G Model
APSUSC-33194; No. of Pages 5
ARTICLE IN PRESS
Applied Surface Science xxx (2016) xxx–xxx
Contents lists available at ScienceDirect
Applied Surface Science
journal homepage: www.elsevier.com/locate/apsusc

Field emission from the surface of highly ordered pyrolytic graphite

Alexandr Knápek[a,*], Dinara Sobola[b], Pavel Tománek[b], Zuzana Pokorná[a], Michal Urbánek[a]

[a] Institute of Scientific Instruments of the ASCR, v.v.i., Královopolská 147, Brno, Czech Republic
[b] Department of Physics, FEEC, Brno University of Technology, Technická 8, Brno, Czech republic

ARTICLE INFO

Article history:
Received 3 December 2015
Received in revised form 29 April 2016
Accepted 1 May 2016
Available online xxx

Keywords:
Field emission
HOPG
Scanning near-field optical microscopy
Scanning electron microscopy

ABSTRACT

This paper deals with the electrical characterization of highly ordered pyrolytic graphite (HOPG) surface based on field emission of electrons. The effect of field emission occurs only at disrupted surface, i.e. surface containing ripped and warped shreds of the uppermost layers of graphite. These deformations provide the necessary field gradients which are required for measuring tunneling current caused by field electron emission. Results of the field emission measurements are correlated with other surface characterization methods such as scanning near-field optical microscopy (SNOM) or atomic force microscopy.

1. Introduction

A simple method utilizing the field emission of electrons has been devised to characterize the sample surface. Electron and probe microscopies were used to determine the structure of both the bulk sample and the partially exfoliated shreds of the uppermost layers of graphite in locations where field emission is observed.

2. Experiment

We used a sample of highly ordered pyrolytic graphite (HOPG), ZYA grade, purchased from MaTecK GmbH. The upper layers of HOPG were mechanically exfoliated using 3 M adhesive tape and the freshly prepared sample was immediately placed via a vacuum feedthrough into a high vacuum chamber with a residual gas pressure of 10^{-7} Pa. This chamber allows for sample cleaning using electron bombardment heating and/or argon ion sputtering prior to electron microscopy observation in the adjacent ultra-high vacuum (10^{-8} Pa) chamber. In order to remove any possible contaminants that may have stuck to the surface, the HOPG slab was annealed to 900° Celsius for 20 min by means of electron bombardment heating.

After the cleaning procedure, the sample was moved via a feedthrough to the ultra-high vacuum chamber equipped for scanning low energy electron microscopy (SLEEM). [1] This technique makes use of the Cathode Lens [2], which is basically an electrostatic decelerating field between the negatively biased sample and a grounded electrode above it.

The sample was connected to a negative DC source providing the necessary extraction voltage. The emitted electrons were collected by a cerium-doped Yttrium Aluminum Garnet (YAG:Ce) scintillator, allowing to measure the emitted current. Additionally, the set-up can be extended to display spatial distribution of defects by adding a CCD chip in the vacuum chamber.

The negative voltage was continually increased until the YAG scintillator detected impinging electrons. A significant part of the total emission current was captured by the thin conductive Indium Tin Oxide layer covering the YAG scintillator, which allowed to measure basic current-voltage (*I-V*) characteristics. In the measurement, the inter-electrode distance was kept constant at 6 mm, which required rather high extractor voltage to gain sufficient electric field intensity *E*. Using a simple diode *I-V* measurement set-up, the current was measured by detector of backscattered electrons the following way: the electrons were converted to photons by the YAG scintillator, the resulting photons then converted back to electrons when entering a photo-multiplier where this current was multiplied by several orders of magnitude, the output was amplified by an amplifier, sampled and recorded in a computer. A schematic arrangement is illustrated in Fig. 1.

In order to interpret the *I-V* behavior, the standard Fowler-Nordheim (F-N) theory was used.

