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Electron beam lithography in thick negative tone chemically amplified resist: Controlling sidewall profile in deep trenches and channels

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ABSTRACT

Exposure characteristics of 30 keV electron beam lithography in 6 μm thick chemically amplified resist SU-8 is reported. The exposure dose variation shows remarkable impact on resolution and sidewall profiles of 3D structures of SU-8. Sidewall profiles of the experimentally realized vertical structures have been analyzed by comparing them with three dimensional electron energy deposition profiles in the resist. The analysis shows that the broadening of the developed structures in excess of a particular energy deposition density contour follows non-monotonic variation. The sidewall profiles indicate that the crosslinking of the resist proceeds in a reaction–diffusion environment in which the photoacid diffusion itself is controlled by crosslink density. We also demonstrate large area fabrication of two types of SU-8 deep channels connected by bridges, by exploiting the energy dependent range of e-beam in the resist.

1. Introduction

Electron beam lithography (EBL) in thick layers of negative tone chemically amplified (CA) resists enables convenient processing in several micro-fabrication applications. The sensitivity and contrast of CA resists are strongly dependent on the thermal processing conditions prior to and, more importantly, after the radiation exposure [1,2]. Under low contrast processing conditions, grey scale electron beam lithography in negative tone CA resists can be used for direct writing of micro-structures with three dimensional (3D) surface profiles [2]. In a different set of applications, EBL on thick films of negative tone resists is used for fabrication of multilayer micro/nano-structures using multiple exposures with electron beams of different energies. The range of an electron beam inside the resist depends on the energy of the incident electrons. By choosing appropriate energy of the beam, a through exposure of the resist, or a partial exposure up to a shallow layer is possible. Combination of such exposures may be used for fabrication of 3D structures with self-supporting parts [3–5]. The control over the sidewall profiles of the developed 3D structures is important in fabricating suitable micro-structures for a particular application. For example, the capillary flow rate of a liquid in an open micro-channel depends on the slope of the sidewall.

SU-8 is an epoxy novolak based negative tone CA resist which has been fruitfully used in the both types of applications mentioned above. SU-8 was originally developed for UV [6–8] and X-ray [9–11] lithography of high aspect ratio 3D micro-structures in thick and ultra-thick resist layers for MEMS and microfluidic applications [12,13]. Later, Wong *et al.* and several other groups have demonstrated the suitability of electron beam lithography for high resolution structures in thin SU-8 layers under suitable choice of processing parameters [14,15]. Grey scale electron beam lithography in thick layers of SU-8 has been used to fabricate micro-structures with 3D surface profiles suitable as refractive optical elements [16].

Electron beam lithography in thick resist layer suffers from inevitable low resolution problem due to broadening of the energy deposition profile of the electron beam inside the resist by electron scattering in the resist and the substrate. Nevertheless, EBL in thick layers of negative resist offers some processing advantages as mentioned above. The loss of resolution due to broadening of the electron energy deposition profile in the resist outside the e-beam addressed area is the so called proximity effect [17–19]. A non-CA polymer resist such as PMMA is based on chain scission events triggered by the electron exposure. The after exposure latent image of a non-CA resist does not change in any subsequent thermal process. Thus a threshold of electron energy deposition density can be associated with such resist systems for development of the resist. Once the spatial distribution of electron energy deposition density

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in the resist for an exposure is obtained, the resolution of the lithography can be estimated by identifying the equienergy deposition density contour corresponding to the threshold energy deposition density [20].

A chemically amplified resist, on the other hand, contains photoacid generators which are decomposed into strong acid upon electron beam exposure, and the acid diffuses in the resist during the post exposure baking process and leads to cationic polymerization (crosslinking) in the resist. Thus the resolution of electron beam lithography in a chemically amplified resist will depend not only on the spread of electron energy deposition in the resist, but also strongly on the photoacid diffusion range and crosslinking which occur during post exposure baking [21,22]. In most cases the proximity correction is performed by assuming the spread in the energy deposition profile of a point beam using a double Gaussian function $f(r)$, which represents the normalized distribution of energy deposition per unit area of the resist, where r is the radial distance from the incident beam [23]. In addition, reaction–diffusion models are employed for doing proximity corrections in CA resists to take into account the effect of photoacid diffusion in the resist [21,22]. To examine the resolution characteristics and the sidewall profiles of vertical structures in a thick resists the radial picture of energy deposition profile is not adequate. The z dependence in the energy deposition function is crucial in such structures and is often ignored. Thus, three dimensional electron energy deposition density profiles in the resist are essential for analyzing the resolution characteristics and sidewall profiles of structures in thick resist layers. The systematic study of exposure dependence of resolution as well as the sidewall profiles of 3D structures in thick negative resist would be of considerable interest as these are important in establishing the required control over achieving the desired structures.

