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Fabrication of intermediate mask for deep x-ray lithography

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Abstract This paper presents the fabrication of intermediate x-ray mask for deep x-ray lithography. In order to have working mask with absorbers thickness larger than 10 μm, the intermediate mask should have absorbers of 0.7 μm in thickness. To demonstrate intermediate mask fabrication, x-ray zone plates are fabricated on the 1.2 μm low-stress silicon-rich silicon nitride (SiN_x) membrane with the tri-layer Chromium-Tungsten-Chromium (Cr–W–Cr) as the x-ray absorbers. The chromium layers both 200 angstroms are used as adhesion and for stress relief. The SiN_x film is deposited with low pressure chemical vapor deposition (LPCVD) and the free standing membrane are formed by KOH silicon backside etching. With the e-beam lithography and reactive ion etching, width of 0.8 μm of outmost zone of the x-ray zone plates has been achieved on the membrane. The scanning electron microscopy (SEM) images of the x-ray zone plates and pictures of intermediate masks are demonstrated.

1
Introduction

For the third generation synchrotron radiation light source with high brightness, small emitting source size and high spatial coherence light, many research perspectives in x-ray region have been reignited [1], especially LIGA in Europe [2]. For deep x-ray lithography LIGA process, the working mask should at least has absorbers of 10 microns thick to expose high aspect ratio structure like nozzles. There are several ways to prepare the working mask. For example, multi-exposure method and UV lithography [3]. For multi-exposure technique, the alignment is a tough task to deal with. For UV lithography, it can only prepare mask with 100 microns structure but the resolution is quite limited. In Synchrotron Radiation Research Center, the storage ring can ramp the electron energy up to 1.5 GeV with 240 mA beam current. The highest photon energy will be in the range of 0.6 nm to 0.4 nm [3]. So, the intermediate mask is adopted for working mask preparation.

For x-ray masks, thinner absorbers have been fabricated for 1:1 proximity x-ray lithography in semiconductor applications [4, 5]. In the case of intermediate mask, the 1.2 μm SiN_x membrane is chosen and 0.7 μm of tungsten is deposited as the absorber. For fine pattern definition, e-beam writer was used. Figure 1 shows the contrast of 1.2 μm membrane of 0.7 μm absorber in the range of 300 eV to 5000 eV. In the 1 KeV to 1.5 KeV and 2 KeV to 3 KeV the contrast can be kept larger than 3.1 which will benefit the exposure results for high aspect ratio structure.

2
Experiments

The 100 mm-diameter p-type ⟨100⟩ silicon wafers were cleaned by standard R.C.A procedure and then deposited with a 1.2 μm thick layer of low tensile stress silicon-rich nitride (∼10 MPa) by low-pressure chemical vapor deposition (LPCVD). The LPCVD SiN_x films were accomplished by varying the furnace temperature from 825 °C to 900 °C and keeping the flow rate ratio of dichlorosilane/ammonia at value of 4.5 such that low stress can be obtained. The pressure during deposition was 120 mtorr.

After silicon-rich nitride has been deposited, the membrane window of 3 cm^2 or larger is opened with photoresist mask on the backside of silicon substrate. The lithography step in conjunction with a sequence of backside reactive ions etch (with CF_4/O_2 gas) were used to strip off the nitride. Then, the wafer was immersed in KOH solution (30 wt%, 68 °C) to etch off silicon from the backside and to form the free standing membrane. The backside silicon etching rate is highly dependent to the concentration of solution and temperature of solution. Figure 2 shows the relationship of etching rate with respect to the concentration of solution as well as temperature.

The subtractive process was adopted for absorber patterning. The tri-layer metal film (Cr–W–Cr) was sputtered and the sputtering conditions must be selected carefully to satisfy the stress control such that the formed membrane will not be destroyed by stress of metal film. To satisfy demanded stress, we select recipes with *dc* power 50 Watt, *Ar* gas flow 80 sccm, pressure 10 mtorr for 200 angstroms Chromium layers and dc power 100 Watt, *Ar*, gas flow 80 sccm, pressure 4 mtorr for 0.7 μm Tungsten absorber.

