A study to reduce surface roughness of a PMMA sheet in the X-ray lithography technique

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Abstract

The conventional LIGA process generates a lot of different kinds of defects on a PMMA sheet after development, which causes a serious problem for fabricating fine structural patterns. A new type of PMMA sheet has been developed, which is composed of double layers with different polymerization. We proved that the PMMA sheet has a highly smooth surface and the etching depth is controllable. Possible applications for optical devices, such as diffraction grating and zone plate, are also addressed.

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Introduction

The LIGA process consists of lithography, plating and mold technology, as shown schematically in Fig.1. X-ray lithography is the process to make some pattern on a photoresist by exposing synchrotron radiation with an X-ray mask. Although X-ray lithography is used popularly in the LIGA process, it might be important to come back to its original role.

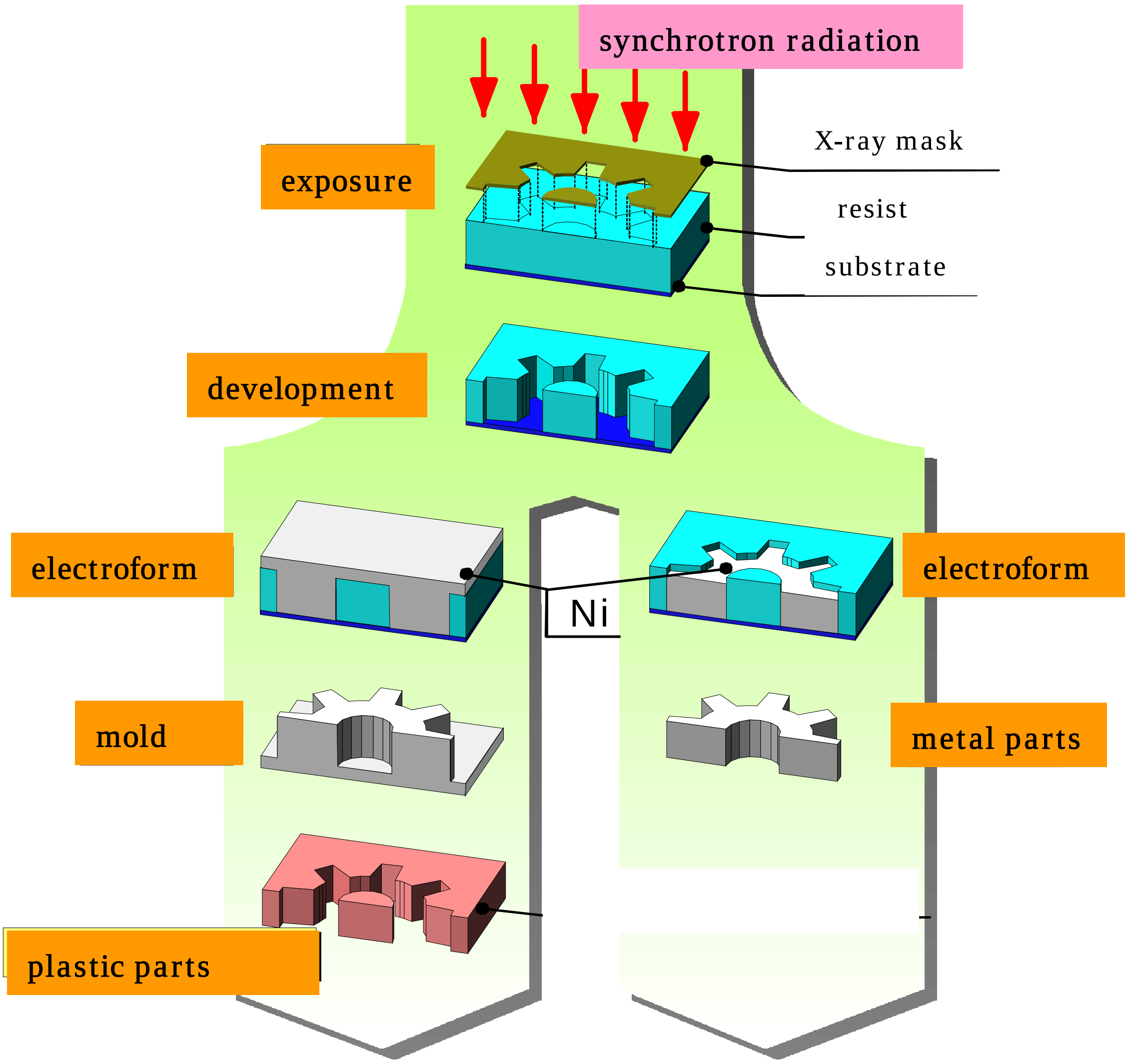

Fig.1. The conventional LIGA process

In the optical industries, X-ray lithography technology has been thought very promising to fabricate optical elements, such as diffraction grating, zone plate and reflection-less film, because of the following advantages.

a. As the resist material, highly transparent PMMA can be used
b. PMMA is a positive-type resist and the exposed area is removed at the development process

c. Several micron thick ditches or concaves should be fabricated for optical elements. This is possible by X-ray lithography for a few minutes exposure.
d. Sub-micron spatial resolution is necessary for optical elements. This is also possible by X-ray lithography.

Basic idea of the process is shown schematically in Fig. 2; (a) choose a PMMA substrate, (b) expose with soft X-rays, (c) develop and (d) separate the substrate. Obtained pieces can be used as optical devices, as shown in Fig. 3. To decrease the cost, manufacturing process should be simple.

