Reactive ion etching of silicon using low-power plasma etcher

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Abstract. The paper is devoted to the study of deep reactive ion etching of silicon using diode plasma etcher system with a low-power source. Silicon wafers were etched in a sulfur hexafluoride plasma and sulfur hexafluoride/oxygen plasma. The maximum achieved silicon etch rate was about 2 μm/min. The expediency of using dry reactive ion etching in combination with wet anisotropic etching of silicon for manufacturing of microelectromechanical systems (MEMS) was demonstrated.

1. Introduction

Vertical etching of silicon is widely used for forming relief structures in MEMS technologies. Traditionally, wet anisotropic etching of silicon is used for this purpose [1]. Its advantages are the ease of operation and low cost. Wet anisotropic etching of silicon also provides high reproducibility of processing modes and the best uniformity of etched surface [2]. Unfortunately, the etching ability of wet etchants decreases during the etching process. This leads to reduction of etch rate and uniformity of the etched surface. Therefore, forming deep cavities in silicon wafers using wet anisotropic etching often requires replacement of etchant. This complicates the production process and reduces the percentage yield.

Further development of MEMS technologies and associated technological equipment leads to increasingly frequent application of dry etching methods. Dry etching of silicon is characterized by some advantages over wet etching. First, dry etching of silicon provides near-vertical sidewalls of the etched cavities [3]. This allows one to increase the quantity of chips on the substrate, thereby reducing their production cost. Dry etching also provides higher etch rate of silicon in comparison with wet etching. In addition, the dry etching rate is less dependent on the silicon crystallographic orientation [4]. However, the application of dry etching requires expensive equipment.

MEMS technologies often require deep silicon etching with high uniformity of the etched surface. In this case, it is convenient to use dry reactive ion etching in combination with wet etching. One of the MEMS technologies is manufacturing dielectric membrane structures for sensitive elements of semiconductor gas sensors. Such structures are fabricated by dielectric film deposition on the surface of silicon substrate and subsequent substrate etching from the reverse side up to form reach-through cavities under the membranes. Initially, deep wet anisotropic etching of silicon is conducted. Then, dielectric film and sensitive layers are formed on the etched film. Finally, deep dry vertical etching of the residual silicon layer is performed to form reach-through cavities under the deposited membranes [5–7]. This allows one to exclude the influence of etchant solution on the formed sensitive layers, increasing the percentage yield. We propose the use of reactive ion etching of silicon as the dry vertical etching method in the described technology. The paper presents study results of deep reactive
ion etching of silicon and expediency assessment of its use in combination with wet anisotropic etching of silicon for manufacture of MEMS based on dielectric membrane structures.

2. Study of deep reactive ion etching of silicon

Phosphorus-doped single crystal silicon wafers of orientation (100) with the resistivity of 4.5 Ωcm were used to study deep reactive ion etching of silicon. 0.5-μm-thick silicon dioxide and 0.3-μm-thick nickel films were used as etching masks. Silicon dioxide films were formed by thermal oxidation of silicon in the dry-wet-dry mode at the temperature of 1150 °C. Initially, oxidation of silicon wafers was conducted in the ambient of dry oxygen, then in the ambient of water vapor. The final stage of silicon oxidation was performed in the ambient of dry oxygen. Nickel films were formed using magnetron sputtering. The masking pattern in the form of square holes with the size of 1 × 1 mm² was obtained using photolithography and subsequent wet etching. After this, silicon dioxide films were etched in buffered oxide etchant at the temperature of 20 °C. Nickel films were etched in 25% aqueous hydrogen nitrate solution of at the temperature of 70 °C.

The study was performed using the reactive ion etcher with planar diode-type reactor. The unit configuration is schematically shown in figure 1. Silicon wafers were placed without fixing on the stationary base electrode equipped with a flowing water cooling system. The ion etcher was equipped with a single stage vacuum system. Vacuum was provided by the Agilent Varian SD-700 dual stage rotary vane vacuum pump. The base electrode was powered by the high-frequency Dressler Cesar 136 generator. The study was carried out at plasma discharge power of 50–140 W. The reaction chamber was equipped with two-channel gas injection system. The working gas was supplied into the reaction chamber through the counter electrode equipped with the gas diffuser. Gas flows into the chamber were controlled by electronic regulators of gas rate. Only high-purity gases were used in this study.

![Figure 1. Reactive ion etcher: 1 - grounded counter electrode with gas diffuser; 2 - vacuum pump tract; 3 - silicon wafer; 4 - base electrode with resistance matching system; 5 - high-frequency voltage source.](image)

Deep reactive ion etching of silicon was studied in a sulfur hexafluoride plasma and sulfur hexafluoride/oxygen plasma. The etching time of all samples in all processing modes was under 10 minutes. The aim of the study was to select the optimal silicon etching modes. Silicon etch rate was calculated from the depth of the etched cavities measured using Veeco Dektak 150 profilometer. The influence of the high-frequency power supply power on silicon etch rate was studied. The influence of sulfur hexafluoride flow rate into the reaction chamber on silicon etch rate was also studied. Dependences of the silicon etch rate on the power of the high-frequency power supply at various flow rates of sulfur hexafluoride were obtained (see figure 2). The influence of oxygen content in the mixture with sulfur hexafluoride on silicon etch rate was also studied. The flow of sulfur hexafluoride into the reaction chamber was assumed constant and equal to 18 cm³/min. Oxygen flow was varied during the study and dependencies of the silicon etch rate on the oxygen content in the mixture with sulfur hexafluoride at various power values of the high-frequency power supply were obtained (see figure 3). The smooth curves in figures 2 and 3 passing through experimental points were obtained with interpolation by the method of cubic spline using Origin Pro 9.1 software (Origin Lab corp.).
3. Results and discussion

