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A STUDY ON ANISOTROPIC ETCHING OF (100) SILICON IN AQUEOUS KOH SOLUTION

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ABSTRACT

This paper reports the temperature and concentration dependence of anisotropic etching of (100) silicon in aqueous KOH solution. Etching rate of wet etching of (100) silicon in pure KOH solution has been experimentally determined with varying KOH concentration and the temperature of the KOH solution. Atomic force microscopy (AFM) and optical microscopy have been employed for the inspection of the quality of the etched surfaces. It has been observed that the etching rate increases with increasing temperature of the KOH solution for concentrations varying from 10% to 45% by weight. However, for a given KOH temperature, the etching rate decreases with increasing KOH concentration. The 10% KOH solution in contrary enhances oxidation of the silicon surfaces leading towards more hydrophilic and therefore a reduced etching rate is resulted.

Key words: MEMS, Anisotropic etching, LPCVD, KOH

INTRODUCTION

Anisotropic etching of silicon refers to the direction-dependent etching of silicon, usually by alkaline etchants like aqueous KOH, TMAH and other alkaline hydroxides like NaOH and LiOH. Due to the strong dependence of the etch rate on crystal direction and on etchant concentration, a large variety of silicon structures can be fabricated in a highly controllable and reproducible manner. Hence, anisotropic etching of (100) silicon has been a key process in common MEMS based technologies for realizing 3-D structures. These structures include V-grooves for VMOS transistors, small holes for ink jets and

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diaphragms for MEMS pressure sensors[1]. The actual reaction mechanism has not been fully understood for a long and a comprehensive physical model for the process has not yet been developed. With increasing numbers of MEMS applications, interest has grown for process modelling, simulation and software tools useful for prediction of etched surface profile.

Chemical etching of silicon depends on crystal orientation, temperature and concentration of the etchant. Geometry of the area to be etched also influences the etch rate owing to the different crystal planes encountered during the etching process. In order to minimize the influence of other chemicals on the etching mechanism and therefore, to obtain more accurate results, pure KOH solution has been preferred over a number of mixtures with moderators like ethylene diamine pyrocatechol (EDP) and isopropyl alcohol (IPA). This has resulted into increasing the temperature of the solution up to its boiling point. Anisotropic etching of silicon has been carried out at temperature near boiling point with smooth surface[2]. 6-8 times faster etching rate has been obtained with reasonable good etched surface quality. The chemical etching mechanism of silicon in KOH is still under debate and therefore, more experimental results under varying conditions are required for preparing accurate physical models. During silicon etching in KOH solution, H_2 bubbles are generated and to a large extent the quality of the etched surface depends on the rate at which these bubbles are removed from the etched silicon surface. Various techniques have been introduced for removing these bubbles during the etching process[3-6]. In the chemical process of anisotropic etching of silicon, surface of the silicon plane undergoes through the stages of hydrophobic to hydrophilic. The formation of large hydrogen gas bubbles depends on the adhesion between the KOH solution and the silicon wafer. If the surface is hydrophilic, only small bubbles are formed and roughness is reduced and if the surface is hydrophobic, large bubbles are formed and hence large hillocks are formed. Concentrated KOH solutions tend to adhere more to silicon surfaces, resulting in smaller bubbles and less roughness. These bubbles cause temporary localized etch stops equivalent to etch masks. The etching rate and the quality of the etched surface have been studied experimentally in the present work. Relevant details of the experiment and the results have been presented in the following sections.

EXPERIMENTAL

Silicon (100) samples were prepared starting with device grade chemical cleaning of the wafer. Thermal oxidation of 0.5-micrometer thickness was grown using wet-dry-wet sequence at 1100 °C, in a horizontal quartz furnace. LPCVD silicon nitride was deposited over that layer, at 780 °C for a thickness of 0.15 micrometer in a horizontal quartz furnace reactor. The combination of silicon dioxide and silicon nitride provides stable masking action in KOH solution at elevated temperatures. Square windows of 2.0 mm size were delineated in an array using photolithography. Dry etching was used to selectively remove silicon dioxide and silicon nitride. The samples were etched in KOH solution in a reflux condenser equipped with a magnetic stirrer to maintain KOH concentration as shown in Fig. 1.

