
CNM 2017

5th Conference on Nano- and Micromechanics

WROCŁAW, POLAND
4-6th July 2014

Dear Colleague,

I am very pleased to welcome you to the 5th Conference on Nano- and Micromechanics (CNM) conference.

The CNM 2017 conference is an international meeting, devoted to micro and nanomechanics, which is held in Poland biennially in July. The conference brings together engineers and scientists from both academia and industry from across the world to discuss the recent progress and future developments in the nano- and micromechanics. Applications in electronics, nanotechnology, biotechnology, photonics environment and life sciences will also be discussed in a friendly and relaxed atmosphere.

The conference has evolved from Krajowa Konferencja Nano- i Mikromechaniki (KKNM). The CNM 2017 will be the 5th in a series, which was started in Krasieczyn in 2008 and was held in Krasieczyn in 2010, in Warsaw in 2012 and in Wrocław in 2014. In July 2017 CNM17 again will be taking place in Wrocław.

I would like to invite you to take part in our conference being fully convinced that your participation will make the program more attractive and the discussions more fruitful.

With best regards,

A handwritten signature in black ink, appearing to read 'J. G. A. A. A.', written in a cursive style.

Scientific Committee

Janusz Badur, Prof.
Tadeusz Burczyński, Prof.
Michał Ciałkowski, Prof.
Marek Dziubiński, Prof.
Paweł Dłużewski, Prof.
Teodor Gotszalk, Prof.
Piotr Grabiec, Prof.
Ling Hao , Dr
Robert Hołyst, Prof.
Tomasz Kowalewski, Prof.
Anna Kucaba-Piętal, Prof.
Henryk Petryk, Prof.
Maciej Pietrzyk, Prof.
Mieczysław Poniewski, Prof.
Ivo W. Rangelow, Prof.
Gwidon Szefer, Prof.
Danuta Szeliga, Prof.
Rafał Walczak, Prof.
Ehrenfried Zschech, Prof.

Organising Committee

Teodor Gotszalk, Prof.
Conference Chair

Anna Kucaba-Piętal, Prof.
Conference Co-Chair

Tomasz Kowalewski, Prof.
Conference Co-Chair

Jacek Radojewski, PhD
Magdalena Moczala, MSc
Karolina Orłowska, MSc
Krzysztof Gajewski, MSc
Michał Świątkowski, MSc

SESSION 1:

4th July 2017

"CARBON NANOTUBES"

- | | |
|-------------|--|
| 09:45-10:15 | [I-01] C. Roman <i>"Nanomechanical sensors based on individual carbon nanotubes"</i> |
| 10:15-10:45 | [O-01] P. Grabiec <i>"MEMS/NEMS challenges in IoT/Industry 4.0 era"</i> |
| 10:45-11:05 | [O-02] Burhanettin Erdem Alaca / Mustafa Yilmaz <i>"Finite Element Modeling for Twist-Bend Buckling of Si Nanowire Structures"</i> |

Nanomechanical sensors based on individual carbon nanotubes

C. ROMAN^{1,*}, L. JENNI¹,
L. KUMAR¹, C. HIEROLD¹

¹*ETH Zürich, Tannenstrasse 3, Zürich, 8092, Switzerland*

*croman@ethz.ch

Keywords: carbon nanotubes, sensors, mechanical resonators, ultra low power, resolution

Carbon Nanotubes have close to ideal mechanical properties, huge stiffness and strength and ultra low mass. All these properties result directly from the strong sp^2 C-C bonds and lightness of the carbon element. Expectedly, researchers have tried to exploit these features in many types of electro-mechanical transducers.

One of the first transducer types based on individual single-walled carbon nanotubes realized in our group was for sensing displacement and force [?],[2]. These transducers showed exceptional piezoresistive gauge factors of 2900 and yield strains above 1.5%. Such performance encouraged us to develop subsequently pressure sensors [3] using carbon nanotubes as miniaturized strain gauges. Although very stiff, nanotubes are still considerably smaller than the pressure sensor diaphragms, meaning that the overall mechanical properties are still determined by the latter. This has allowed us to miniaturize our pressure sensors further down to radii of about 20 μm while retaining gauge factors above 450 and achieving 15 mbar resolution [4],[5].

Most piezoresistive devices mentioned above were fabricated by growing the nanotube directly onto the targeted oxide substrate and subsequently defining the devices via e-beam lithography around AFM-localized nanotubes. However, the presence of the oxide and the exposure of the nanotubes to lithography resists produced undesirable side effects such as gate hysteresis and high excess noise. Another approach was developed in our group to integrate carbon nanotubes into mechanical structures to circumvent these issues. Therein, individual ultraclean nanotubes are grown between contacts, and subsequently metalized by evaporating palladium through self-aligned on-chip shadow masks. Such a process leaves the nanotubes pristine and indeed lead to hysteresis-free nanotube devices in ambient conditions [6].

Ultra-clean, suspended carbon nanotubes are ideally suited for making mechanical resonators. In [7] we report on the fabrication and characterization of such resonators. Capacitive and piezoresistive current modulation readout were compared showing the superiority of the latter method. Resonance at 79.5MHz and Q-factors of 91 have been measured at ambient temperature but $7 \times 10^{-4} Pa$ of pressure. Since the carbon nanotubes are embedded into microactuators, it was possible to tune the resonance frequency mechanically by uniaxial strain applied via actuators. Beam-to-string transition is evidenced in the measurements with a sensitivity of $9.4 \times 10^{10} Hz/\epsilon$ in the latter regime. Tuning of the frequency in the 10–60MHz was achieved together with Q increases upon straining.

The fabrication platform developed so far should allow us to answer some of the open questions still surrounding carbon nanotube resonators. In particular, understanding and ideally reducing energy dissipation, or equivalently improving Q factors is a crucial objective. With those challenge addressed it is possible to achieve mass sensors towards the single-proton resolution limit and ultra-sensitive strain gauges.

Acknowledgments

The authors would like to thank the Swiss National Science Foundation (Project No. 153292) for financially supporting this research. Additionally we acknowledge the co-workers and staff at the Binnig and Rohrer Nanotechnology Center and FIRST clean-rooms for their support.

References

- [1] C. Stampfer, A. Jungen, R. Lindermann, D. Obergfell, S. Roth, C. Hierold, Nano-Electromechanical Displacement Sensing Based on Single-Walled Carbon Nanotubes, *Nano Letters* 6 (2006), 1449–1453.
- [2] C. Stampfer, A. Jungen, C. Hierold, Fabrication of discrete nanoscaled force sensors based on single-walled carbon nanotubes, *IEEE Sensors Journal* 6 (2006), 613–617.
- [3] C. Stampfer, T. Helbling, D. Obergfell, B. Schöberle, M. K. Tripp, A. Jungen, S. Roth, V. M. Bright, C. Hierold, Fabrication of Single-Walled Carbon-Nanotube-Based Pressure Sensors, *Nano Letters* 6 (2006) 233-237.
- [4] T. Helbling, C. Roman, L. Durrer, C. Stampfer, C. Hierold, Gauge Factor Tuning, Long-Term Stability, and Miniaturization of Nanoelectromechanical Carbon-Nanotube Sensors, *IEEE Trans. on Electron Devices* 58 (2011) 4053–4060.
- [5] T. Helbling, S. Drittenbass, L. Durrer, C. Roman, C. Hierold, Gauge Factor Tuning, Long-Term Stability, and Miniaturization of Nanoelectromechanical Carbon-Nanotube Sensors, *IEEE MEMS Conference 2009*, doi:10.1109/MEMSYS.2009.4805447.
- [6] M. Muoth, T. Helbling, L. Durrer, S.-W. Lee, C. Roman, C. Hierold, Hysteresis-free operation of suspended carbon nanotube transistors, *Nature Nano.* 5 (2010) 589–592.
- [7] S.-W. Lee, S. Truax, L. Yu, C. Roman, C. Hierold, Carbon nanotube resonators with capacitive and piezoresistive current modulation readout, *Appl. Phys. Lett.* 103 (2013) 033117.

RF NEMS Based on Suspended Graphene Structures

M. YILMAZ¹, N. WOLLSCHLÄGER², Z. TAŞDEMİR¹,
A. KRANZMANN², Y. LEBLEBICI³, B. ERDEM ALACA^{1,4,*},

¹ *Department of Mechanical Engineering, Koç University, Rumelifeneri Yolu, Sarıyer
 34450 Istanbul, Turkey*

² *Bundesanstalt für Materialforschung und-prüfung (BAM), Unter den Eichen 87,
 D-12205 Berlin, Germany*

³ *Microelectronic Systems Laboratory, EPFL, Bldg ELD, Station 11, CH-1015
 Lausanne, Switzerland*

⁴ *Surface Science and Technology Center (KUYTAM), Koç University, Rumelifeneri
 Yolu, Sarıyer 34450 Istanbul*

*ealaca@ku.edu.tr

Keywords: Si Nanowires, Buckling, FEA

Recently, new and reliable fabrication techniques for nanowires are developed and as a result, nanowires find applications in a variety of fields [1]. Both geometrical aspects and operational requirements of such nanowire architecture can be drastically different from each other. In some instances, successful operation depends on the nanowire's nonlinear behavior which cannot be modeled accurately.

Dry etching is used successfully in the fabrication of top-down Si nanowires [2]. Recent examples of such Si nanowires have been reported [3]-[5]. In one of these example nanowires are fabricated with a trapezoidal cross-section with a 72-nm top surface width increasing to 112 nm over a thickness of 168 nm (Fig. 1), that develops twist-bend buckling upon in-situ midpoint bending test. In this work, an FEA (Finite Element Analysis) model for the simulation of the twist-bend buckling behavior is reported, which is valid for small and large displacements. Utilizing the results of an in-situ mid-point bending test [6], we modeled the behavior of a Si nanowire with the above mentioned cross-sectional features. Due to the specific geometry, twistbend buckling is encountered during the test. The average force-displacement data are given in Fig. 2 (Plot III). First, we used a mathematical model that is suitable for the geometric nonlinearity associated with large displacements (Fig. 2, Plot I). The model was utilized for validating FEA simulations. In the next step, we developed an FEA model for post-buckling interstate integrations for predicting the new iteration parameters such as displacement, required forces and new deformed shapes (Fig. 2. II). Fig. 3 provides a comparison of the involved stress distribution in the absence (Fig. 3.a) and presence of buckling (Fig. 3.b).

The developed FEA model is currently generating acceptable results compared to the bending experiments and could help us understand the mechanical behavior at the nanoscale, such as fracture, in a better way.

Acknowledgments

This work was supported by Tubitak under Grant No. 112E058 and Koc University Rotary Club Fundamental Research Seed Fund Group. The contribution of BAM has received funding from the EMPIR programme StrengthABLE co-financed by the Participating States and from the European Union's Horizon 2020 research and innovation programme.

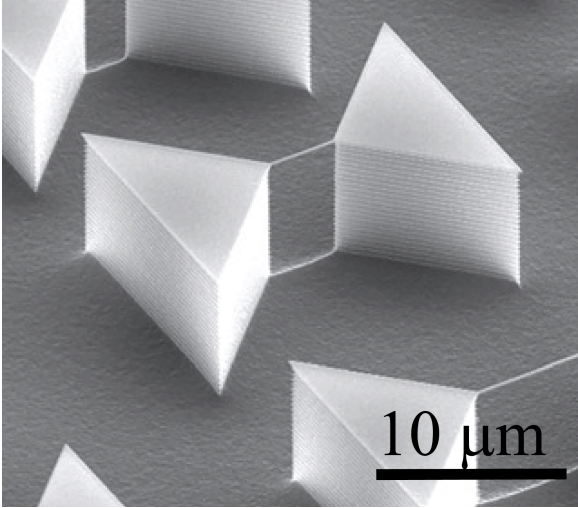


Fig. 1: Fabricated Si NWs that is used in this modelling buckling model (dashed line represents extrapolation study).

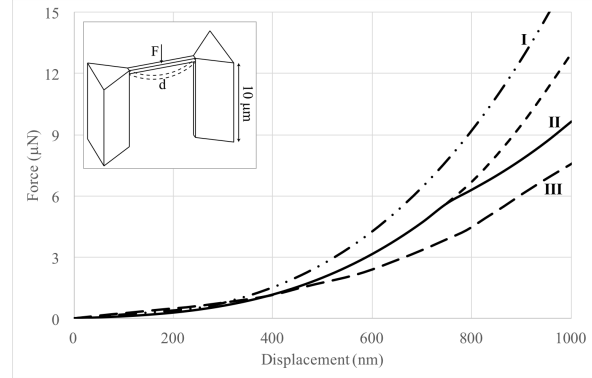


Fig. 2: FEA model results, inset depicts the modeling environment. (I) Large displacement model, (II) Interstate buckling model (dashed line represents extrapolation indicating the absence of buckling), (III) Experimental average results.

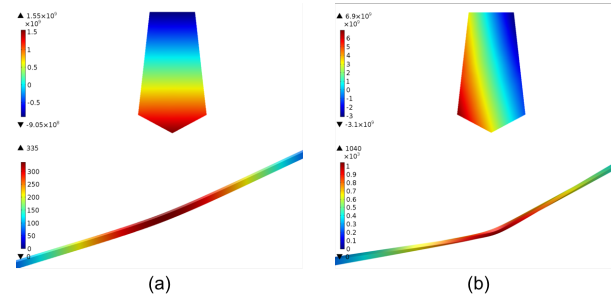


Fig. 3: Stress Distribution in midplane for 1 microNewton applied force (a) Large displacement model (b) Interstate Buckling Mode

References

- [1] N. P. Dasgupta, J. Sun, C. Liu, S. Brittman, S. C. Andrews, J. Lim, H. Gao, R. Yan and P. Yang, 25th Anniversary Article: Semiconductor Nanowires – Synthesis, Characterization, and Applications, *Advanced Materials*, vol. 26, pp. 2137-2184, 2016.
- [2] I. W. Rangelow, Critical Tasks in High Aspect Ratio Silicon Dry Etching for Microelectromechanical Systems, *Journal of Vacuum Science and Technology A*, vol. 21, 1550, 2003.
- [3] Z. Tasdemir, N. Wollschläger, W. Österle, Y. Leblebici and B. E. Alaca, A Deep Etching Mechanism for TrenchBridging Silicon Nanowires, vol. 27, page. 095303, 2016.
- [4] M. Yilmaz, Y. Kilinc, G. Nadar, Z. Tasdemir, N. Wollschläger, W. Österle, Y. Leblebici and B. E. Alaca, TopDown Technique for Scaling Nano in Silicon MEMS, *Journal of Vacuum Science & Technology B*, vol. 35, 022001, 2017.
- [5] M. N. Esfahani, Y. Leblebici and B. E. Alaca, A Monolithic Approach to Downscaling Silicon Piezoresistive Sensors, *Journal of Microelectromechanical Systems*, 2017.
- [6] N. Wollschläger, Z. Tasdemir, I. Häusler, Y. Leblebici, W. Österle, and B. E. Alaca, Determi-

nation of the Elastic Behavior of Silicon Nanowires within a Scanning Electron Microscope, Journal of Nanomaterials, vol. 2016.

SESSION 2:
4th July 2017

"THERMAL"

- | | |
|-------------|--|
| 11:25-11:55 | [I-02] A. Kulik <i>"AFM-IR: new tool for bio- and polymer research (Infrared Spectroscopy with 10 nm spatial resolution)"</i> |
| 11:55-12:15 | [O-03] B. Kaestner <i>"Application of nano-FTIR spectroscopy and infrared near-field imaging for contactless thermography"</i> |
| 12:15-12:25 | [E-01] P. Kasprzycki <i>"AFM and SEEC equipment for nanoscale imaging and measurements"</i> |
| 12:25-12:45 | [O-04] W. Chomicki <i>"Devices to detect oscillations induced by living organisms"</i> |
| 12:45-13:05 | [O-05] M. Rudek <i>"Using piezoresistive NanoHeat cantilevers for quantitative thermal investigations of nanostructures"</i> |

AFM-IR: new tool for bio- and polymer research (Infrared Spectroscopy with 10 nm spatial resolution)

A. KULIK^{1,*}, M. UNGER², E. DILLON², J. MATHURIN³, A. DENISET-BESSEAU³, A. DAZZI³

¹*EPFL, LPMV, Lausanne, 1015, Switzerland*

²*Anasys Instruments, 325 Chapala St., Santa Barbara, 93101, USA*

³*Universite Paris Sud, Av. Jean Perrin - Bat. 350, Paris, 91400, France*

*andrzej.kulik@epfl.ch

Keywords: AFM-IR, nanoIR, IR Spectroscopy, nanoscale

Infrared (IR) spectroscopy commonly used for chemical identification has no, or poor spatial resolution which is diffraction limited (typ. 30 μm). IR sources have limited power, so measuring time is long.

