

INVESTIGATION OF EFFECT OF DIFFERENT ANODISATION CURRENT ON POROUS SILICON BY STM

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ABSTRACT

Effect of different anodisation current on porous silicon is investigated by STM (Scanning Tunneling Microscope). Some porous silicon samples were prepared from p-type, boron doped, [100]- oriented silicon wafers with the resistivity of 5 Ω .cm. A porous layer was obtained by electrochemical etching in hydrofluoric acid (HF), water, ethanol solution with a 1:1:2 ratio.

During the etching, the applied anodisation current has been changed and the effect of anodisation current on porous structure was investigated. Therefore after the etching the STM images of porous silicon have been taken. During the etching process, it was observed that when the applied anodisation current rises, the thickness of wires increases with porosity thickness. It was also observed that the porous depth increases with the applied anodisation current.

Keywords: Scanning Tunneling Microscope (STM), Silicon, Porous, Etching.

FARKLI ANODİZASYON AKIMININ GÖZENEKLİ SİLİSYUM ÜZERİNE ETKİSİNİN STM İLE İNCELENMESİ

ÖZET

Bu çalışmada, farklı anodizasyon akımlarının gözenekli silisyum üzerine etkisi Taramalı Tünelleme Mikroskobu (STM) ile araştırılmıştır. Gözenekli silisyum örnekleri, Boron katkılı, p-tipi, [100] yönlü 5. Ω dirençli silisyum pullardan elde edilmiştir. Gözenek tabakası 1:1:2 oranlarındaki hidroflorik (HF) asit, su, etanol çözeltisi ile elektromekanik aşındırma yoluyla elde edilmiştir.

Aşındırma süresince uygulanan anodizasyon akımı değiştirilmiş ve anodizasyon akımının gözenekli yapı üzerine etkisi araştırılmıştır. Aşındırma sonrasında gözenekli silikonun STM görüntüsü alınmıştır. Aşındırma işlemi süresince uygulanan anodizasyon akımı arttığında, gözenek yapısının boyutları (derinlik ve genişlik) da arttığı gözlemlenmiştir.

Anahtar Kelimeler: Taramalı Tünelleme Mikroskobu (STM), Silisyum, Gözenek, Aşındırma

INTRODUCTION

Porous silicon (PS) is a material with very interesting and well studied optical properties [1]. Visible photoluminescence at room temperature was reported from highly porous Si films formed by anodic etching in aqueous HF, followed by extended immersion in aqueous HF [1]. This result raises the prospect of opto-electronic devices, such as light emitting diodes, based in Si [2]. Strain films on Si wafers produced in solutions of HF:HNO₃:H₂O have been studied over 40 years and have been suggested to be similar in nature to the anodically produced porous Si films first demonstrated by [3,4]. Porous Si was used for formation of thick SiO₂ layer in earl 1980's [5]. In 1984, an optical study was done on porous Si at liquid He temperature, but emission is assumed as amorphous silicon [6]. Formation of porous silicon from crystal silicon by etching in HF solution is called electrochemical method.

Several methods are developed for preparation of porous silicon, but electrochemical process is the most preferable of them. The single crystal Si layers are etched by anodically by solution, which is prepared with HF. A contrivance in order to produce porous silicon is shown in Figure 1.

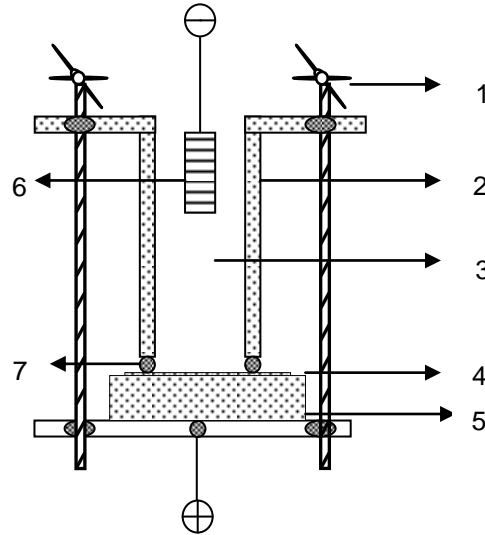


Figure 1. Production system of porous Si. 1. Compression unit 2. Teflon tank 3. Chemical solution 4. Crystal Si electrode 5. Copper plate 6. Pt electrode 7. Rubber packing