Fig. 2 illustrates the *I-V* characteristics showing a nearly exponential increase of the current, where I [A] $= 2 \times 10^{-12}$

* Corresponding author.
E-mail address: knapek@isibrno.cz (A. Knápek).

http://dx.doi.org/10.1016/j.apsusc.2016.05.002
0169-4332/

Please cite this article in press as: A. Knápek, et al., Field emission from the surface of highly ordered pyrolytic graphite, Appl. Surf. Sci. (2016), http://dx.doi.org/10.1016/j.apsusc.2016.05.002

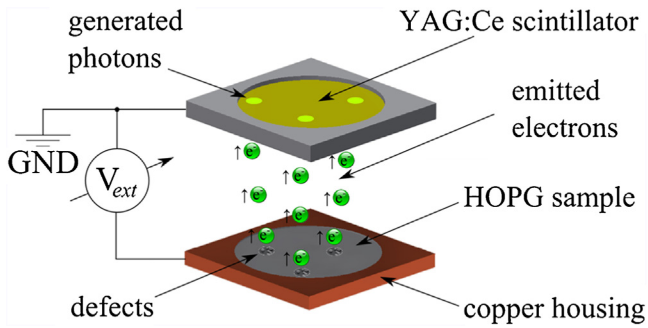

Fig. 1. Experimental setup; the HOPG sample is connected to the negative dc source and the YAG scintillator is grounded, serving as an extractor electrode.

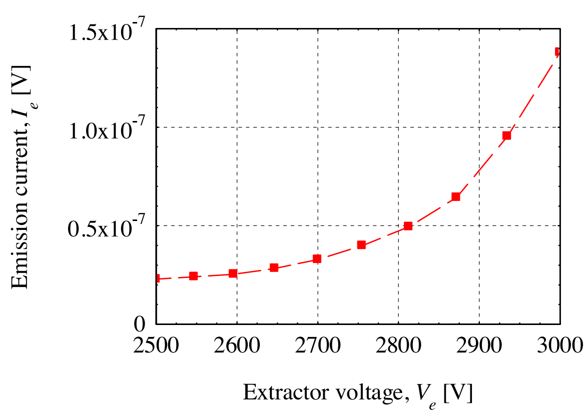

Fig. 2. *I-V* characterstics of the emission current as a function of applied voltage.

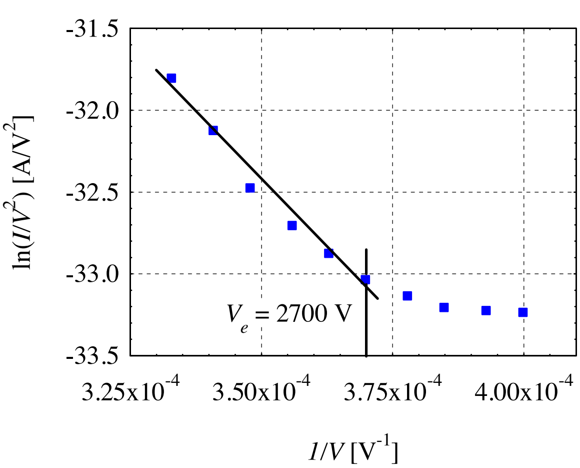

Fig. 3. Corresponding F-N plot for the *I-V* characteristics in Fig. 2.

exp(0.0036 V_e). Also the F-N plot (see Fig. 3) confirms the presence of electron tunneling because of the nearly linear plot.

As shown in Fig. 3, the non-linear behavior starts to appear at V_e = 2700 V suggesting the current contribution of larger defects, i.e. defects with lower field gradient. The nonlinearity of the plot can be explained by the presence of multiple microscopic electron sources. As each of them has a different geometry, the field gradient also differs, and with it the voltage necessary for the onset of field emission. This causes a gradual activation of particular sources making the *I-V* plot non-exponential [3].