Progress in design of tunable IR sources, like OPO (Optical Parametric Oscillator), led to development of AFM-IR method. Short laser pulses of the wavelength within sample's absorption bands induce local thermal expansion. Vertical component of thermal expansion is measured using AFM cantilever in contact with sample surface. Short deformations are exciting resonant frequencies of the cantilever and amplitude of each of the resonant peaks is proportional to local IR absorption. Limit of the sensitivity is determined by the thickness of the specimen and has to be bigger than 100 nm. (Fig. ?? and 2)

Quantum Cascade Lasers (OPO) can deliver fast repetition of output pulses. Therefore, one can tune output pulse frequency to resonant frequency of the cantilever. High mechanical quality factor, enhancing the signal, allow one to measure thermal expansion of the samples having thickness of only 1 nm.

Contact AFM is not always the best choice for soft samples. Recently tapping mode operation was implemented. Tapping cantilever oscillates at the first flexural mode. One tunes QCL laser frequency to the difference between first and second mode of the cantilever. Nonlinear tip-sample interactions act as mechanical mixer, exciting second mode of the cantilever with amplitude proportional to IR absorption. This tapping mode AFM-IR is the best suited to study thin layers of polymers and biopolymers. Spatial resolution of 10 nm allows one to distinguish small features on IR absorption image [2].

As well IR absorption spectra can be obtained at any point of the sample. The fastest experiment is delivering IR spectrum every 100ms, so full information about the sample can be obtained at every pixel within reasonable time.

References

- [1] W. Gruszecki, AJ Kulik, E Janik, J Bednarska, R Luchowski, W Grudzinski, G Dietler, Nanoscale resolution in infrared imaging of protein-containing lipid membranes, *Nanoscale*, 7, 35, (2015)
- [2] Alexandre Dazzi, Craig B. Prater, AFM-IR: Technology and Applications in Nanoscale Infrared Spectroscopy and Chemical Imaging, *Chem. Rev.*, 2017, 117, 5146-5173

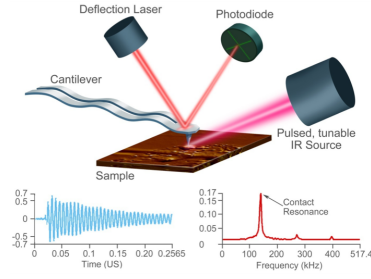
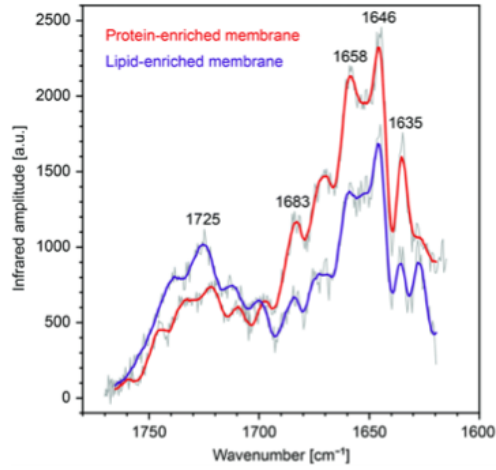
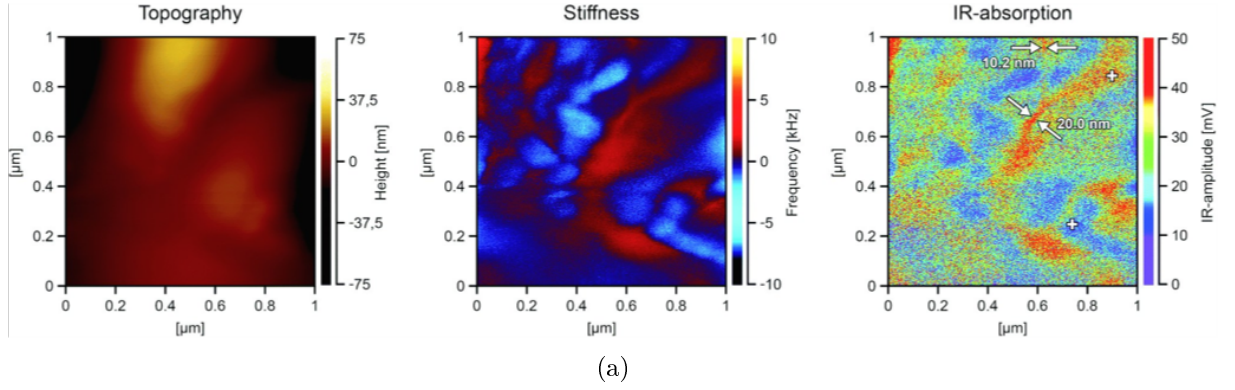


Fig. 1: Setup of nanoIR system



(b)

Fig. 2: High resolution images of artificial membrane (bilipids + LHCI): Topography, Stiffness and IR absorption. Below, IR spectra measured in two different locations. (From [1])

Application of nano-FTIR spectroscopy and infrared near-field imaging for contactless thermography

B. KÄSTNER¹, P. HERMANN¹, A. HOEHL¹,
H. W. SCHUAMCHER², X. K. HU², P. KRZYTECZKO¹, G.
ULRICH¹, G. ULM¹,

¹*Physikalisch-Technische Bundesanstalt (PTB), 10587 Berlin, Germany*

²*Physikalisch-Technische Bundesanstalt (PTB), 38116 Braunschweig, Germany*

Near-field optical techniques such as scanning near-field infrared microscopy and nano-FTIR spectroscopy enable imaging and spectroscopic characterization of samples with both high sensitivity and high spatial resolution. In this work we realized nano-FTIR spectroscopy by elastic scattering of infrared light from a probe tip of an atomic force microscope operated in tapping mode, with synchrotron radiation provided by the Metrology Light Source (MLS) [1]. We apply nano-FTIR spectroscopy and near-field imaging on samples with a well defined temperature profile and material having a temperature dependent phonon resonance. Mapping the spectral position of the phonon resonance allows in principle to determine the temperature distribution on the nanoscale. This method may be regarded as material specific contactless thermography that does not require the establishment of thermal equilibrium between probe tip and sample. We confirm the consistency of the determined temperature distribution with FEM simulations.

References

- [1] P. Hermann et al., Characterization of semiconductor materials using synchrotron radiation-based near-field infrared microscopy and nano-FTIR spectroscopy, *Optics Express* 22, 17948 (2014).

AFM and SEEC equipment for nanoscale imaging and measurements

P. KASPRZYCKI¹

¹*IRtech, Ul. Wyżynna 8H, 30-617 Kraków*
*piotr@irtech.pl

Presentation of new ezAFM+ microscope designed by NanoMagnetics Instruments and its accessories for different applications and N-Lab station using SEEC technique for nanoscale imaging and its applications for material verification.

Devices to detect oscillations induced by living organisms

W. CHOMICKI¹, P. STUPAR¹,
R. FOSCHIA¹, S. KASAS², G. DIETLER³

¹*Laboratoire de Physique de la Matière Vivante (LPMV), Ecole Polytechnique Fédérale de Lausanne (EPFL), Lausanne, CH-1015, Switzerland*

Recently our team demonstrated that living organisms attached to an Atomic Force Microscope cantilevers induce oscillations that reflect their viability and their metabolism [1],[2],[3],[4]. Our team demonstrated a possibility that homemade device can fulfill all the requirements to capture oscillations induced by living organisms. The device operates with noise level similar or better than commercially available AFM, accommodates traditional AFM cantilevers and is extremely simple to operate. In addition the price of the instrument is only a fraction of a traditional AFM. We are seeking to implement similar devices in hospitals and research laboratories for rapid drug screening. There is a need for a device that can be operated remotely in closed spaces, hence the development of a motorized version of the device is desirable. This hardware can be easily upgraded with software for automated alignment or drift correction. In this presentation recent progress will be shown.

Acknowledgments

This work has been supported by Gebert R  f Foundation Project GRS-024/14 and Swiss National Science Foundation Grant Nr. 200021_144321.

References

- [1] G. Longo et al., Nature Nanotechnology 8, 522-526 (2013), doi: 10.1038/nnano.2013.120
- [2] S. Kasas et al., PNAS 112, 378-381 (2015); doi: 10.1073/pnas.1415348112
- [3] P. Stupar et al., Mechanical Sciences 8, 23-28 (2017), doi: 10.5194/ms-8-23-2017
- [4] P. Stupar et al. Clinical Microbiology and Infection 23 (6), 400-405 (2017), doi: 10.1016/j.cmi.2016.12.028

Using piezoresistive NanoHeat cantilevers for quantitative thermal investigations of nanostructures

M. RUDEK^{1,*}, P. KUNICKI¹,
W. MAJSTRZYK¹, T. GOTSZALK¹, P. JANUS², A. SIERAKOWSKI², D.
SZMIGIEL²

¹*Wrocław University of Science and Technology / Faculty of Microsystem Electronics
and Photonics, 50-372 Wrocław, Poland*

²*Institute of Electron Technology, Al. Lotników 32/46, 02-668 Warsaw, Poland*

*maciej.rudek@pwr.edu.pl

Keywords: AFM, SThM, scanning thermal microscopy, thermal nanoprobng, thermal imaging

In 2017 the new record was beaten by IBM which creates a 5nm transistor on new type [1]. In this case the nanoelectronics is one of the fastest expand branch in the modern science. In near future new materials and technology will be used to manufacture fast, small and cheapest integrated circuits (IC). This trend is strong connected to the assumptions of the Moore's Law. Unfortunately with down-scaling of the electronic elements then new problems are showed by pushing the length of the gates in transistors to the physical limits [2]. The main problem is the heat generated in passive and active elements of nano devices that like: resistors, connections patch, transistors, etc. Wherefore there must be made new methodology of investigation such as: mechanical, thermal and/or electrical properties of the fabricated structures and new materials used.

In this paper we present the results of investigation active and passive samples using the scanning thermal microscopy (SThM) which stems directly from AFM. Report technique, permissive high resolution topography and thermal investigations, utilizes novel piezoresistive SThM probes with piezoresistive detector of deflection. Which are manufactured in standard technology: MEMS, CMOS and Focus Ion Beam in postprocessing fabrication steps[3][4]. Moreover the probes allow to obtain high resolution thermal structure measurements. It is possible using novel conductive platinum coated tip with 4-point probe connection wiring for thermal measurement and used in a standard setup in home-made Atomic Force Microscope detection system. Unique construction of SThM cantilever enables measure quantitatively temperature in each point of structure. Further new mode, called Load Force Modulation Method (LoFM), initially was developed for thermal measurements using cantilevers with the piezoresistive deflection sensor to control precisely contact force on each point of scan and measure heat dissipation in and out of the contact with force below 50 nN and resolution 2 nN [5]. Presented methodology and techniques allow to obtain quantitative temperature and thermal conductivity of investigated samples in nanoscale.

Acknowledgments

This work was partially supported by the statutory research grant of the Wrocław University of Technology grant no.: 0401/0234/16.

References

- [1] <https://www-03.ibm.com/press/us/en/pressrelease/52531.wss>

- [2] B. Y. M. M. Waldrop, O. F. M. S. Law, and M. Interesting, More Than Will Soon Abandon Its Pursuit, *Nature*, vol. 530, p. 145, 2016.
- [3] P. Janus, P. Grabiec, A. Sierakowski, T. Gotszalk, M. Rudek, D. Kopiec, W. Majstrzyk, G. Boetsch, B. Koehler, Design, technology, and application of integrated piezoresistive Scanning Thermal Microscopy (SThM) microcantilever, *Scanning Microscopies 2014, Proc. of SPIE Vol. 9236*, 92360R
- [4] P. Janus, D. Szmigiel, M. Weisheit, G. Wielgoszewski, Y. Ritz, P. Grabiec, M. Hecker, T. Gotszalk, P. Sulecki, E. Zschech, Novel SThM nanoprobe for thermal properties investigation of micro- and nanoelectronic devices, *Microelectronic Engineering* 87, 2010, ss. 13701374
- [5] Janus, P., Sierakowski, A., Grabiec, P., Rudek, M., Majstrzyk, W., Gotszalk, T. Micromachined active test structure for scanning thermal microscopy probes characterization, (2017) *Microelectronic Engineering*, 174, pp. 70-73.

SESSION 3:
4th July 2017

"CHEMISTRY & PHYSICS"

- | | |
|-------------|--|
| 14:20-14:50 | [I-03] K. Sozański <i>"How to Break a Molecular Motor: A Study of Kinesin Motion in Crowded Environment"</i> |
| 14:50-15:10 | [O-06] S. Pawłowska <i>"Lateral Migration of Highly Deformable Nanofilaments Conveyed by Oscillatory Flow"</i> |
| 15:10-15:30 | [O-07] R. Pytliński <i>"Complex Stress States in Electromagnetic Field"</i> |
| 15:30-15:40 | [E-03] Ł. Zarodkiewicz <i>"A Closer Look at nanoTechnology"</i> |
| 15:40-16:00 | [O-08] P. Sadowski <i>"Micromechanical modelling of elastoplastic composites: efficient and robust finite-element implementation of Mori-Tanaka model"</i> |
| 16:00-16:20 | [O-09] P. Ziółkowski <i>"On Navier slip and Reynolds transpiration numbers"</i> |
| 18:00-21:00 | WELCOME PARTY AT THE NATIONAL MUSEUM -
plac Powstańców Warszawy 5 |

RF NEMS Based on Suspended Graphene Structures

K. SOZAŃSKI^{*1}, F. RUHNOW², A. WIŚNIEWSKA¹,
M. TABAKA¹, S. DIEZ², R. HOŁYST¹

¹*Institute of Physical Chemistry, Polish Academy of Sciences, Kasprzaka 44/52, 01-224
Warsaw, Poland*

²*B-CUBE Center, Technische Universität Dresden, Arnoldstrasse 18, 01307 Dresden,
Germany*

*ksozanski@ichf.edu.pl

Keywords: molecular motors, nanomechanics, viscosity, diffusion, kinesin

Kinesin is a motor protein, responsible for cargo transport in eukaryotic cells. It is a widely studied model of a biological molecular motor. Each kinesin molecule comprises two motor domains connected by a short, flexible linker. It moves along microtubules – which constitute a cellular scaffolding – in a step-by-step manner. It has been shown that a single kinesin motor can act against forces of up to 7 pN. However, the mechanism of force generation by kinesin is still debated.

In a series of in vitro experiments, based on single molecule tracking, we found that even a moderate increase in the viscosity of the environment – to about 5 mPa·s, i.e. around 5 times the viscosity of water – can cause an utter stall of kinesin motors. This is about 5 orders of magnitude less than expected from Stokes’ law, which predicts that a sphere of 6 nm in diameter (approximately the size of a kinesin motor unit) moving at a velocity of 100 nm/s would experience a stalling drag force in a medium of viscosity as high as 700 Pa·s. We carefully analysed different physical factors that could cause this effect, including kinesin aggregation, influence of disjoining pressure and depletion interactions, as well as limited ATP availability. We point to the viscosity increase as the key factor determining kinesin’s functionality. We quantitatively estimated the increase in activation energy for diffusion of the motor domain, which – combined with the barrier for stretching the protein linker – disrupts the chemomechanical step cycle.

