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LETTER

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Enhanced etching characteristics of Si{100} in NaOH-based two-component solution

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Abstract

Silicon wet bulk micromachining is the most widely used technique for the fabrication of diverse microstructures such as cantilevers, cavities, etc. in laboratory as well as in industry for micro-electromechanical system (MEMS) application. Although, increasing the throughput remains inevitable, and can be done by increasing the etching rate. Furthermore, freestanding structure release time can be reduced by the improved undercutting rate at convex corners. In this work, we have investigated the etching characteristics of a non-conventional etchant in the form of hydroxylamine (NH_2OH) added sodium hydroxide (NaOH) solution. This research is focused on Si{100} wafer as this orientation is largely used in the fabrication of planer devices (e.g., complementary metal-oxide semiconductors) and microelectromechanical systems (e.g., inertial sensors). We have performed a systematic and parametric analysis without and with 12% NH_2OH in 10 M NaOH for improved etching characteristics such as etch rate, undercutting at convex corners, and etched surface morphology. 3D scanning laser microscope is used to measure average surface roughness (R_a), etch depth (d), and undercutting length (l). Morphology of the etched Si{100} surface is examined using optical and scanning electron microscopes. The addition of NH_2OH in NaOH solution remarkably exhibited a two-fold increment in the etching rate of a Si{100} surface. Furthermore, the addition of NH_2OH significantly improves the etched surface morphology and undercutting at convex corners. Undercutting at convex corners is highly prudent for the quick release of microstructures from the substrate. In addition, we have studied the effect of etchant age on etching characteristics. Results presented in this article are of large significance for engineering applications in both academic and industrial laboratories.

Keywords: Silicon, MEMS, Wet bulk micromachining, Wet etching, NaOH, NH_2OH

Introduction

Various kinds of microfabrication techniques such as dry etching, wet etching, surface micromachining, focused ion beam, laser micromachining, etc., are used to fabricate microstructures for microelectromechanical systems (MEMS) [1–3]. Wet anisotropic etching is one of the renowned techniques to fabricate different kinds of suspended structures (e.g., microcantilever beams, diaphragms, etc.) as well as fixed structures (e.g., grooves, trenches, channels, etc.) for applications in MEMS-based sensors and actuators [1–8]. This technique is extensively used due to its numerous advantages, such as relatively low cost, orientation dependent etching, controllable etch rate with addition of additives, ability to fabricate 3D structures with slanted sidewalls, high undercutting to fabricate suspended structures, high selectivity to materials, etc. Most important feature of wet etching is the capability of batch fabrication which makes it useful for industrial fabrication. Hence, silicon wet etching remains an active area of research. Silicon wet anisotropic etching can be performed using many kinds of alkaline solutions such as potassium hydroxide (KOH) [9–14], tetramethyl ammonium hydroxide (TMAH) [15–22], ethylenediamine pyrocatechol water (EDP) [23, 24], ammonium hydroxide (NH_4OH) [25], cesium hydroxide (CsOH)

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[26], hydrazine [27, 28], sodium hydroxide (NaOH) [29, 30], etc. Among these etchants, TMAH and KOH are extensively investigated and employed for silicon wet etching in MEMS and solar cell industries [1–3, 31, 32]. Etching characteristics of silicon using these etchants can be altered by the addition of additives such as a surfactant or an alcohol [5–7, 12, 13, 22, 33–40]. Though a smooth surface can be obtained by the addition of surfactant, it impedes the etch rate, consequently inhibiting the commercial scale fabrication. Different methods such as ultrasonic agitations, microwave irradiation, and by adding additives in wet anisotropic etching have been employed to increase the etch rate [41–55]. However, each method has its pros and cons. For example, ultrasonic agitations and microwave irradiation on the etchants increase etch rate but at the same time damage fragile structures. Other way of increasing the etch rate is etching at (or near) the boiling point of the etchant [11, 56]. Recently, NH_2OH-added TMAH and KOH solutions, which provide very high etch rate in comparison to pure KOH and TMAH, are explored for applications in wet bulk micromachining for the formation of MEMS structures [49–54]. Although NH_2OH-added KOH/TMAH provides very high etch rate, the etch rate is reduced with the age of etchant [54, 55]. NaOH, a low-cost etchant, has been explored for the fabrication of microstructures with vertical walls, removal of the saw-damaged layer from the multi-crystalline as-cut silicon wafer and surface texturing for silicon solar cell [57–59]. To find the potential applications of NaOH as etchant, more study is needed especially with an additive to improve the etch rate.

