MORPHOLOGY DIFFERENCE OF IDENTICAL PREPARED FIELD-EMITTERS

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Abstract: This paper deals with the physical characterisation of experimental field-emission cathodes. These cathodes poses quality source of electrons for devices which uses focused electron beam, such electron microscopes or semiconductor inspection tools. The cathodes with identical constructing parameters have been prepared to estimate theirs reproducibility and define the differentiation in morphology after producing several numbers of than cathodes.

Keywords: identical prepared field-emitters, NaOH solution

1. INTRODUCTION

For the cathode preparation, polycrystalline-tungsten wire of the diameter of 0.38 mm, immersed in the solution of NaOH is mostly used. The NaOH solution is present in two molar concentrations (2 and 0.2 mol·dm^{-3}) for the first and second etching phase. Electrochemical polishing is performed in 0.2 mol·dm^{-3} solution. The moving part of the etching setup is made of micro-metric stepper-motor-controlled device which allows to assure accurate movement. Static part of the etching setup contains of the chemically resistant cylinder in which the electrolyte is located. Paper deals with seeking of the average statistical field-emitter that can be designed using described method.

Basic review of electrochemical etching methods and their description, was published in 1991 by Melmed [1]. All of these methods are based on common principles. During the etching process, the etched wire is immersed in to a grounded metal cylinder. For the cylinder, it is necessary to be manufactured of a proper metal, which would not react with the electrolyte inside since the cylinder acts like cathode in the system. However a Petri dish, with immersed cathode (made of stainless steel

for example) can be used as well. The cylinder is filled with a liquid electrolyte, mostly with NaOH or KOH when working with tungsten wire. The etch procedure is processed inside the cylinder where the etched wire acts like an anode. From the chemical point of view, the process taking place can be described as anodic dissolution. The name of the method is derived from the bottom part of the etched wire, which drops off (down) during the etching procedure. The curvature radius of the tip apex at the moment of the drop off can be expressed as [2]:

$$r = R\sqrt{\frac{\mathrm{L}(\rho_{\mathrm{w}} - \rho_{\mathrm{e}})}{\sigma_{\mathrm{TS}}}},$$

where the R and L are the dropped part radius or length, σ_{TS} is the ultimate tensile strength and ρ_{w} and ρ_{e} are the tungsten and electrolyte densities. This means that the resulting tip sharpness depends on the dropped part dimensions which should be as small as possible. The small mass of the dropped part minimizes some negative effects connected with sudden release of the stored elastic energy when the wire is broken. If the energy release increases to high value it may cause the

tips to recoil, melt or bend causing blunting and tip apex deformationthe cathode surface, that would lower the cathode current.

2. ETCHING METHOD

Main requirement of etching setup was to design fully and self-acting system, which would be capable of driving all the necessary processes centrally by single computer. This means that the computer should measure and record physical quantities along with sending and receiving instrument commands using the GPIB communication interface. All necessary data is acquired continually during the preparation which allows performing flexible control of whole process. Controlling application was completely programmed in MATLAB language which offers easy implementation and communication with external instruments (multimeter, programmable DC source and mainly the micro-metric lifter) as well as easy modification of the current etching algorithm.

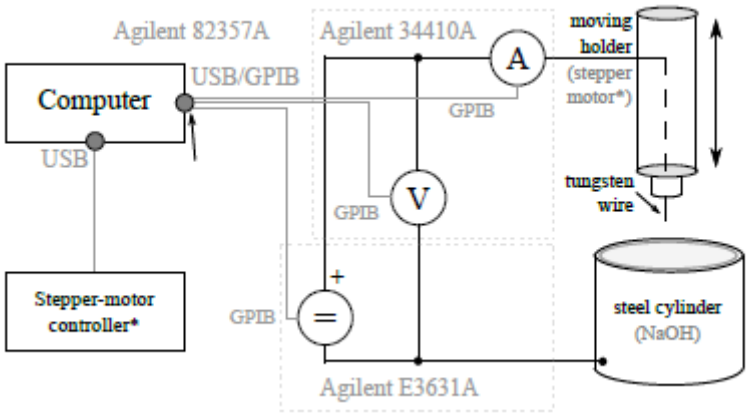

Fig. 1: The experimental setup of the electrochemical etching setup.

