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# Determination of precise crystallographic directions on Si{111} wafers using self-aligning pre-etched pattern

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**Abstract**

Silicon wet anisotropic etching based bulk micromachining technique is widely used for the fabrication of microelectromechanical systems components. In this technique of microfabrication, alignment of mask edges with crystallographic directions plays a crucial role to avoid unwanted undercutting to control the dimensions of fabricated structures. Various kinds of pre-etched designs have been reported to identify the crystallographic directions (e.g. ⟨110⟩ and ⟨100⟩) on Si{100} and Si{110} wafer surfaces. To the best of our knowledge, no pre-etched design has been reported to identify crystal directions on Si{111} wafer. In this work, a self-aligning technique based on pre-etched patterns has been investigated to precisely determine the ⟨110⟩ direction on Si{111} wafer surface. In this technique, a set of circular shape mask patterns close to wafer edge are etched for the identification of ⟨110⟩ direction. On wet anisotropic etching these patterns transform to hexagonal shapes. The notches of hexagonal patterns align precisely along a straight line only when they lie on exact ⟨110⟩ direction. The self-aligned notches can easily be identified by visual inspection using an optical microscope. The major advantages of this technique are simplicity, precision, and self-alignment. In addition, the pre-etched patterns at the wafer periphery occupy very less place.

**Keywords:** Silicon, Wet bulk micromachining, Alignment, Wet anisotropic etching, Si{111}

## Introduction

Micromachining is an integral part of micro/nanofabrication techniques for the formation of micro/nano-electromechanical systems (M/NEMS). Wet anisotropic etching, which is low cost and best suitable for batch process, is a well-established technique in silicon bulk micromachining [1–8]. Potassium hydroxide (KOH) and tetramethylammonium hydroxide (TMAH) are most commonly used etchant in wet anisotropic etching [9–17]. In wet anisotropic etching, the sidewalls of the stable etched profile are formed by {111} planes. In all kinds of wet anisotropic etchants {111} planes exhibit slowest etch rate and therefore the etch selectivity between {111} and non-{111} planes in aqueous alkaline etchants is utilized to fabricate microstructures. The exposure of the number of {111} planes and their angle with wafer surface during etching depend on the orientation of wafer surface. In the case of Si{100} wafer, four {111} planes emerge during etching along ⟨110⟩ directions and make an angle of 54.7° with wafer surface, while on Si{110} wafer {111} planes expose along six directions in which two slanted (35.3°) at ⟨110⟩ directions and four perpendicular at ⟨112⟩ directions [2, 18]. Hence the etching of any arbitrary shaped mask opening on Si{100} and Si{110} wafers results in rectangular and hexagonal shape cavities, respectively.

Figure 1a shows the stereographic projection of {111} silicon. The {111} planes projected from the top and bottom hemispheres are shown by solid (●) and open (○) circles, respectively. Figure 1b presents the ⟨110⟩ directions at which {111} planes appear during wet anisotropic etching process. Hexagonal contour formed by the intersection of ⟨110⟩ directions is exhibited in Fig. 1c. The orientation of ⟨110⟩ directions on Si{111} wafer surface is illustrated in Fig. 1d. These {111} planes represent 6 of 8 {111} planes and the others being the top and bottom

