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A Study of Anisotropic Chemical Etching on Crystalline Silicon

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Abstract

It is known that chemical reaction rates can be different on different crystal planes. KOH is a common etchant for Si and the reaction rate on {100} planes is much faster than the {111} planes. Therefore a square etch window on a (100) Si surface can become an inverse pyramid at some point after KOH etch. However, it is not clear how a 3-dimensional cubic crystal will evolve by KOH etching. In this study we use a dicing saw to create arrays of rectangular crystal pillars of different dimensions in two different orientations followed by 6M KOH etch at 50º C. We use scanning electron microscopy to record the evolution of the pillar morphology with and without an oxide cap layer. We find that the complex geometry is a result of the competition between different crystal planes.

Introduction

Etching is an important process in device fabrication. Alkaline aqueous based etchants include KOH, NaOH, LiOH, CsOH, RbOH, NH_4OH, and TMAH can provide anisotropic etching of crystalline silicon. The etch rates on different crystal planes are different. For KOH etching the 3 common wafer orientations, the etch rate on <110> is slightly greater than that on <100> and both are more than 400 times faster than that on <111>. However, in the planary device fabrication, it is common to create a pit or trench on Si surfaces. (Fig. 1) The final morphology of pit is dictated by the slowest etch planes. In this study, we use a dicing saw to cut a Si(100) wafer into square and diamond shaped pillars. The pillars allow visualization of the competition between many crystal planes. A scanning electron microscope is used to characterize the evolution of the pillar morphology in 6M KOH solution at 50 °C.

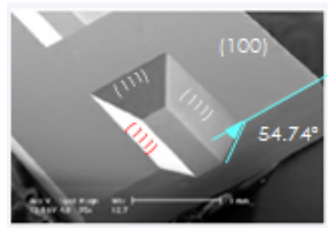

Fig. 1 Si(100) surface etched in KOH.

The chemical reaction of Si etching in alkaline solutions

$Si + 2OH^- + 2H_2O \rightarrow SiO_2(OH)_2^{\,2-} + 2H_2$ [Ref.2]

Purpose

The purpose of this experiment is to investigate how the shapes of 3D crystalline silicone evolve when many crystal planes are exposed to a KOH etch simultaneously.

Experimental Procedure

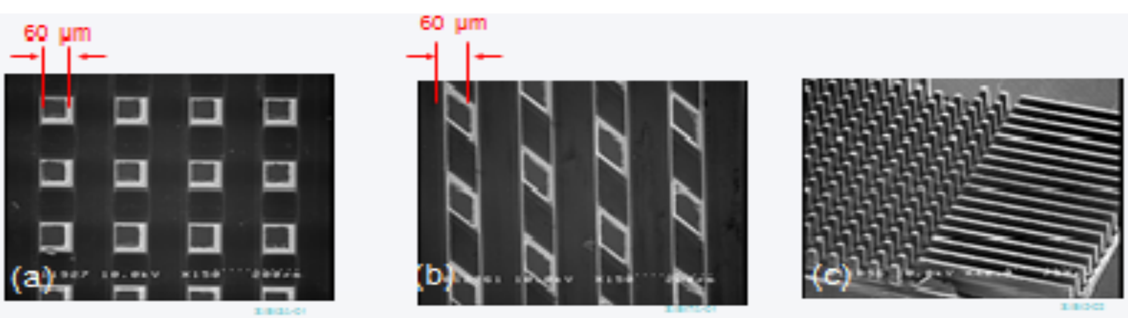

Fig. 2 Top view of the diced pillars (a) square (b) rhombus. (c) Sideview of the square pillars.

Si(100) wafers were diced into different arrays of square and rhombus shaped pillars of 60 µm on the side, 180 µm in pitch, and 210 µm tall.(Fig. 2) Oxide acts as a resist to KOH etch. Thus a Si wafer with a layer of 450 nm oxide is used to compare with wafers without oxide, hence giving 6 different samples. To expose different crystal planes to the etchant, the square samples were diced either along <110> directions (Fig. 3a) or <100> directions (Fig. 3b); the rhombus samples were diced along <100> and <110> directions. (Fig. 3c) Etch was conducted by 6M KOH solution at 50 ºC in 15-min intervals. Etch results were characterized by SEM.

