Etching Methodologies in $\langle 111 \rangle$ -Oriented Silicon Wafers

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Abstract—New methodologies in anisotropic wet-chemical etching of $\langle 111 \rangle$ -oriented silicon, allowing useful process designs combined with smart mask-to-crystal-orientation-alignment are presented in this paper. The described methods yield smooth surfaces as well as high-quality plan-parallel beams and membranes. With a combination of pre-etching and wall passivation, structures can be etched at different depths in a wafer. Designs, using the $\langle 111 \rangle$ -crystal orientation, supplemented with pictures of fabricated devices, demonstrate the potential of using $\langle 111 \rangle$ -oriented wafers in microsystem design. [523]

Index Terms—Bulk micromachining, etch technology, $\langle 111 \rangle$ wafers, reactive ion etching, wet chemical etching.

I. INTRODUCTION

IN MICROSYSTEM technology, bulk etching techniques have found a strong position next to surface micromachining techniques [1]. With bulk etching, rigid high qualitative monocrystalline structures can be etched. In contrast to surface micromachining, pre-stress, creep, and stress relaxation are less pronounced and better fatigue resistance is obtained [2]. Therefore, bulk etching techniques are very well suited for application areas where high demands are put on the mechanical and time-dependent material properties. Another advantage is the possibility to fabricate microstructures, built from rather thick material layers, cavities, and trenches well over a few micrometers in depth. Besides, the bulk processed silicon structures can be used as a mould to grow other construction materials in. Examples are channels for micro total analysis applications [3], [4].

For these applications, mostly $\langle 100 \rangle$ - and $\langle 110 \rangle$ -oriented silicon are used for their fast etch rates in the out-of-plane directions in anisotropic wet chemical etchants like potassium hydroxide (KOH). Also, $\langle 110 \rangle$ -oriented silicon is used since nice deep trenches with straight walls with respect to the wafer sur-

Fig. 1. Spatial positioning of the crystal octahedron, built with $\{111\}$ -planes in a $\langle 100 \rangle$ (left-hand side) and $\langle 111 \rangle$ -oriented (right-hand side) silicon wafer.

Fig. 2. Hexagonal contour formed by the projection of the atomic FCC structure on the $\{111\}$ -plane (left-hand side). The side view of the $\{111\}$ -plane orientations for three plane intersections is shown on the right-hand side (see Fig. 1). The out-of-the wafer-oriented $\{111\}$ -planes are inward and outward directed in an alternating way, with their intersection lines with the in-the-wafer-surface-oriented $\{111\}$ -planes defined by two equilateral triangles, respectively, forming an hexagon. The top triangle of Fig. 1 is accented.

face can be made. However, $\langle 111 \rangle$ -oriented silicon is rarely used since it etches slowly in anisotropic etching solutions [5].

In this paper, we will show the strong points of etching in $\langle 111 \rangle$ silicon and how this type of wafer orientation can be used in microsystem fabrication. The wafer off-axis cut and crystal-orientation-independent pre-etching techniques are used to allow new structures in anisotropic wet-chemical etching. In order to get insight in the etched geometries and possibilities, a good understanding of the etch mechanism and the orientations of the crystal planes is needed. For $\langle 111 \rangle$ wafers, this information is less familiar than for $\langle 100 \rangle$ silicon. Therefore, we shall start with a treatise about the crystal layout.

II. CRYSTAL LAYOUT

The basic spatial structure that describes the $\{111\}$ -planes in monocrystalline silicon is the octahedron, as shown in Fig. 1.

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Fig. 3. Etch mechanism for off-axis cut $\langle 111 \rangle$ wafers: an etch step train travels along the $\{111\}$ -planes bounded by the mask and out-of-plane-oriented $\{111\}$ -planes. The first cross-sectional picture shows roughness due to remaining etch steps. An SEM top view is shown in the right-hand side. Continuing this auto-polish process will finally remove all steps, resulting in a smooth surface.

