

*Full Length Research Paper*

# Topography measurement of nano silicon oxide film

F. M. Nakhei <sup>1\*</sup> and A. Bahari <sup>2</sup>

<sup>1</sup>Department of Physics, Islamic Azad University, Sari Branch, Sari, Iran.

<sup>2</sup>Department of Physics, Faculty of Science, University of Mazandaran, Babolsar, Iran.

Accepted 07 May, 2009.

The Si (111) – 7 x 7 is a semimetal which its surface morphology has been normally studied with using STM (scanning tunneling microscopy) technique, whilst the other kind of silicon, such as Si(100) – 2 x 1 is not a semimetal. For letter surface structure, the AFM (atomic force microscopy) technique can be used as well. There are two main issues which are threatening the use of STM technique as a good technique for studying the silicon surface morphology. First issue is that STM suffers from the impossibility to decouple the anodization bias from the tip – sample separation and second issue is due to heating the sample surface in contact mode. For this purpose, we used AFM technique because it can be applied independently of the feedback and controlled with governing the tip – sample spacing. Moreover, AFM in tapping mode does not touch the sample and can thus eliminate lateral shear forces and overcome tip – sample adhesion forces (e.g. capillarity). In this case, we could grow homogeneous film on the silicon substrate.

**Key words:** Thin film, silicon dioxide, STM, AFM, surface topography.

## INTRODUCTION

Semiconductor is an important class of material both for industrial and for scientific study. Over the last two decades, they have come to be used in a wide range of electronic devices, such as transistors, switching devices, voltage regulation, photocells and photodetectors. The rapid growth of semiconductor industry has stimulated the demands for better material understanding and material quality. One device most widely used in silicon based integrated circuits is the MOSFET (Metal – Oxide – Semiconductor – Field – Effect – Transistor). The success of the device is attributable to several technology important factors. First, silicon can be thermally oxidized to produce a stable oxide which is an excellent insulator. Second, the surface state density at the silicon – oxide interface is sufficiently low to ensure reproducibility. Third, being planner, the structure is amenable to large – scale integration. In addition, nanoelectronic technology is reaching at a point that a little bit imperfection can cause a big problem in controlling the system and stopping it at the favorable time. We have thus demonstrated a series of experiments to find new device principles for continuing the process of miniaturization and integration well into the

current and next century (Quate, 1991; Avouris, 1993; Binh et al., 1993).

STM technique can usually perturb and damage film surface due to the tips (Quate, 1991; Avouris, 1993; Binh et al., 1993). Since the mechanism of the process and the factors that control its rate and resolution remain unclear, we have used an AFM with a conducting tip which is usually employed to produce homogenous oxides. This approach has also been used to fabricate simple device structures (Ma et al., 2001; Irmer et al., 1998).

For high resolution surface investigations, two commonly used technique is AFM. This technique resolves surface structure down to the nanometer scale. AFM consists of scanning a sharp tip on the end of a flexible cantilever across a sample surface while maintaining a small, constant force. By detecting the difference in the photodetector output voltages, changes in the cantilever deflection or oscillation amplitude are determined.

The two most commonly used modes of operation are contact mode AFM and Tapping mode AFM, which are conducted in air or liquid environments. Contact mode AFM consists of raster-scanning the probe (or sample) while monitoring the change in cantilever deflection with the split photodiode detector. The distance the scanner moves vertically at each point is stored by the computer to form the topographic image of the sample surface.

\*Corresponding author. E-mail: [al\\_bahari@yahoo.com](mailto:al_bahari@yahoo.com) or [a.bahari@umz.ac.ir](mailto:a.bahari@umz.ac.ir).

This feedback loop maintains a constant force during imaging. We have also studied the dependence of the reaction rate on the electric field strength and determine the factors that control the depth and the lateral extent of the oxide patterns, that is, the resolution of the process.

## EXPERIMENTAL DETAILS AND DISCUSSION

A scanning probe technique, AFM, which is very effective in visualizing the morphology of surfaces, has been used. It is clear that there is a repulsive force between a scanning tip and the surface. In AFM, forces between the sample and the probe are measured by the binding of a microscopic cantilever that is in the end has an atomically sharp tip. The cantilever bending is detected by the angular deflection of a laser beam reflected from the backside of the cantilever. The scanner raises the sample or lowers the cantilever by a preset amount, the "modulation amplitude" (usually 1-10 nm). In contrast to XPS (X – ray photoemission spectroscopy), excellent lateral resolution ( $\sim 10\text{\AA}$ ) and morphology of top layer can be determined by AFM technique, but it does not take us any chemical information. Of course, the tip effects and image artefacts should be taken into account in contacting mode. Based on the high lateral resolution in AFM, the top layer can be investigated in the detailed shape and even atomic resolution may achieve under ideal conditions. These properties avoid damages on the surface and improve imaging and lithography resolution.