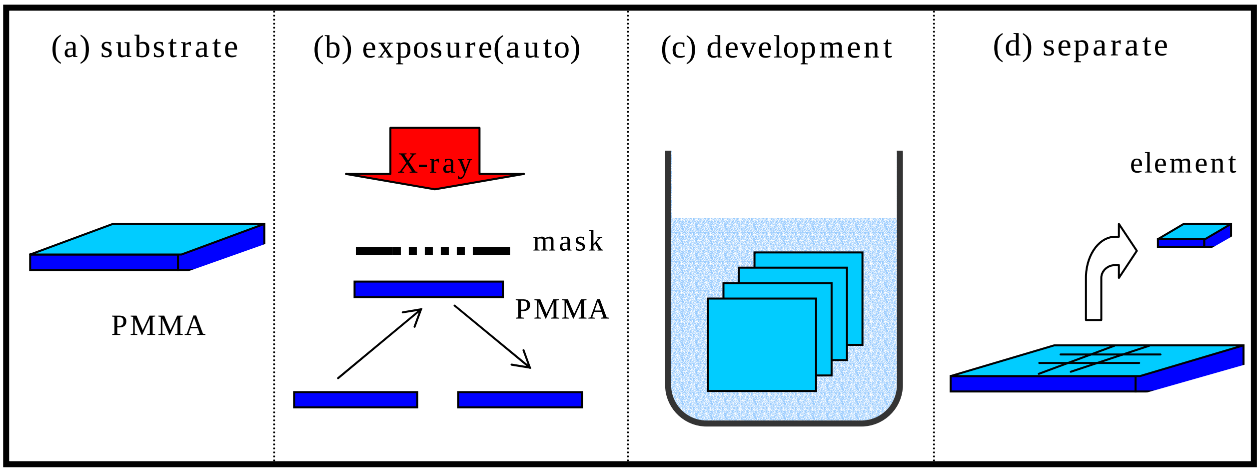

Fig.2 . Basic idea of the process

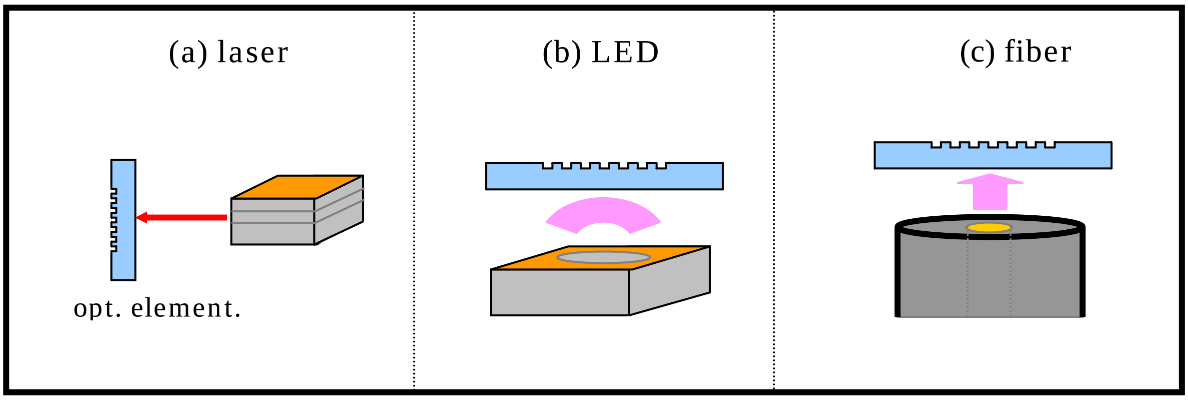

Fig.3. Possible applications

The quality and productivity are important in manufacturing products. In this paper, it is discussed how to prepare a smooth surface in the X-ray lithography, which can be used for optical devices in a specific field. Necessary specifications are micro fabrication, shape, surface roughness and control of etching depth mainly. Improvement of each specification is discussed in the present paper.

Experiments

The experiments were carried out at BL6 in the Ritsumeikan University SR center. We prepared several kinds of photoresist to study the relation between the surface roughness and the resist materials. These resist materials were exposed by soft X-ray (1~7 keV) and immersed in an organic solvent, called 'GG etchant' at 40 °C with the target depth of about 50 μm.

Results and Discussion

A. Surface roughness

Surface of PMMA films after the conventional process looks apparently fine, but detailed looking of the surface with differentiation polarized light shows several ditches and holes, as shown in Fig. 4.

There might be four possible factors which cause surface roughness; (1) radiation exposure process, (2) PMMA material itself, (3) X-ray exposure and (4) development conditions.

Factor (1) mostly comes from non-uniform Be window, membrane of X-ray mask and adhering dust on a PMMA and mask.