For $\langle 100 \rangle$ -oriented silicon, this octahedron structure is oriented with the length axis-oriented perpendicular to the wafer surface. For $\langle 111 \rangle$ -oriented silicon, the structure is rotated over 54.7° such that one of the $\{111\}$ facets is oriented in the wafer plane (Fig. 1, right-hand side).

In the wafer plane, the crystal orientations are governed by the intersection of the out-of-plane, with the in-plane-oriented $\{111\}$ -planes. These intersection lines form a hexagonal shape, as shown in Fig. 2. The corresponding six $\{111\}$ -planes are inward and outward directed in an alternating way [2], [20]. The angle between the in-plane and out-of-plane-oriented planes is 70.5° .

III. ETCH MECHANISM

Anisotropic etching of monocrystalline silicon takes place at local scale by etch-steps traveling along the $\{111\}$ -planes [6]–[8]. This means that for a perfectly cut $\langle 111 \rangle$ wafer without a mask, the steps will start mainly at the wafer edge and etch pits that nucleate randomly all over the wafer. When a wafer with a mask window is etched, nucleation of steps at the wafer edge will be suppressed such that the etch rate is reduced to a minimum. Steps will only be generated at spontaneous nucleated pits and dislocations in the crystal lattice and oxygen precipitates. In practice, a perfect crystal aligning will never occur and often even a predefined off-axis cut is given. For this off-axis cut, situation steps will be initiated within the mask opening until all steps stop at the out-of-plane-oriented $\{111\}$ -planes defined by the mask geometry [9], as shown in Fig. 3.

Unlike $\langle 100 \rangle$ silicon, the surface, parallel to the original wafer surface, will become almost atomically flat after all rough steps have been etched away, which makes etching in $\langle 111 \rangle$ wafers very suitable for optical applications. The remaining low roughness is due to etch nucleation pits distributed over the $\{111\}$ surfaces [7], [10]. This technique of auto-polishing can be used to actively smoothen the surface at a defined area and to obtain a perfect surface alignment with the $\langle 111 \rangle$ orientations. By applying the auto-polishing step to the two sides of the wafer, parallelization of both sides is obtained such that a smooth wafer area with perfectly plan-parallel surfaces, oriented along the crystal orientations, is obtained. This method can only be ap-

Fig. 4. (A) Etch geometries obtained for wafers with a miscut parallel to the flat and the effects of the mask orientation. (B) With use of an adapted mask orientation, free etching of the mask structure can be achieved. (C)–(D) SEM pictures showing free-etched thin nitride beams with metal pads made with mask underetching.

plied to small regions, in order not to etch through the wafer. For example, a 10-mm-wide mask window already results in a maximum etch depth of $175 \mu\text{m}$ for an off-axis cut of 1° . Since most wafers types are cut at angles up to 2° , applying the auto-polishing method to full wafer surfaces is not possible, unless wafers with very small off-axis cut are used.

When a series of mask windows are made, aligned along the $\langle 111 \rangle$ orientations, a stepped structure is obtained, as shown in Fig. 4(A). Mask rotation which causes in-plane crystal misalignment will result in mask underetch such that freestanding mask structures can be obtained. This principle is shown in Fig. 4(A) and (B). With this underetching of the mask, free silicon nitride bridges can be made, for example, with heater elements for flow sensors, as shown in Fig. 4(C).

Fig. 5. Effect on mask layouts and orientation on the shape of the etched cavity: the white area is the mask window, the light gray area indicates the mask underetch. (A1)–(D1) Show the effect of rotation of a rectangular mask. (A2) and (B2) Show that outer polygonal contours are formed with 3–6 sides. (C2) Shows the fusion of two underetched contours to one large cavity. (D2) Shows that matrices of closely spaced hexagonal shapes can be obtained. The latter structure can also be obtained with closely spaced circular equilateral well-oriented triangular- or diamond-shaped mask windows.

Fig. 6. Effects of etching with a matrix of circular holes, differently oriented. Top-left, top-right, and bottom-right figures show mask underetching due to the strong underetching in the $\langle 0\bar{1}1 \rangle$ directions. A part of the bottom still consists of a rough stepped surface, whereas the other part is readily smoothed. The right-hand-side pictures show additional steps at the recently underetched mask areas.