In the present work, we have investigated the etching characteristics of Si{100} in pure and NH_2OH-added NaOH. This orientation is selected due to its importance and popularity for the fabrication of integrated circuits (ICs) and MEMS. The effect of aging on the etching characteristics, especially etch rate and undercutting, is studied.

Experimental

Four-inch, Czochralski grown, p-type, one side polished Si{100} wafers with a resistivity of 1–10 Ωcm are used to study the etching characteristics. An oxide layer (1 µm thickness) is grown using a thermal oxidation process and patterned using lithography. Followed by oxide etching in buffered hydrofluoric (BHF) acid, the wafers are cleaned thoroughly using deionized (DI) water. After oxide etching, the photoresist is removed with acetone and then the wafers are rinsed in DI water. The wafers are diced into 2×2 cm^2 pieces using a dicing saw. The diced samples are cleaned in a piranha bath (H_2SO_4:H_2O_2::1:1) followed by a DI water rinse to remove any trace of organic material and unwanted particles on the surface. The cleaned samples are then dipped into 1% HF for 1 min to remove the oxide which would have grown during piranha cleaning. Following this, the samples are thoroughly rinsed in DI water. Thereafter etching is carried out in pure and 12% NH_2OH-added 10 M NaOH. 10 M of NaOH is selected because it provides high etch rate at its boiling point [29, 30]. Every etching experiment is performed in a 1-L etchant solution. To prepare a 1-L 10 M NaOH solution, 400 gm pellets are dissolved in 1000 ml deionized (DI) water. In the case of 1-L 12% NH_2OH-added 10 M NaOH, 400 grm NaOH pellets, 240 ml of 50% NH_2OH and 760 ml DI water are used. All experiments are performed at 70 ± 1 °C. A constant temperature bath is used to maintain the temperature throughout the experiment. Etching process is carried out in a Teflon made container. To avoid any possible changes in the etchant concentration incurred due to continuous heating of the etchant during experiments, a reflex condenser made of thick glass equipped with a double-layered narrow opening is used. The etched samples are characterized using a 3D measuring laser microscope (OLYMPUS OLS4000), optical microscope (OLYMPUS MM6C-PC) and scanning electron microscope (SEM). The measurements are taken at different locations of the sample/chip after performing the etching process to calculate the standard deviation.

Results and discussion

In this work, we have studied the etching characteristics of Si{100} (etch rate, surface morphology and undercutting) in pure and NH_2OH-added 10 M NaOH to promote the applications of wet etching in MEMS fabrication. These etching characteristics are systematically presented in following subsections.