The silicon samples (n-type, 5 $\Omega$  cm, 3  $\times$  1 cm) were cut out of wafers and introduced in the UHV chamber after a rinse with ethanol in an ultrasonic bath. The chamber was then baked before the experiments. After baking the background pressure was  $2 \times 10^{-10}$  Torr. Of course the pressure inside the vacuum chamber increases during the oxide growth on silicon substrate. All further cleaning was done inside the UHV chamber by heating with a direct current through the sample, initially up to 1200°C and later at higher temperatures to restore a clean Si surface. Earlier measurements with a residual gas mass spectrometer in the line of the beam have shown that a very high proportion (about 50%) of oxygen is produced with this setup. Typical total pressures in the chamber during exposure were around  $2 \times 10^{-7}$  Torr. The Si(100) substrate is kept at room temperature. The AFM-induced oxidation takes place at room temperature and, therefore, no viscous flow can take place.

Moreover, the silicon was cut from a silicon wafer with disorientation angle less than 1° from the (111) plane. The process was done by an image mode of reflection beam in AFM. It allows us to control roughness of the silicon or silicon oxide surface. But the surface was extremely flat. That does mean that large flat terraces were formed on the Si(100) surface. The average roughness of obtained terraces was measured by AFM less than 0.6 Å and the average distance between monatomic steps (terrace width)-as 3-4  $\mu\text{m}$ . The oxide thickness was

about 8 Å (Morgen et al., 2006; Bahari et al., 2005; Morgen et al., 2005). The AFM images were obtained in the resonant mode with platinum coated silicon cantilevers near to 230 Hz.

Hence, we have an insulator, silicon dioxide on silicon. Although we just show how its surface is, but it is possible to measure contact charging of insulators and charge transfer on and in insulators. That is important in nano-molecular electronics. It can be done with using a reproducible and controllable contact charging method (Irmer et al., 1998).

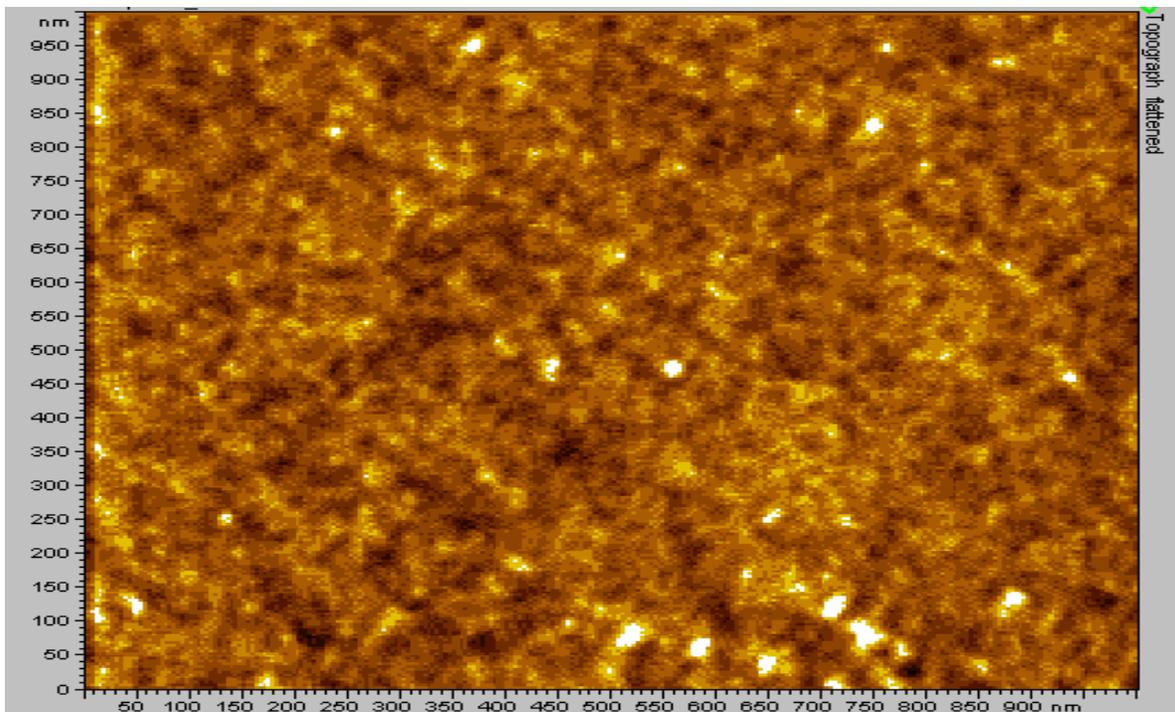
Contact charging should be performed by a contact between the silicon oxide surface and the conductive tip of an AFM cantilever. The thin oxide is a good candidate for this process, because after biasing tip and thin oxide on an electrically grounded Si substrate, contact – electrified charges can be deposited (Christou, 1994).

