

## Nanometer-scale surface modification of Nb thin film by atomic force microscope (AFM) nano-oxidation process and its application to single electron transistors (SETs)

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Nanometer-scale surface modification of niobium (Nb) thin film deposited on SiO<sub>2</sub>/Si substrates was demonstrated by atomic force microscope (AFM) nano-oxidation process for the first time. This technique easily enables us to fabricate the nanometer-scale ultra-small tunnel junction devices such as single electron transistors (SETs), and clear single-electron charging effects such as Coulomb gap, Coulomb staircase and Coulomb oscillation characteristics were observed at such high temperature of 100 K.

### 1. INTRODUCTION

On the recent investigation of ultra-fine material processing techniques, significant efforts have been devoted for fabricating the well-controlled artificial structures with nanometer-scale dimension. Recently, one can clearly see the single-electron charging effects such as Coulomb blockade phenomena through the structure with ultra-small tunnel junctions system. Single electron transistors (SETs), which is based on the Coulomb blockade phenomena, enable us to control the transfer of electron one by one, and lead us to the new frontier of the functional electron devices.

In order to realize such exciting devices with high-operated temperature, it is important to fabricate the tunnel junctions with extremely low capacitance. From this point of view, we have proposed and developed the new nano-lithography techniques for metal/oxide-based electron devices by using atomic force microscope (AFM) in air, which is so called AFM nano-oxidation process. In this paper, nanometer-scale surface modification of niobium (Nb) thin film was described, and its application for fabricating the Nb/Nb oxides-based SETs with ultra-

small tunnel junctions was also reported in detail.

### 2. SURFACE MODIFICATION OF Nb BY AFM NANO-OXIDATION PROCESS

Extremely thin Nb film with the thickness of 3 nm was deposited by DC magnetron sputtering on SiO<sub>2</sub>/Si substrates. AFM-observed surface roughness of as-deposited Nb film was about 1 nm, which is

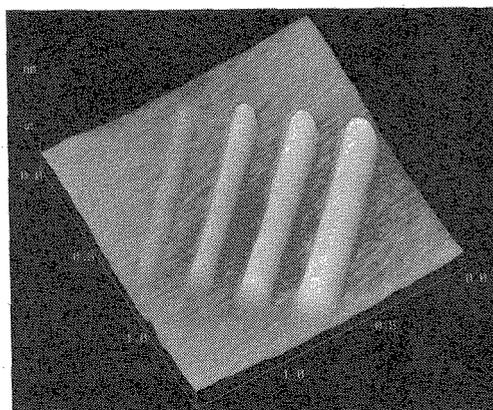


Figure 1. AFM image of the modified structures formed by several applied voltages (-3, -5, -7, -9 V from L to R).

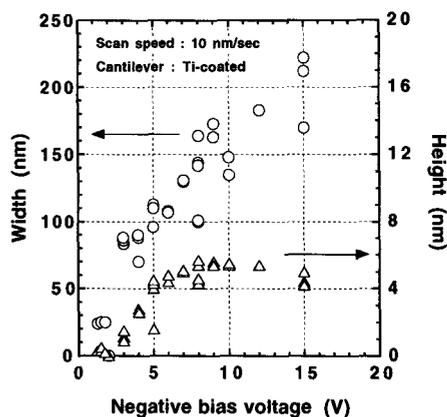


Figure 2. Dependence of size in the modified structures on the applied bias voltages.

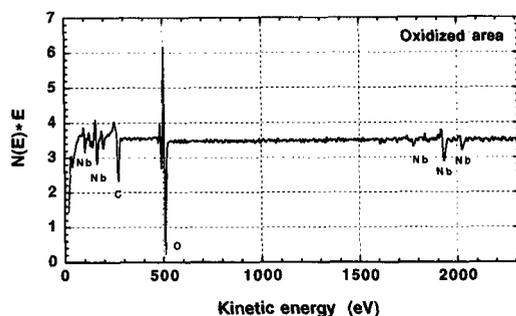


Figure 3. Auger electron spectrum taken from the modified structure.

smooth enough for the AFM nano-oxidation process. Figure 1 shows the modified structure of Nb formed by this technique. As shown in this figure, successful fabrication of the modified structure was performed by applying a negative tip bias voltage between metal-coated conductive cantilever and sample.