The motor stall has not been observed in previous studies. This was due to the fact that those experiments relied on crowders (i.e. viscosity enhancing agents) of high molecular weight and relatively large dimensions. According to the paradigm of length-scale dependence of viscosity, such crowders have practically no influence on the local environment of the motor domain: when the moving object is similar in size or smaller than the crowders, it experiences an effective viscosity much lower than the macroscopic, bulk viscosity of the liquid. Our study is the first one to consider viscosity scaling when analysing kinesin motion in viscous media. Interestingly, the determined stall limit is lower than the effective viscosity usually observed in eukaryotic cell cytoplasm only by a factor of 1.5-3. This opens an intriguing perspective of how tuning the viscosity may constitute a means of global regulation of transport in cells.

The results presented hereby are a strong argument supporting the model of kinesin stepping where motion of the motor domain is realized by diffusion, while ATP-ase activity only provides synchronization and processivity. This implies that even such model system of active transport at the molecular level is in fact based on a passive, spontaneous process of diffusion. An even broader conclusion concerns the key importance of

the viscosity scaling paradigm in nanomechanics – especially in biophysical studies, which often deal with complex environments featuring multiple length-scales.

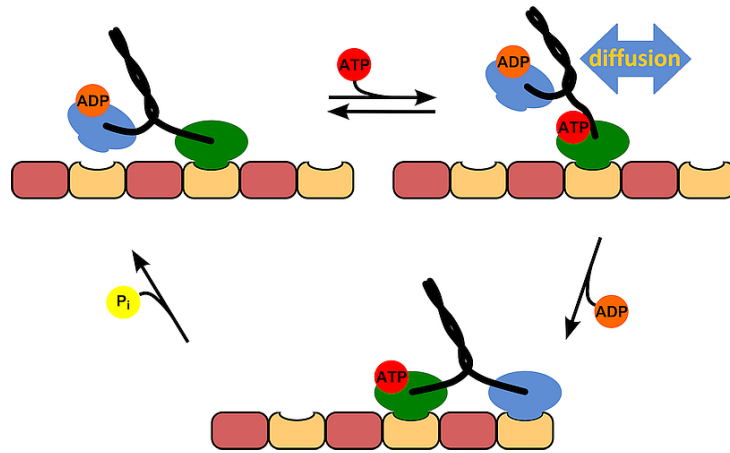


Fig. 1: Simplified scheme of the stepping cycle of a kinesin molecule moving along a microtubule. The key part of the cycle – translocation of the free motor domain to the next binding site – is realized by diffusion. Even a moderate increase in the viscosity of the environment can slow this process down to a point where a backwards reaction becomes favourable and the stepping cycle is disrupted.

Acknowledgments

The authors acknowledge financial support from the National Science Center (Maestro grant 2011/02/A/ST3/00143), the European Research Council (Starting grant 242933, NanoTrans), the European Social Funds (grant 100111059, MindNano and grant 100107464, ChemIT), and the German Research Foundation (Cluster of Excellence Center for Advancing Electronics Dresden and the Heisenberg Program).

References

- [1] K. Sozanski, F. Ruhnaw, A. Wisniewska, M. Tabaka, S. Diez, R. Holyst, *Phys. Rev. Lett.* 115, 218102 (2015).
- [2] S. M. Block, *Biophys. J.* 92, 2986 (2007).
- [3] T. Kalwarczyk, K. Sozanski, A. Ochab-Marcinek, J. Szymanski, M. Tabaka, S. Hou, R. Holyst, *Adv. Colloid Interfac. Sci.* 223, 55 (2015).
- [4] M. Nishiyama, H. Higuchi, T. Yanagida, *Nat. Cell Biol.* 4, 790 (2002).
- [5] T. Kalwarczyk, N. Ziebach, A. Bielejewska, E. Zaboklicka, K. Koyunov, J. Szymanski, A. Wilk, A. Patkowski, J. Gapinski, H.-J. Butt, R. Holyst, *Nano Lett.* 11, 2157 (2011).

Lateral Migration of Highly Deformable Nanofilaments Conveyed by Oscillatory Flow

S. PAWŁOWSKA^{1,*}, F. PIERINI¹, P. NAKIELSKI¹,
I. PIECHOCKA¹, K. ZEMBRZYCKI¹, T. A. KOWALEWSKI¹

¹*IPPT PAN, Polish Academy of Sciences, Pawinskiego 5B, 02-106 Warszawa*

*spaw@ippt.pan.pl

Keywords: thermal fluctuations, lateral migration, flexible filaments

Recently, we demonstrated possibility to produce hydrogel in a form of nanofilaments, the supreme geometry for conducting targeted material delivery to regenerate aligned tissues or perform DNA transport. Hence, in the following we analyze transport properties of such objects conveyed by oscillating flow simulating typical extracellular environment. Due to nanometric size of our nanofilaments both hydrodynamic interactions and Brownian fluctuations have to be considered. The problem has analogy to exhaustive studies of DNA flow dynamics, though the coarse filaments material allows to limit problem description to purely hydrodynamic interactions, neglecting complex molecular and electrostatic interactions.

FITC-fluorescently labeled hydrogel nanofilaments were prepared by co-axial electrospinning of two immiscible polymers (PLCL polymer and NIPAAm/BIS-AAm hydrogel solution), with the former one being used to mold the hydrogel in the core-shell structure [1]. After removal of their shells, hydrogel nanofilaments suspended in water - DMF (4:1) solution were introduced into the PDMS microchannel using a syringe micro-pump. The microchannel was equipped with two additional pre-chambers used to collect nanofilaments and to introduce the selected one into the observation area. Care was taken to analyze nanofilaments solely remaining in the plane of observations. In-house designed squeeze-tube micro-pump was used to produce sinusoidal oscillating flow within the channel. The velocity amplitude V_{max} varied from $100 \mu m/s$ to $900 \mu m/s$, flow oscillations frequency was set around 0.1 Hz. Nanofilaments were imaged using epifluorescence microscope (Leica AM TIRF MC) equipped with 20x/0.40 NA air objective and a mercury lamp light source. The flow-induced migration of nanofilaments was recorded using a high-gain EM-CCD camera (C9100-2, Hamamatsu) with typical frame rate of 15 Hz. The mechanical properties of our nanofilaments such as persistence length (L_p) and bending stiffness were evaluated from their Brownian shape deformations. Selected for the experiment nanofilaments were characterized by flexural modulus of 2 kPa, typical radius R of about 50 nm, and contour length L of several micrometers.

Performed experimental investigations demonstrated presence of lateral migration of nanofilaments, and their complex bending dynamics, being characteristic for long biomolecules. Experimental data are compared with hydrodynamic worm-like beads model of fibers conveyed by shear flow, confirming predicted fiber tumbling and lateral migration. Our electrospun nanofilaments offer a unique capability to mimic dynamics of long flexible object conveyed by a hydrodynamic flow. At the same time, they can be successfully used to validate essential assumptions of computational models used to describe dynamics of complex suspensions.

Acknowledgments

NCN grant 2015/17/N/ST8/02012

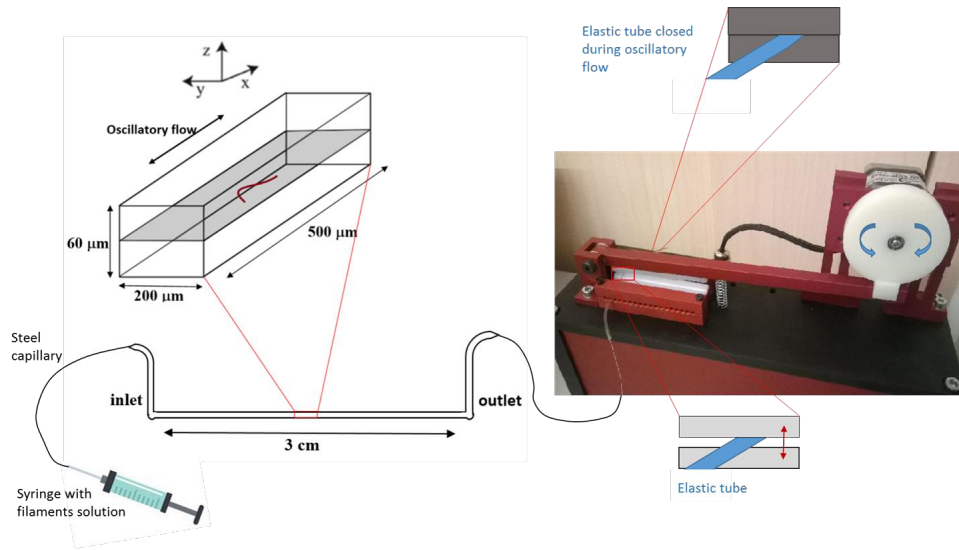


Fig. 1: Schematic of 3 mm long rectangular microchannel with nanofilament conveyed by oscillatory flow; indicated section observed under the microscope.

References

- [1] P. Nakielski, S. Pawlowska, F. Pierini, V. Liwinska, P. Hejduk, K. Zembrzycki, E. Zabost, and T.A. Kowalewski, Hydrogel nanofilaments via core-shell electrospinning, PLOS ONE 10(6):e0129816 (2015).

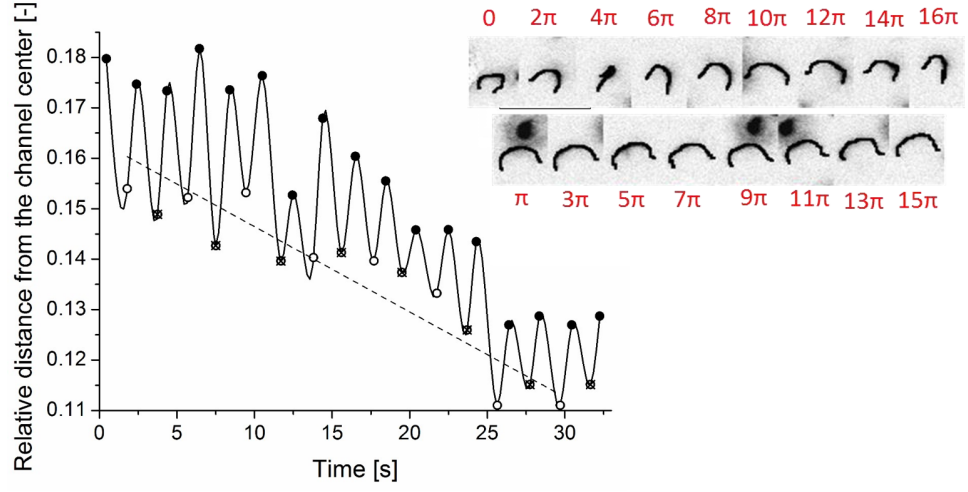


Fig. 2: Lateral migration of nanofilament into the microchannel center; insert – snapshots of filament buckling. $R = 53$ nm; $L = 41$ μ m; $Lp = 2.89$ μ m; $Re = 0.06$

MSA System – A Closer Look at nanoTechnology

Ł. ZARODKIEWICZ¹

¹*MSA System, ul. Jana Cybisa 4/35, 02-784 Warszawa*

**lukasz.zarodkiewicz@msasystem.pl*

Presentation of products delivered by the MSA System.

Micromechanical modelling of elasto-plastic composites: efficient and robust finite-element implementation of Mori–Tanaka model

P. SADOWSKI^{1,*}, K. KOWALCZYK-GAJEWSKA^{1,*}, S. STUPKIEWICZ^{1,*}

¹*Institute of Fundamental Technological Research, Pawińskiego 5B, Warsaw, 02-106, Poland*

*psad@ippt.pan.pl, kkowalcz@ippt.pan.pl, sstupkie@ippt.pan.pl

Keywords: Mean-field homogenization, Mori–Tanaka method, Composite materials, Finite element method

The Mori–Tanaka (MT) model [1] belongs to the wide class of mean-field homogenization methods that have been developed for predicting overall properties of heterogeneous materials. While more elaborate multiscale approaches, such as the FE² method [2], are readily available, the mean-field models are still attractive in view of their simplicity, good predictive capabilities and low computational cost [3].

The MT model is a mean-field model originally dedicated to the estimation of effective properties of linearly elastic two-phase composites. It originates from Eshelby’s solution to the problem of an ellipsoidal inclusion embedded into an infinite linearly elastic matrix [4]. The MT scheme, originally formulated for elastic composites, can be adapted to elasto-plastic composites by applying incremental linearization, as proposed by Hill [5]. In order to use the incremental MT method in practice, e.g., as a two-phase material model in the framework of the finite element method, the rate form of: constitutive equations of phases, so-called interaction equation and averaging rule, must be integrated over time. This leads to an incremental finite-step formulation and several practical realisations, dating back to [6], [7], are known. However, computational efficiency, consistent linearization, and related issues have not attracted sufficient attention yet. The goal of this work is to develop an efficient and robust incremental MT scheme, suitable for large-scale finite-element computations.

Consistent linearization of the macroscopic stress predicted by the MT scheme is considered a necessary condition to achieve the goal of this work. The automatic differentiation (AD) technique [8], [9] has been employed for that purpose. The corresponding compact AD-based formulation of the incremental MT scheme has been introduced. The AD-based formulation constitutes the basis for finite-element implementation and for automation of the related tasks. The incremental MT scheme involves a nested Newton-type algorithm with inner iterations corresponding to the equations of incremental plasticity formulated for the individual phases and with outer iterations corresponding to the micro-macro transition scheme of the MT method. Considering additionally the global equilibrium iterations, the complete computational scheme can be classified as a doubly-nested iteration-subiteration scheme. Exact linearization, which is necessary for achieving quadratic convergence rate, has been performed at each level so that the Newton method can be used to efficiently solve the nonlinear equations at each level.

However, consistent linearization was not sufficient to obtain a fully robust computational scheme. This is because convergence problems were encountered in some situations, especially for higher content of inclusions and in large-scale boundary value problems. It

has been found that the problems were caused by discontinuities in the incremental finite-step response [10]. The discontinuities may occur at the instant of the elastic-to-plastic transition in the matrix phase and result from the related abrupt change of the reference stiffness for which the MT interaction equation is formulated. As a remedy, the incremental MT scheme has been enhanced with a *substepping strategy*.

A highly robust and efficient computational scheme has been finally obtained, as illustrated in Fig. 1. It presents results of a plate with a hole made of a metal-matrix composite stretched in the longitudinal direction. The material is composed of spherical ceramic inclusions uniformly dispersed in an aluminum-alloy matrix. The inclusions are assumed elastic, and the matrix is elasto-plastic, governed by J2-plasticity with isotropic hardening. The finite-element mesh comprises 368,640 trilinear hexahedral elements which corresponds to approximately 1.2 million displacement unknowns. It can be seen that consistent linearization of the incremental Mori–Tanaka scheme was not enough to achieve desired robustness of the computational scheme, Fig. 1(a). Upon employing the substepping strategy, the developed computational scheme [11] can serve as an extremely efficient micromechanical model in large-scale FE problems dedicated to elasto-plastic composite materials, Fig. 1(b).

Acknowledgments

This work has been partially supported by the National Science Centre (NCN) through the Grant No. 2013/09/B/ST8/03320 and by the Centre of Excellence and Innovation of Composite Materials (CDIMK) at the Institute of Fundamental Technological Research (IPPT).

