Electrochemical Etch-Stop Suitable for MEMS Applications

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This paper presents the electrochemical etch-stop characteristics of single-crystal Si(001) wafers in tetramethyl ammonium hydroxide(TMAH):isopropyl alcohol(IPA):pyrazine solutions. The addition of pyrazine to TMAH:IPA solutions increased the etch rate of (100) Si, thus the etching time required by the etch-stop process shortened. The current-voltage(I-V) characteristics of n- and p-type Si in TMAH:IPA:pyrazine solutions were obtained, respectively. Open circuit potential(OCP) and passivation potential(PP) of n- and p-type Si, respectively, were obtained and applied potential was selected between n- and p-type Si PPs. The electrochemical etch-stop method was used to fabricate 801 microdiaphragms of 20 ㎛ thickness on a 5-inch Si wafer. The average thickness of fabricated 801 microdiaphragms on one Si wafer was 20.03 ㎛ and the standard deviation was ±0.26 ㎛. The Si surface of the etch-stopped microdiaphragm was extremely flat with no noticeable taper or nonuniformity.

Keywords : electrochemical etch-stop, MEMS, OCP, PP, microdiaphragm

1. INTRODUCTION

There has been increasing interest in the development of microelectromechanical system(MEMS) using Si micromachining technology. Because of its superior electrical and mechanical properties[1], single-crystal Si has been used to various MEMS. Bulk micromachining technology is a very important technique and making three-dimensional microstructures by anisotropic wet etching of single-crystal Si is of even greater importance. For example, the sensitivities of piezoresistive and capacitive pressure sensors, respectively, are inversely proportional to geometric factors such as thickness and area of diaphragms formed by anisotropic wet etching[2, 3]. In particular, in the case of existing a significant irregularity or nonuniformity on the etched micro-diaphragm surface, the stress distribution in the microdiaphragm will be disturbed. This causes significant variations in the sensitivity, the offset and the dynamic range of the resulting devices[4]. Therefore, accurate control of microdiaphragm thickness with a uniformly etched surface is very important for using micromachined Si structures as sensing or active elements.

Anisotropic etchants that are frequently used for single-crystal Si are KOH, NaOH, ethylenediamine pyrocatechol water(EDP), hydrazine water and tetramethyl ammonium hydroxide(TMAH). EDP and hydrazine water are toxic and unstable. Consequently, they are not easy to handle. KOH and NaOH have excellent anisotropic etching properties, but the use of KOH is usually restricted to postprocessing treatment. In terms of process compatibility, the etchant must be compatible with the CMOS manufacturing process. Since TMAH contains no alkaline ion impurity, it can be used in IC processing. TMAH is similar to KOH in terms of anisotropic etching characteristics and low toxicity[5-7]. TMAH is also used in the removal of positive photoresistors. However, roughly etched surfaces at low concentration and serious undercutting at high concentration are the drawbacks. To overcome these disadvantages, investigations on TMAH:isopropyl alcohol(IPA) solutions were conducted[8]. Though the addition of IPA has improved the smoothness of the etched surface and reduced the undercutting, it has reduced the etch rate of TMAH. On the other hand, when pyrazine ($C_3H_4N_2$) was added to TMAH:IPA solutions, we found, for the first time, that the etch rate of single-crystal Si is enhanced, keeping desirable flatness of the etched surface, also, compensation of undercutting are improved simultaneously[9].

As stated above, the accurate control of microdiaphragm thickness using anisotropic wet etchants is very important. Widely used methods to control the microdiaphragm for achieving the desired thickness are the etched-time stop, the boron etch-stop[10], the Si-on-insulator(SOI) substrate[11] and the electrochemical etch-stop methods[12]. One disadvantage of the etched-time stop method is that the variation of etch rate being occurred with certain etchants causes to a large percentage of thickness error in the etched Si region of the diaphragm or the beam. One disadvantage of the boron etch-stop method is that heavily Boron-doped single-crystal Si introduces compressive stress into other structures, also, it is not compatible with circuit processing techniques. The SOI substrate method shows good etch-stop characteristics, but is not practical because of the price of SOI wafers. Therefore, we focus on the electrochemical etch-stop method, which is based on the anodic passivation characteristics of Si with a reverse-bias pn junction, to provide large etching selectivity for p-type Si over n-type Si in an anisotropic wet etchant[13]. It has the advantages that it can easily control impurity concentration and the thickness of epitaxial layers.

