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Anisotropic Etching of Crystalline Silicon in Alkaline Solutions

I. Orientation Dependence and Behavior of Passivation Layers

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ABSTRACT

The anisotropic etching behavior of single-crystal silicon and the behavior of SiO_2 and Si_3N_4 in an ethylenediamine-based solution as well as in aqueous KOH, NaOH, and LiOH were studied. The crystal planes bounding the etch front and their etch rates were determined as a function of temperature, crystal orientation, and etchant composition. A correlation was found between the etch rates and their activation energies, with slowly etching crystal surfaces exhibiting higher activation energies and vice versa. For highly concentrated KOH solutions, a decrease of the etch rate with the fourth power of the water concentration was observed. Based on these results, an electrochemical model is proposed, describing the anisotropic etching behavior of silicon in all alkaline solutions. In an oxidation step, four hydroxide ions react with one surface silicon atom, leading to the injection of four electrons into the conduction band. These electrons stay localized near the crystal surface due to the presence of a space charge layer. The reaction is accompanied by the breaking of the backbonds, which requires the thermal excitation of the respective surface state electrons into the conduction band. This step is considered to be rate limiting. In a reduction step, the injected electrons react with water molecules to form new hydroxide ions and hydrogen. It is assumed that these hydroxide ions generated at the silicon surface are consumed in the oxidation reaction rather than those from the bulk electrolyte, since the latter are kept away from the crystal by the repellent force of the negative surface charge. According to this model, monosilicic acid $Si(OH)_4$ is formed as the primary dissolution product in all anisotropic silicon etchants. The anisotropic behavior is due to small differences of the energy levels of the backbond surface states as a function of the crystal orientation.

Anisotropic etchants for crystalline silicon have been known for a long time (1-3). Their first applications included the etching of V-grooves on <100> silicon or U-grooves on <110> silicon, in order to fabricate MOS transistors for high power and high current densities (4). Increasing attention has been paid to this etching technology, after recognizing its unique capabilities for micromachining three-dimensional structures (5-9). Due to the strong dependence of the etch rate on crystal direction and on dopant concentration, a large variety of silicon structures can be fabricated in a highly controllable and reproducible manner. Typical structures include thin membranes, deep and narrow grooves, and cantilevers with single or double sided suspension. Important fields of application include the fabrication of passive mechanical elements, sensors, and actuators, as well as micro-optical components (8, 10). Among the best known examples are sensors for pressure (8), acceleration (11), and flow (12), as well as ink jet nozzles (13), connectors for optical waveguides (14), and major components of a gas chromatograph (15).

All anisotropic etchants are aqueous alkaline solutions, where the main component can be either organic or inorganic. The first organic system was proposed in 1962 and consisted of hydrazine (N_2H_4) and water with the addition of pyrocatechol ($C_6H_4(OH)_2$) (16). It was shown that pyrocatechol is not a necessary component, and might well be omitted (2). Experiments were made with iso-2-propyl alcohol as a third component, which was shown to act as a moderator (3). In a later work, hydrazine was substituted by ethylenediamine ($NH_2(CH_2)_2NH_2$), which is more stable and less toxic than the former (2).

Purely inorganic aqueous solutions of KOH and NaOH have been known to etch silicon anisotropically for a long time (1). A different system with improved etching behavior was obtained by the addition of isopropyl alcohol (17)

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In general, all aqueous solutions containing hydroxides of other alkali metals, like LiOH, and CsOH (18) perform in a similar manner. Aqueous solutions of ammonium hydroxide (NH_4OH), were also reported to etch anisotropically (19). The same type of solution with the addition of H_2O_2 is frequently employed for the cleaning of silicon wafers (20). More complicated derivatives of ammonium hydroxide, *e.g.*, so-called quaternary ammonium hydroxides like tetramethyl ammonium hydroxide ($N(CH_3)_4OH$) and choline ($(CH_3)_3N(CH_2CH_2OH)OH$) can also be used as anisotropic etchants (21).

