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Influence of Processing Parameter on Nanoscale Anodic Oxidation by AFM

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***Abstract*—Combining the pressure sensor's oxidation insulating nanostructure fabrication this paper studied the impact of bias voltage, ambient temperature and humidity on the size of oxide dots during the AFM-based anodic oxidation nano-fabrication. Experimental results show that the size of oxide dots increases with the increasing bias voltage and ambient humidity, but too high bias voltage and ambient temperature will cause staircase phenomena on the surface of oxide dot; ambient temperature 22 ℃, bias voltage 8V, humidity 50% and oxidation time 8s are relatively suitable processing parameters for the oxidation fabrication of n-type Si (100).**

Keywords-* *AFM; anodic oxidation; nanoscale

I. INTRODUCTION

Since the advent of atomic force microscope (AFM), it has been used as a new type of surface analysis instrument [1]-[5], providing a powerful tool for people to study matter structures and their interactions on nanometer scale. With the development of modern science and technology, there is a higher requirement for nano-fabrication technology and AFM-based anodic oxidation fabrication technology has become a hotspot of research, such as nano-dot and nano-gap fabrication, particularly such nano-electronic devices as single-electron transistor, point contact quantum devices and ultra-fast optical switches. Therefore, the paper used this method to fabricate pressure sensor's oxidation insulating nanostructure based on n-type Si (100), and researched the impact of bias voltage and humidity on the size of oxide dot.

II. EXPERIMENT

A. Principle

AFM-based anodic oxidation fabrication is similar to the conventional electrochemical anodic oxidation, that is, AFM conductive probe through chemical reaction between electric field induction and sample surface forms nano-scale oxidation structure. In the oxidation process, the probe's tip serves as the cathode of electrochemical oxidation [6]-[11], sample surface as the anode of the reaction, water molecules adsorbed on the sample surface as the electrolyte. Take Si fabrication for example, as shown in Figure 1, when the probe approaches Si surface, the water film between the two contacts to form water bridge, applying positive bias on the sample surface, and electrons from the tip's Fermi level transits into water film, reducing the water molecules on tip-sample surface to produce H^+ and OH^-, which under the effect of the electric field flow to Si surface and react:

$$Si + 2H_2O \rightarrow SiO_2 + 2H_2 \uparrow$$

SiO_2 is 10 ~ 100nm wide, 1 ~ 10nm high, and then by the moving tip can form the corresponding micro-structure graph.

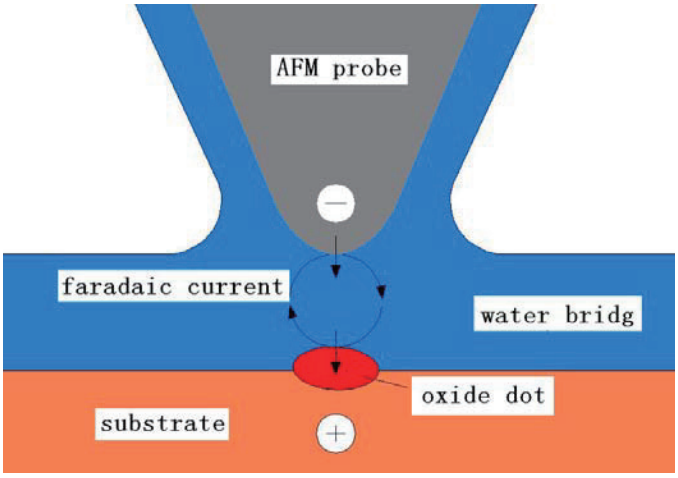


Figure 1. Schematic of AFM anodic oxidation nano-fabrication.

B. Process

The experiment used CSPM5500 multi-functional SPM system from Being Nano-Instruments LTD., NSG11/WuC conductive probe from Russian NT-MDT Company, used n-type Si (100) as experimental substrate, resistance rate of 5~7Ω·cm and surface Ra ≤ 0.5nm. The Si substrate was immersed in 2% HF solution for 5min for passivation to reduce its hydrophilicity and the number of water molecules adsorbed on the surface in order to reduce the interference caused by capillary action of water molecules [12]-[20].