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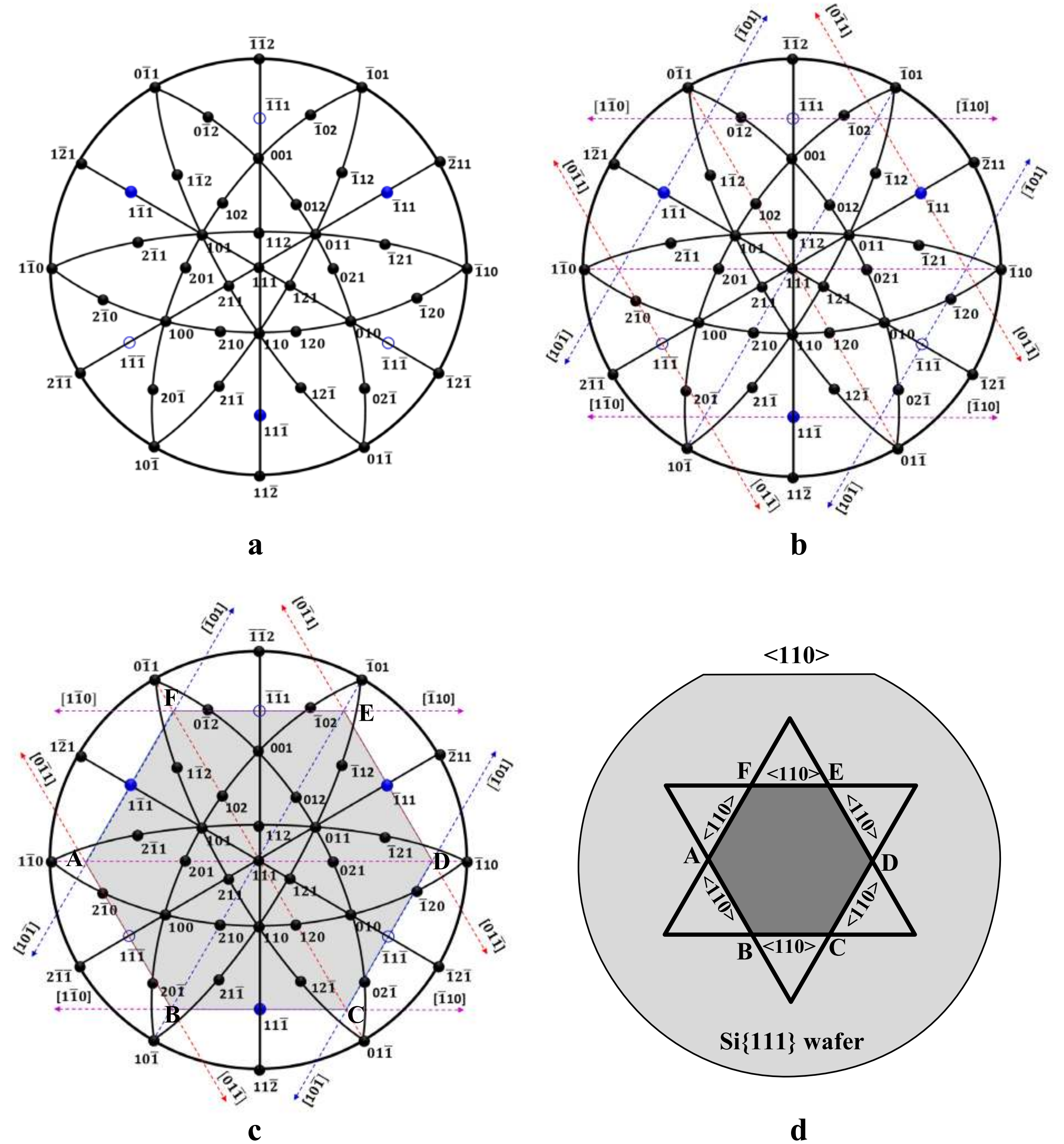



**Fig. 1** **a** Stereographic projection of {111} silicon. The {111} planes projected from the top and bottom hemispheres are shown by solid (●) and open (○) circles, respectively. **b** ⟨110⟩ directions at which {111} planes appear during wet anisotropic etching process. **c** Hexagonal contour formed by the intersection of ⟨110⟩ directions at which {111} planes emerge. **d** ⟨110⟩ direction at Si{111} wafer surface with primary flat oriented along ⟨110⟩ direction. Three {111} planes (●) are 109.5° to the surface plane and are 60° to each other, while another three {111} planes (○) are 70.5° to the surface plane and 60° to each other

planes. Three {111} planes (●) are 109.5° to the surface plane and 60° to each other, while another three {111} planes (○) are 70.5° to the surface plane and 60° to each other. In other words, six {111} planes on Si{111} surface are tilted at ± 19.5° from the vertical. Hence in the case of Si{111} wafer, six {111} planes expose at ⟨110⟩ directions which form a hexagonal shape. Figure 2 presents wet anisotropically etched profiles of different shapes of