General Considerations

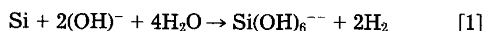
Solutions consisting of ethylenediamine, water, and pyrocatechol (EDP) are among the most widely employed. Many essential results concerning the composition of this system and its crystal orientation dependence were reported in the work done by Finne and Klein (2). They found that pyrocatechol can be omitted, so that in its most primitive form, the etchant solely consists of ethylenediamine and water. With nonaqueous ethylenediamine no etching was achieved, indicating that water is an active and necessary component. The maximal etch rate occurred at an ethylenediamine to water molar ratio of approximately 1:2. By the addition of pyrocatechol, a strong increase of the etch rate by a factor of three was obtained, saturating at a concentration of approximately 4 mole percent (m/o). For the three main silicon crystal orientations <100>, <110>, and <111>, Finne and Klein found an etch rate ratio of 17:10:1.

The effects of further additives were studied by Reisman *et al.* (22). They found that when exposing the EDP solution to oxygen, 1,4-benzoquinone and other products are formed, leading to an increase of the etch rate and a darkening of the solution. This effect can be avoided by continuously purging the etching apparatus with an inert gas (2, 22). They also found that trace quantities of pyrazine ($C_4H_4N_2$) lead to an increase of the etch rate. However, similar to pyrocatechol, this effect nearly saturates at a concentration of 3g pyrazine per liter ethylenediamine. Since commercially available ethylenediamine usually contains an unknown trace amount of pyrazine, Reisman *et al.* (22) proposed to intentionally add enough pyrazine to the solution so that the saturation level is reached.

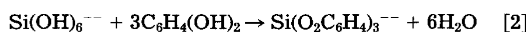
For the ethylenediamine-water-pyrocatechol system several recipes were proposed. Reisman *et al.* developed two specific solutions optimized for use where either a high etch rate is required ("F"), or where slower etch rates and/or lower temperatures are desired ("S") (22). Their specific compositions are listed in Table I, together with a recipe used by Finne and Klein ("T") (2) and another one proposed by Bassous ("B") (23).

For both solutions suggested by Reisman *et al.* (22) an activation energy of 0.36 eV on <100> silicon was found, which increased to 0.47 eV when no pyrazine was added. Furthermore, they determined an anisotropy ratio for the <100>/<111> silicon etch rates of 19 and 13.5 with and without pyrazine, respectively.

Finne and Klein were the first authors to publish reaction equations for the etching process (2). Based on a chemical analysis of the reaction products and on the observation that hydrogen evolves during etching in a stoichiometric ratio of approximately 2 H_2/Si, they proposed an oxidation-reduction step with hydroxide ions and water reacting with the silicon surface, followed by a chelation stage involving pyrocatechol



$$Si + 2(OH)^- + 4H_2O \rightarrow Si(OH)_6^{--} + 2H_2 \quad [1]$$



$$Si(OH)_6^{--} + 3C_6H_4(OH)_2 \rightarrow Si(O_2C_6H_4)_3^{--} + 6H_2O \quad [2]$$

They assumed the chelation to be the slow step, unless pyrocatechol was added at a concentration exceeding 4 mole percent (m/o). In that case they considered the oxidation reaction to be rate limiting. Pyrocatechol was assumed to act mainly as an agent to increase the solubility of the silicon compound, thus increasing the reaction rate.

The above mentioned oxidation-reduction equation was used by several authors in later publications without

Table I. Composition of different EDP solutions published by Finne and Klein (2), Reisman *et al.* (22), and Bassous (23)

Type		S (22)	F (22)	B (23)	T (2)
Water	ml	133	320	320	470
ED	l	1.0	1.0	1.0	1.0
Pyrocatechol	g	160	320	160	176
Pyrazine	g	6	6		

major modifications (22, 24). The gross reaction proposed in this equation does not provide an obvious explanation for the anisotropic behavior of the etchant. For this purpose it must be broken up into its fundamental reaction steps.

It was noticed by several workers that residues might appear on the silicon surface. The occurrence of this phenomenon depends on the composition of the solution and on its saturation level with silicon. For EDP solutions, Reisman *et al.* (22) have found that this tendency increases with the water content of the solution. With respect to the aging of the solution, quantitative results were given by Wu *et al.* (25). They found that in a one liter solution (type F and type B) at a temperature of 100°C a maximum amount of 10g silicon can be etched without producing solid residues. This value was slightly higher for the F etchant which was attributed to its larger content of pyrocatechol. A chemical analysis of the residues showed that they consisted mainly of SiO_2 with additional trace amounts of reaction products.