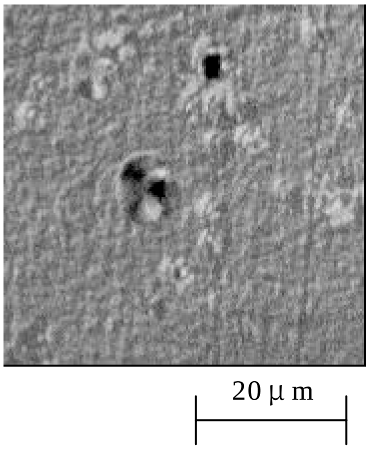

Fig.4. PMMA surface obtained by the conventional process taken by differentiation polarized light.

Factor (2) is illustrated in Fig. 5, in which the photo after development is shown without X-ray mask. Diffused spots disappeared, but a concave with a hole is observed and the surface is still rough.

We suppose that the concave was originated by a crack or defect in the commercial PMMA sheet, since it appears at the same position of the defects before etching. Carefully observation revealed that the rough surface is due to a thin film (about 10 μm thick) on the commercial PMMA.

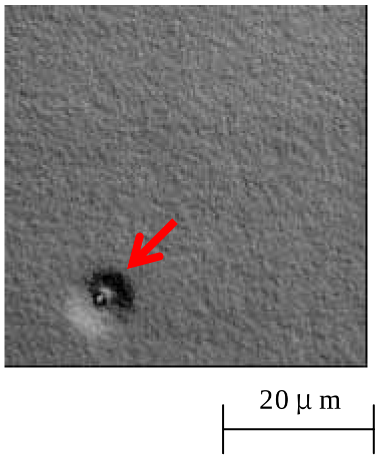

Fig.5. PMMA surface without X-ray mask.

To examine factor (3), we prepared a sample, in which about 3 μm thick PMMA film was coated on a Si substrate. A 10 weight% PMMA resist (MicroChem. Corp.) was used and coated on Si substrate by spin coater and annealed at 160 °C for 1 hr. The surface was observed after a little exposure and development, as shown in Fig. 6, where the etching depth is about 1 μm, similar condition to Fig. 5. The surface was smooth before development but the surface was still rough due to etching. This is considered that

PMMA damage by X-ray was revealed at development. Above result suggests that only etching an X-ray exposed PMMA is not enough to prepare a perfect smoothing surface and some improvement in the PMMA material itself is necessary.