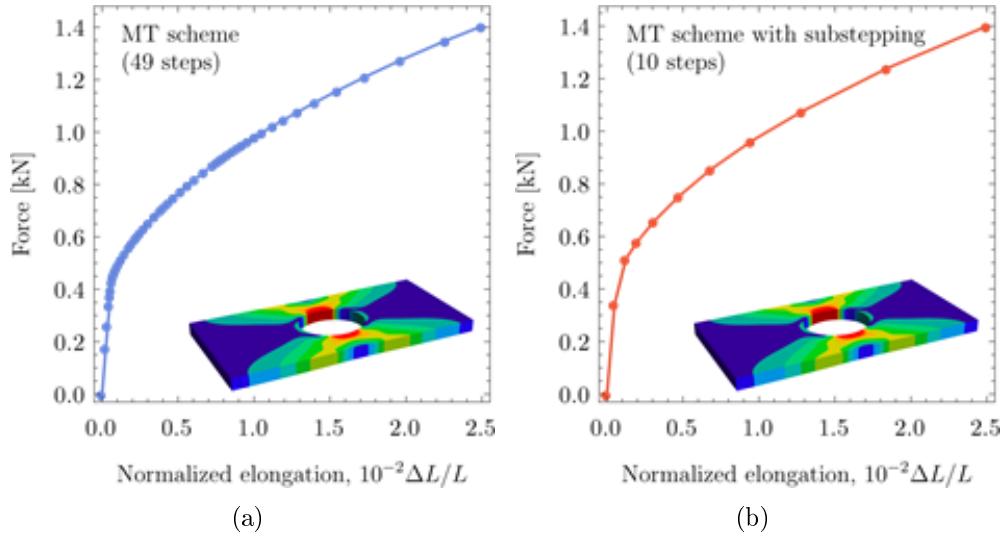


Fig. 1: The impact of response discontinuities on the convergence behaviour and robustness of the incremental MT scheme employed in finite-element computations for: (a) basic MT scheme exhibiting severe convergence problems that are caused by the discontinuities and (b) MT scheme enhanced with the substepping strategy [10]

References

- [1] T. Mori, K. Tanaka, Average stress in matrix and average elastic energy of materials with misfitting inclusions, *Acta Metall.* 21 (1973) 571–574.
- [2] F. Feyel, J. L. Chaboche, FE2 multiscale approach for modelling the elastoviscoplastic behaviour of long fibre SiC/Ti composite materials, *Comput. Methods. Appl. Mech. Eng.* 183 (2000) 309–330.
- [3] J. Qu, M. Cherkaoui, *Fundamentals of micromechanics of solids*, (Wiley, New Jersey, 2006).
- [4] J. D. Eshelby, The determination of the elastic field of an ellipsoidal inclusion, and related problems, *Proc. R. Soc. Lond. A* 241 (1957) 376–396.
- [5] R. Hill, A self-consistent mechanics of composite materials, *J. Mech. Phys. Solids* 13 (1965) 213–222.
- [6] H. E. Pettermann, A. F. Plankensteiner, H. J. Böhm, F. G. Rammerstorfer, A thermo-elasto-plastic constitutive law for inhomogeneous materials based on an incremental Mori–Tanaka approach, *Comput. Struct.* 71 (1999) 197–214.
- [7] I. Doghri, A. Ouair, Homogenization of two-phase elasto-plastic composite materials and structures: Study of tangent operators, cyclic plasticity and numerical algorithms, *Int. J. Solids. Struct.* 40 (2003) 1681–1712.
- [8] J. Korelc, Automation of primal and sensitivity analysis of transient coupled problems, *Comput. Mech.* 44 (2009) 631–649.
- [9] J. Korelc, P. Wriggers, *Automation of finite element methods*, (Springer, Switzerland, 2016).
- [10] P. Sadowski, K. Kowalczyk-Gajewska, S. Stupkiewicz, Response discontinuities in the solution of the incremental Mori–Tanaka scheme for elasto-plastic composites, *Arch. Mech.* 69 (2017) 3–27.
- [11] P. Sadowski, K. Kowalczyk-Gajewska, S. Stupkiewicz, Consistent treatment and automation of the incremental Mori–Tanaka scheme for elasto-plastic composites, *Comput. Mech.* (2017) doi:10.1007/s00466-017-1418-z.

On Navier slip and Reynolds transpiration numbers

P. ZIÓŁKOWSKI^{1,*}, J. BADUR¹

¹*Energy Conversion Department, Institute of Fluid Flow Machinery, Polish Academy of Sciences, Fiszera 14, Gdańsk, 80-231, Poland*

*pziolkowski@imp.gda.pl

Keywords: micro and nano flows, slip velocity, pressure driven flow, thermally driven flow

In the paper, based on the original argumentation of Reynolds and Maxwell with consideration previous experiences in nano and micro-flows area [1]-[4], a general form of boundary forces that, consist a contributions from the friction and the mobility components: $\mathbf{f}_{\partial V} = \mathbf{f}_r + \mathbf{f}_m$ has been extended to common effects of the bulk and surface motion. Hence, adopting Reynolds reasoning to a porous media at whole, we reexamine the Poiseuille-Knudsen-Reynolds equation in terms sum of three contributions: the bulk pressure driven flow, and two mobility surface forces, mainly: a Knudsen surface slip driven flow and a Reynolds surface thermally driven flow. The main motivation of our work is to find the nondimensional contribution of Navier slip number and Reynolds thermal transpiration number in materials with the high volumetric surface density.

Though the continuous mechanics theory is enormously useful, it does not take into account all the phenomena that occur in the fluid solid interface at the micro and nanoscale. So new approaches such as: continuum thermodynamics with slip velocity and thermal transpiration [1]-[4] or molecular dynamics [5]-[6] are developed with consideration to experimental result. A peculiar difficulty of modeling of flow in porous media arises often if there is impressed to apply some extension of Darcy equation like, for instance, Brinkmann-Darcy-Forchheimer equation [7]. Treating that a nano-pipe is equivalent to a single porous domain and looking for common effects of the bulk and surface motion, one may consider of a following momentum flux integral in any cross section of a porous media oriented by tangential component of unit vector $\mathbf{n}_{\tan}$:

$$\iint_{bulksection} (\rho \mathbf{v} \otimes \mathbf{v} + \mathbf{p}) \mathbf{n}_{\tan} dA + \oint_{\mathcal{C}} (\rho_S \mathbf{v}_S \otimes \mathbf{v}_S + \mathbf{p}_S + \mathbf{n}_{nor} \mathbf{p} + \mathbf{f}_{\partial V}) \mathbf{n}_{\tan} d\mathcal{C} = 0 \quad (1)$$

In the above $\rho \mathbf{v}$ and $\rho_S \mathbf{v}_S$ are the bulk and the surface momentum density vectors, ρ and ρ_S are the gas density in the bulk and on the boundary. Next, $\mathbf{p} = p_{ij} \mathbf{e}_i \otimes \mathbf{e}_j = \mathbf{p}^T$ and $\mathbf{p}_S = \mathbf{p}_S^T$ are the bulk and the surface flux of momentum. The boundary force can be separated on: surface friction and surface mobility, thus: $\mathbf{f}_{\partial V} = \mathbf{f}_r + \mathbf{f}_m$ [1]-[4]. A total momentum influx as to be :

$$\mathfrak{M} = \iint_{bulksection} \rho \mathbf{v} \mathbf{v}_{\tan} dA + \oint_{\mathcal{C}} \rho_S \mathbf{v}_S \mathbf{v}_{S \tan} d\mathcal{C} \quad ; \mathfrak{M} = \dot{m} \mathbf{v}_r \quad (2)$$

where $\iint_{bulksection} \rho \mathbf{v} \mathbf{v}_{\tan} dA$ - contribution from bulk velocity and $\oint_{\mathcal{C}} \rho_S \mathbf{v}_S \mathbf{v}_{S \tan} d\mathcal{C}$ - contribution from the slip velocity. Using arguments of homogenization, there is assume an existence of a resultant velocity $\mathbf{v}_r$, which is parallel to the vector of total momentum which is located somewhere in a geometrical center of a velocity profile. In many cases, independently on the shape of the cross section the bulk profile of velocity is nearly flat

and ending with value of $\mathbf{v}_{Stan} \approx \mathbf{v}_S$ - the magnitude of slip velocity. In the above, according with traditional Reynolds notation, denotes resultant mass flow rate. Taking into account that $\mathbf{f}_{\partial V} = \mathbf{f}_r + \mathbf{f}_m = \nu \mathbf{v}_S - c_{m\theta} grad_s \theta_S$ there can be reorganized the integral (1), expressing explicitly the thermal mobility part $c_{m\theta} grad_s \theta_S$ with the thermo-mobility coefficient $c_{m\theta}$ and the slip friction part $\nu \mathbf{v}_S$ with ν Navier surface friction:

$$\dot{m} \mathbf{v}_r = \underbrace{\iint_{Poiseuille} [p\mathbf{I} - 2\mu \mathbf{d}] \mathbf{n} dA}_{Darcy} - \underbrace{\oint \nu \mathbf{v}_S \mathbf{I}_S d\mathcal{C}}_{Reynolds} - \underbrace{\oint c_{m\theta} grad_s(\Theta_S) \mathbf{I}_S d\mathcal{C}}_{Reynolds} \quad (3)$$

Since the cross section of porous media is quite arbitrary that a known procedure of homogenization can be applied, than eq.(3) leads to 3D resultant equation [8]:

$$\mathbf{v}_r = -\left(\frac{P}{\nu} \mathbf{B} + \mathbf{D}_K\right) \frac{grad P}{P} + \mathbf{D} \frac{grad T}{T} \quad (4)$$

where $\mathbf{v}_r$ - the resultant filtration velocity, μ - a gas viscosity, $\mathbf{B}$ - the permeability tensor, $\mathbf{D}_K$ - the Knudsen accommodation diffusion tensor and $\mathbf{D}$ - thermal transpiration coefficient tensor. Here, does not appear single surface pressure tensor $\mathbf{p}_S$, slip velocity $\mathbf{v}_S$ or the surface temperature Θ_S , since after homogenization their role takes the capillarity pressure P and the capillarity temperature T . Also, the two-dimensional surface gradient, due to homogenization turned into the three-dimensional gradient $grad(\cdot)$. This phenomenological model is based on the already averaged equations for the bulk flow resistance and the surface mobility forces.

References

- [1] J. Badur, M. Karcz, M. Lemański, On the mass and momentum transport in the Navier-Stokes slip layer, *Microfluid Nanofluid*, 11 (2011) 439 – 449 DOI 10.1007/s10404-011-0809-2
- [2] P. Ziółkowski, J. Badur, Navier number and transition to turbulence, *Journal of Physics: Conference Series*, 530 (2014) 012035. doi:10.1088/1742-6596/530/1/012035
- [3] J. Badur, P.J. Ziółkowski, P. Ziółkowski, On the angular velocity slip in nano flows, *Microfluid Nanofluid*, 19 (2015) 191–198. DOI 10.1007/s10404-015-1564-6
- [4] P. Ziółkowski, J. Badur, A theoretical, numerical and experimental verification of the Reynolds thermal transpiration law, *Int. J. Num. M. for Heat and Fluid Flow*, accepted 2017.
- [5] T.A. Kowalewski, P. Nakielski, F. Pierini, K. Zebrzycki S. Pawłowska, Micro and nano fluid mechanics. [in]: *Advances in Mechanics: theoretical, computational and interdisciplinary issues*, Edt. Kleiber et al., (2016) 27-34.
- [6] A. Kucaba-Piętal, Z. Walenta, Z. Peradzyński, Molecular dynamics computer simulation of water flows in nanochannels. *Bull Polish Acad Sci Tech Sci* 57 (2009) 55–61.
- [7] K. Hooman, Heat and fluid flow in a rectangular microchannel filled with a porous medium, *Int. J. Heat Mass Transfer* 51 (2008) 5804–5810.
- [8] G.L. Vignoles, P. Charrier, C. Preux, B. Dubroca, Rarefied pure gas transport in non-isothermal porous media: effective transport properties from homogenization of the kinetic equation, *Transp. Porous Media* 73(2), (2008) 211–232.

SESSION 4:

5th July 2017

"NANOSCALE"

09:00-09:30	[I-04] E. Manske <i>"RF NEMS Based on Suspended Graphene Structures"</i>
09:30-09:50	[O-10] L. Koenders <i>"Self-organized and FIB modified silicon surface used as a measurement standard for the calibration at the nanoscale"</i>
09:50-10:10	[O-11] A. Yacoot <i>"Metrological applications of x-ray interferometry"</i>
10:10-10:20	[E-04] F. Ciceri <i>"MCL nanopositioning and instrumentation: down to 10pm resolution"</i>
10:20-10:40	[O-12] M. Kühnel <i>"Spring constant measurement of small cantilevers"</i>
10:40-11:00	COFFEE BREAK SPONSORED BY THE OPTOTOM

RF NEMS Based on Suspended Graphene Structures

E. MANSKE¹

¹*Technische Universität Ilmenau, Institute of Process Measurement and Sensor Technology, Competence Centre Nanopositioning and Nanomeasuring Machines*

The semiconductor industry has been following Moore's Law for over 40 years with amazing continuity. In spite of enormous improvements made in the field of optical lithography, it can be expected that structure sizes smaller than 20 nm can be achieved by conventional technology only at considerable expense. Consequently, the fundamental challenge is to develop alternative fabrication technologies in particular for micro and nanotechnologies that are capable of measuring and patterning at the atomic scale in growing operating volumes of several hundred millimetres in diameter. Tip-based nanofabrication methods offer high potential in this context. They already enable patterning in sub-10 nm range, but so far only in small processing areas ($< 100 \mu m^2$), at low speed and with limited precision.

Optical methods, due to non-linear effects, already allow the generation of subwavelength structures in the plane even in large areas for surface functionalisation. The challenge addressed by our research consortium is to realise such functionalizations on non-planar surfaces, such as aspheres or freeform surfaces.

The aim of the new Research Training Group "Tip- and laser-based 3D-Nanofabrication in extended macroscopic working areas" (GRK 2182) funded by the German Research Foundation (DFG) from 2017-2021 is to synergistically combine sophisticated nanofabrication techniques with the outstanding capabilities of Nanopositioning- and Nanomeasuring machines (NPM machines) developed at the Technische Universität so that new, multi-scale solutions arise for nanofabrication in large areas. It is to be examined to what extent smallest features can be produced efficiently in large areas by combining the latest AFM tip-based nanofabrication techniques with the NPM technique. Likewise, laser-based subwavelength processing methods in conjunction with the NPM-technology shall open up the possibility to enable truly 3D nanofabrication with highest precision on optical and particularly curved precision surfaces.

Compared with nanometrology the particular challenge of nanofabrication is that static and dynamic deviations of position lead to errors (geometry errors, deviations in shape, roughness) in the produced nanostructures or nanoscale objects. A post generation correction is possible only in the case of measurement, but not during the production processes. The research consortium can build on a successful multi-year collaboration in the SFB *Nanopositioning- and Nanomeasuring Machines (SFB 622)*, the Research Training Group *Lorentz Force* and the research project *Inno Profiles Force Measurement* and the DFG device-center *Micro-Nano-Integration* at IMN MacroNano[®] of the TU Ilmenau.

Self-organized and FIB modified silicon surface used as measurement standard for the calibration at the nanoscale

O. LENCK¹, I. BUSCH^{1,2},
T. DZIOMBA², A. FELGNER¹, M. HEMMLEB², L. KOENDERS¹

¹*Physikalisch-Technische Bundesanstalt (PTB), Bundesallee 100, 38116 Braunschweig, Germany*

²*2m2c calibration, Alt Nowawes 83a, 14482 Potsdam, Germany*

Atomic force microscopes, optical measurement instruments and tactile probes used for measurement of surface topography need to be calibrated in their axes x, y and z to be traceable to the SI unit of length. However, besides the calibration some of the properties of the instruments depending on the measurement task, need to be determined additionally: noise, flatness and roughness.

However optical flats are mainly prepared by polishing thick glass or silicon substrates. The polishing results in flatness deviation, i.e. a waviness of $\lambda/20$ or better, strongly depends on the amount of work invested in polishing. Thus such optical flats are expensive and for many applications in AFM too large. So there is a need for crystalline flats, which are by nature flat due to the crystalline order. For roughness measurements samples are necessary to probe the instruments properties and to verify that the instrument can perform the required task.

For all these cases where flat surfaces are needed, we have developed new measurement standards based on crystalline silicon and its nature properties, i.e. we can prepare very flat, step free terraces which are separated by monoatomic steps of 0.31 nm in height. Due to a special annealing process, we can extend the width of the step free region up to diameters of 150 μm , which allows both, to check the guiding errors of high-resolution scanning instruments as well as the quality of reference waves in optical measurement microscopes, like phase shifting interference microscopes.

