

Testing the performance of tungsten-graphite cathodes as MEMS Electron microscope electron source

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Abstract

The cold field emission (CFE) performance of tungsten-graphite single-tip field emitters (WG-STFE) has been under study in the past few years. WG-STFEs have shown outstanding emission characteristics and stability. Moreover, this type of cathode has been reported previously to have exceptional performance in scanning tunneling microscopy outside ultra-high-vacuum environments. This work presents preliminary CFE results of exfoliated graphene layers deposited on the nanotip of a tungsten field emission cathode. The obtained WG-STFEs were operated in a 3D-printed MEMS electron microscope testing model for later applications in MEMS electron microscopy. The results were promising, where a maximum emission current of 25 μA was obtained with an extraction potential difference of 0.8 kV, in addition to the possibility of focusing and controlling the generated electron beam using a 3D printed Einzel lens system. The conductivity of the 3D prints was obtained by coating the 3D electrodes with exfoliated graphite coatings of the same material.

Introduction

MEMS electron microscope represents a pioneering methodology for miniaturizing transmission electron microscopy using the technology of microelectromechanical systems. In this technology, field-emission electron guns are used and designed to produce a stable electron beam of small spot size, high emission current density, and long life and operational times. This beam is crucial for obtaining high-quality images at the microscopic level [1, 2].

Despite their wide availability and compatibility with MEMS fabrication, silicon-based field emission cathodes have several limitations for MEMS applications. This is because silicon tips, as semiconductors, are prone to blunting or melting when exposed to strong electric fields over time. In addition to their low thermal conductivity and melting point, they are less robust under ion bombardment and require a complex fabrication procedure for sharpened tips [3].

Moreover, recent studies showed that traditional uncoated tungsten field emission cathodes have several limitations when operated outside of ultra-high vacuum conditions, such as high threshold voltages, short lifetime and operation time, and widespread electron beam. However, it was also shown that coating the nanoapex of such cathodes with graphene nanolayers helped to improve their performance in scanning tunneling microscopy, along with excellent field emission performance and characteristics of exfoliated graphite coatings when deposited on a glass substrate [4, 5].

This study presents the implementation methodology of a tungsten-graphite single-tip field emitter as the electron source of a MEMS electron microscope. In addition, preliminary results are presented.

Methodology

A. Samples preparation

The tungsten nanotips were fabricated from 99.9% high-purity polycrystalline tungsten wires of 1 cm in length and 0.3 mm in diameter and following the procedure described in [6], along with an additional cleaning step in isopropanol (IPA). To prepare tungsten-graphite cathodes, clean tungsten tips were dipped inside colloidal conductive graphite liquid, which is prepared by diluting the liquid KONTAKT CHEMIE graphite 33 conductive paint (CRC Industries Europe, Zele, Belgium) in an ultrasonic IPA bath as presented in **Figure 1**.