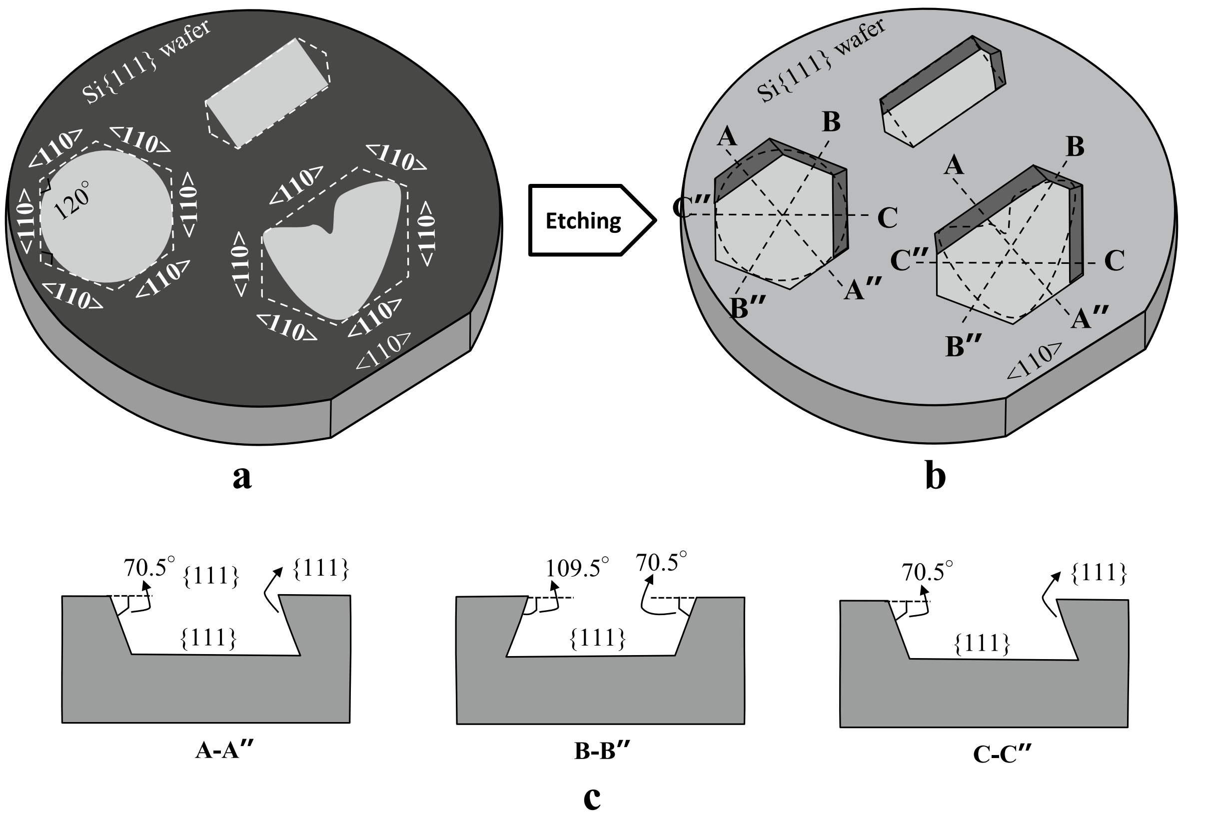



**Fig. 2** Schematic representation of the wet anisotropically etched profiles of different shapes mask geometries on Si{111} wafer: **a** mask pattern on wafer surface, **b** etched profile after wet anisotropic etching, and **c** cross sectional view of etched profiles. Dashed lines in **a** indicate the directions where wet etch will terminate due to the appearance of {111} planes

mask patterns. Three {111} planes out of six are slanted at 70.5° with wafer surface, while other three make an angle of 109.5° to the wafer surface. Among three principle orientations namely {100}, {110} and {111}, {100}-oriented wafers are most frequently used. Si{110} wafers are employed for specific applications such as microstructures with vertical sidewalls. As the Si{111} planes have slowest etch rate in all kinds of wet anisotropic etchants, therefore Si{111} wafers are used for specific applications and to fabricate complicated structures using deep reactive ion etching (DRIE) assisted wet anisotropic etching [19–34]. In these structures, gap between freestanding structure and bottom surface can be controlled precisely.

In wet anisotropic etching, least lateral undercutting takes place at the mask edges where {111} planes expose. Therefore, any arbitrary shape mask opening in prolonged wet anisotropic etching results in a microstructure bounded by {111} oriented sidewalls. Hence the alignment of mask edges with crystallographic direction in silicon wet bulk micromachining is utmost important to control the dimensions of microstructures. Figure 3 shows the effect of misalignment on the dimensions of

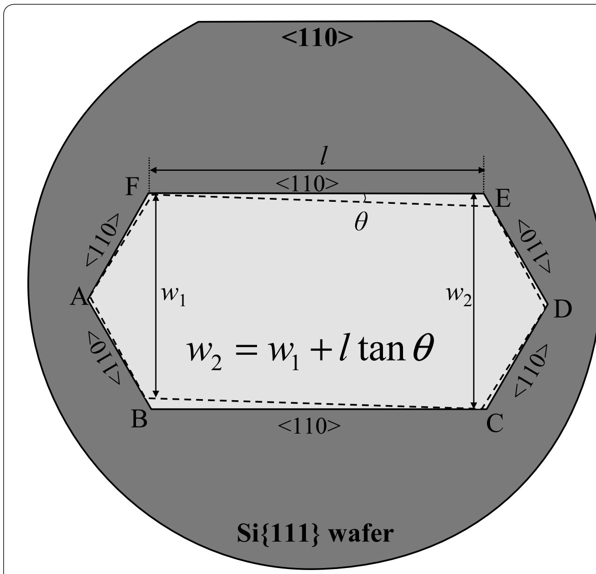



**Fig. 3** Effect of misalignment on the size of the structure fabricated using wet anisotropic etching on Si{111} wafer. θ is the misalignment angle of mask edge from ⟨110⟩ crystallographic direction. Dashed lines indicate the edges of original hexagonal shape mask pattern