As demonstrated, a rectangular mask window will result in a mask undercut. When off-axis cut is present, both the off-axis angle and in-plane orientation of the mask windows to the crystal orientation determine this mask underetching. Assuming zero off-axis cut and an infinitesimal deep initial cavity in the silicon to initialize sideways etching, a fully developed mask underetch is established, bounded by the out-of-the-wafer-plane-oriented $\{111\}$ -planes, defined by the $\langle 10\bar{1} \rangle$, $\langle 0\bar{1}1 \rangle$, and $\langle \bar{1}10 \rangle$ directions given in Fig. 2. The pre-etching technique will be discussed in more detail in the following sections. The etch cavity can be determined by moving lines parallel to these orientations from the outside toward the mask center until they intersect with the mask window. Applying this step for six lines and drawing straight lines between the neighboring intersection lines will give the resulting cavity, as demonstrated for different mask geometries in Fig. 5. The maximum number of sides of the etched cavities must be between 3–6.

These examples show that the information, added to the mask layout by the crystallographic orientation, is substantial. Convex corners are fully etched away [see Fig. 5(A2)] and

cavities formed by two individual mask windows can fuse into one large cavity with much mask underetch [see Fig. 5(C2)]. A hexagonal equilateral triangular shape or diamond shapes built out of equilateral triangles, aligned along the crystal orientations, will show no mask underetch such that closely spaced matrices can be built with thin $\langle 111 \rangle$ -oriented plates are obtained [see Fig. 5(D2)]. In Fig. 6, the result is shown of etching with a mask, consisting of a matrix of circular windows, with different orientations to the crystal. At certain orientations, mask underetch will occur. At the bottom of the etched hexagonal cavities, etch steps are still visible, although a part of the bottom is ready smoothed. The given rules and examples also hold for fabrication methods where a pre-etch step is used, as will be discussed later.

IV. PRE-ETCHING WITHOUT WALL COATING

Applying a noncrystal orientation-dependant etching technique prior to anisotropic wet chemical etching increases the number of possible geometries. Hereafter in this paper, we will refer to this as a “pre-etch step” [24] although different nonetching methods can be used for this, such as mechanical and laser drilling [7], sawing, melting [12], [13], and powderblasting. We used cryogenic deep reactive ion etching (DRIE) [14], [15]. The complete process consists of successively patterning the KOH etch mask (siliconnitride, SiN) and the DRIE resist mask, followed by DRIE, resist stripping, KOH etching, and SiN stripping.

In Fig. 7 a three-dimensional art impression is made of the cavity that arises when anisotropic etching with a hexagonal mask is preceded by a pre-etch step with varying aspect ratios and at different depths with zero off-axis cut.

The situation shown in Fig. 7(A1) will not etch since the wafer plane is perfectly oriented with the $\{111\}$ crystal plane and no pre-etching is applied. Fig. 7(A2)–(A5) show that the “pre-etch” depth (dotted lines) determines the depth of the final etched cavity. The cross sections of the structures are given for the plane AA'. For BB' and CC', the cross sections are similar. For different depths, the white areas [see Fig. 7(A)] within the hexagonal shape define the boundaries of the DRIE etch mask to

Fig. 7. Cavity shapes, existing after pre-etching (dotted lines) at different depths and with different aspect ratios, defined at the wafer surface by a hexagonal mask for zero off-axis wafer cut. (A) Top view and mask geometry. (B) Cross-sectional AA'. (C) Three-dimensional art impression. The limiting situation (infinitely large aspect ratio of the pre-etch hole) is given in (C5). In this situation, a sharp point exists bounded by three $\{111\}$ -planes.

get geometries of the alternating $\{111\}$ sidewalls. Hence, a combination of the position, aspect-ratio, and depth of the pre-etch cavity relative to the mask determines the resulting anisotropically etched cavity shape. In case the pre-etched cavity has an infinitely small diameter, which is exactly positioned, as shown in Fig. 7(A5), the octahedron structure will change into a heptagon with one sharp tip. The bottom $\{111\}$ -plane has vanished.