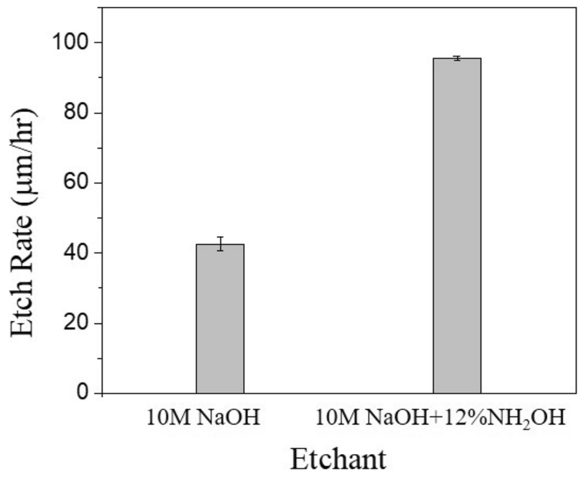

Fig. 1 Etch rate of Si{100} in 10 M NaOH without and with addition of 12% NH_2OH

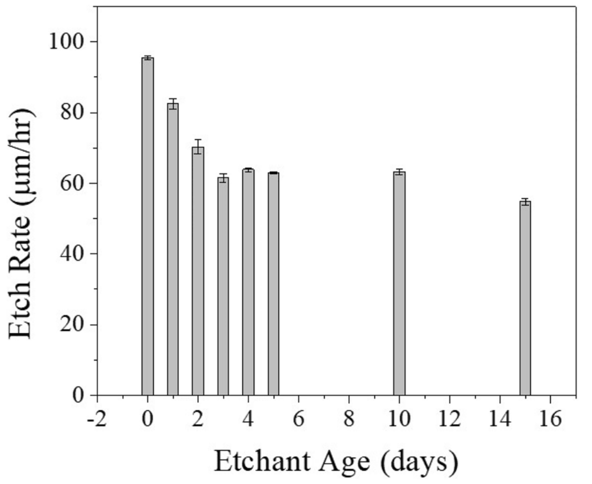

Fig. 2 Effect of aging of 10 M NaOH + 12% NH_2OH on the etch rate of Si100}

Etch rate

Etch rate is a key parameter when etching is involved in silicon micromachining for the formation of microstructures for MEMS-based sensors and actuators. It is defined as the vertical distance etched per unit time (i.e., etch depth (d) per unit time (t) or d/t). The etch depth on the samples etched in pure and modified NaOH is measured using a 3D laser scanning microscope. Figure 1 presents the etch rate of the Si{100} surface in pure and modified NaOH at 70 ± 1 °C. The standard deviation is determined by taking six measurements on the same chip at different locations. It can easily be noticed that the etch rate increases remarkably by the addition of NH_2OH to pure 10 M NaOH. The etch rate becomes double in 12% NH_2OH + 10 M NaOH than that of pure 10 M NaOH.

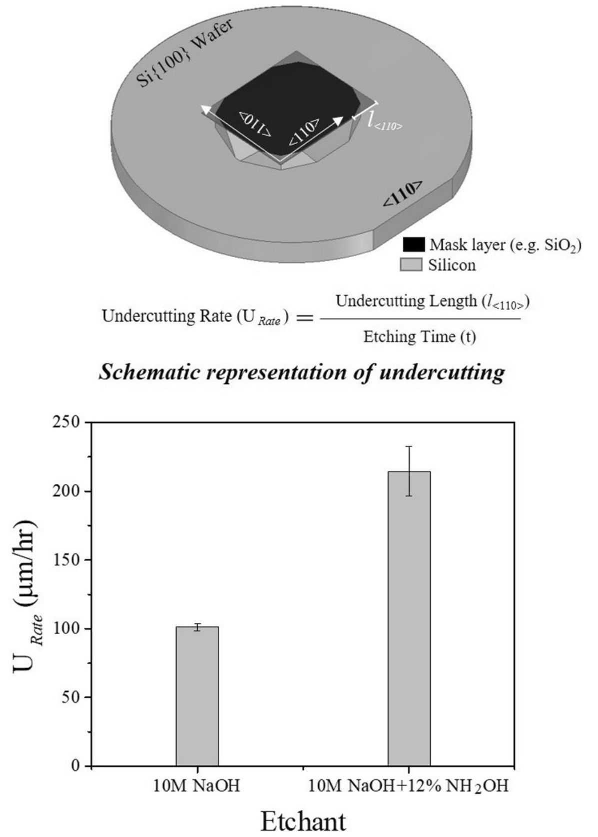

Fig. 3 Undercutting rate in 10 M NaOH without and with addition of 12% NH_2OH at 70 ± 1 °C on the mask pattern formed by < 110 > directions on Si{100} surface