Thus, we inserted an X-ray resistant polymer beneath the etched PMMA. It is expected that etching stops at the interface, where a smooth surface can be obtained. The basic concept is illustrated in Fig. 8. We used PMMA with high polymerization as X-ray resistant polymer in this experiment. Molecular weight of the lower layer PMMA was 1,540,000, and the upper one was 130,000. As shown in Fig. 7, a very smooth surface was obtained when the etching stopped at the interface. In addition, it is necessary to optimize the exposure dose and the development conditions.

Fig.6. PMMA resist

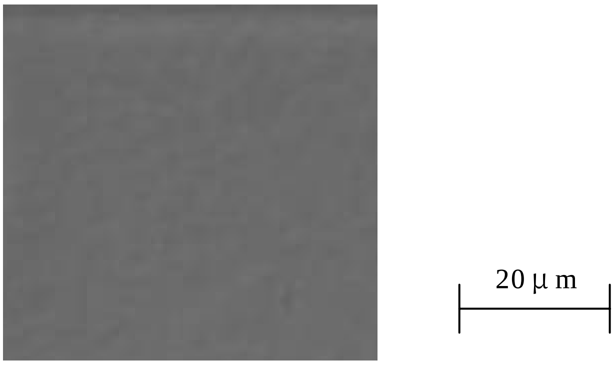

Fig.7. improved surface

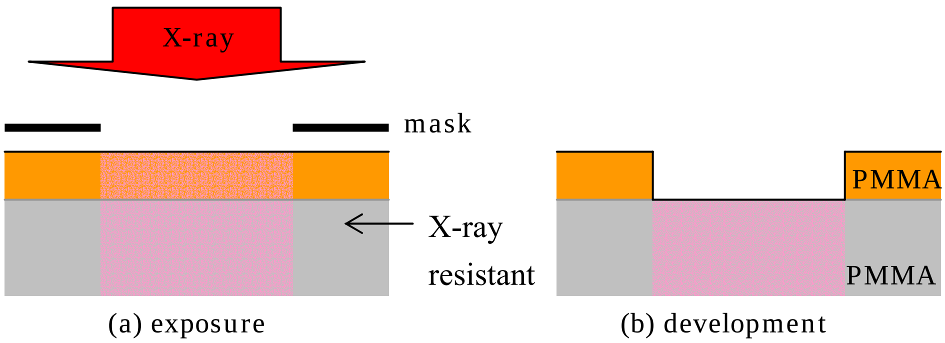

Fig.8. The concept to obtain a smooth surface

B. Depth control

In fabricating optical elements, to control the etching depth is an important requirement. Unlike the usual LIGA process using Si substrate, it is expected that this process conditions must be managed. But there was not in detail data of several-micron order up to now, and I studied the etching depth change as functions of the exposure dose and the development conditions using commercial PMMA sheets. Figure 9 shows the relation between the depth and development time at the exposure dose of 4.4 and 8.5(mAh/cm).

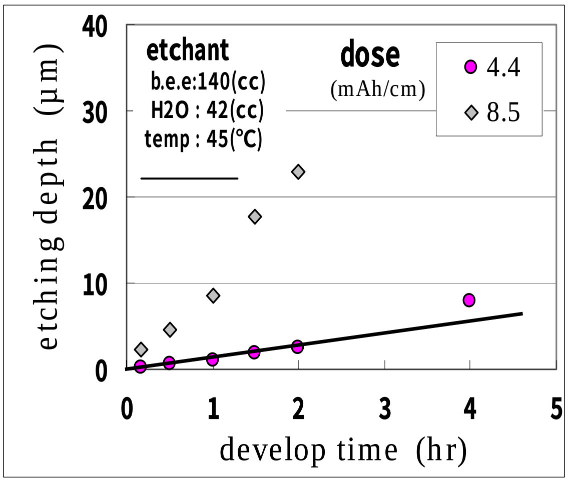

Fig.9. Etching depth dependence on the developing time for the commercial PMMA

In case of low exposure dose, the etching depth increases linearly with the development time. Temperature effect at the development process was also examined. We found that the temperature affects very sensitively and should be controlled precisely.

