FABRICATION AND NOISE DIAGNOSTICS OF SCHOTTKY CATHODES

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ABSTRACT

The paper introduces a method for fabrication and diagnostics of microscopic cathode based on Schottky field emission. Schottky emission is the predominant electron source technology in actual focused electron beam equipment, including scanning electron microscopy (SEM), transmission electron microscopy (TEM), Auger systems, and semiconductor inspection tools. Achieving proper results requires an electron source with the following ideal properties: small source size, low electron emission energy spread, high brightness (beam current per solid angle), low noise and long-term stability, simple and low-cost operation. Electrochemical etching procedure, used for producing extra-sharp tungsten cathode tips, together with suitable noise based analysis method are presented in the paper.

1. INTRODUCTION

The scanning electron microscope (SEM) is a type of electron microscope that displays the sample surface by scanning it with a high-energy beam of electrons. The electrons are interacting with the atoms which are making the sample producing signals that contains information about the samples surface topography, composition and other properties such as electrical conductivity. Mostly, in the standard detection mode the secondary electron imaging (SEI) can produce very high-resolution images of a sample surface, revealing details about 1 to 5 nm in size. Due to the way of creation of images, the SEM micrographs have a very large depth of field yielding a characteristic three-dimensional appearance useful for understanding the surface structure of a sample. In a typical SEM, an electron beam is thermionically emitted from an electron gun fitted with a tungsten filament cathode. Tungsten is normally used in thermionic electron guns because it has the highest melting point and lowest vapour pressure of all metals, thereby allowing it to be heated for electron emission, and because of its low cost. Fine pointed, an atomically sharped tip is necessary to gain atomic resolutions (less than nm).

Electrochemical etching procedures, used to product atomically sharp tips from polycrystalline wires, have been well established for more than 60 years and are described in several books and articles. On the present, mostly motivated by the industry competition, etching techniques are being explored and further refined. In this article, a technique for etching a tip from a tungsten wire is described.

2. ELECTROCHEMICAL ETCHING BASICS

A basic review of different etching techniques intended for sharp tips making was published by Melmed [4]. The etching technique is based on presence of the tungsten wire and of a stainless steel counter electrode, both immersed into an electrolyte of NaOH (or KOH). For etching, a DC voltage of about 2.5–9 V is applied to the electrodes, the tungsten wire acts here as the anode, the stainless steel electrode then as the cathode. An etching current on the order of mA leads to the following chemical reaction (simplified):

$$\mathbf{W} + \mathbf{H_2O} + 2\mathbf{NaOH} \rightarrow 3\mathbf{H_2} + 2\mathbf{Na_2WO_4} \tag{1}$$

As a matter of fact, this reaction is much more complicated. After a proper etching time (from 30 to 50 sec), the wire at the air/electrolyte interface becomes so thin that the bottom part of the tungsten simply drops off. Finally, the etched wire terminates in a uniform taper because gravity is the only significant physical force effecting on the wire. In order to prevent further etching of the upper part, the constant voltage power supply is equipped with a fast electronic switch set to switch the current off when it falls below a preset value. The value of the etching current then drops drastically when the lower end of the tungsten wire drops off. Sophisticated methods to cut off the current have been developed [3,4]. Electrochemical etching is widely used because of its simplicity, but is strongly dependent on the experience of the manufacturer due to its empirical nature. Therefore, a drop-off technique based on electrochemical etching was introduced.

3. ETCHING VOLTAGE CONTROL TECHNIQUE

It has been observed that a smaller radius of curvature is necessary for high resolution images and also that a lower aspect ratio is preferable for the reduction of noise induced by flexible vibration. The concept of drop-off technique is mainly focused on obtaining the tip apex with small radius of curvature in a reproducible way.