In Figure 1, platinum electrode is used as anode and crystal silicon as cathode. Surface morphology of etched crystal silicon depends on the type of doping, anodisation current and compound of solution. The mechanism, including the pore formation process is not fully understood [7]. Dispersion mechanism of porosity is well understood with the electric field line concentration at porosity edges and by way of charge carrier's diffusion from crystal Si to porous Si [8]. Many experiment results have been reported which mainly suggested the mechanism of light emission or the relation between the porous structure and photoluminescent (PL) characteristic [9]. Many studies contain surface defects, impurity, morphology and hydrogen contribution.

During the investigations it has been come clear that porous silicon is a vastly diverse and complex material with its properties depending on the details of its preparation, temperature and chemical treatment and condition of storage before measurement [10].

The anodisation of Si wafers at low current densities in HF-based solutions can be used to generate, an array of extremely small holes that run orthogonal to the surface. Bulk Si can be made in micro-porous (pore

width ≤ 20 Å), mesa porous (pore width $\leq 20-500$ Å) or macro porous (pore width >500 Å) depending on substrate resistivity and anodisation conditions.

Silicon in contact with HF as an electrochemical system has been used in microelectronic technology, but it has moved only into the scientific focus after it was shown that various porous layers with dimensions scaling from nanometers to micrometers can be obtained easily [11].

Electroluminescence has been observed in porous Si [12] and light emitting diodes fabricated from porous Si [13]. Larger current density results in stronger photoluminescence[9]. Thickness depends linearly on the current density at 100-1000 nm thickness range.

Application of porous silicon in different field of technology are outlined below [14].

LEDs, photodetectors, photonic crystals, waveguides and optical logic gates at optoelectronic application. Cold cathodes, intercomponents isolation of integrated circuits by oxidated porous silicon, epitaxial growth of crystalline silicon films on surface of porous silicon at microelectronic applications. Micromachining at sensors and actuators. Sensors based on electrical conductivity effect and sensors employing photoluminescence quenching at chemical sensor applications. Biological application of porous silicon.

EXPERIMENT

The PS layers were performed on n-type [100] oriented Si wafers (p doped) with resistivity 5-8 Ω .m. Samples were anodized in HF:H₂O:C₂H₅OH=1:1:2 electrolyte at 10-50 mA/cm² current density. Different samples and anodisation current densities are given in Table 1. Etching time is 5 minutes for all samples. After the etching process, samples are cleaned by deionised water to remove the localized HF in porosities. Production system of porous Si is shown in Figure 1. STM

pictures of porous Si, etched under different anodisation currents, are shown in Figure 2, 3, 4, 5, 6. Since porous silicon has low electrical conductivity, surfaces are excited with 150 watt Xenon arc lamp to increase the surfaces' electrical conductivity.

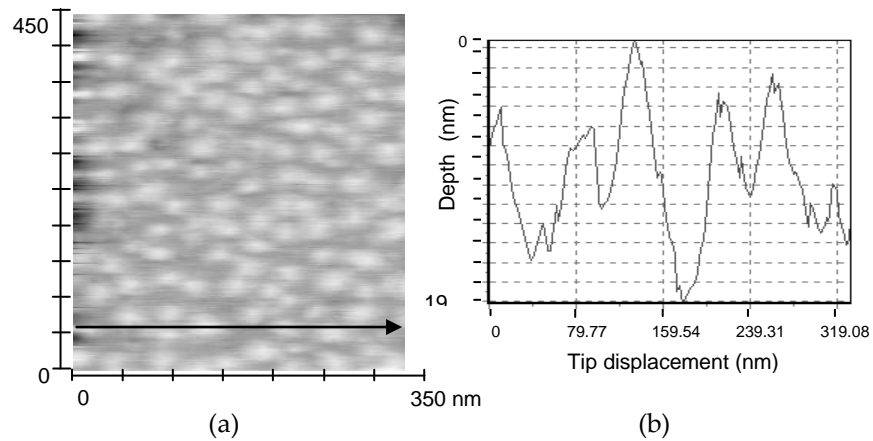


Figure 2. (a) Surface picture of porous Si etched by 10 mA/ cm² anodisation current. Scan size : (x=332.4nm)×(y=450.6nm) (b) Surface depth data

