BEHAVIOR OF SU-8 EPOXY BASED NEGATIVE UV-PHOTORESIST AS NEGATIVE TONE RESIST FOR ELECTRON-BEAM LITHOGRAPHY

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ABSTRACT

This article deals with an epoxy based UV-photoresist SU-8. It analyses the behavior of SU-8 resist as negative resist for e-beam lithography. E-beam lithography in thin layers of SU-8 is demonstrated. An etching mask of the SU-8 resist for a silicon reactive ion etching (RIE) in oxygen and tetrafluoromethane is fabricated. Two reactive ion etching processes are performed in order to show both the SU-8 resist etching resistance in RF oxygen plasma and the selectivity of RIE process in CF_4/O_2 between silicon and the SU-8 resist.

1. INTRODUCTION

SU-8 resist is a relatively cheap chemically amplified epoxy based negative UV-photoresist [1]. It was designed for versatile spectrum of use as micromachining (MEMS) [2, 3], microelectronic, microoptical systems [4] and other applications where a thick, chemically and thermally stable image is desired. The SU-8 resist is most commonly known as a negative resist for a conventional UV lithography (350-400 nm) [5], however recently capabilities of SU-8 as negative e-beam lithography (EBL) resist have been discovered [6].

E-beam lithography uses a polymer resist mostly for the recording of the image information. The behavior of these polymer electron resists, under influence of electron of a given energy, is expressed by the sensitivity curve. The sensitivity curve is constructed as a dependence of the relative change of the functional resist layer thickness on the exposure dose [7]. In order to examine the behavior of the SU-8 resist, silicon wafers with a thin layer of the SU-8 resist has been prepared. These samples were used for the sensitivity determination. Prepared testing structure layout was exposed using the 15kV shaped ebeam writer. In the second experiment an etching mask for CF₄/O2 based reactive ion etching (RIE) of silicon was fabricated. The Etching resistance and the etching selectivity of the SU-8 resist layer were defined.

2. SAMPLE PREAPARATION

For all experiments the commercially available resist SU-8 2000.5 were used. The SU-8 2000.5 resist with a solid content 14 wt.% is from all offered formulation least viscous, therefore very thin layers could by deposited. With this solution is possible spin a layer with nominal thickness 500 nm at a spin speed of 3000 rpm. In order to archive good resolution with e-beam exposition the thickness of the SU-8 resist layer should by below 300 nm. Hence, the base solution of the SU-8 2000.5 resist was thinned with cyclopentanone to 9 wt.% concentration solution. Before the spin coating, the silicon substrates were cleaned in the RF oxygen plasma. With respect to the relatively fair adhesing properties, of the base SU-8 2000.5 substance on silicon, no adhesion promoters were used. For the experimental part, two silicon substrates Si[100] 3" in diameter was spin coated with the 9wt.% SU-8 solution. The resulting thicknesses of deposited films were 342 nm and 265 nm. After the spin coating, silicon substrates with the SU-8 resist film were baked at 90°C for 1min on a hotplate (soft baking). Exposures of a sensitivity test and a grid test structures for RIE were carried out with the BS600 15kV shaped e-beam writer. Since the SU-8 resist is an chemically amplified resist, exposed samples were baked at 90°C for 1 min on the hotplate (PEB) and subsequently developed in a SU-8 developer (propylene glycol monomethyl ether acetate, PGMEA) for 30 sec. The Final step were hard baking of both substrates at 150°C for 5min on the hotplate to annealing surface cracks.

3. EXPERIMENTAL

3.1. SU-8 SENSITIVITY EVALUATION

The exposition test was designed to measure the SU-8 resist sensitivity. The Sensitivity test was composed from a square exposition stamps with size $2 \times 2 \mu m$ (see on Fig.1). The exposure dose was specified from measuring of current on a given exposition stamp size in a Faraday cage. Measuring was performed before and after sample exposure to ensure that a current density didn't change during exposition. The thickness of the SU-8 resist in the sensitivity test was measured by a profilometr Talystep Taylor-Hobson.

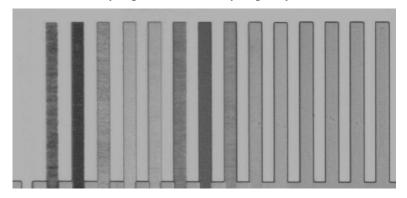


Fig. 1: Image of the sensitivity test designed for the profilometr Talystep Taylor-Hobson measurement (exposition dose increase onwards from left to right).

The normalized sensitivity curve is shown in Fig 2. It shows, that the SU-8 resist have relatively high sensitivity $S^{0,5} = 0.67 \mu C/cm^2$, however low contrast $\gamma = 0.99$.

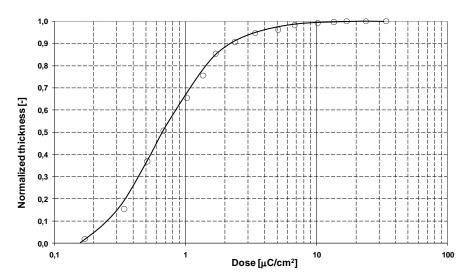


Fig. 2: The normalized sensitivity curve for SU-8 resist layer with thickness of 265 nm. Exposure parameters: U=15kV, I_0 =0.34A/cm², development time: 30sec. Sensitivity $S^{0.5}$ = 0.67 μ C/cm²; contrast γ = 0.99.