Etching efficiency for PMMA materials was studied and found to be greatly dependent on the manufacturing company. Such a difference might come mainly from the polymerization level of PMMA. Accordingly, we prepared a double-layer PMMA sheet and examined the etching rate. In this process, a PMMA resist was spin coated on the commercial PMMA sheet, whose molecular weight was 1,540,000. A molecular weight of the coated PMMA is 130,000 and the thickness can be controlled by the rotation speed of spin coater. Figures 10 and 11 show the results for the PMMA resists with different thickness.

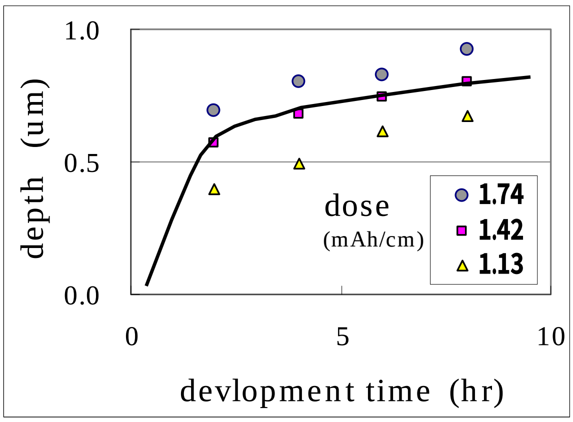

Fig.10 Etching depth dependence on the developing time for the 0.7 μm thick PMMA

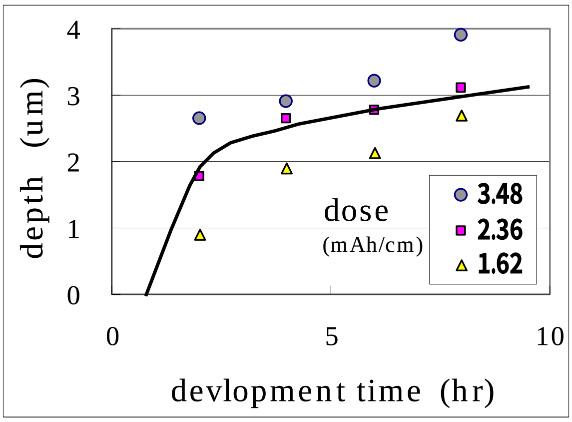

Fig.11. Etching depth dependence on the developing time for the 2.6 μm thick PMMA

In case of Fig.10, the thickness of the soft PMMA was 0.7 μm, and it was 2.6 μm for Fig. 11, in which etching depth is plotted as a function of development time for three different doses. The etching rate difference between the upper and lower PMMA sheets was clearly seen in these figures. Though etching does not stop at the interface completely, these results prove it possible to control the target depth within ±10%.

C .Micro fabrication and shape

For the visible optical elements, we should fabricate sub-micron structures with sharp edges. They are strongly dependent on the X-ray's wavelength, X-ray mask structure and the gap between X-ray mask and the resist. Optimum conditions have been studied by Sugiyama Lab. Ritsumeikan University. For example, an array of lines with 0.25 mm thick and 1.7 mm high was prepared on a Si substrate [1]. This clearly shows that the X-ray lithography technique is enough to make visible optical elements.

Summary

Surface roughness of the PMMA sheet after development is a serious problem in the X-ray lithography technique. The roughness is much improved by forming a double layer PMMA film with different polymerization by spin coating of the PMMA resists. Etching depth is also controllable for the double layer film. This improvement of the PMMA sheet will enable us to apply the X-ray lithography technique to new fields, such as optical devices.

References

[1] F.Kato, S.Fujinawa, Y.Hotta and S.Sugiyama: Memors of the SR Center, Ritsumeikan University, June 2007, No.9, pp.25-30 (2007.6)