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# Nanometer E-Beam Lithography Using 2-Layer Resist System Composed of Silicone-Based Negative Resist (SNR)

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As an E-beam fabrication process in deep-submicron - nanometer region, 2-layer resist system using Silicone-based Negative Resist (SNR) was proposed. A high resolution, high aspect ratio, large E-beam dosage margin and little proximity effect in this system allows various high resolution E-beam lithographies on solid substrate. Fine gratings with minimum pitch of 0.18  $\mu$ m, minimum linewidth of 0.07  $\mu$ m, and aspect ratio of 6.5 were delineated. SNR 2-layer resist patterns were applied to the fabrication of high contrast x-ray mask by RIE and small windows for Josephson junction by lift-off.

#### \$1. Introduction

There have been extensive studies where nanometer structures are fabricated by E-beam lithography.<sup>1)</sup> These patterns are normally defined in positive electron resist such as polymethylmethacrylate (PMMA), because of its high resolution. The exposure of thin PMMA film on thin Si3N4 membrane with 1 nm diameter E-beam, which can minimize the resolution loss due to beam size and electron scattering in resists and from the substrate, revealed that the resolution limit of this resist is 10 nm in linewidth and 40 nm in resolved minimum pitch. 2) PMMA resist, however. suffers from its poor resistance against various dry-etching reactions and low sensitivity to E-beam, which severely restrict wide application of the resist to nanometer lithography.

Though negative electron resists have been believed to show lower resolution than positive type, some negative resists could have high adaptability to device fabrication process, because of excellent dry-etching durability. Furthermore, in order to delineate complex and arbitrary nanometer structures for future devices, both negative and positive type resists will be inevitably used. We have already delineated nanometer structures as small as 23 nm by exposing a high resolution negative resist aM-CMS, but this resolution was achieved only in very thin resist on thin film substrate. 3) Such thin resist and/or

thin substrate clearly cannot construct a comprehensive device fabrication process, in which thick and dense resist patterns are often required on various substrates.

The trade-off between resolution and resist or substrate thickness can be solved by introducing multi-layer resist process. In this paper we demonstrate nanometer E-beam lithography using 2-layer resist composed of Silicone-based Negative Resist (SNR), which was newly developed by NTT.<sup>4)</sup>

## \$2. SNR 2-layer resist system

Figure 1 depicts the fabrication process of SNR 2-layer resist systems. The characteristic features of SNR are its excellent durability to 02 reactive-ion-etching (RIE) owing to the siloxane



Fig. 1 SNR 2-layer resist processes

main chain structure, as well as high contrast and useful sensitivity. This durability to 0<sub>2</sub>RIE enables a double layer resist structure, which consists thin top SNR layer and thick organic bottom layer. High resolution patterns which can be defined in very thin SNR layer, will be transferred to the thick bottom resist.

The good solubility of SNR allows wide selection of film as a bottom resist, because various solvents can be used for spinning and developing SNR on the bottom resist. Solid top resist patterns can be obtained because of the high glass transition temperature( $T_g$ ) of SNR. These features make it possible to use SNR 2-layer resists for lift-off process with negative type E-beam exposure. This lift-off process is very suitable for defining small holes such as Josephson junction windows or contact holes with little fabrication damage.

These SNR 2-layer resist system has various advantages over previous resist systems proposed for nanometer lithography, such as a high aspect ratio, reduced proximity effect, the use of solid substrate and the minimum increase of process step number in multilayer resist processes.

#### \$3. Experiment

As bottom resists, phenylmethacrylatemethacrylic acid copolymer( $\phi$ -MAC)<sup>5)</sup> and AZ-1350J were used, owing to their high dry-etching durability. SNR was spin coated on a cured organic bottom layer, exposed by 20kV electron beam and then developed in the xylene solution.



Fig. 2 The elctron beam sensitivity of SNR on Si substrate.

The electron beam sensitivity curve of SNR on the Si substrate is shown in Fig. 2. The sensitivity was  $27 \,\mu$ C/cm<sup>2</sup> as D<sub>0.5</sub> and the contrast 2.4. To estimate the resolution limit of SNR 2-layer resist, grating patterns with various pitches were written in 0.08  $\mu$ m thick SNR on 0.4  $\mu$ m thick  $\phi$ -MAC resist over 40  $\mu$ m square. The patterns were evaluated after 0, RIE by SEM photographs.

The x-ray mask with 0.8 µm thick Tantalum(Ta) as an x-ray absorbing layer was fabricated with SNR(0.05 µm)/AZ(0.4 µm) resist system. The resist patterns were transferred to SiO2(0.35 um) layer, then Ta layer was etched with SiO2 mask using RIE with a CBrF3 gas. The sensitivity of SNR on Ta was increased by 20% as compared with that on Si. For a lift-off process, SNR(0.37  $\mu$ m)/ $\phi$ -MAC(0.42  $\mu$ m) resist was used. The 02 pressure was chosen to 10 mTorr to restrain side-etching of bottom layer which reduces the resolution limit. The rf power density was 0.1 W/cm<sup>2</sup> and the etching rates of φ-MAC,AZ, and SNR were 1000,800, and 10 Å/min, respectively. The overhang structure for lift-off patterns was controlled by an etching time. Lift off was carried out using methylethylketone in an ultrasonic bath.

