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Effects of ultrasonic agitation and surfactant additive on surface roughness of Si (1 1 1) crystal plane in alkaline KOH solution

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ABSTRACT

In the silicon wet etching process, the "pseudo-mask" formed by the hydrogen bubbles generated during the etching process is the reason causing high surface roughness and poor surface quality. Based upon the ultrasonic mechanical effect and wettability enhanced by isopropyl alcohol (IPA), ultrasonic agitation and IPA were used to improve surface quality of Si (1 1 1) crystal plane during silicon wet etching process. The surface roughness R_q is smaller than 15 nm when using ultrasonic agitation and R_q is smaller than 7 nm when using IPA. When the range of IPA concentration (mass fraction, wt%) is 5–20%, the ultrasonic frequency is 100 kHz and the ultrasound intensity is 30–50 W/L, the surface roughness R_q is smaller than 2 nm when combining ultrasonic agitation and IPA. The surface roughness R_q is equal to 1 nm when the mass fraction of IPA, ultrasound intensity and the ultrasonic frequency is 20%, 50 W and 100 kHz respectively. The experimental results indicated that the combination of ultrasonic agitation and IPA could obtain a lower surface roughness of Si (1 1 1) crystal plane in silicon wet etching process.

1. Introduction

Anisotropic etching of silicon in alkaline solutions is widely used for bulk micromachining in micro-electro-mechanical systems (MEMS) [1]. Anisotropic wet etching of single crystal silicon, which depends on the differential etching rate of its crystallographic planes, has been used to fabricate diverse MEMS devices. Of all the anisotropic etchants, the inorganic KOH (potassium hydroxide) is the most popularly used because of its easily preparation and smaller toxicity [2]. In the process of the chemical reaction between the silicon and KOH solution, the hydrogen bubbles generated during the etching process logged on the etched surface to form the 'pseudo-mask' phenomenon [3–5], which will obstruct the chemical reaction between the etchant and the silicon atoms at the surface and lower the etching rate while increasing surface roughness, even causing hillocks to form.

In the semiconductor industry, the magnetics stirring is a common method to reduce the surface roughness of the silicon devices during the monocrystalline silicon wet etching process [6]. However, the limitation of this method lies in the fact that the solution will be layered and the distribution of temperature is uneven, which is difficult to realize the accurate control of microstructure size and uniform distribution of roughness [7]. Recent studies show that the ultrasonic agitation could improve the etching rate and smoothness of the Si (1 0 0) and (1 1 0) crystal plane [8–10]. On the other hand, increasing the wettability on the surface of the silicon wafer through using different kinds of surfactant additives also could reduce the surface roughness of the silicon (1 0 0) and (1 1 0) crystal plane [11–18]. IPA is one of the most commonly used surfactant additives, which could improve the surface quality of silicon (1 0 0) crystal plane and reduce the lateral erosion of convex corner [19,20].

The literatures mentioned above are all focused on the studies of the relationship among the surface roughness of the Si (1 0 0) and (1 1 0) crystal plane and ultrasonic agitation and surfactant additives, and literatures studying the effects of ultrasonic agitation and surfactant additive on surface roughness of Si (1 1 1) crystal plane in alkaline KOH solution have not been reported. In the present paper, we discuss the relationship between the surface roughness of Si (1 1 1) crystal plane and ultrasonic agitation, IPA and combination ultrasonic agitation with IPA respectively. The results have shown that the methods mentioned above are all the effective ways to reduce the surface roughness of Si (1 1 1) crystal plane and the combination ultrasonic agitation with IPA could achieve more smoother, "mirror-like" surface on the whole wafer.

2. Experimental procedure

The silicon wafer used in our experiment is high-purity float zone silicon boules up to 76.2 mm in diameter and 1 mm in thickness with resistivity of approximately 2000 Ω cm, and the supplier

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of mono-Si is China Electronics Technology Group Corporation No. 46 Research Institute. The surface of silicon wafer will be affected by contamination of organic impurities and metal ions after different process. Based upon this, SC-1 liquid (H_2O:H_2O_2:NH_4OH = 5:1:1) and SC-2 liquid (H_2O:H_2O_2:HCl = 6:1:1) are used to clean organic impurities and metal ions respectively. A SiO_2 layer with thickness of 70 nm is deposed on silicon wafer using thermal growth. The wafer is cleaned in accordance with the order of the toluene, acetone, alcohol and water before mask fabrication.

Method used to coat photoresist is spin coating, and spin speed, coating time and photoresist thickness is 3000 rpm, 30 s and 300 nm respectively. The silicon wafer is harden into the oven after coating with hardening temperature of 120 °C and hardening time of 20 min. It is so important to align mask to crystal orientation because of crystallographic orientation dependence of silicon wet etching. If not be aligned well, the groove will be very rough and not completely collimating. The alignment of mask and crystal orientation can be thought of completely well when the dislocation between mask and crystal orientation is smaller than 0.01° [21]. The lithography is undergoing with the help of ultraviolet exposure machine whose model is KARLSUSSMA6/BA6 and the precise alignment of mask and crystal orientation can be realized due to its alignment accuracy of ±1 µm, which can solve the problem of big surface roughness of crystal plane. We then develop the photoresist, immersing the entire wafer for 8 min in a 25 °C developer solution that we stir constantly to insure uniform concentration over the entire wafer surface. The developer removes the UV-exposed photoresist, leaving a pattern of photoresist lines on the wafer that is a 1:1 positive image of the contact mask pattern. The SiO_2 mask is formed on silicon substrate by exposure and development of photoresist and etching on SiO_2 layer using BHF and RIE methods [22].