In this paper, we describe the electrochemical etch-stop characteristics of single-crystal Si in TMAH :IPA:pyrazine solutions suitable for MEMS applications. The electrochemical etch-stop method is used for the fabrication of microdiaphragms at reverse-biased pn junctions. The thickness reproducibility of 801 microdiaphragms fabricated on a 5-inch Si wafer and the surface smoothness of the etch-stopped microdiaphragms are presented.

2. EXPERIMENTAL

2.1. Samples

The starting materials consisted of 550-㎛-thick, <100>-oriented 5-inch p- and n-type Si wafers. The electrical resistivities were 13~18 Ωcm and 4~6 Ωcm, respectively. Since the thermal oxide etch rate of TMAH is very low, for all samples a 4000-Å-thick thermal oxide was used as masking material.

Depending upon the experiment, two groups of Si wafer samples were prepared:

(a) For current-voltage(I-V) measurements, one side contact metallized n- and p-type Si wafers were used. Eight hundered and one rectangular openings of 1.5×1.5 mm^2 were made on the other side of the wafer.

(b) For etching diaphragms, we used wafers that had 20-㎛-thick n-type Si grown epitaxially on p-type substrate. In the n-type Si layer, we implanted boron to form a contact, and 801 diaphragm patterns were also made on the other side of the wafer.

The optimum anisotropic etch condition is TMAH(20 wt.%):IPA(8.5 vol.%):pyrazine(0.5 g/100 ml) solutions. Under this condition, the etch rate is higher than that in TMAH:IPA solutions and the surface quality is excellent[9].

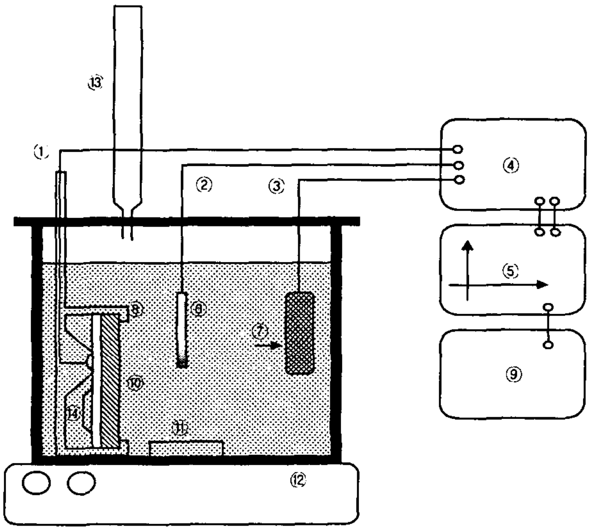
2.2. Etching set up

Electrochemical etching was controlled using an EG&G 362 potentiostat. An Ag/AgCl-type electrode, whose working temperature extends up to 100℃, was used as the reference electrode and a Pt mesh, which supports high currents, was used as the counter electrode. All voltages presented in the paper refer to that of the Ag/AgCl electrode.

Etching was performed in the dark using a tank equipped with a reflux condenser, into which nitrogen was bubbled. A temperature of 80℃ was chosen for all experiments, and was controlled during the process to within ±1℃.

The experimental setup is shown in Fig. 1. The I-V curves were obtained using a three-electrode system with the voltage sweep rate set at 2 mV/s. The holder that prevented leaking out of the solution to the back of the wafer was made of Teflon and O-ring.

The etch-stop voltage was selected between n- and p-type Si passivation potentials(PPs), and the electrochemical etch-stop characteristics of a 5-inch Si wafer were studied. The thickness variation of microdiaphragms fabricated by the etch-stop method and the sufrace roughness of etch-stopped microdiaphragms were evaluated using scanning electron microscopy(SEM) and atomic force microscopey(AFM), respectively.



1. WE
2. RE
3. CE
4. Potentiostat
5. Plotter
6. Ag/AgCl
7. Pt mesh
8. Teflon holder
9. PC
10. Sample
11. Magnetic stir-bar
12. Hot plate
13. Reflux condenser

Fig. 1. The configuration for electrochemical etch-stop.