Almost all AFMs can measure sample topography in two ways: by recording the feedback output ("Z") or the cantilever deflection. The sum of these two signals always yields the actual topography, but given a well-adjusted feedback loop, the error signal should be negligible.

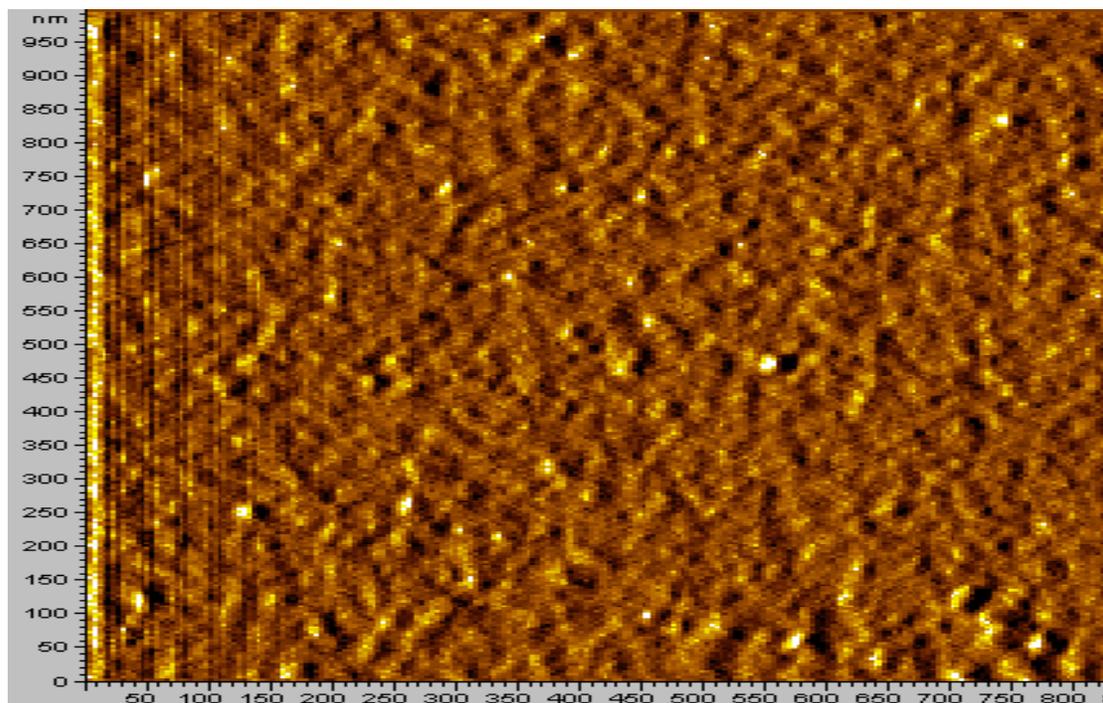
Figure 1 gives an example of patterned oxidation of silicon with an AFM in tapping mode. Given the different chemical properties of the oxide, subsequent chemical reactions can be used to produce patterns of different composition. But in Figure 2 aqueous HF is used to preferentially etch the silicon oxide surface. One of the most important pieces of information needed in considering the use of this oxidation process in fabrication is its intrinsic rate. It is also essential for elucidating the mechanism of the oxidation process. To determine the reaction kinetics, we stopped the tip over a surface site at a point and changed voltage between –2 and –20 V while the tip could move to a new position before each pulse (Irmer et al., 1998; Morgen et al., 2006; Bahari et al., 2005; Morgen et al., 2005; Bahari et al., 2006; Bahari et al., 2006; Christou, 1994).

We then imaged the oxide which selectively etched away by aqueous HF (Figure 2). In this way, we found an apparent volume expansion upon oxidation, which is higher than the anticipated increase of 2.3 for formation of amorphous SiO<sub>2</sub>. Furthermore, we measured the width and height of the resulting oxide dots. These kinetic measurements are shown in Figure 3 where the height of the oxide dots under the tip apex is plotted as a function of the new point position of the film which is achieved by changing the applied voltage. Therefore, the lateral oxidation and the line width determine that the lateral resolution is found proportional to the oxide height (Figure 3).

