

Plasma dry etching of monocrystalline silicon for the microsystem technology

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Plasma dry etching is the main process utilized in the microelectronics technology. This technique can be useful in the micromachining of single crystal silicon objects for the microsystem technology. Reactive ion etching (RIE) procedures of isotropic and anisotropic fabrication of the three-dimensional (3D) silicon microstructures, in SF₆-base plasmas, were studied. Applications of the developed dry plasma etching processes are presented.

1. Introduction

Microsystem – a miniature smart system – consists of micromechanical sensors and actuators, and of the electronic circuits (microprocessor), which ensures the connection with informatics environment. The pressure and acceleration transducers, generally used in automotive industry, *e.g.*, air bags, ABS, EPS systems, are the best known, mass manufactured microsystems. The second wide application field of microsystems is medicine. Now, smart endoscopes, miniature devices for angioplasty, microinstruments for non-invasive surgery, and microdevices for drug delivery have been developed.

Most often, a sensor and an actuator, as parts of a microsystem, are made of monocrystalline silicon, and the characteristic dimensions of these elements are about single micrometers or less. Micromechanical sensors and actuators are fabricated by use of the integrated microelectronics techniques, and also by use of such new techniques like bulk or surface micromachining of silicon. The following methods are used for the fabrication of 3D silicon structures:

- wet, deep, anisotropic or isotropic silicon etching (for example: water solutions of KOH – potassium hydroxide, EDP – ethylene diamine pyrochatechol, TMAH – tetramethylammonium hydroxide, HF+HNO₃+CH₃COOH – hydrofluoric acid + nitric acid + acetic acid),

- dry, deep, anisotropic or isotropic silicon etching (ion beam, freon plasma).

Generally, plasma dry etching is applied in VLSI (very large scale integration) technology, because this technique makes it possible to obtain the width of pattern line less than 1 μm.

In the paper, results of the investigations of reactive ion etching (RIE) of silicon for the microsystem technology applications are presented. Two main goals of the works were to test the parameters of the dry etching of silicon in SF₆ plasmas (RIE), and to chose the optimum etching procedures for the 3D silicon microstructures fabrication.

2. Plasma etching

Plasma, reactive ion etching, is used for the formation of cavities, deep trenches and free elements in a silicon wafer – it is a bulk micromachining. The dry etching process is also applied to release the free-standing parts of sensors and actuators, like beams, rotors, cantilevers, membranes, springs – it is a surface micromachining [1]. RIE of silicon is carried out in ionised freons, usually SF₆ is used [1]–[3]. SF₆ plasma in comparison with chlorine and bromine compounds is characterized by the high etching rate of silicon and low harmfulness for the environment. For SF₆, dissociation, ionisation and attachment reaction release the fluorine free radicals that carry out etching of the bulk silicon. Silicon is removed from not-masked wafer surface mainly as SiF₄, a volatile product of the chemical process, which is pumped out by a vacuum system. Due to ions presence in plasma, RIE proceeds also as a physical process. Then the gas mixture besides SF₆ includes also other gases, *e.g.*, oxygen (a photoresist or an aluminium mask covers the silicon surface), deposition of the thin polymer layers SiO_xF_y and AlO_xO_y is observed [2]. This effect influences the value of etching rate, and consequently, the shape of the obtained silicon microstructures.

Today, in the microsystem technology, plasma etching allows semiconductors, insulators, and metals to be etched anisotropically (*i.e.*, with high directionality, the material is removed in vertical direction only). The anisotropy A can be defined in terms of the vertical ($V_{\text{et}\downarrow}$), and lateral ($V_{\text{et}\rightarrow}$) etching rates as: $A = 1 - V_{\text{et}\rightarrow} / V_{\text{et}\downarrow}$. This parameter depends on the electrodes configuration of the plasma etcher, on the process parameters, like composition and pressure of reactive gases, the flow rate of the gases, and on the frequency and power of the plasma supply. An accurate pattern transfer requires the substrate material to be high rate etched with respect to the masking layer. This parameter is called the etch selectivity S , and may be defined in terms of the first material V_1 and the second material V_2 etching rates as: $S = V_1 / V_2$.

The SF₆/freonTM 115 was chosen for the study on the basis of literature data. SF₆ plasma is known as an isotropic etchant of silicon. Anisotropy can be improved by the modification of the gas mixture composition. Elaboration of isotropic and anisotropic procedures for silicon microstructures fabrication is the goal of this work.

