

Three-Dimensional High-order Nano-rod Formation Using a Hydrogen-assisted Deep Reactive Ion Etching

Samaneh Soleimani, Somaieh Zanganeh, Soheil Azimi, Zeinab Sanaee, Shams Mohajerzadeh[#] and H. Taghinejad*

*Nano-Electronics and Thin Film Lab, School of Electrical and Computer Engineering,
University of Tehran, P.O. Box 14395/515 Tehran, Iran.
Fax: +98-21-88013201; Tel: +98-21-61114905

[#]mohajer@ut.ac.ir

ABSTRACT

We report the formation of three-dimensional nano-sized vertical features on silicon and amorphous silicon films by means of a hydrogen-assisted deep reactive ion etching. The thickness of amorphous silicon has been varied between 5 and 10 μm . The adhesion quality of relatively thick amorphous Si films has allowed deep micro and nano-machining features on silicon and glass substrates. Apart from optical photo-lithography, high precision nano-sphere colloidal lithography and electron beam lithography has been exploited to realize ultra-small features on the amorphous silicon. Scanning electron microscopy has been extensively used to study the evolution of three-dimensional features. Such 3-D structures are suitable for future MEMS and NEMS-based devices on glass and silicon substrates.

Keywords: Silicon nanorods, amorphous silicon films, DRIE, colloidal lithography, three-dimensional features.

1 INTRODUCTION

The use of hydrogenated amorphous silicon (a-Si:H) has found great applications such as thin film transistors (TFTs), solar cells, lithium ion batteries and RF-ID. The main advantage of a-Si:H over its crystalline silicon counterpart is its low temperature processing which is an important requirement for devices containing materials with high temperature limitations. Such structures are most suitable for large area devices which is mainly owing to its compatibility with glass and plastic substrates. Most recently the use of textured silicon-based structures has been of great attention for large area solar cell fabrication. Although silicon micro and nano-machining techniques have shown great advance in the past few years, the use of amorphous silicon as a medium for micro and nano-machining has not progressed in the past. Both the growth of thick amorphous silicon films as well as successful large area nano-machining of such material have been the reason behind this limitation.

As stated, amorphous silicon films enjoy the advantage of their large area deposition and processing steps and whence, the immediate application of conventional deep etching processing techniques such as Bosch-process and cryogenic etching is not well suited for this material. In this paper, we report the creation of high aspect ratio 3-D amorphous silicon microstructures using hydrogen-assisted deep reactive ion etching compatible with large area applications. Since the processing is achieved on amorphous silicon layers, it can be performed on SiO_2/Si layers as well as on glass substrates, although the use of flexible substrates as PET is conceivable. These 3-D amorphous silicon microstructures are speculated to have great applications in solar cells, lithium ion batteries, vertical transistors and soft lithography in which they can be used as desired molds, suitable for tissue and bio-engineering [1-3]. In addition to standard photolithography, we have used nanosphere colloidal lithography to fabricate vertical nanoholes and nanorods of amorphous silicon on SiO_2 underlayers. The optical spectroscopy, performed on such array of amorphous silicon nano-rods shows their potential applications in solar cells. To realize highly ordered arrays of amorphous silicon nano-rods, electron beam lithography has been exploited which is most suitable for photonic crystal applications.

2 EXPERIMENTAL STEUP

The a-Si:H films were deposited by a radio-frequency PECVD unit operated at 13.56 MHz. The substrates temperature is set at 300 $^\circ\text{C}$, heated from the bottom of the substrate via a ceramic heater. The pressure and power density were 2 torr and 0.3 W/cm^2 , respectively. A mixture of SiH_4 and H_2 gases with respective flows of 30 and 100 sccm is used as the source gas during the deposition. The thickness of the film depends on various parameters including gas pressure, plasma power and more importantly on the duration of the deposition process. Figure 1 shows a scanning electron microscopy image of a sample with a typical thickness of 11 μm of a-Si:H which has been deposited on glass at a temperature of 300 $^\circ\text{C}$ and for a duration of 5 hours. By reducing the deposition time, one can achieve thinner layers on glass or Si/SiO₂ substrate. Thick amorphous silicon layers are prone to peel-off and

crack formation and a suitable pretreatment is needed to achieve smooth layers with good adhesion to the substrate, a necessary condition for subsequent lithography and micro and nano-machining steps.

