## Fabrication and Characterization of Nb/Nb Oxides-Based Single Electron Transistors (SETs)

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Niobium (Nb)/Nb oxides-based single electron transistors (SETs) were fabricated by atomic force microscope (AFM) nano-oxidation process, which is based on the selective oxidation of metal thin film due to the anodization. Successful surface modification of Nb thin film deposited on the SiO<sub>2</sub>/Si substrates was demonstrated by using this process, and from Auger electron spectroscopy (AES) analysis it was confirmed that the modified structure consists of Nb and large amount of oxygen (O), suggesting the formation of Nb oxides. Moreover, this technique was applied to the fabrication of SETs with side-gate structure. Single-electron charging effects such as Coulomb gap, Coulomb staircase and Coulomb oscillation characteristics were observed at such a high temperature of 100 K.

### 1. Introduction

On the progress of ultra-fine material processing and device fabricating techniques, single-electron charging effects such as Coulomb blockade phenomena have been easily and clearly observed in the well-controlled artificial structures with ultra-small tunnel junctions. Single electron transistors (SETs), which is based on the principle of the Coulomb blockade phenomena, enable us to control the transfer of electron one by one, meaning that the SETs will open the new frontier of the functional electron devices.

In order to realize such exciting devices with highoperated temperature, we have proposed and developed the new nano-lithography techniques by using scanning tunneling microscope (STM) / atomic force microscope (AFM) in air, which is so called STM/AFM nano-oxidation process<sup>1-3</sup>). In this process, water and/or oxygen containing species adsorbed on the planar metal surface could electrochemically react with the metal, resulting in the formation of metal oxides<sup>4,5)</sup>. Also, since niobium (Nb) is one of the supercoducting materials, Nb-based SETs will provide the exciting stage to study the competition of the two energy scales between Coulomb charging energy and Josephson coupling energy at the superconducting temperature range. In addition, the Nb-based SETs may also operate as conventional ones at the normal-metal temperature range. In this paper, we focus our attention first on the higher temperature operation of the Nb/Nb oxidesbased SETs.

## 2. Selective anodization of Nb thin film by AFM nano-oxidation process

Nanometer-scale surface modification of Nb thin film deposited on SiO<sub>2</sub>/Si substrates was investigated by AFM nano-oxidation process in air. The thickness of the film is about 3 nm. Figure 1 shows the AFM image of the modified structures formed with several applied negative voltages. When the oxidation occurs, the modified structure expands its volume on the surface because of the large amount of the incorporated oxygen. Therefore, we can easily confirm the structure by AFM observation directly after the oxidation was done. In this oxidation process, it is important to get the smooth surface on the deposited metal because the size uniformity on the modified structures depends on the surface roughness of the metal. AFMobserved surface roughness is about 1 nm, which is smooth enough for this process. Successful fabrication of the modified structures was performed by applying the negative tip bias voltage between the conductive cantilever and the sample. When the polarity of the applied bias was inverted, any structures could not be obtained, implying that the reaction on the Nb may also be due to the anodization as



Figure 1. AFM image of the modified structures formed by several applied voltages (-3, -5, -7, -9 V from L to R).



Figure 2. Size dependencies of the modified structures on the applied bias voltage.

well known in the cases of titanium  $(Ti)^{1-5}$  and chromium  $(Cr)^{6,7}$ .

Figure 2 shows the size dependencies of the modified structures on the applied bias voltage. Width and height of the modified structures could be controlled ranging from about 25 nm to 220 nm and about 0.5 nm to 6 nm, respectively, by varying the applied voltage from -1 V to -15 V. With increasing the applied negative bias voltages, the width was linearly increased but the height was saturated at the fixed value. As the bottom of the modified structure reaches at the Nb/SiO<sub>2</sub> interface, the oxidation no longer occurs perpendicularly. Therefore, it enhances the oxidation to the lateral direction, keeping the height constant.

Auger electron spectroscopy (AES) analysis was also performed to confirm the elements on the modified structures. From this analysis, it was revealed that the structure consists of Nb and the large amount of oxygen, suggesting the formation of Nb oxides.

# 3. Nb/Nb oxides-based single electron transistors with side-gate structure

Next, we present the fabrication and characterization results of metal-based ultra-small tunnel junction devices with Nb/Nb oxides system. The side-gate SETs with double junction structure were fabricated by AFM nano-oxidation process. The sample is the same structure as one used in Sec.2. Figure 3 shows the typical schematic of a side-gate SET with double junction structure. In the fabrication of the SETs, at first, a narrow Nb metal wire is defined as a channel. Then, two ultra-narrow Nb oxides wires with 25 nm wide and 30 nm long, which act as tunnel barriers, were formed by AFM nano-oxidation process in the narrow channel region. As a result, we can see the ultra-small island between them. AFM-observed typical size of the island was found to be approximately 30 nm wide, 40 nm long and 2 nm thick. Figure 4 also shows the AFM image of the side-



Figure 3. Typical schematic of a side-gate SET with double junction structure. The size of an island surrounded by Nb oxides is approximately 30 nm wide, 40 nm long and 2 nm thick.



Figure 4. AFM image of a side-gate SET with double junction structure. Bright and dark areas show the Nb oxides and Nb regions, respectively.

gate SET. In this image, bright area shows the Nb oxides region, and dark one for the Nb metal region. Source, drain and gate are placed as shown in the figure, and connected to the contact pads.

Electrical measurements were performed in threeterminal arrangement. Figure 5 shows the drain current-drain voltage characteristics with a side-gate voltage of 0 V and a temperature of 100 K. As shown in this figure, clear Coulomb gap of +15~20 mV and Coulomb staircase with 30~40 mV periodicity were observed. The Coulomb staircase was also observed at a temperature of up to 123 K. Total capacitance ( $C_{\Sigma}$ ) deduced from this periodicity (30~40 mV) is 2.7~2.0 aF, which agrees well with the value ( $C_{\Sigma} =$ 2.6 aF) obtained from the AFM-observed structural parameters of the device.

Figure 6 also shows the drain current-gate voltage characteristics with a drain-source voltage of 100  $\mu$ V and a



Figure 5. Current-voltage characteristic of a side-gate SET with double junction structure measured at 100 K. Coulomb gap of +15~20 mV and Coulomb staircase with 30~40 mV periodicity are clearly observed.



Figure 6. Dependence of the drain current on the gate bias voltage of a side-gate SET at the temperature of 100 K and the drain-source voltage of 100  $\mu$ V. Coulomb oscillation with about 50 mV periodicity is clearly observed.

measurement temperature of 100 K. In the gate bias voltage ranging from -0.4 V to -0.7 V, current oscillation characteristics were clearly observed with about 50 mV periodicity. These results clearly suggest that AFM nanooxidation process is suitable for fabricating the metal-based ultra-small tunnel junction devices and Nb/Nb oxides system may expand the function of the SET from superconducting region to normal one, depending on the operation temperature.

### 4. Conclusion

AFM nano-oxidation process was applied for the fabrication of Nb/Nb oxides-based SETs. Since Nb is a

superconductor, Nb-based SETs may expand its function to the wide range of the operation temperature. Successful surface modification of Nb thin film was demonstrated by using this process, and from AES analysis it was confirmed that the modified structure consists of Nb and large amount of oxygen, suggesting the formation of Nb oxides. The sidegate SETs with Nb/Nb oxides system showed the clear single-electron charging effects such as Coulomb gap, Coulomb staircase and Coulomb oscillation characteristics at such a high temperature of 100 K. This technique easily enables us to fabricate the SETs with ultra-small metal/insulator/metal tunnel junctions.

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