resultant structure fabricated using wet anisotropic etching. Silicon wafer manufacturers provide the orientation (i.e. crystallographic direction) of wafer flat. Hence the primary flat of silicon wafer is commonly used as reference for all kinds of wafer orientation. Although wafer manufacturers typically specify the misalignment of primary flat from accurate crystallographic directions to 1°, it could be larger or smaller than this value. Thus, the wafer flat is not sufficient to use as reference direction to precisely control the dimensions of microstructures fabricated using wet anisotropic etching based bulk micromachining. Hence the precise determination of crystallographic direction is desirable to avoid unwanted undercutting at mask edges due to misalignment from crystallographic direction. Precise crystallographic direction on silicon wafer can be identified by X-ray diffraction method by mounting X-ray diffraction unit on a mask aligner. However, this method is expensive and makes mask aligner very bulky. In addition, pre-etched patterns formed by wet anisotropic etching are employed for precise determination of crystallographic direction [35–44]. Various kinds of pre-etched designs have been reported to find out the precise crystallographic direction on {100} and {110} orientations as these orientations are mostly used to fabricate MEMS structures. To the best of our knowledge, no pre-etched design is reported for Si{111} wafer to find the precise crystallographic directions.

In this paper, we have investigated a pre-etched method to find accurate $\langle 110 \rangle$ crystallographic direction on Si{111} wafer. The proposed method does not require any measurement to determine the precise direction. The pre-etched patterns are designed in such a way that the notches of these patterns after wet anisotropic etching align with each other along precise $\langle 110 \rangle$ direction.

## Design details

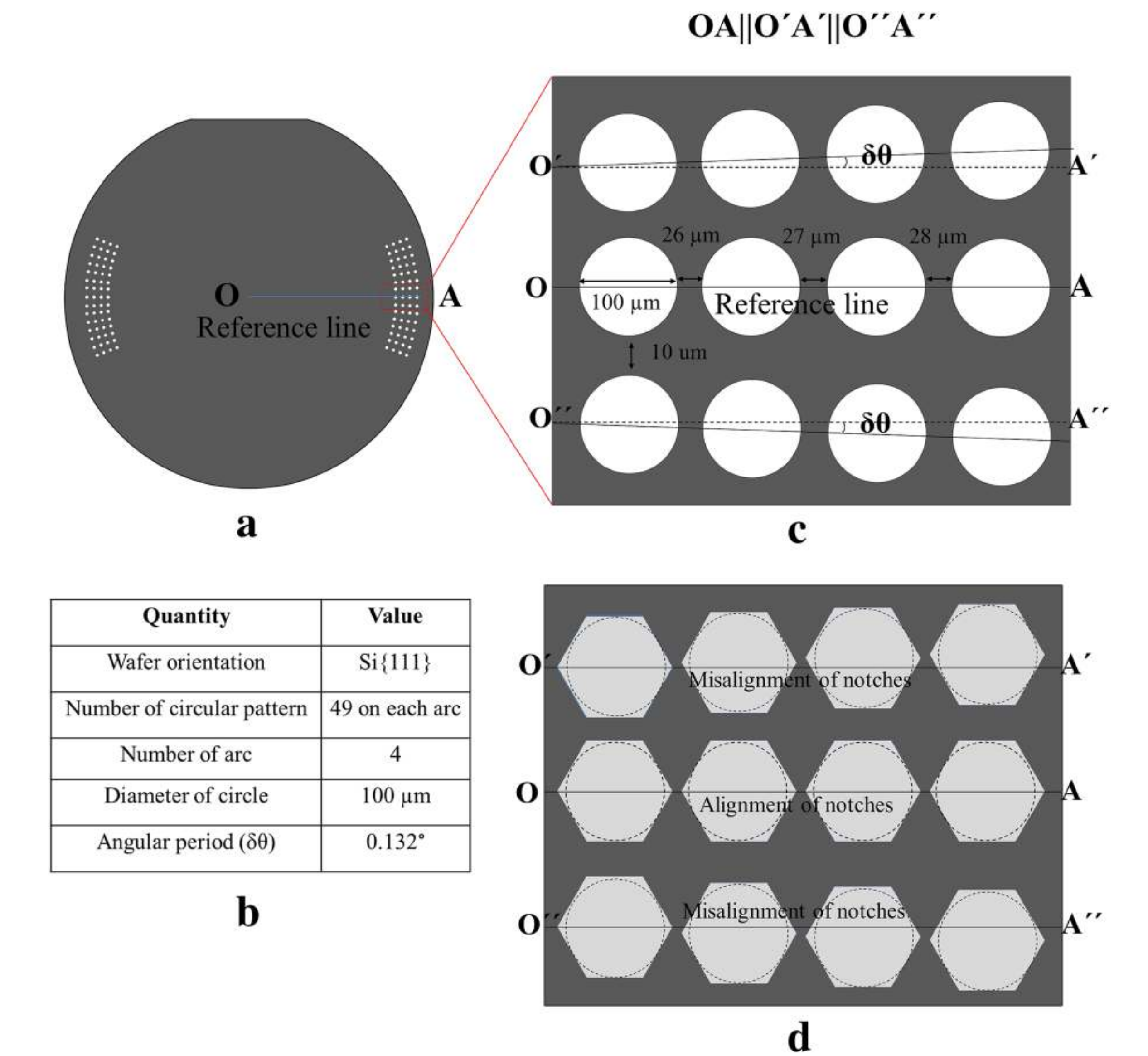
To design pre-etched pattern, 100 µm diameter of circular openings pattern with four concentric arcs are used. The number of circles in each arc is chosen based on the inaccuracy of the wafer flat. In present experimental study, 49 circles in each arc are incorporated. Lines passing through four radially arranged circles intersect at centre ('O') of the wafer. Angle of tilt with neighbour sets of four radial circles (along OA) is $\delta\theta$ and shown in Fig. 4. The dimensions of the proposed pre-etch pattern are presented in Fig. 4b. Optical image of etched profile of pattern on oxide layer is shown in Fig. 5. The primary flat of the wafer is used as a reference direction to transfer the pre-etched pattern on the wafer surface. For getting simple visual investigation, we selected angular period ($\delta\theta$) of 0.132° with same diameter (100 µm) circles. In order to obtain the better accuracy, the spacing of circular patterns is calculated in such a way so that the notches of hexagon patterns should emerge close to each other, but should not merge with each other. Closer spacing of notches enables to identify most accurately aligned notches to determine most precise $\langle 110 \rangle$ direction. Figure 4 presents the schematic diagram of pre-etched pattern before and after etching. The line OA passes through the notches of all the four radial hexagons indicates precise alignment of notches with each other. The lines O′A′ and O″A″ illustrate the misalignment of notches. The precise alignment of notches along a direction represents $\langle 110 \rangle$ direction, while the misalignment of notches indicates non- $\langle 110 \rangle$ directions.