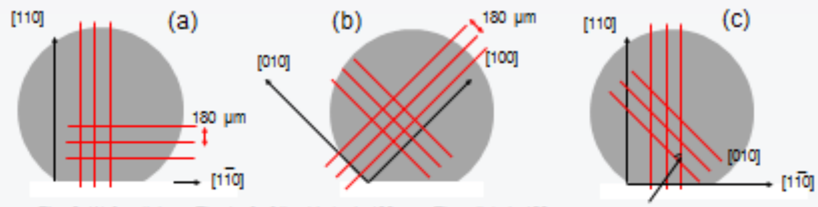

Fig. 3 Wafer dicing. The kerf of the blade is 120 μm. The pitch is 180 μm.

Results

1. Dicing along the [110] direction on (100) wafers

The cutting planes are the {110} planes. It is the fastest etching plane. Therefore the initial {110} facets gradually reduce its size to some slower facets. (Fig. 5) The sidewalls in the {110} planes are very rough. (Fig. 6) The cutting marks reveals different crystal planes and they could be slower than the {110} plane. This is consistent with Ref[1]. The curved base created by the cutting blade provides a preferred slow crystal plane very quickly. That plane could be {111}. The same holds for the sidewalls of the long trenches cut in the directions of <110>. (Fig. 5b) The corner provides two different crystal planes which should have even slower rate that {111} so that the {111} facets totally disappear at the end. (Fig. 7) The top is a {001} plane which is a slower etching plane than the sidewall {110} planes. We would expect the shrinking of the pillar shaft faster than the height reduction. The surface of the (001) forms hillocks. The structure of the hillock could be very complicated. [1] After etching for a long time, the result is octagonal pyramids. (Fig. 7) The final crystal plans could be determined by measuring the height of the pyramid.

15 minute etch

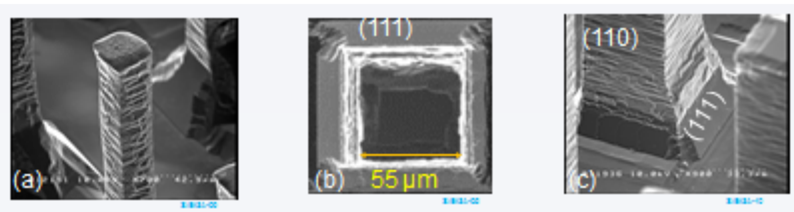

Fig. 5 (a) Tip, (b) top view. and (c) base of a square pillar diced along <110> directions after 15 min etch .

Significant etch occurred on the {110} sidewalls in 15 min. (Fig. 5a, 5b) Downward etch on the top (001) surfaces is not obvious, but new facets initiated by dicing on the edges are developed. (Fig. 5a) Initiated by dicing, (111) surfaces are developed at the base, but new facets are also developing at the corners. (Fig. 5c)

60 min etch

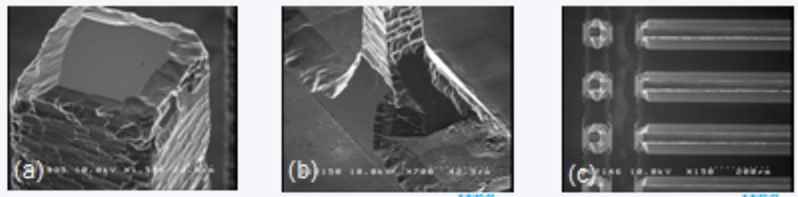

Fig. 6 (a) {110} sidewalls were etched to 35 µm and very rough. Hillocks developed on the top (001) surface. (b) {111} facets at the base are reduced at the corners. (c) {111} facets developed at the base of walls.

Significant etch occurred on the {110} sidewalls at ~25 µm/h. (Fig. 6a) The {111} facets at the base are losing to the new facets developed at the corner. Hillocks on the top (001) surface are observed [2]. (Fig. 6b) Walls along the [110] direction developed {111} facet at the base. (Fig. 6c) Also the {311} faces are becoming visible and the octagonal pyramid begins to become visible. (Fig 7)

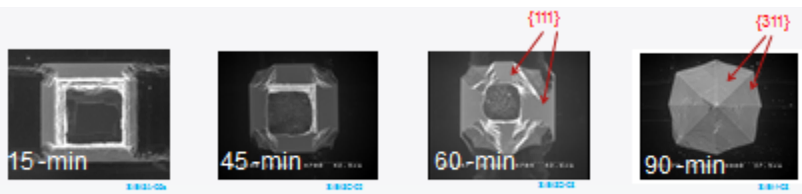

Fig. 7 The {111} planes disappear completely from the pillar bases after 90-min etch.