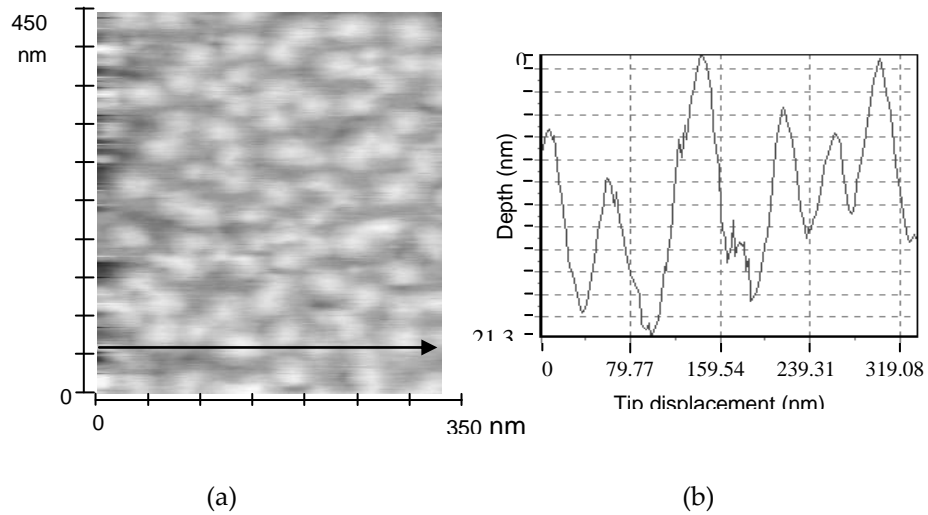


Figure 3. (a) Surface picture of porous Si etched by 20 mA/ cm² anodisation current. Scan size : (x=332.4nm)×(y=450.6nm) (b) Surface depth data

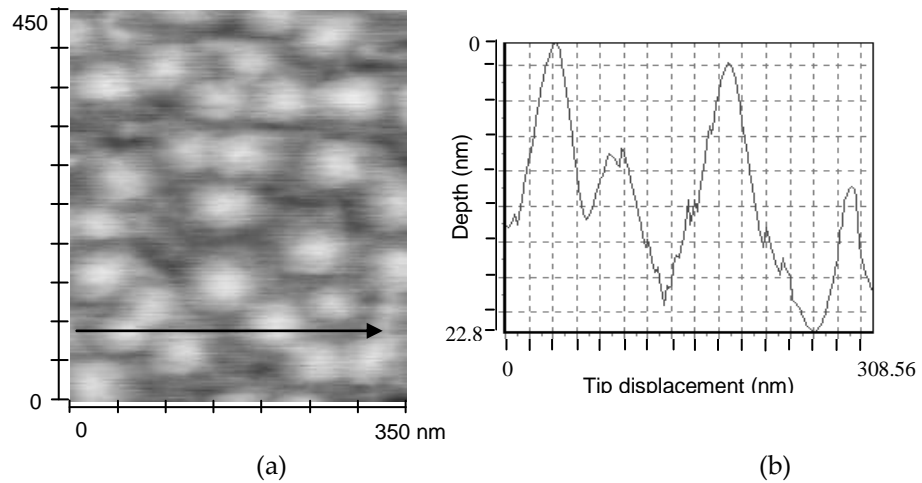


Figure 4. (a) Surface picture of porous Si etched by 30 mA/ cm² anodisation current. Scan size : (x=332.4nm)×(y=450.6nm) (b) Surface depth data