The fits to the data show some peaks. It indicates that the electric field plays an important role in the oxidation process. In estimating the field we have assumed that the entire voltage drop takes place across the oxide. In this view, the role of the electric field is to lower the activation



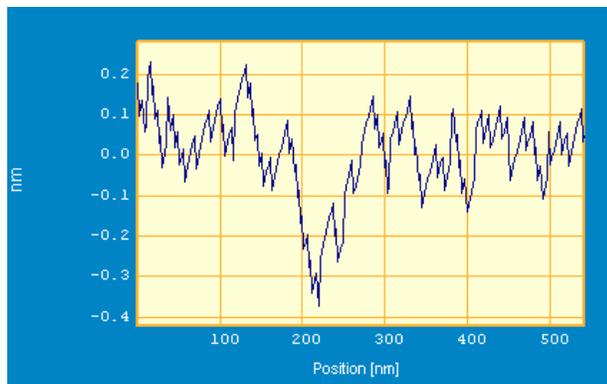
**Figure 1.** The topography patterned of oxidation.



**Figure 2.** The topography of silicon oxide surface which is selectively etched away by aqueous HF.

barrier for transport of ionic species across the oxide. The minima points observed here would then be attributed to the reduction of the electric field strength as the oxide thickness increases. An exponential dependence of the

oxidation rate on oxide thickness at the early stages of oxidation has also been observed in the case of thermal oxidation (Bahari et al., 2005). It is believed that the development of stress during oxidation causes the



**Figure 3.** AFM image of an identical type of sample; amplitude and frequency along the lateral line.

observed thickness dependence of the oxidation rate. The molecular volume of  $\text{SiO}_2$  is  $45 \text{ \AA}^3$  and that of Si is only  $20 \text{ \AA}^3$  (Aviouris et al., 1998). It is this doubling in volume which is responsible for the development of stress. New oxide formed at the Si /  $\text{SiO}_2$  interface must push against the already formed oxide. The observed discrepancy between the measured and expected volume expansion may be an indication that stress relief involves the formation of a rather open and defect-rich oxide. Furthermore, the increase of oxide height in Figure 2 is nearly constant over the whole dot.

We attribute the observed discrepancy to the finite electrical conductance of the water film that forms between the hydrophilic oxidized regions of the surface and the tip apex. Therefore, the large field gradients present at grain boundaries help in this transport and maximize these locations and help activate the chemical reactions.

## Conclusion

The electric field of a conducting AFM tip was used to induce oxidation due to arising of stress during oxidation, which in turn leads to thickness dependent activation energy. This procedure can be used for growing silicon oxide film without any defects of tip and anodization. Furthermore, we suggest using this procedure for other silicon orientations such as Si(100) –  $1 \times 1$  and so on.

## REFERENCES

- Aviouris P, Martel R, Hertel T, Sandstrom R (1998). AFM – tip – induced and current – induced local oxidation of silicon and metals, *Applied Physics A*, 66: 659 -68.
- Avouris P (1993). *Atomic and Nanometer Scale Modification of Materials: Fundamentals and Applications*, Kluwer Academic Publishers, Dordrecht.
- Bahari A, Morgen P, Li ZS (2006). Valence Band Studies of the Formation Ultra thin Pure Silicon Nitride Films on Si(100), *Surface Science*, 600: 2966- 74.
- Bahari A, Morgen P, Li ZS, Pederson K (2006). Growth of a Stacked Silicon Nitride /Silicon Oxide Dielectric on Si (100), *J. vacuum Sci. Techno. B* 24 :2119- 27.
- Bahari A, Robenhagen U, Morgen P (2005). Growth of Ultra thin Silicon Nitride on Si(111) at low temperatures, *Modern physical review B* 72: 205323 - 31.
- Binh VT, Garcia N, Dransfeld K (1993). *Nanosources and Manipulation of Atoms under High Fields and Temperatures*, Kluwer Academic Publishers, Dordrecht.
- Christou A (1994). *Electromigration and Electronic Device Degradation*, Wiley-Interscience, New York.
- Irmer B, Kehrl M, Lorenz H, Kotthaus JP (1998). Nanolithography by non-contact AFM induced local oxidation: Fabrication of tunneling barriers suitable for single electron devices, arXiv:cond-mat/9805350 v1 27 May.
- Ma YR, Yu C, Yao YD, Liou Y, Lee SF (2001). Tip-induced local anodic oxidation on the native  $\text{SiO}_2$  layer of Si(111) using an atomic force microscope, *Modern physical review B* 64:195324 - 33.
- Morgen P, Bahari A, Pederson K (2006). *Functional properties of Nano structured Material*, Springer 223: 229-257.
- Morgen P, Bahari A, Rao MG, Li ZS (2005). Roads to Ultra thin Silicon Oxide, *J. Vacuum Sci. Techno. A* 23: 201 - 9.
- Quate CA (1991). *Highlights in Condensed Matter Physics and Future Prospects*, Plenum Press, New York.