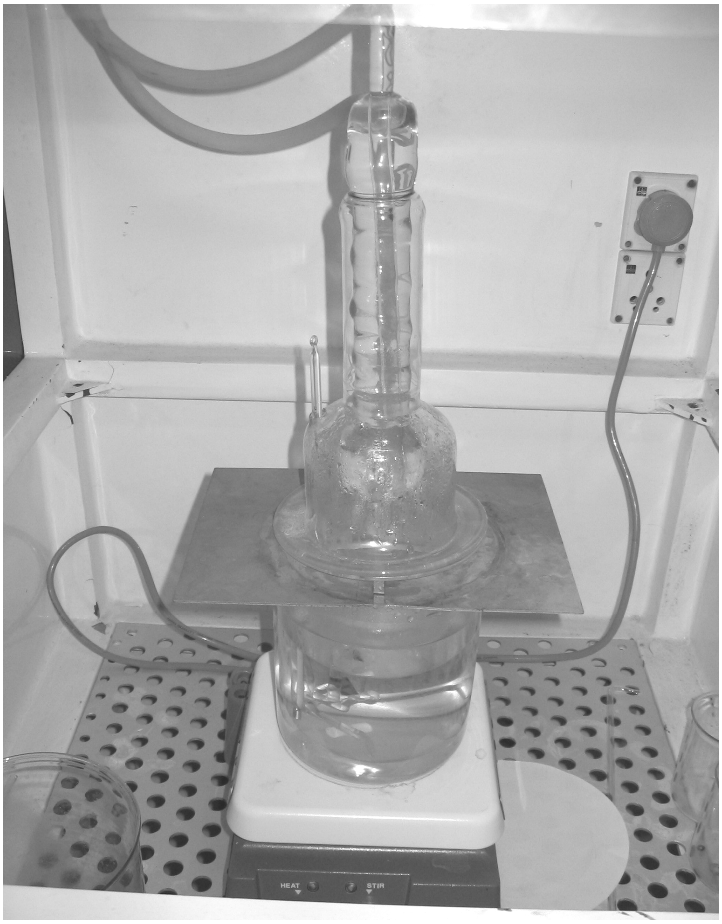

Fig. 1:

Initially KOH solutions of concentrations varying from 10% to 45% by weight were prepared. For each concentration of the KOH solution, samples were identified for etching with varying temperature of the KOH solution starting from 50^{o}C to 100^{o}C. Optical microscope was used to determine the etch depth and thereby etch rate was calculated for each sample. Quality of the etched surfaces was inspected using AFM in contact mode.

RESULTS AND DISCUSSION

Boiling point of the KOH solution for varying concentrations was experimentally determined and plotted as shown in Fig 2.