### \$4. Results and Discussion

In the case of thick resist pattern formation on solid substrate, SNR 2-layer resist system shows superior performances both in resolution and sensitivity to PMMA. The minimum grating pitch clearly resolved in SNR(0.08  $\mu$ m)/ $\phi$ -MAC(0.4  $\mu$ m) was 0.18  $\mu$ m, of which SEM picture is shown in Fig. 3.



Fig. 3 0.18  $\mu$ m pitch grating in SNR/ $\phi$ -MAC(0.4  $\mu$ m thick) resist.

On the other hand, the minimum pitch achieved in PMMA (0.5  $\mu$ m thick) under the almost same exposure conditions, was 0.3  $\mu$ m, and the required dosage was 4-5 times higher than in SNR/ $\phi$ -MAC.

Figure 4 shows the line dosage margin as a function of pitch width for SNR 2-layer resist. The optimum dosage does not change significantly for various pitch widths and the relatively large dosage margin is obtained for gratings of wider than 0.18 µm pitches. The rapid reduction of dosage margin under 0.18 µm in pitch probably corresponds to lowering of contrast which is due to increased back-scattered electrons from the substrate. Though such fine gratings have been normally written by holographic exposure, this large dosage margin and much higher aspect ratio in SNR 2-layer resist indicates the future possibility of practical writing of various fine gratings such as those required in DFB laser.

The finest linewidth obtained in this system was 0.07 µm with an aspect ratio of 6.5. The patterns narrower than 0.07 µm tend to incline or fall down, uniform and rigid patterns could not be obtained. This means the minimum linewidth of SNR 2-layer resist is limited not by the resolution of SNR itself, but by the rigidity or adhesion of bottom 1ayer resist. To achieve further improvements in resolution, SNR with a higher contrast and tough and adhesive bottom resist will be required.



Fig. 4 Dosage margin for pitch resolution of  $SNR/\phi$ -MAC 2-layer resist.(SNR: 60 nm,  $\phi$ -MAC: 0.4µm)

With dry-etchig durable bottom resist such as AZ, various fabrications by using dry-etcing technology can be accomplished in deep-submicron to nanometer region. The SNR/AZ resist system was applied to high contrast x-ray mask fabrication for Synchrotron Radiation lithography. Since a thick film made from heavy atom such as Au or Ta is used for x-ray absorbing layer, it has been difficult to fabricate very high contrast x-ray mask because of large backscattering effect. Bv applying SiO<sub>2</sub> middle layer with SNR 2-layer resist system, the minimum grating pitches of 0.25 µm and 0.3 µm were produced as resist patterns and as Ta patterns, respectively, without proximity effect The resolution limit of grating correction. patterns on the Ta substrate slightly decreased in comparision with that on Si substrate. Figure 5 shows the SEM picture of 40 nm gaps between 0.26 µm wide Ta patterns, and the aspect ratio of the narrow gap is about 20.



Fig. 5 40 nm gaps in 0.26 µm wide, 0.8 µm thick Ta patterns.

1 µm

Although dry-etching technology has rapidly grown as a main LSI fabrication process, lift-off is still important process for fabrication of various test devices, particularly nanometer structure devices, because of less damage to substrate and no restriction of film to be patterned, and so on. SNR 2-layer resist system also has a high adaptability to lift-off process, which is demonstrated in the fabrication of small window patterns.

Figure 6 shows this process. (a) the overhang structure suitable for lift-off is formed in 2-layer resist patterns by  $0_2$ RIE. (b) 0.27 um thick Si0 was evaporated. (c) by lift-off in methylethylketone windows in the insulated Si0

layer are produced. The openings were clearly formed, and a very smooth slope of 0.25  $\mu m$  square assures a high step-coverage in contacting upper layer. Slope angles can be controlled with varing the thicknesses of both of the resists. 0.1 µm diameter SiO holes can be so far fabricated.

The overhang structure in resist was obtained by overetching in O2RIE at relatively low gas pressure. As this method may cause fabrication damages to substrate, we propose the 2-step etching technique: a bottom layer etched at lower pressure to form vertical side wall, and then over etched at higher pressure to reduce substrate damage, as shown in Fig.7.



a : 2 layer resist pattern



b : after SiO evaporation



Fig. 6 Fabrication of SiO window by negative lift-off process.



Fig. 7 The overhang structure fabricated with the 2-step etching technique. The resist thickness: SNR;0.15  $\mu$ m ,  $\phi$ -MAC; 0.75 µm. Etched time: 7.5 min. at 10 mTorr 8 min. at 100 mTorr.

#### \$5. Conclusion

In conclusion, E-beam lithography using SNR 2-layer resist process allows various fabrications of thick, dense and arbitrary deep-submicron nanometer structures on thick substrates for future nanometer devices.

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