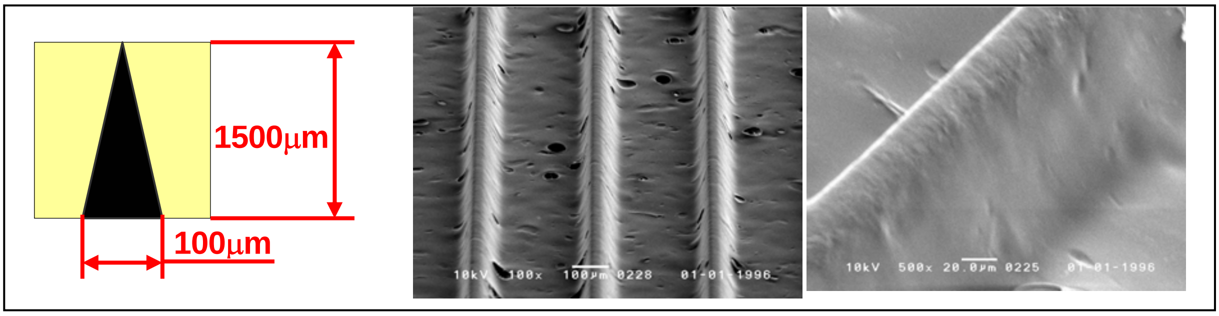

Fig.9 Mask pattern and SEM images of resulting structure from triangular mask (140°C)

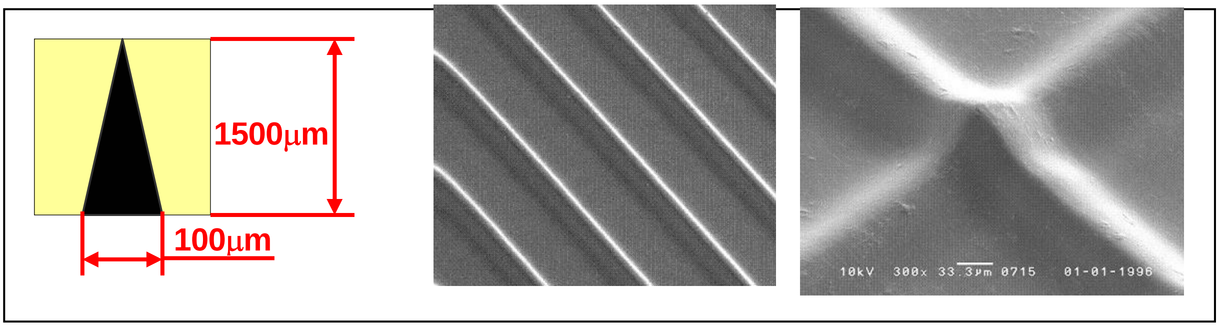

Fig.10 Mask pattern and SEM images of resulting structure from triangular mask (200°C)

The result when using the semicircular mask at the PTFE temperature of 200°C is shown in Fig. 11. The result when using the sine-curved mask at the PTFE temperature of 200 degrees C is shown in Fig. 12.