3. Experiments

The investigations of RIE plasma etching of n-type, (100) and (111) silicon wafers for various compositions of SF₆, Cl₂, O₂ were carried out. A GIR-300 Alcatel (France) asymmetric parallel plate RIE equipment was used. A GIR-300 contains 8 independent

gas lines, which allow etching and depositing of different materials. Lower electrode is driven by radio frequency (RF) generator at 13.56 MHz. The chamber is evacuated by a turbomolecular pump enabling to reach base pressure of 5×10^{-7} kPa. After each etching process the micrographs were taken on a Hitachi scanning electron microscope. Anisotropy, selectivity and etching rate were estimated directly from the micrographs.

3.1. Silicon etching in SF₆ plasma

The etching was initially optimized using SF₆ alone, and varying the pressure, flow rate and mask material. As a mask, an array pattern consisting of 1 μm thick thermal oxide circles ($\phi = 5, 8, 15 \mu\text{m}$) was used. The following parameters of the etching

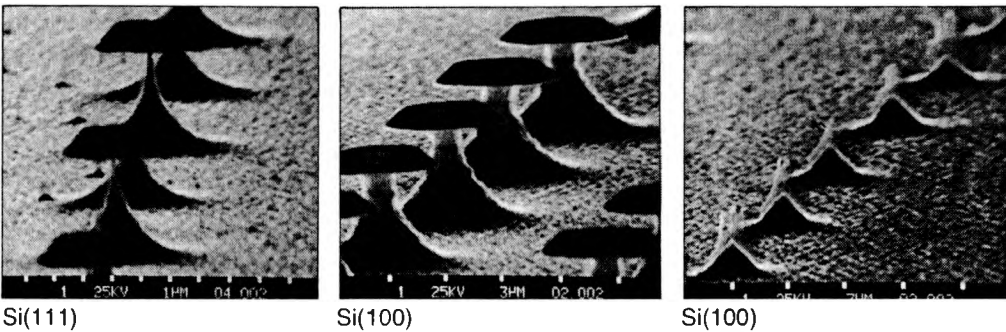


Fig. 1. Silicon structures with SiO₂ masks, etched with 12 sccm SF₆, 4×10^{-3} kPa, 100 W. The obtained results: $V_{\downarrow} = 0.56 \mu\text{m}/\text{min}$, $V_{\rightarrow} = 0.5 \mu\text{m}/\text{min}$, $V_{\text{SiO}_2\downarrow} = 40 \text{ nm}/\text{min}$, $A = 0.11$, $S_{\text{Si}/\text{SiO}_2} = 14:1$.

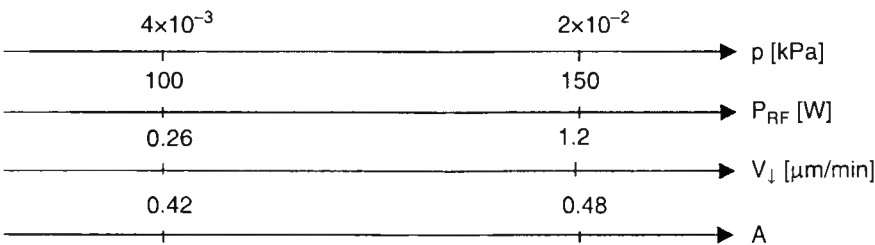
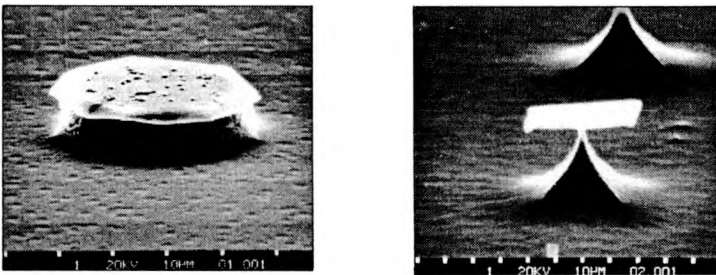


Fig. 2. Illustration of the parameters of (100) silicon etching processes carried out in SF₆ plasma, and obtained results vs. pressure and power supply. Aluminium masks (0.3 μm) were used ($S_{\text{Si}/\text{Al}} = 100:1$).

process were set at: pressure $p = 4 \times 10^{-3}$ kPa, flow rate $f_{\text{SF}_6} = 12$ sccm, power of a glow discharge $P_{\text{RF}} = 100$ W. It has been confirmed that etching rate of (100) Si, and (111) Si is similar, and is $V_{\text{et}\downarrow} = 0.56$ $\mu\text{m}/\text{min}$ in perpendicular direction, $V_{\text{et}\rightarrow} = 0.5$ $\mu\text{m}/\text{min}$ laterally (Fig. 1). Anisotropy is low ($A = 0.11$), selectivity of silicon and silicon dioxide etching is $S = 14:1$. For above process parameters the thermal dioxide silicon layer was etched with velocity $V_{\text{SiO}_2\downarrow} = 40$ nm/min. An increase in the power supply causes a rise in the SiO_2 mask-layer rate etching. Thus, this process cannot be applied to deep silicon etching for the microsystem technology.

