

Realization of Three-Dimensional Micro and Nano-Structures on Silicon Substrates

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Abstract. *The realization of nano and micro-sized, highly-featured, 3-dimensional structures on Si substrates is reported where a single mask in a hydrogen-assisted deep reactive ion etching is exploited. Three main gases of oxygen, hydrogen and SF₆ are used in a sequential passivation and etching process to achieve high aspect ratio features. The flows of gases, and the plasma power and timing of each subsequence are the main parameters to achieve the desired three-dimensional etching by controlling the under-etching and recovery steps, which leads to the formation of unique features directly on silicon substrates. Depending on the plasma power, etch-rates as high as 0.75 μm/min can be obtained. The plasma power can reach a high value of 1 W/cm² over a large area of 20 × 15 cm². In addition, features with a controllable under-etching and a recovery with more than 8 μm in the sidewall recession have been achieved. Furthermore, values of aspect ratios higher than 40-50 can be obtained. The formation of nano-wall features is also reported.*

Keywords: *Vertical etching; Deep reactive ion etching; 3-D structures; Curved nano-walls.*

INTRODUCTION

Deep vertical etching and especially three-dimensional (3-D) structures are promising features for optical and mechanical applications. Conventionally, 3-D structures refer to those where height, as the third dimension of the feature, is controlled during the vertical etching process. These structures can be regularly realized using multiple lithographies. Recently, however, a single masking layer fabrication process has been introduced using deep reactive ion etching combined with gray scale lithography [1-4]. The evolution of Micro-Stereo-Lithography (MSL) has been shown to be a powerful technique to achieve high-aspect-ratio, three dimensional structures on polymers, where the height remains constant but where the structure possesses complex features in the *z*-direction. In this technique, a UV laser illumination is used to create sections on a UV-curable polymer to form the desired shape [5].

MSL enjoys high accuracy and miniaturization power but it is not immediately applicable to silicon

substrates and requires a polymeric coating. Due to its natural polymerization and exposure, it is considered as a serial fabrication process, making it time consuming and expensive. Nevertheless, it can be used as a method to form three-dimensional features on silicon substrates after a micro-molding step is carried out [6]. Apart from micro-scale features, sub-micro and nanostructures with three-dimensional features are realized using a glancing low angle deposition and layer engineering technique [7,8]. Such three-dimensional features are suitable candidates for optical applications, where wavy metallic nanowires are formed on glass and silicon substrates during film deposition.

The advent of silicon micromachining has been an important step towards the realization of various 3-D features [9]. Complex devices, such as pressure sensors, accelerometers, gyroscopes, optical mirrors and switches, RF devices, filters and micro-motors, lend their success to the advent of silicon micromachining and its outcomes, where the anisotropic effects of etching chemistry are combined with the isotropic plasma-based chemistry to achieve desired shapes and features [10-16]. Apart from wet chemical etching processes, the use of a dry etching tool, especially plasma-based reactive ion etching, is a complementary technique to achieve high aspect ratio 3-D structures [9].

The invention of Deep Reactive Ion Etching

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(DRIE) has been a great milestone in modern micro/nano-electromechanical systems. A process which was first reported by the Bosch Company in 1998 utilizes a sequential etching and passivation steps to form vertical trenches and walls onto silicon substrates with high-aspect ratio and controllable sizes and depths [17]. The consecutive use of a polymeric coating during the passivation cycle guarantees the protection of the sidewalls of the etched craters, while the bottom of the etched region is removed with the aid of high density Inductively Coupled Plasma (ICP). The etching subsequence is also achieved using RF-plasma with a reactive gas like SF_6 . The formation of vertical features with aspect-ratios as high as 100:1 has been reported [18]. A non-sequential cryogenic process has also been introduced, taking advantage of a mixture of oxygen and SF_6 for the etching of silicon substrate with no need for a polymeric coating step. The key parameter in this technique is to maintain the substrate at a cryogenic temperature (-110°C) during the etching [19]. The concurrent use of SF_6 and oxygen during the etching, with the addition of chlorine-based gases, allows the formation of high-aspect ratio features at ambient temperatures. The use of inductively-coupled plasma is a critical component in all the reported etching techniques [20].