Whole preparation process is driven and evaluated by the computer in the real-time which allows detection of momentary preparation state continually in time, and to return appropriate action based upon newly occurred events. The zero position of the wire (position when the wire touches the solution surface) is set with micrometric precision according to the measured conductivity. The main idea is to trigger the software, whenever the etched wire touches electrolyte surface, which causes quick current increase for about tens of microamperes. This principle is used in whole preparation whenever is necessary to set the bottom part on the electrolyte surface.

Using that etching method 3 experimental field-emission cathodes have been prepared. All three has identical etching conditions: the length of the first and second phases, bias voltage, NaOH solution, current limit for the second phase, diffusion coefficient, etc. Additionally has been designed one experimental cathode the only difference from others consists in changed current limitation for the second phase. It was performed to observe the influence of current limitation parameter on forming the cathode`s tip.

3. RECEIVED EXPERIMENTAL SAMPLES

To design a proper method able to control the etching current, it is necessary to describe the etching current and to understand its behavior during the pursuit of the wire etching. As mentioned above, the tip etching consists of two phases. The first phase is performed until the wire is significantly constricted and thus prepared for the second phase. The slope of linear regression of the etching current for all four cathodes is almost the same and can be seen in fig. 2, where the slope is equal to -12.07×10^{-3}. Consequently, we are able to estimate the drop off time from the initial current

based on the result of linear regression. However this is not used for the first phase, since for the two-phase etching, the phase one is always stopped prematurely and continued in different electrolyte as a phase two. So this approach can be used only when carrying on the single-phase etching. The current for the second phase can be also modeled using linear regression-function, where the average meaning of the slope for the 2nd phase equals to -7.69×10^{-3} for field-emitters with I_{limit} = 10mA and equals to -0.091×10^{-3} for field-emitters with I_{limit} = 5mA. Etching continues until the bottom part drops off. As it`s demonstrated in figure 2 the limitation current has been saved during the whole period of the 2nd phase only for the last prepared cathode whereas for others it presents a linear regression.

