NO<IA

Science Day PhD thesis presentation Krzysztof Gajewski 21.01.2025

Agenda

- 1. Who am I?
- 2. What did I do?
- 3. Summary



Who am I?

MN, Network Management

- Nokian since 05.2020 as a SW developer
- Since 11.2022 as a SW archtiect
- Involved in 3 projects



4



Wrocław University of Science and Technology

PL: Grafenowe przetworniki nanoelektromechaniczne EN: Graphene nanoelectromechanical systems



Supervisor prof. dr hab. inż. Teodor Gotszalk







This work was supported by the Foundation for Polish Science TEAM Programme "Highresolution force and mass metrology using MEMS/NEMS devices - FoMaMet" (Grant No. TEAM/2012-9/3), co-financed by the European Regional Development Fund resources within the framework of Operational Program Innovative Economy



This work was supported by the National Science Centre, Poland by the PRELUDIUM 9 programme "Investigations of the properties of graphene nanoelectromechanical systems with Raman spectroscopy" (grant the use of scanning probe microscopy and Nr 2015/17/N/ST7/03850)



Some of presented works were supported by the FP7 project STREP NanoHeat (Nr 318625) ..MultidomaiN plAtform for iNtegrated MOre-tHan-MoorE/Beyond CMOS systems charActerisation & diagnosTics." financed using the european funds.



Graphene

- Carbon atoms connected by in-plane strength sp² bonds
- Free 🕺 electrons
- Lattice constants $a_{AB} = 1.41$ Å, $a_{AA} = 2.45$ Å
- Electron velocity $\hat{\mathbb{R}}_{\text{F}} \sim 10^{6} \text{ ms}^{-1}$
- Electron mobility ~ 200 000 cm²V⁻¹s⁻¹
- Graphene ambipolarity in dependence of external electric field it can be "n" or "p" type
- Thermal conductivity 3080 5150
 W/mK





Graphene and GNEMS



Zande AMVD, et al., *Nano letters* 2010, 10 (12), 4869-73.



(a) 67 www. 1 2 3 4 5nm 3 4 5 nm ΔY 20 2 3 4 5 nm Xu P, et al., PRB 2012, 85(12): 121406(R).





M. Kumar, et al., *Nano Lett.*, 2015, 15 (4), 2562-2567







The main goal of the thesis

- Development of methods and techniques for graphene nanoelectromechanical systems investigation using microscopic and spectroscopic technologies
- The main focus was put on scanning probe microscopy and Raman spectroscopy



The main goal could be achieved by:

Project and GNEMS preparation

Scanning tunneling microscope preparation which will be able to measure GNEMS

Atomic force microscopy techniques adaptation for GNEMS

GNEMS investigations



The main goal could be achieved by:

Project and GNEMS preparation

Scanning tunneling microscope preparation which will be able to measure GNEMS

> Atomic force microscopy techniques adaptation for GNEMS

> > **GNEMS** investigations



Cooperation during PhD





Typical GNEMS sample processing

Substrate

Gajewski, K. R., et al., Proc. SPIE 8902, Electron Technology Conference 2013, 89020G (July 25, 2013).

- 300 nm SiO₂ / Si.
- Apply photoresist
- Photolitography
- Photoresist removal
- SiO₂ etching

Graphene growth



Goniszewski, S.,..., **K. Gajewski,** et al., IET Circuits, Devices & Systems, 2015, 9, (6), p. 420-427.

- Growth on copper foil
- Low pressure (110 mbar), high temperature (1035 °C). •
- Methane as a carbon source
- Hydrogen used to minimize oxidation

Graphene transfer



- Graphene covered by PMMA
- Graphene etching from the bottom side using nitric acid.
- Copper etching using ammonium persulfate.
- Graphene cleaning.
- Transfer.
- PMMA etching in dichloromethane
- Annealing (350 °C ,10 h, P≈ 2 mbar, hydrogen flow100 sccm



The main goal could be achieved by:

Project and GNEMS preparation

Scanning tunneling microscope preparation which will be able to measure GNEMS

> Atomic force microscopy techniques adaptation for GNEMS

> > **GNEMS** investigations



STM capable to investigate GNE



0.00 V

Movable tip
 Movable carrier

- Movable sample
- Sample movement in X/Y axis
- Constant current and constant height modes
- STM related modes introduced:
 - STP
 - z-U_{bias} spectroscopy

Gajewski, K., et al., Microel. Eng., 212 2019, 1-8 **Gajewski, K.**, et al., MST, 28(3) 2017, 034012



17



Introduced measurement



Gajewski, K., et al., Elektronika LIV, 06, 2013, 14 - 9.

Gajewski, K., et al., Applied Surface Science, vol 510, 30 April 2020, 145504



Optical observation and STM tip etching

Small sample requires capability of its optical observation







Gajewski, K., Microelectronic Engineering, 212 2019, 1-8





Tamulewicz, M., Kutrowska-Girzycka, J., **Gajewski, K.**, et al., Nanotechnology. 24 (30) 2019, s. <u>1-8</u>.