## Experimental details

P-type doped (boron) Czochralski-grown Si{111}wafers with 5–10 Ω-cm resistivity of 4-in. diameter are used. Silicon dioxide layer of 1 µm thickness grown by thermal oxidation method (Supplier: MicroChemicals GmbH) is used as a mask to protect unwanted places from etching. Simple and low cost wet anisotropic etching process has been used to obtain pre-etched pattern of hexagonal shape on wafer surface. The process starts with coating of positive photoresist on oxidized silicon wafer using spinning method. Thereafter mask patterns are transferred on photoresist using photolithography process followed by oxide etching in buffered hydrofluoric (BHF) acid. The wafer is then rinsed in running de-ionized (DI) water. Subsequently, photoresist is removed using acetone followed by thorough rinse in DI water. Now the wafer is cleaned in piranha bath ($H_2O_2$:$H_2SO_4$::1:1) to remove organic impurities on the wafer surface. This step is followed by DI water rinse. In order to remove the oxide layer grown during piranha bath, wafer is dipped in 1% HF for 30 s followed by rinsing in DI water. After this step, wafer is transferred in 25 wt% TMAH (99.99%, Alfa Aesar) to achieve hexagonal shape etched profiles as presented in Fig. 2. TMAH exhibits high etch selectivity between silicon and silicon dioxide and therefore used as an etchant. Silicon etching process is performed at $70 \pm 1$ °C. This temperature is achieved by heating a solution in constant temperature water bath. The etch rate of Si{111} at this temperature in our experiment was around 0.6 µm/h. The etching process is continued until the circular mask openings take the hexagonal shape with sharp corners. In order to analyse the pre-etched patterns, optical photographs are taken using 3D measuring laser microscope (Olympus, OLS4000).

## Results and discussion

As discussed earlier, in wet anisotropic etching the circular mask openings on {111} surface take the hexagonal shape with sharp corners. Figure 5 shows the optical images of mask pattern in oxide layer and the




| Quantity | Value |
|---|---|
| Wafer orientation | Si{111} |
| Number of circular pattern | 49 on each arc |
| Number of arc | 4 |
| Diameter of circle | 100 μm |
| Angular period (δθ) | 0.132° |