Although our focus in this work is on the etching characteristics of silicon in NaOH based solutions, which is useful for academic and industrial applications. Yet we

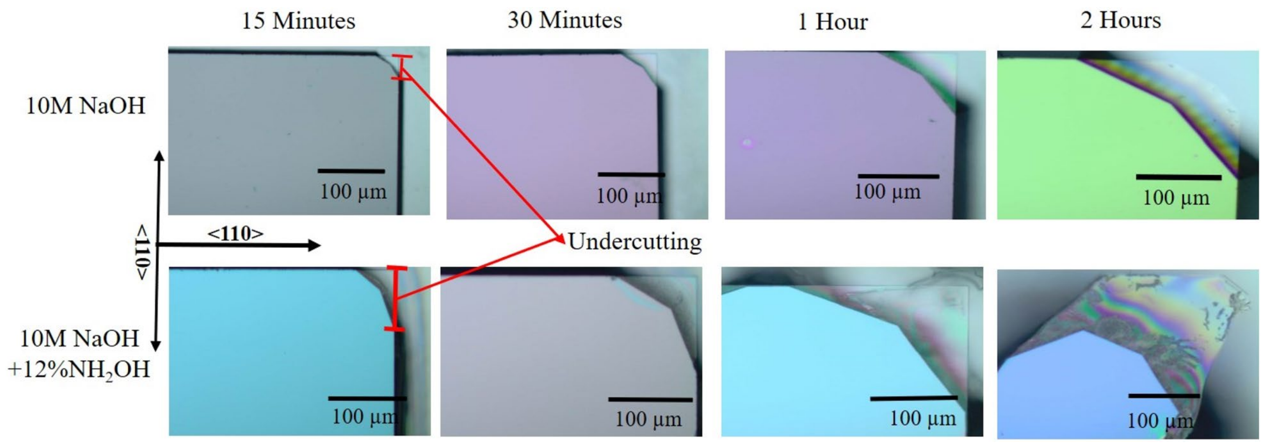

Fig. 4 Optical micrographs of undercutting at convex corners on Si{100} surface

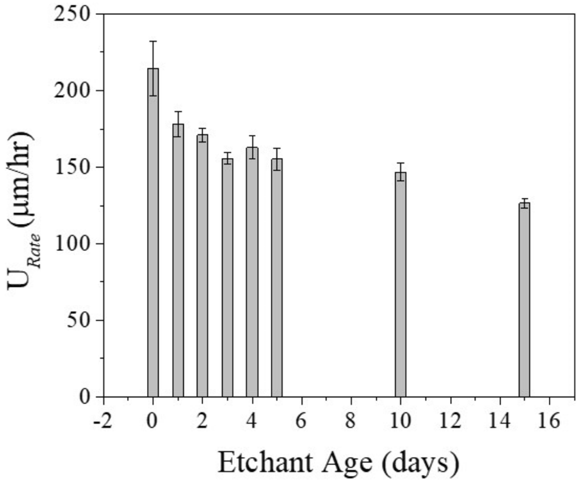

Fig. 5 Effect of etchant age on the undercutting rate in 10 M NaOH + 12% NH_2OH at convex corners

attempt to give an insight behind increase in etch rate in NH_2OH-added NaOH solution. We speculate that the etch rate of silicon increases owing to high accessibility of OH^- ions and H_2O in NH_2OH-added NaOH [49–55]. In wet chemical etching of silicon, the OH^- ions and H_2O plays an important role as a catalyzing and active etching species, respectively [2, 55, 60]. These extra OH^- ions and H_2O molecules might be produced from the chemical decomposition of NH_2OH as intermediate and final products in the presence of alkaline solutions.