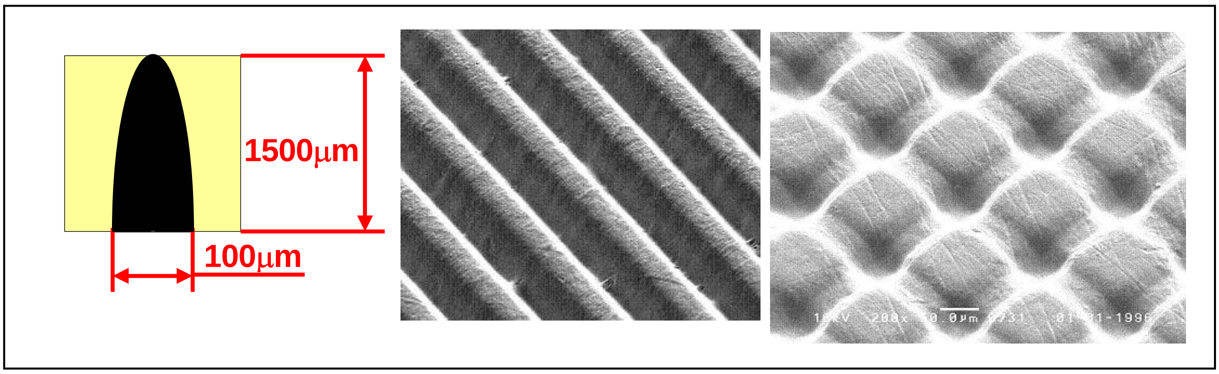

Fig.11 Mask pattern and SEM images of resulting structure from semicircular mask

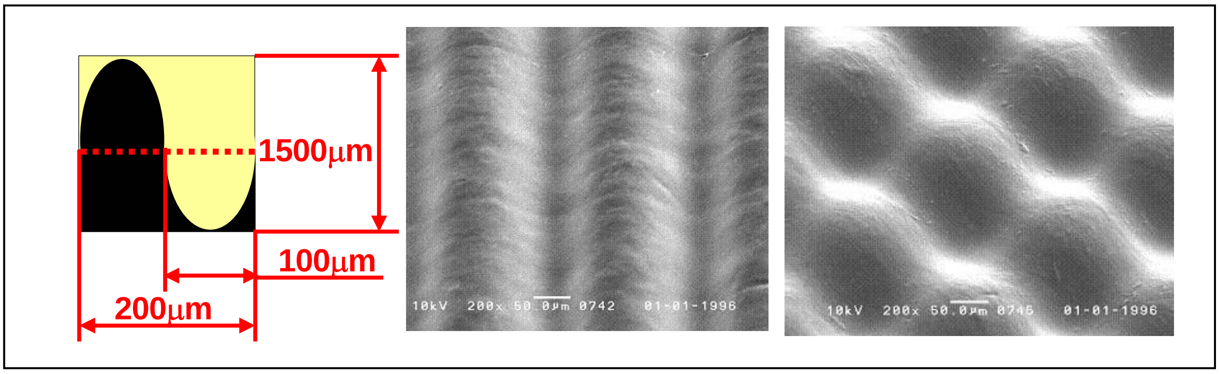

Fig.12 Mask pattern and SEM images of resulting structure from sine-curved mask

5. Conclusion

PTFE shows excellent material characteristic especially chemical resistance, so these researches will make to apply fabrication of μ-TAS (Micro Total Analysis System) for Bio-medical applications. In addition, this technology will be expected not only as the parts of microdevices, but also matrix structures for the metal mold fabrication.

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SR Lithography for Optical Device Applications : Sub-wavelength Structure and Diffractive Optical Elements

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Abstract

This paper introduces the 3D-micro/nano optical devices such as applying to Diffractive Optical Element (DOE), Wavelength Division Multiplexing (WDM) device, Sub-Wavelength Structure (SWS), as the examples. The 300 nm and 500 nm pitch grating with high aspect ratio were fabricated by a static X-ray lithography. The 500 nm-pitch grating has the aspect ratio of about 10, and 300 nm-pitch grating has the aspect ratio of about 7. The blazed grating was fabricated by a dynamic X-ray lithography so called Plane-pattern to Cross-section Transfer (PCT) technique. The PCT method was also performed with various proximity gaps ranking from 30 µm to 60µm to fabricate a sub-wavelength structure.

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1. Introduction

Recently, the information infrastructure has been growing higher and higher. The computer systems and the optical fiber net require a high-speed and mass system. There is a number of research and development in the field of optical element such as the interface element between electrical and optical, the wave front control elements, etc. The fabrication technology of an optical element is the micromachining or optical lithography. However, the production of the structure that has the narrow pitch and high aspect ratio is difficult. Therefore, SR lithography was employed to produce these optical elements. In addition, a mass production is also possible by the use of replication technology [1, 2]. As for the SR lithography, x-ray has a high penetration rate. The thick resist can also be exposed. SR beam is straight penetration, the various thickness of substrate and high aspect ratio structure can be exposed. Not only the grating that has two dimensional structure of wavelength size (hundreds of nanometer up to several microns) but also the type of blazed grating can be fabricated using this technology. SR lithography can also be employed for a fabrication of the sub-wavelength structure used for the anti-reflection film.