**Fig. 4** Schematic view of pre-etched patterns in oxide layer **a** on Si{111} surface with **b** quantitative details. Zoomed-in view of pre-etched pattern **c** before and **d** after wet anisotropic etching. The line OA indicates where the centres of all circles lie on ⟨110⟩ direction and therefore the notches of hexagon pattern perfectly aligns along ⟨110⟩ direction. The lines O′A′ and O″A″ are parallel to OA indicate the misalignment of the notches when the centres of all circles do not lie on ⟨110⟩ direction

etched profile with 3 μm depth after wet anisotropic etching. As Si{111} is a slowest etch rate plane in wet anisotropic etching, there is an obvious question about the formation of hexagon pattern on the surface of Si{111} wafer if only wet anisotropic etching is used. In other words, how does hexagonal shape develop on wet etching of a circular pattern on Si{111} surface? There are two ways to explain the formation of hexagon pattern on the etching of a circular pattern. First explanation is based on the off-axis cut of silicon surface. It is not possible to get the wafer surface perfectly parallel to the crystal orientation. Commercial standard wafers usually are cut ± 0.5 off-axis. Although the wafers of more precise orientation can be ordered, these can never be cut perfectly parallel to crystal orientation at the atomic scale. So when the circular pattern is etched in wet anisotropic etchant, crystal planes other than {111} orientation expose at the mask edges that lead to lateral etching under the mask layer and proceed till it finds the ⟨110⟩ directions where {111} planes appear.

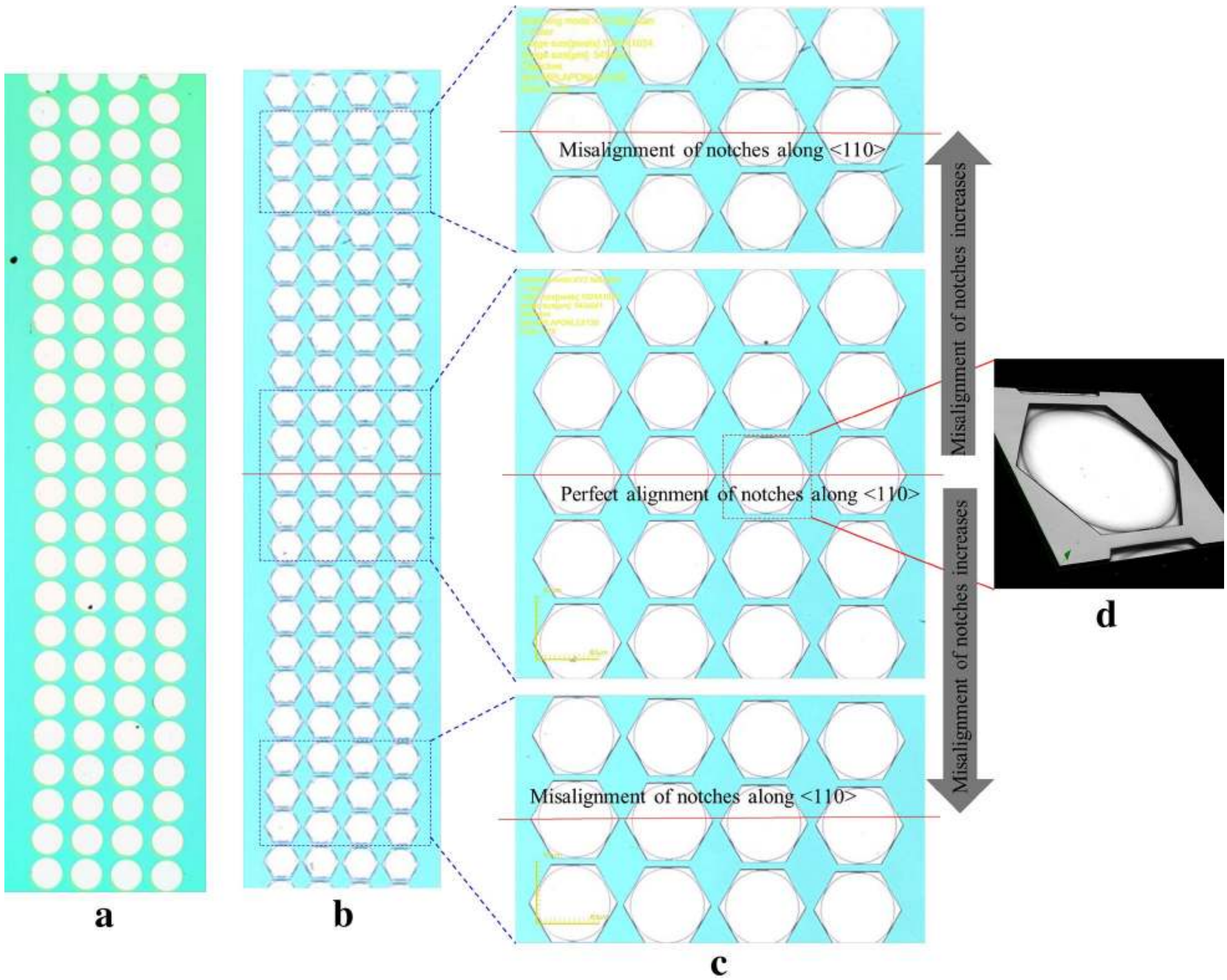



**Fig. 5** Optical images of the pre-etched pattern on Si{111} surface: **a** before etching, **b** after etching, **c** zoomed-in view to show the perfectly aligned and misaligned notches of hexagon patterns and **d** zoomed-in view of hexagon structure. The notches of the four radial hexagons align in a straight line along the precise ⟨110⟩ direction, while the misalignment of notches increases above and below the precise ⟨110⟩ direction