The effect of aging of 12% NH_2OH-added 10 M NaOH on the etch rate of Si{100} is investigated. To perform this study, etching experiments are performed every day for next 5 days. After 5th day, etching experiments are carried out on 5 days interval. The results are shown in Fig. 2. It can obviously be noted that the etch rate significantly decreases up to two days, but there is a minor change after third day. Though the etch rate is decreasing with etchant age, it is more than that of pure NaOH solution presented in Fig. 1. It can be stated that the addition of NH_2OH enhances the etch rate significantly and very useful for industrial application to improve the throughput. As stated earlier, the present work is focused on engineering applications of wet etching. However, we attempt to explain the reason behind reduction of Si{100} etch rate with etchant age. As the etchant age is increases, the availability of OH^- and H_2O may decrease, which leads to reduction in the etch rate [60].

Undercutting rate

Undercutting is the lateral etching which takes place under the masking layer. Undercutting at the convex corners is an essential parameter to fabricate freestanding

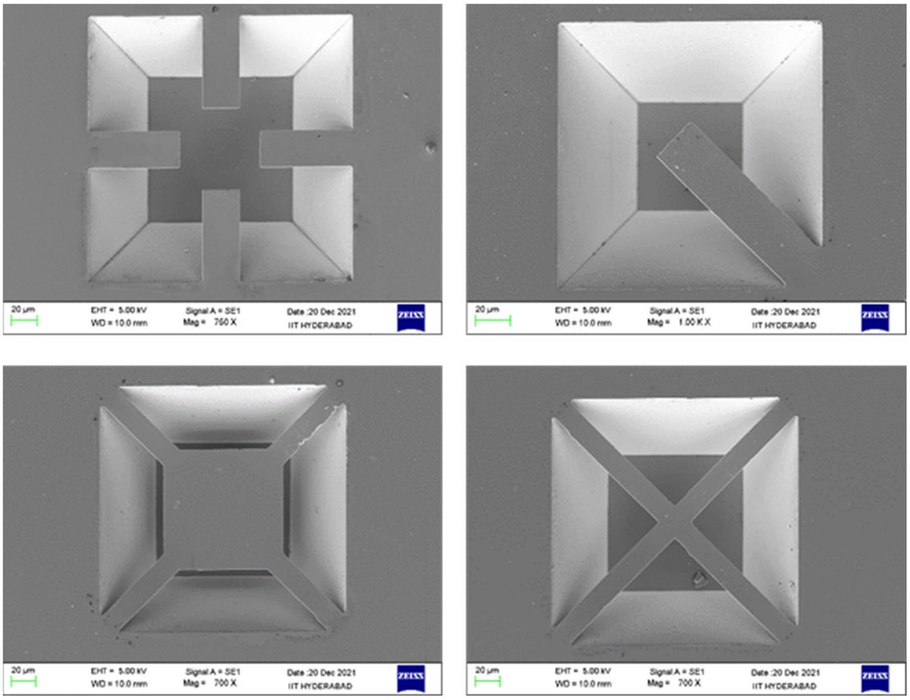

Fig. 6 SEM images of fabricated structures in NH_2OH-added NaOH

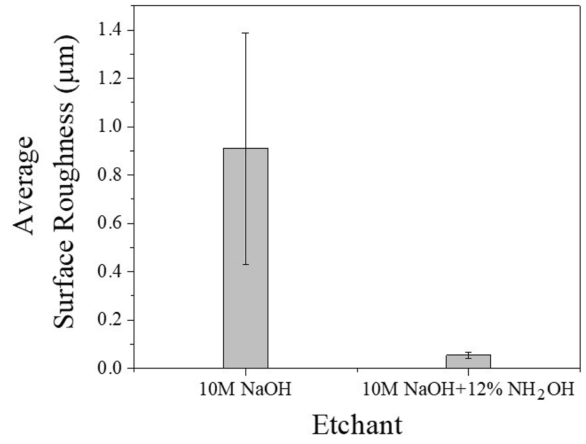

Fig. 7 Average etched surface roughness of silicon in 10 M NaOH without and with addition of 12% NH_2OH at 70 ± 1 °C