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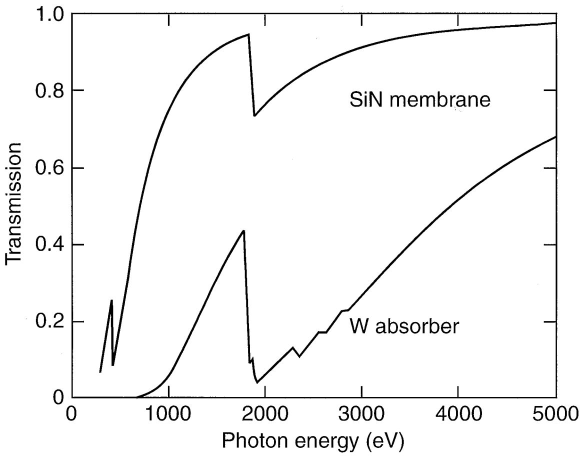

Fig. 1.

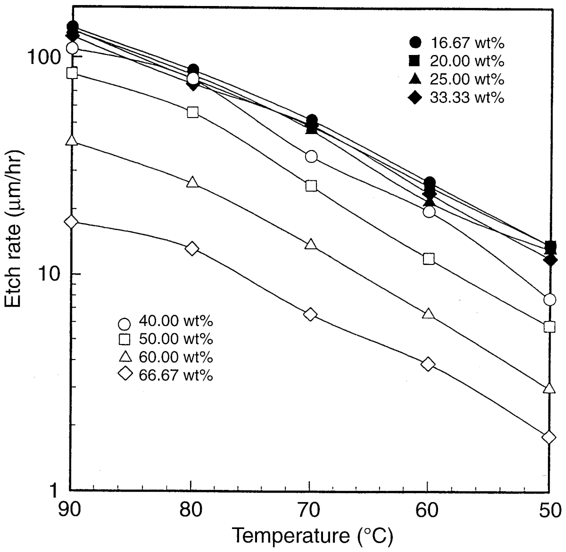

Fig. 2.

The pattern definition is accomplished by E-beam direct writing. The spin speed of coating of photoresist must be carefully controlled to keep the membrane from ruin during the unstable start acceleration. The resist used for e-beam direct writing is ZEP-520.

In the dry etching of absorbers, the reactive ions etch (RIE) of Plasma Tech system has been utilized to obtain anisotropic results. To etch these absorbers, there are different etching gases can be applied. In this system, $Cl_2 + O_2$ is used for Chromium layer etching and NF_3 is used for Tungsten absorber etching. The details of etching recipes are Cl_2 40 sccm, O_2 5 sccm, *rf* power 100 Watt, pressure 100 mtorr for Chromium etching and NF_3 21.6 sccm, rf power 150 Watt, pressure 100 mtorr for Tungsten etching. The residual photoresist during absorbers etching must be stripped off in advance to reduce the contamination in process afterward. For resist strip off, wet etching ($H_2SO_4{:}H_2O_2 = 3{:}1$ at 85 °C) solution usually used is not applicable because of metals will be attacked by the solution. The ultrasonic wafer cleaning system with ACE solution can be used either because of its ultra-high frequency

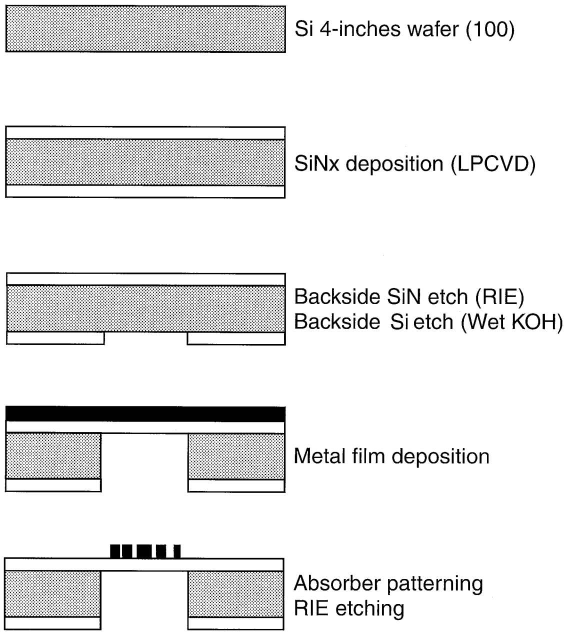

Fig. 3.

