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Fabrication of Silicon Menbranes for MEMS Applications

MEMS용 실리콘 멤브레인의 제작

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Abstract

This paper presents the electrochemical etch-stop characteristics of single-crystal silicon in a tetramethyl ammonium hydroxide(TMAH):isopropyl alcohol(IPA):pyrazine solution. Addition of pyrazine to a TMAH:IPA etchant increases the etch-rate of (100) silicon, thus the elapsed time for etch-stop was shortened. The current-voltage(I-V) characteristics of n- and p-type silicon in a TMAH:IPA:pyrazine solution were obtained, respectively. Open circuit potential(OCP) and passivation potential(PP) of n- and p-type silicon, respectively, were obtained and applied potential was selected between n- and p-type silicon PP. The electrochemical etch-stop is applied to the fabrication of 801 microdiaphragms having 20 ㎛ thickness on a 5-inch silicon wafer. The averge thicknesses of 801 microdiaphragms fabricated on the one wafer were 20.03 ㎛ and standard deviation was ±0.26㎛. The silicon surface of the etch-stopped microdiaphragm was extremely flat without noticeable taper or other nonuniformities. The benefits of the electrochemical etch-stop in a TMAH:IPA:pyrazine solution become apparent when reproducibility in the microdiaphragm thickness for mass production is considered. These results indicate that the electrochemical etch-stop in a TMAH:IPA:pyrazine solution provides a powerful and versatile alternative process for fabricating high-yield silicon microdiaphragms.

key words: *electrochemical etch-stop, tetramethyl ammonium hydroxide, isopropyl alcohol, pyrazine, silicon, open circuit potential, passivation potential, microdiaphragm*

1. Introduction

There have been increasing interests, recently, in the development of MEMS(microelectro mechanical systems) using silicon micromachining technology. Since single-crystal silicon has superior electrical and mechanical properties,(1) silicon is applied various MEMS applications. The bulk micromachining technology is a very important technique and making three-dimensional microstructures in the use of the anisotropic wet etching of single-crystal silicon is even more important. For example, sensitivities of piezo-resistive and capacitive pressure sensors, respectively, are inversely proportional to the square and the cubic of the thickness of diaphragm formed by anisotropic wet etching.(2, 3) In the case of microdiaphragms, especially, if there exists a significant irregularity or non-uniformity in the etched microdiaphragm surface, the stress distribution over the microdiaphragm will be disturbed. These cause significant variations in the sensitivity, the offset, and the dynamic range of the resulting devices.(4) Therefore, accurate control of the microdiaphragm thickness with a uniform etched surface is very important to use micromachined silicon structures as sensing or active elements.

Anisotropic etchants, frequently used for single-crystal silicon are KOH, NaOH, ethylenediamine pyrocatechol water(EDP), hydrazine-water and tetramethyl ammonium hydroxide (TMAH). EDP and hydrazine-water are toxic and unstable. Conseqently, they are not easy to handle. KOH and NaOH have excellent anisotropic etching properties, but the use of KOH is usually restricted to postprocessing. For the process compatibility consideration, the

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etchant must be compatible with the CMOS manufacturing process. Since TMAH has no alkaline ion contamination, it can be used in IC processing. The anisotropic etching characteristics of TMAH are similar to those of KOH in etching characteristics and low toxicity.(5-7) TMAH is also used as the remover. of positive photoresistor. However, the rough etched surfaces at low concentration and serious undercuttings at high concentration are the drawbacks. To overcome these disadvantages, investigations on TMAH: isopropyl alcohol(IPA) solutions had been launched.(8) Though addition of IPA has improved the smoothness of the surface and the undercuttings, it has reduced the etch rate of TMAH. On the other hand, when addded pyrazine($C_3H_4N_2$) to a TMAH:IPA solution, we kown, for the first time, that the etch rate of single-crystal silicon is enhanced and flatness of etched-surface and compensation of undercutting are improved simultaneously.(9)

As stated above, the accurate control of a microdiaphragm thickness using anisotropic wet etchants is very important. Widely used methods to controll the desired microdiaphragm thickness are the etched-time stop, the boron etch-stop,(10) the SOI(silicon-on-insulator) substrate(11) and the electrochemical etch-stop.(12) A disadvantage of the etched-time stop is that the variations of etch-rate which can occur with certain etchants and variations of silicon thickness can lead to a thickness error which may be a large fraction of the desired diaphragm or beam thickness. A disadvantage of the boron etch-stop is that single-crystal silicon containing a heavy boron diffusion introduces compressive stress into the structures and are not compatible with the circuit processing techniques. Using the SOI structure method shows good etch-stop characteristics, but is not suitable because of high price of SOI wafers. Therefore, we have turned to the electrochemical etch-stop technique based on anodic passivation characteristics of silicon with a reverse-bias pn junction to provides a large etching selectivity of p-type silicon over n-type in anistotropic wet etchant, which has the advantage that it can easily control the impurity concentration and the thickness of epitxial layers.