3. RESULTS AND DISCUSSION

3.1. I-V characteristics

By conventional I-V measurements, open circuit potential(OCP) and PP were determined in a three-electrode configuration. The sweep rate was 2 mV/s and the range was from -2 V to 0 V.

Figure 2 shows the I-V characteristics of n- and p-type Si obtained in TMAH:IPA:pyrazine solutions, respectively. The etchant is TMAH(20 wt.%):IPA(8.5 vol.%):pyrazine(0.5 g/100 ml) solution at 80℃. The addition of pyrazine to TMAH:IPA solutions shifted both OCP and PP of Si toward the positive direction.

OCPs for n-type and p-type Si were measured to be -1.4 and -1.1 V, respectively, PPs were measured to be -0.9 and - 0.74 V, respectively. Thus, we have selected the potential, in which selective etching is realized. Since this potential is formed between the two PPs, one must choose the potential from values between - 0.9 and - 0.74 V.

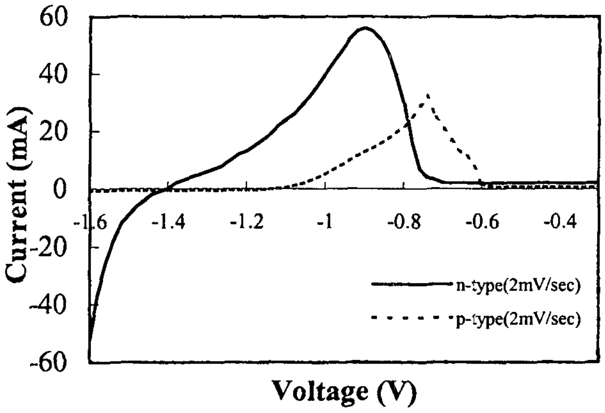


Fig. 2. I-V chacteristics of n- and p-type Si obtained in TMAH:IPA:pyrazine solutions, respectively.

3.2. T-I characteristic

When etching reached the space-charge layer of the n-region, a typical current peak appeared to indicate the end of the process. After the peak appeared, the wafers were overetched for 10 min.

Figure 3 shows the typical time-current(T-I) characteristic for a 5-inch Si wafer with a 20-㎛-thick epitaxial layer in TMAH:IPA:pyrazine solutions during the electrochemical etch-stop process. The increase in the etch rate due to the addition of pyrazine reduced the time required for the etch-stop process[14]. The current peak indicates increasing current with decreasing thickness of the diaphragms. Thus, the leakage current under reverse bias was increased. The n-type Si that was exposed to the etchant when all p-type Si was etched off, behaved as a resistor only. Therefore, large current flow occurred. This current induced the anodic oxidation of the surface of n-type Si[15]. Hydroxide ions in the etchant react with the silicon surface to form silicon dioxide(SiO$_2$). Since the etch rate of SiO$_2$ in TMAH solution is very low, etching does not proceed further and finally stops. Moreover, because SiO$_2$ is a good insulator, the current decreases to zero.

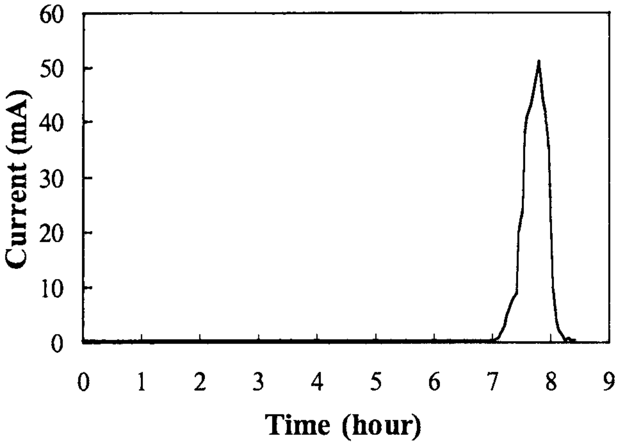


Fig. 3. I-t characteristic for a 5-inch Si wafer in TMAH: IPA:pyrazine solutions during the elelctro-chemical etch-stop process.

3.3. Flatness of etched surface

Surface smoothness of the etch-stopped microdiaphragms is one of the main requirements for the fabrication of high-quality micromachining devices.