The samples were etched in an ultrasonic bath. The temperature was 60 °C and was controlled with an accuracy of ±0.2 °C. The mass fraction of KOH solution is 10%. In order to keep solution concentration uniform, the condenser pipe is added during the wet etching process. Ultrasonic with different frequency and power and IPA with different concentration were introduced in the experiment process. For the convenience of discussion, a parameter named ultrasound intensity is defined, which is the ratio of ultrasound intensity divided by volume of the liquid. The range of ultrasonic frequency and ultrasound intensity is 40–100 kHz and 10–50 W/L respectively and the concentrations of IPA are 5%, 10%, 15% and 20%. The morphology, roughness and etching rate of etched surfaces of Si (1 1 1) crystal plane were inspected with the atomic force microscopy (AFM) whose model is "Dimension Icon" made by the United States Bruker company (Z sensor noise level: 35 pm RMS typical imaging bandwidth (up to 625 Hz); 50 pm RMS, force curve bandwidth (0.1 –5 kHz)). The size of the tip that was used in the AFM experiments is "RTESP" (MPP-11100-10, 40 N/m, 300 kHz, Rotated Tip, No coating) and the mode which AFM operated in is "Tapping mode". Fig. 1 is the sketch showing experimental equipment in silicon wet etching process.

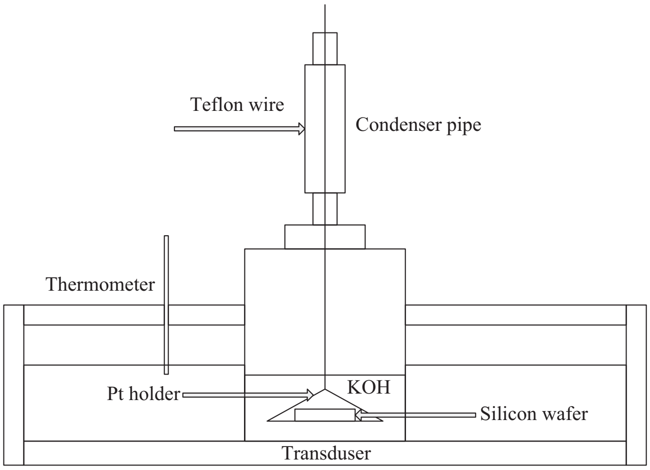

Fig. 1. The sketch showing experimental equipment in silicon wet etching process.

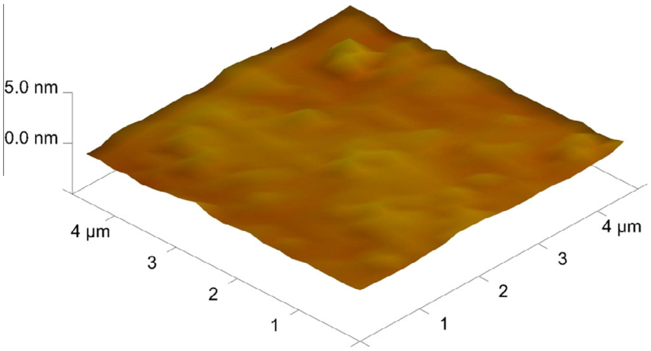

Fig. 2. AFM image of Si (1 1 1) surface etched in KOH with ultrasonic agitation.

Table 1
Measurement results of surface roughness and etching rate of Si (1 1 1) crystal plane under conditions with ultrasonic agitation and without ultrasonic agitation.

Ultrasonic frequency (kHz)	Surface roughness on R_q (nm)	Etching rate on (1 1 1) plane (nm/s)
40	13.41 ± 0.02	0.033
60	11.54 ± 0.05	0.036
80	9.57 ± 0.05	0.044
100	7.34 ± 0.03	0.048
0	40.28 ± 0.04	0.025

3. Result and discussion

3.1. The effect of ultrasonic frequency on surface roughness of Si (1 1 1) crystal plane

The AFM image of Si (1 1 1) surface etched in KOH with ultrasonic agitation is shown in Fig. 2. The surface roughness of Si (1 1 1) wafers under conditions with ultrasonic agitation and without ultrasonic agitation was measured by using surface profiler, where the ultrasound intensity is equal to 50 W/L, the measurement range is 5 µm × 5 µm and the ultrasonic frequency is 40 kHz, 60 kHz, 80 kHz, and 100 kHz respectively. It can be seen from the measured results shown in Table 1 that the surface roughness of Si (1 1 1) crystal plane decreases with the increasing of ultrasonic frequency and the surface roughness of Si (1 1 1) wafers with different ultrasonic frequencies are all less than that without ultrasonic agitation. The etching rate of Si (1 1 1) crystal plane is shown in Table 1, it can be seen that the etching rate increases with the increasing of ultrasonic frequency, which means the ultrasonic enhancement on etching rate increases with the increasing of ultrasonic frequency when ultrasound intensity is fixed. During the reaction process between silicon wafer and KOH solution, the hydrogen bubbles generated will log on the etched surface to form the 'pseudo-mask' phenomenon, which will obstruct the chemical reaction between the etchant and the silicon atoms at the surface and lower the etching rate while increasing surface roughness of silicon wafer. The aim of introducing ultrasound is to shock the hydrogen bubbles on the basis of the mechanical effects of it, which will decrease the duration for which the hydrogen bubbles are attached to the etched surface, and then