Figure 2 also shows the size dependencies of the modified structure on the applied bias voltage. By varying the applied voltage from about -1 V to above -15 V, the size of the modified structure was controlled ranging from about 25 nm to 220 nm in width and about 0.5 nm to 6 nm in height. From Auger electron spectroscopy (AES) analysis of the modified structure as shown in Fig. 3, it was revealed that the structure consists of Nb and the

large amount of oxygen (O), suggesting the formation of Nb oxides. As well known in the cases of surface modification on titanium (Ti)[1-5], chromium (Cr)[6,7], Si[8] and GaAs[9] by AFM-based oxidation process, the mechanism of oxidation on Nb may also appear to be tip-induced anodization, i.e., water and/or oxygen-containing species adsorbed on the sample surface could electrochemically react with Nb by applying the bias voltage between cantilever and sample.

### 3. METAL/INSULATOR/METAL (MIM) DIODES WITH Nb/Nb OXIDES SYSTEM

This technique with the possibility on fabricating the ultra-small tunnel junction devices such as SETs was preliminarily applied for the fabrication of planar-type metal/insulator/metal (MIM) diodes. Figure 4 shows the room temperature current-voltage (I-V) characteristics of Nb/Nb oxides/Nb-based MIM diodes with different width of Nb oxides. From this result, nonlinear I-V characteristics were clearly observed, suggesting that Nb oxides formed by AFM nano-oxidation process act as insulating barrier material for the electron. Furthermore, from the temperature dependencies of the I-V characteristics with the consideration of barrier height lowering effect due to the image force, barrier height ( $\phi_B$ ) and relative dielectric constant ( $\epsilon_r$ ) in Nb/Nb oxides system were determined as 133 meV and 64, respectively.

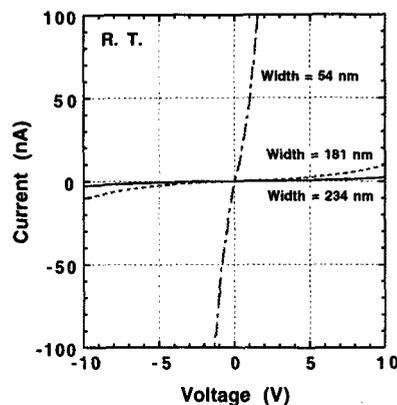


Figure 4. Current-voltage characteristics of the planar-type MIM diodes measured at room temperature.

#### 4. SINGLE ELECTRON TRANSISTORS (SETs) WITH Nb/Nb OXIDES SYSTEM

As mentioned above, Nb is successfully oxidized by the AFM nano-oxidation process, and Nb oxides show the electrical properties as an insulator. This AFM-based oxidation process has nanometer-scale controllability and easily enables us to fabricate the nanometer-scale ultra-small tunnel junction devices such as SETs. Since Nb is a superconductor, the SET with Nb/Nb oxides system may show the new function depending on the operating temperature, i.e., room temperature operated SETs in higher temperature region and superconducting SETs in lower one. In this paper, we propose new functional metal-based SETs with Nb/Nb oxides system,

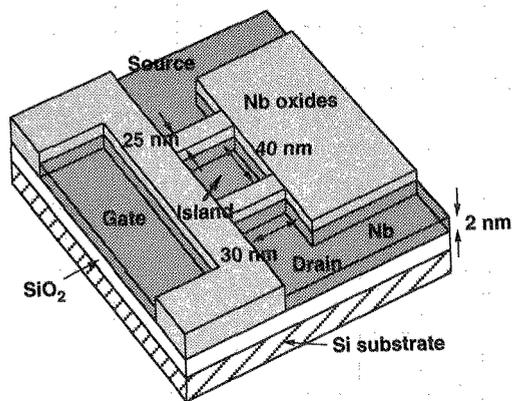


Figure 5. Schematic of Nb/Nb oxides-based SETs with side-gate structure.

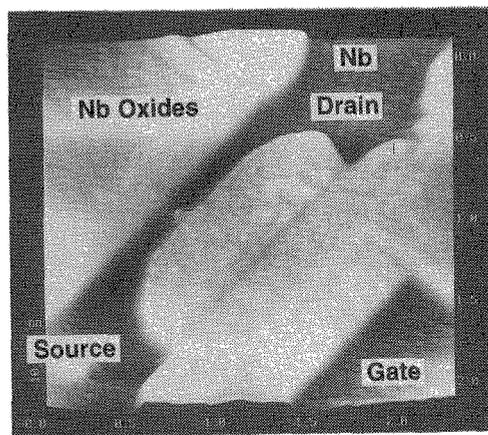


Figure 6. AFM image of Nb/Nb oxides-based SETs with side-gate structure.

especially focusing on the high temperature operation.