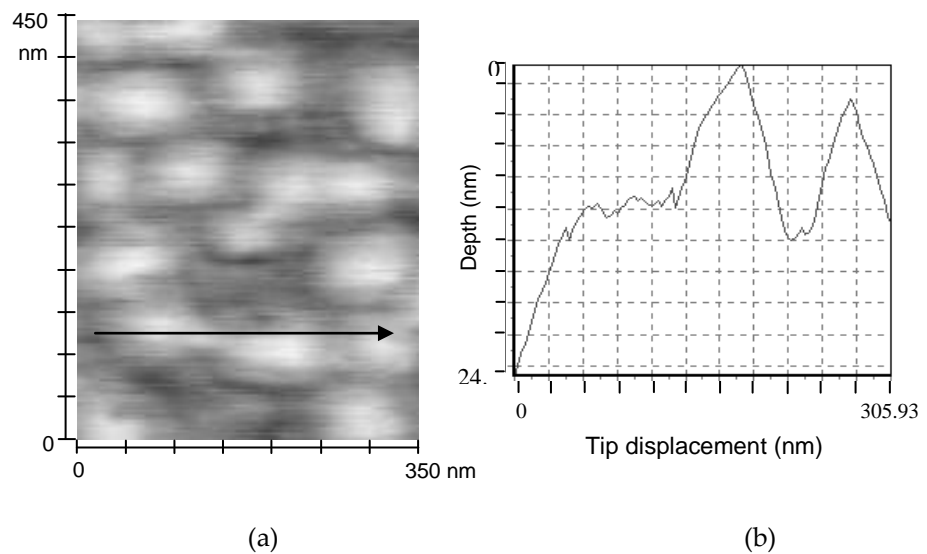


Figure 5. (a) Surface picture of porous Si etched by 40 mA/ cm² anodisation current. Scan size : (x=332.4nm)×(y=450.6nm) (b) Surface depth data

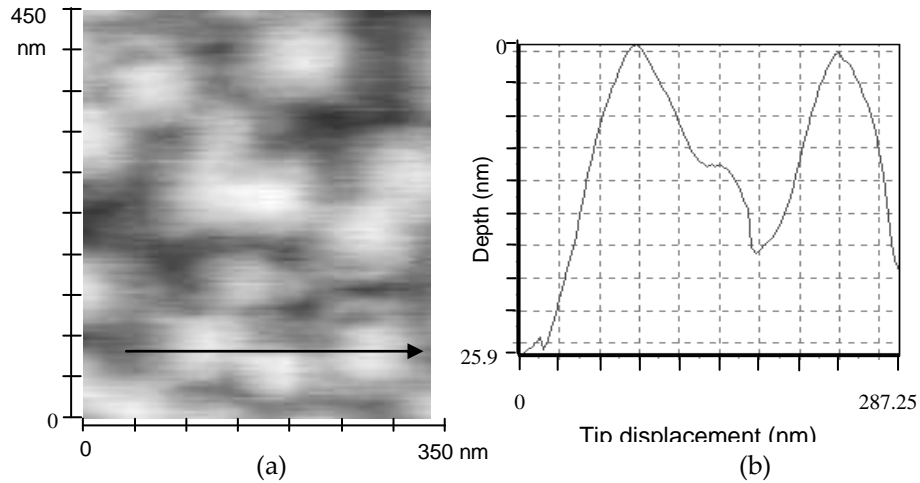


Figure 6. (a) Surface picture of porous Si etched by 50 mA/ cm² anodisation current. Scan size : (x=332.4nm)×(y=450.6nm) (b) Surface depth data

In the experiments, tunneling current (I_t) is adjusted to 5 nA and tunneling voltage (V_B) to 7 volt. In order to get pictures which are given in Figures 2,3,4,5 and 6, potential difference that is applied to the piezotube's electrodes has changed from 0 Volt up to ± 6 Volt. In this situation, displacement for x-axis which is done by electrodes is $(55.4 \text{ nm/V}) \times (6 \text{ Volt}) = 332.4 \text{ nm}$. Displacement of y-axis done by electrodes is $(75.1 \text{ nm/V}) \times (6 \text{ Volt}) = 450.6 \text{ nm}$.