The diffraction optical element (DOE) is an accurate device for controlling and operating information, the wave front, wavelength, the inclination, and these complex functions by using of light. DOE can be classified into four: the lens function element, the branching/combination wave function element, the optical strength distribution change element, and the wavelength filter function element. Only optical combination/branching filter can use twice or more diffraction light, the refraction and reflection element cannot be achieved. In this work, the diffractive optical elements for planar grating lens, the blazed grating for optical combination/branching filter, and sub-wavelength structure are introduced.

2. Fabrication methods

2.1 Nanograting

The diffraction optical element is used as a lens for reading or writing the signal from the optical disk that installed as the optical pickup. Grating that has a high aspect ratio and near a suitable pitch for wavelength 405 nm of Blue-Ray next generation DVD standard was produced. However, the lens corresponding to two or more wavelength is required if the interchangeability between media with a different standard as a picking up lens is taken into account. It is necessary to produce the structure with a high aspect ratio to raise the diffraction efficiency by two or more wavelength. Deep X-ray lithography was employed in fabrication of the grating with process improvements by the influences of some parameters e.g. X-ray dose, developing time and PMMA casting conditions.

2.2 Blazed Grating

Since in a slit grating as above mentioned, light passes to many diffraction orders simultaneously, half of the amplitude/a quarter of the intensity goes straight through without being diffracted. In order to solve this problem, the blazed grating that its inclined surface opened for the light and put most of the light into one diffracted order was produced. Plane pattern to Cross section Transfer (PCT) technique [3] was used as an extension method of the conventional X-ray lithography for realizing a complete 3-Dimensional structure. The X-ray mask was made of Ta absorber with a thickness of 0.5 µm. The submicron mask patterns were formed on a 2.2 µm-thick silicon nitride (SiN) membrane as a set of triangles placed in a double row facing each other as shown Fig.1 where the light-gray area is Ta absorber, and the black area is SiN membrane. The designed mask for blazed grating has a set of right triangles placed in a double facing row. Fig. 2 shows the schematic of PCT process. The mechanism is, when X-ray transmitted through the mask membrane and absorber, if the x-ray resist moves, the 2-D pattern on the mask will be exposed repeatedly and deposited dosage on the profiled area will form the 3-D structure on the resist with the similar cross section to the mask pattern.

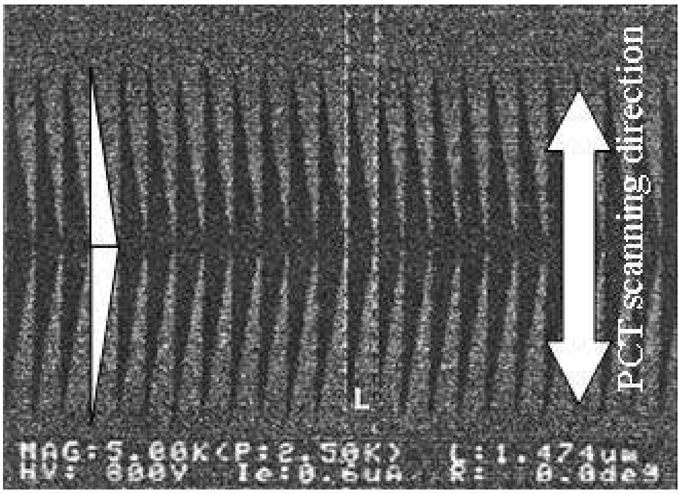


Fig. 1 The SEM image of x-ray mask pattern for blazed grating

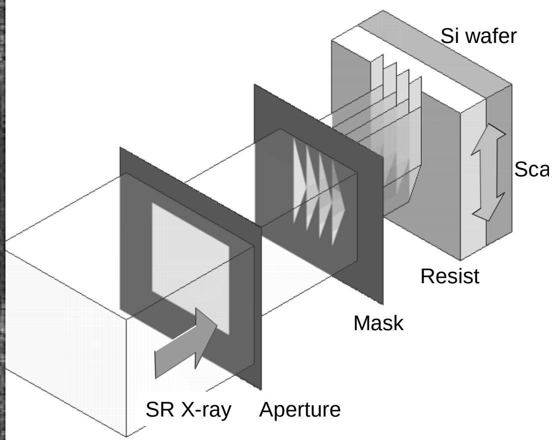


Fig. 2 The schematic of the PCT exposing process

2.3 Sub-wavelength Structure

The PCT method has also been applied to the fabrication of a sub-wavelength structure. Fig. 3 shows a schematic of an x-ray exposure and axes guides. The SR light was exposed to the resist while expanding the distance between mask and resist which is called the proximity gap. The diffraction influence is generally raised by increasing the proximity gap. [4-6] Therefore, the small proximity gap is assumed to be applicable for fabrication of tiny structures such as electric circuits, micro/nano parts for Micro Electro Mechanical Systems (MEMS). Although this process is not complicate since only the movement of resist stage in Z-axis was designed, the collected data for process requirement and simulation is useful for fabrication of a number of 3D-micro/nanostructures.