In the following works, the silicon etching was carried out through the 0.3 μm thick aluminium mask layer. Mask pattern contains a series of the circles and squares with dimensions 50, 75, 120, 150 μm . Processes were done for: SF_6 flow rate $f_{\text{SF}_6} = 12$ sccm, total gas pressure $p = 4 \times 10^{-3}$ kPa, and 2×10^{-2} kPa, power supply $P_{\text{RF}} = 100$ and 150 W, respectively (Fig. 2). It was confirmed that the silicon etching rate is larger for high pressure of SF_6 , $V_{\text{et}\downarrow} = 1.2$ $\mu\text{m}/\text{min}$ in comparison with 0.26 $\mu\text{m}/\text{min}$ for lower pressure. Etch anisotropy was slightly better, $A = 0.48$ in comparison with $A = 0.42$. Thus, the etching process with the following parameters: SF_6 , 2×10^{-2} kPa, 150 W, Al mask, can be used for the silicon structures formation in the microsystem technology.

3.2. Silicon etching in SF_6/Cl_2 plasma

Etching processes were carried out for n- and p-type, (111) and (100) oriented silicon wafers. Silicon structures were formed through the square and circular masks ($\phi = 5, 10, 15$ μm), made of 1 μm thick, thermal dioxide layer. The following process parameters were applied: $\text{SF}_6:\text{Cl}_2 = 24$ sccm:6 sccm, $p = 4 \times 10^{-3}$ kPa, $P_{\text{RF}} = 150$ W. For this process the parameters $A = 0.6$, and $V_{\text{et}\downarrow} = 0.5$ $\mu\text{m}/\text{min}$ were obtained. In the SEM photos a thin polymer layer can be seen (Fig. 3a). When the mask layer is removed, a rough surface of the silicon is visible (Fig. 3c). High reactivity of the chlorine ions, and the thin polymer layer have a large influence on the quality of the silicon surface. It was estimated that the thermal silicon dioxide mask was etched at

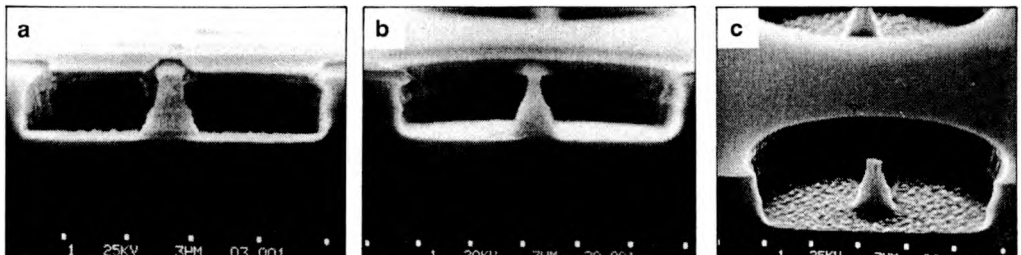


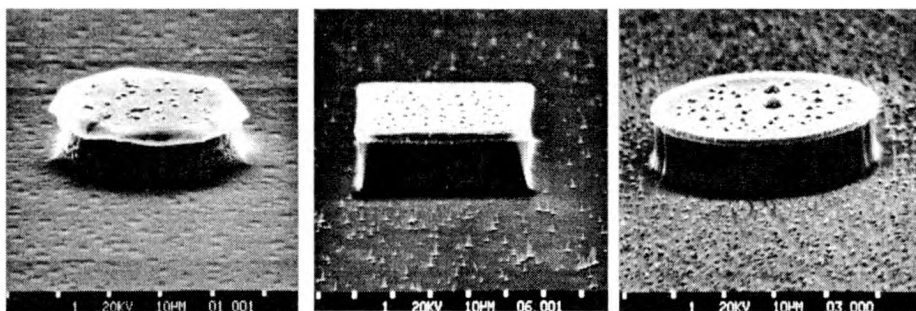
Fig. 3. Silicon structures etched in SF_6/Cl_2 plasma ($p = 4 \times 10^{-3}$ kPa, $f_{\text{SF}_6} = 24$ sccm, $f_{\text{Cl}_2} = 6$ sccm, $P_{\text{RF}} = 150$ W): a – directly after etching process, b – after etching $\text{HF}:\text{H}_2\text{O} = 1:100$ solution, c – after SiO_2 mask layer removing. The obtained results: $V_{\downarrow} = 0.5$ $\mu\text{m}/\text{min}$, $V_{\rightarrow} = 0.2$ $\mu\text{m}/\text{min}$, $V_{\text{SiO}_2\downarrow} = 60$ nm/min. $A = 0.6$, $S_{\text{Si}/\text{SiO}_2} = 8:1$.

