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Wet anisotropic etching characteristics of Si{111} in NaOH-based solution for silicon bulk micromachining

S. Purohit, V. Swarnalatha, A. K. Pandey and P. Pal* 

Abstract

Silicon bulk micromachining is extensively employed method in microelectromechanical systems (MEMS) for the formation of freestanding (e.g., cantilevers) and fixed (e.g., cavities) microstructures. Wet anisotropic etching is a popular technique to perform silicon micromachining as it is low-cost, scalable, and suitable for large scale batch processing, which are the major factors considered in the industry to reduce the cost of the product. In this work, we report the wet anisotropic etching characteristics of Si{111} in sodium hydroxide (NaOH) without and with addition of hydroxylamine (NH₂OH). 10M NaOH and 12% NH₂OH are used for this study. The effect of NH₂OH is investigated on the etch rate, etched surface roughness and morphology, and the undercutting at mask edges aligned along <112> direction. These are the major etching characteristics, which should be studied in a wet anisotropic etchant. A 3D laser scanning microscope is utilized to measure the surface roughness, etch depth, and undercutting length, while the etched surface morphology is examined using a scanning electron microscope (SEM). The incorporation of NH₂OH in NaOH significantly enhances the etch rate and the undercutting at the mask edges that do not consist of {111} planes. To fabricate freestanding structure (e.g., microcantilever) on Si{111} wafer, high undercutting at <112> mask edges is desirable to reduce the release time. Moreover, the effect of etchant age on the abovementioned etching characteristics are investigated. The etch rate and undercutting reduce significantly with the age of the modified NaOH. The present paper reports very interesting results for the applications in wet bulk micromachining of Si{111}.

Keywords: Silicon, Si{111}, Anisotropic etching, MEMS, NaOH, NH₂OH

Introduction

Wet anisotropic etching is a fundamental process for the fabrication of variety of components in the field of microelectromechanical systems (MEMS) [1–5]. Many kinds of MEMS components (e.g., cantilever, cavity, diaphragm, etc.) are fabricated through wet anisotropic etching-based silicon bulk micromachining of {100}, {110} and {111} oriented silicon wafers for different applications [1–14]. In addition, wet anisotropic etching of Si{111} is utilized to fabricate complex structures using deep reactive ion etching (DRIE) assisted wet etching

[7–12]. The Si{111} wafers can also be used to control the gap between the freestanding structure and the bottom surface [10, 11]. Silicon wet anisotropic etching is usually carried out in alkaline solution, which can be either an organic (e.g., tetramethylammonium hydroxide (TMAH)) or an inorganic compound (e.g., potassium hydroxide (KOH), and sodium hydroxide (NaOH)) [15–32]. TMAH and KOH are comprehensively studied alkaline solutions for silicon wet anisotropic etching. However, limited number of studies have been reported for other kinds of alkaline solutions such as NaOH, ethylenediamine pyrocatechol water (EDP), cesium hydroxide (CsOH), ammonium hydroxide (NH₄OH), Hydrazine, etc. [25–36]. Recently, we reported the etching characteristics of Si{100} and Si{110} in NaOH without and with

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addition NH_2OH . A significant improvement in etching characteristics is observed when NH_2OH is added into NaOH solution [31, 32]. Hence, it is very important to investigate the etching characteristics of Si{111} in NaOH-based solution for application in MEMS.

In all kinds of alkaline solutions, Si{111} planes are the slowest etch rate planes. Hence, the mask edges aligned along a crystallographic direction comprising {111} planes exhibit least undercutting. When an arbitrary mask opening is etched in alkaline solution, severe undercutting takes place at the edges that do not contain {111} planes. The undercutting continues till it encounters {111} planes. To fabricate an etched profile of controlled dimensions, the edges of the mask patterns must be aligned along the directions comprising {111} planes (e.g., $\langle 110 \rangle$ directions on Si{100} and Si{111} surfaces, $\langle 112 \rangle$ and $\langle 110 \rangle$ directions on Si{110} surface). As stated in previous paragraph, Si{111} is an important orientation to fabricate complicated microstructures using wet anisotropic etching or DRIE assisted wet anisotropic etching [10, 11]. Stereographic projection, which is schematically presented in Fig. 1, provides very important information about the crystallographic planes on wafer surface that appear at specific crystallographic directions. Moreover, it is very useful to know the angles between different planes and directions. In Fig. 1, solid blue circle represents the {111} planes projected from the top hemisphere, while open blue circle indicates the {111} planes projected from the bottom hemispheres. {111} planes denoted by solid blue circle make an angle of 109.5° with wafer surface plane and 60° with each other, however the {111} planes depicted by open blue circle form an angle of 70.5° with the top surface of the wafer and 60° with each other as illustrated in Fig. 1. The $\langle 110 \rangle$ crystallographic directions on Si{111} surface, as shown in Fig. 1, comprise {111} planes that emerge during wet anisotropic etching process. Various types of shapes formed by $\langle 110 \rangle$ crystallographic directions at which {111} planes appear during wet anisotropic etching process are described in Fig. 2. These shapes are schematically presented on Si{111} wafer surface in Fig. 3a. Sidewalls of the etched profiles are created by {111} planes when these shapes are etched in an anisotropic etchant as shown in Fig. 3b. The cross-sectional views of the etched profiles are exhibited in Fig. 3c.

