AFM OF HOPG: CASE STUDY OF HOPG MILLING

Josef Komínek

Bachelor Degree Programme (3), FEEC BUT

E-mail: j.kominek10@seznam.cz

Supervised by: Dinara Sobola

E-mail: sobola@vutbr.cz

Abstract: The purpose of this work is study of highly oriented pyrolytic graphite (HOPG) processed by focused ion beam (FIB) using atomic force microscopy (AFM) and Raman spectroscopy. Due of its properties, HOPG have a promising potential and wide range of applications in nanotechnology. AFM demonstrates quality of the pattern created by FIB: topography of patterned area, shape of the edges, etc. Raman spectroscopy indicates defectiveness of the near surface area occurred due to FIB processing.

Keywords: EEICT, template, guide

1 INTRODUCTION

Surface analysis is an important part of nanotechnology research [1]. Among all existing methods, scanning probe microscopy (SPM) can be highlighted. This method quickly transformed from being available only to small groups to a widespread tool that is used by almost every laboratory that works in the field of nanotechnology. The correct choice of method and sample for calibration is a basis for obtaining reliable data [2]. One of such samples is HOPG. Surface analysis of HOPG by scanning tunnelling microscopy allows depicting of individual atoms even in air at normal conditions.

HOPG is one of allotropic modification of graphite. Oriented layers provide option of observing one undivided layer or multiple layers. Atoms in single layer create hexagonal grid. The symmetry can be broken and distance between carbon atoms can change due to irradiation, mechanical fissures and various impurities. Its layer structure also causes that its conductivity is anisotropic. Covalent bond in this composition provides better conductivity. Forced electron can travel through layers. In non-oriented graphite, layers are composed in various directions causing its conductivity is isotropic.

One single layer with hexagonally composed grid layer is called graphene. Graphene provides promising technology and research potential. If individual atoms are cut out of graphene grid correctly, residual charge caused by electrons that originally constituted covalent bonds causes layer to bend in certain ways creating various carbon forms hitherto unobserved, like fullerene, nanotubes, etc.

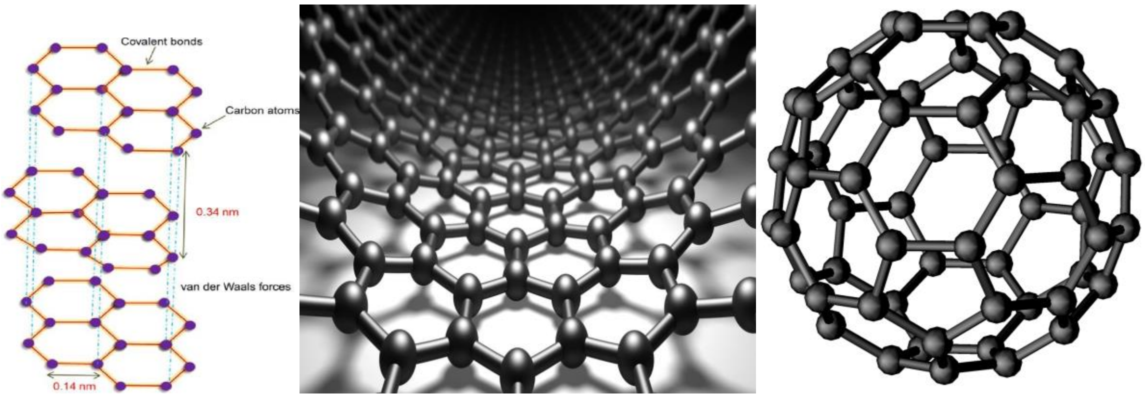

Figure 1: Graphite (left) [5], graphene (middle) [6] and one of fullerenes (right) [7] are shown.

2 EXPERIMENT

HOPG sample of ZYA quality was chosen (from NT-MDT SI). The first step was preparation of sample itself: using adhesive tape, top layer was separated. Focused Ion Beam/Scanning Electron Microscope FEI Helios NanoLab 660 was used for processing. The area of the sample was localized using electron imaging. Short time irradiation by ions, even with low intensity, can cause local heating and inter-layer ionization resulting in temporal or permanent local surface bending. Series of dots with different depth were prepared using FIB at 30kV and 2.5 nA. (Fig.2).