In contrast to the situations of Fig. 5, where only mask underetching occurs due to mask-to-crystal alignment, the application of pre-etching also initializes the effects of the out-of-the-wafer-oriented $\{111\}$ -planes on the etched geometry. The outward-directed $\{111\}$ -planes create a wider cavity than defined by the mask. Now, the mask orientation in combination with the pre-etched cavity size and depth determines the obtained structure. In Fig. 8, three typical situations are shown for a pre-etch cavity size according to Fig. 7(3).

To get insight in designing structures with the pre-etch step, we will only focus on an extruded two-dimensional trench structure of the situation sketched in Fig. 7(A2). As shown in Fig. 8(A) and (B), the effect of the size and position of the mask relative to the initial trench can be of importance for the mask underetch. In Fig. 8(C), the underetch depends on the depth of the pre-etched trench and by using two mask windows, freestanding structures over adjustable channel depths can be

obtained. Also, rotation of the mask to create a mask-to-crystal misalignment can be used for this.

To design geometries, which are bounded by the KOH-etch mask, the depth has to first be defined. The second step is to determine the bottom width of the “pre-etch” at that specific depth and add a margin. The last step is to calculate the width of the mask opening and the position relative to the “pre-etch” structure. Notice that the roughness of the shape geometry of the initial trench will not play a role in the definition of the anisotropically etched sidewalls. After anisotropic wet chemical etching, all walls will be bounded by $\{111\}$ -planes.

By etching two trenches and using two mask openings, thin $\langle 111 \rangle$ -oriented membranes can be fabricated. Provided that the mask is precisely aligned with respect to the crystal orientation, the thickness of these membranes can be less than $1\ \mu\text{m}$. In Fig. 8(D), an example of a cross section of such a membrane, anodically bonded at one side to a glass wafer is shown. The orientations of these membranes will be along the in-plane-oriented $[1\ 0\ \bar{1}]$, $[\bar{1}\ 10]$ and $[0\ \bar{1}\ 1]$ directions (see Fig. 2). The membranes can be used as in-plane deflecting membranes for creating check valves by selectively bonding to, for example, glass, as was demonstrated with $\langle 100 \rangle$ wafers in [16], [15], and [18], although the steep angle of 70.5° is disadvantageous compared to the 54.7° -oriented membranes in $\langle 100 \rangle$ silicon.

Fig. 8. (A)–(C) Results of the mask positioning relative to the pre-etched cavity and the cavity depth on the final geometry. (D) Layout and SEM pictures of a thin $\langle 111 \rangle$ -oriented membrane. The dotted line indicates the pre-etched trench. In the lower SEM picture, a glass wafer is anodically bonded on top. The off-axis cut is clearly observed by the stepped bottom surfaces.

Fig. 9. Top view of typical etch cavity shapes for equilateral triangular mask windows for two different orientations and a circular mask window, respectively. The pre-etch cavity is indicated by the dotted circle and etched down to a depth according to the situation of Fig. 7(3). Due to a combination of inward- and outward-directed $\{111\}$ -planes, the bottom shape becomes hexagonal for situation (B) and (C). In case of situation (A), the triangular cavity is bounded by inward directed planes only.

The $\langle 111 \rangle$ -oriented membranes can be etched along the six sides of the hexagon, three inward and three outward oriented. Notice the effects of the mask layout and orientation on the orientation of the planes, as illustrated in Fig. 9. Designing the plates such that a hexagon is formed with membranes at all sides, outer corners will arise such that the structures are etched

Fig. 10. Three-dimensional impression of a matrix of three seven-hexagonal-shaped anisotropically etched cavities, respectively.

Fig. 11. (A) Inside etched and (B) outside etched geometries, respectively. For situation (B), a time stop is needed in order not to underetch the posts. Fig. 1(A) and (B) show the mask layouts, Figs. 2–6 show cross sections.

away. Though with a combination of several closely spaced hexagons, rotated equilateral triangles or diamonds built with two equilateral triangles, stable thin walled structures are obtained such as the designs shown in Fig. 10.