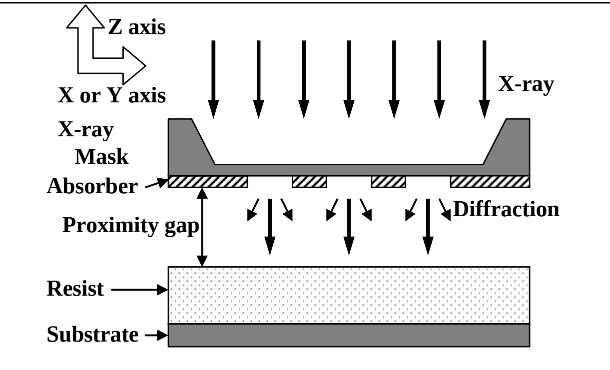


Fig. 3 The dimension of exposure

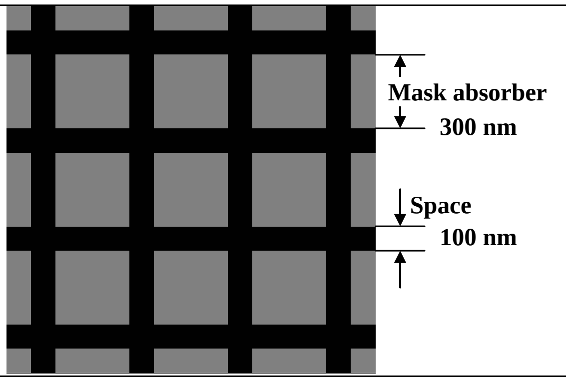


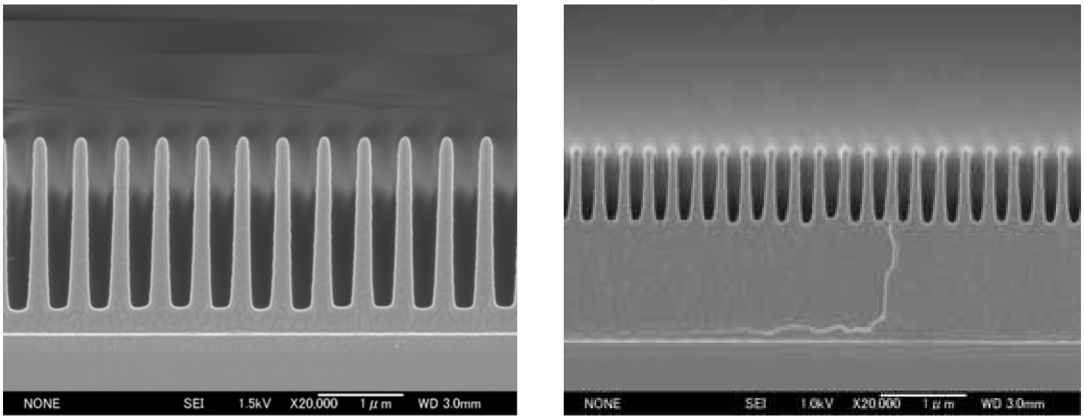
Fig. 4 The mask pattern

Fresnel and Fraunhofer diffractions provide 3D energy intensity distribution in the resist at the expanding proximity lithography method. The influence of diffractions was simulated, and shown in the form of intensity distribution. The diffraction energy spectrum at the surface of resist without any influence was calculated from beam line parameters. The parameters of the mask used are 100 nm-width and 400 nm-pitch as shown in Fig. 4. The x-ray wavelength is between 0.1 nm to 0.6 nm at peak of 0.35 nm. Main parameters of diffraction simulation related to mask are the slit width and slit pitch.