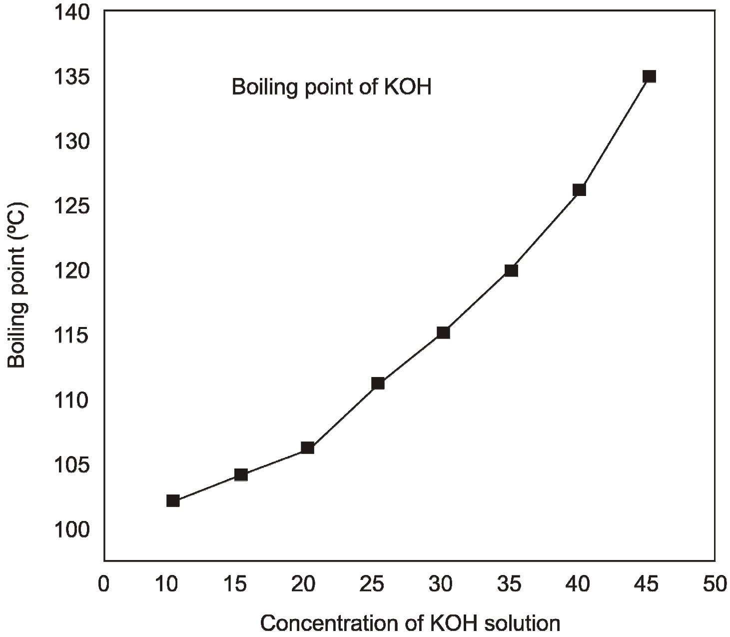

Fig. 2

The boiling point of the KOH solution set the upper limit for the allowable maximum temperature for a given concentration. The boiling point of the KOH solution almost increases linearly with increasing KOH concentration. The temperatures identified for the present work were 80, 90, and 100°C for which KOH concentration was varied from 10% to 45% by weight. The variation in etch rate of (100) silicon with temperature for 45% by weight of KOH is shown in Fig. 3. A step increase in etch rate is observed with temperature. This graph verifies the Arrhenius between the etch rate and temperature[7-8]. The variation of etch rate of (100) silicon with concentration at the temperatures of 50 °C, 60 °C and 70 °C is shown in Fig. 4. The graph shown in Fig. 4 indicates that variation of etch rate with concentration is less pronounced at lower temperatures like 50 °C and 60 °C than at 70 °C, since the etch rate itself at these temperatures is less. The variation in etch rate at 70 °C shows first, an increase with concentration and then a decrease.

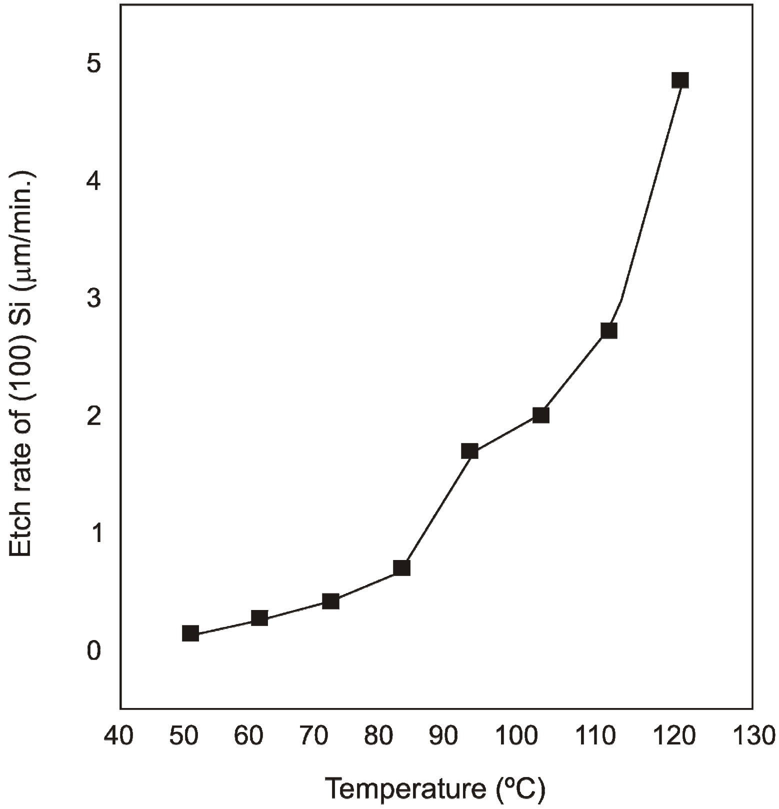

Fig. 3

An explanation provided by research conducted much earlier suggested a model where both OH^- and H_2O participate in the reaction with silicon as follows:

$$Si + 2\,H_2O + 2\,OH^- \longrightarrow Si(OH)_4 + H_2$$

Such a reaction would proceed at a lower rate in the absence of any one of the reactants H_2O and OH^-. Since at low KOH concentration, OH^- is in low concentration, and at high KOH concentration, H_2O is available in low concentration; the reaction would slow down at both; high and low KOH concentrations. However, this is not a comprehensive explanation capable of explaining all the experimental results obtained.