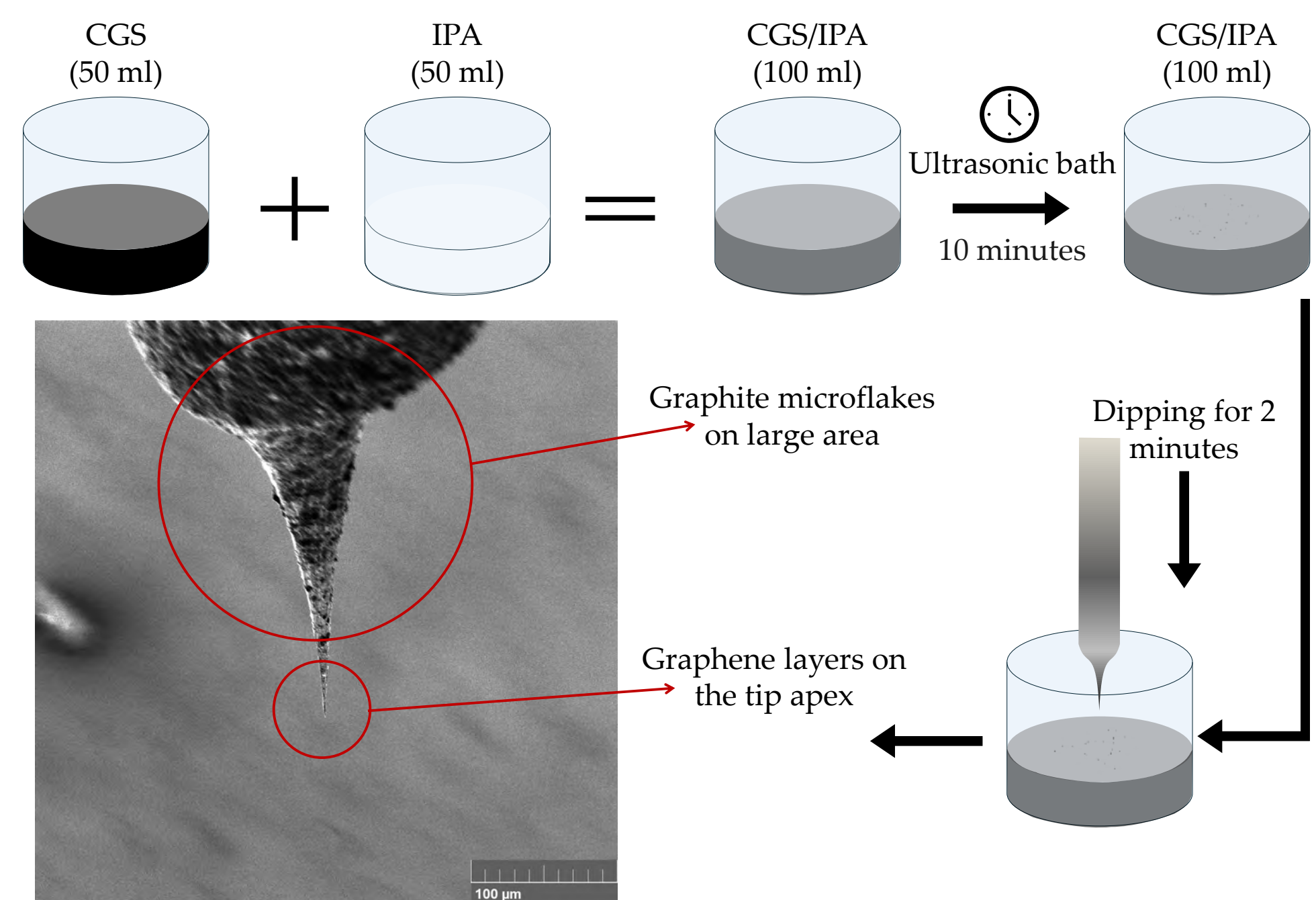


Figure 1. Schematic diagram to present the preparation methodology of exfoliated graphite solution and tungsten-graphite samples.

B. 3D printed field emission microscope

ASIGA Fusion GRAY V2 3D resin was used to print a class-wise constructed field emission microscope as presented in **Figure 2**. The electrodes were prepared in a cylindrical shape of 2 mm thickness and coated with the same exfoliated graphite solution. The extractor aperture had a diameter of 2 mm, while the Einzel lens electrodes had an aperture of 4 mm in diameter. The cathode was mounted to the base stage electrode holder, and the entire set-up was then installed inside a high-vacuum chamber of pressure of 10^{-3} Pa. Electrical conductivity for the cathode, electrodes, and collection screen was established using conductive carbon tapes. The emission current was measured using the cathode using a special home-programmed software.