Recently, we have developed a deep reactive ion etching process, which neither needs high-density plasma nor uses external polymeric coating or cryogenic temperature arrangements. In our novel technique, the vertical etching of silicon substrate is achieved using a sequential etching and passivation sub-cycles, as reported elsewhere [21]. Our process exploits a combination of three main gases of hydrogen, oxygen and SF_6 in a reactive ion etching unit with a RF plasma source, although nitrogen or argon can also be incorporated to purge the chamber between gas-switching. The etching is divided into two or three sub-cycles (etching/passivation) where a mixture of $\text{H}_2/\text{O}_2/\text{SF}_6$ constituents is used during the passivation cycle, while the next subsequence (etching) is achieved merely with SF_6 as the feed gas. In this paper, for the first time, the creation of ultra-small, highly featured, three-dimensional structures with curved sidewalls is reported.

Unique nano-metric features suitable for electronic and photonic devices, are reported. The etching and passivation sub-sequences are programmed in order to achieve the under-etching and recovery steps which form the desired three dimensional features. A potential application of these 3-D silicon structures is in soft lithography, where they can be used as micro/nano-molds, suitable for tissue and biocompatible engineering [22,23]. Next, we describe our deep reactive etching technique followed by the experimental procedure for the formation of three-

dimensional structures. Images have been obtained using a SEM unit operating at 15-30kV and with a secondary electron detector.

EXPERIMENTAL SETUP

P-type 1-5 Ωcm single side polished silicon wafers are used for this investigation. The etching and fabrication processes are based on our recently reported deep reactive etching technique, where a sequential etching/passivation procedure is employed [21]. The etching is achieved in a Reactive Ion Etching (RIE) machine operating at 13.56 MHz plasma with an effective area of $20 \times 15 \text{ cm}^2$. A RCA # 1 solution ($\text{NH}_4\text{OH}:\text{H}_2\text{O}_2:\text{H}_2\text{O}$, 1:1:5) is used to clean the samples prior to any lithographic step. The cleaning step is critical to avoid grass or black silicon formation. The cleaned samples are placed in an electron-beam evaporation system to deposit a $0.1 \mu\text{m}$ -thick layer of Cr. The base pressure for this deposition step is 1×10^{-6} torr. The Cr-coated samples are patterned using standard high precision photolithography (projection lithography) to act as a mask for the subsequent etching process and to realize the desired micro and nano-structures.

Figure 1 shows the schematic of the etching process, illustrating both the passivation and etching sub-cycles. For a three-dimensional etching, the underetching is controlled by adjusting the power, gas mixture and duration of the passivation sub-cycle. The evacuation of the reactor is systematically practiced by means of a mechanical pump and the mixture of hydrogen and oxygen gases is fed into the reactor. These two steps are repeated as many times as required to achieve the desired depth. We believe that during the passivation step, the existence of the hydrogen atoms not only assists in the formation of a thin protective layer on the side-walls of the etched pit, but also bombards the bottom of the crater to impart defects to the exposed silicon surface, hence improving silicon removal in the subsequent etching step. The total etch rate depends on the etching depth during the etching subcycle, as well as on the total duration of a complete cycle. By controlling the etching parameters, one is able to achieve etch-rates as high as $0.75 \mu\text{m}/\text{min}$, while achieving an aspect ratio of 10 and more. If the etch-rate is lowered to values less than $0.4 \mu\text{m}/\text{min}$, a high aspect ratio up to 40-50 can be obtained.

Since this technique does not use Inductive-Couple-Plasma (ICP), an accurate control over important parameters of the process, i.e. gas-flows, plasma power and cycle-duration, are crucial to achieve complex and high-aspect ratio features. Figure 1b represents a schematic gas-flow versus time for the whole etching/passivation cycle. This cycle is repeated as many times as required to achieve the desired

