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SU-8 etching in inductively coupled oxygen plasma ☆

Kristian Hagsted Rasmussen [a,*], Stephan Sylvest Keller [a], Flemming Jensen [b], Anders Michael Jorgensen [b], Ole Hansen [a,c]

[a] Department of Micro- and Nanotechnology, Technical University of Denmark, DTU Nanotech Building 345E, DK-2800 Kgs. Lyngby, Denmark
[b] DTU Danchip, Technical University of Denmark, Oersteds Plads Building 347, DK-2800 Kgs. Lyngby, Denmark
[c] CINF – Center for Individual Nanoparticle Functionality, Technical University of Denmark, DK-2800 Kgs. Lyngby, Denmark

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ABSTRACT

Structuring or removal of the epoxy based, photo sensitive polymer SU-8 by inductively coupled plasma reactive ion etching (ICP-RIE) was investigated as a function of plasma chemistry, bias power, temperature, and pressure. In a pure oxygen plasma, surface accumulation of antimony from the photo-initiator introduced severe roughness and reduced etch rate significantly. Addition of SF_6 to the plasma chemistry reduced the antimony surface concentration with lower roughness and higher etch rate as an outcome. Furthermore the etch anisotropy could be tuned by controlling the bias power. Etch rates up to 800 nm min^{-1} could be achieved with low roughness and high anisotropy.

1. Introduction

SU-8 is an epoxy based, photo sensitive polymer developed by IBM in the late 1980s [1]. SU-8 negative photo resist is derived from EPON™resin [2], where the monomer consists in average of eight epoxy groups and eight aromatic benzene groups as indicated by the name. The viscous polymer contains between 5% and 10% photo-initiator enabling cross linking by standard I-line lithography. The photo-initiator used for the SU-8 resin is based on triarylsulfonium–hexafluoroantimonium, adding fluorine, sulfur, and antimony to the carbon, hydrogen, and oxygen from the monomers as elements in the SU-8 resin.

SU-8 in microtechnology was developed for use in LIGA[1] [3] where the polymer is used to define a structured mold. Furthermore, SU-8 was interesting as etch mask, due to the patterning by standard photolithography. The chemical resistance of SU-8, however, complicates the removal of the resist in both applications, with plasma removal as one of the only reliable option. Therefore, the most thoroughly discussed subject in SU-8 etching is complete removal of SU-8 after its use as masking material.

More recently, SU-8 has been used as a device layer rather than a sacrificial layer. Fabrication of devices in SU-8 can in general be accomplished by photo-lithography, for a large number of applications. Lab on a chip (LOC) systems with microfluidic channels made in SU-8 [4,5], have advantages such as biological compatibility and easy fabrication. Devices for optical applications such as polymer waveguides [6] and optical transducers [7] have been shown. Furthermore, the mechanical properties of SU-8 make it an obvious choice for cantilever sensors [8].

Plasma treatment of all of these devices can be used for several purposes. Probably the most relevant cases of plasma treatment of SU-8, in addition to removal, is functionalization or activation of a surface. This can for example be used to tune the hydrophobicity of a surface or change the surface termination to alter the bonding capabilities [9]. For some applications further patterning of the SU-8 after the initial photo-lithography in the form of etching might be interesting. For instance an isotropic etch can be used to increase the aspect ratio or decrease the line width of lithographically defined structures.

In the scarce literature on SU-8 etching available, most authors agree on the need for fluorine in the plasma chemistry. However, there has not been offered a satisfying explanation for this observation.

Dentinger et al. [10] presented a study on different methods for SU-8 removal, including removal using solvents, chemical removal

* Corresponding author. Address: Building 344, Room No. 208, Oersteds Plads, DK-2800 Kgs. Lyngby, Denmark. Tel.: +45 4525 5848; fax: +45 4588 7762.
E-mail addresses: khara@nanotech.dtu.dk, kristian.rasmussen@nanotech.dtu.dk (K.H. Rasmussen).

[1] Lithographie, Galvanoformung, Abformung.

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in different plasma etching configurations, and other more exotic methods. For our study the chemical removal in any type of plasma setting is interesting. Both, results from reactive ion etching (RIE) as well as downstream chemical etching (DCE) can increase the understanding of the different mechanism involved in the process. Etch rates of 1–4 μm min^{-1} were obtained in RIE using a mixture of CF_4/O_2 in approximately equal proportions [10].

In DCE, Dentinger et al. observed that only 2–4% of CF_4 was needed to obtain etch rates as high as 10 μm min^{-1}. However, to obtain such high rates the temperature was elevated to 275 °C. Such high temperatures will introduce thermal stress in the polymer, increasing the risk of cracking and peeling. It will also cause compatibility problems with some materials in practical applications. Furthermore, surface contamination with antimony was observed after complete SU-8 removal. Dentinger et al. ascribed the surface antimony contamination to residues left from the photo initiator.