The lateral undercutting under the mask layer of circular pattern results in hexagon shape pattern. In the second case, if the surface of silicon wafer is perfectly parallel to {111} crystal orientation, how will hexagon shape form on the etching of circular mask pattern? In this situation, removal of the first layer of surface atoms will expose non-{111} planes at the mask edge of the circle that will result in lateral etching. The lateral etching will end up when it encounters {111} planes which appear at ⟨110⟩ directions and these directions form hexagon shape on {111} surface.

In order to find out the accurate ⟨110⟩ direction, the optical image shown in Fig. 5b is inspected. If the mask patterns are transferred in mask layer without any mask edge distortion (i.e. fabrication error), all notches of four hexagons along radial direction come in a straight line i.e. ⟨110⟩ direction. In addition to fabrication error, crystal defects may affect the alignment of the notches of hexagon pattern. In the pre-etched pattern, we should find a set of four hexagons whose notches along radial direction lie on a straight line most accurately. It can easily be noticed in Fig. 5 that the notches of pre-etched hexagon patterns misaligned above and below the set of most accurately aligned hexagons. The notches of the four radial hexagons align to each other at precise crystallographic direction and the misaligned sets of radial hexagons can simply be identified with closer visual inspection using optical microscope. We can say that this method does not require any measurement to identify ⟨110⟩ direction. It is completely free

