Review Report on PhD Thesis

Faculty: **Central European Institute of Technology**
Brno University of Technology in Brno

Academic year: **2024/2025**

Student: **Oleh Martyniuk**

Doctoral study program: **Advanced Materials and Nanosciences**

Supervisor: **doc. Dr. Ing. Petr Neugebauer**

Reviewer: **Dr. Yahya Moubarak Meziani**

PhD thesis title: New generation of graphene bolometers

Topicality of doctoral thesis:

In the present work, the Ph.D. student fabricated and characterized new types of bolometers that are based on CVD-grown graphene from Graphenea-Spain. He implemented arrays of quantum dots on graphene (GQD) and characterized them under 100 GHz radiation excitation and at low temperature. Two devices were successfully fabricated and characterized: two and eight parallel GQDs. A clear bolometric effect was observed from both devices at 4.5K and versus both drain-to-source voltage and gate voltage. The signal was related to the quantum confinement and the Coulomb blockade effect. The thesis was organized in 6 chapters as follows:

Chapters 1 & 2 were dedicated to the introduction and to exhibiting the aim of the work. Chapter 3 deals with the background of the bolometric detection and their main keys parameters (Responsivity and Noise Equivalent Power) as well as state of the art of graphene-based bolometers. In chapter 4, the main part of the thesis, he introduces the different steps of the fabrication process including failures and proposed solutions. He started by introducing the different steps: lithography techniques, deposition, and etching process. He showed the results obtained at each step and the observed failure with different doses till obtaining the right one. Then he started the implementation of the quantum dots and processing of the contacts for wire bonding. This chapter shows the heavier works of the present thesis and highlights the skills of the student in the fabrication process. Chapter 5 describes the characterization techniques that were used along the fabrication process. Raman spectroscopy to ensure the monolayer property of the CVD-based graphene after transferring graphene from Copper to the Si/SiO2 substrate. The Scanning Electron Microscope was also used to explore the GQD and their quality. Finally, the main system, THz FraScan Spectrometer, was introduced and described in detail. This system was used to measure the resistance of the GQDs from room temperature down to 2K and its bolometric behavior under excitation of 100 GHz source from Virginia diode. In chapter 6, he describes and discusses the results obtained. First, the devices were characterized at room temperature to ensure their good behavior. Resistance of

each device was measured versus gate voltage and at a fixed drain-to-source bias. Both devices exhibit good behavior with more variation of resistance for negative gate voltage which indicates that the devices are p-type with holes as majority carriers. Then it follows measurements of resistance as function of temperature. Increase of resistance was observed from 50K down to 2K which was used to extract the activation energy of each device. Resistance shows different behaviors upon the applied gate bias. High increase in the resistance was observed for positive biases for both GQDs. At 4.5K & Vds=26mV, the measured current versus gate bias shows different spikes that were related to the quantum confinement which induces Coulomb blockade and conductivity suppression in a specific range of gate bias, known as the transport gap. The measurements of drain-to-source current versus gate voltage with and without excitation show a clear increase of the current (decrease in resistance) when the THz source was switched on. The increase of current was more pronounced for the 8-dots than for the 2-dots which mainly related to the impedance matching since the 8-dots has lower impedance. This observation is a clear demonstration of bolometric behavior as when the radiation excites the device, the electronic temperature increases which activates the electrons across the dot increasing the current. Finally, he presented some results of THz detection by using SiC epitaxial graphene where similar behavior was observed under excitation at 150 GHz and at 3K. From those data, the responsivity around 10^9V/W and thermal conductance around 25 pW/K were obtained showing competitive values.

Meeting the goals set:

The main objective of this work was the fabrication of a bolometer using CVD-graphene based quantom dots. Graphene is a single layer of graphite with thickness around 0.35nm and highly transparent in visible range. Handleing and fabrication of quantum dot from graphene is a highly challeng. The process of fabrication is time cosuming and involve an import workload with many failures. The candidate succeed in this task and fabricate two devices one with 2-dots and other with 8-dots that were used as a new bolometric detector. One of the goals was to increase the active area through an array of quantum dots which was alsove achieved with the 8-dots. Both devices were used as bolometer and detection of THz radiation at 100 GHz was obtained.

Problem solving and dissertation results:

The candidate faced many failures during the fabrication process as was presented extensively in chapter 4. He could manage all the problems and resolved them where he succeed in defining the right process (PMMA dose, exposition, etching,...) and obtained working devices.