In the fabrication of microstructures, etch rate is one of the most important characteristics as it influences the fabrication time, which eventually affects the overall production cost. High etch rate is advantageous for the higher industrial throughput. The salient factors that control the etching characteristics are etching time,

etching temperature, agitation during etching, and the presence of an additive in the etchant. Lately, the influence of hydroxylamine (NH_2OH) on the etching properties of KOH and TMAH is reported [37–43].

In this paper, we present the wet anisotropic etching characteristics of Si{111} in NaOH-based solution. NaOH solution is modified by addition of NH_2OH to alter the etching characteristics. The main objective of the present work is to enhance the undercutting rate at the mask edges aligned along $\langle 112 \rangle$ direction for the fast release of the microstructures such as microcantilever. Moreover, the effect of etchant age on the etching characteristics is methodically investigated.

Experimental details

In this work, Cz-grown p-type doped Si{111} one side polished four-inch wafers with a resistivity of 1–10 Ωcm are used for the investigation of etching characteristics. An oxide layer of 1 μm thickness deposited using thermal oxidation method is employed as mask and/or structural layer. This oxide layer is patterned using photolithography in which positive photoresist (AZ1512HS) is used as mask for selective etching of oxide layer in buffered hydrofluoric acid (BHF). A thorough rinse in DI water is performed once the photoresist is removed using acetone. Subsequently, the wafer is diced into small samples. These samples are cleaned in a piranha bath ($\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2::1:1$) followed by a DI water rinse. A thin layer of oxide is chemically deposited in piranha bath during cleaning process that delays silicon etching process. The cleaned samples are therefore dipped in 1% HF for 1 min to remove chemically grown oxide layer. Thereafter, the samples are thoroughly rinsed in DI water. Now the etching is performed in pure and 12% NH_2OH -added 10M sodium hydroxide (NaOH) at $70 \pm 1^\circ\text{C}$ temperature. 10M of NaOH is selected because this concentration provides the high etch rate [29]. The etching experiments are conducted in 1-L etchant solution. To prepare a 1-L solution of 10M NaOH, 400 g of pellets are dissolved in 1000 ml deionized (DI) water. In the case of 1-L 12% NH_2OH -added 10M NaOH, 400-gm NaOH pellets, 240 ml of 50% NH_2OH and 760 ml DI water are used. To keep the temperature constant during the etching experiment, a constant temperature water bath is used. An etching container made of Teflon is used for the process. The continuous heating of the etchant during etching process changes the concentration of the etchant due to the evaporation of water. Henceforth, a reflex condenser made of thick glass equipped with a double-layered narrow opening is used. A 3D measuring laser microscope