The ambient humidity within AFM chamber is achieved by the external humidity controller with the control range of 40% to 70%, the smallest change in humidity of 1%. Detection of humidity is completed by AFM's own humidity sensor with the measurement range of 0 to 100%, accuracy of 0.5%. The experiment selected ambient humidity respectively 40%, 45%, 50%, 55%, 60%, 65% and 70%; external bias voltage 6.5V, 7V, 7.5V, 8V, 8.5V, 9V and 9.5V. The experiment had always maintained the contact mode, the temperature at 22 ℃, scanning speed 5μm/s. In order to ensure the accuracy of the experiment, oxidation was conducted at different positions on the substrate without replacing it in order to compare the oxidation effect of the dots.

III. RESULTS AND ANALYSIS

A. *The impact of bias voltage*

According to Cabrera and Mott's film oxidation theory [12], we know that the relationship between oxide dot's height h and bias voltage V is:

$$h = \frac{qa/KT}{\lg\left(\frac{h_L^2 vkt}{uW_{ox}qa}\right) - \lg V} V \qquad (1)$$

h is the actually measured height of the oxide dot; h_L is the maximum height of oxidation under the effect of electric field; v is scanning rate; k is Boltzmann constant; a is half of the width of the barrier; T is temperature; q is the electronic charge; W_{ox} is energy required for gap ion to overcome the diffusion; u is the change in temperature; V is bias voltage.

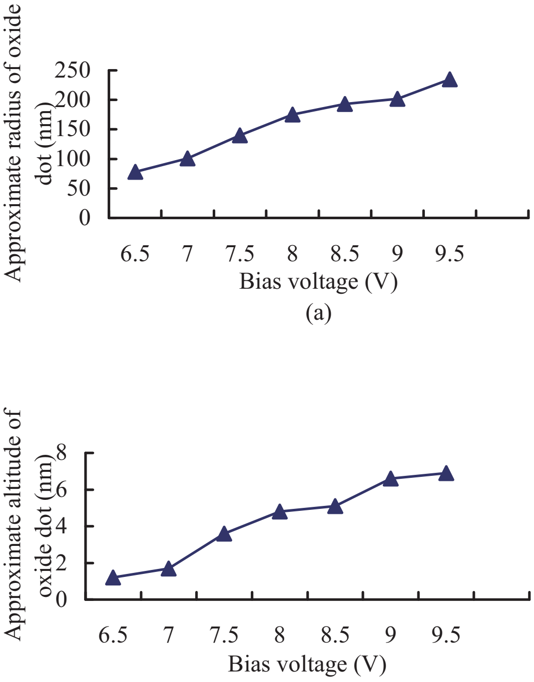
Formula (1) shows that the oxide dot's height h increases linearly with the increasing bias voltage V, which means the oxide dot's growth rate depends on the bias voltage.

In the experiment to verify the relationship between bias voltage and size of oxide dot, when the same conditions maintained that ambient temperature was 22 ℃, humidity 50% and oxidation time 8 s, different bias voltages (each change of 0.5 V) obtained such size of oxide dots as shown in Table 1:

TABLE I. SIZES OF OXIDE DOTS UNDER DIFFERENT BIAS VOLTAGES

Bias voltage (V)	*Approximate radius of oxide dot (nm)*	*Approximate altitude of oxide dot (nm)*
9.5	235	6.9
9.0	202	6.6
8.5	193	5.1
8.0	175	4.8
7.5	140	3.6
7.0	101	1.7
6.5	78	1.2

Table 1 shows the oxide dots size that the radius and altitude of the oxide dots was direct radio to bias voltage. And we can actually better visualize this if we plot how that oxide dots size changes as a function of bias voltage (Figure 2).



(a)

(b)

The relation between bias voltage and size of oxide dots. Bias voltage against (a) dot radius and (b) altitude.

The surface morphology of oxide dots obtained under different bias voltage conditions is shown in Fig. 3:

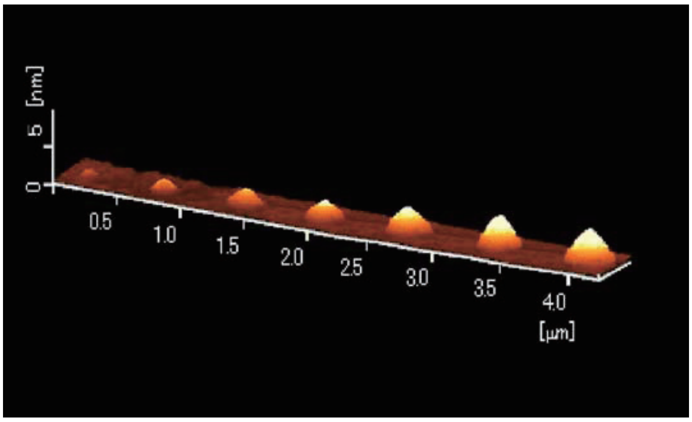


Figure 2. Oxide dot surface morphology under different bias voltages

Experimental results are consistent with expectations that the size of oxide dots increases with the increasing bias voltages.