The work of Abu-Zeid *et al.* (26) showed that, for ethylenediamine-based solutions, the silicon etch rate can be increased considerably by stirring. They also showed that the etch rate depends on the effective silicon area being exposed to the solution and its geometry. An increase can be observed, when the area of the active region gets smaller. These results indicate that diffusion processes influence the silicon dissolution rate considerably.

For hydrazine water solutions a very similar behavior to the one observed in EDP solutions was found (3, 27, 28). At a temperature of 118°C, an etch rate ratio of 16:9:1 for the {100}:{110}:{111} planes was determined, which is comparable to EDP (28). When underetching convex corners, (211) was identified as an etch bordering plane with a very high etch rate (3, 27).

Among the inorganic solutions, the one most frequently used is based on KOH. The first detailed study on a ternary mixture of KOH, water, and isopropyl alcohol was given by Price in 1973 (17). His major observations were the following: the maximal etch rate occurred at a KOH concentration of 10-15 weight percent (w/o) when no alcohol was added, and around 30% KOH with alcohol. In general, the addition of isopropyl alcohol leads to a decrease of the etch rate. On <100> silicon the activation energy was found to be between 0.52 and 0.69 eV. He found no effect of stirring on the etch rate, indicating that the reaction is not diffusion limited. Under optimum conditions, Price observed an etch rate ratio of 35:1 for the {100}/{111} crystal planes (17). A much higher anisotropy ratio of up to 500:1 for the <110> to <111> etch rates in a highly concentrated 55% KOH solution was reported by Kendall (29, 30). Further data on the etch rates of <110> and <111> silicon as well as SiO_2 were given by Clark and Edell (31). For KOH solutions with a concentration between 9 and 54 w/o, they found the following ranges of activation energies: 0.6-0.8 eV for <110>, 0.4-0.9 eV for <111>, and 0.8-1.0 eV for SiO_2.

In a work done by Palik *et al.* (32) the etching process of KOH on silicon was monitored *in situ* by Raman spectroscopy. From these experiments the main reaction species was determined to be OH^-. They proposed $SiO_2(OH)_2^{--}$ to be the primary etching product with subsequent polymerization. The following overall gross reaction was suggested by them (33)

$$Si + 2H_2O + 2\,OH^- \rightarrow Si(OH)_2O_2^{--} + 2H_2 \quad [3]$$

From experiments done with isopropyl alcohol added to the KOH solution, they conclude that the alcohol does not

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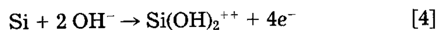
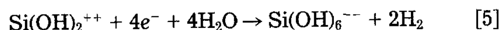
participate chemically in the reaction. In a later work (34), Palik *et al.* included energy level considerations, stating that the etching reaction transfers an electron from OH^- into the silicon surface bond and then back to the etch products.

A more detailed break down of the reaction equations, including the transfer of charge was suggested by Raley *et al.* (24). They assumed that four electrons are injected into the conduction band by an initial oxidation reaction, which are later consumed in a reduction step



$$Si + 2\,OH^- \rightarrow Si(OH)_2^{++} + 4e^- \quad [4]$$



$$Si(OH)_2^{++} + 4e^- + 4H_2O \rightarrow Si(OH)_6^{--} + 2H_2 \quad [5]$$

In the literature published to date several attempts were undertaken to explain the anisotropic behavior of these etchants. Price indicated that a correlation between the available bond density of different crystal planes and the etch rate could exist (17). However, it is difficult to explain etch rate ratios of about 100:1 when the bond density only varies by a factor of two. Another proposal was made by Kendall who argues that {111} planes get oxidized more rapidly than others and therefore could be covered with a thin oxide layer immediately after immersion into the etchant (29). Palik *et al.* assume that the anisotropy might be attributable to differences in activation energies and in backbond geometries on different surfaces (33).