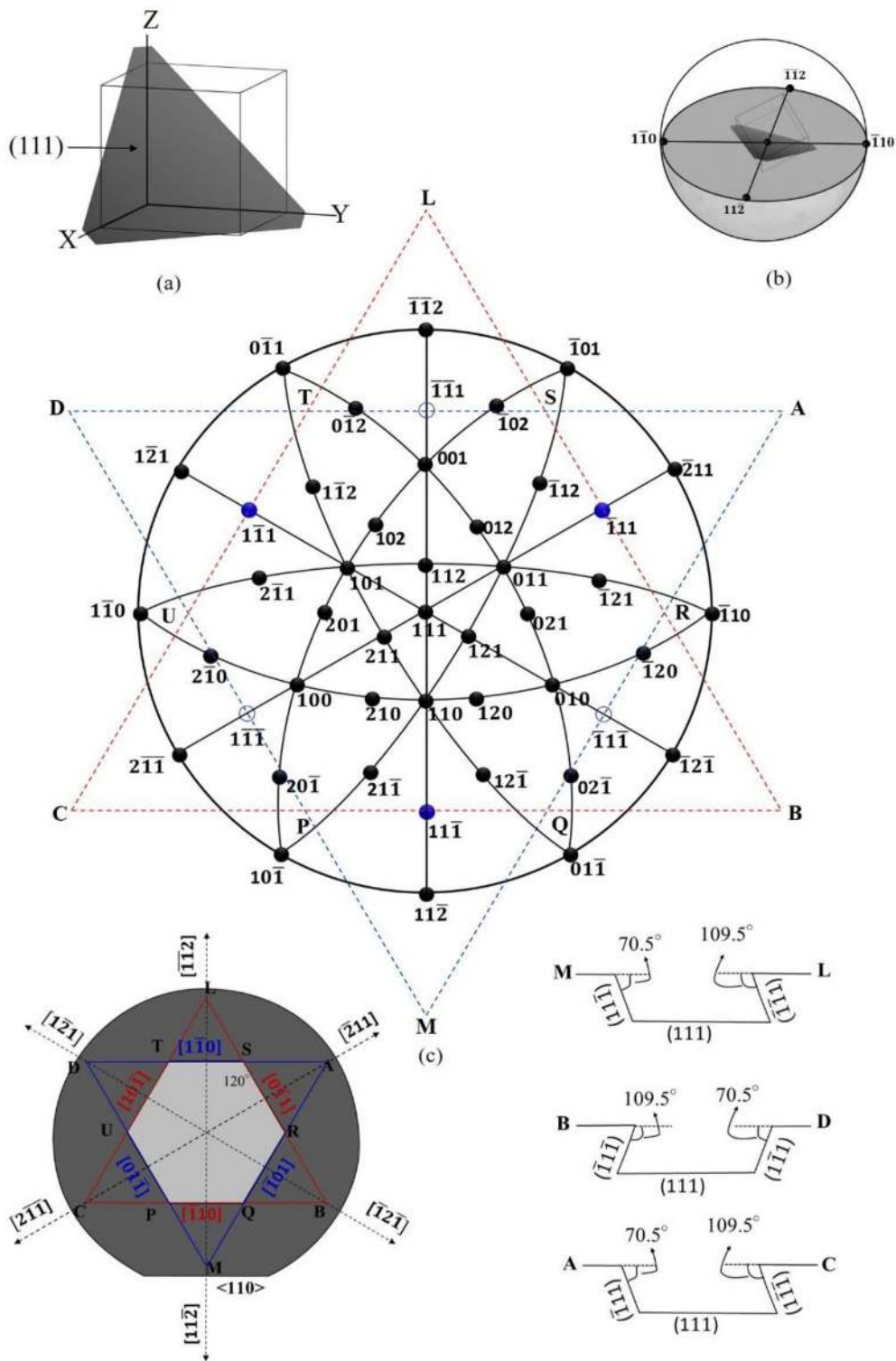


Fig. 1 Schematic representation of stereographic projection of {111} silicon: **a** (111) plane inside a unit cell, **b** unit cell at the center of a sphere which is used to project different crystallographic planes on 2D surface, and **c** stereographic projection exhibiting different planes. The {111} planes projected from the top and bottom hemispheres are shown by solid and open blue circles, respectively. Three {111} planes shown by blue color dots make an angle of 109.5° to the silicon wafer surface plane and 60° with each other, while another three {111} planes indicated by open blue circle form an angle of 70.5° to the surface plane and 60° to each other

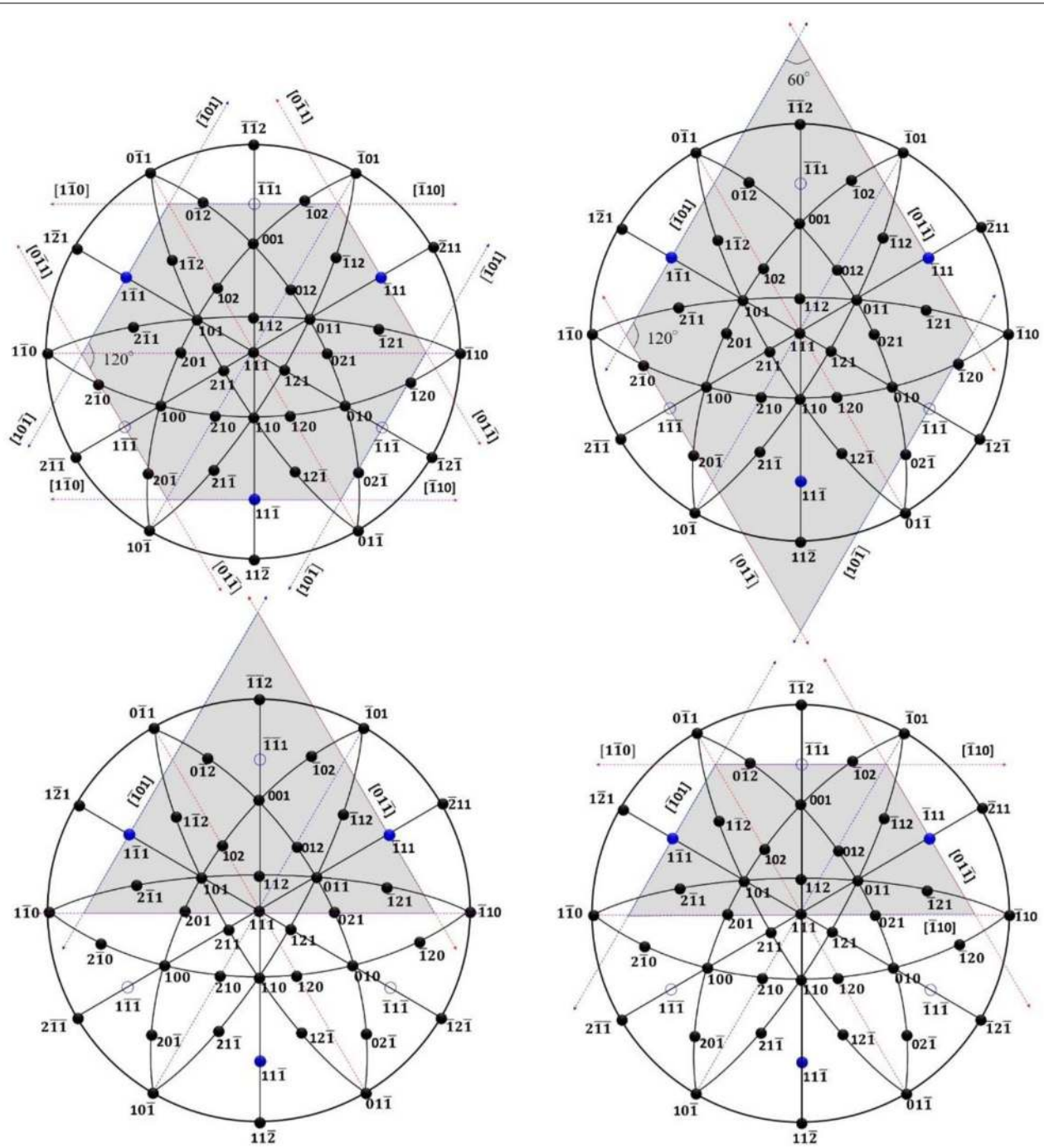
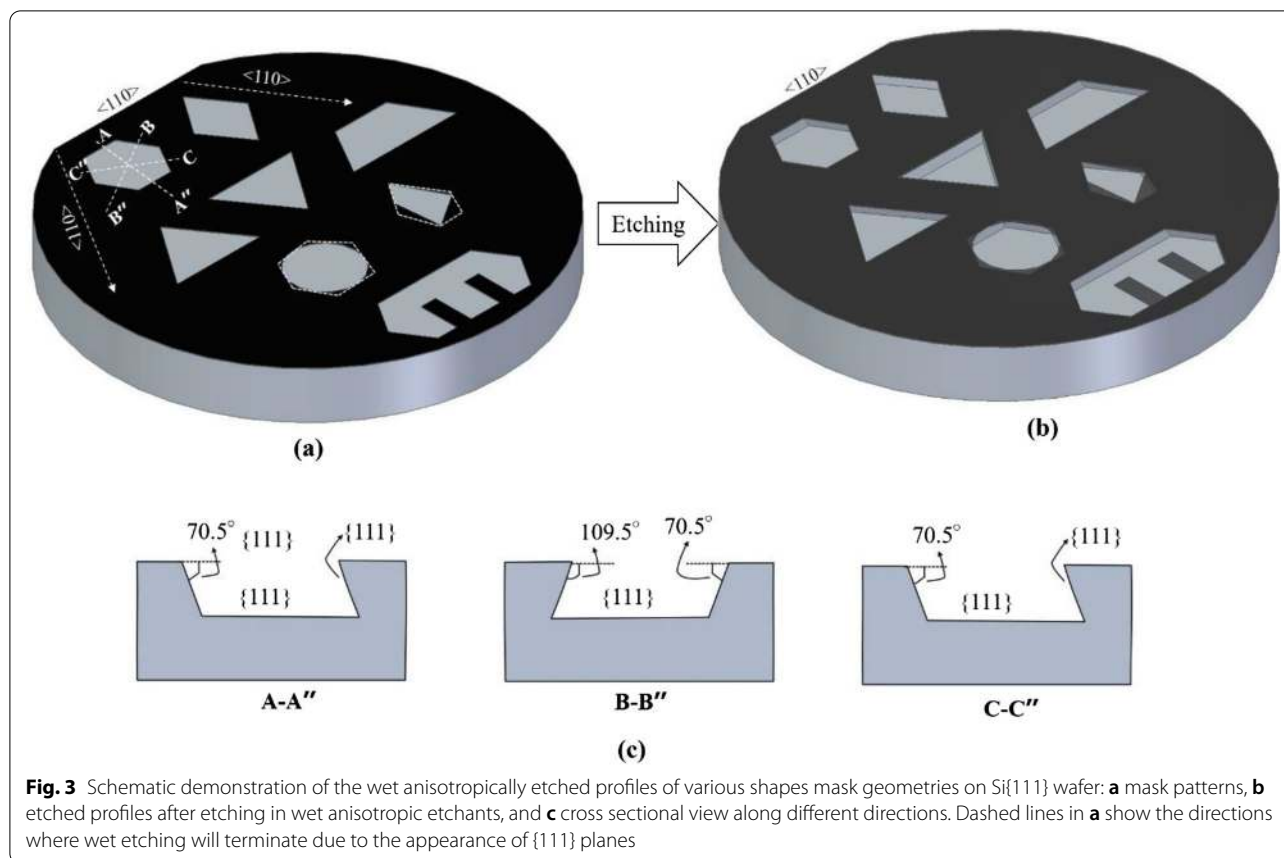