This paper describes the electrochemical etch-stop characteristics of single-crystal silicon in a TMAH:IPA:pyrazine solution. Then, the etch-stop at reverse-biased pn junctions was used for the fabrication of microdiaphragms. The reproducibility of 810 microdiaphragm thickness fabricated on a 5-inch silicon wafer and the surface smoothness of the etche-stopped microdiaphragms are also presented.

2. Experimental

2.1 Samples

The starting material consisted of 550 ㎛ thick, <100>-oriented 5-inch p- and n-type wafers, respectively. 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 4000Å thermal oxide was used as masking material.

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

(a) For current-voltage(I-V) measurements, one side contact metallized n- and p-type wafers were used. 801 rectangular openings of 1.5 ㎜ × 1.5 ㎜ were made in the other side of the wafer.

(b) For etching diaphragms, we used wafers which have 20 ㎛ thick n-type silicon epitaxial on p-type substrate. In n-type silicon layer we implanted boron to make a contact, and the other side of the wafer 801 diaphragm patterns were also made.

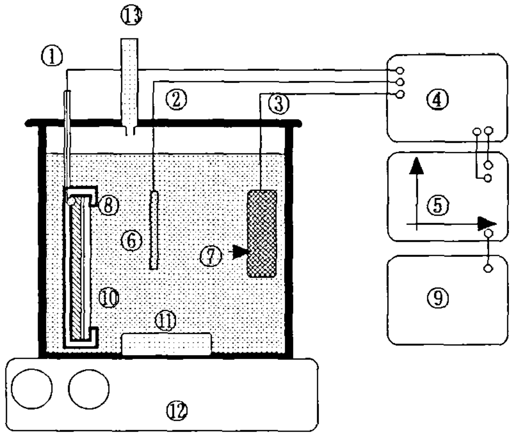
Optimum anisotropic etch condition is TMAH(20 *wt.%*):IPA(8.5 *vol.%*):pyrazine(0.5 *g*/100 *ml*), under this condition the etch-rate is faster than that in a TMAH:IPA solution and the surface quality is excellent.(9)

2.2 Etching set-up

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

The etching processes were performed in the dark, using a tank equipped with a reflex condenser and with nitrogen bubbling. A temperature of 80℃ was chosen for all experiments, controlled during the process to withing ±1℃.

The experimental set-up can be seen in Fig. 1. The I-V curves were obtained using three-electrode system with the sweep rate of



① Working Electrode ② Reference Electrode
③ Counter Electrode ④ Potentiostat ⑤ PC
⑥ Ag/AgCl ⑦ Pt mesh ⑧ Teflon holder
⑨ Plotter ⑩ Sample ⑪ Magnetic stirrer
⑫ Hot Plate ⑬ Reflux condenser

Fig. 1. Experimental set-up for electrochemical etch-stop.

voltage set at 2 *mV/sec*. The holder which prevented leaking out of the solution to the backside of the wafer was made of Teflon and O-ring.

The etch-stop voltage was selected between n- and p-type silicon passivation potential(PP), and electrochemical etch-stop characteristics of a 5-inch silicon wafer was also studied. The thickness variation of microdiaphragms fabricated by etch-stop and the sufrace roughness of etche-stopped microdiaphragms were evaluated using SEM and AFM, respectively.

3. Results and Discussion

3.1 The I-V characteristics

With the usual I-V measurements the open circuit potential(OCP) and the PP were determined in the three-electrode configuration. The sweep rate was 2 *mV/sec* and the range was -2 *V* to 0 *V*. Fig. 2 shows I-V characteristics obtained from a TMAH:IPA:pyrazine solution. The condition of etchant is a TMAH(20 *wt.%*) :IPA(8.5 *vol.%*):pyrazine(0.5 *g/100 ml*) solution at 80℃. Addition of pyrazine to a TMAH:IPA solution made the OCP and PP of silicon to move in the positive direction.