the rate of $V_{SiO_2\downarrow} = 60$ nm/min. Mask erosion is larger near the edge (Fig. 3b). Thus, the dry etching process in SF_6/Cl_2 plasma is not suitable for deep silicon etching, because aluminium masks cannot be used, and oxide masks are characterized by low etch selectivity with regard to silicon.

3.3. Silicon etching in SF_6/O_2 plasma

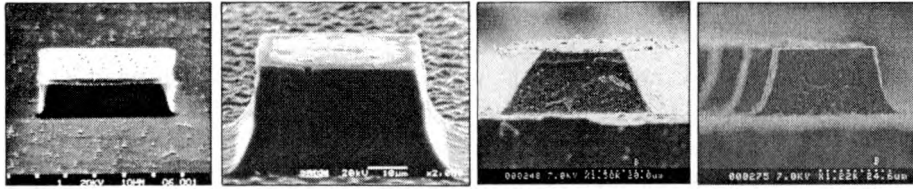
The works were continued for SF_6/O_2 gas mixtures. It has been expected that oxygen addition to SF_6 will cause an increase in gas ionisation efficiency and, subsequently, in the etching rate of silicon. The base parameters of the etching processes were: total gas pressure $p = 4 \times 10^{-3}$ kPa, and 2×10^{-2} kPa, SF_6 flow rate $f_{SF_6} = 12$ sccm. For different oxygen additions to SF_6 , in the range from 0 to 8 sccm, anisotropy and etching rate were estimated. The mask pattern was composed of circles and squares made of $0.3 \mu m$ thick aluminium layer (50, 100, 150 μm).

In the SEM pictures taken for etched structures a thin polymer layer can be seen (Fig. 4). This polymer layer covers the sidewalls of silicon structures, and the whole surface of a silicon wafer. The silicon surface development is observable. Etch anisotropy was larger for a higher oxygen flow rate. The best etch directionality for the oxygen flow rate of 4 sccm was obtained. For large quantity of oxygen in the gas mixture and for higher power supply ($P_{RF} > 150$ W) a decrease in the etching rate was observed (Fig. 5). A similar study was carried out for the higher pressure of SF_6/O_2 gas mixture (Fig. 6). Maximum etching rate for the oxygen flow rate of 2 sccm was



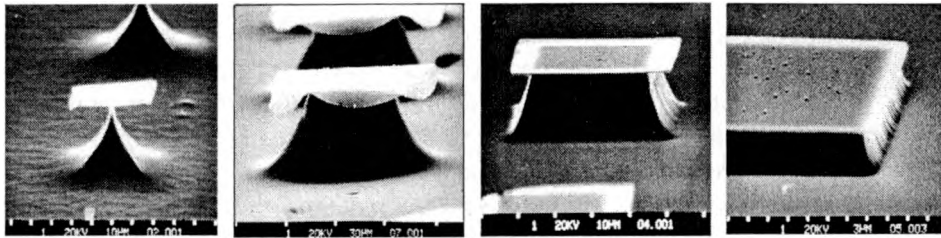
0	2	4	f_{O_2} [sccm]
100	100	100	P_{RF} [W]
0.26	0.46	0.30	V_{\downarrow} [$\mu m/min$]
0.42	0.80	0.86	A

Fig. 4. Illustration of the parameters of (111) silicon etching processes carried out in SF_6/O_2 plasma, and obtained results vs. oxygen flow rate, $f_{SF_6} = 12$ sccm, total gas pressure $p = 4 \times 10^{-3}$ kPa. Aluminium masks ($0.3 \mu m$) were used ($S_{Si/Al} = 100:1$).



100	150	175	200	→ P _{RF} [W] V _↓ [μm/min] A
0.46	1.25	0.80	0.76	
0.80	0.80	0.63	0.60	

Fig. 5. Illustration of the parameters of (111) silicon etching processes carried out in SF₆/O₂ plasma, and obtained results vs. power supply, f_{SF₆} = 12 sccm, f_{O₂} = 2 sccm, total gas pressure p = 4 × 10⁻³ kPa. Al masks (0.3 μm) were used (S_{Si/Al} = 100:1).