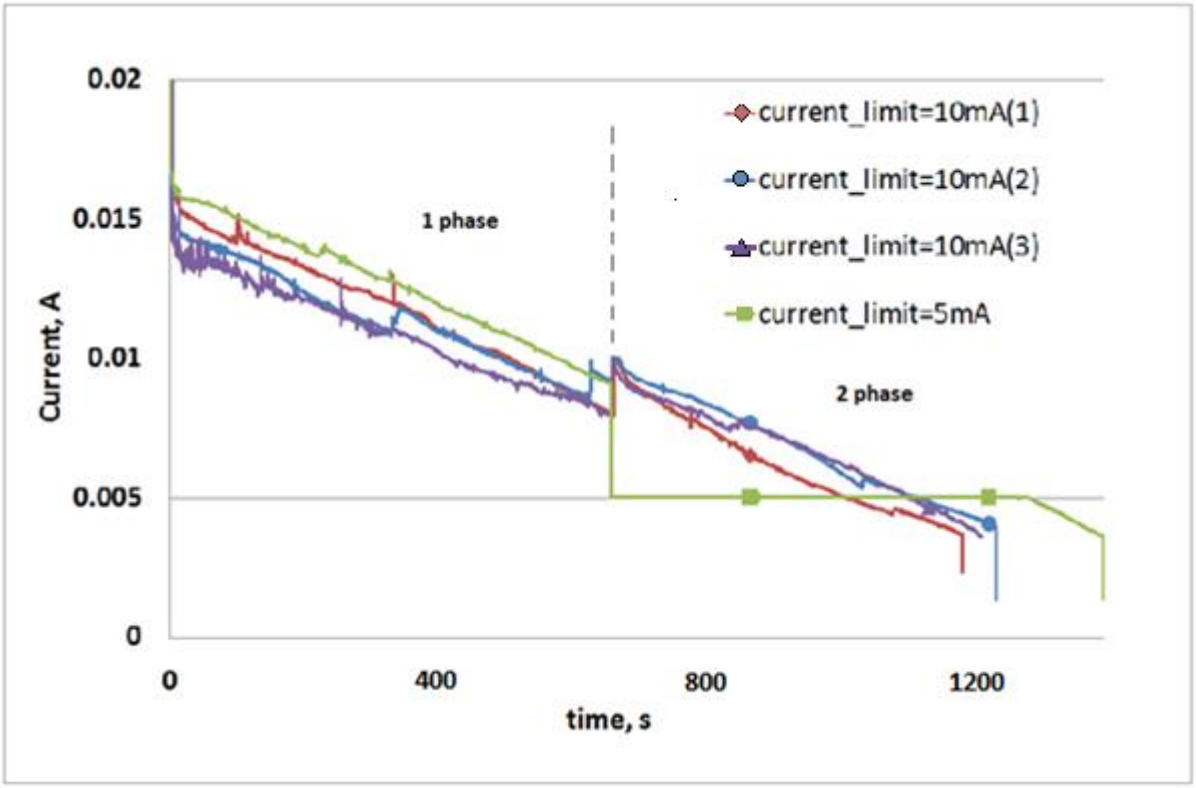

Fig. 2: Variation of the etching current as a function of etching time

According to our experiments, there is a clear relation between the length of these two etching phases and the diameter of the etched wire in order to reach desired tip geometry and avoid tip blunting. This ratio was named as phase-length ratio it can be expressed as:

$$\mathrm{plr} = \frac{t_1 - t_0}{t_2 - t_0},$$

where t_0 is the initial etching current, t_1 is the current at the time of start of the second phase, and t_2 is the drop off time. Obtained data for *plr* are illustrated in figure 3. The values of the *plr* coefficient for identical prepared cathodes varies from 0.540 to 0.564 that shows good accuracy and reproducibility of experimental device. The root mean square (rms) has been calculated and was defined as plr=0.551. Considering the field-emitter with 5 mA current limitation it rather noticeably differs from received meaning, taking the value of 0.472.

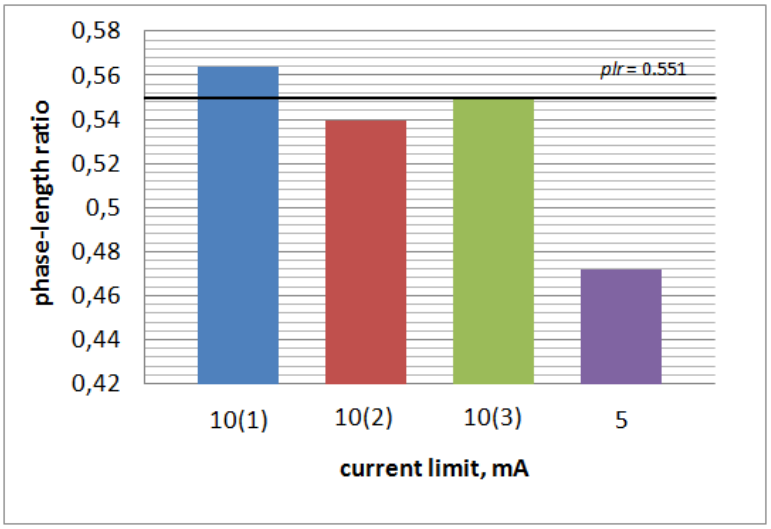

Fig. 3: Variation of the phase-length ratio as a function of current limitation for the second etching phase

4. MORPHOLOGY OF DESIGNED CATHODES

It is well known that a smaller radius of curvature (ROC) is required for high resolution images and that a lower aspect ratio is preferable for the reduction of noise induced by flexible vibration. The aim of the drop-off technique has mainly focused on obtaining a small radius of curvature of the tip apex in a reproducible way. The basic topography analysis has been performed using scanning electron microscopy (SEM) which shows a slightly visible distinction in shapes of the cathodes, which have been fabricated under different current limits (5 and 10 mA). Nevertheless, first three field-emitters demonstrated in figure 4 definitely have a more sharper tip and more expressed lateral etching then the last one, that suggests an improvement in output characteristics.