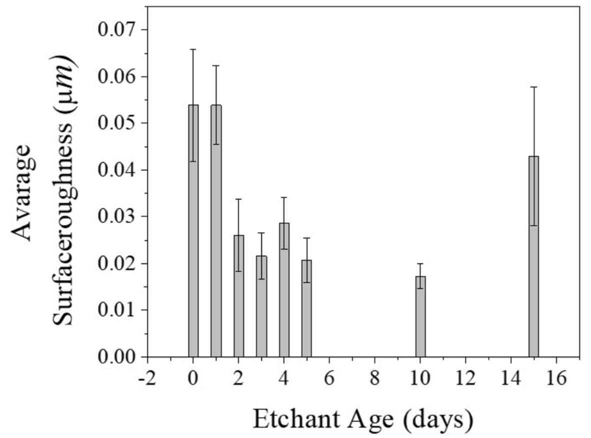

Fig. 9 Effect of etchant (10 M NaOH + 12% NH_2OH) age on the etched surface roughness

structures like cantilever beams for applications in MEMS/NEMS-based sensors and actuators [61, 62]. However, convex corner undercutting is undesirable for fabricating the mesa structures for some applications such as accelerometers and other sensors. Undercutting length along the < 110 > direction ($l_{<110>}$) and etch time (t) is used to define the undercutting rate ($U_{\mathrm{rate}} = l_{<110>}/t$), which is a very important parameter to estimate the release time. Figure 3 presents the undercutting rate at the convex corners in pure and NH_2OH-added 10 M NaOH. It clearly indicates that the undercutting rate increases significantly when NH_2OH is added to 10 M NaOH. The undercutting rate in NH_2OH-added NaOH becomes more than double to that in pure NaOH. The optical micrographs of the undercut convex corners for different etching duration are shown in Fig. 4. The similar explanation as the etch rate can be applicable behind the significant increase of the undercutting rate at convex corners on the addition of NH_2OH. The availability of catalytic species OH^- and H_2O near the convex corner are more in modified NaOH solution, which improves the undercutting considerably.

Effect of etchant age on the undercutting rate is studied and presented in Fig. 5. The undercutting rate follows the same trend as the etch rate. It decreases with etchant age and becomes stable after two days of the etchant age. The reactive species in the etchant solution decrease with etchant age that results in the decrease of undercutting with etchant aging [55]. Although the undercutting rate decreases with the age of the solution, it is higher in comparison to pure NaOH. It can be concluded that the etchant should be used immediately after its preparation to obtain higher undercutting. However, in terms of undercutting rate, NH_2OH-added NaOH solution is better