Figure 5 shows the typical schematic of SET with side-gate structure. At first, ultra-narrow metal wire as a channel with 30 nm wide and 10 nm long was defined by the AFM nano-oxidation process. Then, narrow Nb oxides wires with 25 nm wide and 30 nm long, which act as tunnel capacitances, were formed in the channel region identifying the ultra-small island between them. Typical size of the island decided by AFM observation was found to be approximately 30 nm wide, 40 nm long and 2 nm thick. AFM-observed Nb/Nb oxides-based side-gate SETs are shown in Fig. 6. In the AFM image, bright region shows the Nb oxides, and dark one for Nb metal. Tunnel junctions were placed at the center of the constrict channel region.

Current-voltage characteristics of the devices we measured with a three terminal arrangement. Figure 7 shows the drain current-drain voltage characteristic of double junction ("one island") device measured at 100 K, with the gate bias voltage of 0 V. Clear Coulomb gap of +15~20 mV and Coulomb staircase with 30~40 mV periodicity were observed at temperature of 100 K. Total capacitance ( $C_{\Sigma}$ ) deduced from this periodicity (30~40 mV) is 2.7~2.9 aF, which agrees well with the value obtained from the AFM-observed structural parameters of the device ( $C_{\Sigma} = 2.6$  aF).

In addition, dependence of the drain current and corresponding differential conductance on the gate

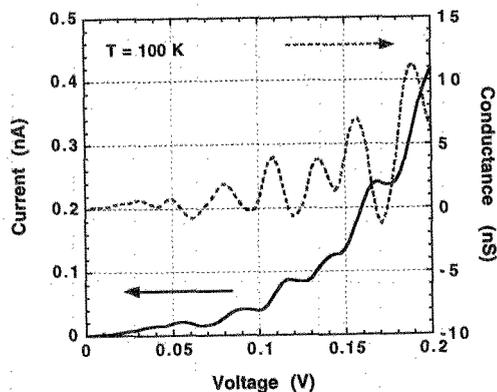


Figure 7. Drain current-drain voltage characteristic of the side-gate SET with double junction structure measured at 100 K.

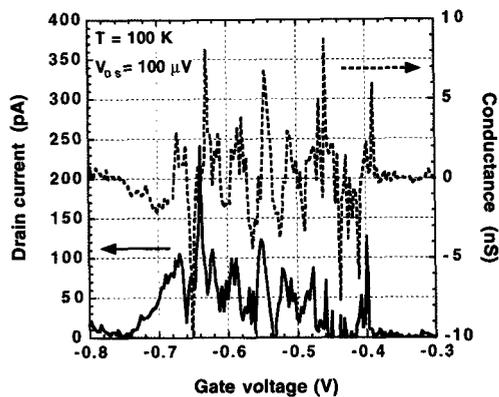


Figure 8. Dependence of the drain current on the gate bias voltage of the side-gate SET at temperature of 100 K and drain-source voltage of 100  $\mu$ V.

bias voltage was measured at a drain-source voltage of 100  $\mu$ V and a temperature of 100 K, as shown in Fig. 8. Current oscillation characteristics were clearly observed with about 50 mV periodicity. These results clearly suggest that AFM nano-oxidation process is suitable for fabricating the metal-based ultra-small tunnel junction devices and Nb/Nb oxides system may expand the function of SET to the wide range of operation temperature.

## 5. CONCLUSION

Successful surface modification of Nb thin film was performed by AFM nano-oxidation process for the first time. From AES analysis, it was revealed that the structure consists of Nb and the large amount of oxygen, suggesting the formation of Nb oxides. By adjusting the oxidation parameters, Nb oxides wires with nanometer-scale dimension could be obtained, and its electrical properties show the insulator characteristics.

Metal-based side-gate SETs with Nb/Nb oxides system were fabricated by using this unique process, and clear single-electron charging effects such as Coulomb gap, Coulomb staircase and Coulomb oscillation characteristics were observed at such high temperature of 100 K. This technique easily enables us to fabricate the nanometer-scale ultra-small tunnel junction devices such as SETs.

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