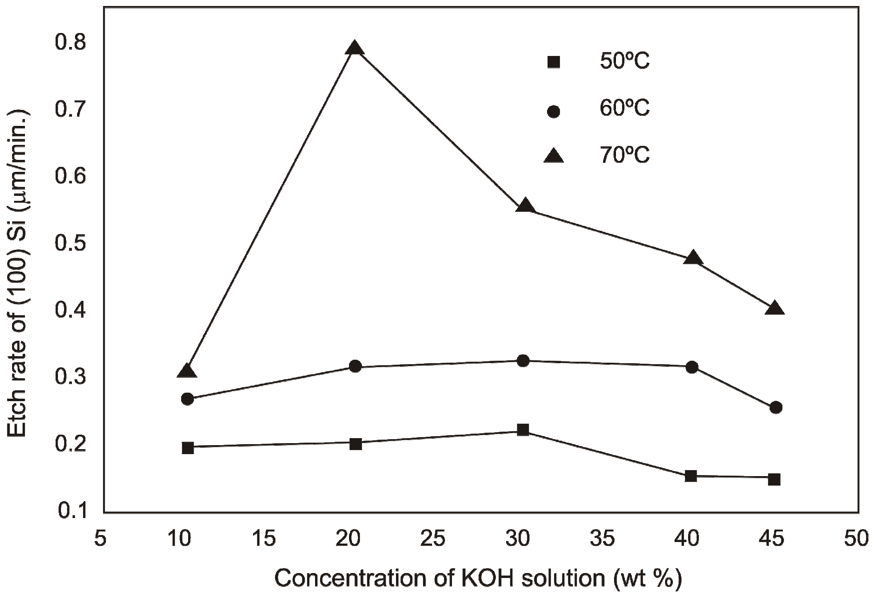
Etch rate of (100) Si (μm/min.)
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
5
10
15
20
25
30
35
40
45
50
Concentration of KOH solution (wt %)
■ 50ºC
● 60ºC
▲ 70ºC

Fig. 4

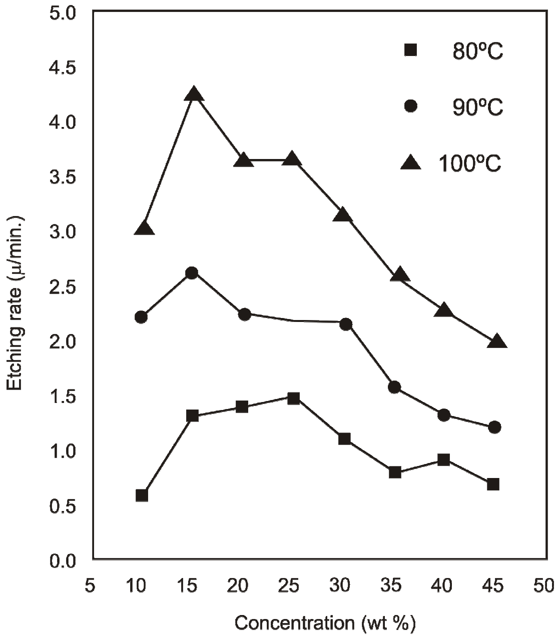
Etching rate (μ/min.)
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5
10
15
20
25
30
35
40
45
50
Concentration (wt %)
■ 80ºC
● 90ºC
▲ 100ºC

Fig. 5

Fig. 5 shows the result obtained for the variation of etch rate of (100) silicon with concentration at the temperatures of 80 °C, 90 °C and 100 °C. The etching rate shows a peak for each temperature curve that is observed at lower concentration with increasing temperature. At 80 ^{0}C, a maximum etching rate of 1.5 µm/min is obtained for 25 wt % KOH solution. For temperatures above 80^{0}C, the peak of the maximum etch rate is observed at 15 wt % KOH solution. At 100 ^{0}C the maximum etch rate is about 3 times of that at 80 ^{0}C.