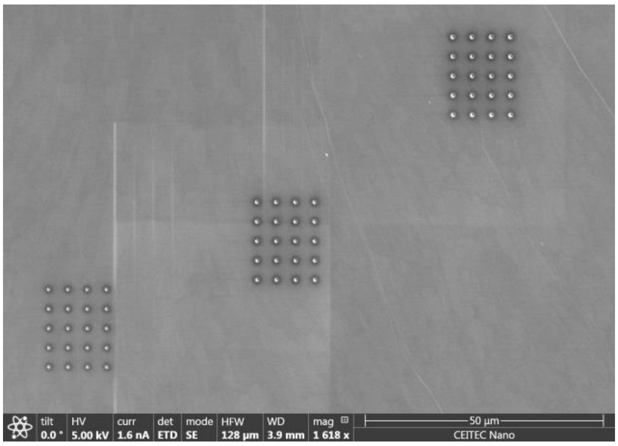

Figure 2: SEM image of the patterns prepared by FIB 0.5 µm diameter with depths of 200 nm (left), 400 nm (middle) and 600 nm (right)

Raman spectroscopy was done using WITec confocal Raman imaging system alpha300 R. Typical phonon for undamaged HOPG surface, called G phonon, lies at approximately at 1600 cm^{-1}. The D-peak can be observed at HOPG Raman spectra at 1350 cm^{-1}. This peak corresponds to disorder of the HOPG layers. Relative intensity of G and D peak provides information about quality of HOPG surface. Radiated dots of different depths and 0.5 µm diameter were investigated by Raman. Resulting spectra presented in Figure 3. Increasing of D-peak for irradiated areas is observed. It is caused by defects in the layer arrangement caused by ion beam.

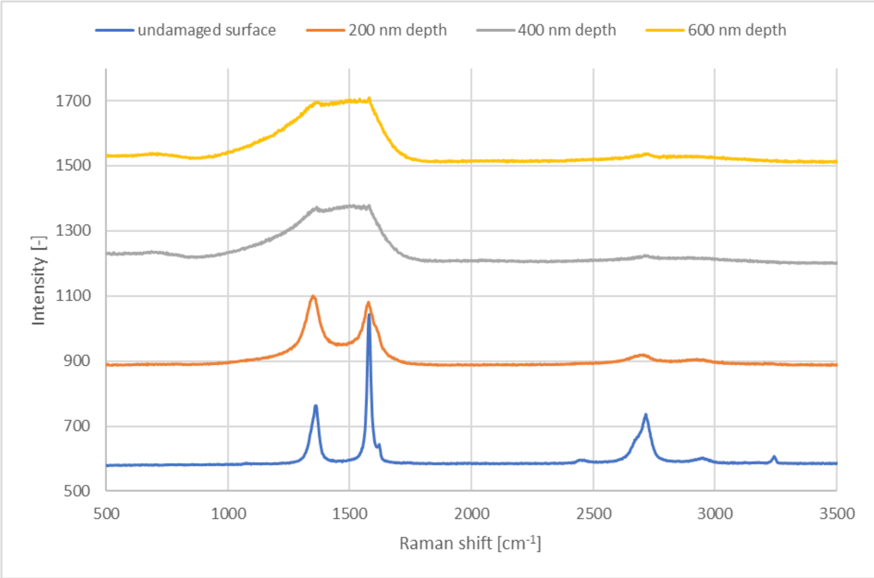

Figure 3: Raman spectra of pure HOPG and HOPG processed by FIB

Resulting topography was observed by SPM NTEGRA PRIMA (from NTMDT SI). Dimensions of the pattern were studied using atomic force microscopy (AFM) in tapping mode. The holes with 200 nm depth were chosen (Fig.4).