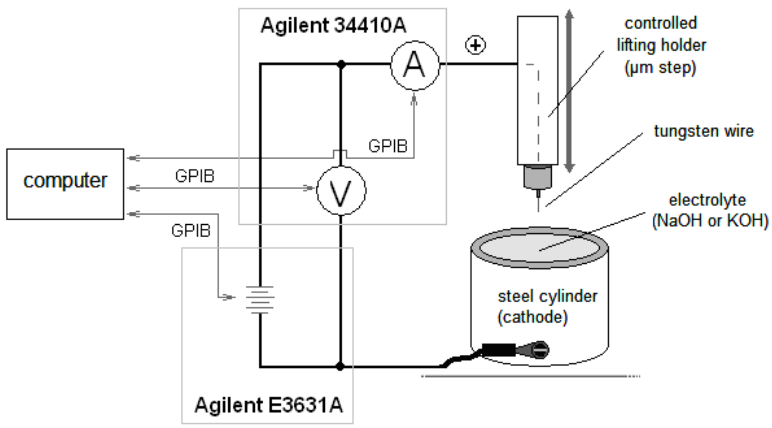

Fig. 1: The experimental setup of the electrochemical etching installation

It is well known that the radius of curvature of the etched tip is mainly influenced by the cut-off time whereas the aspect ratio of the etched tip is inversely proportional to the etching voltage and electrolyte concentration [2,3]. Chemical and physical properties can also influence the quality of scanning. As for the tungsten wire, a dark oxide layer (most probably WO_3), is covering the tips surface when a high etching voltage is applied.

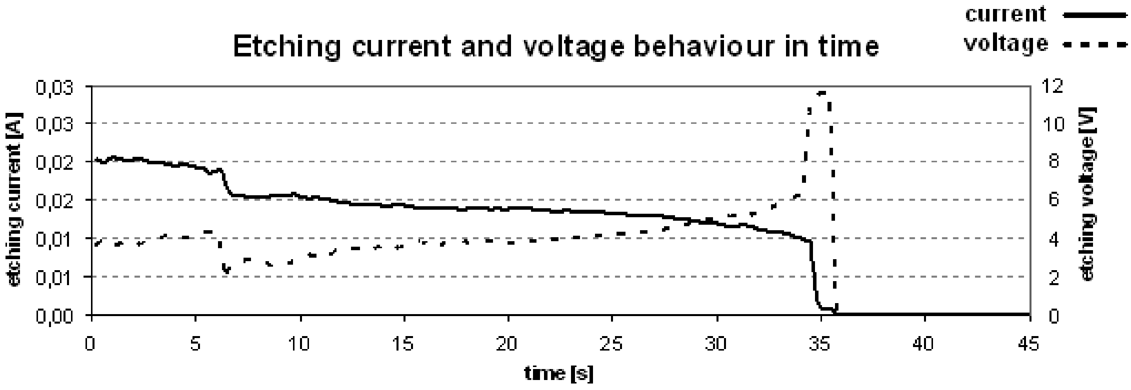

Fig. 2: Etching current and voltage time behaviour

The experimental setup is schematically shown in Fig. 1. An etching voltage in the range of 2.5–9V DC, supplied from a GPIB controlled power source, is connected between two electrodes and both, the voltage and the etching current are measured by GPIB controlled multimeter. Whole measurement is controlled by the SCPI commands through the USB/GPIB interface from the Matlab script. The Multimeter acts here as the Amperemeter and Volt-meter. Resulting values are continually sent back to Matlab development environment, when they are processed and evaluated. It has been experimentally observed, that the etching current is rising fluently (almost linear) until the wire drops off. In this moment etching current decrease rapidly and settle down on zero value (fig 2). In contrary, the etching voltage is increasing and gaining its highest value just a short time before the wire has been etched over (fig. 2).

4. NOISE DIAGNOSTICS

Noise diagnostics has been performed on the cathode, under the ultra high vacuum conditions (UHV) in order to provide basic analysis of manufactured cathodes. On the basis or reached results (fig. 3), it is evidently that measured noise has characteristics of so called