Importance for practice or development of the discipline:

Developpment of new type of bolometers for terahertz technology is a challenging and hot topic. The last decades, extensive investigations were made to close the so called THz gap (0.1-10THz). The other topic is the 2D materials which are new generation of materials with excelent propoerties like high carriers mobility, thermal conductance, light-matter interaction, mastering the gap,...

Mastering the technology of fabrication using graphene (semi-metal 2d material) toward bolometric detection is very important for practical aplications within

Formal adjustment of the thesis and language level:

The overall level of english is good and the thesis is well organized in different chapters and sections. The candidate published his work in J. Phys. D: Appl. Phys. 58 (2025) 135103 with IF of **3.2** (Q1 in scopus) and used his skills for other papers ***Mater. Chem. Front.***, 2025,**9**, 866-883 (Q1) & Applied Nanoscience 8, 931–935 (2018). This work was also presented in the well peer reviewed conference IRRMW-THz 2023.

Questions and comments:

Despite the good level of the thesis, there is some comments that needs to be addressed. Here is a list of my comments:

- In general, the figures needs to be better commented along the thesis. Many figures were provided without description.

- Chapter4: the candidate should clarify at the begining of the chapter the design that he is looking to implement with the dimension and all informations (CAD design). He talks about small and big (Fig.4-9) structures but its not clear. Are they the cross bar with different dimensions?

- There is no information of the size of the dots fabricated as well as the contacts

- Figs 3.4 & 3.5: More details on the Area Throughtput and describe better this figure.

- p53: Two steps for metalization were used. In the first one, only Ti/Au were used and for the second one Ti/Cu/Au was used. In which parts of the device they were used. I guess that the last one was used for the contact made by optical lithogrphy for bonding purpose. More details are needed.

- p63, Fig. 5.2: highlight the different peaks of raman spectra (SiO2, G, D, 2D,...) as youdid in fig. 5.1

- p66 & p76: provide the emitted power from the THz source.

- p67, Fig.5.5: how the radiation was guided to the device? What is the purpose of the optical window at the side-load? Was the step motor used in the experiment?

- p71, 2nd paragraph: it stated that the SEM was performed before the bonding wire. This step didn't affect the device as electron beam is impinging on the surface and could damage the device?

- p73: it's said that at 2K, dR/dT is around 76Mohm/K. This is slope and should be measured in a range of temperatura but not at fixed temperature as stated. Please comment on this and how this value was exctracted from the measurements.

- p79, 1st paragraph: it's stated that the electron-phonon scattering is dominant, what is the time scale of this effect and if you performed any measurement to highlight the speed response of the device.

- p48: provide more information on the Si/SiO2 wafers: thikness of oxide, resistivity, roughness,...

- p56: Fig. 4.25 highlight the area of graphene flake to be more clear.

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Here are other tipos and minor comments:

- p17,2nd paragraph: indicate the reference for the following statement: *"Vacuum-suspended graphene, obtained through exfoliation, exhibits remarkably high carrier mobilities exceeding 200.000 cm²/Vs at room temperature."*

- p18, first line: provide reference for the statement:" *...exceptional thermal conductivity, which is around 10 times that of copper and 2 times that of diamond*"

- p24,1st paragraph: correct the sentence "*resulting in the discrete quantization of energy levels. The discrete energy levels in graphene are markedly*" to "*resulting in the discrete quantization of energy levels in graphene that are markedly*"

- p25, 2nd paragraph: provide the reference for the statement: "*responsivity. If the thermal energy is lower than the charging energy kBT < e2C, the current flowing through the dot is restricted by thermal activation across this energy barrier.*"

- p27: provide the reference for the statement "*Reported GQD-based devices demonstrate ....*"

- p36: change puttering to sputtering

- p51: change remnants to remanants

- p61: complete the [ref]

- p72, fig6.2: Add the indices a,b,c,d to the figure and put a label for the 2 or 8 GQDs for clarity.

- Ref 24 is not completed, refs 102, 104, 105: add the day of access to the webpage.

- p98: change Ecola to Ecole Central de Casablanca

Conclusion:

In conclusion, the candidate shows high skills in fabrication process of complcated structure and demostrated their use as bolometric detector for terahertz detection. The result obtained were published in good level peer reviewed journal, J. Phys. D and presented in different peer reviewed internal conference

In my opinion, the reviewed thesis fulfill all requirements posed on theses aimed for obtaining PhD degree. This thesis is ready to be defended orally, in front of respective committee.

In ...Salamanca....., date...15/07/2025.....

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Dr. Yahya Moubarak Meziani

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