Fig. 2 Different shapes formed by $\langle 110 \rangle$ crystallographic directions at which $\{111\}$ planes appear during wet anisotropic etching process such as hexagonal, rhombus, triangle, isosceles trapezoid

(OLYMPUS OLS4000) is used to measure the etch depth, undercutting length and surface roughness. The measurements are carried out at different locations of the

samples. The etched surface morphology is characterized using scanning electron microscope (SEM).



Results and discussion

The etching characteristics (e.g., etch rate, surface roughness, and undercutting) of Si{111} in 10M NaOH without and with the addition of 12% NH₂OH are investigated. Etch rate and undercutting are determined by measuring the etch depth and lateral undercutting length, respectively, for different etching times. Surface roughness is measured at different locations of the same sample, and then its mean and standard deviation are calculated. The etched surface morphology is analyzed using a SEM. In the subsequent subsections, etching characteristics of Si{111} are systematically presented. To determine the impact of etchant aging on the etching characteristics, successive etching experiments are performed in the same solvent for next 15 days.

Etch Rate

The etch rate is one of the most important parameters to be measured when the etching characteristics of an etchant are investigated. It is characterized as vertical etch depth per unit time as illustrated in Fig. 4. It is required to estimate other parameters such as etching time for the formation of different kinds of microstructures such as cavities/grooves. To calculate the etch rate in pure and