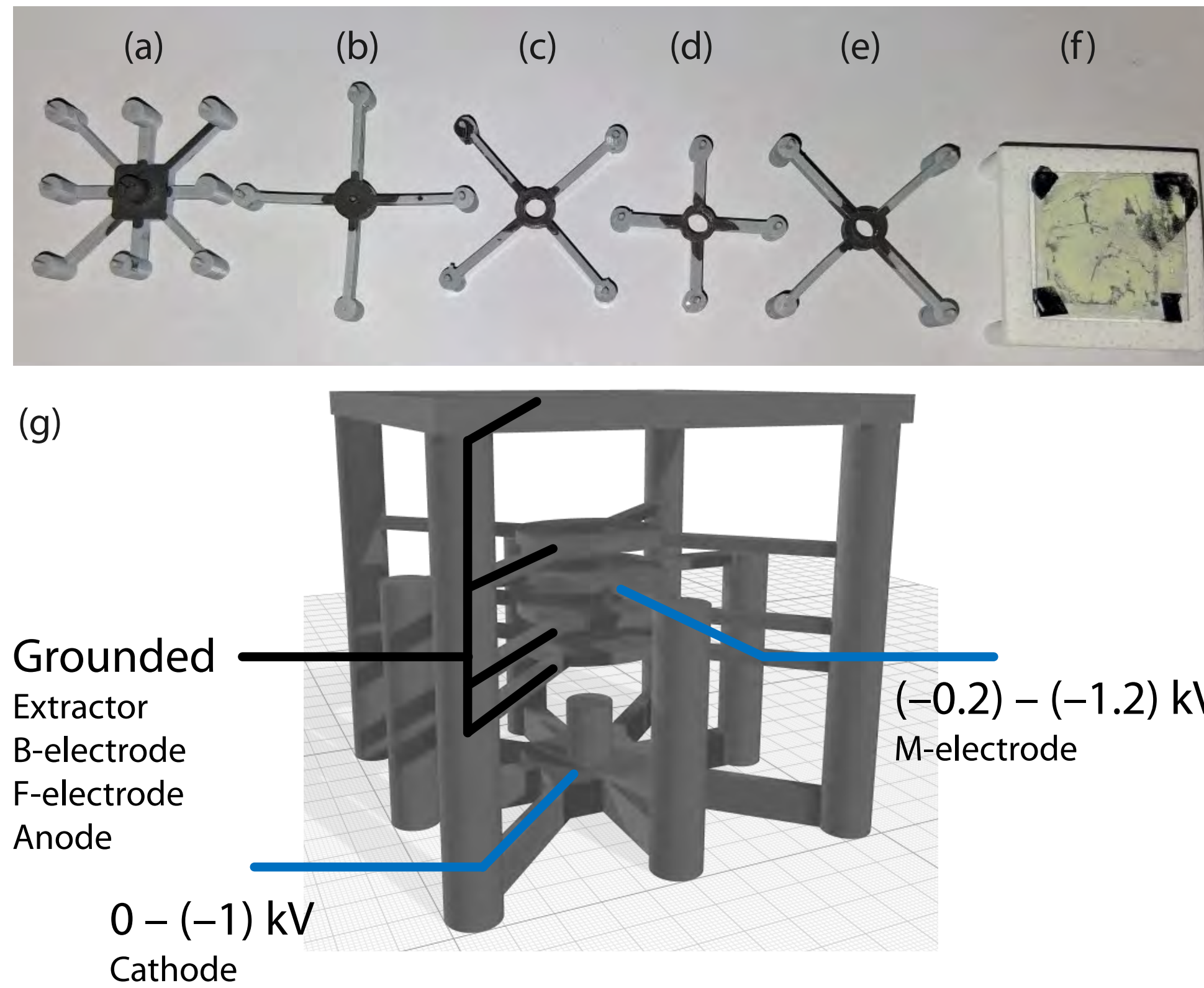


Figure 2. Parts of 3D printed class-wise constructed field emission microscope that presents its (a) stage and cathode holder, (b) extractor, (c) backward Einzel electrode, (d) middle Einzel electrode, (e) forward Einzel electrode, and (f) the collection screen (anode). The distribution of electrical contact is explained in the complete structure described in (g).

Results and discussion

Figure 3 shows the possibility of focusing the emitted electron beam by setting the middle electrode potential at different values close to the cathode potential. The potential was set from 400 - 1000 V for two cases where the anode was grounded in the first case, while it was biased to 1500 V in the second case.

Moreover, **Figure 4** describes the cold field emission characteristics represented by the current-voltage characteristics, Murphy-Good analysis plots for the voltage increasing part of the measurement, and the current-time characteristics for stability testing over 2 hours of operation time. The first sample was operated at 800 V with average current of 25.98 μA , the second sample was operated at 700 V with average current of 8.35 μA , while the third sample was operated at 600 V with average current of 3.79 μA .

Furthermore, the work function for colloidal graphite was measured as 5.09 eV using the ultraviolet photoelectron spectroscopy technique, where the $\kappa(\phi)$ function for Murphy-Good plots was 1.273361. The extracted parameters are presented in **Table 1**.

Table 1. The Murphy-Good analysis results related to Figure 4(b). In this table, OTR is the Orthodox test result, ζ is the voltage conversion length, f is the extracted scaled field range, and A_f is the formal emission Area.