In this paper, experimental results on the orientation dependence of the silicon etch rate for several solutions as a function of composition and temperature will be given. An attempt will be made to give a model valid for all anisotropic silicon etchants, explaining the underlying mechanism. Furthermore, results on the etch behavior of the most widely used passivation layers SiO_2 and Si_3N_4 will be reported.

The effects of dopants on the silicon etch rate will be discussed in an accompanying paper (35).

Experimental

In the experiments, n- and p-type Czochralski grown 3 in. wafers with <100> and <110> orientation were used. The resistivity was 1-10 Ω-cm, corresponding to an impurity concentration of approximately $1 \cdot 10^{15} - 1 \cdot 10^{16}$ cm^{-3}. For passivation, these wafers were either thermally oxidized or a CVD silicon nitride layer was deposited.

In order to obtain detailed data on the crystal orientation dependence of the etch rate, a fan shaped or wagon wheel shaped masking pattern was employed, consisting of radially divergent segments with an angular separation of one degree (36-40). This pattern was transferred to the passivation layer. Thus, alternating segments of bare silicon and regions covered with silicon dioxide or silicon nitride were obtained. The size of a chip containing one such pattern was $16 \cdot 16$ mm^2. In order to achieve a more accurate angular resolution, particularly in the neighborhood of {111} planes, where the etch rate is a very sensitive function of the angular orientation, a second pattern with segments and spaces differing by an angle of 0.1° and with a total angular spread of 4° was used.

For etch rate studies on passivation layers, wafers covered with thermal SiO_2 (1000°C, wet), CVD-SiO_2 (SiH_4, N_2O, 800° and 900°C) and CVD-Si_3N_4 (SiH_4 and NH_3, 900°C) were prepared and diced to the same chip size mentioned above.

The samples were etched in an oil-heated, double-walled glass vessel. The temperature was varied between 20° and 115°C and was controlled with an accuracy of ±0.2°C. In order to keep the composition of the etchant constant, the vapors were recondensed in a water cooled reflux condenser. In the case of EDP solutions, an additional nitrogen purge was applied, in order to prevent changes of the etch properties due to contact with atmospheric oxygen (2, 12).

The inorganic etchants employed in the experiments were KOH, NaOH, and LiOH with concentrations in the range of 10-60, 24, and 10%, respectively, where all values are in weight percent. In some experiments, isopropyl alcohol was added to a 20% KOH solution according to the following recipe: 1 liter H_2O, 312g KOH, 250 ml isopropyl alcohol. The only organic etchants used were EDP solutions, among which a composition type "S" introduced by Reisman *et al.* was employed most frequently (Table I). This choice was made because it is applicable in a wide temperature range which is important for obtaining reliable data on activation energies.

After etching the chips masked with a wagon wheel pattern, a blossom-like figure developed. This phenomenon is due to the total underetching of the passivation layer in the vicinity of the chip center, leaving an area of bare exposed silicon. The radial extension of this area depends on the crystal orientation of the individual segments, leading to a different amount of lateral underetching. The pattern developed on a <100> test sample etched in an EDP solution is shown in Fig. 1.

Visual inspection of such blossom-like patterns was only used for a qualitative evaluation of the anisotropy. In order to obtain quantitative results, the lateral etch rates were determined by measuring the width w of the overhanging passivation layer with an optical linewidth measurement system (LEITZ Latimet). This is illustrated in Fig. 2, showing a schematic cross section of the test chip after etching. Additionally, the depth d of the etch grooves and the orientation of the etch bordering planes were determined by means of a mechanical stylus profiler and laser reflection methods, respectively (41). Similar measurements were performed on chips with the 0.1° pattern.

For determining the etch rate of the passivation layers, their thicknesses were measured by ellipsometry before and after etching.

Results

Morphology of the etched silicon surfaces.—After etching a partially masked silicon surface, two regions of interest arise. One is the bottom surface obtained from vertical etching, the other consists of the laterally under-etched sidewalls developing along the edges of a masking pattern.

When using EDP solutions, the surfaces generally tend to be quite smooth, particularly on etched bottom surfaces of <100> wafers. Also, the laterally under-etched sidewalls of the passivated segments are generally smooth. However, as indicated in Fig. 3, a certain waviness can be observed on fast etching sidewall crystal planes.

