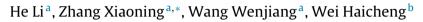
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The influence of oxidation properties on the electron emission characteristics of porous silicon



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ABSTRACT

In order to investigate the influence of oxidation properties such as oxygen content and its distribution gradient on the electron emission characteristics of porous silicon (PS) emitters, emitters with PS thickness of 8 μ m, 5 μ m, and 3 μ m were prepared and then oxidized by electrochemical oxidation (ECO) and ECO-RTO (rapid thermal oxidation) to get different oxidation properties. The experimental results indicated that the emission current density, efficiency, and stability of the PS emitters are mainly determined by oxidation properties. The higher oxygen content and the smaller oxygen distribution gradient in the PS layer, the larger emission current density and efficiency we noted. The most favorable results occurred for the PS emitter with the smallest oxygen distribution gradient and the highest level of oxygen content, with an emission current density of 212.25 μ A/cm² and efficiency of 59.21‰. Additionally, it also demonstrates that thick PS layer benefits to the emission areas of PS emitters can be enlarged and electron emission thresholds is decreased because of the higher oxygen content and smaller distribution gradient, which were approved by the optical micrographs of top electrode of PS emitters before and after electron emission.

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1. Introduction

The electron emitters based on porous silicon (PS) outperform conventional cold electron emitters in various aspects such as relatively simple structure, general processing, low operation voltage, ballistic emission, and insensitivity to gas pressure and gas species when emitting electrons [1]. It means that PS electron emitters have potential applications on flat panel display [2], optoelectronic device [3], image sensor [4], electron beam lithography [5], and electroplating [6], etc. The basic structure of the electron emitter is top electrode/PS layer/Si substrate and bottom electrode. The PS layer plays a key role in determining the emission characteristics because it provides tunneling to accelerate electrons injected from the substrate [7]. Because the PS layers are usually prepared in the HF solution, the silicon dangling bonds and vacancy defects would be formed when the corrosion current are abruptly interrupted at the end of the corrosion [8,9], only partial silicon dangling bonds are saturated by hydrogen in the HF solution. Additionally, the PS is particularly easy to absorb gas molecules due to its high specific

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http://dx.doi.org/10.1016/j.apsusc.2016.04.129 0169-4332/© 2016 Elsevier B.V. All rights reserved. surface area and the bonding requirements caused by the fracture of the Si-Si. Much of the impurity levels would be introduced into the PS forbidden band and subsequently lead to a large number of electron traps in the PS layer [10,11]. The electrons injected from metal bottom electrode are easily captured by the electron traps and scattered in the PS layers, which directly affects the electron emission characteristics of PS emitters. Therefore, the passivation treatment is required to stabilize the microstructure of PS layer and eliminate the defect levels and electron traps in the PS layers [12].

Oxidation is one of the most effective passivation methods for the PS layers. It can stabilize the microstructures of PS by changing its physical properties and achieve more suitable electric filed distribution in the PS layers which are helpful to electron acceleration [13,14]. In addition, the indispensable tunneling barriers for the electron emission can be formed after oxidation [15]. The PS layers with different thickness can be completely oxidized by selecting suitable oxidation methods [16–18]. Electrochemical oxidation (ECO) and rapid thermal oxidation (RTO) are the common oxidation methods for the PS layers. Because of the different thickness and microstructure of the PS layers, there is great difference in the oxygen content and the oxygen content distribution among PS emitters, which may produce significant influence on the distribution of the accelerating electric field and the electrons emission







Table I	
The prepared	parameters of PS samples A1 to C2.

Samp	e Corrosion			Oxidation	Oxidation	
	Volume (I	mAmin) Thick	ness (µm) Porosi	ty Method	Time (min)	
A1	90	8	48%	ECO	18	
A2	90	8	48%	ECO-RTO	18	
B1	60	5	67%	ECO	10	
B2	60	5	67%	ECO-RTO	10	
C1	45	3	53%	ECO	6	
C2	45	3	53%	ECO-RTO	6	

[19,20]. A suitable oxidation property is the key issue to obtain excellent electron emission characteristics for the PS emitters.

The PS emitters with thickness of 8 μ m, 5 μ m, and 3 μ m were prepared. Each thickness of PS emitters have two samples and they were oxidized by ECO and ECO-RTO (ECO before RTO), respectively. The influence of the oxygen content and its distribution gradient on the electron emission characteristics were investigated through analyzing oxidation properties of the PS layer and measuring the electron emission of PS emitters in the vacuum. The experimental results indicated that the higher oxygen content and smaller distribution gradient can enlarge the effective emission area and decrease threshold voltage of electron emission, it also benefits to the emission current density, efficiency, and stability. Electron emission characteristics are determined by the oxygen properties, which were approved by the FN plots.