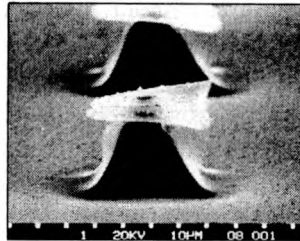
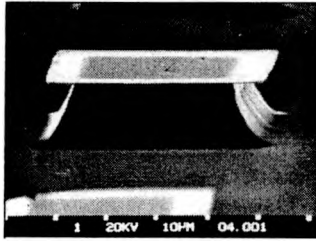
0	2	4	8	f _{O₂} [sccm] P _{RF} [W] V _↓ [μm/min] A
150	100	100	100	
1.20	1.48	0.48	0.20	
0.48	0.26	0.54	0.63	

Fig. 6. Illustration of the parameters of (111) silicon etching processes carried out in SF₆/O₂ plasma, and obtained results versus oxygen flow rate, f_{SF₆} = 12 sccm, total gas pressure p = 2 × 10⁻² kPa. Al masks (0.3 μm) were used (S_{Si/Al} = 100:1).

obtained. For a larger oxygen flow rate the etch anisotropy was better. Next, the etching process for the oxygen flow rate of 4 sccm, and P_{RF} = 200 W was carried out (Fig. 7). A significant increase in the etching rate and slight decrease in the etch anisotropy were obtained.

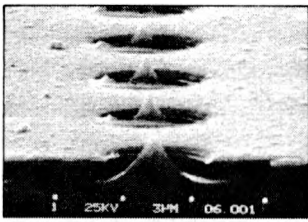
4. Summary

The control of gas mixture plasma composition allows to obtain an isotropic, as well as an anisotropic profile of dry RIE silicon microstructures. To obtain a selective process of the deep silicon etching in SF₆ plasma, an aluminium mask should be

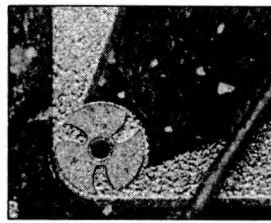


100	200	→ P _{RF} [W]
0.48	1.37	→ V _d [µm/min]
0.54	0.46	→ A

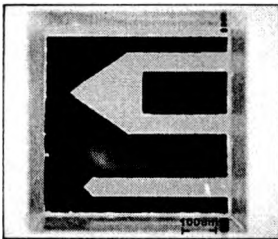
Fig. 7. Illustration of the parameters of (111) silicon etching processes carried out in SF₆/O₂ plasma, and obtained results vs. power supply, $f_{SF_6} = 12$ sccm, $f_{O_2} = 4$ sccm, total gas pressure $p = 2 \times 10^{-2}$ kPa. Al masks (0.3 µm) were used ($S_{Si/Al} = 100:1$).



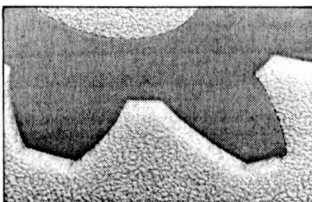
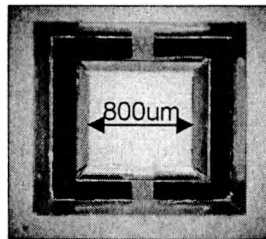
a



b



c



d

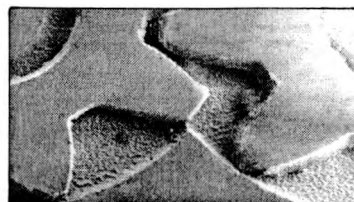


Fig. 8. Applications of SF₆ plasma etching: silicon emitters array with Cr–Au metallic gate (a), silicon micromachine with a dry etched flow channel (b), dry-release free-standing silicon structures (c), fragment of the silicon turbine, and an assembled gear (d).

applied. Etching in SF₆ plasma without any additions makes it possible to obtain the isotropic profile of an etched structure and a smooth surface of the silicon wafer. Oxygen addition into SF₆ plasma causes an increase in the etch anisotropy, decrease in the etching rate and the deterioration of the silicon surface quality.

An elaborated isotropic dry etching procedure was applied to the fabrication of the silicon emitter array with Cr–Au steering gate (Fig. 8a) [4]. A flow microchannel with an axle, the element of a silicon micromachine (with a metallic microturbine, $\phi = 100 \mu\text{m}$), was produced with the aid of the SF₆ RIE process (Fig. 8b) [5]. The anisotropic dry etching procedure was used to release the micromechanical silicon structures (Fig. 8c) and for fabrication of a silicon microgear with silicon rotors ($\phi = 1 \text{ mm}$, Fig. 8d) [6].

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