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ARTICLE

Electron Emission from High-Purity Copper Wires

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Abstract: Field electron emission measurements were carried out on copper of 99.95% purity emitters, with apex radii in the nano and micrometer range. Samples have been prepared by electrochemical etching using phosphoric acid (H_3PO_4) solution. Measurements have been carried out under high vacuum environments with a base pressure of 10^{-7} mbar. Samples were installed in a field electron microscope (FEM) with an anode (tip)-to-cathode (screen) distance of 10 mm. Scanning electron microscope images, electrons spatial distribution behavior, and the current-voltage characteristics (*I-V*) have been studied and analyzed. Copper emitters have been prepared as electron sources and tested. The results showed a typical current-voltage characteristic from a coat emitter. The scanning electron microscope showed the sample geometry and cleanness. These copper electron emitters have been found to have significant aspects (high voltage breakdown mechanism) affecting the performance of advanced systems such as the electronic accelerator of The European Organization for Nuclear Research, known as CERN. This work aims to study this type of material to understand the high voltage breakdown phenomena in copper and the reasons behind it to provide a solution to such phenomena.

Keywords: Field electron emission, Current-voltage characteristics, Copper tips, High vacuum.

Introduction

Field electron emission (FE) is the mechanism of electron emission via tunneling from a material's surface into a vacuum under the effect of a high voltage, and consequently a high surface field, typically a few V/nm [1].

The emitter is commonly shaped into a tip, with an apex radius ranging from several nanometers to hundreds of nanometers. When making extremely sharp emitters, it is required to utilize the material of the highest quality possible. Field electron emission source for vacuum electronics is considered to provide faster response, high performance, and reduced energy consumption in contrast to traditional thermionic emitters. Electron sources used as a cold cathode are of great interest to researchers and industries for their technological applications. There have been many important efforts to study and understand the field emission characteristics from various materials [2-11]. Several attempts were made not only to investigate the microstructure and geometry of the samples but also to test the validity of theoretical models of field emission [12-18]. The field electron emission behavior of various materials has already been investigated. Such attempts showed that several factors affect the field emission behavior, including the geometry and the degradation effect at high current density and harsh working environments. Further investigation of the field emission characteristics and the behavior of the electron emission from Copper wires is accomplished. Furthermore, the application of such emitters and the theoretical background are to be discussed in future work.

Experimental Methods

Copper wire of 0.25 in diameter and 99.95% pure was used in this work. The micro-apex copper emitters utilized for this work have been electrolytically etched using phosphoric acid (H₃PO₄) solution. The etching process was controlled by selecting a suitable initial etching current of ~ 40 mA, and a voltage of 15V for the first sample and 10V for the second sample. After the etching process, the etched tips were cleaned with distilled water in an ultrasonic device. Afterward, the surface of the etched copper tips was checked using a digital electron microscope. Additionally, scanning electron microscopy (SEM - FEL QUANTA FEG 450) was employed to capture closer and more detailed images, enabling measurements for estimating the apex radius of the tips.

The emitters were then installed in a field emission microscope (FEM) with a pressure of 10^{-7} mbar. The axial tip-to-screen distance was maintained at approximately 10 mm. The applied voltage was supplied by an external power supply and the current was measured using a picoammeter. The emission spatial distribution was captured from the phosphor screen using a digital camera (Nikon DX).

Results and Discussion

A successful chemical etching process produced a sharp emitter tip. Figure 1 shows the scanning electron microscope images of two samples. The apex of the tip shows different geometry between the two samples, which can be attributed to both the etching process and the subsequent storage conditions. Despite the meticulous handling of the samples throughout the entire process, precise control is difficult, because of the very small size of the samples (nano-micrometer). However, the investigation of the different shapes and geometries of the samples is the primary objective of this study. Fibrils on the sample surface are seen in Figs. 1 (c) and 1(d), these fibrils are expected to be residuals of the etching solution and/or contamination from the atmosphere during the sample transportation.

The first sample, seen in Figs. 1(a) and 1(b), has a radius of \sim 386 nm and the second sample, in Figs. 1(c) and 1(d), has a radius of \sim 243 nm.



FIG. 1. Shows the scanning electron microscope images of the two samples at two different magnifications. 1 (a) and 1 (b) show the first sample at 5000 and 25000 magnification, respectively. 1 (c) and 1 (d) depict the second sample with the same magnifications as the first sample.