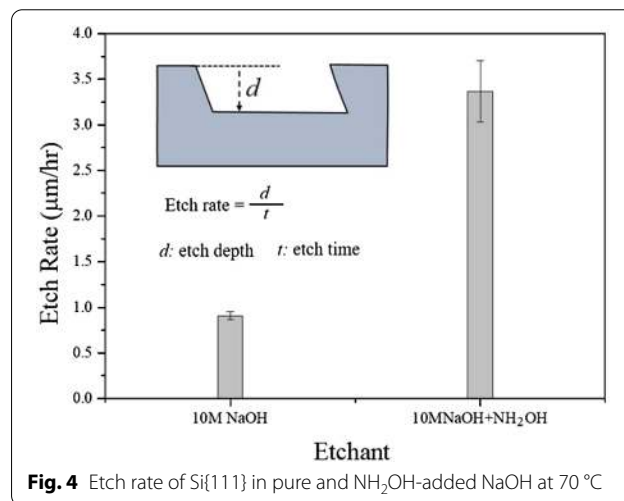
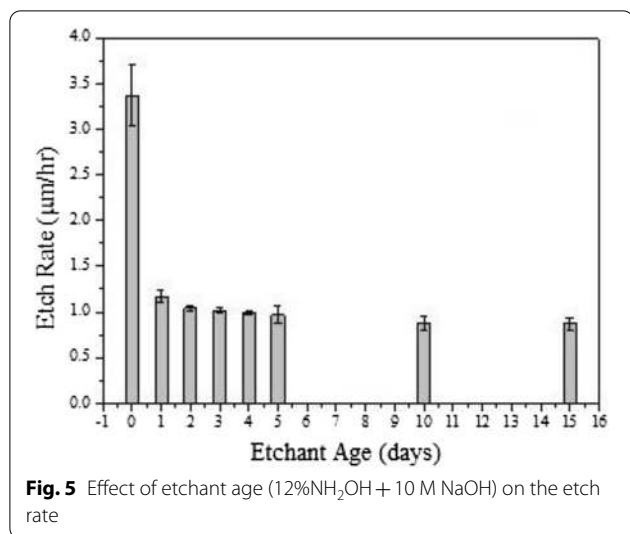


Fig. 4 Etch rate of Si{111} in pure and NH₂OH-added NaOH at 70 °C

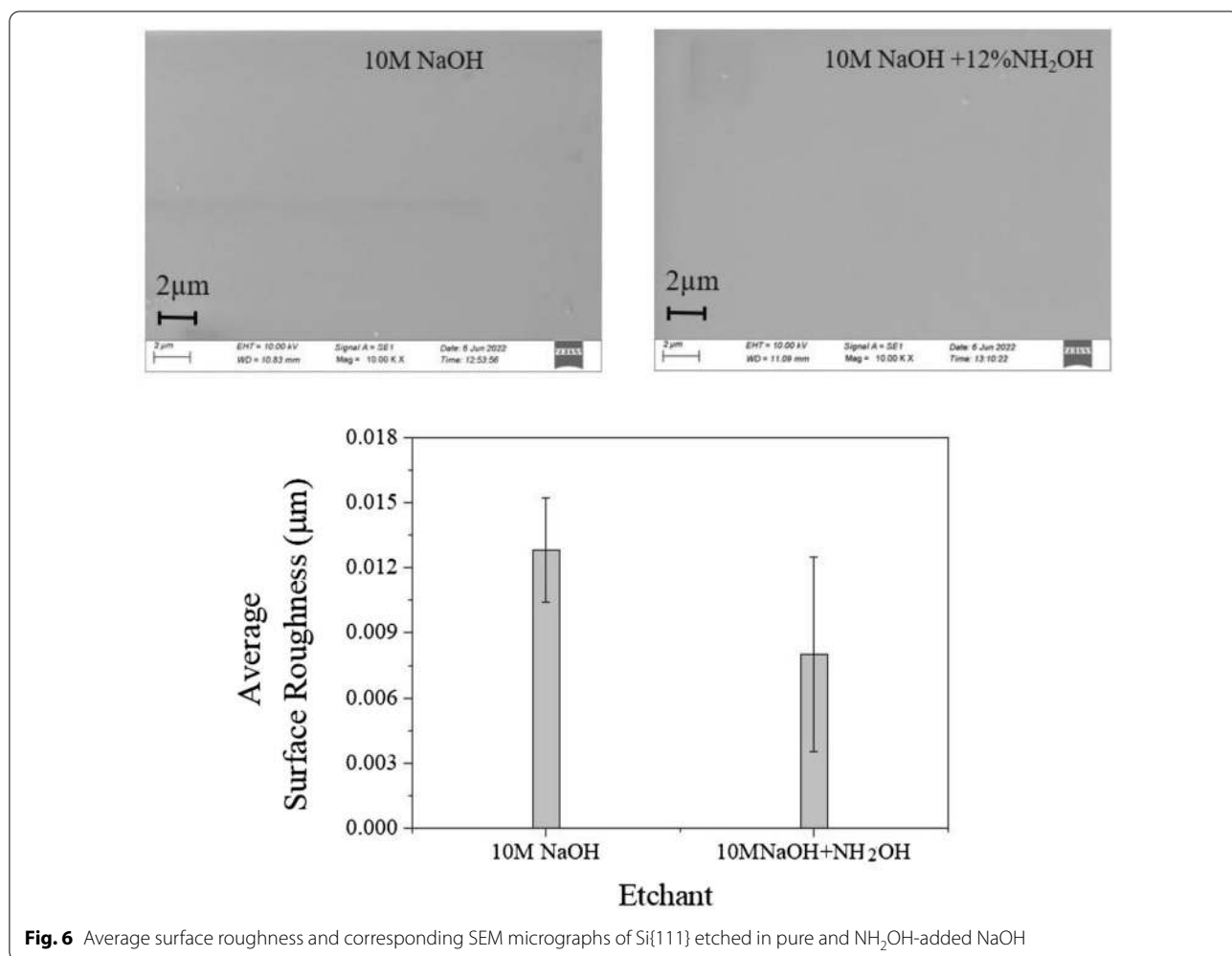
NH₂OH-added NaOH, etch depths are measured on Si{111} samples etched for different etching times. The results are presented in Fig. 4. The etch rate of Si{111} significantly increases when the NH₂OH is added in NaOH solution, which is useful to reduce the etch time to form the microstructures, for instance, cavities. Several factors



may affect the etch rate in an etchant such as etchant concentration, etching temperature, loss of reactive species (H₂O, OH⁻, etc.), the amount of substrate dissolved in the etchant, impurities/additives in the etchant, etc.

