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

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7 **Abstract**

8
9 In this work, the effects of the N₂ addition to the SF₆ plasma used in the isotropic silicon etching of Micro-
10 electromechanical systems (MEMS) with Au components are investigated. A four-variables Doehlert
11 design was implemented for optimizing the etching parameters (power, pressure, gas flow rate, and N₂/SF₆
12 ratio) to maximize the lateral etch rate of Si using SF₆/N₂ gas mixture. The optimized etch condition
13 founded for a lateral etch rate of 1.8 μm/min was: power=143 W, chamber pressure=86 mTorr, flow
14 rate=22 sccm, and N₂/SF₆ ratio=0.1. Furthermore, it was demonstrated that the established etching
15 process avoids the structure damage of Au components.

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

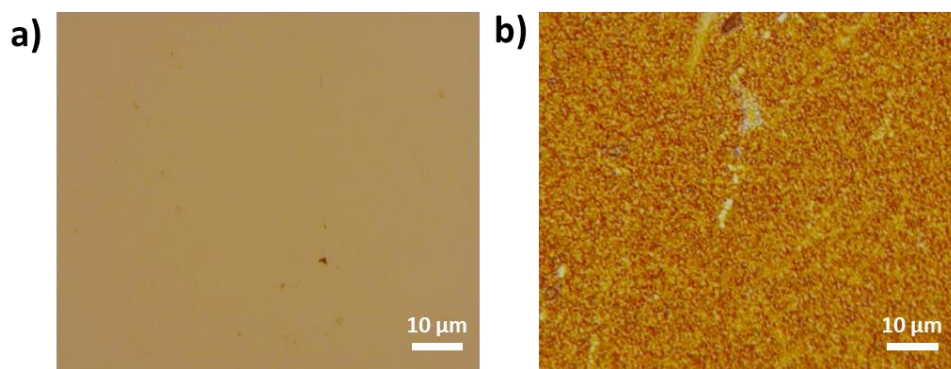
17 **1. Introduction**

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

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

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

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



42
43 Fig. 1. Optical microscopy image of an Au thin film (a) before and (b) after 5 min of RIE using SF_6 gas.

44 2. Materials and methods

45

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

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

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

61

62 **3. Results and discussion**

63

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

$$70 \text{ Lateral etch rate} = 3,556 - 1,633Y_1 - 0,05148Y_2 - 0,00508Y_3 - 0,02024Y_4 + 0,01158Y_1Y_3 +$$

$$71 0,0006531Y_2^2 + 0,0001203Y_4^2 \quad (1)$$

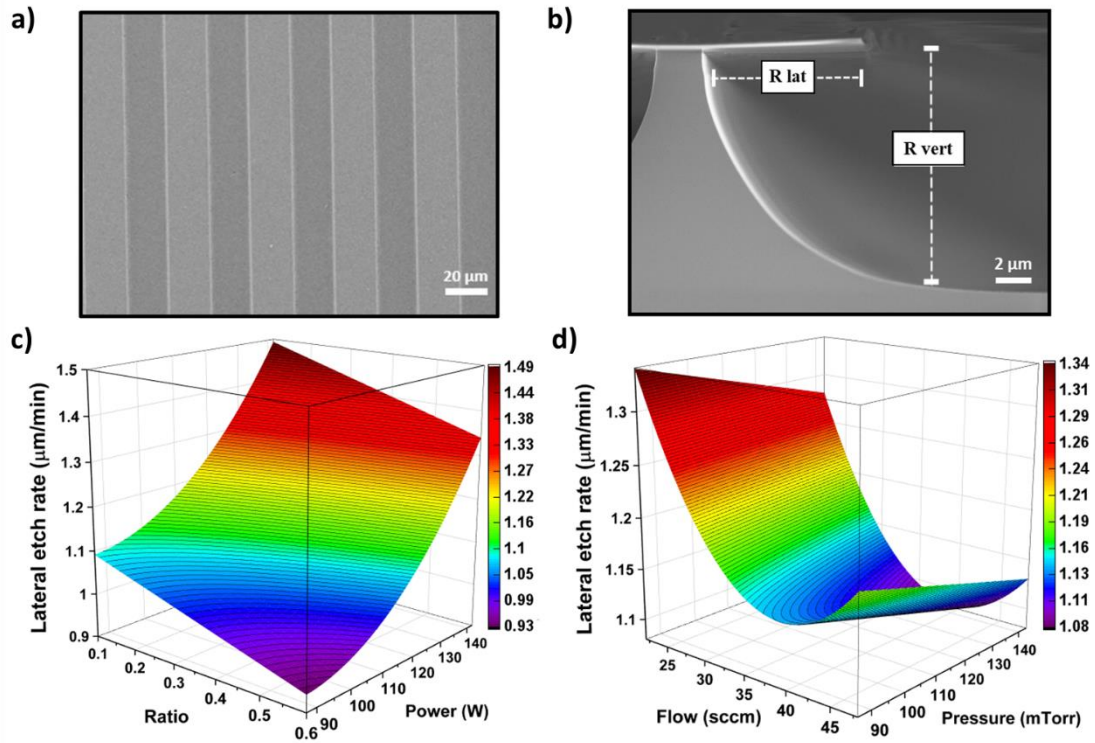
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73 where Y_1 =Ratio; Y_2 =Flow; Y_3 =Pressure and Y_4 =Power.

