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# ARTICLE

# **Characterizing a New Composite Material: Effect of NaOH Coating of Variable Thickness on the Properties of a Tungsten Microemitter**

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Abstract: Tungsten based microemitter tips have been prepared with various tip radii ranging from 30 to 100 nm. These tips were manufactured by electrochemical etching of a 0.1 mm diameter high purity (99.95%) tungsten wire at the meniscus of two molar NaOH solutions. Contrary to the standard procedure, the tips' surfaces have not been cleaned off NaOH solution by ultrasonic cleaning in distilled water. Only a coarse cleaning by dipping the electro-polished samples a few times in distilled water has been performed. Thus, a layer of NaOH remained on the surface, which acts like a coating. The thickness of this coating layer left on the core material depends on the number of dips of the sample in water after etching. This procedure produced composite microemitters which consisted of a tungsten core with three different thicknesses of coating - thick, medium or thin consecutively produced by dipping the etched samples in water for one, six or twelve time(s). A conventional field emission microscope with a tip (cathode) – screen (anode) separation standardized at  $\sim 10$  mm was used to characterize the electron emitters. The system was evacuated down to a base pressure of  $\sim 10^{-8}$  mbar when baked at up to  $\sim 180$  °C overnight. This allowed measurements of typical Field Electron Emission (FE) characteristics; namely the current - voltage (IV) characteristics and the emission images on a conductive phosphorus screen (the anode).

Keywords: Nickel ferrite; Lattice parameters; Magnetic hysteresis; Cationic distribution.

### Introduction

Due to its favorable emission properties and simple operating principle, the field emitter is particularly attractive as an electron source. In the past decades, there has been sustained interest in field emitters as high brightness electron sources for various technological devices. For applications, such as electron microscope sources, that require point-geometry emitters, the most widely used substrate material is tungsten [1,2]. Metallic micropoint emitters have faced extra interest by the emergence of microfabrication technology, especially in the form of planar field emitter arrays (e.g. Refs. [3-7]) which were incorporated into devices such as flat-panel cathode luminescent display [8].

To avoid metallic tip degradation from the ion sputtering processes during emission and to obtain an electron source with long lifetime and high beam brightness, a wide range of composite micropoint cathodes has been manufactured. This involved coating the tungsten tips with a variety of dielectric materials which was started in 1986 by Latham and Mousa [9]. Recently, several types of dielectric coating on tungsten were reviewed by Mousa [10]. A guidance to the interpretation of the measured electron emission characteristics is given by Forbes and Deane [11], along with detailed analysis of the transmission coefficients for the potential energy barrier.

In this work, we consider the probably simplest way of coating microemitters which is utilizing NaOH. This material is deposited on the tips' surfaces by the standard etching process and usually washed off before any further processing. Here, we only coarsely wash the surface allowing a more or less thick layer of NaOH to remain on the surface and subsequently we analyze its properties as a coating material.

#### Experimental

The preparation of tungsten microemitters of a tip radius of less than 100 nm is generally performed by an electrolytic etching technique as described in the literature [2,9]. The tips here are produced from a 0.1 mm high purity tungsten wire by electrolytic etching at the meniscus of two molar sodium hydroxide (NaOH) solutions. According to the standard procedure, the tip would be cleaned directly after the etching by repeatedly immersing it in distilled water and finally subjecting it to ultrasonic cleaning.

Contrary to this standard procedure, we avoided the thorough cleaning leaving a layer of NaOH behind on the tips' surfaces. The thickness of this NaOH layer was influenced by partial cleaning through immersing the tips in distilled water for one, six or twelve time(s), respectively. The resulting layers will be referred to as thick, medium or thin in that order.

The analyses were essentially carried out using a field emission microscope. This system could be evacuated to a base pressure of about  $10^{-8}$  mbar after baking at ~180 °C overnight. The cathode was mounted ~10 mm away from the phosphorus screen and a current limiting resistor of 20 M $\Omega$  was used.