The influence of fluorine on etching of cured SU-8 is also discussed by Hong et al. [11] and Mischke et al. [12]. Mischke et al. used CF_4 just as Dentinger et al. did, while Hong et al. added SF_6 as fluorine source to the plasma. Hong et al. limit the discussion to etch rate and anisotropy without discussing chemical composition. However Mischke et al. [12] used Energy-dispersive X-ray spectroscopy (EDX) on etched SU-8 surfaces to identify antimony and fluorine in addition to the expected carbon and oxygen. Mischke et al. conclude that fluorine is introduced by the etch chemistry, neglecting the fact that the photo initiator in SU-8 is triarylsulfonium hexafluorantimonium which includes SbF_6^+ ions.

De Volder et al. [13] used plasma etching to produce nanowires in SU-8. Their process is basically an oxygen plasma etch where they also see an accumulation of antimony at the surface; the antimony is believed to act as local masking agent and starting point of the nanowires. X-ray photoelectron spectroscopy (XPS) analysis of the surface shows up to 19%$_{atom}$ antimony surface concentration in their experiments. Similar to Mischke et al. no external source for antimony was present, and the antimony must hence originate from the SU-8 photo-initiator. For removal of SU-8 this will result in rough surfaces and low etch rates and should be avoided.

The presence of antimony in plasma treated surfaces is a problem for biological applications since antimony is toxic. This does not only apply to samples structured by plasma etching, but also surfaces cleaned or primed in an oxygen plasma will have increased concentrations of antimony in the surface after a shallow etch. Small amounts of antimony may not be critical since the toxicity is weaker than e.g. that of arsenic [14,15]. However, since etching generates thin hairlike structures it can be assumed that the antimony present in the surface is on nanometer scale, for which Bregoli et al. [16] has evaluated the toxicity and found it poisonous. It is important to minimize the antimony concentration to achieve relevant results for biological experiments performed on SU-8 chips.

In this work we will discuss structuring of SU-8 in an ICP-RIE oxygen plasma with varying SF_6 content. Control of antimony concentration and surface roughness will be discussed, together with measurements of etch anisotropy and rate. We will in more detail discuss the influence of antimony on the surface quality obtained and link it to the etch chemistry.

2. Experimental

All SU-8 etching experiments were done in a turbo pumped, inductively coupled plasma (ICP) system, Advanced Silicon Etcher (ASE HC250M) from STS, refitted for polymer etching. The system is fitted with two RF power supplies; the main power supply, the Coil Power, controls the intensity of the plasma, while the secondary power supply, the Bias Power, controls the ion energy of the ion flux to the etched substrate. In the experiments reported here, the feed gasses oxygen (O_2) and sulfur hexafluoride (SF_6) were used at flow rates controlled using mass flow controllers. The pressure in the etch chamber is controlled by a throttle valve and measured using a pressure gauge. All sample preparation and characterization except XPS was carried out in a cleanroom environment.

Since plasma etching, in general, is a very complicated process involving many parameters, Design of Experiments (DoE) was used to reduce the number of experiments necessary to identify the most important parameter relations in etching of SU-8.

2.1. Design of experiments

The number of experiments conducted was reduced by selecting the four most important parameters for variation, Table 1 while the remaining parameters were kept constant. The O_2 flow rate (Q_{O_2}) was keep constant at 99 sccm, while the SF_6 flow rate (Q_{SF_6}) was varied between 0 and 20 sccm. The pressure in the etch chamber was controlled to keep the gas density stable. Since the pressure has a pronounced effect on etch characteristics, the pressure (p) was varied between 20 and 40 mTorr. It should be noted that the system was run in automatic pressure control mode, which continuously adjusts the throttle valve to keep a constant pressure during etch. The coil power (P_C) was fixed at 1000 W, while the bias power (P_B) was varied between 0 and 30 W. Finally, the substrate chuck temperature (T) was controlled between 10 and 50 °C. This design resulted in a full factorial screening in four parameters, where three center points were used to check for quadratic curvature, where the quadratic term of a parameter is needed to generate a valid model. The total number of experiments in this setup is 19, which were processed for 20 min each. The experiments in the design were carried out in random order.

After completion of the first set of experiments it was evident that curvature was present in the response. To enable data analysis and generation of a valid model for the system, the curvature was addressed by adding eight face centered points with two additional center points to the design. A face centered point is a center point with one parameter value at min or max. The ten extra experiments were also carried out in random order, and the center points were used to check for variations between the two sets of experiments. The final dataset comprises the 19 initial experiments combined with the 10 additional, giving a total of 29 experiments to characterize.

2.2. Sample preparation

Samples were prepared by spinning 25 μm SU-8 2075 resist on 100 mm silicon wafers with a 2 μm thick thermal silicon dioxide followed by 1 h of baking on a hotplate at 50 °C [17]. The samples were exposed with 150 mJ cm^{-2} at the I-line, through a test mask with line arrays of different widths, and baked for 2 h at 50 °C on a hotplate, followed by development in PGMEA. Finally, to completely crosslink the polymer, samples were flood exposed

Table 1
Parameters used for DoE design. Center denotes the value used for center points and face centered points.

Parameter		Min	Center	Max
Coded value		−1	0	1
SF_6 flow rate (sccm)	Q_{SF_6}	0	10	20
Pressure (mTorr)	p	20	30	40
Bias power (W)	P_B	0	15	30
Temperature (°C)	T	10	30	50