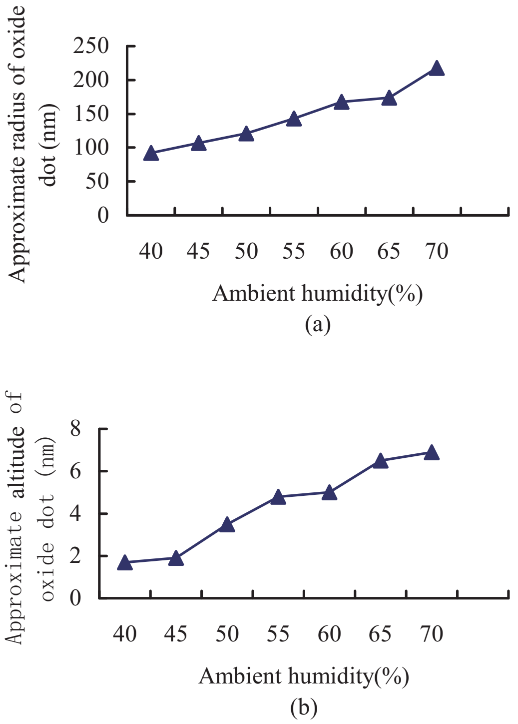
B. *The impact of humidity*

In order to learn the impact of humidity on the size of oxide dots, when the same conditions maintained that ambient temperature was 22℃, bias voltage 8V and oxidation time 8s the sizes of oxide dots obtained at different ambient humidity (each change of 5%) are shown in Table 2:

TABLE II. SIZES OF OXIDE DOTS UNDER DIFFERENT AMBIENT HUMIDITY

Ambient humidity (%)	*Approximate radius of oxide dot (nm)*	*Approximate altitude of oxide dot (nm)*
70	218	6.9
65	174	6.5
60	168	5.0
55	143	4.8
50	121	3.5
45	107	1.9
40	92	1.7

Table 2 shows the oxide dots size that the radius and altitude of the oxide dots was direct radio to ambient humidity. We also can plot how that oxide dots size changes as a function of ambient humidity (Figure 4).



(a)

(b)

Figure 3. The relation between ambient humidity and size of oxide dots. Ambient humidity against (a) dot radius and (b) altitude.

The surface morphology of oxide dots obtained under different humidity conditions is shown in Figure 5:

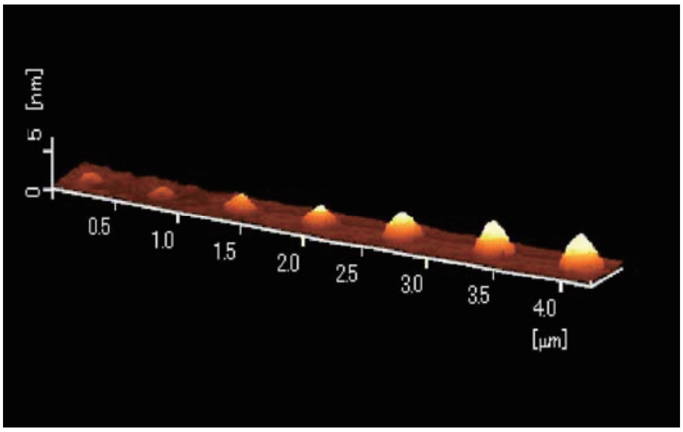


Figure 4. Oxide dot surface morphology under different ambient humidity

C. *Optimization of experimental parameters*

AFM-based anodic oxidation nano-fabrication technology should have a relatively appropriate parameters rather than blindly increase bias voltage and ambient humidity in order to get a larger size of oxide dots. In order to verify this conclusion, the author conducted oxidation under the ambient temperature of 22℃, the bias voltage 9.5V, humidity 70% and oxidation time 8 s to get the surface morphology of oxide dots shown in Figure 6:

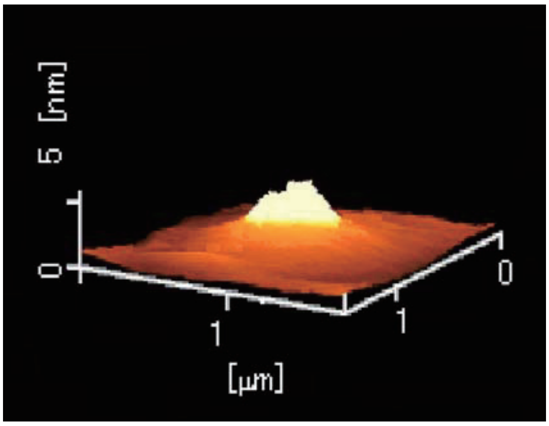


Figure 5. Oxide dot surface morphology under temperature 22 ℃, bias voltage 9.5V

Obviously, the higher humidity and bias voltage can indeed increase the size of oxide dot, but produces staircase phenomenon at the top. In this case, according to the results of the tests, by analyzing the size of oxide dots and variation curve of oxidation conditions, the author found that we keep the ambient temperature to be 22℃ and humidity to be 50%, and oxidation time is 8s, when bias voltage reaches 8.5V, it starting showing staircase slightly. However, when we keep temperature to be 22℃, bias voltage to be 8V and oxidation time is 8s, as soon as the humidity reaches 55%, it will also shows staircase slightly. Increased bias voltage leads the increasing of ions in water bridge, while increased humidity will also lead the increasing of oxygen negative ions during the reaction. Meanwhile, the size of oxide dot also depends on the sample material, probe shape and processing speed and other factors21-25. Based on the above analysis, the author under the conditions of ambient temperature 22 ℃, bias voltage 8 V, humidity 50% and oxidation time 8s fabricated pressure sensor's oxidation insulating nanostructure, shown in Figure 7.

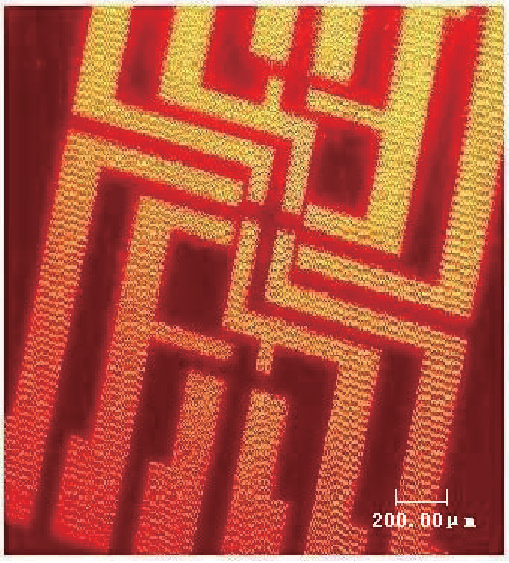


Figure 6. Oxidation insulating nanostructure of the sample

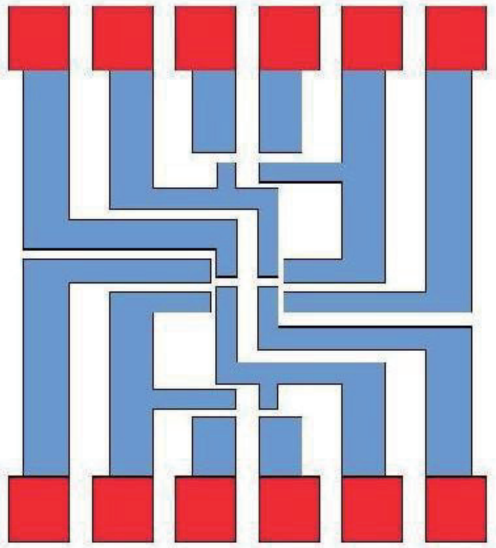


Figure 7. Design drawing of the sample

Fig. 8 is the design drawing. Contrasted with the sample we found that the oxidation insulating nanostructure is consistent with expectations. The surface roughness of it is not good, but its insulating efficiency is able to meet the requirement. In consequence, we conclude that the oxidation process is very well.

IV. CONCLUSION

During the AFM-based anodic oxidation nano-fabrication, the bias voltage, ambient humidity (and other environmental conditions), sample material, probe shape and the processing speed, etc. will all affect the size of oxide dots. Experimental results show that the size of oxide dots increase with the increasing bias voltage and ambient humidity, but too high bias voltage and ambient temperature will cause staircase phenomenon on the surface of oxide dot. Ambient temperature 22 ℃, bias voltage 8 V, humidity 50% and oxidation time 8 s are relatively suitable processing parameters for the oxidation fabrication of n-type Si (100).

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