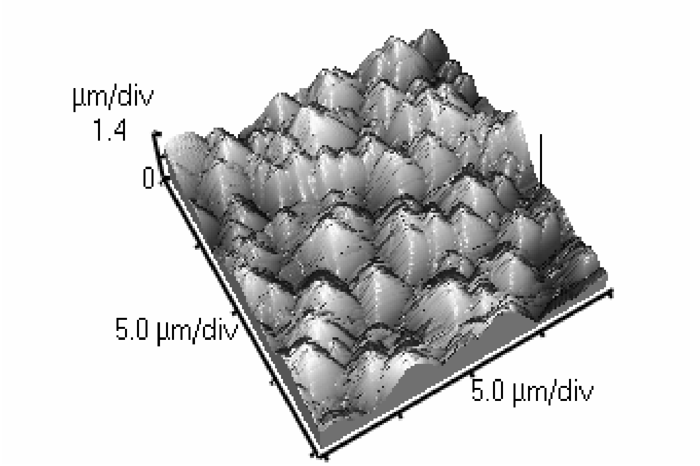

Fig. 6(a)

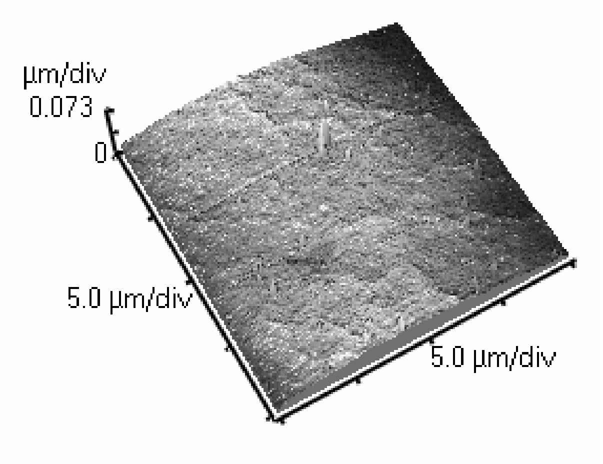

Fig. 6(b)

The morphology of the etched surface obtained with an AFM and optical microscope at typically two concentrations of the KOH solutions has been shown in Fig. 6 and 7. In 10% wt KOH solution, surface of the silicon becomes more hydrophilic, which further reduces to less hydrophilic with increasing temperature. The silicon (100) surface is subjected to changes from hydrophilic to hydrophobic and again hydrophilic with increasing KOH concentration. However, the higher temperature has been found to be preferred one with low KOH concentration.

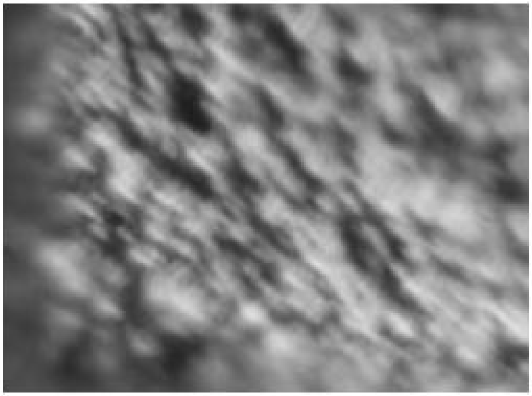

Fig.7 (a)

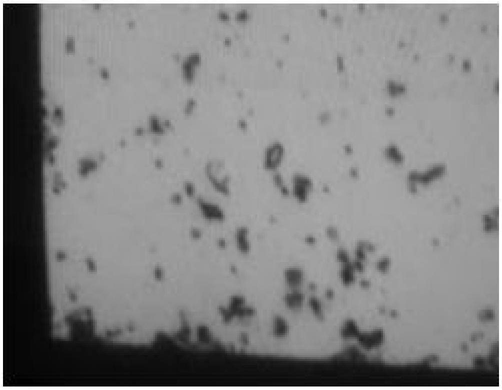

Fig. 7 (b)

CONCLUSION

Faster etching rate with smooth silicon surface has been observed at higher temperature in low KOH concentration solutions. The data has been found useful to formulate a physical model for the anisotropic etching of (100) silicon in KOH solution. The boiling point of the KOH solution decreases with decreasing concentration and thereby limits the use of higher temperature at low concentration KOH solutions. However, 100 ^{o}C is the most preferred temperature for 15% wt KOH solution for smooth etched surface and faster etching rate. At higher temperature of the KOH solution, hydrophobic nature of silicon surface tends to prevail giving rise the enhanced etched rate with smooth silicon surface. Pure KOH solution provides improved process control resulting into added advantage for MEMS applications.

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