[1, 2]. When the NH₂OH is incorporated, the reactive species H₂O, OH⁻, and NH₂O⁻ increases [44]. These extra species might be produced from the decomposition of NH₂OH as intermediate and final products in the presence of alkaline solutions [44–47]. Therefore, it is speculated that the etch rate in NH₂OH-added NaOH increases due to the presence of extra reactive species.

To investigate the effect of etchant age on the etching characteristics, the same etchant solution is continuously used for the next 15 days. Silicon etching is carried out after every one day for the next five days. Thereafter, etching is performed after every five days. The impact of etchant age on the etch rate is presented in Fig. 5. It can easily be noticed that the etch rate is considerably reduced with etchant age during etching process. It is speculated that the accessibility (or production) of extra reactive species may decline as the etchant age increases, that may lead to decrease in the etch rate [44].



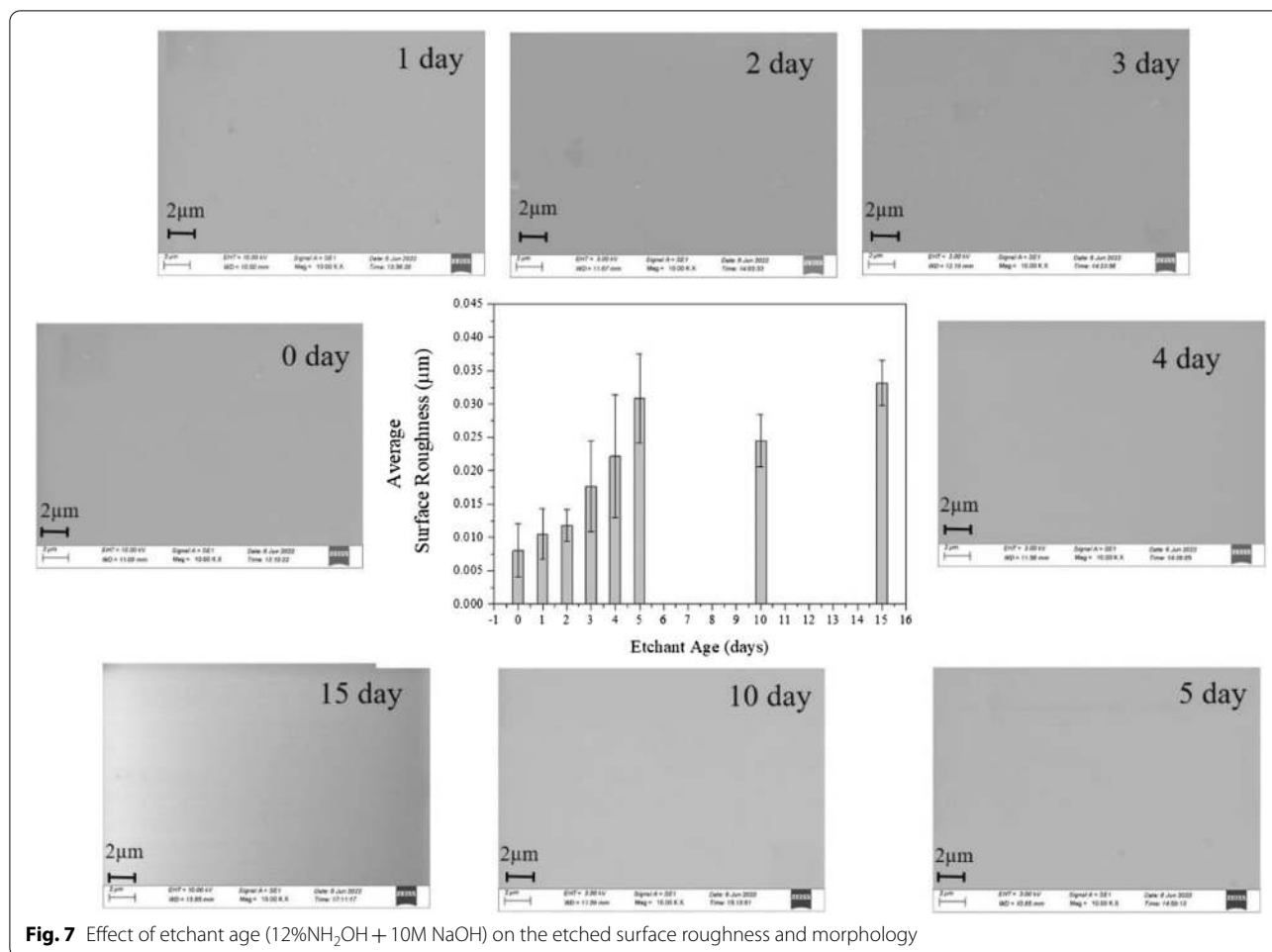


Fig. 7 Effect of etchant age (12% NH_2OH + 10M NaOH) on the etched surface roughness and morphology