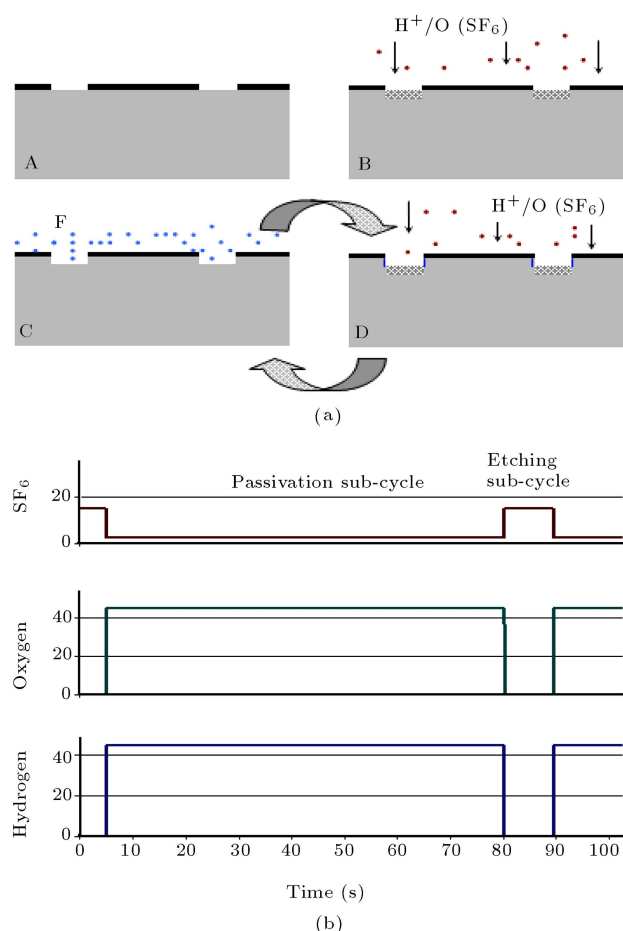


Figure 1. (a) Process steps of deep silicon vertical etching (from A to D). The etching requires a passivation subcycle plus an etching sub-cycle to achieve a deep vertical etching. (b) A schematic gas flow for three gases used in this technique. Typical gas flow rates are 45:45:3 sccm for $H_2:O_2:SF_6$ gases during the passivation subcycle, respectively. During the etching subcycle, only SF_6 flows into the chamber with a flow of 15 sccm.

shape and depth of the crater. As seen from this figure, during the passivation step, all three gases are introduced into the reactor. Typical values for the O_2/H_2 mixture as the main constituent in this period are 45-50 sccm, whereas SF_6 flows with a low value of 3 sccm. The plasma power is another important parameter in this subcycle and it has a typical value ranging from 150 to 250 W during this subcycle. The duration of this subcycle can be widely varied, ranging from a short treatment of 30 seconds in high speed etching to 120 seconds for high resolution and high aspect ratio etching processes. The etching subcycle is achieved by switching off the H_2/O_2 gases and increasing the value of the SF_6 flow rate to 15-20 sccm. The duration of this subcycle is less and ranges between 10 and 20 seconds, with a typical value of 15 seconds and a typical plasma power of 140 W. Usually, two intermediate purging steps are needed to

ensure that proper switching between gases is achieved. These steps might be as short as 5 seconds, although typical values are 15 seconds for each. So the total duration of an etching cycle can range from 55 to 160 seconds. The overall etch rate can be between 0.25 to 0.75 $\mu m/min$, although higher etch-rates up to 1 $\mu m/min$ are expected.

RESULTS AND DISCUSSIONS

Vertical Etching

Figure 2 collects several SEM images for the etching process prepared for this study. The top-right image in this figure corresponds to a high aspect ratio etched thin rod. The thickness of the rod is 150 nm and the

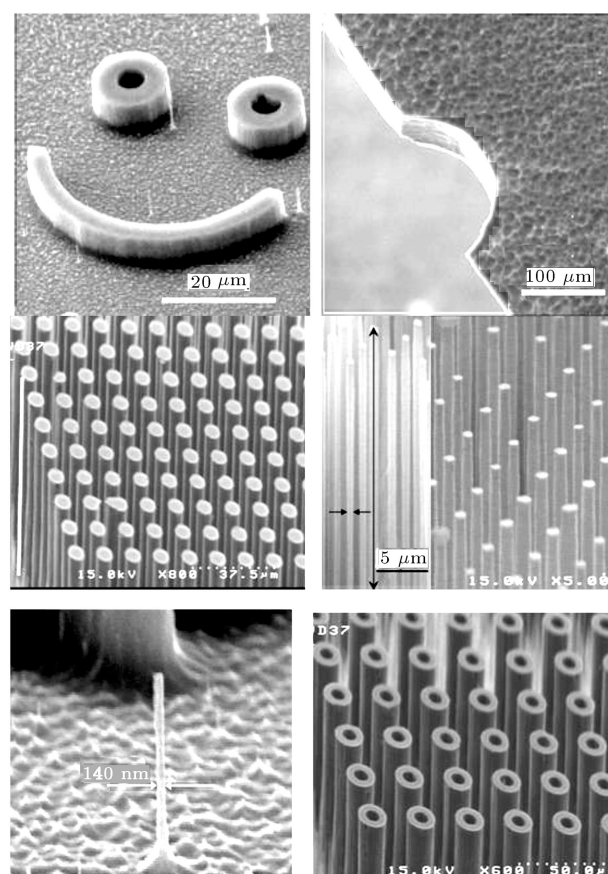


Figure 2. The collection of several SEM images of samples processed by silicon vertical etching with various heights and sizes. The smiley face structure has 8 μm height and aspect ratio of 30:1. Deep etching down to 100 μm is also observed in this figure. Arrows in the middle images show the height (25 μm) and width (500 nm) of rods. These images represent a highly ordered array of deep sub-micrometer rods and needles with sizes below 300 nm and heights over 20 μm . The bottom-left image belongs to vertical nano-metric features with different sizes down to 140 nm and an aspect ratio of the order of 50.