74

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

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

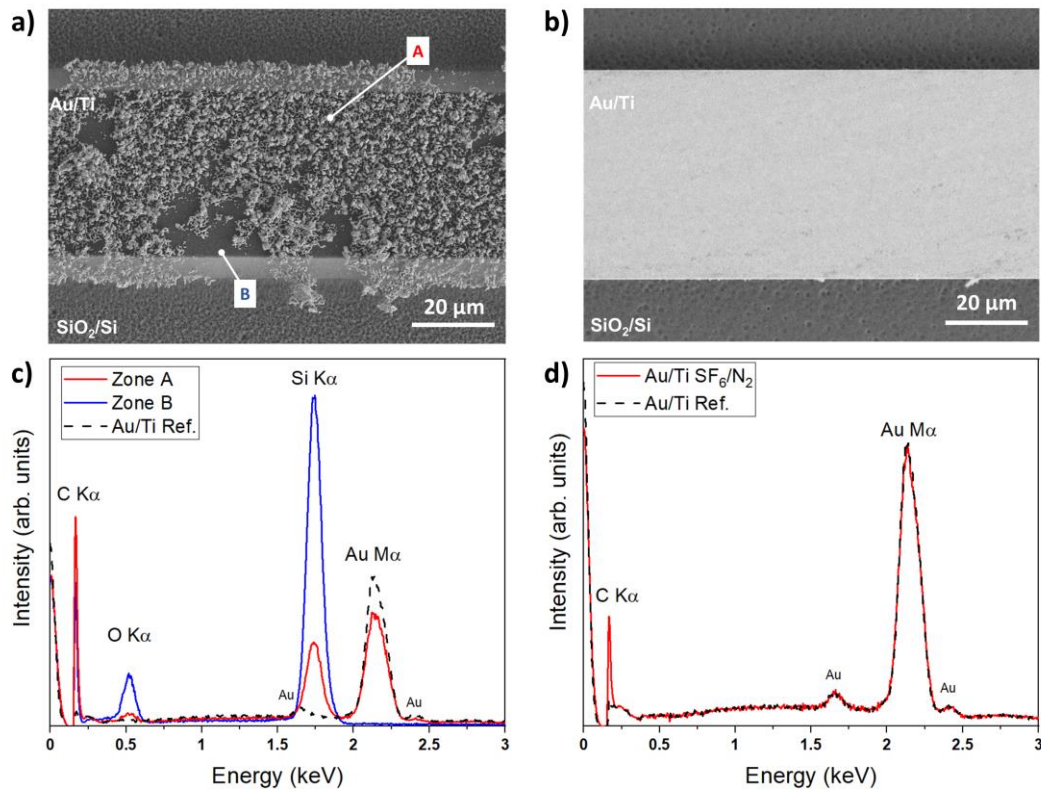


80
81 Fig. 2. (a) Top-view and (b) cross-sectional SEM images of the samples. 3D surface plot of (c) Lateral rate
82 vs. Ratio vs. Power and (d) vs. Flow rate vs. Pressure.

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

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

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



95
 96
 97 Fig. 3. SEM images of the Au/Ti thin films etched by (a) SF₆ and by (b) SF₆/N₂. EDX spectra obtained (c)
 98 from zone A and B and (d) from a sample etched by SF₆/N₂ and not etched (reference).

99 Our model generated the optimized values for each parameter to maximize the lateral rate. The
 100 optimization predicted a lateral rate of 1.8 μm/min for: Flow rate=22 sccm; Ratio=0.1; Pressure=86 mTorr
 101 and Power=143 W. A validation experiment was performed and obtained a lateral rate of 1.79 μm/min,
 102 which shows good accuracy of the model. For comparison, a sample was etched applying the same
 103 parameters however using only SF₆ and a lateral rate of 1.66 μm/min was obtained. This result shows that
 104 the addition of 10% N₂ does not cause a significant F-atom density reduction and probably helps the
 105 formation of SF₅ and F radicals [7].

106 Finally, an Au/Ti thin film deposited on SiO₂ was exposed to the plasma using the optimal
 107 parameters founded, to evaluate the impact of this new process on the Au structure. A comparison
 108 between the thin film exposed to SF₆ plasma or SF₆/N₂ plasma was performed using SEM images and EDX
 109 spectra. Fig. 3(a) shows that the SF₆ plasma importantly damages the Au film. After 10 min of etching, the

110 film becomes porous (zone A) and the SiO₂ is highlighted (Zone B). This effect is confirmed by the EDX
111 spectra of the different zones (Fig. 3(c)) which shows that the Au M α peak is not detected on Zone B in
112 comparison to A confirming that the metallic layer was fully etched leaving only the oxide. In turn, Fig.
113 3(b) shows that the N₂ addition protects the metallic layer during the etching process. In this case, the Au
114 surface is homogenous not showing a significant roughness caused by the plasma. In addition, the EDX
115 spectrum obtained from the sample etched using SF₆/N₂ (Fig. 3(d)) is similar to the spectrum obtained
116 from a reference sample (not etched) which evidences that this method does not influence significantly
117 the Au thin film composition. The SEM image of the sample before etching (reference) is shown in Fig. S2
118 (Supporting Information).

119 **4. Conclusion**

120
121 In this work, a DOE was employed to optimize an SF₆/N₂ gas mixture based etching recipe to
122 obtain the maximum Si lateral etching rate. It was found that the optimum conditions are: power=143 W,
123 chamber pressure=86 mTorr, flow rate=22 sccm and N₂/SF₆ ratio=0.1 for a lateral rate=1.8 $\mu\text{m}/\text{min}$. The
124 experimental etch rate measured (1.79 $\mu\text{m}/\text{min}$) proved to be in agreement with the predicted one.
125 Furthermore, this recipe proved to be adapted to be applied in MEMS with Au components. The addition
126 of 10% N₂ on the SF₆ plasma avoids the Au damage and increases the lateral etching rate of silicon under
127 the studied conditions.

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