3. Results and Discussion

3.1 Nanograting

The 300 nm and 500 nm pitch grating with high aspect ratio were produced as shown in Fig. 5. These gratings made from such a condition: exposure dose was 0.0065 A·h for 500 nm-pitch and 0.0050 A·h for 300 nm-pitch, proximity gap was 20 μm, and it was developed by GG developper at 23°C for 10 min. The SEM images of each grating are below.



(a) (b)

Fig. 5 The grating with (a) 500 nm pitch and (b) 300 um pitch

The 500 nm-pitch grating has such a property: the height of line was 2070 nm and line width was 208 nm which made the aspect ratio of about 10. The 300 nm-pitch grating has such a property: the height of line was 910 nm and line width was 128 nm which made the aspect ratio of about 7. These grating were in the area of 2×2 mm^2.

3.2 Blazed Grating

The fabricated blazed structure was shown in Fig. 6. This grating made from such a condition: exposure dose was 0.0028 A·h/area, proximity gap was 100 μm, scanning speed was 0.00025 mm/sec and it was developed by GG developper at 37°C for 10 min.

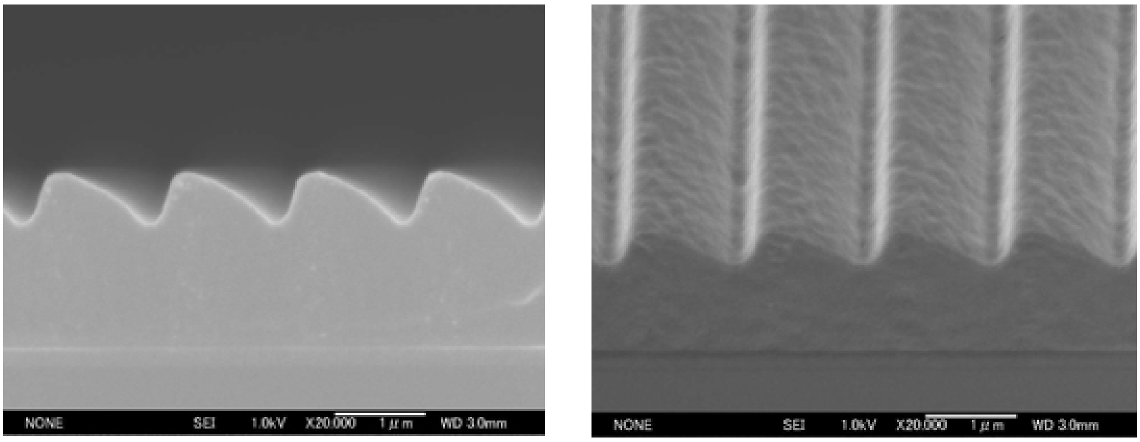


Fig. 6 The blazed grating with pitch of 1.5 μm

The triangular pattern was successfully transferred, but the vertical side of the triangles was deformed as a slope, and each tip of peak and valley are not sharp tip.

