Use of SU-8 photoresist for very high aspect ratio x-ray lithography

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This paper describes the process of deep x-ray lithography using epoxy negative photoresist SU-8. Coating, softbake, exposure, post exposure bake, and development of the resist is characterised. Influence of x-ray source spectrum on the lithographic image contrast is calculated and optimal x-ray mask layers compositions for the spectrum in use are proposed. Method for resist film thickness control during casting step is reported. Temperature limit for the post exposure bake was found to ensure safe post bake to obtain maximum resist sensitivity. Optimised development and rinsing process is presented. Resist structures with aspect ratio as high as 100:1 (height : width) are demonstrated.

1. INTRODUCTION

SU-8 abilities to produce high aspect ratio resist masks in photolithography have been demonstrated in recent five years 1-5. SU-8 is an epoxy resin with very high epoxy functionality. Its main feature is the ability to form dense 3D network of cross-links, provided the irradiation dose is high enough. That results in highly mechanically and thermally stable resist mask. The high concentration of the cross-links makes the resist less susceptible to swelling during the development. The photolithography, however, appears to be incapable of producing resist structures with aspect ratios higher than 25:1(H:W), which are of great value in fabrication of MEMS and MOEMS. The reason for that is absorption of the UV light in the resist and optical diffraction, though the diffraction influence is decreased by the light wave-guide effect in SU-8.

Deep x-ray lithography (DXRL) is physically the most natural method to produce high structures. For radiation with wavelength of 0.3-0.5 nm penetration depth into a polymer like SU-8 is 1-1.5 mm. The diffraction divergence would produce a diffraction blur of just 0.5 µm at 2 mm distance from the mask. These numbers suggest a theoretical limit for possible aspect ratio - 1000:1 or more.

In this work we investigated the process of very deep x-ray lithography with SU-8 resist. The idea was to find out realistic limit for the aspect ratio and resolution for the resist and to optimize its processing parameters.

2. EXPOSURE STATION

The exposure station is described in a greater detail elsewhere6. Synchrotron radiation from MAX II third generation 1.5 GeV source was used. The beam cross-section at the mask plane was 2.5x76 mm² (HxW). The beam divergence was less than 1 mrad both horizontally and vertically. The exposure area was 76x76 mm².

3. X-RAY MASK PARAMETERS EVALUATION

The choice of mask materials and the layers thickness is based firstly on mask contrast and secondly on mask technological workability. X-ray absorption spectrum of almost any material can easily be fetched via Internet 7. Knowing the incident radiation spectrum and the spectra of resist and mask absorption one can calculate the image contrast vs. depth in the resist. The contrast is defined as:

\[ C(d) = \frac{E_1(d) - E_0(d)}{E_1(d) + E_0(d)} \]

where \( E_1(d) \) and \( E_0(d) \) are the doses absorbed by the resist at the depth \( d \) in bright and dark image areas respectively. Figure 2 shows the contrast-vs.-depth curves for different mask compositions. In our in-house mask production material of choice for mask membranes is Si. The absorber material is Au. As it appears from the \( C(d) \) curves...
even the mask with the thinnest absorber (Au 2/Si 10) will be good enough for lithography with \( d \leq 150 \mu m \) \( (C > 0.8) \). With thicker absorber layers Si mask is almost as good as the Be membrane mask.

Figure 2. Image contrast vs. resist thickness for various mask compositions. Solid line - 16 \( \mu m \) Au on 500 \( \mu m \) Be; \(- 10 \mu m \) Au on 10 \( \mu m \) Si; \(- 6 \mu m \) Au on 10 \( \mu m \) Si; \(- 4 \mu m \) Au on 10 \( \mu m \) Si; \(- 2 \mu m \) Au on 10 \( \mu m \) Si

Beryllium is an excellent membrane material, though being extremely toxic it is quite tough to machine. Masks made of 500 \( \mu m \) thick Be with 10-20 \( \mu m \) Au absorber possess the best contrast \(-0.98 at 1000 \mu m \) in our exposure conditions. Therefore, to achieve the best lithography results Au/Be (16/500 \( \mu m \)) mask was used. The mask was made at IMM Mainz and had various micromechanical test structures with the minimum feature size down to 4 \( \mu m \).

4. RESIST PROCESSING

4.1. Substrate coating

XPSU-8 5 with solid content 52% provided by Microlithography Chemical Corporation (MCC) was used in all experiments. The substrates were 2” and 3” diameter Si wafers. For spin coating we used the following routine: In two stage spinning the first stage had duration of 3 seconds at 50 rpm. The second stage was 15 s long with 450÷1500 rpm. For this resist the optimal spinning conditions for thick film coating were found to be at 550 rpm. At this speed the resulting thickness after 20 min bake at 90\(^{\circ}\)C on a hotplate was \(-60 \mu m \) and the quality of the film was sufficient. Using mentioned above spinning procedure it was possible to spin on up to 5 subsequent layers thus obtaining \(-300 \mu m \) thick films.