#### Results

The emission characteristics obtained from three sets of etched tungsten tips - coated with NaOH layers of different thicknesses - show a generally interesting behavior. As the applied voltage is slowly increased, the emission current "switches on" from approximately zero (i.e., a very low value, usually a few nA) to a stable saturated value of 10 to 30  $\mu$ A. By slowly reducing the voltage, the current decreases smoothly until it vanishes at a certain threshold voltage. For all samples, the IV plots (Fig. 1) show the usual exponential - like increase at low voltages (< 1000 V). At higher voltages, the current-voltage-relations turn into straight lines. This indicates that the emission current in this region is bounded by limited electron supply.



FIG. 1: The emission characteristics of micropoint cathodes coated with different layers of NaOH. The IV plots show the usual exponential - like increase at low voltages (< 1000 V). At higher voltages, the emission currents appear to be bounded by electron supply limits, resulting in rather straight current - voltage relations in this region. Comparing the three graphs, we find that the current increases with the thickness of the coating layer. Note that the data have been recorded during voltage decrease. (The connecting lines in the graphs are for eye-guidance only.)

Comparing the three graphs in Fig. 1, we find that the current (at a given voltage) generally increases with the thickness of the coating layer. Thus, the NaOH coating apparently enhances the electron emission at the surface.

The FN plots of the emission characteristics (Fig. 2) show a variety of effects. In the right hand part, the relation is mostly linear in all graphs indicating the usual field emission behavior. However, for higher electric field strengths (left of about 1.7 in 1000/U) the graphs bend and finally start decreasing slightly. This again indicates that the emission currents are bounded by an electron supply limit. The graphs of the tips coated with the medium or thick layer

show a specialty. The two maxima found at about 0.8 and 1.3 for the thick coating as well as 0.7 and 1.7 for the medium coating (each in values of 1000/U) might imply field dependent structural reorganization of these NaOH layers. As the data have been recorded during voltage decrease, this effect was found to be reversible upon cycling the voltage.

Additionally, we find protective properties of the NaOH coating; i.e., all of the tips were able to serve rather high currents of up to 20  $\mu$ A or more with good stability and without being blown out. In Fig. 3 the highest current recorded during these measurements (which is 28.7  $\mu$ A) is shown.



FIG. 2: The FN plots of the emission characteristics of the composite cathodes show several effects. In the right hand part of these plots, we find a mostly linear relation indicating the usual field emission behavior. However, at increasing field strengths the graphs bend (at about 1.7 in 1000/U) and finally start slightly decreasing, which indicates that the emission currents are bounded by an electron supply limit. The graphs of the tips coated with the medium or thick layer show two maxima (each found at about 0.8 and 1.3 for the thick coating as well as 0.7 and 1.7 for the medium coating), which might imply field dependent structural reorganization of these NaOH layers. Note that the data have been recorded during voltage decrease. (The connecting lines in the graphs are for eye-guidance only.)



FIG. 3: The IV plots of the emission characteristics during the voltage decrease of another cycle for the emitter coated with a medium NaOH layer. Note that the tip could be operated at a maximum emission current of 28.7 μA without being destroyed.

#### Conclusions

Tungsten based microemitters have been prepared by electrolytically etching a 0.1 mm high purity tungsten wire at two molar NaOH solutions; i.e., using the same technique as employed by Müller in 1937. Contrary to the standard procedure, the tips' surfaces have not been cleaned off NaOH solution by ultrasonic cleaning in distilled water. Thus, a layer of NaOH remained on the surface, which provides a novel coating material.

The presence of emission current switch-on effects during initial voltage increase and the rather non-linear FN plots indicate the presence of a dielectric coating layer on the emitters' surfaces. The nature of the current saturation phenomenon with stable charge distribution on the emitters' surface is not fully understood. Amongst other possible explanations, it could indicate a new field emission mechanism. Further research will be needed to clarify this point. Nonetheless, it appears that such tips might provide an electron source with stable supply mechanism of saturated current for technological application.

All of the tips were able to serve rather high emission currents of up to  $20 \,\mu\text{A}$  or more

without being destroyed. This indicates protective properties of the NaOH layer on the tips' surfaces.

As a further step, it might be interesting to investigate the long-term storage stability of this coating material. This seems particularly relevant as – in a related investigation – we found that TCNE, MgO and ZnO coated tips can maintain their original properties while being stored under standard atmosphere conditions for more than a decade.

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