
Fabrication of Nanotip arrays Based on AAO Template

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1. Introduction

During the times in which thermionic vacuum tubes were the dominant part for low-power electronic circuits, various efforts were carried out reducing their size and power consumption. In the last from 1955 to 1965, vacuum tubes were rapidly replaced with solid-state microelectronic devices. There are two major reasons for the decline of thermionic vacuum tubes. First, significant power waste to eject the electron through vacuum region occurs. Second, obtainable highest current density from thermionic vacuum tube is very small.

Recently, field emission based vacuum devices have recently attracted considerable attention. The reasons why field emission based vacuum devices have several advantages over solid-state devices. It is independent of ambient temperature and radiation environment. Also it has no power dissipation while electrons transport through medium, because the electron transport in the vacuum device is ballistic[1]. On the other hand, electron transport of solid-state devices is collision-dominated transport. Consequently, field emission based vacuum device can generate high power at high frequencies. These characteristics guarantee the many applications, including active elements for integrated-circuit[2], flat-panel display[3], electron guns[4], and microwave power tube[5].

Most of the field-emission-based vacuum devices are on the basis of conventional Spindts process. However, it requires very complicate process and high cost equipment such as selective etching and electron beam lithography. Also high voltage is required for operating devices because inter-electrode distance is several hundred micrometers. In addition, it is difficult to apply to a large area. To overcome these difficulties, we have used anodic aluminum oxide(AAO) technology. This process is possible to control template dimensions in nanometer size easily such as pore diameter, length, and density without using electron beam lithography. Also, it can be applied to a large area easily.

In this study, we have fabricated field emission arrays(FEAs) on the basis of AAO template. In our structure, we have obtained the high current densities at low operating voltage by reducing of the distance between field emitter array and anode electrode compared to conventional vacuum devices.

2. Experimental

First, a high purity aluminum sheet (99.999 %) of 1 mm thickness was degreased in acetone by ultrasonication and rinsed in an ethanol solution. Thereafter, the Al sheet was electropolished in a

mixture of perchloric acid and ethanol (HClO_4 : $\text{C}_2\text{H}_5\text{OH}$ =1:4 in volumetric ratio) to remove surface irregularities. A two-step anodization was performed to obtain a regular pore array. The first-step anodization was carried out in a 0.3 M oxalic acid solution at 15 °C for 12 h. After the first anodization was completed, AAO layer was removed by immersing the Al sheet in a mixture of 1.8wt. % chromic acid and 6wt. % phosphoric acid at 65 °C for 3 hrs. Finally, AAO template was obtained by the second anodization under the same condition as the first one for 5 min. After second anodization, the AAO template was treated in a 0.1 M phosphoric acid solution at 30 °C for 40 min. Ni nanowires were partially filled by electrochemical deposition in the pores of AAO template. The electrodeposition in the AAO template was accomplished in an electrolyte consisting of 5 wt. % $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ and 2 wt. % H_3BO_3 by applying an alternating voltage. To get the different aspect ratio of Ni nanowires, we have varied the electrodeposition time 30 sec and 3 min, respectively. The AAO template containing metal nanowires was sealed at a pressure of about 10^{-6} torr using evaporation of titanium which served as an anode electrode.

The morphology and structure of AAO template containing metal nanowires were observed by field emission scanning electron microscope (FE-SEM, Hitachi S-4200). The measurement of field emission current was carried out using a diode-type configuration. 4155 analyzer (Hewlett Packard, 4155 parameter analyzer) was used to apply voltage up to 5 V and to measure the emitting current with pA sensitivity up to 100 mA. Figure 1 presents the overall experimental scheme.