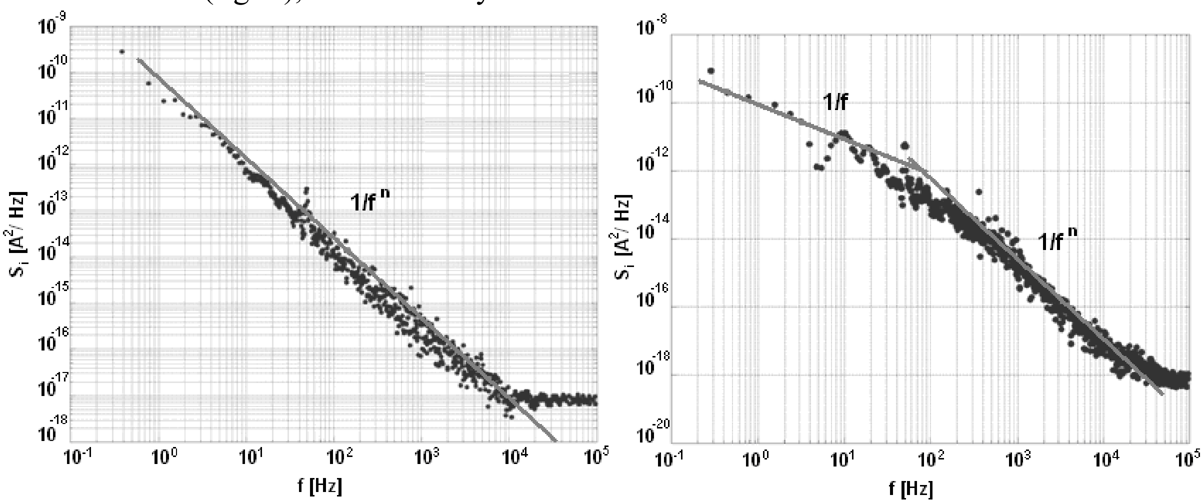

Fig. 3: Manufactured cathode noise diagnostics under the UHV conditions (P = 4.10^{-5} [Pa])
a) voltage connected U_a = 250V b) voltage connected U_b= 280V

1/*f* (flickering) noise. The 1/*f* noise is a process with a frequency spectrum such that the power spectral density is proportional to the reciprocal of the frequency. In terms of power at a constant bandwidth, 1/*f* noise falls off at 3 dB per octave. There can be also found noise caused by adsorption and desorption of different atoms of residual gas in vacuum chamber. This is generation-recombination noise. Positive ions created in vacuum chamber are accelerated back to the cathode and bombard the emission area. Ion bombardment is mechanically deforming an emitter's surface.

The $1/f^n$ noise (where n > 1) originates from the superposition of particular 1/*f* and G-R processes. At high enough frequencies the 1/*f* noise is never dominant, on these frequencies the thermal noise is dominating. The source of 1/*f* noise is not very clear, in other words, there is not a theory, which could explain all the experiment results. Because Schottky cathodes operate at high temperature, the surface mobility is high enough to anneal such deformations in a reasonable time. The room temperature of cold field emission cathode will not anneal such deformations. To repair the cold field emission, it is necessary to periodically "flash" the cathode.

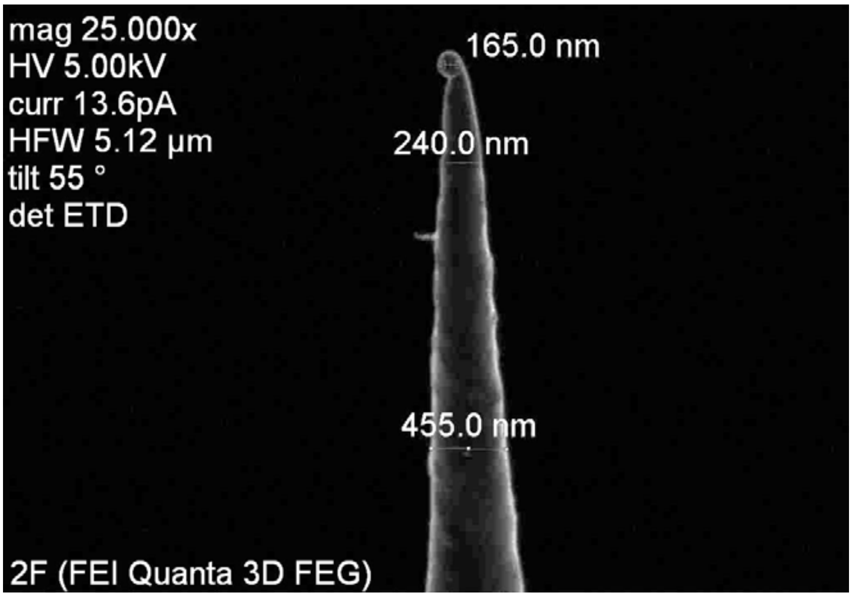

Fig. 4: The geometry of the manufactured cathode’s tip (magnification 25.000x)

5. CONCLUSION

The experiment resulted in creation of active Schottky cathode with very sharp tip (fig. 4) using the computer controlled electrochemical etching technique. This method can be extended for other metal wires as well (tantalum, niobium). Anodic oxidation, used to prepare thick oxide layer of thickness about 30 to 100 nm, can be modified further to achieve optimized value of tunnelling current. As far as the diagnostics new experimental results of noise spectral density in this frequency range has been obtained. The main sources of noise are: $1/f^n$ noise (which generates from particular 1/*f* and G-R processes superposition) and from generation recombination noise with thermal noise. The 1/*f* noise is dominant in ultra low frequency range (mHz region). The noise spectroscopy in time and frequency domain is one of the promising methods to provide a non-destructive characterisation of semiconductor materials and devices.

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