As can be seen from figure 2, the silicon etch rate gradually increases with the increase in the power of the high-frequency power supply and sulfur hexafluoride flow rate into the reaction chamber. The maximum achieved silicon etch rate in the sulfur hexafluoride plasma was about 0.6 μm/min. This etch rate was obtained when the sulfur hexafluoride flow rate into the reaction chamber was of 18 cm³/min and the power of the high-frequency power supply was of 120 W. Adding oxygen into the sulfur hexafluoride flow was seen to increase the silicon etch rate. The maximum achieved silicon etch rate in the sulfur hexafluoride/oxygen plasma was about 2 μm/min. This etch rate was obtained when the oxygen content in the sulfur hexafluoride flow was about 10%. Taking into account the mathematical approximation, the maximum silicon etch rate is achieved when the oxygen content in the sulfur hexafluoride flow is about 14%. Further increase in the oxygen content in the mixture leads to the reduction of silicon etching rate, as shown in figure 3.

In order to explain this, it is necessary to consider the processes occurring in the reaction chamber during the reactive ion etching of silicon. Plasma discharge activates successive dissociation of sulfur hexafluoride resulting in the formation of fluorine atoms that have one unpaired electron. These atoms
are adsorbed by the wafer surface and react with the silicon. The product of this chemical reaction is silicon tetrafluoride which is then desorbed from the wafer surface. When feeding the mixture of sulfur hexafluoride and oxygen into reaction chamber, the following processes also occur. The plasma discharge activates oxygen dissociation in addition to the sulfur hexafluoride dissociation which leads to formation of oxygen atoms that have one unpaired electron. These oxygen atoms clean the wafer surface of the deposited sulfur, forming gaseous sulfur oxide. The oxygen atoms are also adsorbed by the wafer surface and react with silicon. As a result, a thin film of silicon oxide forms on the wafer surface.

The increase in silicon etch rate at low oxygen content in the mixture with sulfur hexafluoride is caused by the cleaning of the wafer surface through sulfur oxidation that is transferred into the gaseous state. Further increase in the oxygen content in the mixture leads to increase in silicon oxidation rate. If silicon oxide film forms on the wafer surface, fluorine ions are expended on its removal. Therefore, the resultant etch rate of silicon is reduced.

The maximum rate of reactive ion etching of silicon was obtained when the power of the high-frequency power source was of 120 W. Further increase in plasma discharge power significantly reduces the silicon etch rate. This is explained by the sputtering of the base electrode and chamber walls taking place at higher discharge power. The sputtered materials are partially deposited on the wafer surface and result in masking the silicon etching surface.

The studied silicon etching modes revealed that the 0.5-μm-thick silicon dioxide mask is mostly etched off the wafer surface. Therefore, such a mask can only be used when silicon etching is performed on the depth of several tens of micrometers. In this study, the nickel mask was characterized by high etch resistance both in the sulfur hexafluoride plasma and in the sulfur hexafluoride/oxygen plasma. This means that for deep reactive ion etching of silicon it is necessary to use metal masking layers, in particular, nickel masks.

4. Conclusions
A mode of deep reactive ion etching of silicon was found which provides sufficiently high silicon etch rate and acceptable uniformity of the etched surface. Feeding sulfur hexafluoride flow with oxygen content of 10% into the reaction chamber resulted in the silicon etch rate of about 2 μm/min. This mode allows etching 100-μm-thick silicon layers using a 0.3-μm-thick nickel mask. It was also shown that it is expedient to use dry reactive ion etching in combination with wet etching to form deep relief in silicon wafers for manufacture of MEMS. The use of deep reactive ion etching of silicon at the final stage of dielectric membrane structures formation for sensitive elements of semiconductor gas sensors improves the percentage yield. This is achieved through the absence of etchant contact with sensitive layers, and also through the absence of rapid changes in temperature and pressure typical for wet silicon etching at the final stage of the membranes formation.

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References
[1] Rouhi J, Mahmud S, Hutagalung S D, Naderi N, Kakooei S and Abdullah M J 2012 *Semiconductor Sci. Tech.* 6 27 065001
[2] Shikida M, Inagaki N, Sasaki H, Amakawa H, Fukuwara K and Sato K 2010 J. *Micromech. Microeng.* 1 20 015038
[3] Azimi S, Sandoughsaz A, Amirsoleimani B, Naghsh-Nilchi J and Mohajerzadeh S 2011 J. *Micromech. Microeng.* 7  21 074005
[4] Nozawa Y 2010 J. *Vac. Soc. Japan* 7 53 446
[5] Veselov D S 2015 *Phys. Procedia* 72 500
[6] Veselov D S and Voronov Yu A 2015 *Phys. Procedia* 71 423
[7] Veselov D S, Voronov Yu A and Vanyukhin K D 2015 *Phys. Procedia* 72 495