3.3 Sub-wavelength Structure

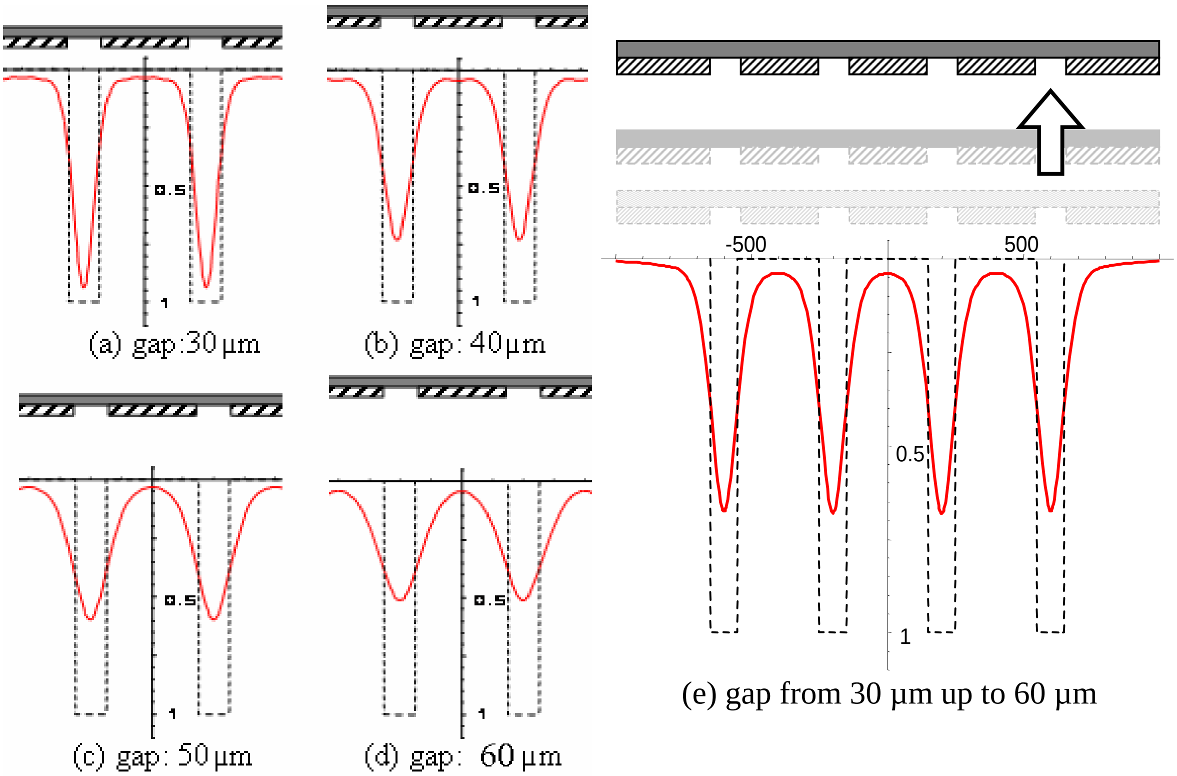
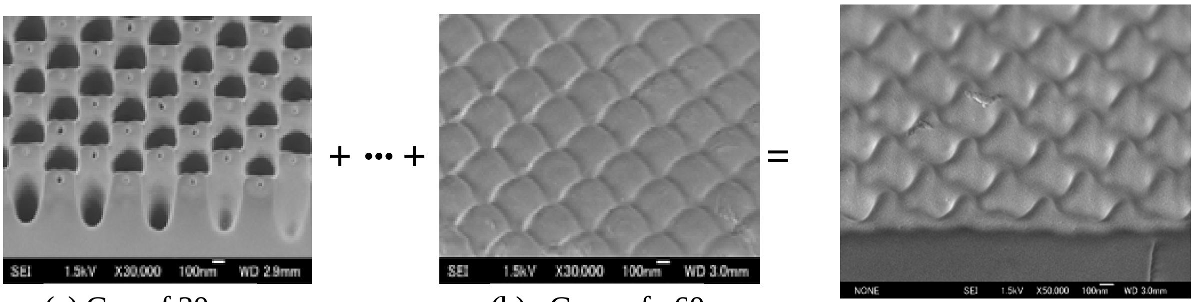


Fig. 7 The simulation results of x-ray intensity distribution

The influence of diffraction and the proximity gap are shown in Fig. 7(a) - (d). The gaps used in the simulation were at 30 µm, 40 µm, 50 µm, 60 µm. As a result of the simulations, the intensity distribution is appeared to be the shape of Fresnel diffraction at the 30 µm gap, and that of Fraunhofer diffraction at 60µm gap. During the expanding proximity gap, the simulation result of aerial intensity distribution is shown in Fig. 7(e)

The fabrication was performed at the same condition as in the simulation. Fig. 8(a) and 9(b) are the results of exposure at the gap of 30 µm and 60 µm respectively which are corresponding to the results of the simulation properly. In addition, the result after applying the method to scanning exposure in Z-axis is shown in Fig. 8(c).



(a) Gap of 30 µm (b) Gap of 60

Fig. 8 SEM photographs of 3D surface submicron array.

4. Conclusion

The 300 nm and 500 nm pitch grating with high aspect ratio were produced as nano grating. The blazed grating was fabricated with PCT technique. According to the result, Z-axis scanning exposure can be expected to be available for making various 3D micro/nanostructures, such as SWS. In the future if technologies are combined, SWS will be produced on grating so that it will be possible to develop more efficient diffraction gratings.

Moreover the structure of these high aspects can be applied to not only optical device but also MEMS sensors and actuators.

References

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