RESULT AND DISCUSSION

Average size of wires, obtained from STM pictures is calculated. Size of wires is measured from the mid point of maximum depth.

The STM investigations show the nanometer scale Si wire substructure on the surface of the P-Si sample prepared at $I_a = 10 \text{ mA/cm}^2$ with the size of wires being equal nearly to 20-30 nm (Figure 2.a.) The average size of wires is 23.34 nm. At this anodisation current maximum porosity depth is observed as 19.824 nm from Figure 2.b.

At $I_a = 20 \text{ mA/cm}^2$ with the size of wires being equal nearly to 30-40 nm (Figure 3.a) the average size of wires is 34.48 nm. At this anodisation current maximum porosity depth is observed as 21.348 nm from Figure

3.b. At $I_a=30\text{mA/cm}^2$ with the size of wires being equal nearly to 50-60 nm (Figure 4.a) the average size of wires is 56.28 nm. At this anodisation current maximum porosity depth is observed as 22.87 nm from Figure 4.b. At $I_a=40\text{mA/cm}^2$ with the size of wires being equal nearly to 70-85 nm (Figure 5.a) the average size of wires is 78.53 nm. At this anodisation current maximum porosity depth is observed as 24.4 nm from Figure 5.b. At $I_a=50\text{mA/cm}^2$ with the size of wires being equal nearly to 70-110 nm (Figure 6.a) the average size of wires is 89.47 nm. At this anodisation current maximum porosity depth is observed as 25.92 nm from Figure 6.b.

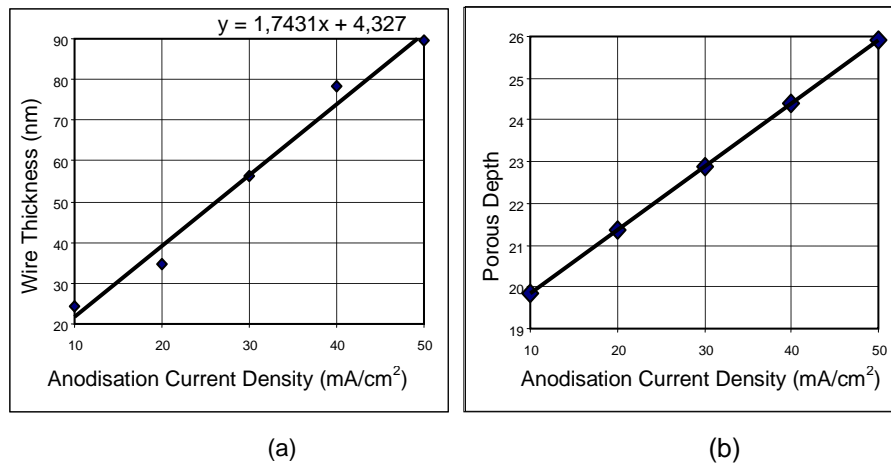


Figure 7. (a) Change of thickness of wires versus anodisation current density. (b) Change of depth of porous versus anodisation current density.

According to these data, Table 2 is prepared. According to the data, which is given in Table 2, the change of average size of wires and maximum porous depth with respect to the anodisation current are shown in Figure 7.a. and 7.b, respectively.

Table 1. Samples and anodisation current.

Sample No	Current Density (mA/cm ²)
1	10
2	20
3	30
4	40
5	50

Table 2. Changing of porous structure with respect to the anodisation current.

Current Density (mA/cm ²)	Size of Wires (nm)	Average Size of Wires (nm)	Maximum Porous Depth (nm)
10	20-30	23.34	19.824
20	30-40	34.48	21.348
30	50-60	56.28	22.870
40	70-80	78.53	24.400
50	70-110	89.47	25.920

As a result of STM pictures, it is observed that during process of obtaining porous Si when the applied anodisation current increases porosity and porosity depth increase. Porosity width changes with applied anodisation current depending on $y=1.7341.x+4.327$ function.

CONCLUSIONS

This study showed that by using STM, thickness and porosities change with current density linearly at 20-100nm thickness ranges.

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