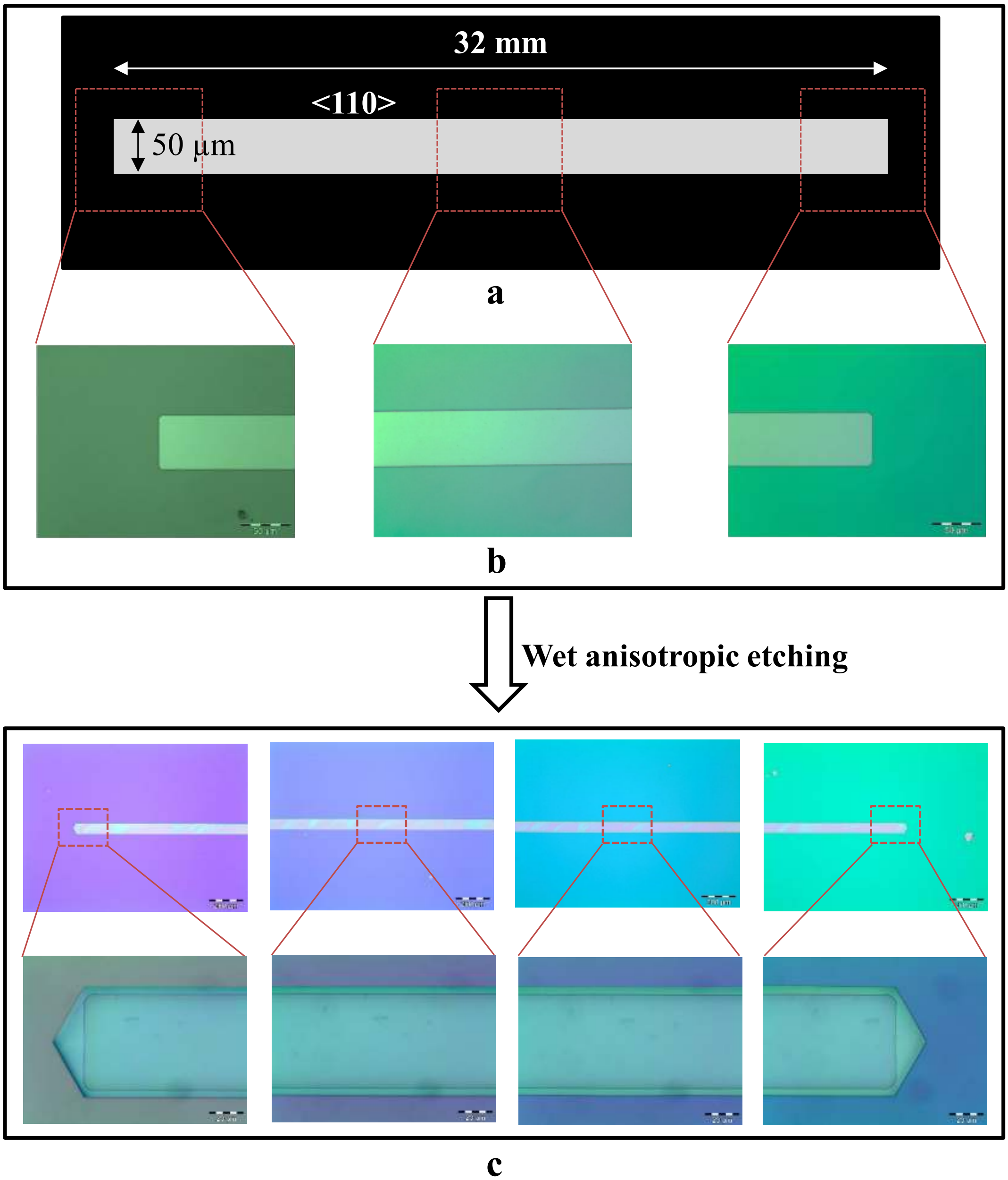



**Fig. 6** A 50 µm wide and 32 mm long rectangular shape mask opening with longer edge aligned along the ⟨110⟩ direction on {111} wafer surface to test the accuracy of proposed method: **a** schematic diagram, **b** optical image of the mask pattern after oxide etching, **c** etched profile with zoomed-in images of different portions. Pre-etched patterns are used as reference for the precise alignment of the mask edges along ⟨110⟩ direction. The undercutting at the mask edges takes place due to finite etch rate of {111} plane and the uniform undercutting indicates the accurate alignment of mask edges along ⟨110⟩ direction

from measurements and therefore called a self-aligned technique in the sense of the self-alignment of notches along ⟨110⟩ crystallographic directions on Si{111} wafer.

## Accuracy of the technique

In silicon bulk micromachining based on wet anisotropic etching, the mask edges comprising {111} planes (e.g. ⟨110⟩) exhibit least undercutting. If the mask edges are precisely aligned along ⟨110⟩ direction, uniform undercutting takes place at the mask edges. To demonstrate the accuracy of the proposed method, a rectangular shape mask opening, as presented in Fig. 6, is patterned in oxide layer on Si{111} wafer. In order to align the mask edges along ⟨110⟩ crystallographic direction, most precisely aligned notches of pre-etched hexagon patterns are used as the reference. After transferring the pattern in oxide layer, etching is performed in 25 wt% TMAH for 8 h to observe the undercutting at ⟨110⟩ mask edges, which occurs due to the finite etch rate of {111} planes. This undercutting is measured at different locations along the longer edges of the mask pattern. As can be observed in Fig. 6, the undercutting at mask edge is measured to be uniform, which indicates that the mask edges are precisely aligned along ⟨110⟩ direction. Thus, the proposed method to determine the ⟨110⟩ direction on Si{111} wafer is accurate and can effectively be used to fabricate microstructures with high dimensional accuracy.

In general, the accuracy of this technique depends on the angular separation between the groups of four circles and how precisely one identifies the set of hexagons whose notches are most precisely aligned along a line. In addition, when the alignment notches look equally good (or bad) for two contiguous rows of circles, the radial direction half way between them should be selected. If an experimentalist chooses one row (of four circles) above or below the best aligned notches of the pre-etched hexagon patterns, it will add the alignment error equal to angular separation. Hence the accuracy of the alignment is limited by the ability to find out the best aligned hexagon patterns and angular spacing between the groups of four circles.

Most notably, this method provides best control on the dimensions of fabricated structure if one uses most precisely aligned notches of pre-etched hexagon patterns as reference ⟨110⟩ direction for the alignment of subsequent mask patterns. In order to improve the identification of most precise crystallographic directions, the size of the circles should be small and must be placed close to each other in such way so that the notches of hexagons can be attained at least separation, but should not merge with each other. It helps to find out the most accurately aligned notches in pre-etched patterns.

## Conclusions

A simple and self-aligned technique is presented to determine the precise ⟨110⟩ direction on Si{111} wafer surface. Circular mask patterns, which are easy to design, are used to get hexagon shapes pre-etched pattern. The notches of hexagonal shape mask patterns align with each other along accurate ⟨110⟩ direction which is identified by visual inspection using an optical microscope. It does not require any measurement to find out the crystallographic direction. In addition, pre-etched patterns occupy very small space close to wafer edge. To the best of our knowledge, it is the first time, a simple and measurement free technique is studied for the identification of crystallographic directions on Si{111} wafer, which is used for the fabrication of special types of microstructures.

**Authors' contributions**

AVNR and VS did experiments. AVNR and PP wrote the manuscript. AKP reviewed/edited the manuscript. All authors read and approved the final manuscript.

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**Competing interests**

The authors declare that they have no competing interests.

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