3.2. REACTIVE ION ETCHING OF SU-8 RESIST

In the first experiment etching rate of the SU-8 resist layer in the RF oxygen plasma, in the barrel type reactor at pressure 1000 Pa, frequency 13.57 MHz and HF power 100 W, were investigated. Because of etched substrate temperature and reactor temperature changes during etching, it was ensured that in each experiment of etching, both the etched substrate and the reactor had same initial temperature 25°C. The decrease of thickness was measured by means of the profilometr Talystep Taylor-Hobson. The result of whole experiment is shown in the graph on Fig.3, which expresses dependence of the etching rate on the etching time. The etching rate of SU-8 in the RF oxygen plasma increase with etching time this is probably caused by temperature increase in the reactor chamber. The high resistance of the SU-8 layers during the oxygen based RIE process was confirmed.

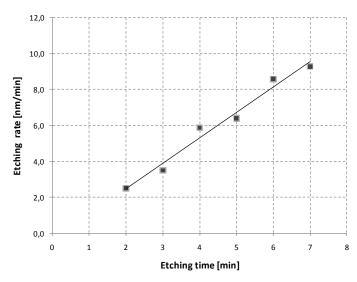


Fig. 3: Dependence of the etching rate on the etching time for the SU-8 resist layer at RIE in O₂.

For the second experiment special testing layout for exposition into SU-8 resist were developed (see on Fig.4). This test contained an horizontal and an vertical diffraction grids with periodicity of 4 μ m, 2 μ m, 1.6 μ m and 1 μ m to test resolution and some other testing

structures for example for proximity effect observation. In order to transfer structure of the grids created in the SU-8 resist into the silicon substrate, CF₄/O₂ based RIE process were applied.

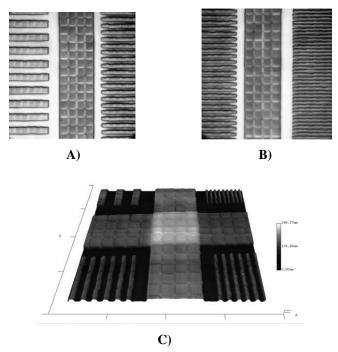


Fig. 4: Image of a gird structure formed in the SU-8 resist serving as the etching mask in the RIE process. A) grid with periodicity of 4μm and 2μm. B) grid with periodicity of 1.6μm and 1μm. C) AFM image.

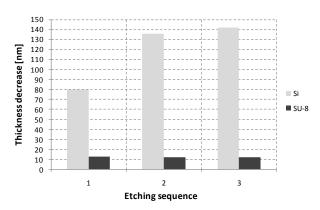


Fig. 5: The thickness decrease in the Si and the SU-8 resist within three steps CF_4/O_2 based RIE (P = 200W; p = 1000 Pa; t = 1 min).

The RIE processing were carried out with the barrel type reactor TESLA 214VT at these conditions: 84% CF₄ + 16% O₂ etching agent, frequency 13.57 MHz, pressure 1000 Pa and HF power 200 W. Overall, three etching cycle were performed each with 1 min duration so etching process repeatability might by confirmed. The result of second experiment is shown in the graph on Fig.5, which expresses a thickness decrease of the SU-8 resist and the silicon in three step etching. The etch rate of the silicon in the RIE process is 1.98 nm/s whereas the etch rate in the SU-8 resist is only 0.205 nm/s this resulting in etching selectivity approximately 9.7. On the second and the third etch is shown that the repeatability of the silicon RIE process is very high. It also enables high control in the etch process and allow an adjustment of the final deepness of the etched layout. Displays of the low etch rate in the first etching is probably caused by the thin layer of SiO₂ at the surface of the silicone substrate.

4. CONCLUSION

The e-beam lithography in thin layers of the SU-8 resist has been demonstrated. The sensitivity and the contrast of the SU-8 resist were defined and the sensitivity curve was drawn. It was experimentally found the etching rate of the SU-8 resist layer in oxygen based RIE. It was found that the RIE of the SU-8 resist in oxygen occurs isotropically. The grids pattern in the SU-8 resist was successfully transferred into a silicon substrate by isotropic CF_4 based RIE process. The etching selectivity and the silicon etching rate at the RIE of the SU-8 resist in CF_4/O_2 were found.

5. ACKNOWLEDGMENT

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REFERENCES/LITERATURA

- [1] SU-8 2000 processing guidelines by MicroChem Corp., Newton, Massatchusses, www.microchem.com
- [2] ZHANG, J., TAN, K.L., GONG, H.Q., Characterization of the polymerization of SU-8 photoresist and its applications in micro-electro-mechanical systems (MEMS), *Polymer Testing*, (2001) vol .20, p. 693-701
- [3] DESPONT, M., LORENZ,M., FAHRNI, N., RENAULD,P., BRUGGR,J., VETTIGER,P., "High aspect ratio ultrathick, negative-tone near-UV photoresist for MEMS applications", in Proc. MEMS'97, IEEE, Nagoya, (1997): p. 518-522
- [4] LING, Z., LIAN, K., SU-8 3D microoptic components fabricaated by inclined UV lithography in water, *Microsyst Technol* (2007) vol. 13, p. 254-251
- [5] SHAW,J.M., GELORME,J. D., LABIANCA,N.C.,CONLEY, W.E.,HOLMES, S.J., "Negative photoresists for optical lithography", IBM Journal of Research and Development vol. 41(1997) p. 81-94
- [6] BILENBERG,B.,JACOBSEN,S., SCHMIDT,M.S., SKJOLDING,L.H.D., SHI,P., BOGGILD,P., TEGENFELDT,J.O., KRISTENSEN,A., "High resolution 100kV electron beam lithography in SU-8". Elsevier Science (2001).
- [7] MATĚJKA, F., MATĚJKOVÁ, J., "Optimization of reliefs of thin polymer films of resist for phase diffractive optical elements", Proceeding of the international summer school on the new trends in surface and thin film physics and engineering. 14-18 June 1999, Tři Studně, Czech republic.