Choosing other process parameters, we can grow several single atomic steps in form an amphitheaterlike or a staircase-like structure. The former structures are useful for accurate calibration, but the latter have the advantage to reduce measurement uncertainty of the mean silicon step height to values down to $U(k=2) = 20$ pm.

Another technique developed to produce surfaces with well-defined roughness is using Focus Ion Beam (FIB) technology in combination with silicon and other materials. We are able to write structures in surfaces which can be used for the measurement of profile-like roughness or in an advanced mode as areal-like roughness topography. The big advantage against other techniques is the possibility to repeat the same profile or areal structure several times, what significantly improves the applicability. Some examples will be given.

Metrological applications of x-ray interferometry

A. YACOOT^{*1},

¹*National Physical Laboratory, Hampton Road, Teddington, Middlesex TW11 0LW, UK*

^{*}andrew.yacoot@npl.co.uk

Keywords: Dimensional nanometrology, x-ray interferometry, traceability

An x-ray interferometer can be regarded as a ruler or translation stage where the graduations or displacements are based on the spacing between atoms in a crystal from which x-rays are diffracted: in this case the (220) planes in a silicon crystal with a spacing of 0.192 nm. The lattice parameter of silicon is known to a few parts in 10^8 and is regarded as a secondary length standard that is traceable to the metre. The x-ray interferometer can therefore be used for sub-nanometre positioning and has successfully been used both for the determination of picometre scale errors in optical interferometers and as a high resolution one dimensional translation stage for atomic force microscopy. Quadrature signal process of XRI signals enables picometre sub fringe positioning.

MCL nanopositioning and instrumentation: down to 10pm resolution

F. CICERI¹

¹*Mad City Labs GmbH, Balz-Zimmermann Str 7, 8302 Kloten, Switzerland*

**ferdi@madcitylabs.eu*

Presentation of products delivered by the MadCityLabs company.

Spring constant measurement of small cantilevers

M. KÜHNEL^{1,*}, O. DANNBERG¹, T. IVANOV¹,
I. W. RANGELOW¹, T. FRÖHLICH¹

¹*Technische Universität Ilmenau, 98693 Ilmenau, Germany*

*michael.kuehnel@tu-ilmenau.de

Keywords: small cantilever, spring constant, calibration

The measurement of (sub)nanonewton forces in nanostructures is of rising interest in the field of biology, biophysics, medical science [1]-[3]. A further instance is the material science and here especially the nanoindentation, where surface and layer properties, such as Young's modulus or hardness, are determined in the nanonewton scale [4]-[6]. The atomic force microscopy (AFM) can deliver data at the atomic-scale and is capable to probe the interaction forces between surfaces gained. However, reliable quantitative measurements are difficult to obtain because a very precise knowledge of the cantilever spring constant is necessary.

Several facilities for a traceable and precise calibration of AFM-cantilevers spring constants have been developed by different national metrology institutes [7]-[10]. The cantilever to calibrate is pushed on a precise force sensor (electromagnetic force compensated balance). Based on the measured force and deflection the static spring constant of standard AFM cantilevers have determined with an expanded measurement uncertainty of 1 – 4 % ($k = 2$).

A calibration facility based on this method has been developed at the TU Ilmenau [11]. Measurements and investigations have shown, that uncertainties of the static spring constant calibrations of standard AFM Cantilevers in the range of 1 - 3 % ($k = 2$) can be achieved [12],[13].

The described calibration facilities of the national metrological institutes and of the TU Ilmenau have in common, that the force resolution is limited to ≥ 1 nN. Consequently, for proper calibrations of the cantilever spring constant, the calibration force should be $\geq 1\mu N$. This force range is acceptable for calibrations of standard length cantilevers (100 – 400 μm length) with spring constants of $\geq 1N/m$. For smaller cantilevers with lower spring constants even lower calibration forces must be applied to avoid high deflections which would result in high nonlinearities of the force-deflection curve and consequently in rising measurement uncertainties of the spring constant calibration.

Standard sized cantilevers are usually too stiff to image at high speeds with high resolution. Herein, a high sensitivity to small forces is required resulting in an aggressive cantilever-size scaling [14]. Within the new project "Novel Small cantilevers" cantilevers with lengths $\leq 20\mu m$ and spring constants $\leq 0,1N/m$ but high resonance frequencies will be developed and investigated. Those cantilevers are prospectively needed for fast AFM measurements in bioscience where a low mechanical impact is desired.

For investigations and calibrations of the small cantilevers spring constants a new calibration facility with a higher resolving force sensor will be developed. The force sensor

will base on the electromagnetic force compensation principle and should be capable for spring constant calibrations without increased demands on laboratory environmental surroundings. To calibrate at forces $\leq 100nN$ the desired force resolution is $\leq 0.1nN$.

Acknowledgments The authors acknowledge the financial support from Deutsche Forschungsgemeinschaft (DFG) of the project “Novell Small Cantilever” at Technical University Ilmenau.

References

- [1] Müller D.J., Dufrêne Y. F.: Force nanoscopy of living cells *Current Biology* (2011) Vol 21 No 6, 212–216
- [2] Bull M.S. et al.: Improved Single Molecule Force Spectroscopy Using Micromachined Cantilevers, *ACS Nano* (2014) 8 (5), pp 4984–4995
- [3] Plodinec M. et al. (2012), *Nature Nanotechnology* 7, 757–765
- [4] Bhushan B and Koinkar V N: Nanoindentation hardness measurements using atomic force microscopy *Appl. Phys. Lett.* (1994) 64 1653–5
- [5] Kim S-H and Boyd J G: A new technique for measuring Young’s modulus of electroplated nickel using AFM *Meas. Sci. Technol.* 17 (2006) 2343
- [6] Lee, C., Wei, X., Kysar, J.W. and Hone, J., "Measurement of the elastic properties and intrinsic strength of monolayer graphene," *Science* 321, pp. 385–388, 2008.
- [7] Hoffmann, W. et al., Methods of characterising micro mechanical beams and its calibration for the application in micro force measurement systems, in: *Proc. of the MICRO.tec 2000*. Presented at the MICRO.tec 2000 Conf., Hannover, Germany, pp. 819–823.
- [8] J. Pratt, J. Kramar, D. Newell, D. Smith, Review of SI traceable force metrology for instrumented indentation and atomic force microscopy, *Meas. Sci. Technol.* 16 (11) (2005) 2129–2137.
- [9] M.-S. Kim, J.-H. Choi, J.-H. Kim, Y.-K. Park, Si-traceable determination of spring constants of various atomic force microscope cantilevers with a small uncertainty of 1
- [10] M.-S. Kim, J. Pratt, U. Brand, C. Jones, Report on the first international comparison of small force facilities: a pilot study at the micronewton level, *Metrologia* 49 (2012) 70–81.
- [11] C. Diethold, F. Hilbrunner, T. Fröhlich, M. Kühnel and E. Manske, Determination of force to displacement curves using a nanopositioning system based on electromagnetic force compensated balances, *Measurement* (2014), <http://dx.doi.org/10.1016/j.measurement.2014.02.034>
- [12] C. Diethold, M. Kühnel, Tzvetan Ivanov, Ivo W. Rangelow and T. Fröhlich, Determination of AFM-Cantilever spring constants using the TU Ilmenau force displacement measurement device *Proceedings of the XXI Imeko world Congress*, S.175-180, (2015)
- [13] M. Kühnel, T. Fröhlich, R. Popadic, U. Brand, National Comparison of spring constant measurements of atomic force microscope cantilevers, *Proceedings of the IMEKO 23rd TC3, 13th TC5 and 4th TC22 International Conference 2017*, Helsinki, Finland
- [14] Kaestner et al., Scanning probes in nanostructure fabrication, *J. Vac. Sci. Technol. B*, Vol. 32, No. 6, Nov/Dec 2014

SESSION 5:
5th July 2017

"GRAPHENE"

11:00-11:30	[I-05] N. Klein <i>"Graphene for nanoscale sensor devices"</i>
11:30-11:50	[O-13] A. Kucaba-Piętal <i>"Wall material effect on water transport coefficients in nanoconfinement"</i>
11:50-12:00	[E-04] I. Mościcki <i>"Innovative solutions for Surface and Nanoscience"</i>
12:00-12:20	[O-14] Z. Liao <i>"In-Situ Stretching Patterned Graphene Nanoribbons in the Transmission Electron Microscope"</i>
12:20-12:30	[E-05] D. Gnatek / M. Foltyniak <i>"AGP"</i>
12:30-12:50	[O-15] K. Gajewski <i>"Electromechanical studies of graphene based devices"</i>

Graphene for Nanoscale Sensor Devices

**N. KLEIN¹, S. GONISZEWSKI¹, M. ADABI¹,
D. KWONG HONG TSANG¹, O. SHAFOROST¹, S. HANHAM¹, P. PETROV¹, J.
LISCHNER¹, I. DUNLOP¹,**

¹Imperial College London, Department of Materials

As transparent and flexible semimetal with strong control of its electrical conductivity by surface modifications and by external gate voltages, graphene is one of the most promising materials for the possible realization of low cost multisensor platforms. Wafer scale graphene prepared by chemical vapour deposition can either be transferred onto silicon /SiO₂ using Si as bottom gate of a simple field effect transistor (FET) structure, alternatively heterostructures based on thin layers of aluminium nitride embedded between 2 graphene layers provide a wafer scalable technology for flexible and optical transparent FET sensors and arrays [1]. Using non-covalent 1-pyrenebutyric acid N-hydroxysuccinimide ester (PBASE) as linker molecule between graphene FET structures and antibodies, macromolecular biological species and cells can be immobilized and concentration dependent shifts of the Dirac Point in graphene enables sensitive detection via charge. Recently, we demonstrated exosome detection by a graphene FET structure which forms one wall of a microfluidic channel. As a new concept of a dual-mode biosensor, we have secured the funding to develop a nanomechanical graphene aluminium nitride free standing structure which should enable combined mass and charge detection – a viable approach to tackle the high false positives and false negatives of current biomarker- based early state cancer diagnostic tools.

References

- [1] M. Adabi et al., Scientific Reports 7,44202 (2017)

Wall material effect on water transport coefficients in nanoconfinement

A. KUCABA-PIĘTAL^{1,*}

¹*Rzeszow University of Technology, Powstańców Warszawy 12, Rzeszów, 35-959, Poland*
 *anpietal@prz.edu.pl

Keywords: Molecular dynamics simulation, Nanoflows, Viscosity, Nanoconfinement

Nanoconfinement is known to have several significant structural effects on fluid and some researchers have reported that the confined phases are highly inhomogeneous. The structure and dynamics of confined fluids can be vastly different from their bulk behavior. The liquids often tend to be structured near a solid surface, with typically higher local density than that in bulk. The transport properties of fluids through nanochannels and nanopores are the most important phenomena from both a practical and theoretical point of view. Bitsanis et al. [1] studied the flow of fluids confined in a slit pore and found that the diffusivity decreases as the pore width decreases and viscosity increases at very narrow pores. The viscosity, similarly as the other transport coefficients (i.e. the thermal conductivity, the diffusion coefficient) describe the material properties of fluid within the framework of continuum theory. Mass diffusion is a stochastic process, i.e. a consequence of local heterogeneity of matter distribution and of the energy at a molecular level and makes a contribution to molecules transport and is often coupled with phenomena which can be observed in nanoscale flows

The paper presents the values of water viscosity calculated by use molecular dynamic simulation method (MD). There are several ways to obtain the values of the viscosity by means of MD results: the Green-Kubo method, the Stokes-Einstein method or by simulating Couette shear flow or Poiseuille flow. The last method was used to predict the argon viscosity coefficient and the water viscosity coefficient [2]. In this paper the water viscosity and diffusion coefficient is determined for the water flowing through the copper and the quartz channels which height is equal to 10 and 5 water molecular diameters. The simulation were performed at T= 300 K using an in-house modified version of Moldy solver which permits to add the force to each water molecule to drive the flow. MD computation cell was the same as described in [3]. For a water the model TIP4P was used. Results show that when fluid is confined in nanochannels, the transport coefficients can differ significantly from the bulk value and it depends from wall material. A similar observation for shear flow of a water has been reported in [4].

Acknowledgments

Acknowledgment. I would like to thank to ICM Warsaw University for access to high power computers (Research Project No. G49-1)

References

- [1] Bitsanis S, Yingchun Liu, Qi Wang, and Linghong Lu : Transport Properties and Distribution of Water Molecules Confined in Hydrophobic Nanopores and Nanoslits: Langmuir (2004), 20, 6921-6926
- [2] A.P.Markenstein R.Hartkamp, S.Luding, J.Westerweel: A comparison of the value for several water models using Poiseuille flow in nano-channel: J.Chem.Phys.136,(2012) 134-144
- [3] A. Kucaba-Piętal, Z. Peradzyński and Z. Walenta: MD Computer simulation of water flows in nanochannels. Bull. Acad. Polon. Sci. Ser. Sci. Techn. 57(1), (2009) 1-15

- [4] Truong Quoc Vo, BoHung Kim, Transport Phenomena of Water in Molecular Fluidic Channels Scientific Reports 6, Article number: 33881 (2016) doi:10.1038/srep33881

Innovative solutions for Surface and Nanoscience

I. MOŚCICKI¹

¹*Labnatek (IGHT s.c.), ul. Marszałkowska 80, 00-517, Warszawa, Poland*

**info@labnatek.pl*

Presentation of products delivered by the Labnatek (IGHT s.c.) company.

In-Situ Stretching Patterned Graphene Nanoribbons in the Transmission Electron Microscope

Z. LIAO^{1,2,*}, L. MEDRANO SANDONAS², T. ZHANG³,
M. GALL¹, A. DIANAT², R. GUTIERREZ², U. MÜHLE¹, J. GLUCH¹, R.
JORDAN³, G. CUNIBERTI², E. ZSCHECH¹

¹ *Fraunhofer Institute for Ceramic Technologies and Systems (IKTS), 01109 Dresden, Germany*

² *Institute for Materials Science and Max Bergmann Center of Biomaterials, Technische Universität Dresden, 01069 Dresden, Germany*

³ *Professur für Makromolekulare Chemie, Department Chemie, Technische Universität Dresden, 01069 Dresden, Germany*

*Zhongquan.liao@ikts.fraunhofer.de

Keywords: : Graphene nanoribbons, In-situ stretching, TEM

As the first isolated two-dimensional (2D) material,[1] graphene leads an unprecedented boom in interdisciplinary research in the past decade. It shows tremendous potential applications. One of them is in the area of the novel electronic devices due to the superior charge carrier mobility ($2 \times 10^5 \text{cm}^2 \text{V}^{-1} \text{s}^{-1}$) and very high thermal conductivity (3000WmK^{-1}).[2], [3] However, it seems that the application of graphene in transistors as a planar channel material is unlikely to happen within the next decade because of the absence of a bandgap. Bandgap engineering and increasing the on/off ratio are very essential for its application in transistors. One of the approaches to tune the bandgap of graphene is strain engineering. On the other hand, potential applications of graphene in electronic devices and sensors inevitably involve sophisticated structure fabrications (patterning).[4] Therefore, the investigation of strain engineering on patterned graphene-based nanostructures is a necessary step towards its practical application for future electronics.

In this study, focused ion beam and focused electron beam are used to directly pattern graphene into nanoribbons. Focused electron beam shows relatively slow patterning speed, but preserves much better crystallinity of graphene. An in-situ experimental methodology in the transmission electron microscope (TEM) is demonstrated to stretch the patterned graphene nanoribbons. The mechanical response of the graphene nanoribbons is monitored in-situ, and the low-loss electron energy loss spectrum (EELS) of the stretched nanoribbons under strain is recorded as an attempt to reveal the tuning of bandgap. The correlation from simulations are provided as well. Density Functional Based Tight Binding (DFTB) simulation is conducted to predict a feasible bandgap opening as a function of width in graphene nanoribbons at low strain levels.