V. PRE-ETCHING WITH SIDEWALL COATING

Passivation of the sidewalls of an etched structure further increases the possibilities, as shown by Ensell [19], Chou *et al.* [20], Lee *et al.* [21], Park *et al.* [22], and Fleming [23]. By subsequently directional etching a structure into $\langle 111 \rangle$ silicon, passivating the trench with an anisotropic etching solution resistant material (SiN , SiO_2), removing the passivation layer at the trench bottom, and deepening the silicon with a second mask, anisotropic wet chemical etching will not attack the passivated structure from below since this structure is protected by a $\{111\}$ -plane. After stripping the passivation layer, monocrystalline freestanding structures like cantilever beams [19] or suspended plate structures [20] remain. The structure height is limited by the photoresist step coverage at the second lithography step. To overcome the lithography problems, Fleming [23] proposed a one-mask process, which makes the process suitable for directionally etched high-aspect-ratio structures. The key step is a mask-less removal of the passivation layer at the bottom of the etched trenches and successive silicon etching. Released structures up to $\sim 25\text{-}\mu\text{m}$ thickness, and an underetch gap of $\sim 10\text{ }\mu\text{m}$ were realized in this way.

There are two ways to release monocrystalline structures: 1) by undercutting, based on unstable convex corners or 2) by using the smart mask aligning procedure, as discussed

Fig. 12. SEM photographs showing the silicon underetching in $\langle 111 \rangle$ -oriented wafers applied to spring element for micro check valves. (A) Circular plate suspended by straight beams, which are underetched only when a proper crystal (mis-)alignment is used. The spiral shaped beams of (B) will always be released. When the three beams are oriented along the $\{111\}$ -planes, no underetching might occur, as shown in (C). (D) Zoom-in of the slight negative tapering during DRIE and the resulting rim of mask material due to the shadow effect.

in Sections III and IV. With these techniques, “mask” under etching occurs by considering the passivated monocrystalline silicon structure now as an etch mask, as illustrated in Fig. 11. For convex-bounded cavities, the under etching will stop at the out-of-plane-oriented $\langle 111 \rangle$ -planes [see Fig. 11(A)], whereas cavities etched from the outside in the inward direction will fully remove the silicon since the convex corners will be attacked such that a time stop is needed. This situation occurs for free-hanging structures suspended by posts, as shown in Fig. 11(B).

With the etching method of Fig. 11(A), different devices have been made. In Fig. 12(A) and (B), suspended plates are shown, which form the spring structures of check valves. The outer geometry of the gap is bounded by six alternating $\{111\}$ -planes. Since the underetch depth can be determined by the second directional etch step, the spacing and, thus, the amount of volume in the valve, can be varied. The spiral-shaped valve element will always be released, whereas straight beams will not be etched under when they are aligned along the $\{111\}$ -planes. This situation is shown in Fig. 12(C) and (D). Notice the fact that, for this situation, underetching of the circular plate will proceed until silicon walls have been formed along the intersection lines of the three beams. These beams and circular plate can only be etched free when the second DRIE etch step is deeper than the

beam width times $\tan(70.5^\circ)$. With DRIE, a high-aspect ratio mono-crystalline silicon structure can be created with a profile that is tuned to give a slightly negative tapered structure. After applying the passivation layer, a mask-less directional trench bottom etch step and anisotropic wet chemical etching, an overhanging mask roof at the bottom of the initial trench is created due to the shadow effect [see Fig. 12(D)].

From this figure, a clear wafer mis-cut and the resulting running steps at the right-hand side are also observed. Longer etching times will remove these steps, resulting in a smooth surface. This mis-cut also gives rise to a thickness tapering of the machined structures since the off-axis-oriented bottom $\{111\}$ -plane will be auto-polished as well. To overcome this problem, perfectly cut wafers need to be applied or an auto-polishing step must be introduced. The latter method is used to fabricate a thin monocrystalline high-precision plan-parallel double-sided clamped beam with low surface roughness to be used as an optical waveguide [25]. Fig. 13 shows such a beam of $1\text{-}\mu\text{m}$ thickness and the applied process scheme.

