Optimization of Reactive-Ion Etching (RIE) parameters to maximize the lateral etch rate of silicon using SF₆/N₂ gas mixture: an alternative to etching Si in MEMS with Au components

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Abstract

In this work, the effects of the N₂ addition to the SF₆ plasma used in the isotropic silicon etching of Micro-electromechanical systems (MEMS) with Au components are investigated. A four-variables Doehlert design was implemented for optimizing the etching parameters (power, pressure, gas flow rate, and N₂/SF₆ ratio) to maximize the lateral etch rate of Si using SF₆/N₂ gas mixture. The optimized etch condition founded for a lateral etch rate of 1.8 μm/min was: power=143 W, chamber pressure=86 mTorr, flow rate=22 sccm, and N₂/SF₆ ratio=0.1. Furthermore, it was demonstrated that the established etching process avoids the structure damage of Au components.

Keywords: Isotropic Si RIE, Microstructure, Au Thin films, MEMS, SF₆/N₂ plasma, DOE

1. Introduction

Isotropic silicon (Si) etching is an important step for the fabrication of substrate-free Micro-electromechanical systems (MEMS) such as cantilever beams [1], membrane-based structures [2], or suspended thermal sensors [3]. This process is indeed used to release the microstructure from the underlying Si support or from a sacrificial Si layer. Meanwhile, gold (Au) is widely used as a component of this type of device, mainly for its good electrical and thermal conductivity. Considering a device where the design does not support a step of lithography to protect the Au components, like micro-bridge structures, the employed isotropic etching must be adapted so as not to damage the metal components. Wet etching

techniques are usually applied to obtain isotropic etching but are not adapted for the fabrication of very small devices considering the difficulty of controlling the process. Vapor phase etching using xenon difluoride (XeF_2) is an alternative method exclusively based on chemical reactions between the silicon and the etchants. Even though this method is a good option for Si isotropic etching, the XeF_2 is expensive and rare [4]. Reactive-Ion Etching (RIE) based on the physical/chemical process using SF_6 plasma is also an effective method for the isotropic etching of silicon due to a high density of radicals (SF_5^* and F^*) responsible for the Si lateral etching [5,6]. However, preliminary tests have shown that SF_6 plasma causes damage to Au thin films as shown in Fig. 1.

In this letter, we report the investigation of an RIE process using the SF_6/N_2 gas mixture as an alternative for the isotropic etching of silicon while decreasing the impact of the process on the Au structure. N_2 was chosen since it is an inert gas and contrary to the effect of O_2 addition, it does not cause a significant increase in anisotropy [7].

We used a four-variables Doehlert Design of Experiment (DOE) [8] to determine a relationship between etching parameters and the lateral etch rate minimizing the number of experiments. This kind of DOE is widely used for multivariate optimization of different processes and technologies [9–11]. In this study, the parameters were optimized to obtain a high lateral etch rate.

Fig. 1. Optical microscopy image of an Au thin film (a) before and (b) after 5 min of RIE using SF_6 gas.

2. Materials and methods

This study was performed on silicon patterned samples fabricated with a (100) silicon wafer with a 500 nm thick silicon dioxide (SiO_2) mask. The masking pattern consisted of equal lines and spaces both

with 20 μm -width as shown in Fig. 2(a). The SiO_2 mask layer was deposited using Plasma Enhanced Chemical Vapor Deposition (PECVD) and was patterned by laser lithography and RIE using CF_4/CHF_3 plasma gas. In order to determine the optimal parameters to obtain the maximum lateral etch rate of Si using SF_6/N_2 gas mixture, a Doehlert DOE [8] was implemented using the statistical analysis software Ellistat's DOE [12]. Four input factors have been selected for this study: pressure (mTorr), power (W), gas flow rate (sccm), and N_2/SF_6 ratio. Table 1 shows the maximum and minimum values set in the software for each selected factor and also the total number of experiments with the different etch parameter values generated by the DOE. The number of experiments (N) was determined according to $N = F^2 + F + N_0$, where F is the number of factors and N_0 the number of experiments at the central point: $N = 4^2 + 4 + 1 = 21$. The experiments were carried out in OIPT Plasmalab 80 RIE etcher at room temperature with an etching time of 7 minutes.

We have also investigated the impact of the etching process on Au (400 nm)/Ti (100 nm) thin films (50 μm -width) deposited by evaporation on a SiO_2/Si substrate.