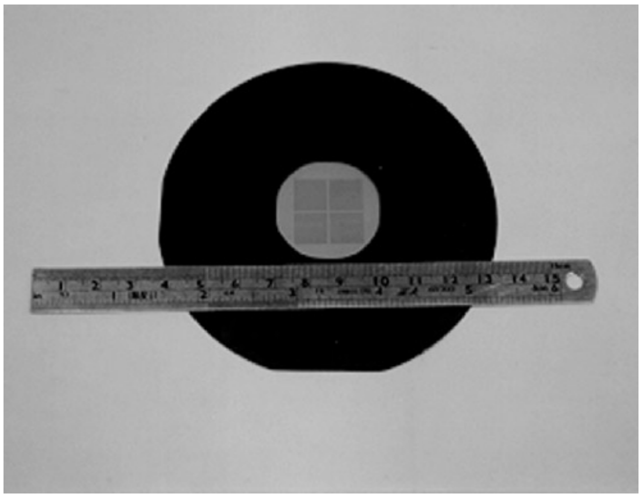

Fig. 4.

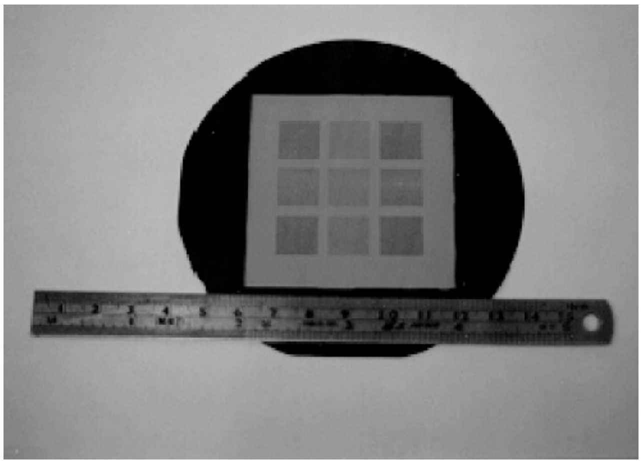

Fig. 5.

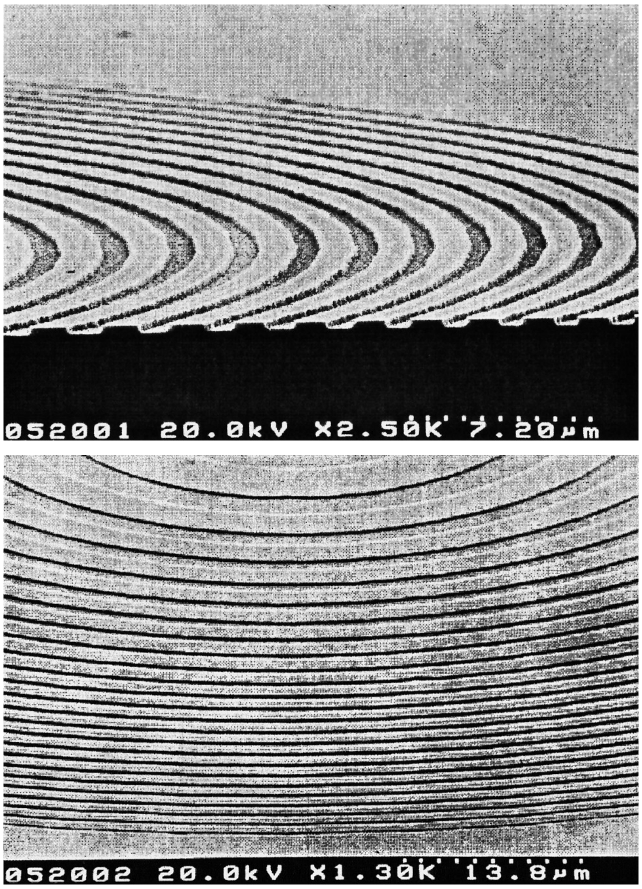

Fig. 6.

(~850 kHz) will destroy the membrane. Therefore, oxygen plasma, Samco UV & Ozone dry stripper were used to strip off the photoresist. The process procedure of intermediate mask fabrication is shown in Fig. 3. Figure 4 and Figure 5 show pictures of the intermediate masks with 3 cm^2 in diameters and 6 by 6 cm^2, respectively. The top view and crosssection view of tungsten zone plate pattern are shown in Fig. 6a and 6b.

3
Results and conclusion

Intermediate mask with zone plates patterns have been fabricated on 1.2 μm low-stress silicon-rich silicon nitride membrane successfully. The thickness of Tungsten (W) absorbers is 0.7 μm. The width of outmost zone is 0.8 μm and around 200 μm in diameter for all zone plates. Working masks for hard x-ray microscopy applications, wavelength less than 0.6 angstrom, zone plates can be duplicated by using intermediate mask as x-ray mask and exposed in the x-ray lithography beamline with thicker resist like PMMA such that thicker absorbers can be obtained by electroplating techniques.

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