After installing the samples inside the vacuum chamber, voltage is applied gradually on emitter. Fig. 2(a) the shows the I-V characteristics of the first sample, during the voltage increase cycle, a stable current is measured of $\sim 0 \ \mu A$ up to $\sim 1.5 \ kV$ followed by an increase to a 6.5 µA at 1.7 kV in the so-called 'switch-on' phenomena [1,19,20]. The second sample shows a similar behavior, i.e. the constant current is followed by an instant increase to a higher voltage as shown in Fig. 2(b). It should be noted, that although the overall I-V characteristics of the two samples are similar, a closer examination reveals variations in the magnitude of current increase between the two samples. This difference is worth to be discussed more thoroughly. The switch-on phenomena in the current study is a result of the residual etching media on the sample surface, which acts as a coating media.



FIG. 2. Shows the current-voltage characteristics of both samples (a) sample 1 and (b) sample 2, during voltage increase (black square) and voltage decrease (red circle).

The electron field emission is a process where an electron leaves the sample surface into the vacuum (under high vacuum) and hits the screen. Therefore, the geometry of the sample plays a critical role in this process. Figures 1(a) and 1(b) show that the sample apex is semi-circular with a non-polished surface. This surface will require a higher voltage to emit electrons, since the bigger the apex area is, the more energy is required to overcome the potential barrier and release the electrons. Figure 4 shows the electrons' spatial distribution on the screen during the voltage decrease. The emission recorded at high voltage exhibits a single spot, indicating that the electron emission occurs from a specific point. The intensity of the emission lessens as the measured current decrease. This behavior indicates that the current has created a 'tunnel' on the emitter surface through which the electrons pass during the voltage decrease.



FIG. 3. Shows the Fowler-Nordheim plots corresponding to the I-V plots in Fig.2. Green stars correspond to the voltage increase cycle and L1, L2, and L3 correspond to the voltage decrease cycle.

Comparison of Figs. 1(a) and 1(b) on the one hand and Figs. 1(c) and 1(d) on the other, shows the difference between the two samples. The second sample has a more regular and sharper apex than the first sample. Unlike the first sample, the second one has multiple spikes on its surface as seen in Fig. 1(d). These spikes are expected to play a role in the electron emission process. Now, let's consider the I-V behavior of the second sample, as shown in Fig. 2(b). Although the switch-on phenomenon is present, the magnitude of the change is smaller than that exhibited by the first sample. The switch-on of the second sample is only 1.7 µA, whereas the corresponding number for the first sample is 6.5 μ A. The main reason for such a difference is the sample geometry: the second sample is sharper and smoother, which leads to an easy flux of electrons through the emitter.

Figures 3(a) and 3(b) show the Fowler-Nordheim (FN) type plots calculated from the I-V data presented in Figs. 2(a) and 2(b), respectively. A clear deviation in the linearity between the low and high voltage is observed. Thus, some distinct linear lines with different slopes in the low- and high-voltage regions can be observed in the decreasing cycle in Figs. 3(a) and 3(b). Such nonlinear behavior has been predicted theoretically and observed experimentally (even for large-area field emitters) by several researchers [3, 7, 21, 22]. This behavior can be related to several effects: (a) the local enhancement of the electrical field affected by the statistical distribution of the emitters' geometrical parameters [2], (b) the high current density (a field-induced mechanism) and its effect on the emitter degradation [23], and (c) at high emission current, the space charge and adsorption effects might lead to a nonlinearity [24]. The mixture and overlapping of these effects make it a challenging mission to relate the nonlinearity to a single reason. Thus, it is advisable to combine I-V characterization and emission spatial distribution with F-N plots for clearer data interpretation.

Figure 5 shows the second sample's emission distribution during voltage decrease. The multispot pattern indicates that the spikes on the sample surface might be working as emitters as well.



FIG. 4. Shows the electron emission spatial distribution of the first sample during the voltage decrease. The applied voltage and measured current are given in each image.



FIG. 5. Shows the electron emission spatial distribution of the second sample during the voltage decrease. The applied voltage and measured current are given in each image.

Conclusions

High-purity copper wires have been prepared using an electrochemical etching process and investigated using a field electron microscope and scanning electron microscope.

The current-voltage characteristics and the electron emission spatial distribution were recorded and discussed. The results showed that not only does the emitter geometry affect the emission behavior, but also the surface quality and cleanness play a major role.

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The switch-on phenomena were found as well as a result of the residual of the etching media, which works as a coating on the sample surface.

Future work will be on investigating the orthodoxy test from samples made of copper and comparing it with the literature.

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