Without corners {111} planes are quite stable against KOH etch. (Fig. 8a) The cross-section images indeed confirm the planes are {111}. (Fig. 8b)

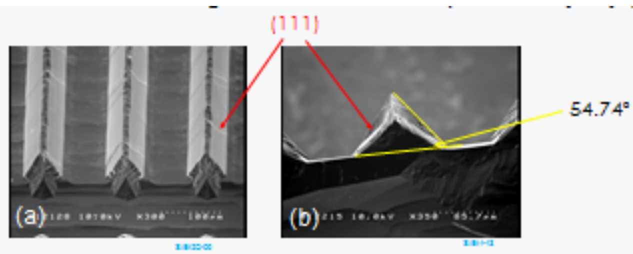

Fig. 8 (a) Top view and (b) sideview of the one-dimensional walls after 60-min KOH etch.

2. Dicing along the [100] direction on (100) wafers

The cutting plane should be the {100} plane. It should be a faster etching plane than the {111} direction. Also, The {100} sidewalls are smoother but with hillocks. The etch rate is ~ 25 µm/h. (Fig. 9a) Facets of {111} were developed at the base. (Fig. 9b, 9d) Two new facets were developed in each <110> direction. (Fig. 9c) Top (001) surface is etched smaller but no sharp point yet. (Fig. 9c)

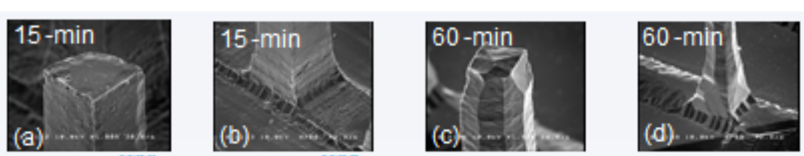

Fig. 9 The evolution of {100} sidewalls in KOH etch.

3. Dicing along the [010] and [110] directions on (100) wafers

The cutting plane reveals the contrasts between two surfaces under KOH etch. The [100] surface is smooth with hillocks but [110] surfaces are rough. (Fig 10a-10c) Each {100} facet develops a new facet in the <110> direction, the rhombus cross-section is significantly reduced to ~12 µm in the middle. (Fig. 10d)

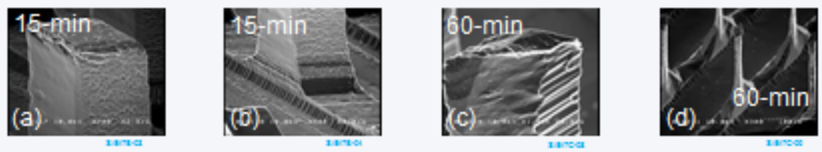

Fig. 10 Different etch characteristics of {100} and {110} sidewalls of a rhombus array lead to very thin pillars (d).

4. Effect of an oxide top layer

The oxide layer provide a mask for the KOH etch and a "bias". The pillars with oxide caps will be taller after the same etch time than those without oxide caps. The oxide can also be etched but at a much slower rate. When the Si substrate was etched, the oxide cap breaks off. (Fig. 11a, 11b) Facets such as {311} are developed under the oxide. (Fig. 11c) Pillars from <110> dicing are quite different from <100> dicing. (Fig. 11d, 11e

Fig. 11 (a) Top view of a <100> diced pillar after 60 min KOH etch. The irregular oxide cap is on the top. (b) Oxide capped octagonal pillar arrays. (c) Sideview of the oxide capped Si pillars after etching. Hillocks on the sidewalls. (d) Top view of a <110> diced pillar after 60 min KOH etch. (e) Oxide capped square pillar in the <110> directions but the base is developed into octagonal pyramids.

Conclusions

From the behavior of the etching of the three different arrays, both with and without oxide, we can tell that anisotropic etching is dictated by the etch rates of crystal planes. Namely, the {111} plane etches slower than the {100} and the {311} etches slower than the {111}. In addition, oxide can block the etching from occurring at all. Because of this, in order to create sharp points with high aspect ratio, multiple oxidation-etching cycles will be needed.

Reference

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