Etched surface morphology

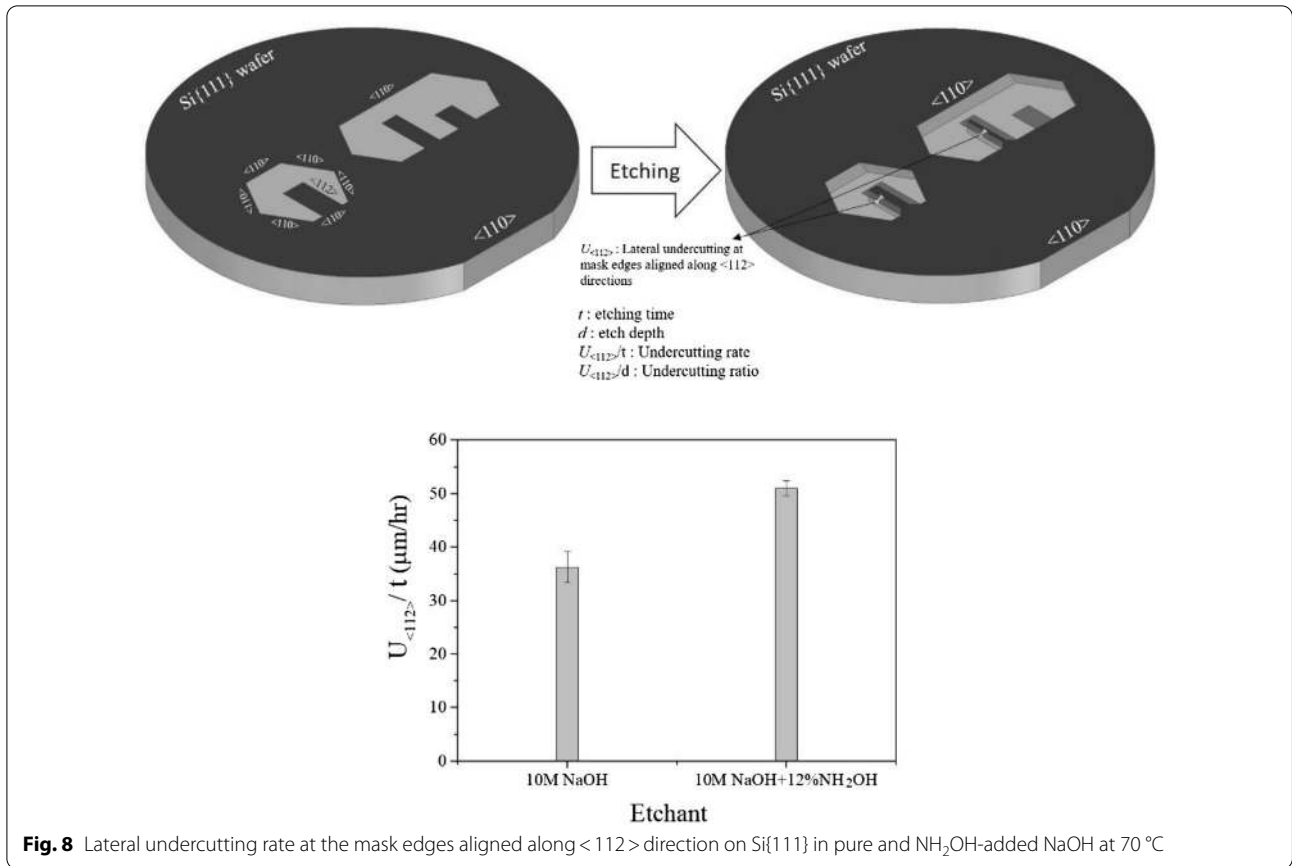
Etched surface morphology is a major concern specifically when the microstructures with uniform depth need to be fabricated or the surface is used for optical applications. The etching parameters, which affect the etch rate, also influence the etched surface roughness and morphology. It is basically a result of non-uniform removal of silicon atoms from the surface during etching process. It happens mainly due to the micro-masking effect [1, 2]. Surface contaminations and hydrogen bubbles predominantly inhibit surface reactions and therefore act as micro-mask during etching process [1, 2, 48–51].

Figure 6 presents the average surface roughness (R_a) and corresponding surface morphologies of Si{111} etched in pure and NH_2OH -added NaOH. The measurement is obtained by scanning a $200 \times 200 \mu\text{m}^2$ area across different locations on the same sample using a 3D laser scanning microscope. It can be noticed from the graph that the surface roughness is decreased when NH_2OH is added to NaOH. As the etched surface morphology depends on various factors, the results may

vary from one laboratory to another laboratory and sample to sample in the same type of etchant under similar etching parameters. To investigate the effect of etchant age, the same methodology, which is used for etching rate study, is followed. Figure 7 exhibits the consequence of etchant aging on the average surface roughness. It can be seen from the results that the aging of the etchant deteriorates the etched surface roughness.

