Patterning process of SiO_2 film and fabrication of Si V-groove

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In the process of silicon wet etching, the SiO_2 film formed by thermal oxidation is firm and compact, and it is an excellent mask material. However, there are some difficulties in its patterning process. Considering the high density of the SiO_2 film, its etching time is so long that the protective layer photoresist wrinkles and floats. In this letter, a novel way is used to achieve the graphical process resorting to the MgO film. A layer of the MgO film as the protective layer is deposited on the surface of the SiO_2 film, and inductively coupled plasma etching is used to etch the SiO_2 film. Then the chip is put in the KOH solution to fabricate the Si V-groove. The results show that the patterning process is easy to control.

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The use of the KOH solution for anisotropic etching of silicon is an important technology in micromachining, with a very wide range of applications in sensors, actuators, and micromaches. Anisotropic etching of silicon is simple in principle. There are numerous studies and applications of the anisotropic etching of silicon chip technology. In the actual process, we usually take the SiO_2 film that forms in thermal oxidization as the masking layer in the KOH solution. The SiO_2 film is very dense and is a good material as the masking layer. However, the patterning process of SiO_2 films involves some difficulties. This letter mainly deals with the use of inductively coupled plasma (ICP) etching to realize the patterning process of SiO_2 films, as well as the anisotropic wet etching of silicon with the SiO_2 film as the masking layer in the KOH solution.

A silicon single crystal has a diamond structure and is anisotropic. Some crystallographic directions or crystal planes are very important. The commonly used crystallographic directions are (100), (111), and (110). In the crystal, the arrangements of atoms are dissimilar in different directions, and the density degrees are also different. When the atomic arrangement is most compact on some crystal face, this crystal face is called the closepacked plane. The close-packed plane of Si is the (111) plane. The atom on the close-packed plane is the most compact plane, and one of its features is slower etching rate than that of other crystal planes in the anisotropic etching solution. Under certain conditions, the etching rate of the (111) crystal plane is only a few hundreds of the (100) crystal plane^[1].

Grooves are made along the (110) crystallographic direction on the (100) crystal plane of silicon, whose structure is shown in Fig. 1. In the V-groove, the between angle the (111) crystal plane and the (100) crystal plane is 54.74° . The different atomic arrangements lead to silicon anisotropy or different etching rates, so the etching rate of the (111) crystal plane is far less than that of the (100) crystal plane^[2]. Therefore, we can make the Si V-groove as long as we choose the proper etching temperature and etching time. The (100) n-type silicon chips were used in the fabrication of the V-groove. We also used the thermal oxidation method to form a layer of 200-nm-thick SiO_2 films grown on two surfaces. Then we used the vacuum thermal evaporation technique to deposit a layer of 500-nm-thick MgO films on the surface to be etched. After lithography, corrosion, and ICP plasma etching, the sample formed an exposed silicon structure of the SiO_2 film. Finally, the silicon chip was eroded by KOH solution to obtain a Si V-groove structure under the protection of the SiO_2 film.

The SiO₂ thin-film growth process was carried out in Northwest Polytechnical University. Moreover, lithography, ICP etching, and chemical etching were conducted in the Micro-optoelectrical System Laboratory of Xi'an Technological University. The ICP-98A high-density plasma etching machine and the JKG-2-based lithography machine were employed in the experiments.

In the traditional process of silicon wet etching, the photoresist is used as the protective layer of the SiO₂ film while etching. Considering the high density of SiO₂, its etching time is so long that the photoresist wrinkles and floats. This affects the quality and the finished product rate of the V-grooves. Therefore, we used the ICP plasma etching method which involves the deposit of a layer of the MgO film on the SiO₂ film using the vacuum thermal evaporation technique. The photoresist became the masking layer of the MgO film. The etching time of the MgO film is short, so the photoresist corrosion resistance is sufficient. After the etching of the MgO film, we used



Fig. 1. V-groove geometry for (100) silicon oriental.



Fig. 2. Schematic diagram of photolithography.



Fig. 3. Bird's eye view of the Si V-groove structures with different structural sizes over (100) silicon wafer.



Fig. 4. Cross section's contour of a V-groove.

ICP etching to etch SiO_2 film with MgO as the protective layer. The gas of ICP plasma etching is SF_6 . In the process of glow discharge, the fluoride ions released from SF_6 reacted with SiO_2 , producing SiF_4 and O_2 . The chemical reaction formula is^[3]

$$4\mathbf{F}^- + \mathrm{SiO}_2 = \mathrm{SiF}_4 \uparrow + \mathrm{O}_2. \tag{1}$$

The fluoride ions are difficult to react with MgO, so the SiO₂ etching rate is far greater than the MgO etching rate. The HCl solution was used to remove MgO, and the residual MgO on the SiO₂ layer was completely corroded away. Figure 2 shows the details. The silicon chip was put into the mixed silicon etching solution, with the SiO₂ layer having been localized and etched. According to Refs. [4], [5], the formula for the silicon etching solution is KOH: IPA: $H_2O =$ 23.4%:13.3%:63.3%(mass percentage). The etching rates to the (100) crystal plane, the (111) crystal plane, and the SiO₂ film are 1, 0.7, and 0.0028 µm/min, respectively.

$$\text{KOH} + \text{H}_2\text{O} \to \text{K}^+ + 2\text{OH}^- + 2\text{H}^+,$$
 (2)

$$\mathrm{Si} + 2\mathrm{OH}^{-} + 4\mathrm{H}_{2}\mathrm{O} \to \mathrm{Si}(\mathrm{OH})_{6}^{-2} + 2\mathrm{H}_{2}\uparrow, \qquad (3)$$

$$Si(OH)_6^{-2} + (CH_3)_2CHOH \rightarrow$$

 $[Si(OC_3H_7)_6]^{-2} + 6H_2O.$ (4)

From the above chemical equations, we can see that the KOH solution oxidizes silicon into the silicon compounds containing water. Then it reacts with IPA, producing soluble complex compounds that constantly leave the silicon surface. The water is an activator, supplying OH⁻ ions for the chemical reaction.

The etching rate of the KOH solution increases as the temperature increases. However, the temperature should not be higher than 82 °C since the boiling point of organism I is 82.5 °C. Owing to the anisotropy of silicon etching, we made the V-grooves have different sizes after a certain etching time. In addition, to avoid the small islands at the bottom of the V-grooves, we used ultrasound to clean the silicon chip for 3-5 min.

Figures 3 and 4 show the experimental results. We can see that the shape of the V-grooves is whole and that their edges are straight and clear. Moreover, their symmetry is good.

In conclusion, the experimental results show that for the patterning process of the SiO_2 film in silicon anisotropic wet etching, ICP etching is much simpler than the traditional method. Correspondingly, the quality of the Si V-groove is good, and the finished product rate is high. That is a new method to achieve Si anisotropic wet etching is found.

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