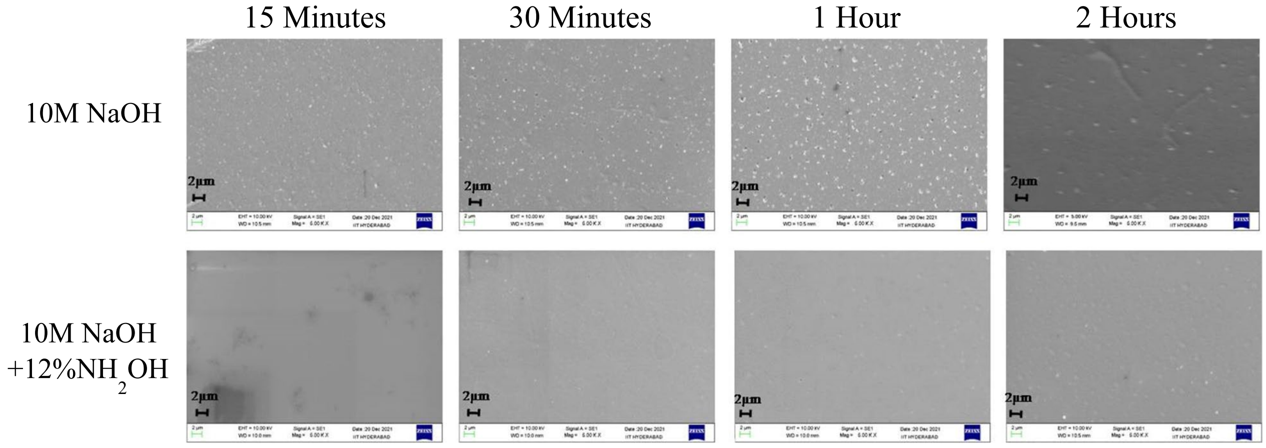

Fig. 8 SEM images of etched surface morphology of silicon {100}

than NH_2OH-added TMAH/KOH as the effect etchant age on its etching characteristics is less.

To demonstrate the applications of high undercutting rate in NH_2OH-added NaOH in the fabrication of MEMS components, various kinds of microstructures are released in this etchant. The SEM micrographs of the fabricated structures are exhibited in Fig. 6.

Etched surface morphology

Surface morphology is another important parameter to be considered in MEMS, especially for optical applications (e.g., MOEMS) [1–3, 37, 39, 63]. It plays a crucial role in silicon micromachining for the fabrication of components for micro-devices such as cavities, gratings, diaphragms, micro-mirrors, cantilevers, etc. The average surface roughness on the samples etched in pure and NH_2OH-added NaOH is measured using 3D laser scanning microscope at different locations of the sample. The results are presented in Fig. 7. As can be seen in the figure, the surface roughness substantially decreases by the addition of NH_2OH. The SEM micrographs of surface morphology etched at different times in pure and NH_2OH added 10 M NaOH solutions are presented in Fig. 8. Based on the results presented in Figs. 7 and 8, it can be stated that the modified etchant is a good choice for the fabrication of microstructures where a smooth surface is needed. The surface roughness at the microscopic scale occurs due to the non-uniform removal of atoms from the surface or lattice defects which are present on the surface and extend into the bulk crystal. It is characterized by the formation of pyramids or hillocks on the surface. In wet etching, surface roughness is the result of various factors; one is the formation of a hydrogen bubble during etching which hinders the surface reactions and acts like a micro mask on the surface, and another is the deposition of etching by-products on the surface [2].

The effect of etchant age on the surface roughness is presented in Fig. 9. It can simply be noticed that the surface roughness is fluctuating with the etchant age. As the etched surface morphology depends on various parameters and can be influenced by any kinds of surface contamination on the silicon surface during etching. However, we can claim from the results that the etched surface roughness is not deteriorated with etchant age.

Conclusions

In the present work, we have presented a detailed investigation of a non-conventional etchant in the form of NH_2OH-added into 10 M NaOH, which possesses the capability of exhibiting a higher etch rate along with high undercutting at convex corners and improved etched surface morphology on Si{100} wafers. High etch rate is beneficial to decrease the etching time and hence the higher throughput, which is inevitable for industrial application to reduce the product cost. In addition, the high undercutting is very useful to reduce the etching time to release the structure from the substrate. Moreover, the effects of etchant age on the etching characteristics are investigated. It is found that the etch rate and the undercutting at convex corners are significantly decreased with the etchant age. At the same time surface roughness fluctuates with etchant age. Hence, it is recommended that the NH_2OH-added NaOH must be use after its preparation to take the advantage of higher etch rate and undercutting.

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Author contributions
VS and SP did experiments. VS, SP, and PP wrote the manuscript. RKS reviewed and edited the manuscript. All authors read and approved the final manuscript.

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Declarations

Ethics approval and consent to participate
Not applicable.

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All authors agreed to this publication.

Competing interests
The authors declare that they have no competing interests.

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