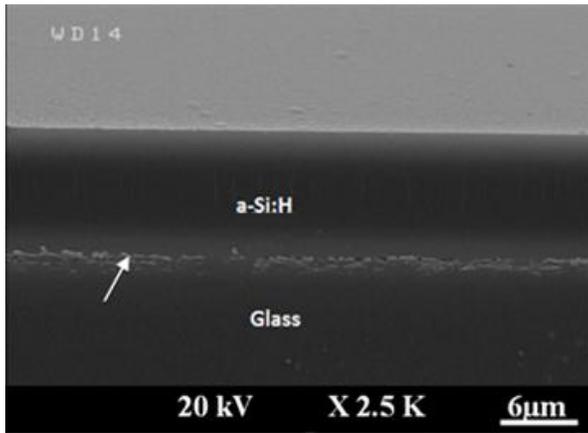


Figure 1: (a) The SEM image of 11 μm-thick a-Si:H film deposited on glass, indicating a smooth surface. Arrow points at the glass-film interface.

To improve the adhesion of amorphous silicon film on silicon substrate a pre-texturing process has been performed where a sequential two-cycle process is used. For this purpose, a thin silicon-oxide layer is first deposited using RF-PECVD method and it is then exposed to reactive ion etching to further process the layer. The SEM of the textured surface is shown in the following figure. Further details about the nanotexturing will be presented.

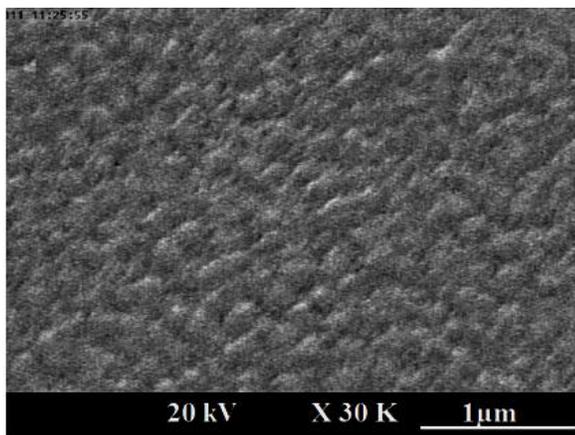


Figure 2: The SEM image of a highly textured oxide layer on silicon substrates to improve the adhesion of the subsequent processing. The deposition of a thick silicon layer (amorphous) is feasible on this textured surface.

3 THREE-DIMENSIONAL SILICON-BASED STRUCTURES

In this part, we report the formation of vertical and three-dimensional silicon-based structures on silicon and oxide substrates. The silicon substrates are coated with a layer of

Cr with a thickness of 10 nm and patterned using photolithography to act as the future hard mask in the DRIE process. In the case of amorphous silicon films, a strong adhesion is needed to endure the subsequent etching steps. The evolution of three-dimensional feature is possible by means of a recently reported hydrogen-assisted deep reactive ion etching [4] where a sequential etching and passivation process is used. Essentially this technique employs one step of passivation which is typically achieved by a combination of three gases of hydrogen, oxygen and SF₆. The process is achieved in a 13.56 MHz radio-frequency reactive ion etching unit. The plasma power is set at a value of 150W and the duration of the passivation sub-cycle is between 10 and 50 seconds. The etching sub-cycle is achieved using SF₆ as the feed gas where the plasma power is around 100 W. The duration of the etching could be 9 to 20 seconds. Since a time-multiplexed (sequential) etching process is exploited, the underetching and recovery steps can be highly programmed. The evolution of three-dimensional features on silicon substrates or amorphous silicon films is based on deep etching of silicon for which a programmable mixture of three gases of oxygen, hydrogen and SF₆ is used. More details about the etching process will be presented.

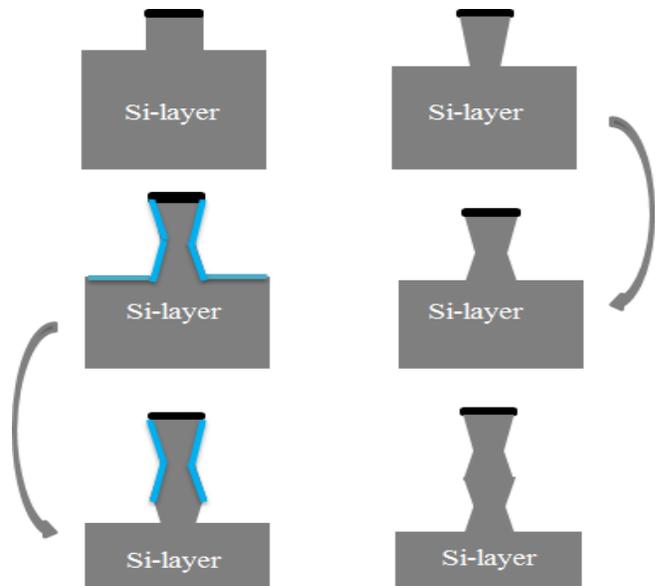


Figure 3: Schematic of 3-D etching process. After the mask definition (Cr here), the deep etching is performed. A suitable recovery step is practiced to come back to the original mask-size and shape. Then the silicon film is coated with a thin oxide and the process continues to form the second (and more) level of three-dimensionality. This process has been performed both on crystalline silicon as well as amorphous films.