height of the structure is around $6\text{ }\mu\text{m}$. In addition, other small features, like a smiley face, have been vertically etched with a high aspect ratio of more than 30:1. The evolution of vertical holes with a height of $100\text{ }\mu\text{m}$ is observed in the bottom-right image. The opening of the hole is about $120\text{ }\mu\text{m}$ and the under-etching is less than $5\text{ }\mu\text{m}$. The bottom images in this figure belong to arrays of silicon nano-rods where highly ordered features with a height of more than $20\text{ }\mu\text{m}$ and width less than 300 nm are observed. Figure 3 depicts the result of the vertical etching of silicon using this technique to study the size-dependent etch rate or the so-called RIE lag. As seen from this image, the etch depth for an opening of $1\text{--}2\text{ }\mu\text{m}$ is slightly less than the etch depth for a higher opening of $20\text{ }\mu\text{m}$. For this experiment, the etch depth is around $20\text{ }\mu\text{m}$ and a modest drop is observed for smaller openings. Figure 4 shows the SEM images of ultra small features down to 100 nm .

In order to observe the effect of the passivation time on the total etch rate, we have selected the etch

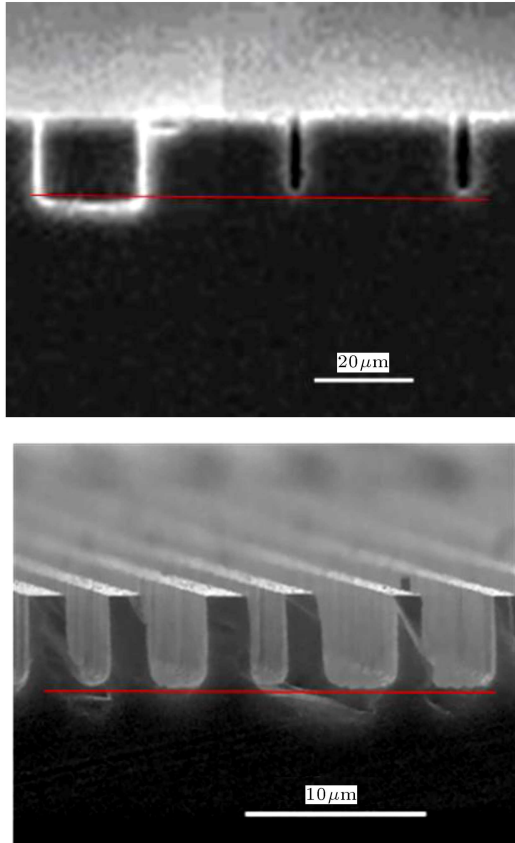


Figure 3. The observation of RIE lag for small openings (top) and channels (bottom). For a small opening of $1\text{--}2\text{ }\mu\text{m}$, there is a slight drop in the etch rate. The red line in both figures compares the bottom of a large opening with smaller features. As seen, the depth of the narrower opening is slightly less than the larger windows, which is called “RIE-lag”.