Acknowledgments

The financial support from the Free State of Saxony of Germany (SMWK, Project Number 4-7531.50/1125/1) is gratefully acknowledged

References

- [1] A. K. Geim, K. S. Novoselov, Nat. Mater. 6 183 (2007).
- [2] S. V. Morozov et al., Phys. Rev. Lett. 100 016602 (2008).
- [3] A. A. Balandin, Nat. Mater. 10 569 (2011).
- [4] K. S. Novoselov et al., Science 315 1379 (2007).

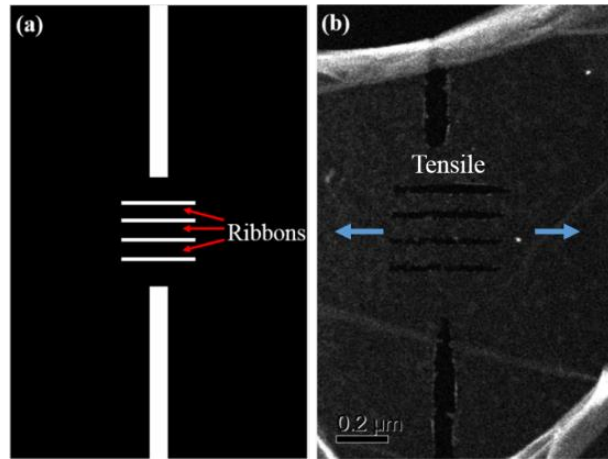


Fig. 1: An experimental sample. (a) Schematic picture, and (b) HAADF STEM image.

New approach to large-area graphene - HSMG ®

D. GNATEK¹

¹*Advanced Graphene Products, Nowy Kisielin - A. Wysockiego 4, 66-002, Zielona Gora,
Poland*

*d.gnatek@agp-corp.com

Advanced Graphene Products (AGP) is not only a manufacturer and supplier of high quality graphene materials but also a research centre focused on the development of graphene-related applications. AGP holds international patents for the unique method of graphene production – growth on liquid metal matrix which yields in graphene characterized by exceptional strength and durability (HSMG ®- High Strength Metallurgical Graphene). The Company is actively working on the implementation of graphene products in various areas, such as sensor devices, composite materials or photovoltaics.

Electromechanical studies of graphene based devices

**K. GAJEWSKI^{1,*}, L. HAO², A. M. IONESCU³,
W. STRUPIŃSKI⁴, P. NIEDZIELSKI⁵, P. GRABIEC⁶ T. GOTSZALK¹**

¹ *Wrocław University of Science and Technology, Faculty of Microsystem Electronics and Photonics, ul. Janiszewskiego 11/17, 50-372 Wrocław*

² *National Physical Laboratory, Teddington, Middlesex, TW11 0LW*

³ *École Polytechnique Fédérale de Lausanne, CH-1015, Lausanne, Switzerland*

⁴ *Institute of Electron Materials Technology, Wólczyńska 133, 01-919 Warszawa, Polska*

⁵ *Łódź University of Technology, ul. Stefanowskiego 1/15, 90-924 Łódź, Polska*

⁶ *Institute of Electron Technology, Al. Lotników 32/46, 02-668 Warsaw, Poland*

*krzysztof.gajewski@pwr.edu.pl

Keywords: Graphene, STM, AFM, Raman spectroscopy

Two-dimensional materials (2D), due to their interesting physical properties, are at the center of interest of modern material engineering. The best known 2D material is graphene, combining excellent electrical and thermal conductivity and significant mechanical strength.

Graphene research has led to a number of discoveries such as fractional Hall effect, "massless" electrons, gas impermeability, or applications such as conductive pathways for flexible electronics, graphene transistors, hydrogen storage.

The following paper presents the results of studies on the electromechanical properties of two-dimensional nanostructures based on graphene. Graphene was obtained by chemical or physical vapor deposition. Graphene was transferred (PMMA method or marker-free method) to substrates like:

- SiO₂ / Si, containing microcavities, which formed a membrane;
- low density polyethylene (LDPE) for elastic electronics;
- SiO₂ / Al₂O₃;
- metals, etc.

Raman spectroscopy techniques and scanning probe microscopy (scanning tunneling microscopy and atomic force microscopy), have been used in this study to determine the parameters of the manufactured devices such as the stress state, doping level or change in deformation resistance of the structure. Fig. ??,1 and 2 showed selected results obtained during the investigations of the electromechanical properties of graphene devices.

Acknowledgments

This work was financed from WrUST statutory grant no. 0401/0234/16. KG work was partially supported by the National Science Centre, Poland by the PRELUDIUM 9 programme (grant Nr 2015/17/N/ST7/03850).

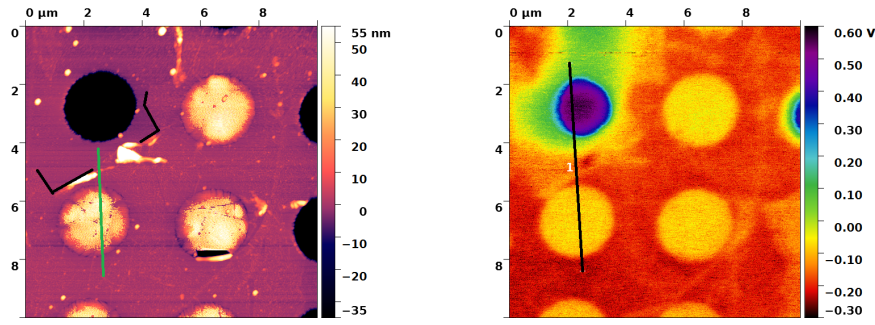


Fig. 1: Kelvin probe microscopy observation of the graphene membranes. Left - topography; right - CPD[1]



Fig. 2: Kelvin probe microscopy observation of the graphene-LDPE composites. Left - topography; right - CPD[2]

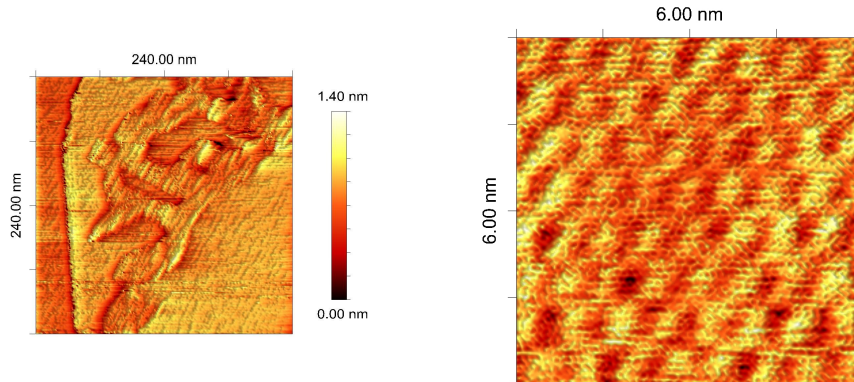


Fig. 3: Scanning tunneling microscopy studies of graphene on SiC substrate [3]

References

- [1] Gajewski, et al., Raman Spectroscopy and Kelvin Probe Force Microscopy characteristics of the CVD suspended graphene, (2016) *Diamond and Related Materials*, 64, pp. 27-33.
- [2] Gajewski, K., et al., Kelvin Probe Force Microscopy investigations of High Strength Metallurgical Graphene transferred on low-density polyethylene, (2016) *Microelectronic Engineering*, 157, pp. 71-77.
- [3] Gajewski, K., et al., Scanning probe microscopy investigations of the electrical properties of chemical vapor deposited graphene grown on a 6H-SiC substrate, (2015) *Micron*, 68, pp. 17-22

SESSION 6:

5th July 2017

"DIAMOND & CARBON"

- 13:40-14:10 [I-06] R. Bogdanowicz *"Nanocrystalline-diamond thin films for MEMS and NEMS devices"*
- 14:10-14:30 [O-16] M. Kaestner *"Mix match approach for high-throughput field-emission scanning probe lithography with active cantilever"*
- 14:30-14:40 [E-06] M. Włodowski *"OPTOTOM"*
- 14:40-15:00 [O-17] C. Lenk *"Single electron transistors for room temperature operation fabricated by FE-SPL and cryogenic plasma etching"*
- 15:00-15:20 [O-18] M. Ficek *"Diamond-phase (sp^3 -C) rich boron doped carbon nanowalls (sp^2 -C): hay carbon"*
- 15:20-16:20 POSTER SESSION & COFFEE BREAK SPONSORED BY THE MSA SYSTEMS

Nanocrystalline-diamond thin films for MEMS and NEMS devices

R. BOGDANOWICZ^{1,*}, M. SOBASZEK¹, M. FICEK¹, Ł. GOŁUŃSKI¹, D. KOPIEC², M. MOCZAŁA², K. ORŁOWSKA², M. SAWCZAK³, T. GOTSZALK²

¹*Department of Metrology and Optoelectronics, Faculty of Electronics, Telecommunications and Informatics, Gdańsk University of Technology, 11/12 G. Narutowicza St., 80-233 Gdańsk, Poland*

²*Faculty of Microsystem Electronics and Photonics, Wrocław University of Technology, 11/17 Janiszewskiego St., 50-372 Wrocław, Poland*

³*Polish Academy of Sciences, The Szewalski Institute of Fluid-Flow Machinery, 14 Fiszerza St., 80-231 Gdańsk, Poland*

*rbogdan@eti.pg.gda.pl

Keywords: Boron-Doped Diamond, NEMS and MEMS devices, STM; C-AFM; KPFM

Surface modification of the micro- and nanocantilevers is a very interesting chance for modification of mechanical, electrical and physical properties of these micro- and nanodevices. Modified cantilevers can be attractive tools in atomic force microscopes (AFM) as a scanning probes and in biosensor applications [1],[2].

Fabrication processes of thin boron-doped diamond films on silicon-based micro- and nano-electromechanical structures will be presented. Nanocrystalline boron doped -diamond (B-NCD) films were deposited using Microwave Plasma Assisted Chemical Vapour Deposition (MW PA CVD) method [3]. The variation of B-NCD morphology, structure and optical parameters were particularly investigated. The sp^3/sp^2 ratio was calculated using Raman spectra deconvolution method. The resistivity of B-NCD coatings was studied using four-point probe measurements.

We present several kind of silicon cantilevers which surface was coated with boron-doped diamond films. The change of mechanical parameters was investigated with laser vibrometer. The analysis of a spring constant and resonance frequency shift connected with mass addition are shown. Electrical parameters of boron-doped diamond films were investigated by electrical mode of AFM – Kelvin probe force microscopy (KPFM). Work function and electrostatic forces of deposited materials were measured [4].

Acknowledgments

The authors gratefully acknowledge financial support from the Polish National Science Centre (NCN) under Grant No. 2014/14/M/ST5/00715 and 2015/16/T/ST7/00469. The DS funds of the Faculty of Electronics, Telecommunications and Informatics of the Gdansk University of Technology are also acknowledged.

References

- [1] R. Bogdanowicz, M. Sobaszek, M. Ficek, D. Kopiec, M. Moczala, K. Orłowska, M. Sawczak, and T. Gotszalk. "Fabrication and characterization of boron-doped nanocrystalline diamond-coated MEMS probes." *Applied Physics A* 122, no. 4 (2016): 1-9.
- [2] R. Raiteri, Micromechanical cantilever-based biosensors, *Sensors and Actuators B Chem.* 79, 115, (2001)

- [3] R. Bogdanowicz, A. Fabiańska, L. Golunski, M. Sobaszek, M. Gnyba, J. Ryl, K. Darowicki, T. Ossowski, S.D. Janssens, K. Haenen, E.M. Siedlecka, *Diamond and Related Materials*, 39, 82, (2013)
- [4] W. Melitz, J. Shen, A.C. Kummel, and S. Lee, Kelvin probe force microscopy and its application, *Surface Science Reports* 66, 1, (2011)

Mix and match approach for high-throughput field-emission scanning probe lithography with active cantilever

M. KAESTNER^{1,*}, C. LENK¹, Y. KRIVOSHAPKINA¹,
T. IVANOV¹, A. AHMAD¹, A. REUM², M. HOLZ², E. GULIYEV¹, I. W.
RANGELOW¹

¹*Department of Micro- and Nanoelectronic Systems (MNES), Ilmenau University of Technology, Gustav-Kirchhoff-Str. 1, 98693 Ilmenau, Germany*

²*nano analytik GmbH, Ehrenbergstraße 1, 98693 Ilmenau, Germany*

*marcus.kaestner@tu-ilmenau.de

Keywords: Nanofabrication, Nanolithography, Active Scanning Probes

The ability of rapid manufacturing of features in the single nanometer regime has been identified as one of the most important step to enable future nanoelectronic, NEMS, photonic, and bionanotechnology-based devices. In order to address this challenge we are working on low energy electron exposure emitted from scanning proximal probe tips [1] - [4], Fig. 1. Here, the electron energy is close to the lithographic relevant excitation and the electrons penetration volume is minimized. In consequence, a much more spatially confined lithographic interaction is enabled. Based on the thermally actuated, piezoresistive cantilever technology we have developed a fieldemission scanning probe lithography (FE-SPL) platform, which is able to image, inspect, align and pattern features down to single digit nano regime.

However, due to the serial operation principle the throughput capability is rather limited. Mix and match lithographic strategies [1],[3] are a promising path in order to overcome this trade-off. In this term, all large scale features are defined by conventional lithography, whereas only the nanoscale features are patterned via closed loop FE-SPL. By incorporation of in-situ imaging and alignment procedures a nanometer precise step-and-repeat, multi-step and multi-layer lithography is enabled. In such a manner we have recently demonstrated the combination of FE-SPL with optical lithography (Fig. 3)[1], electron beam lithography (EBL, Fig. 2) [4] as well as with nanoimprint lithography (NIL). As a result single electron transistor (SET) devices are patterned, transferred as well as replicated successfully. The electrical characterization reveals quantum dot sizes of < 2 nm giving Coulomb diamonds at room temperature operation (Fig. 3). Within the talk an overview about the applied technology, from basics towards application, in terms of beyond CMOS device fabrication is given.

Acknowledgments The research leading to these results has received funding from the European Union's Seventh Framework Programme FP7/2007-2013 under Grant Agreement No. 318804 (Single Nanometer Manufacturing for beyond CMOS devices – acronym: SNM).

References

- [1] M. Kaestner et al., J. Micro/Nanolith. MEMS MOEMS 12, 031111 (2013).
- [2] M. Kaestner et al., J. Vac. Sci. Technol. B 32, 06F101 (2014).
- [3] M. Kaestner et al., J. Micro/Nanolith. MEMS MOEMS 14, 031202 (2015).

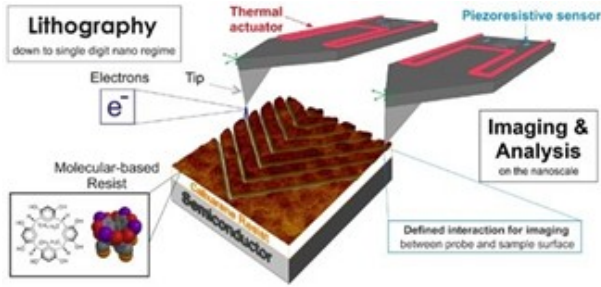


Fig. 1: FE-SPL principle of operation

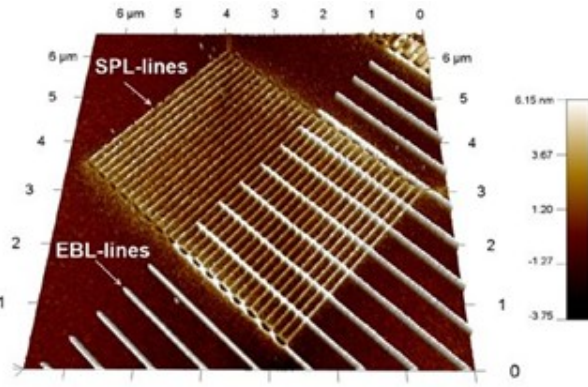


Fig. 2: Mix & match lithography combining incorporating active cantilever systems [1]. Due to field emission scanning probe lithography (FEthe combination of imaging and lithography a SPL) and standard electron beam lithography closed loop lithography is enabled. (EBL) within the same resist layer.