Figure 4 shows a typical three-dimensional feature on crystalline silicon substrates. The array of highly-ordered 3-D features indicates the ability of the process to highly control the under-etching and reverse recovery steps. Inset demonstrates the cup-like structure made of hollow oxide around silicon stems. It must be born in mind that the presented result in this figure pertains to a crystalline

substrate where the etching has been performed on (100) silicon substrates.

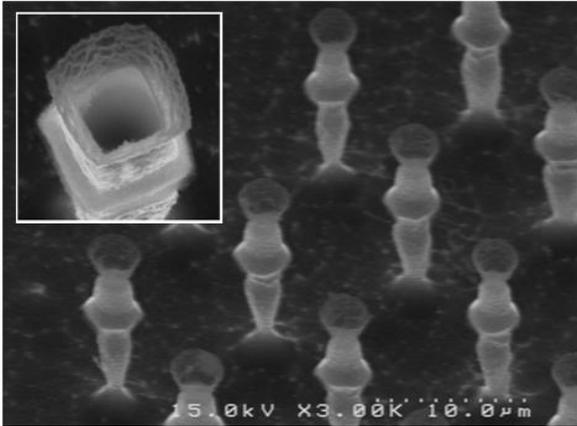
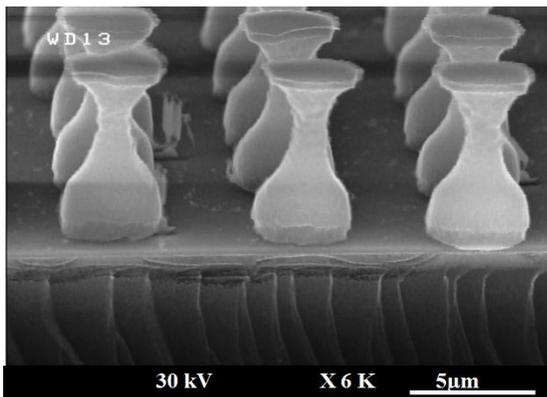
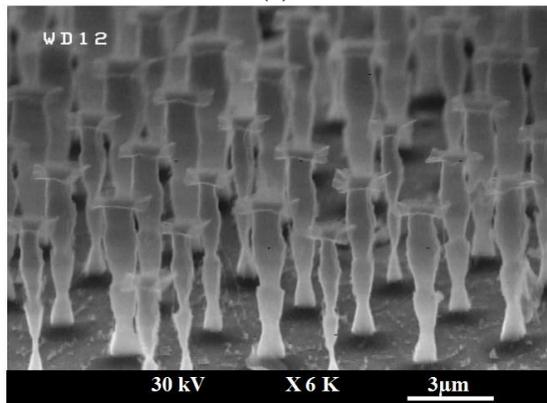


Figure 4: The formation of highly ordered three-dimensional features on silicon substrates. The curvatures on the vertical features can be controlled by the process parameters.

The etching process has been highly modified to be feasible on amorphous silicon layers which have been deposited on silicon or glass substrates. Extreme care should be taken into account to avoid peel-off of the amorphous silicon film as well as damage creation to the layer.



(a)



(b)

Figure 5: The evolution of highly featured structures made of amorphous silicon films. (a) Structures with one level of curvature and (b); a two-level curvature formation.

Figure 5 demonstrates, for the first time, the evolution of highly curved features made of amorphous silicon on an insulator (SiO_2 in here) underlayer. Since the underlayer is oxide, it is not much affected by the etching process. Apart from three-dimensional structures, we have processed ultra-small nano-rods on amorphous silicon films. Figure 6 shows the evolution of vertical nano-rods of amorphous silicon on silicon-oxide and glass substrates. Such structures have great potential for three-dimensional transistor fabrication on an insulating underlayer. For lithography purposes, we have used a nano-sphere colloidal lithography. Since both the lithography as well as the etching processes are compatible for large area applications, the evolved structures can be used for future solar-cell panels based on amorphous silicon films.