For this reason it is also not possible to determine the exact work function and field enhancement factor β (the ratio of maximum electric field on the shred and the average electric field on the HOPG

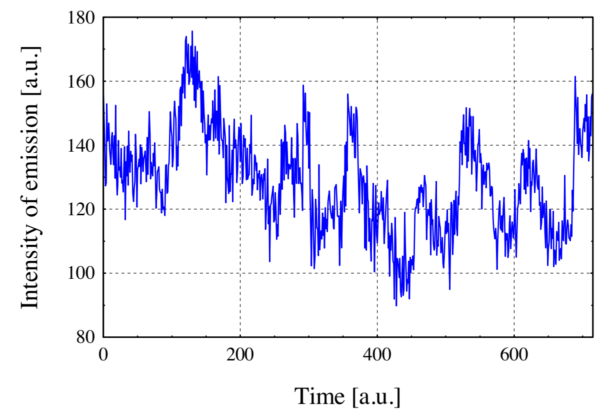

Fig. 4. Example of fluctuation of the total emission current of the HOPG slab at one particular voltage, V = 3010 [V].

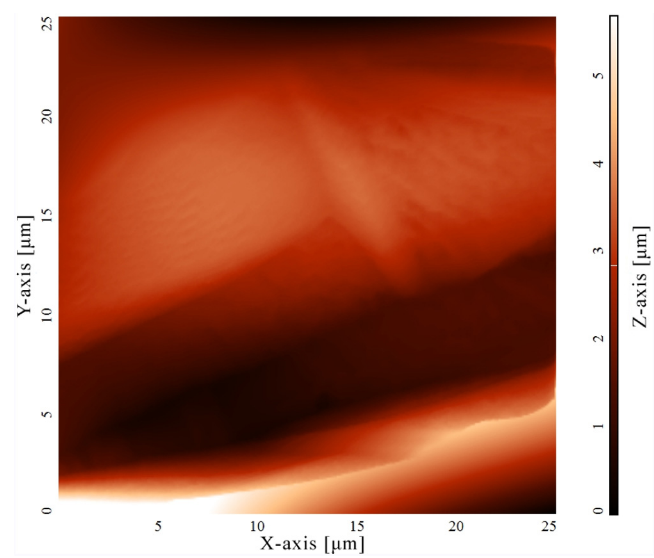

Fig. 5. Topography image of the HOPG surface, obtained by SNOM.

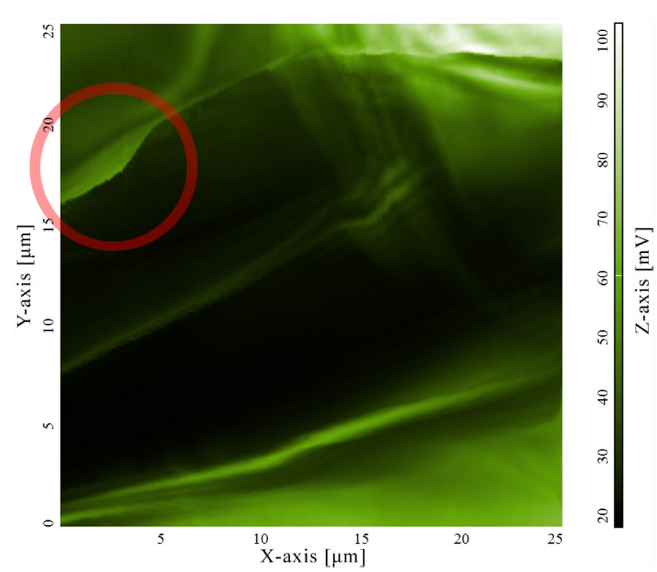

Fig. 6. Reflectance image from the same surface area as in Fig. 4, obtained by SNOM. The marked area shows a partly exfoliated shred of graphite surface. The milivolt scale denotes the photodetector signal intensity.

Please cite this article in press as: A. Knápek, et al., Field emission from the surface of highly ordered pyrolytic graphite, Appl. Surf. Sci. (2016), http://dx.doi.org/10.1016/j.apsusc.2016.05.002