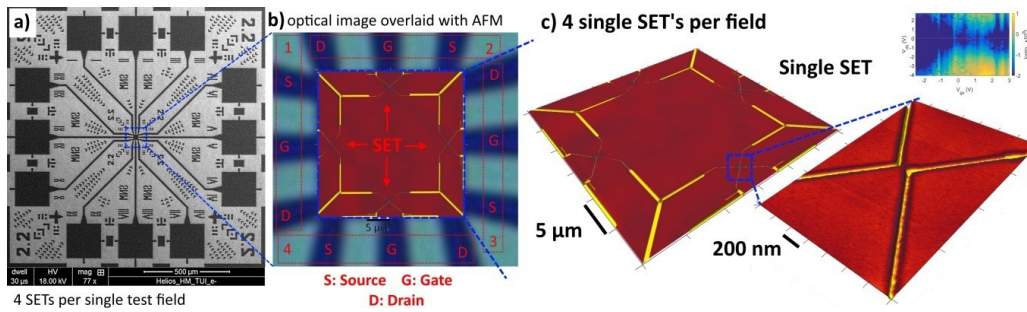


Fig. 3: Single electron transistor (SET) devices fabricated by mix & match optical lithography and FE-SPL, cryogenic plasma etching and pattern-dependent oxidation. [4]

- [4] I. W. Rangelow et al., J. Vac. Sci. Technol. B 34, 06K202 (2016).

Optical Techniques in Measuring Surface Topography

M. WŁADOWSKI¹

¹*OPTOTOM, ul. Głębocka 54c, 03-287 Warsaw, Poland*

^{*}*mariusz.wladowski@optotom.pl*

Presentation of products delivered by the OPTOTOM company.

Single electron transistors for room temperature operation fabricated by FE-SPL and cryogenic plasma etching

C. LENK^{1,*}, M. KAESTNER¹, Y. KRIVOSHAPKINA¹, C. AYDOGAN³,
D. NECHEPURENKO¹, M. HOFMANN¹, B. VOLLAND¹, W. M. MAJSTRZYK⁴,
T. IVANOV¹, S. LENK¹, M. HOLZ²,
A. AHMA¹, T. ANGELOV^{1,2}, I. ATANASOV¹, A. REUM^{1,2},
Z. DURRANI⁵, M. JONES⁵, I. W. RANGELOW¹

¹*Department of Micro- and Nanoelectronic Systems (MNES), Ilmenau University of Technology, Gustav- Kirchhoff-Str. 1, 98693 Ilmenau, Germany*

²*Nano Analytik GmbH, Ehrenbergstraße 1, 98693 Ilmenau, Germany*

³*Electronics and Cryptology Department TUBITAK - YITAL, Gebze, Kocaeli, Turkey*

⁴*Department of Metrology Micro- and Nanostructures, Wroclaw University of Science and Technology*

⁵*Department of Electrical and Electronic Engineering, Imperial College London, South Kensington, London SW7 2AZ, United Kingdom*

*claudia.lenk@tu-ilmenau.de

Keywords: Single Electron Transistor, Scanning Probe Lithography, cryogenic plasma etching

Single electron transistor (SET) devices are promising as post-CMOS era technology due to their small energy consumption and high switching speed. This opens up the possibility to build low- power and high-density integrated circuits. A bundle of methods for their fabrication was introduced, e.g. by chemical assembly of molecules combined with electron beam lithography, optical lithography with subsequent thermal oxidation, extreme ultraviolet lithography, atomic force microscopy based placement of carbon nanotubes, building heterojunctions or using metallic grains or nanoparticles [1, 2]. However, these methods mainly rely on a statistical arrangement and thus exhibit low reproducibility.

Here, we are introducing the fabrication of SETs by mix and match lithography (optical and field emission scanning probe lithography (FE-SPL)), subsequent cryogenic etching and thermal oxidation patterning [1]. Therefore, SOI samples are pre-structured by optical lithography defining contact pads and the centered patterning area. A 15 nm thick resist film is spin-coated onto the sample. By FE-SPL the SET device is defined. FE-SPL, which is based on thermally actuated, piezoresistive cantilever technology (fig. 1 left) allows imaging, inspection, alignment and patterning of features in the range of single-nanometers [2] while fields of up to $200\mu\text{m} \times 200\mu\text{m}$ can be written. Thus, the full field consisting of four SETs and their connections to the contacts is written within a single patterning process. An overlay alignment and stitching accuracy in the range of single-nanometres were demonstrated [3]. The alignment of the pattern with contacts and line quality are counterchecked directly after lithography using the same cantilever. In the subsequent plasma etching process the resist works as local protection mask. In order to obtain optimal pattern transfer results plasma etching of silicon at cryogenic environment is applied. Fig. 1 (middle) shows an AFM image obtained after etching of a field with 4 SETs and their connections to the contacts.

A pattern-dependent thermal oxidation step is applied afterwards at Imperial College London (ICL) for quantum dot definition and, subsequently, SETs are electronically characterized (at ICL). Coulomb diamonds were identified at room temperature. The effective

quantum dot size calculated from these measurements is ranging below 2 nm. Different layouts were tested and optimized regarding gate leakage.

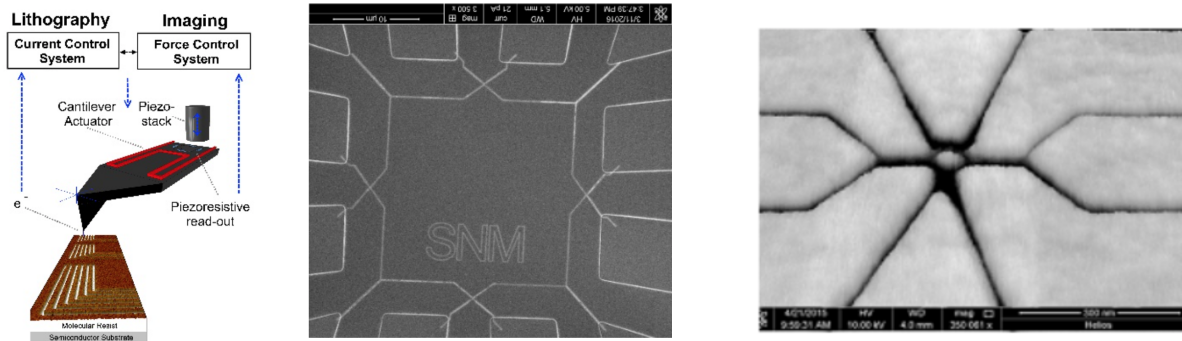


Fig. 1: (left) Scheme of field-enhanced, current-controlled SPL method (middle) Scanning electron microscopy (SEM) image of etched field with 4 SETs and connections to the contacts. (right) SEM of SET with dot layout.

Acknowledgments

The research leading to these results has received funding from the European Union's Seventh Framework Programme FP7/2007-2013 under Grant Agreement No. 318804 (Single Nanometer Manufacturing for beyond CMOS devices – acronym: SNM).

References

- [1] Z.A.K. Durrani, H. Ahmed, Nanosilicon (Elsevier), ch. 10 (2007), 335-359.
- [2] M. R. Islam, D. Jounga, and S.I. Khondaker, Nanoscale 7 (2015), 9786-9792.
- [3] I.W. Rangelow et al., J. of Vacuum Science and Technology B34 (2016) 06K202.
- [4] M. Kaestner et al., J. of Micro/Nanolith. MEMS and MOEMS Vol. 14(3) (2015) 031202.
- [5] V. Ishchuk et al., Applied Physics A 123 (2017), 1–12.

Diamond-phase ($\text{sp}^3\text{-C}$) rich boron doped carbon nanowalls ($\text{sp}^2\text{-C}$): hay carbon

M. FICEK^{*1}, K. SIUZDAK², J. RYL³,
M. SAWCZAK², M. SOBASZEK¹, B. DEC¹, K. DAROWICKI³, R. BOGDANOWICZ¹

¹*Department of Metrology and Optoelectronics, Faculty of Electronics, Telecommunications and Informatics, Gdańsk University of Technology, 11/12 G. Narutowicza St., 80-233 Gdańsk, Poland*

²*Polish Academy of Sciences, The Szwedzki Institute of Fluid-Flow Machinery, 14 Fiszera St., 80-231 Gdańsk, Poland*

³*Department of Electrochemistry, Corrosion and Materials Engineering, Faculty of Chemistry, Gdansk University of Technology, 11/12 Narutowicza St., 80-233 Gdansk, Poland*

¹*Department of Metrology and Optoelectronics, Faculty of Electronics, Telecommunications and Informatics, Gdańsk University of Technology, 11/12 G. Narutowicza St., 80-233 Gdańsk, Poland*

²*Polish Academy of Sciences, The Szwedzki Institute of Fluid-Flow Machinery, 14 Fiszera St., 80-231 Gdańsk, Poland*

³*Department of Electrochemistry, Corrosion and Materials Engineering, Faculty of Chemistry, Gdansk University of Technology, 11/12 Narutowicza St., 80-233 Gdansk, Poland*

*matficek@pg.edu.pl

Keywords: Boron Doped Carbon Nanowalls, Growth mechanism, XPS, Raman spectroscopy

Our recent report [1] growth and application of Boron-Enhanced Carbon Nanowalls (B:CNWs) as biosensors was shown. The B:CNW samples were deposited by the microwave plasma assisted CVD using a gas mixture consisting of H_2 , CH_4 , B_2H_6 and N_2 . The time of growth was 4, 7 and 6h for B-CNW-1.2k, B-CNW-2k and B-CNW-5k, respectively. All samples were doped by using diborane (B_2H_6) as acceptor precursor. $[\text{B}]/[\text{C}]$ ratios in plasma were 1.2k, 2k and 5k ppm resulting in various content of boron incorporation into nanowalls. To investigate morphology of deposited samples the high-resolution scanning electron microscope was used. The left hand side of Figure 1 is showing the surface morphology of obtained B-CNW samples. For all deposited samples obtained walls are nearly straight which is difference with others reports showing maze-like CNW. As can be seen the influence of boron presence has significant impact on wall length and quantity. The average length of walls for sample B-CNW-1.2k is approximately $1.5\mu\text{m}$, and decreases with increasing boron level in gas phase, obtaining length below 500 nm for B-CNW-5k sample. At the same time, the quantity of walls resulting in denser film e.g. B-NCD-5k. This can lead to increasing specific capacitance due to increasing amount of small pores between nanowalls. On the other hand, the boron has also influence onto the film thickness (right panel of Figure 1). To achieve the same thickness of the different doped film, the times of deposition varies highly. Even a small change in gas phase can almost double the deposition time. This phenomena shows direct impact of boron atoms on growth kinetics during MWCVD process. The growth process of CNW in presence of nitrogen involves H, CHx, and CN radicals [2].

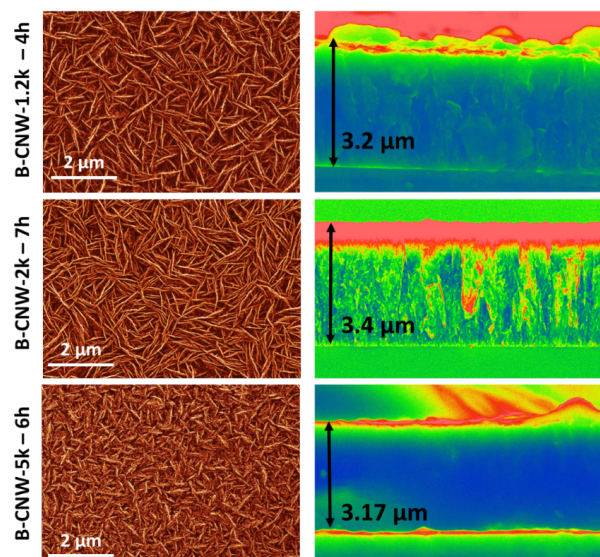


Fig. 1: The HR-SEM images of B-CNW vs. various boron doping level ($[B]/[C]$: 1.2k, 2k and 5k ppm): (left) top-view morphology, (right) cross-sectional micro-images with specific wall thickness

The boron incorporation level in diamond lattice is confirmed by the correlation of its composition in the gas phase (see Figure 2) with boron content as determined experimentally by LIBS or XPS. Positively, the re-nucleation processes proving shorten walls, which results in higher specific surface area, while larger boron concentration produces more carriers / acceptors enhancing electrochemical performance of nanowalls, as will be shown later on

Acknowledgments

The authors gratefully acknowledge financial support from the Polish National Science Centre (NCN) under Grant No. 2014/14/M/ST5/00715 and 2015/16/T/ST7/00469. The DS funds of the Faculty of Electronics, Telecommunications and Informatics of the Gdansk University of Technology are also acknowledged.

References

- [1] K. Siuzdak et al., "Boron-Enhanced Growth of Micron-Scale Carbon-Based Nanowalls: A Route toward High Rates of Electrochemical Biosensing," *ACS Appl. Mater. Interfaces*, vol. 9, no. 15, pp. 12982–12992, Apr. 2017.
- [2] M. Hiramatsu and M. Hori, *Carbon Nanowalls*. Vienna: Springer Vienna, 2010.
- [3] Y. Wu, B. Yang, B. Zong, H. Sun, Z. Shen, and Y. Feng, "Carbon nanowalls and related materials," *J. Mater. Chem.*, vol. 14, no. 4, pp. 469–477, Feb. 2004.
- [4] M. Hiramatsu and M. Hori, "Fabrication of carbon nanowalls using novel plasma processing," *Jpn. J. Appl. Phys.*, vol. 45, no. 6S, p. 5522, 2006.
- [5] N. G. Shang et al., "Catalyst-Free Efficient Growth, Orientation and Biosensing Properties of Multilayer Graphene Nanoflake Films with Sharp Edge Planes," *Adv. Funct. Mater.*, vol. 18, no. 21, pp. 3506–3514, Nov. 2008.

SESSION 7:

5th July 2017

"BIOTECHNOLOGY"

- 16:20-16:50 [I-07] A. Ptak *"Single-molecule force spectroscopy: theoretical models, simulations, experiments"*
- 16:50-17:10 [O-19] A. Montazeri Hedesh *"The role of spherical voids on the deformation mechanism of Cu/Ag nanocomposites via MD simulation"*
- 17:10-17:20 [E-07] J. Budzioch *"TBA"*
- 17:20-17:40 [O-20] K. Herman *"Quantitative characterization of the specific interactions of syndecans for cancer diagnosis with the dynamic force spectroscopy"*
- 17:40-18:00 [O-21] W. Majstrzyk *"Electromagnetically actuated silicon microcantilever arrays for multicantilever force- distance spectroscopy"*
- 18:30-23:30 CONFERENCE DINNER ON THE CRUISE

Single-molecule force spectroscopy: theoretical models, simulations, experiments

A. PTAK^{*1}

¹*Institute of Physics, Faculty of Technical Physics, Poznan University of Technology,
Piotrowo 3, 60-965 Poznan, Poland*

^{*}arkadiusz.ptak@put.poznan.pl

Keywords: adhesion, Bell-Evans model, Dudko-Hummer- Szabo model, dynamic force spectroscopy, receptor–ligand interaction, steered molecular dynamics

Force experiments using optical tweezers, magnetic tweezers and atomic force microscopy (AFM) are widely used to probe adhesive and mechanical properties of materials at the nano and molecular scale [1]. Physicists and chemists study molecular mechanisms of surface interactions, biologists investigate processes of protein unfolding and ligand dissociation, and engineers are interested in practical aspects of nanocontacts for construction of micro- and nanodevices. AFM has become the most familiar tool applied for studying the mechanical properties and adhesion of nanometer-sized contacts. Its major advantages over competing techniques are: direct way of force measurements, simplicity of sample preparation (biological samples can be studied under near-physiological conditions), imaging capability and many others. In the force spectroscopy mode, the AFM tip approaches the sample surface and subsequently retracts while the force is measured. From the retracting part of the force-displacement curve, it is possible to detect adhesion and mechanical effects like molecular conformational changes.