Undercutting at mask edges

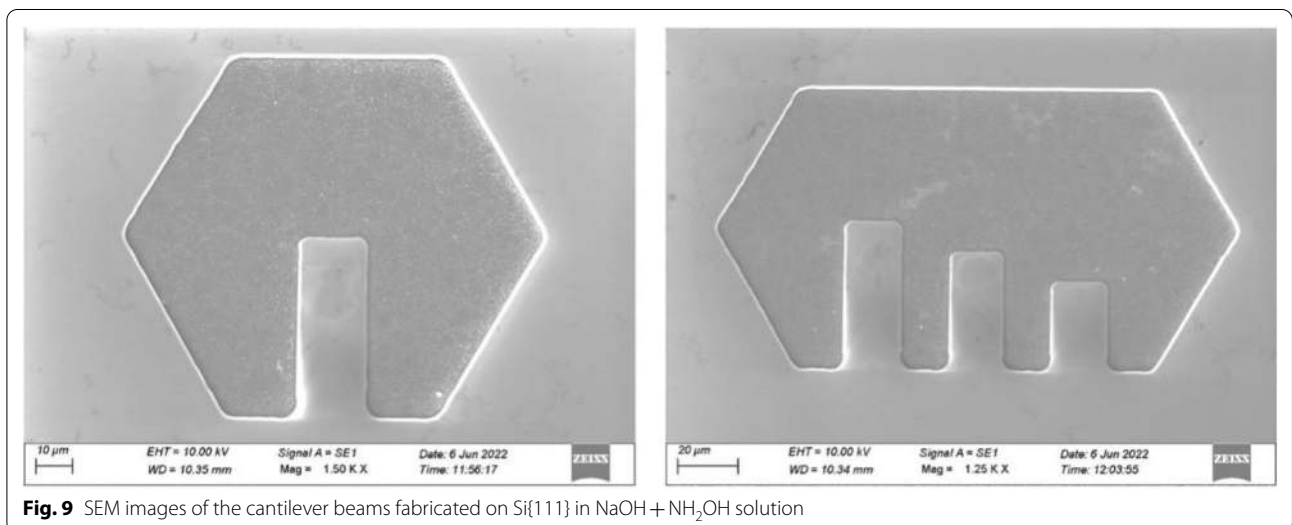
Undercutting refers to the lateral etching that occurs under the masking layer [52]. It has its own advantages and disadvantages. It is a desirable feature for the fabrication of overhanging structures made up of materials exhibiting high etch selectivity with silicon such as SiO_2 , Si_3N_4 as schematically illustrated in Fig. 8. In this work, the undercutting rate is investigated at the mask edges aligned $\langle 112 \rangle$ directions. The results are presented in Fig. 8. It can easily be noticed from

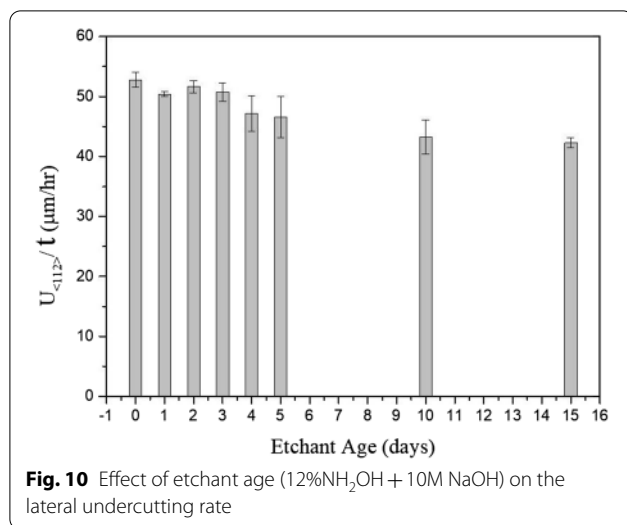


the experimental results that the undercutting rate increases significantly with the addition of NH₂OH. The primary reason behind the increase of undercutting is the same as explained for etch rate in "Etch Rate" section i.e., the reactive species increase on addition of NH₂OH that results in the increase of the undercutting rate at mask edges aligned along $\langle 112 \rangle$ direction. To

demonstrate the application of modified etchant in silicon micromachining, freestanding cantilever beams are fabricated, and the results are presented in Fig. 9.

It is imperative to examine the effect of etchant aging on the undercutting process to understand the variation in etching characteristics as the etchant ages. The same experimental procedure, which is employed





for etch rate study in "Etch Rate" section, is followed to investigate the aging effect. The results are shown in Fig. 10. It can obviously be observed that the lateral undercutting is lessened with the age of the etchant. As discussed previously, the reactive species in NH_2OH -added NaOH are reduced with etchant age, which is the main reason behind the decrease in undercutting rate with the age of the etchant. Although the undercutting rate decreases with time, it is still higher than that in pure NaOH. As the etchant age adversely affects the undercutting rate, NH_2OH -added NaOH should be used immediately after its preparation to obtain the advantages of higher undercutting.

Conclusions

A 10M NaOH solution without and with 12% NH_2OH is used to investigate the etching characteristics (etch rate, etched surface morphology, and undercutting at $\langle 112 \rangle$ mask edges) of Si $\{111\}$ wafers. The incorporation of NH_2OH significantly improves the etch rate, which is vital for enhancing productivity. Moreover, the undercutting rate is increased when NH_2OH is added in NaOH, which is crucial for the quick release of the microstructures from the substrate. Etched surface morphology is not significantly affected by the addition of NH_2OH . Furthermore, the effect of etchant aging on etching characteristics is examined. The etch rate and undercutting rate are suppressed with the etchant age. In addition, the etched surface roughness is adversely impacted by the aging of the solution. Based on the results presented in this paper, it can be concluded that the modified NaOH solution should be used immediately after the addition of NH_2OH to achieve a higher undercutting rate at $\langle 112 \rangle$ mask edges.

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Author contributions

SP and VS did experiments and have made equal contributions. SP, VS and PP wrote the manuscript. AKP reviewed and edited the manuscript. All authors read and approved the final manuscript.

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Availability of data and materials

Not applicable.

Declarations

Ethics approval and consent to participate

Not applicable.

Consent for publication

All authors agreed to this publication.

Competing interests

The authors declare that they have no competing interests.

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