On etched surfaces of <110> wafers with relatively large areas exposed to the etchant, a linearly textured structure develops which can be recognized with the naked eye. When viewed under a scanning electron microscope, as shown in Fig. 4, these textured surfaces appear to be bounded by well-defined, slanted crystal planes.

As has been observed in numerous previous studies, {111} crystal planes form perfectly smooth lateral sidewalls, both on <100> and on <110> wafers, and for all etchants investigated (5, 6).

In case of alkaline etchants like KOH, the vertically etched bottom surfaces of <100> and <110> wafers turn out to be relatively smooth. However, when using a solu-

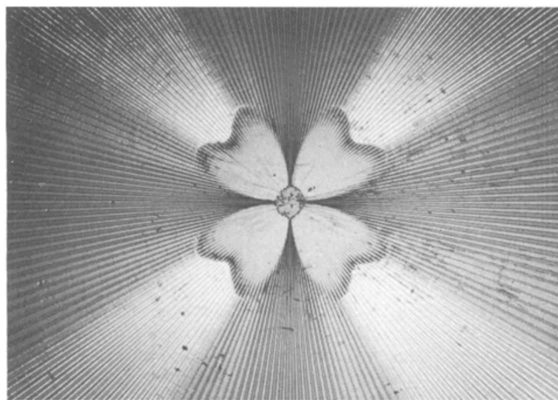


Fig. 1. Etch pattern emerging on a masked test wafer with the orientation <100> after etching in an EDP solution type S. The residual oxide at the center is due to the finite resolution of the mask.

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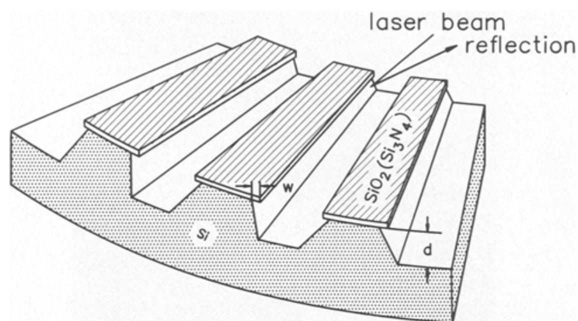

Fig. 2. Schematic cross section of a silicon test chip covered with a star-shaped masking pattern after etching.

tion of low concentration (below 30%), a tendency for the formation of pyramids can be observed on <100> wafers. The laterally underetched sidewalls of the masked segments generally appear to be fragmented, as demonstrated in the micrographs of a <110> wafer shown in Fig. 5a and b. Both pictures show a steep-walled mesa rising over a more gently sloped shoulder. Within this formation patches with smooth {111} surfaces can be observed. They are surrounded by jagged surfaces which could not be identified as any particular crystal plane.

Etch limiting crystal planes.—By means of laser reflection measurements, as indicated in Fig. 2, the crystal orientation of the planes bounding the laterally underetched segments was determined for the case of EDP etchants. These planes are shown in Fig. 6 for <100> and <110> wafers in the form of stereographic projection diagrams. For both wafer orientations they can be characterized by the Miller indexes $\{hhk\}$ where h and k are integers with $h \geq k$. This was found for all EDP compositions and temperatures applied.

As mentioned above and indicated in Fig. 4, a linearly textured structure develops on bottom surfaces of etched <110> wafers. The crystal orientation on the long steps in this texture was found to be close to {331}. On the basis of these results, it is surprising to observe the formation of smooth, laterally underetched {110} surfaces on <100> wafers, when using EDP solutions. A similar surprise is the formation of smooth, flat-etched bottoms when etching narrow grooves bounded by vertical {111} planes on <110> wafers. This phenomenon is probably attributable to the different geometrical situation of a flat, large surface, as compared to a small sidewall bounded by concave corners.