Segment	OTR	Slope	f range	ζ (nm)	A_f
L_1	Pass	-9700.00	0.27-0.30	123.65	1.02 μm^2
L_2	Pass	-9217.00	0.31-0.37	171.50	4.63 μm^2
L_3	Inconclusive	-3521.00	0.48 - 0.62	44.89	0.05 nm^2
L_4	Pass	-7521.66	0.3 - 0.37	95.89	265.89 nm^2
L_5	Pass	-12947.92	0.16 - 0.19	165.06	8.67 μm^2

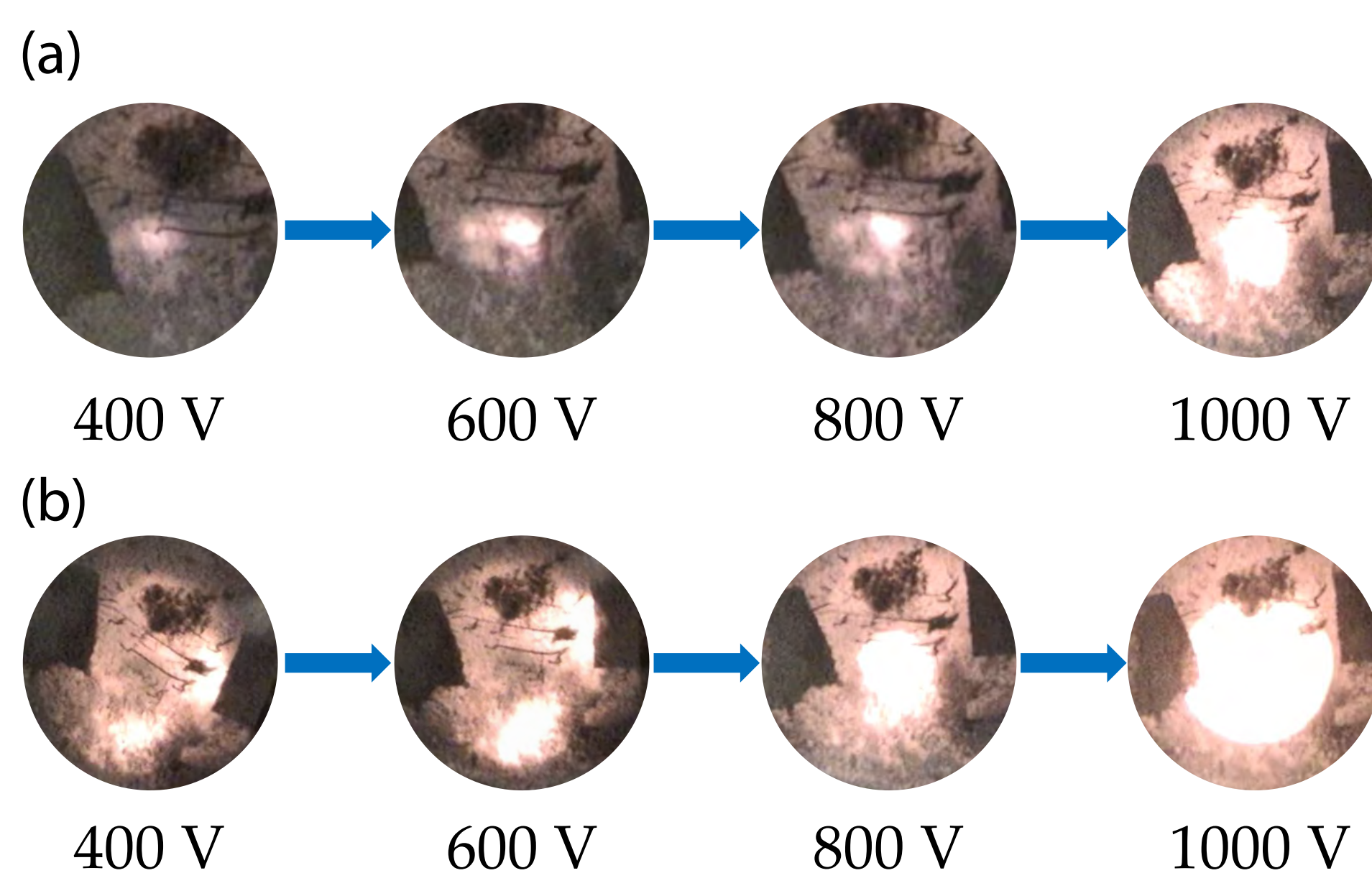


Figure 3. Changing of electron beam spot shape on the anode screen when changing the middle Einzel lens electrode potential for two cases: (a) when the anode is grounded and (b) when the anode is at potential of 1500 V.

Conclusions

Coating tungsten tips with colloidal graphite has several benefits for field emission applications. This procedure enhances the electrical conductivity of the oxidized nanotip region, providing higher and more stable emission current densities.

Moreover, the successful coating of the nanotip with graphenic layers provided enhanced hardness and other mechanical parameters along with higher resistance to environmental factors compared to tungsten oxide, which makes it more suitable for lower vacuum conditions.