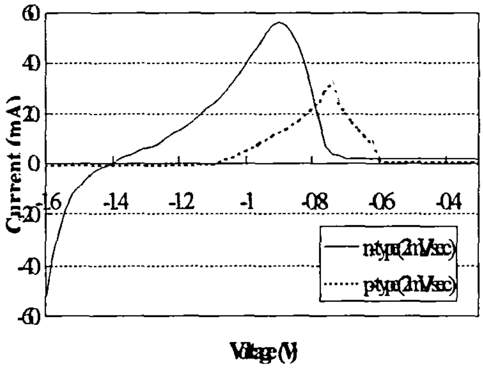


Fig. 2. Current-voltage characteristics of n- and p-type silicon obtained in a TMAH:IPA:pyrazine solution.

OCP's for n-type and p-type silicon have been measured to be -1.4 *V* and -1.1 *V*, respectively, and PP have been measured to be - 0.9 *V* and - 0.74 *V*, respectively. Thus, we have selected potential, which the selective etching is enable. Since this potential is placed between two PP's, one must choose the potential between - 0.9 *V* and - 0.74 *V*.

3.2 The T-I characteristics

When the etching reaches the space-charge layer of the n-region, the typical current peak appears, indicating the end of the process. After the peak the wafers were overetched for 10 min. Fig. 3 shows typical time-current(I-T) charac-teristics obtained on a 5-inch silicon wafer having 20 ㎛ thickness epitaxial layer in a TMAH:IPA:pyrazine solution during the electrochemical etch-stop. The boost of the etch-rate

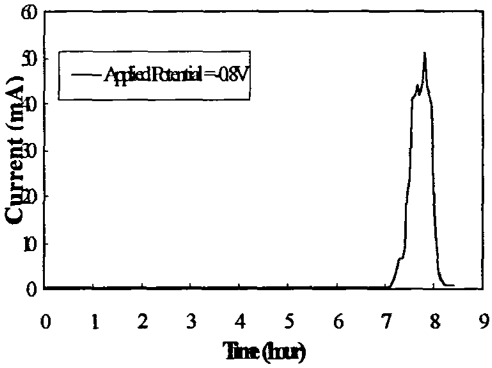


Fig. 3. Time-current characteristics obtained on a 5-inch silicon wafer in a TMAH:IPA:pyrazine solutions during elelctrochemicaa etch-stop.

due to addition of pyrazine reduces the time needed for etch-stop processes.(13) The peak of curve means increased current value with thinning thickness of diaphragms. Thus, the leakage current under reverse bias has increased. The n-type silicon which expose to the etchant, when all p-type silicon was etched away, only behaved as a resistor only. Therefore, large current flow was occured. This current causes anodic oxidation phenomena on the surface of the n-type silicon.(14) Hydroxide ions in the etchant react with silicon surface and silicon dioxide(SiO_2) is formed. Since the etch-rate of SiO_2 in TMAH solution is very low, etching is not proceed any more and it finally stopped. Moreover, as SiO_2 is a good insulator, the current value would decrease to zero.

3.3 Flatness of the etched surface

Surface smoothness of the etche-stopped microdiaphragms is one of major requirements for the fabrication of high quality micromachining devices. Fig. 4 shows a cross-sectional view SEM photgraph of a cleaved silicon microdiaphragm fabricated by the electrochemical etch-stop method in a TMAH(20 *wt.%*):IPA(8.5 *vol.%*):pyrazine(0.5 *g*/100 *ml*) solution at 80℃. Etching was stopped precisely at pn junction and a micordiaphragm having the approximately 20 ㎛ thickness was fabricated. In spite of the unpolished intial backside silicon wafer, the smoothness of the etch-stopped microdiaphragm surface was extremely flat without noticeable taper or other nonuniformities. The etched-stopped suface 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 measured as 13.55, 5.48 and 5.42 nm. In spite of etch-rate decreasing, addition of IPA to a TMAH solution improved the etched-stopped surface flatness.(8) On the other hand, addition of pyrazine to a TMAH:IPA solution, the etch rate is increased and the etched-stopped surface is improved simulateously.(9) Fig. 5 shows a AFM image of the etch-stopped silicon surface which the microdiaphragm from unpolished surface was etched in a TMAH(20 *wt.%*):IPA(8.5 *vol.%*): pyrazine(0.5 *g*/100 *ml*) solution. The inital surface roughness of about 5 ㎛ was smoothed to less than 5.42 nm after etch-stop. Owing to very good flatness of etched silicon surface and accurate electrochemical etche-stopped characteristics in a TMAH:IPA:pyrazine solution, this is a sginificant improvement over conventional methods of etching monitoring and control technique.(15) Therefore, the etch-stop approach using the elelctrochemical etch-stop in a TMAH:IPA:pyrazine solution provides a great promise due to the capability of obtaining very flat and uniform microdiaphragm surface.(16)