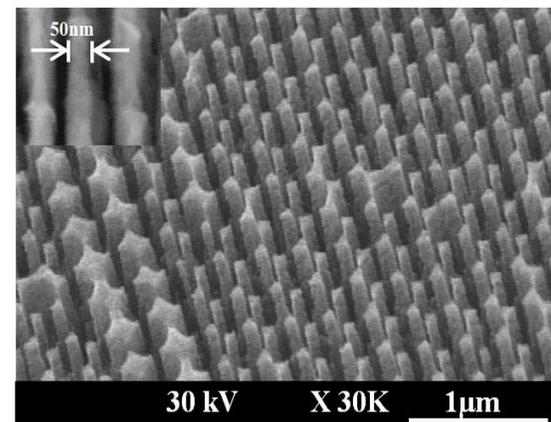
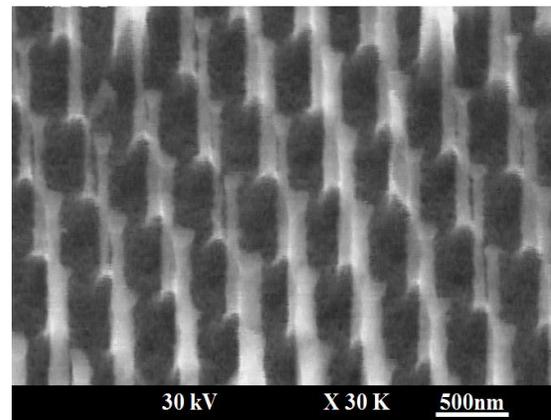


Figure 6: The evolution of high order vertical nano-rods made of amorphous silicon films on glass or oxide underlayers. As seen in the inset, the evolution of features of the order of 50nm with an aspect ratio of 10 is easily possible using this hydrogenation-assisted etching process.

In addition to SEM analysis, we have used transmission electron microscopy to observe the nano-rods and their amorphous silicon structure. Since a low power density plasma etching process is used, the amorphous silicon film is etched without being damaged. In addition, the outer surfaces of the nano-rod does not show any scallop. Figure

7 presents the TEM image of one of the rods. A random cleaving has been used for specimen preparation.

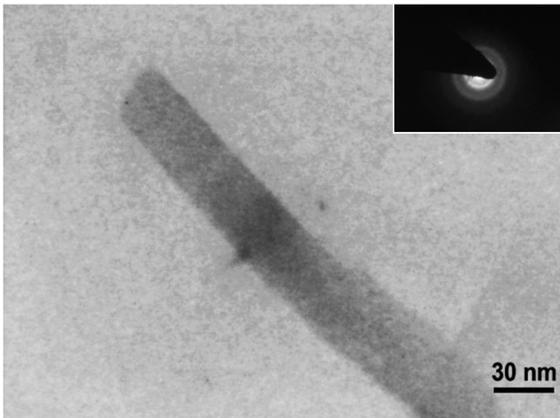


Figure 7: Transmission electron microscopy image of one of the process nano-rods indicating its amorphous structure.

4 CONCLUSION

In summary, we have fabricated three dimensional vertical structures on amorphous silicon by means of a hydrogen-assisted deep reactive ion etching. These 3-D amorphous silicon microstructures can have many applications in solar cells, lithium ion batteries, vertical transistors and soft lithography in which they can be used as desired molds, suitable for tissue and bio-engineering.

It has been found that by applying extra high power long time passivation subsequence, more complex three dimensional amorphous silicon structures can be obtained while upper structure is preserved by excess protective layer formed by extra passivation subsequence. Moreover, nanosphere lithography has been used for nano machining of amorphous silicon layer. Ordered amorphous silicon nanoholes and nanorods were successfully fabricated using a scalable and IC-compatible process. This work has been supported with a grant from Research Council of the University of Tehran.

References

- [1] J. Zhu, Z. Yu, G. F. Burkhard, C.M. Hsu, S.T. Connor, Y. Xu, Q. Wang, M. McGehee, S. Fan, Y. Cui, *Nano. Lett.* 9 (2009) 279-282.
- [2] S. Murugesan, J. T. Harris, B. A. Korgel, and K. J. Stevenson, *J. Chem. Mater.* 24 (2012) 1306-1315.
- [3] K. H. Cherenack, A. Z. Kattamis, B. Hekmatshoar, J. C. Sturm, and S. Wagner, *IEEE ELECTRON DEVICE LETTERS* 28 (2007) 1004-1006.
- [4] S. Azimi, A. Sandoughsaz, B. Amirsoleimani, J. Naghsh-Nilchi and S. Mohajerzadeh, *J. Micromech. Microeng.* 21 (2011) 074005.