Figure 4 shows a cross-sectional SEM image of a cleaved Si microdiaphragm fabricated by the electrochemical etch-stop method in TMAH(20 wt.%):IPA(8.5 vol.%):pyrazine(0.5 g/100 ml) solutions at 80℃. Etching was stopped precisely at a pn junction and a microdiaphragm of approximately 20 ㎛ thickness was fabricated. In spite of the unpolished initial back of the Si wafer, the etch-stopped microdiaphragm surface was extremely flat and had no noticeable taper or nonuniformity. The etched-stopped surface smoothness in TMAH(20 wt.%), TMAH(20 wt.%):IPA(8.5 vol.%) and TMAH(20 wt.%):IPA(8.5 vol.%):pyrazine(0.5 g/100 ml) solutions, respectively, was 13.55, 5.48 and 5.42 nm. In spite of the decrease in etch rate, the addition of IPA to TMAH solution improved the etch-stopped surface flatness[8] On the other hand, the addition of pyrazine to TMAH:IPA solutions increased the etch rate and improved the etch-stopped surface simulateously[9].

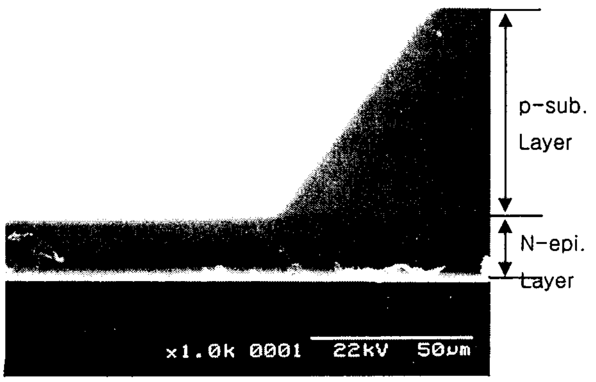


Fig. 4. Cross-sectional SEM image of an etch stopped microdiaphragm.

Figure 5 (a) and (b) show an AFM images of the mirror surface of Si wafer the etch-stopped Si surface in which the microdiaphragm from the unpolished surface was etched in TMAH(20 wt.%):IPA(8.5 vol.%):pyrazine (0.5 g/100 ml) solutions, respectively. The degree of surface roughness of non-mirror Si backside surface

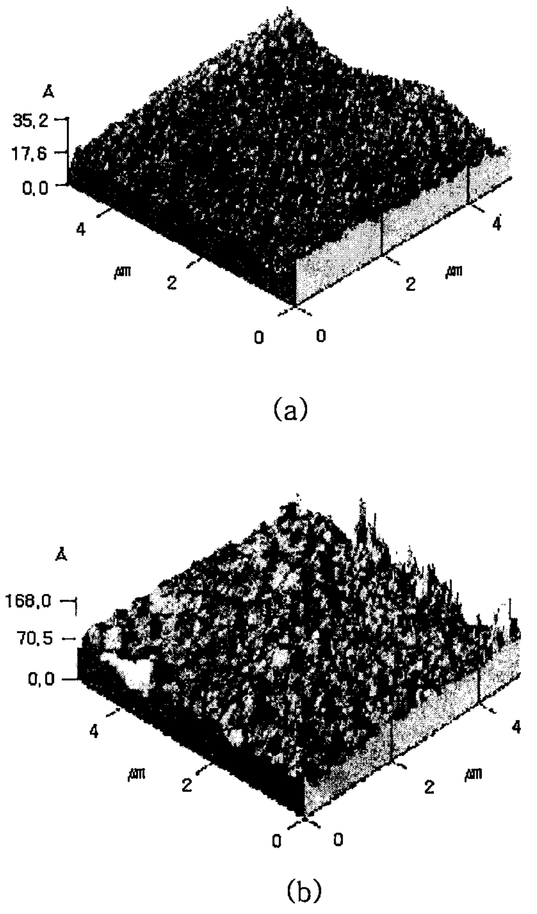


Fig. 5. AFM images of (a) the mirror surface of Si wafer and (b) the etch-stopped Si surface and electrochemical etch-stopped microdiaphragm surface in TMAH:IPA:pyrazine solutions.