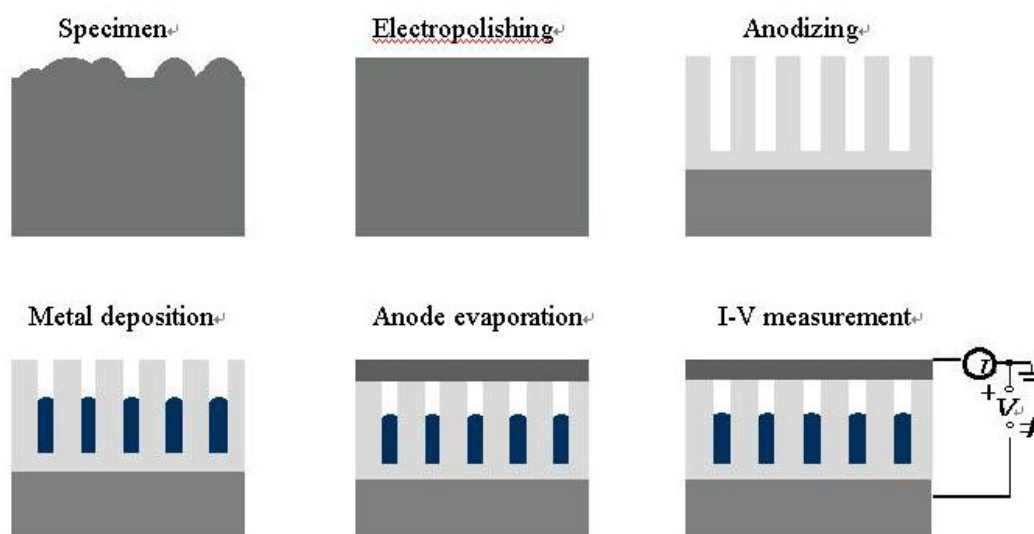


Figure 1. Experimental scheme for fabrication of nanotip arrays based on AAO template and their field emission measurements

3. Results and discussion

The self-organization conditions reported by different groups[6-8] were employed to obtain hexagonal closed-packed pore structure. It can be obtained highly ordered AAO template at 40 V in a 0.3M oxalic acid at 15 °C solution by 2-step anodization. It can be obtained AAO template with pore diameter of 24 nm, interpore distance of 100 nm, and pore density of $10^{10}/\text{cm}^2$. It can be fabricated the AAO template with 600 nm length. Subsequently, pores were treated in a 0.1 M phosphoric acid

solution for 40 min. After chemical treatment of the AAO template, the pore diameter of AAO template is about 50 nm. Ni nanowires were partially filled by electrochemical deposition in the pores of AAO template. The diameter of the Ni nanowires is exactly the same as that of the pores in the AAO template. Subsequently, angled evaporation of titanium was performed to seal the structure and to obtain the anode electrode. Angled evaporation was performed with 30 ° angle. It is known that angled evaporation is essential to prevent penetration of evaporation metal inside pores. The vacuum inside the pores is the same as the evaporation environment.

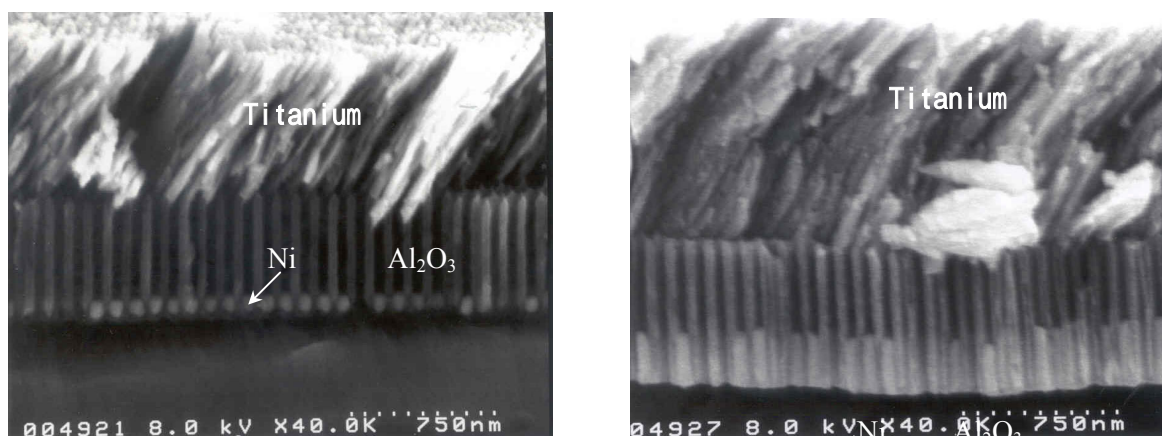


Figure 2. 2nd anodization was performed for 5 min. Ni nanowires were electrodeposited at the pore bottom of a AAO template for (a) 30 sec, (b) 3 min and titanium and titanium was sealed the structure by angled evaporation.

Field emission characteristics of nickel nanowires deposited the pores of the AAO template were measured using the diode-type field emission measurement unit. Figure 2 shows the SEM images of integrated diode structure. It is clearly shown that the aspect ratio of Ni nanowires increased with deposition time and titanium is well sealed the diode structure. The aspect ratios of Ni nanowires with 30 sec and 180 sec electrodeposition time and 180 sec are 1.22 and 4.52, respectively.