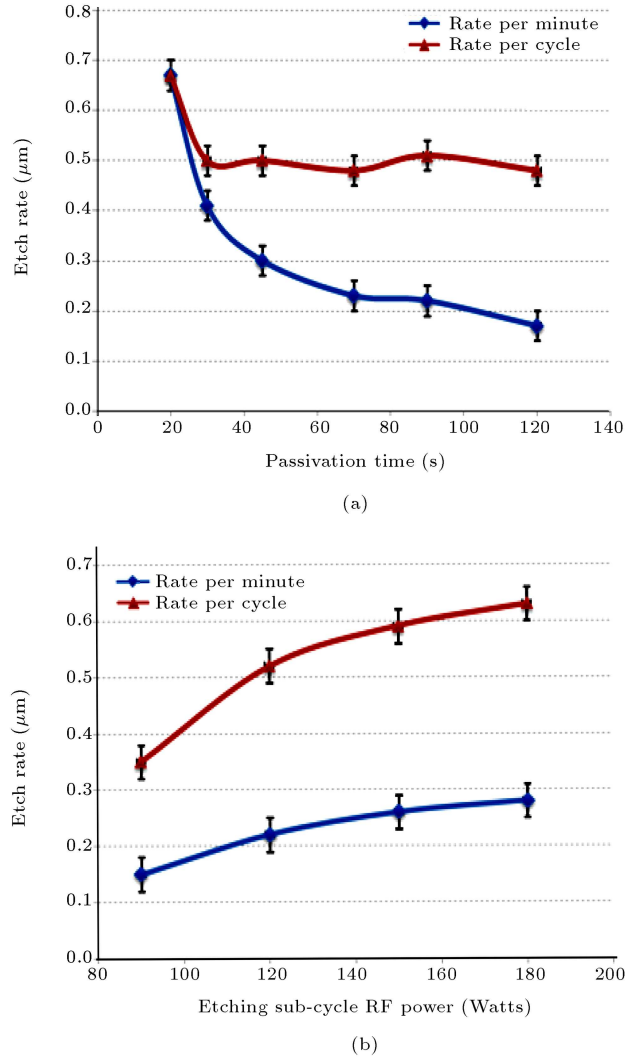


Figure 4. (a) Etch rate versus passivation time where the duration of the etch sub-cycle has been fixed at 15 seconds. If the passivation time exceeds 2 minutes, etch rate drops considerably to less than $0.2\text{ }\mu\text{m}/\text{min}$. (b) Etch rate versus RF power during the etching sub-sequence. As seen by raising the plasma power, the total and sequence etch rates greatly increase.

parameters as given in Table 1. The etching sub-cycle has a determining effect on the total etch rate and mask under-cut. An important parameter in obtaining three-dimensional structures is the passivation time whose effect on the etch rate is given in Figure 4. As seen from this figure, by going to higher passivation times, the rate per sequence drops due to further passivation of the crater bottom. However, the total etch rate shows a more magnified drop mainly due to the addition of extended time to the total etching period. A maximum etch rate of $0.65\text{ }\mu\text{m}/\text{min}$ is observed from this curve obtained for a passivation sub-cycle of 15 seconds. The etching sub-cycle has been set at 15 seconds. with a plasma power of 150 W over an area of $15 \times 20\text{ cm}^2$. It is worth mentioning that higher etch rates can be

Table 1. Etching parameters selected to investigate the effect of sweeping the passivation time.

Sub-Cycle	Duration (Sec)	RF Power (Watts)	SF ₆ Flow (sccm)	O ₂ Flow (sccm)	H ₂ Flow (sccm)	Chamber Pressure (mTorr)
Passivation	20-120	200	3	45	45	380
Etching	10-15	150	16	0	0	120

obtained by increasing the duration of the etching of the sub-cycle. For instance, by raising this time to 20 seconds and keeping the passivation time at 30 seconds, it has been possible to obtain a total rate of $0.75 \mu\text{m}/\text{min}$.

Three-Dimensional Etching

Figure 5a depicts the SEM image of a sample with a severe under-etch, which leads to complete damage of the original mask and the obtaining of an undesired crown-like structure out of the gear mask. To obtain such a sample, the hydrogen gas has been switched off during the passivation sub-cycle and no sign of vertical etching is observed. In Figure 5b, the same structure has been examined with a lower amount of underetch, suitable for three-dimensional fabrication. For this sample, the hydrogen gas flow has been set at 15 sccm, as opposed to 45 sccm for a normal vertical etching

process. Controlling the underetching is an important factor in the formation of highly featured, three-dimensional structures. By properly adjusting the H₂/O₂ plasma conditions during the passivation sub-cycle, the desired 3-D shapes are realized. Interestingly, it is possible to realize a reverse underetching, which we refer to as recovery, by similar controlling tools. In Figure 5d, a combination of underetching with vertical removal is depicted. The arrow in this figure points out the recessed region due to a desired underetching step. Once the gas flows have been set back to their optimum values for vertical etching, sharp walls are obtained.