Having approximately 5 μm significantly smoothen to be bout 5.42 nm via the etch-stop process. The remarkably enhanced flatness of the etched silicon surface and the accurate electrochemical etch-stopped characteristics in TMAH:IPA:pyrazine solutions are significant improve-ments over these of the conventional method of etching monitoring and control technique[16]. Therefore, the etch-stop approach using the electrochemical etch-stop method its capability of obtaining a very flat and highly uniform microdiaphragm surface[17].

3.4. Diaphragm thickness distribution

For mass production, all devices should have identical mechanical properties. Hence, the reproducibility of microdiaphragm thickness is an important fabrication feature. In order to evaluate the reproducibility of microdiaphragm thickness across a wafer, 801 microdiaphragms were fabricated, equally spaced over a 5-inch Si wafer with 20 μm-thick n-type Si grown epitaxially on a p-type substrate. After the electrochemical etch-stop process in TMAH:IPA: pyrazine solutions at a reverse bias of 0.8 V, the thickness of the microdiaphragms was measured by microscopy.

Figure 6 shows a histogram of the thickness variation obtained of microdiaphragms on one Si wafer. Microdiaphragms with a thickness of 20±0.2 μm comprise 77.9 % of the total number of microdiaphragms on one wafer. The average thickness of 801 microdiaphragms is 20.03 μm, and the standard deviation is only ± 0.26 μm. This result is markedly improved from that of previous microdiaphragms fabrication method[18]. In this work, the thickness of microdiaphragms in the central part of the silicon wafer was slightly less than 20 μm, as a result of electrical contacts. Because of the attachment of four point electrodes on each of the corners of the Si wafer, the central part of the Si wafer is not passivated to sufficiently n-type Si at the pn junction. The thickness of microdiaphragms at the edge of the Si wafer was greate than 20 μm because of the etching holder. Since hydrogen bubbles that were generated during etching were not eliminated completely, they gathered around the holder. If an additional point electrode was added and the structure of the etching holder was changed, these problems would be solved. Moreover, this thickness distribution reflects the thickness variation of the epitaxial Si layer in the wafers used in these experiments, which is about 0.2 μm. Between different wafers, the thickness of the epitaxial layer can vary by more than 0.5 μm, yielding a large microdiaphragm thickness variation. Therefore, the reproducibility of the electrochemical etch-stop method is shown to be limited only by the reproducibility of the epitaxial layer growth process.

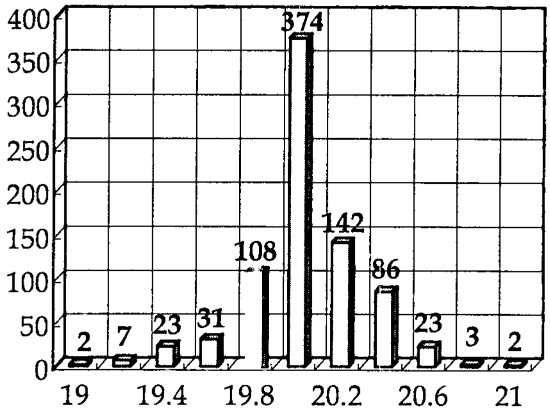


Fig. 6. Thickness distribution of 801 micro-diaphragms fabricated on a 5-inch Si wafer using the electrochemical etch-stop process in TMAH: IPA:pyrazine solutions.

4. CONCLUSIONS

The electrochemical etch-stop characteristics of single-crystal Si in TMAH(20 wt.%):IPA(8.5 vol.%):pyrazine(0.5 g/100 ml) solutions at 80℃ are presented. The I-V curves, OCP and PP were also obtained for n- and p-type Si. Selective etching of p- and n-type Si is possible by electrochemical etching at -0.8 V. This potential is between n- and p-type Si PPs. The T-I curve indicates the etch-stop point. The increase in etch rate by the addition of pyrazine reduces the time required for the etch-stop process. The thickness variation of 801 microdiaphragms fabricated on a 5-inch Si wafer by the etch-stop method and the sufrace roughness of etch-stopped microdiaphragms are evaluated using SEM and AFM, respectively. The average thickness of microdiaphragms is 20.03 μm and the standard deviation is ±0.26 μm. The etch-stopped microdiaphragm surface is extremely flat without any noticeable taper or nonuniformity. These results are satisfactory for fabricating high-yield microdiaphragms for MEMS applications.

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