Furthermore, this study shows that it was possible to control and focus the generated electron beam using Einzel lens system built in the form of small structures. Moreover, 3D printed field emission microscopes shows that this technology has promising applications in testing the prototypes of more complicated systems.

Future plans are arranged for better field emission characteristics and enhanced system alignment for full implementation of tungsten-graphite and tungsten-epoxy single-tip field emitters in MEMS electron microscopes.

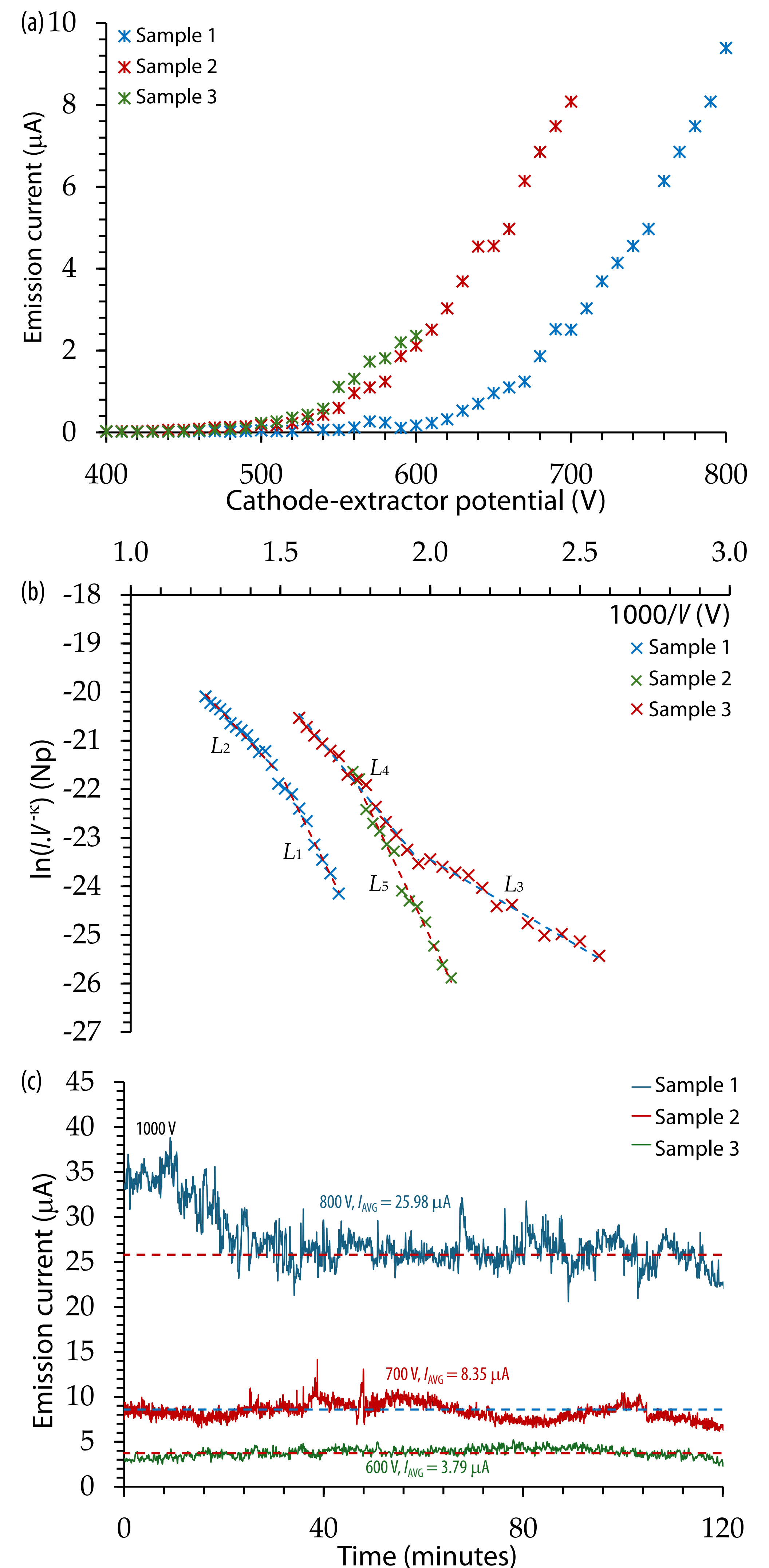


Figure 4. Field emission characteristics of three samples operated at three different voltages. In this figure, (a) represents the current-voltage characteristics, (b) represents the Murphy-Good plots, and (c) represents the current-time characteristics.

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