3. Results and discussion

To investigate the influence of the etching parameters on the lateral silicon etching, we established a response surface methodology to obtain a predictive model. The lateral etch rate (R_{lat} /etching time) of the silicon was determined by cross-sectional SEM analysis measuring the R_{lat} (Fig. 2(b)). Table 1 presents the obtained measured responses. The 21 values of each response were used to compute the following second-order model that concerns the linear and quadratic effects of the factors and their interactions:

$$\text{Lateral etch rate} = 3,556 - 1,633Y_1 - 0,05148Y_2 - 0,00508Y_3 - 0,02024Y_4 + 0,01158Y_1Y_3 + 0,0006531Y_2^2 + 0,0001203Y_4^2 \quad (1)$$

where Y_1 =Ratio; Y_2 =Flow; Y_3 =Pressure and Y_4 =Power.

The coefficient of determination ($r^2=0.972$) indicates that the model is at first adequate to describe the lateral etch rate. Moreover, all terms present on Eq. (1) were found to be significant ($p\text{-value} \leq 0.05$). The predicted values generated by Eq. (1) are presented in the last column of Table 1. The

DOE analysis concerning the vertical etch rate is shown in Eq. (S1), Table S1 and Fig. S1 in the supplementary information.

Fig. 2. (a) Top-view and (b) cross-sectional SEM images of the samples. 3D surface plot of (c) Lateral rate vs. Ratio vs. Power and (d) vs. Flow rate vs. Pressure.

The 3D response surface plots (Fig. 2(c) and (d)) are the graphical representations of the equation and provide a visualization of the relationship between the etch rate and each parameter. The effect of the power and ratio variation (Fig. 2(c)) was analyzed setting the other parameters at their center point values (Flow rate=35 sscm and Pressure=114.5 mTorr). We can observe that power has a significant influence on the rate variation. The increase of the power causes an increase of ion energy and in the F-atom density that has an impact on the physical and chemical effect of the etch. On the other hand, the increase of the N₂ content causes a decrease in the lateral rate, probably due to a consequent SF₆ dilution[5].

Fig. 2(d) shows the response in function of the flow rate and pressure by fixing Power=114.5 W and Ratio=0.35 (center point values). It is observed that the increase of pressure causes a small linear

decrease of the lateral rate. In turn, the flow rate variation causes a non-linear variation of the lateral rate with a minimum value around 41 sccm.

Fig. 3. SEM images of the Au/Ti thin films etched by (a) SF_6 and by (b) SF_6/N_2 . EDX spectra obtained (c) from zone A and B and (d) from a sample etched by SF_6/N_2 and not etched (reference).

Our model generated the optimized values for each parameter to maximize the lateral rate. The optimization predicted a lateral rate of $1.8 \mu\text{m}/\text{min}$ for: Flow rate=22 sccm; Ratio=0.1; Pressure=86 mTorr and Power=143 W. A validation experiment was performed and obtained a lateral rate of $1.79 \mu\text{m}/\text{min}$, which shows good accuracy of the model. For comparison, a sample was etched applying the same parameters however using only SF_6 and a lateral rate of $1.66 \mu\text{m}/\text{min}$ was obtained. This result shows that the addition of 10% N_2 does not cause a significant F-atom density reduction and probably helps the formation of SF_5 and F radicals [7].

Finally, an Au/Ti thin film deposited on SiO_2 was exposed to the plasma using the optimal parameters founded, to evaluate the impact of this new process on the Au structure. A comparison between the thin film exposed to SF_6 plasma or SF_6/N_2 plasma was performed using SEM images and EDX spectra. Fig. 3(a) shows that the SF_6 plasma importantly damages the Au film. After 10 min of etching, the

film becomes porous (zone A) and the SiO₂ is highlighted (Zone B). This effect is confirmed by the EDX spectra of the different zones (Fig. 3(c)) which shows that the Au M α peak is not detected on Zone B in comparison to A confirming that the metallic layer was fully etched leaving only the oxide. In turn, Fig. 3(b) shows that the N₂ addition protects the metallic layer during the etching process. In this case, the Au surface is homogenous not showing a significant roughness caused by the plasma. In addition, the EDX spectrum obtained from the sample etched using SF₆/N₂ (Fig. 3(d)) is similar to the spectrum obtained from a reference sample (not etched) which evidences that this method does not influence significantly the Au thin film composition. The SEM image of the sample before etching (reference) is shown in Fig. S2 (Supporting Information).

4. Conclusion

In this work, a DOE was employed to optimize an SF₆/N₂ gas mixture based etching recipe to obtain the maximum Si lateral etching rate. It was found that the optimum conditions are: power=143 W, chamber pressure=86 mTorr, flow rate=22 sccm and N₂/SF₆ ratio=0.1 for a lateral rate=1.8 μ m/min. The experimental etch rate measured (1.79 μ m/min) proved to be in agreement with the predicted one. Furthermore, this recipe proved to be adapted to be applied in MEMS with Au components. The addition of 10% N₂ on the SF₆ plasma avoids the Au damage and increases the lateral etching rate of silicon under the studied conditions.

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