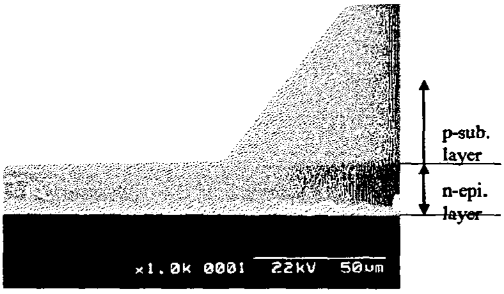


Fig. 4. A cross-sectioanl view SEM photograph of an etch-stopped micordiaphragm.

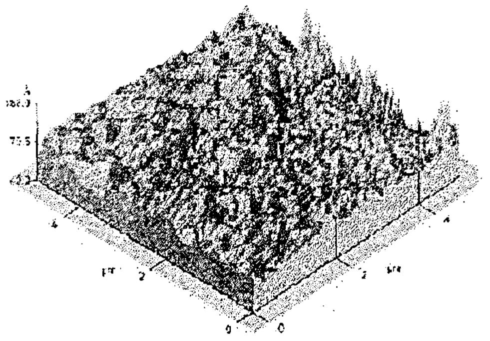


Fig. 5. AFM images of electrochemical etch-stoppoed microdiaphragm surfaces in a TMAH: IPA:pyrazine solution.

3.4 Diaphragm thickness variation

For mass production, all devices should have identical mechanical properties. Hence, the reproducibility of the microdiaphragm thickness is another important fabrication feature. In order to evaluate the microdiaphragm thickness reproducibility cross a wfer, 810 microdiaphragms were fabricated, equally spaced over a 5-inch silicon wafer with 20 ㎛ thick n-type silicon epitaxial on p-type substrate. After electrochemical etch-stop in a TMAH:IPA: pyrazine solution at a reverse diode bias of 0.8 V, the thickness of the microdiaphragms was

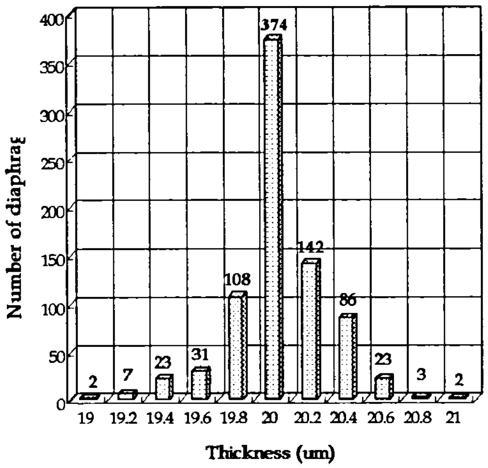


Fig. 6. Thickness variation distribution of 801 microdiaphragms fabricated on a 5-inch silicon wafer using electrochemical etch-stop in a TMAH:IPA:pyrazine solution.

measured by microscope for all of micro-diaphragms. Fig. 6 shows a histogram distribution of the thickness variation obtined for micro-diaphragms of one silicon wafer. Microdiaphragms having thickness of 20 ± 0.2 ㎛ are 77.9 % for a full wafer scale. The average thickness of 801 micordiaphragms is 20.03 ㎛, and the standard deviation is only ± 0.26 ㎛. This result is a great improvement as compared to previous fabrication methods of microdiaphragms. (17) In this work, the thickness of micro- diaphragms in central part of silicon wafer was slightly smaller than 20 ㎛, this is a result of electrical contacts. Due to attached point electrode to 4 point on silicon wafer edge area in this study, the central part of silicon wafer dose not sufficient to passivate n-type silicon at pn junction. The thickness of microdiaphragms at the edge of silicon wafer was a little thicker than 20 ㎛ because of etching holder. Since hydrogen bubbles generated during etching dose not eliminated sufficiently, hydrogen bubbles gathered around the holder. If an additional point electrode is added and the structure of the etching holder is changed. these problems will be resolved. Moreover, This distribution reflects the thickness variation of epitaxial silicon for the wafers used in these experiments, which is about 0.2 ㎛. Between different wafers, the thickness of the epitaxial layer can vary more than 0.5 ㎛, yielding a large microdiaphragm thickness variation. Therefore, the electrochemical etch-stop reproducibility is shown to be limited only by the reproducibity of the epitaxial layerr growth process.

4. Conclusion

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

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