2. Experiment methods

2.1. Sample preparation

The PS layers were formed by anodizing heavily doped $(0.02 \ \Omega \ cm)$ single-crystalline n type silicon wafers with an ohmic back contact in a solution of HF (40 wt%): ethanol = 1:1 at room temperature. The PS emitters with the PS thickness of 8 μ m, 5 μ m, and 3 μ m were prepared corresponding with electrochemical corrosion volume of 90 mA min, 60 mA min, and 45 mA min, respectively. In order to obtain different oxidation properties for all samples, two samples with same thickness were oxidized by RTO and ECO-RTO, respectively. At a certain porosity, RTO and ECO usually begin from the top and bottom of PS layer, respectively [18,21]. The ECO-RTO was chosen in order to reduce the interactive effect between ECO and RTO. If RTO-ECO (RTO before ECO) was chosen, the permeability of ECO solution would be reduced due to the PS surface passivation caused by RTO, which would directly affect the oxidation properties of the ECO.

The RTO and ECO were carried out at 900 °C under 1000 sccm oxygen, and 15 mA/cm² in 1 mol/L H₂SO₄ solution, respectively. The prepared parameters of PS emitters A1 to C2 are shown in Table 1. The porosity of the PS samples shown in Table 1 were measured by the gravimetric method. Both the adonization and oxidation were carried out under illumination by a 300 W tungsten lamp located at a distance of 20 cm to supply the holes that the reaction required. We hope to obtain different oxidation properties for the PS emitters with same thickness and thus can achieve the regular influence of oxidation properties on the electron emission characteristics based on this. Therefore, we did not try to optimize the oxidation parameters to oxidize all samples completely and uniformly. Subsequently, all PS emitters were annealed in nitrogen gas at 550 °C for 1 h. Finally, a 10-nm Au film (as top electrode) was sputtered onto the PS surface and a 90-nm Ni-Cu-Ni film (as bottom electrode) was formed at the backside of the wafer. An electron emitter with a metal/PS/Si/metal structure was obtained.

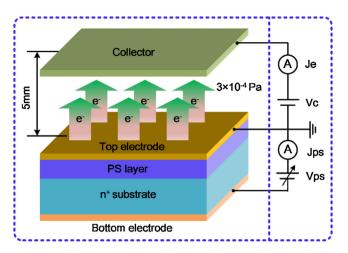


Fig. 1. The structure of PS electron emitter and measurement system for electron emission characteristics.

2.2. Electron emission measurements

The surface and section morphologies of the oxidized PS samples were measured by the scanning electron microscope (SEM) and their distribution properties of oxygen content were calculated by the energy dispersive spectrometer (EDS). The structure of PS electron emitters and the measurement system for electron emission characteristics is shown in Fig. 1. The device current density J_{ps} , emission current density J_e , and the electron emission stability σ were measured in a vacuum system ($\sim 3 \times 10^{-4}$ Pa). Emission efficiency η is defined as the ratio of J_e – J_{ps} . DC positive bias voltage V_{ps} was applied on the Au electrode relative to the negative electrode. An anode electrode was kept at a positive potential of V_c = 200 V to collect the emitted electrons. The distance between the electron-emitting surface of the PS electron emitter and the collector was 5 mm.

3. Results and discussion

3.1. The oxidation properties of the PS emitters

The probability histograms of the pore diameters, morphologies, and oxygen content distribution of PS samples A1/A2, B1/B2, and C1/C2 are shown in Fig. 2. The insets in the upper-left of the 2nd and 3rd rows subgraphs are the corresponding surface morphologies. It can be found that the diameters of the holes in the PS surfaces decrease gradually, the densities of holes increase. The holes are more clear with the decreasing of the electrochemical corrosion volume. The average diameter sizes of samples A1/A2, B1/B2, and C1/C2 are 64.5 nm, 57.3 nm, and 32.8 nm, respectively. Due to the passivation of RTO, comparing with samples A1, B1, and C1, the surfaces of sample A2, B2, and C2 are more blur and the corresponding uniformities were improved [15,21]. The other parts of each subgraph are section morphologies of the oxidized PS layers and the curves of oxygen content distribution. It shows that the incomplete oxidation samples A1, A2, and B1 present the distinct oxidation stratification phenomenon. The other PS samples were oxidized completely from top to bottom. Additionally, from the profile of the unoxidized part in the sample B1, the pore size is larger and pore direction is upright than that in other samples. The pore distribution of the samples with the corrosive volume of 90 mA min and 45 mA min is more disorder. There are some differences in the microstructures for all samples even with the same corrosion parameters, which related to the fluctuation of doping concentration among the same batch of wafers.

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