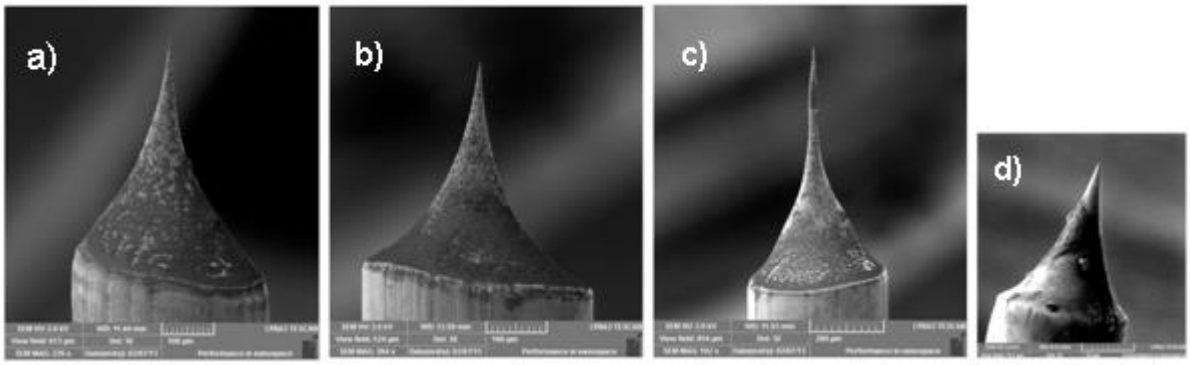

Fig. 4: Morphologies of W tips after etching at NaOH solution with current limit for the 2nd phase equals 10 mA (a, b, c) and 5 mA (d)

It is well known that a smaller radius of curvature (ROC) is required for obtaining high-quality field-emitters and that a lower aspect ratio is preferable for the reduction of noise induced by flexible vibration. The aim of the drop-off technique has mainly focused on obtaining a small radius of curvature of the tip apex in a reproducible way. A very similar sharpening behavior was observed for three cathodes constructed using identical parameters. It can be better catch sight of minimal difference in curvature radiuses of two such prepared tips in figure 5. The estimated values of those radiuses us about 120-140 nm.

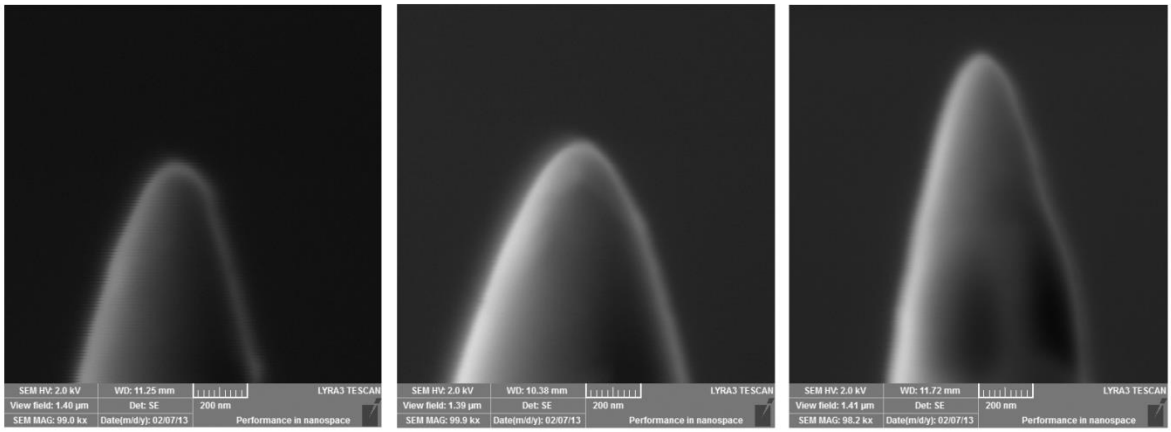

Fig. 5: Morphologies of W tips curvatures after etching with identical parameters

5. CONCLUSIONS

Morphology analysis of the ultra-sharp tungsten field-emission cathode`s has been performed. The cathodes with identical constructing parameters approved the relative accuracy of reproducibility and suitable tips radius of curvature. After the theoretical introduction and sample description, specimens have been prepared using method of the two-phase etching, the phase one has been stopped prematurely and continued in different electrolyte as a phase two. The basic topography analysis has been performed using scanning electron microscopy (SEM) which shows minimal distinction in shapes of the tips, which have been designed with identical construction parameters. The future receiving of tungsten field-emission tips with more quality parameters proposed to make changes in conditions of the experiment.

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