For silicon surfaces etched by KOH, the resulting crystallography depends on the composition of the etchant. Furthermore, as mentioned above, only a limited number of crystal planes can be identified. For relatively high KOH

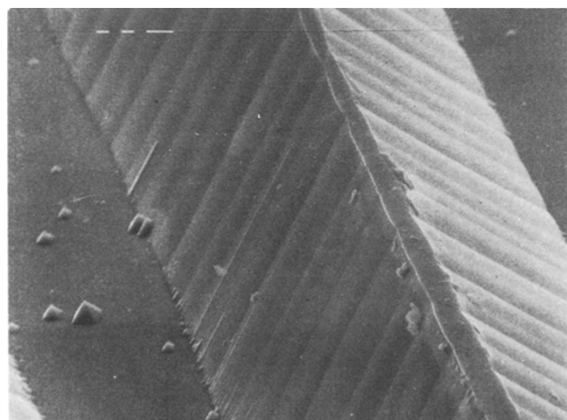

Fig. 3. Fast etching sidewalls on a <100> silicon wafer after expo-

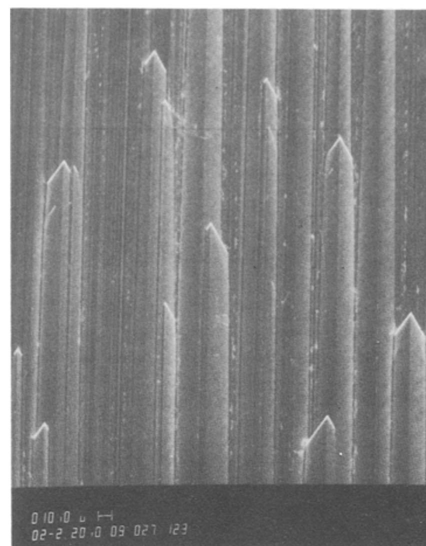

Fig. 4. Main surface of a <110> silicon wafer after etching in an EDP solution type S.

concentrations, exceeding 35%, these planes are shown in the stereographic projection diagrams in Fig. 7. In contrast to EDP solutions, vertical {100} planes emerge on <100> wafers (Fig. 7a) when a masking pattern with an angle of 45° to the flat is used (39). When misaligning such a pattern by a few degrees with respect to this 45° line, the vertical smooth sidewall remains, but an inclined shoulder begins to form in the corner. When misalignment exceeds a few degrees, a similar situation as shown in Fig. 5a develops.

The crystal planes marked in the central region of the <110> diagram, Fig. 7b, correspond to the slanted shoulders depicted in Fig. 5b. The vertical {111} planes on <110> wafers rise over an inclined shoulder of {311} planes. Such a shoulder does not arise when using EDP solutions.

Lateral etch rate as a function of crystal orientation.—As mentioned above, it was found for EDP solutions that etch bordering crystal planes develop, which are characterized by the indexes $\{hhk\}$ ($h \geq k$). The lateral underetch rates of these planes on <100> and <110> wafers for a temperature of 95°C are shown in the polar coordinate diagrams in Fig. 8. Apart from the sharp absolute minima at {111} planes, relative minima occur at {110}. The maximal lateral etch rate is observed for {331} planes.

The differences of the lateral underetch rates of crystal planes occurring several times on the same diagram, *e.g.*, in Fig. 8b, are due to different slanting angles of these planes. The immediate result of the measurement is the projection of the etch rate onto the surface of the wafer. The true etch rate can be calculated by multiplying the apparent lateral underetch rate, as measured by the overhanging film of the passivation layer, by the sine of the inclination angle Θ. This factor can be given as a function of the lateral alignment angle ϕ in the wafer plane. When the <110> direction, which corresponds to the intersecting line of the {100} wafer plane and the resulting etch bordering {111} plane, is taken as $\phi = 0°$, the following formula applies

$$\sin\Theta = \sqrt{1 - \frac{1}{\tan^2(45° - \phi) + 2}} \qquad 0° \leq \phi \leq 45° \qquad [6]$$

For <110> wafers again with the <110> direction taken as $\phi = 0°$, which in this case corresponds to the intersecting line of the {110} wafer plane and the {111} planes with an inclination angle of 35.3°, the correction factor is given by

$$\sin\Theta = \sqrt{\frac{\tan^2\phi + 1}{2\tan^2\phi - 2\sqrt{\tan\phi} + 3}} \qquad 0° \leq \phi \leq 54.7° \qquad [7]$$

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