Figure 6 collects many samples prepared, using this underetching/recovery technique. Figure 6a shows an unusual reversed glass-like structure where the initial intentional underetching has been fully recovered

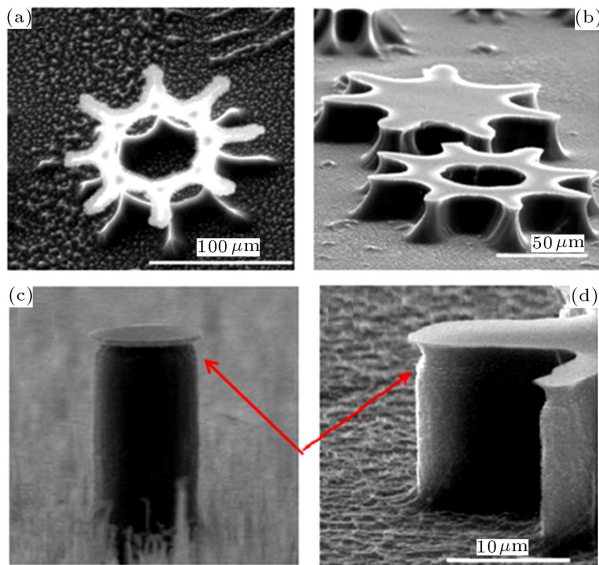


Figure 5. (a) Severe under-etch results in a crown-like structure. The gear-like chromium mask is remained on top of the structure. (b) The same structure with a lower amount of under-etch suitable for three-dimensional fabrication. The control over underetching is an important factor for the formation of highly featured three-dimensional structures. (c) and (d) Similar structures etched with some under-etching at the first steps, followed by high aspect ratio vertical etching. Arrows show where the under-etch has recovered.

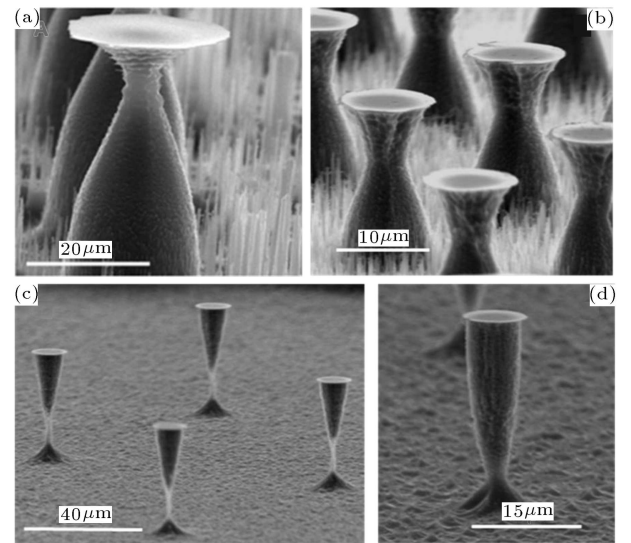


Figure 6. Some silicon 3-D structures obtained from controlled under-etch during the vertical (anisotropic) etching process. (a) An unusual sample, where the initial underetching has been recovered and the remainder of the process has been vertical. Elimination of hydrogen during the passivation process for the first five etching sequences has led to the severe underetching, whereas for the remaining 45 sequences, the recipe is the same as that used for the results in Figure 2. (b) The same shapes, but in this case, the first 5 steps of etching are vertical and, thus the underetching is observed at a lower height. (c) and (d) are opposite in the sense that initial etching sequences (40) are vertical and the last 10 sequences are without a hydrogen flow to create underetching.

and a vertical etching has been evolved. For this sample, the initial etching has been carried out by turning “off” the hydrogen gas to minimize the passivation of the side-walls. As observed, the sample has undergone a severe underetching over this period, which took “ten” etching cycles. After this underetching period, the hydrogen gas has been turned “on” and the normal etching process has been set. Under such conditions, the further progress of underetching has stopped and a recovery is observed. After 25 cycles of normal etching, the original round mask has been recovered and the further progress of the etching results in an almost vertical structure. The presence of grass-like features or black-silicon at the very bottom of the sample surface is observed in this figure, which could be diminished by a quick exposure to SF_6 plasma with a low power of 100 W. Other images of this figure belong to cases where a vertical etching process has been practiced, initially followed by a low hydrogen gas flow to ensure moderate underetching over the subsequent cycles. It is worth mentioning that similar shapes can be obtained using a micro-stereo-lithography technique. However, a polymeric substance is used for such a 3-D configuration instead of silicon substrates.