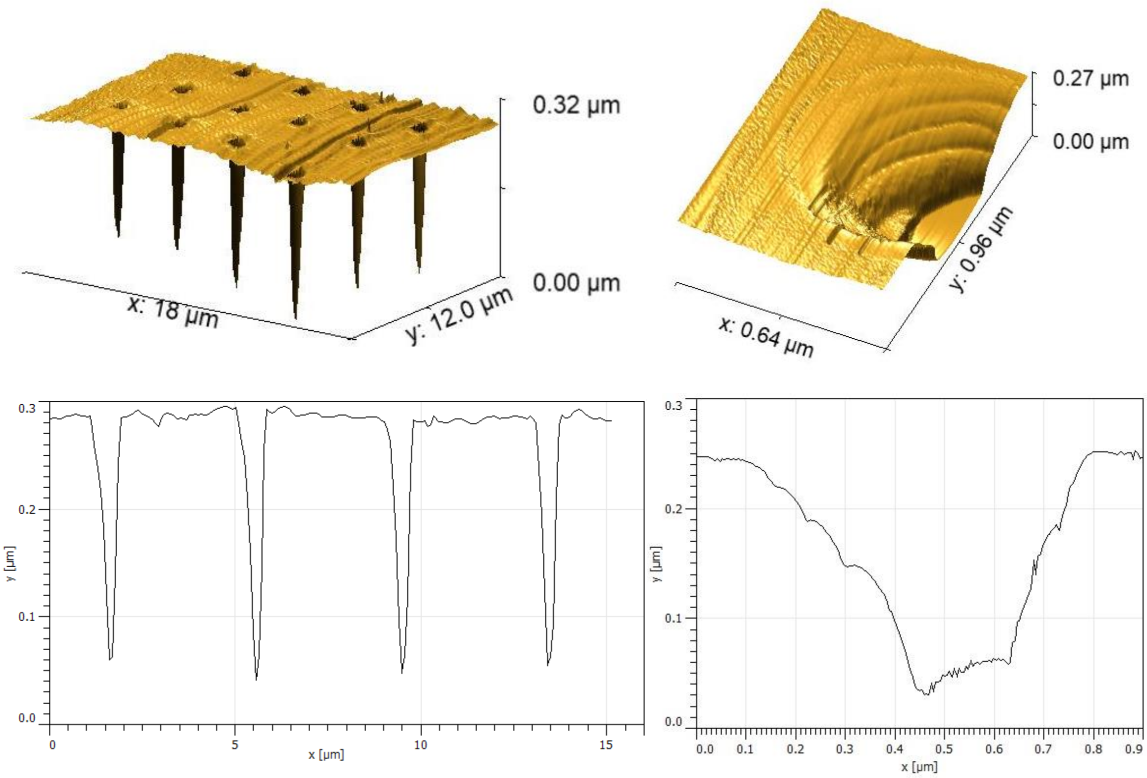

Figure 4: AFM images of series of dots with diameter of 0.5 µm and depth of 200 nm. 3D topography and single raster are shown.

Total height of the sample topography 0.32 μm is explained by presence of surface roughness (upper left). The estimated depth of milled areas is 200 nm (lower left). From lower left and lower right pictures can be observed, that lateral size of the pattern is larger than defined during milling 0.5 μm. Local heating [3] and cascade effect [4] could be responsible for this.

3 CONCLUSION

The performance of FIB on HOPG surface was studied here. Raman spectroscopy of milled areas indicates presence of broken bonds and defects appearance. AFM shows difference between demanded and achieved geometrical sizes of patterns. AFM data can be used for adjusting of FIB parameters for pattering of the surface.

ACKNOWLEGEMENT

Research described in the paper was financially supported by the Ministry of Education, Youth and Sports of the Czech Republic under the project CEITEC 2020 (LQ1601). Part of the work was carried out with the support of CEITEC Nano Research Infrastructure supported by MEYS CR (LM2018110).

REFERENCES

[1] A. Knápek, D. Sobola, P. Tománek, Z. Pokorná and M. Urbánek, „Field emission from the surface of highly oriented pyrolytic graphite“, Appl. Sufr. Sci., vol. 395, pp. 157 – 161, 2017

[2] D. Sobola, Ş. Ţălu, S. Solaymani and L. Grmela, „Influence of scanning rate on quality of AFM image: Study of surface statistical metrics,“ Microsc. Res. Tech., vol. 80, no. 12, 2017.

[3] Zhenhai Wang, Lijiang Gui, Danhong Han, Zhuang Xu, Li Han & Shengyong Xu, „Measurement and Evaluation of Local Surface Temperature Induced by Irradiation of Nanoscaled or Microscaled Electron Beams“, Nanoscale Research Letters, volume 14, Article number: 31 (2019)

[4] Archanjo BS, Maciel IO, Ferreira EH, Peripolli SB, Damasceno JC, Achete CA, Jorio A. Ion beam nanopatterning and micro-Raman spectroscopy analysis on HOPG for testing FIB performances. UI-tramicroscopy. 2011 Jul;111(8):1338-42. doi: 10.1016/j.ultramic.2011.04.007

[5] Structure of Graphite. In: https://researchgate.net/figure/Structure-of-graphite_fig24_283081307

[6] Structure of Graphene. In: https://www.ctimaterials.com/resources/graphene-battery-users-guide

[7] Structure of Fullerene. In: https://www.novarials-store.com/products/fullerene-c60-1