In this work, we study the resolution characteristics and sidewall profiles of the thick CA resist SU-8 using electron beam lithography. In particular, we have studied the exposure characteristics of 30 keV electron beam lithography in 6 μm thick SU-8 resist layer by systematic observation of the profiles of the developed structures. Three dimensional electron energy deposition density profiles in the resist have been obtained using a Monte Carlo program to analyze the evolution of the sidewall profile with exposure dose. Comparison with experiment is used to distinguish the effect of photoacid diffusion on the structure profile. We also demonstrate a simple process for large area fabrication of two different types of deep micro-structures with self-supporting components using multiple exposures of different e-beam energies.

2. Experimental

The main part of the SU-8 resist comprises of the resin Bisphenol-A Novolak epoxy oligomers, which in general contain eight epoxy groups hence the name SU-8. The resist also contains mixed triarylsulfonium hexafluoroantimonate salts up to 10 wt.%, which are the photoacid generators (PAG). In the present experiments we have used the commercially available resist SU-8 3005 purchased from MicroChem. This formulation uses cyclopentanone as the organic solvent. SU-8 films of thickness 6 μm were spin coated over RCA cleaned indium tin oxide (ITO) coated glass substrates. After spin coating, the samples were prebaked on a level hot plate at 95 °C for 3 min. The electron beam exposures were carried out using the dual-beam SEM/FIB setup Nova 600 NanoLab (FEI) with exposure control and pattern generation kit Elphy Quantum (Raith GmBH). The post exposure baking (PEB) temperature was kept fixed at 95 °C while the electron beam energy, dose and the duration of PEB were varied from case to case. Exposures were carried out using well focused beam of 30 keV electrons (current 44 pA). The exposures with 5 keV electron beam were carried out with 98 pA beam current. The electron beam exposure induces generation of hexafluoroantimonic acid by decomposing the triarylsulfonium hexafluoroantimonate salt. During the post exposure bake process the acid diffuses in the resist and causes acid catalyzed crosslinking of the epoxy groups of the resin which leads to polymerization. The exposed patterns were developed in the solvent 1-methoxy-2-propanol acetate for 2 min in a petri dish with gentle agitation, and dried by dry N_2 blow followed by hard baking at 95 °C for 1 min. The developed micro-structures have been characterized using SEM and optical surface profiler NanoMap-D.

3. Results and discussion

3.1. Sidewall broadening of SU-8 walls and resolution limit

Fig. 1 shows SEM images of SU-8 walls written with 30 keV electron beam by making uniform exposures in parallel lines of width 1 μm separated by 20 μm. The area dose of electron beam exposure was 12 μC/cm^2 and the post exposure bake was carried out for 2 min at 95 °C. The SEM image of the tilted view as well as the top view shows that the width of the developed walls is larger than the 1 μm wide scanned lines. The width of the walls keeps on getting broadened towards the bottom, and at the bottom the width is 13.6 μm. We call the development of the walls beyond the 1 μm wide e-beam addressed lines as the sidewall broadening. Thus the sidewall broadening of the walls in Fig. 1 is 6.3 μm, which merely implicates poor resolution of 30 keV electron beam lithography in 6 μm thick SU-8 resist. The loss of resolution is due to intrashape proximity effect as the separation between the lines is large (20 μm). The sensitivity of 30 keV EBL in 8 μm thick SU-8 layers for 90 s PEB at 95 °C was reported [16] earlier to be around 4–6 μC/cm^2. The structure in Fig. 1 was written with relatively higher dose and longer post exposure bake time. To find the resolution limit of 30 keV EBL in such wall-like structures we varied the e-beam dose in the range 2–18 μC/cm^2 and the PEB duration was reduced to 1 min.

The lowest exposure dose at which the pattern formation occurred without loss of resist thickness was 6 μC/cm^2. Fig. 2(a) shows the SEM image of the top view of the pattern. The height and sidewall profile of the walls is obtained from the optical surface profile of the pattern shown in Fig. 2(b). The line profile (Fig. 2(c)) across a single wall shows that the height of the walls is 6 μm which is equal to the resist thickness. The line profile also shows that the width of the top of the wall is 1 μm, but the sidewall is broadened by 2.7 μm at the base. Fig. 3(a) shows SEM images of the top view of SU-8 walls written with e-beam doses in the range 6–18 μC/cm^2. The variation in the sidewall broadening with e-beam exposure dose is plotted in Fig. 3(b). The sidewall

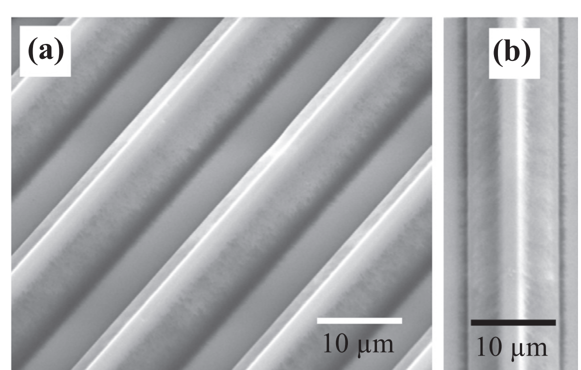

Fig. 1. SEM images of SU-8 walls written with 12 μC/cm^2 dose of 30 keV e-beam: (a) view at 30° angle. (b) Top view.