The advantage of this method over the sacrificial layer etching techniques is the freedom in choosing the structure height and underetch depth (“sacrificial layer”) during processing, as in micromachining techniques such as SCREAM [26]. Since SCREAM uses an isotropic etch step to release

Fig. 13. Process scheme and SEM photographs of a monocrystalline high-quality plan-parallel silicon waveguide. After the auto-polishing step, pre-etching with wall protection is used to underetch the silicon beam.

Fig. 14. Cross sections of contours etched at different levels in the wafer using wall protection and under-etching. The SEM photograph shows the most elementary membrane structure identical to (A1). The plates of (A2), for example, have a triangular shape. In order to gain a large underetch distance, wide slits can be pre-etched.

the structures, this technique can only be applied when narrow structures need to be underetched. At large underetch widths, long etching times are needed and, thus, much thickness variation will occur. Thus, the attainable geometry size is strongly limited by this process. The $\langle 111 \rangle$ pre-etching method with wall coating, however, allows free etching of large structures without dimensional constraints.

The process of underetching can be applied to two sides of the wafer to obtain plan-parallel buried membranes. In Fig. 14, an SEM photograph of a cross section is shown. The process of etching, passivation, deepening, and anisotropic under etching can be performed several times on different depth levels to obtain a series of stacked membranes. Design possibilities with stacked structures are shown schematically in this figure as well.

A last, but very important application is for researching anisotropic wet chemical etching. As discussed at the begin-

Fig. 15. Steps, which are initiated at the mask interface travel along the surface until the out-of-plane-oriented $\{111\}$ -planes are reached. The membrane is fully surrounded by $\{111\}$ -planes due to which it becomes independent of nucleation of steps at the mask. Only after all silicon above and below the membrane have been removed, the etching speed of the $\{111\}$ -planes of the membrane becomes dependent of the step nucleation at the mask interface.

ning of this paper, steps are initiated and etched along the $\{111\}$ -planes. After cavities have been etched, bounded by

the $\{111\}$ -planes, the net etch velocity in the $\langle 111 \rangle$ direction is defined by the step nucleation. Nucleation at the mask interface can be an important mechanism [10]. Elimination of this mechanism in the observed etch process is necessary. This can be achieved with the membrane design, shown in the SEM photograph of Fig. 14. After all steps are etched from the membrane, the membrane edges are fully bounded by $\{111\}$ -planes. Nucleated steps at the mask interface will travel along the inner sides of the overhanging parts of the cavities, as shown in Fig. 15, but stop at the out-of-the-wafer-oriented $\{111\}$ -planes. Only after the top and bottom layers are fully etched away, the $\{111\}$ -planes of the membrane will be etched. Thus, the membrane design can be used for researching the etch behavior without the influence of step nucleation at the mask interface over a long period of time.

VI. CONCLUSIONS

Monocrystalline $\langle 111 \rangle$ -oriented wafers are rarely used for microsystem applications since the $\langle 111 \rangle$ -oriented top and bottom planes slowly etch in an anisotropic etching solution. However, when using the off-axis cut and pre-etch steps, many new interesting possibilities arise additionally to conventional etching techniques, such as in $\langle 100 \rangle$ silicon. One of these interesting aspects is the possibility to obtain high-quality monocrystalline plan-parallel beams and membranes with low surface roughness, which are very useful in optical applications.

To process the $\langle 111 \rangle$ wafers, the wafer off-axis cut or an isotropic or directional pre-etch can be utilized. With these techniques combined with a smart mask to crystal positioning, underetching can be obtained or prevented. Different examples and design "tricks" have been demonstrated such as thin plan-parallel beams, stacked membranes, in-plane deflecting membranes, mask bridges, and check valve suspensions. We believe the possibilities of $\langle 111 \rangle$ wafers in microsystem technology are underestimated, but this wafer type offers much potential for constructing high-quality monocrystalline silicon structures.

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