Further improvement in coating process was gained when casting was employed instead of spinning. Prior to the casting Si substrates were ashed in oxygen plasma to remove organic contaminants. The casting was done on a precisely leveled hotplate at 90\(^{\circ}\)C. The wetting of the substrate surface by the resist appeared to be not very strong. The contact angle was close to 90\(^{\circ}\). It can be derived by elementary calculation that the small, compared to the wafer area, drop of the resist is spreading over the wafer surface until it reaches the minimum height defined by the resist surface tension and the contact angle. The minimum drop height is:

\[
h_{\text{min}} = 2 \cdot \frac{\sigma}{\rho \cdot g} \cdot \sin \frac{\theta}{2},
\]

where \( \sigma \) is the surface tension of the resist, \( \rho \) is the resist density, \( \theta \) is the contact angle, and \( g \) is the gravity constant. When the volume of spilled resist is such that the meniscus can reach the edges of the wafer the thickness of the film becomes proportional to the volume as there is a certain energy threshold for the resist to go over the substrate edge. The maximum height of the resist meniscus, which can be kept on the substrate, is defined by the substrate edge radius of curvature and substrate and resist surface tension. It has to be mentioned that if the temperature of the substrate during resist spreading is raised above \( T_g \), like in our case, the resist viscosity gets considerably low, which helps quick resist spreading. Our typical thickness values varied between \( h_{\text{min}} = 300 \mu m \) and \( h_{\text{max}} = 1300 \mu m \).

4.2. Softbake

The conditions of thick SU-8 softbake are very important for its lithographic performance. For the films thicker than 20 \( \mu m \) the most important condition is the substrate leveling. As the resist is baked over its glassing temperature and the amount of solvent is quite high, during the first minutes of
baking the resist can easily flow forced by the force of gravity.

Another important parameter is the baking time. We chose our baking time so that residual concentration of solvent in the resist was 5-10%. Small amount of solvent remaining in thick SU-8 films seemed to decrease the resist cracking after the development. About 10 hours of baking at 90°C were found to be optimal for 500 µm thick SU-8 5 films.

4.3. Exposure parameters

The minimum dose absorbed by resist to begin cross-linking was found to be 52 J/cm³. The incidence dose of 7 J/cm² was required for 480 µm thick SU-8 resist to make it fully exposed. In our exposure conditions (MAX-II current 180 mA, He pressure 200 mbar) the time required to expose the film was about 3 minutes. To protect x-ray mask 25 µm thick capton film was used as a separator between mask and resist.

4.4. Post-exposure bake (PEB)

To obtain good resist mask edges we followed PEB method suggested for SU-8 by MCC. The substrate was put on the hotplate set to 50°C for 3-5 min. This step is believed to be necessary to avoid the image flowing before resist becomes at least slightly cross-linked. Then the temperature of the hotplate was gradually (in 10 min) ramped to 90°C and there it remained for 20 - 30 minutes. After that the hotplate was switched off and cooled down on its own with the substrate still on it.

To be able to predict behavior of the resist at the temperatures higher than 90°C we conducted a simple experiment. Using 25 µm thick SU-8 we measured cleaning time in a solvent for unexposed films which were baked for the same length of time but at different temperatures. SU-8 solvent gamma-Butylacetone (GBL) was used for film removal. Dependency of the time needed to remove the film upon the baking temperature is shown in Figure 3.

As it may be seen in the graph the temperature at the cross-linking onset is around 135°C. This result allows to suggest that post-exposure bake temperature may be increased up to 120-130°C without risk of unexposed cross-linking. The elevated temperature regime can not only decrease required baking time but may as well increase the resist sensitivity.

4.5. Development

Structures which are shown in the micrographs (Figure 4) were developed using the following procedure: To ensure complete dissolution of partially cross-linked areas at the resist mask edges we used GBL as a developer. 2-3 minutes of ultrasound excitation were preceding 20-30 minutes of development in a slightly stirred GBL. Although we tried development at the temperatures of up to 50°C, no dramatic change compared to the development at room temperature was observed. The ultrasound excitation step did not any noticeable difference too. The most important factor for SU-8 deep structure development was refreshing of the developer approximately once in 10 minutes. Rinsing after the development was done in GBL and then IPA. Noticeably white flaky deposits could have been observed after the IPA rinsing in cases when the first rinsing with GBL was insufficient.

5. RESULTS

Resist films for the structures shown on the micrographs 1-5 were cast onto the hot Si substrates and baked on the leveled hotplate for 5-10 hours. Absorbed exposure doses at the bottom of the resist was estimated as 55-60 J/cm² while the dose at the top of the resist films was ~3.5 times higher. Development times were not longer than 1 hour in all cases. Structures were tested for mechanical and thermal endurance. Grids shown in Figure 4(b) have
Figure 4. (a) SU-8 thickness 400 µm. The fence thickness is 4 µm. Aspect ratio (AR) is 100:1; SEM photo is taken at 45° tilt. (b) Resist thickness is 360 µm, lines are 20 µm wide/s spaces are 15 µm. AR is 20:1. SEM photo is taken at 60° tilt. (c) 8 µm diameter pillars in 480 µm thick SU-8. Selected fragments are shown in micrographs d, e. AR = 60:1; SEM photo is taken at 60° tilt. (d) Diameter of the pillar at the top is 8 µm. Roughness of the pillar walls looks less than 0.2 µm, and walls verticality is almost perfect. (e) Foot of the pillar is slightly wider. This may be a result of some additional exposure by photoelectrons emitted from the substrate. Diameter of the pillar at the foot is 9 µm.

taken ultrasonic bath during rinsing. Pillars (Figure 4c) were baked at 190°C without any noticeable change of shape. During SEM inspection the same pillars underwent hard exposures by electrons. Magnifications of up to 40000x were used while looking at the uncoated SU-8 structures. Nevertheless, no bubbling effects or melting of the resist, typical for PMMA under such conditions, were observed.

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REFERENCES


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