In Figure 7 we have shown schematically how complex 3-D structures are realized and repeated. Steps “A” to “F” are needed to form a curved structure. It must be borne in mind that the depth of the curved sidewalls can be as much as $20\text{ }\mu\text{m}$, where many etching/passivation cycles are involved. After obtaining the first curved feature, the sample is thermally oxidized in a dry-oxide furnace and the bottom SiO_2 is removed using direct-current plasma hydrogenation carried out in a dc-plasma reactor. This step is crucial

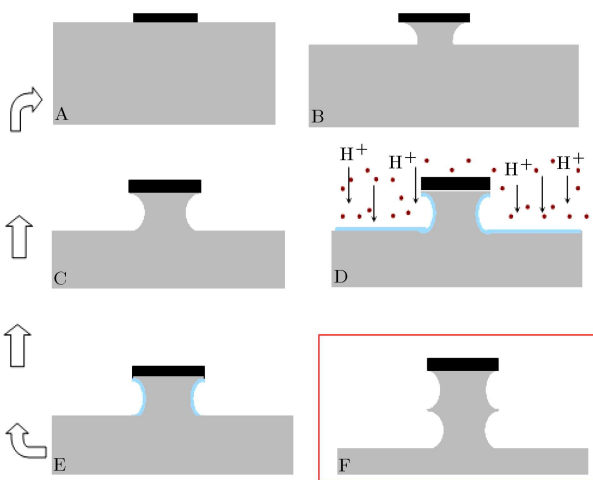


Figure 7. The process steps of a desired 3-D shape silicon etching, starting with mask patterning (A), followed by etching with desired under-etching (B) and recovery (C), oxide passivation (D) and removal from the bottom (E). The final structure depends on the number of times these steps are repeated (F).

to remove the oxide layer from the very bottom of the craters and can be obtained by a dc power of $1\text{ W}/\text{cm}^2$ in hydrogen plasma at a pressure of 1-5 torr. Once this oxide layer is partially removed, a quick dip in buffered HF (hydrofluoric acid) ensures the complete removal of the bottom oxide. Care should be taken not to affect the side-oxide to make sure that the subsequent etching will not affect the remaining features of the previous under-etching/recovery stages. At the present time, we are trying to avoid such an external oxidizing step by better optimization of the etching process.

Figure 8 collects a set of SEM images for several samples, which have been prepared with three

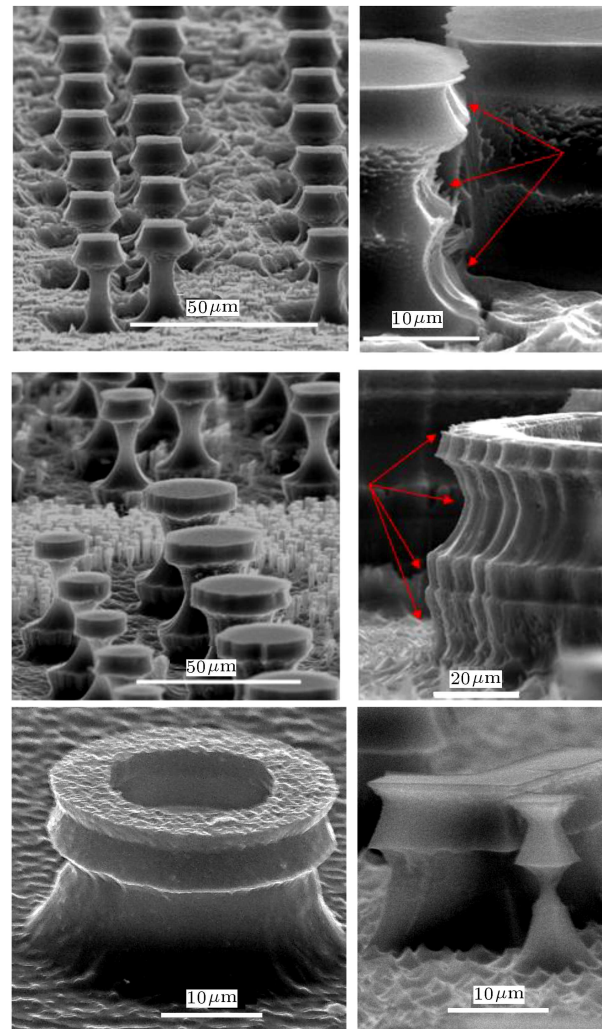


Figure 8. SEM images of desired 3-D shape silicon etching. The under-etching has been as much as $8\text{ nm }\mu\text{m}$. By controlling the main gases, it has been possible to recover the recessed side-walls and obtain the original mask. The results presented in this figure correspond to one initial step of vertical etching (top), followed by one or two steps of under-etching/recovery. The curved and highly featured edges are evident from this figure.