Optical camera, cutted tip



SEM



STM Microscope scaling



Scanlield (240.0 × 240.0) nm Resolution (128 × 128) pixels B Height 0.3 am Position: 8.327 nm Heights difference: 0.00 nm Positions difference: 0.000 nm Height 0.31 nm Position: 39.334 nm Heights difference: -0.34 nm Positions difference: 31.006 nm

6.08 nm/V, Moiré pattern on HOPG X/Y





Atomic scale 33 Å x 33 Å



Minimal detectable potential, CVD graphene/Al₂O₃



I.e. Keithley 2400, minimal error $> 300 \mu V!!!$



The main goal could be achieved by:

Project and GNEMS preparation

Scanning tunneling microscope preparation which will be able to measure GNEMS

Atomic force microscopy techniques adaptation for GNEMS

GNEMS investigations





KPFM of the elongated sample – grapgene/LDPE







KPFM of the elongated sample – grapgene/LDPE HSMG – grapgene/LDPE



CVD graphene exhibited additional details caused by the sample elongation, HSMG WF \sim 4.59 - 4.62 eV, CVD WF \sim 4.52 - 4.43 eV GF \sim 70 (HSMG) / 30 (CVD) Gajewski, K., et al., Diamond and Related Materials, 82 2018, 143-149

GF from publications 10 – 35 Amjadi et al., Advanced Functional, Materials, vol. 26, no. 11, pp. 1678–1698, 2016



The main goal could be achieved by:

Project and GNEMS preparation

Scanning tunneling microscope preparation which will be able to measure GNEMS

> Atomic force microscopy techniques adaptation for GNEMS

> > GNEMS investigations





Electromechanical properties investigations using AFM





$$F = \left[\frac{4\pi t^3}{3(1-\nu^2)R^2}E + \pi T\right]\delta + \frac{tE}{q^3R^2}\delta^3$$









Observation of graphene membrane deformation during STM measurement.





 $\Delta U \sim 1V \rightarrow \Delta z \sim 35 \text{ nm}$

Alternative for TEM?



www.jeol.co.jp/en/

products/detail/JEM-

https://

2100.html



H. Rasool, Nature Comm., vol. 4, 2013

TEM





Observation of the natural resonance of a graphene <u>membrane</u> – laser vibrometry

Membrany

EPFL

BLG



Cooperation with mgr inż. Piotr

Kunicki





Natural

vibration

208 Frequency (Hz)

(b)

- Reference

Measurements in vacuum





5e-4 Pa





EPFL BLG

How far it was / it from Nokia?

STRENGTH-FLAW RELATIONSHIP OF CORRODED PRISTINE SILICA STUDIED BY ATOMIC FORCE MICROSCOPY

QIAN ZHONG, DARYL INNISS, AND CHARLES R. KURKJIAN AT&T Bell Laboratories, Murray Hill, NJ 07974

APPLIED PHYSICS LETTERS

VOLUME 77, NUMBER 3

17 JULY 2000

ABSTRACT

Glass streng flaw relationshi difficulty in ider surface. In thi presented. Th spatially-resolve Excellent streng modeled as part to the corrosion have assumed th

Metal-insulator-semiconductor tunneling microscope: two-dimensional dopant profiling of semiconductors with conducting atomic-force microscopy

S. Richter^{a)} Bell Laboratories, Lucent Technologies, Murray Hill, New Jersey 07974 and Optoelectronics Center, Lucent Technologies, Breinigsville, Pennsylvania 18031

M. Geva

Optoelectronics Center, Lucent Technologies, Breinigsville, Pennsylvania

J. P. Garno and R. N. Kleiman

Bell Laboratories, Lucent Technologies, Murray Hill, New Jersey 07974

(Received 20 March 2000; accepted for publication 24 May 2000

A method for two-dimensional carrier profiling is presented, based atomic-force microscope (AFM) probe tip to a semiconductor si taken during the AFM scan on a cross-sectioned sample consisting results show a clear dependence of the current–voltage characteri and different behavior for *n*-and *p*-type InP. Modeling of the data of a quantitative tool for high-resolution two-dimensional dopant Institute of Physics. [S0003-6951(00)03329-5]

Scanning capacitance microscopy imaging of silicon ^{*nii*} metal-oxide-semiconductor field effect transistors*

R. N. Kleiman,^{a)} M. L. O'Malley, F. H. Baumann, J. P. Garno, and G. L. Timp Bell Laboratories, Lucent Technologies, Murray Hill, New Jersey 07974

(Received 17 January 2000; accepted 31 May 2000)

We have studied cross-sectioned *n*- and *p*-metal-oxide-semiconductor field effect transistors with gate lengths approaching 60 nm using a scanning capacitance microscope (SCM). In a homogeneous semiconductor, the SCM measures the depletion length, determining the dopant concentration. When imaging a real device there is an interaction between the probe tip and the built-in depletion of the *p*-*n* junction. With the help of a device simulator, we can understand the relation between the SCM images and the position of the *p*-*n* junction, making the SCM a quantitative tool for junction delineation and direct measurement of the electrical channel length. © 2000 American Vacuum Society. [S0734-211X(00)08604-2]

VO<IY



PhD thesis presented (just extract)

Wants more?

https://dbc.wroc.pl/dlibra/publication/141090/edition/72968