Figure 3 presents typical I-V characteristics of the nickel nanowires with AAO template. Useful parameter for the comparison of the field emission performances of two samples is turn-on voltage, V_{to} , and the threshold voltage V_{thr} , the voltage needed to produce an emission current density of 10 $\mu\text{A}/\text{cm}^2$ and 10 mA/cm^2 , respectively. Our V_{to} and V_{thr} values are in the range of $\sim 0.1\text{V}$ and 1.4~2.8V, respectively. They are much lower than those of other groups [9-10]. This is due to the short interelectrode distance and high packing density of field emitters. While interelectrode distances of other groups were about several hundred micrometers [9-10], the interelectrode distance of our structure is less than 500 nm and a number of field emitters are more than $10^9/\text{cm}^2$. Short interelectrode distance can make sufficient electric field for field emission at low applied voltage.

It is shown in figure 3 that emission current density for Ni_{180} is higher than that of Ni_{30} at the same operating voltage. In Ni_{180} , it appears that the interelectrode distance is shorter than in Ni_{30} , so it can be induced the higher electric field than that of Ni_{30} at the same voltage.

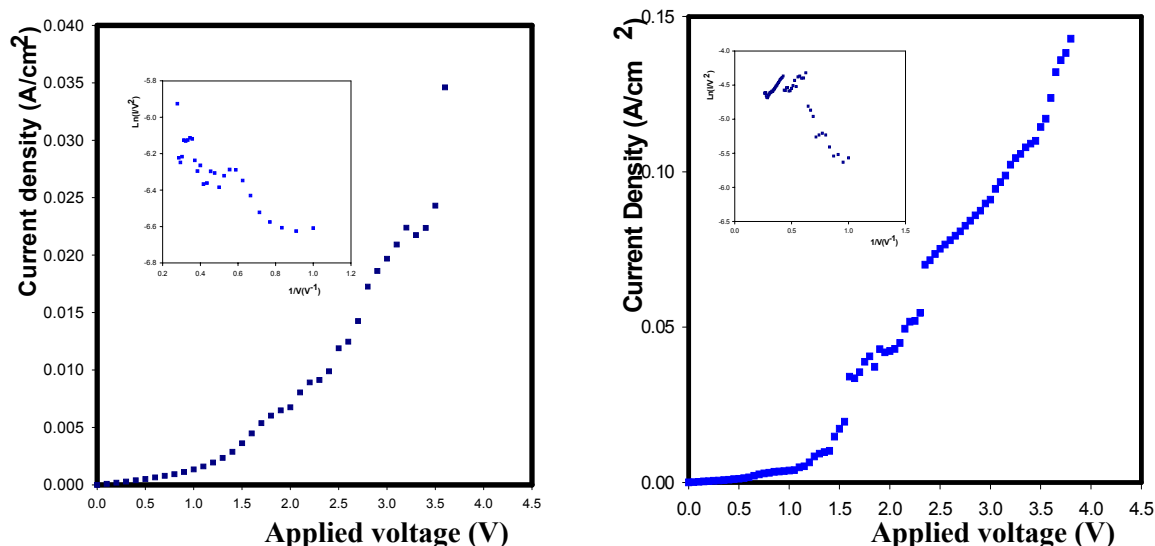


Figure 3. I-V characteristics of nickel nanowire arrays (a) for 30 sec deposition (Ni_{30}), and (b) for 180 sec deposition (Ni_{180}). Insets in (a) and (b) are the Fowler-Nordheim(FN) plots for each Ni nanowire field emitters.

Fowler-Nordheim (F-N) plots confirmed that the mechanism of emission current was indeed caused by field emission. The slope of the F-N plot is equal to $B\phi^{3/2}d/\beta$, where the constant $B = 6.87 \times 10^9 \text{ V eV}^{-3/2} \text{ m}^{-1}$, is a work function, d is the interelectrode distance. Insets in figure 3(a) and (b) show the Fowler-Nordheim(FN) plots for each Ni nanowire field emitters. It shows that the mechanism of emission current comes from the field emission of Ni nanowires.

In conclusion, we have fabricated integrate diode structure for the template based Ni nanowires as field emitters. The emission characteristics were affected by the aspect ratio of Ni nanowire. We observed the significant high current density at low operating voltage. To obtain the low operating voltage, interelectrode distance was diminished less than 500 nm.

4. Reference

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