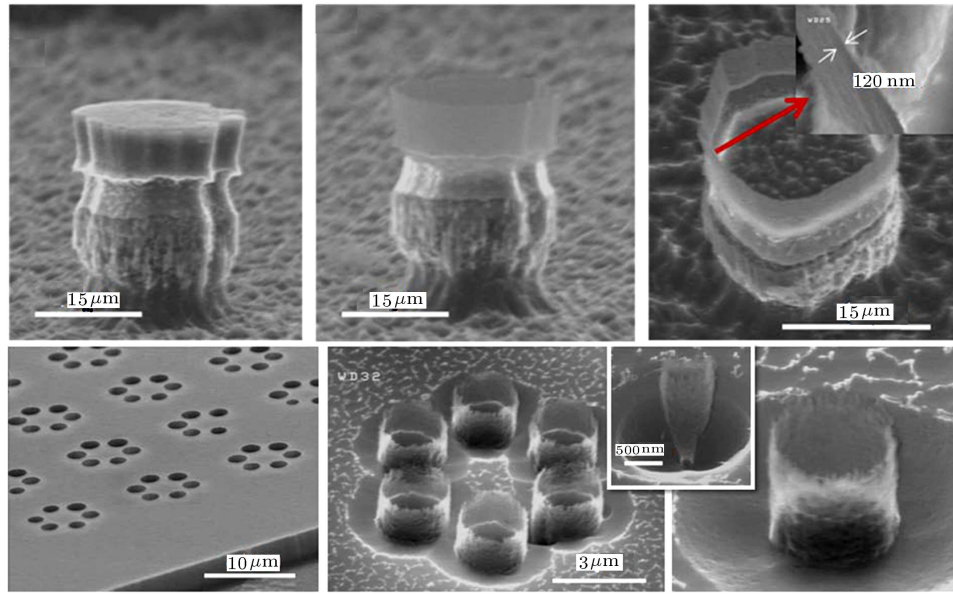


Figure 9. The evolution of highly featured, three-dimensional nano-wall structures. The top images belong to the formation of nano-metric featured Si/SiO₂ with a wall thickness below 120 nm. The bottom images show the formation of ultra thin curved cup-like structures. The inset in the bottom line of the images depicts an ultra fine 3-D glass like SiO₂ nanostructure, where features around 100 nm are observed.

sequences to form highly featured structures. As seen from these images, the overall underetching has been as much as 7 μm in some of the samples. By controlling the main gases, it has been possible to recover the recessed sidewalls and obtain the size of the original mask. The results presented in this figure correspond to one initial step of vertical etching (top) followed by one or two steps of underetching/recovery. The curved and highly featured edges are evident from this figure. In addition, Figure 9 shows a collection of several SEM images of the samples, where 3-D nano-walls are easily observed. In this image, the thickness of the SiO₂ walls has been measured to read a value of 120 nm. Such 3D structures can be used for polymeric molding, photonic crystal fabrication and mechanical resonators, in which the complex geometry of the vertical rods would affect their resonance behavior. Molded polymeric 3D micro-structures obtained from this technique can be used as scaffolds for tissue engineering [22-23]. Further investigation into this technique to obtain nanometric features, to integrate the passivation in the same reactive ion etching machine, and to fabricate polymeric 3D structures is being pursued.

SUMMARY AND CONCLUSION

We have successfully demonstrated three-dimensional nano and micro-structures on silicon substrates using a novel deep reactive ion etching technique. This technique allows the high aspect ratio vertical etching of silicon substrate with no need for a polymeric coating or high-density plasma. Instead of a polymeric coating

for the passivation subcycle, a mixture of hydrogen, oxygen and SF₆ gases is used, which not only protects the sidewall, but also improves the verticality by bombarding the exposed surface of the silicon substrate. The use of these gases allows a unique feature which allows the recovery of the underetched sidewalls. By controlling the gas flows during the passivation subcycle, it is possible to achieve the desired underetching, which can be recovered by proper setting of the gases in subsequent etching steps.

The formation of highly featured three-dimensional structures with SiO₂ nanowalls has also been demonstrated. These structures can be used in designing the photonic crystal arrays of vertical-standing rods, where apart from the desired order in the X – Y plane, the order in the Z-direction adds up to the filtration capabilities of the array. The formation of ultra small features sheds light on the formation of more complex nano-structures suitable for single-electron and nano-electromechanical (NEMS) applications. By combining the polymeric molding and 3-D features of the fabricated structures, useful structures for tissue engineering can be realized. The fabrication of more complex structures and smaller features is being pursued.

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BIOGRAPHIES

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