Models based on Kramers' theory for bond dissociation rates [2] show that association and dissociation rates of a bond depend on the shape of the tip-sample interaction potential, from which both rates can be extracted. Thus, the interaction potential determines not only the strength of interactions but also their kinetics. The conventional way to reconstruct a tip-sample interaction potential from force-distance curves would be to integrate the force times the distance covered by the tip retracting the sample surface. Unfortunately, this approach will not allow full reconstruction of the tip-sample interaction potential due to instabilities, which occur during the contact formation between the tip and sample. Therefore another method is applied. Measuring the bond rupture force (or adhesion force) over a range of loading rates, i.e., performing the so-called dynamic force spectroscopy (DFS) measurements, and using a model of thermally activated unbinding (Bell-Evans model [3, 4] or Dudko-Hummer-Szabo model [5]), provides a way to determine such parameters as the kinetic dissociation rate constant, the position and height of the activation barrier. An example of DFS data for receptor-ligand interactions is shown in Figure 1. The idea of the models of thermally activated unbinding (treated as an escape from the potential well) facilitated with an external force is presented in Figure 2.

During my talk I will discuss various interpretations of DFS data, including those assuming the existence of an inner barrier in the interaction potential and rebinding processes [6]. I will also present the idea and some results of simulations of single molecule stretching performed with the steered molecular dynamics method. I hope that my talk will contribute to better understanding of the issue of single-molecule interactions and the interaction potential reconstruction and will prevent misinterpretations of force spectroscopy data.

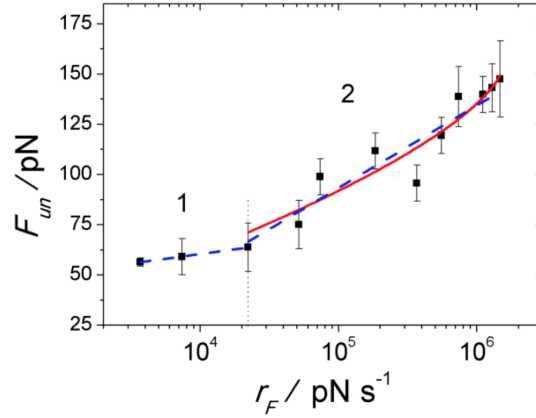


Fig. 1: DFS data (i.e. the dependence of the unbinding force on the loading rate) for the concanavalin A-carboxypeptidase Y complex showing two regions (1 and 2) for low and high loading rates, respectively. The relation was fitted with the Bell-Evans model – separately for two regions, 1 and 2 (blue dash lines) – and with the Dudko-Hummer-Szabo model applied only to region 2, i.e. for loading rates larger than $\tilde{2} \cdot 10^4$ pN/s (red solid curve).

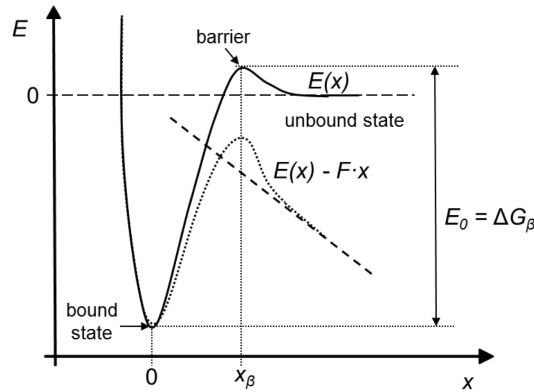


Fig. 2: Conceptual one-barrier interaction potential and its deformation induced by external force

Acknowledgments

Authors thank the Ministry of Science and Higher Education in Poland for financial support within Project No. 06/62/DSPB/2173.

References

- [1] K. C. Neuman, A. Nagy, Nat. Methods. 5 (2008) 491
- [2] H. A. Kramers, Physica 7 (1940) 284
- [3] G. I. Bell, Science 200 (1978) 618
- [4] E. Evans, K. Ritchie, Biohys. J. 72 (1997) 1541
- [5] O. K. Dudko, G. Hummer, A. Szabo, Phys. Rev. Lett. 96 (2006) 108101
- [6] A. Ptak, H. Gojzewski, M. Kappl, H.-J. Butt, J. Phys. Chem. C 114 (2010) 21572

The role of spherical voids on the deformation mechanism of Cu/Ag nanocomposites via MD simulation

ABBAS MONTAZERI^{1,*}, A. KARDANI¹

¹*Faculty of Materials Science and Engineering, K.N. Toosi University of Technology, Tehran, Iran*

*a_ montazeri@kntu.ac.ir

Recently, metal matrix nanocomposites have been widely used due to their availability and commercializing the manufacturing process. Among them, Cu-Ag nanocomposites have attracted great attention in the community of microbiology field. It has been well established that copper and silver can destroy the bacterial cell wall resulting in bacteria's death and thus, prevention from inflammation. Since this type of nanocomposites intensifies the antibacterial effect owing to the large interfacial contact area with the bacteria, it can find applications in medical devices and body implants. However, the most important conditions for proper use of materials in medical applications are to possess high yield strength and lack of presence of plastic deformation. Therefore, the deformation mechanism of these materials is important. Since the variations in the properties of these materials result from the changes in the atomic scale, molecular dynamics (MD) simulation method can be implemented to survey the phenomenon successfully. Hence, in the present research, copper matrix nanocomposites reinforced with spherical silver nanoparticle are examined using MD simulation. Accordingly, to study the deformation mechanism, the samples are studied under uniaxial tension test at the room temperature. In the next step, to capture the role of spherical voids on the deformation mechanism and thus, mechanical properties, some spherical voids are created within the bulk matrix and then, tensile tests are repeated to compare the results with the perfect sample.

The results reveal that presence of silver nanoparticles in the copper matrix causes a mismatch strain in the interface resulting from the different atomic radius of Cu and Ag. Hence, with increasing the local stresses in this zone, a significant number of edge and screw dislocations nucleated in this area. Since the perfect dislocations in a material with the grain size of bigger than 10 nm, follow partial sliding and dividing into partial dislocations, the stacking fault areas will be formed between two divided partial dislocations. These stacking fault areas can decrease the material instability during the tensile deformation. On the other hand, the presence of voids as structural defects in the material bulk can increase the stress concentrations in their wall. This phenomenon makes voids to be the preferred areas for the formation of dislocations. The results obtained from the stress-strain curve shows that the void formation in the bulk causes a significant decrease in the yield strength, elastic modulus and fracture strength of the sample. Also the presence of voids can accelerate the fracture process drastically.

Combined Optical Tweezers and AFM. The Ultimate Force Toolbox for Life Sciences

J. BUDZIOCH¹

¹*MeasLine sp. z o.o., ul. Rozrywka 20/9, 31-417 Krakow, Poland*

**mariusz.wladowski@optotom.pl*

Measurement of both techniques is based on interaction of probe and the sample and thanks to very sensitive detection system is possible to measure very small forces (starting at 0,1 pN for single molecules deforming, break of the chemical bonds, ending by tens of nano newtons for cell deformations). Optical tweezer covers to lower range of this forces, upper limit is defined by laser power and is several hundred of pN.

Quantitative characterization of the specific interactions of syndecans for cancer diagnosis with the dynamic force spectroscopy

K. HERMAN^{1,2,*} J. DANILKIEWICZ², J. PABIJAN², A. PTAK¹, M. LEKKA²

¹*Institute of Physics, Poznan University of Technology, Piotrowo 3, 60-965 Poznan, Poland*

²*Laboratory of Biophysical Microstructures, The Henryk Niewodniczanski Institute of Nuclear Physics, Polish Academy of Sciences, Radzikowskiego 152, 32-341 Cracow, Poland*

*katarzyna.p.herman@doctorate.put.poznan.pl

Keywords: syndecan, bladder cancer cell, force spectroscopy, DFSs

Receptor-ligand interactions play a crucial role in various aspects of living systems, and have been applied in medicine and biotechnology. Ligands can be molecular objects from ions to small molecules to peptides or proteins. The interaction of the receptor protein with a ligand leads to formation of various molecular complexes whose type, property, and the distribution on cell surface determine the strength of adhesion [1]. Multiple receptors present in a cell membrane mediate a great number of versatile interactions with the extracellular matrix (ECM). Syndecans are a family of transmembrane heparan sulphate proteoglycans with ectodomains modified by glycosaminoglycan chains. The ectodomains can interact with different molecules, including cytokines, proteinases, adhesion receptors, and ECM components [?]. Importantly, they can act as co-receptors of other cell surface receptors like growth factor receptors and integrins. In this context, syndecans can bind, immobilize, concentrate, and induce conformational changes in growth factors and other signaling molecules via their heparan sulfate chains, thus facilitating their receptor interaction [4, 5, 6]. In some cancers, syndecans expression may regulate tumor cell function and serve as a prognostic marker for tumor progression. Low expression of syndecan-1 in tissue is related with a bad prognosis in head and neck cancer. In turn, high syndecan-1 expression correlate with negative predictions in lung cancer [7]. The aim of this work is the quantitative characterization of the unbinding processes of syndecans (1 and 4) with suitable monoclonal antibodies, vitronectin and living cells in different stage of cancer mutation. Human bladder cancer cells originated from non-malignant epithelial cells of ureter (HCV29), malignant grade 2 (HTB9), grade 3 (HT1376) and transitional (T24) carcinoma were studied. Single molecule force spectroscopy was performed using Force Robot (JPK) and Nanowizard 4 (JPK) heads depending on a type of the sample. Dynamic force spectroscopy measurements were carried out for various values of the separation rate ranging from 0.1 to 19.0 $\mu\text{m/s}$ for syndecan-antibody and syndecan-vitronectin interactions and from 0.5 to 20.0 $\mu\text{m/s}$ for syndecan-bladder cancer cell interactions. The most probable value of adhesion force of every unbinding process was determined for each separation rate. Subsequently, Worm Like Chain model was used to identify specific binding and to obtain loading rates enabling to reconstruct profile of energy landscapes of examined complexes. The dependence of the specific adhesion force on the loading rate was obtained and analyzed with two thermal activation models: the Bell-Evans and the Dudko-Hummer-Szabo (DHS) model for quantitative characterization of the specific interactions

citeherman8. DHS model provides information about the position and the height of activation barrier ΔG . Our research show that the unbinding properties of similar molecular

complexes differ in distinct bladder cancer cell lines indicating their alterations linked with cancer progression.

Acknowledgments

Authors thank the Ministry of Science and Higher Education in Poland for financial support within Project No. UMO-2014/15/B/ST4/04737.

References

- [1] A. Woods et al.: Syndecans: Synergistic activators of cell adhesion. *Trends in Cell Biology*, 1998, 5: 189–192.
- [2] M. Bernfield et al.: Functions of cell surface heparan sulfate proteoglycans. *Annual Review of Biochemistry*, 1999, 68: 729–777.
- [3] S. Saunders et al.: Molecular Cloning of Syndecan, an Integral Membrane Proteoglycan. *The Journal of Cell Biology*, 1989, 107: 1547–1556.
- [4] M.R. Morgan et al.: Synergistic control of cell adhesion by integrins and syndecans. *Nature Reviews Molecular Cell Biology*, 2007, 12: 957–969.
- [5] J. R. Couchman et al.: Transmembrane Signaling Proteoglycans. *The Annual Review of Cell and Developmental Biology*, 2010, 26: 89–114.
- [6] A. C. Rapraeger et al.: Synstatin: a selective inhibitor of the syndecan-1-coupled *IGF1R* – $\alpha v \beta 3$ integrin complex in tumorigenesis and angiogenesis. *The FEBS Journal*, 2013, 280: 2207–2215.
- [7] Y. H. Teng et al.: Molecular functions of syndecan-1 in disease. *Matrix Biology*, 2012, 31:3–16.
- [8] A. Ptak et al.: Influence of humidity on the nanoadhesion between a hydrophobic and a hydrophilic surface. *Chemical Physics Letters*, 2011; 503: 66–70.

Electromagnetic, piezoresistive cantilever as virtual deflection reference

W. MAJSTRZYK ^{*1}, M. GRAMAŁA¹, M. RUDEK ¹,
T. GOTSZALK ¹, A. SIERAKOWSKI ², P. GRABIEC ², I. RANGELOW ³

¹ *Wroclaw University of Science and Technology, Janiszewskiego 11/17, 50-372 Wroclaw, Poland*

² *Institute of Electron Technology, Al. Lotników 32/46, 02-668 Warsaw, Poland*

³ *Ilmenau University of Technology, Gustav-Kirchhoff-Str. 1, D-98684 Ilmenau, Germany*

*wojciech.majstrzyk@pwr.edu.pl

Keywords: AFM Cantilever, Lorentz Force, Piezoresistive Bridge, Virtual Reference

Since development of atomic force microscopy, there was necessity to calibrate response of its crucial elements – the cantilever spring constant and sensitivity of the detector. Today most of the cantilevers used in AFMs are provided with the value of nominal spring constant and range, which can be even more than 100 % of the nominal value. This makes quantitative force investigations difficult. Very common solution is to perform two calibrations in AFM. First an Inverse Optical Lever Sensitivity [1] is calibrated by pushing the cantilever against hard surface and record both piezoelectric scanner extension and AFM signal. After this second thermal noise method is used to estimate cantilever spring constant. This method has the drawback that it is prone to error propagation.

A solution is to use a device called piezosensor transfer standard [2]. This solution was used to transfer an absolute force standard, the NIST electrostatic force balance (EFB) to the AFM. We have developed an virtual reference standard device by enclosing a piezoresistive cantilever with Lorentz loop into a special brass or stainless steel holder, where we added a powerful neodymium magnet (Figure 1). The device is compact and for full operation it requires only accompanying electronics. In such configuration this sensor is not only capable of purely serving as deflection sensor, but it can be also used to exert known force to the investigated cantilever. This is done by the means of the Lorentz force exerted at the end of the cantilever:

$$\vec{F}_L(t) = \vec{I} \times \vec{B} \cdot L \quad (5)$$

This gives us ability to precisely calibrate the AFM InvOLS and unknown cantilever spring constants. Moreover this sensor is calibrated to be a deflection even for sub-nanometer resolution. This is done by precise SIOS Nano Vibration Analyzer with our custom software. We have investigated cantilevers to be applied as virtual deflection references with spring constants ranging from 1 N/m up to 400 N/m.

Acknowledgments

This research has been supported by the NCN OPUS 9 Grant - “Metrology of molecular interactions using electromagnetically actuated MEMS force sensors-MetMolMEMS” (Grant No. 2015/17/B/ST7/03876).

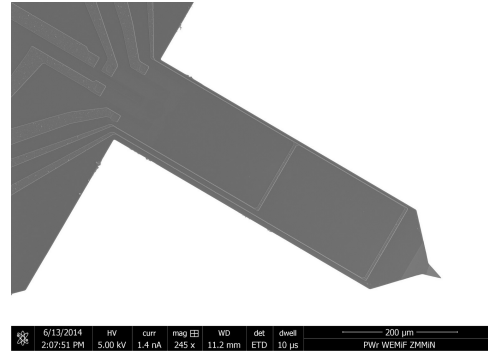
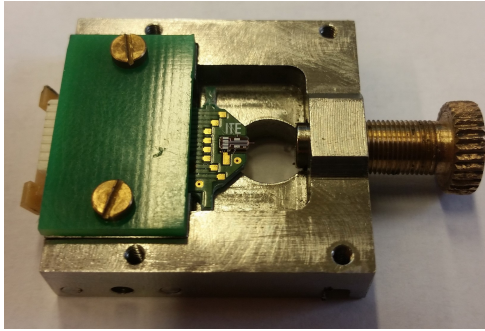


Fig. 1:) Image of the proposed electromagnetic, piezoresistive virtual reference standard within stainless steel holder and neodymium magnet on screw

Fig. 2: SEM image of the piezoresistive cantilever used as virtual reference

References

- [1] M. J. Higgins, R. Proksch, J. E. Sader, M., Polcik, S. Mc Endoo, J. P. Cleveland and S. P. Jarvis, Noninvasive determination of optical lever sensitivity in atomic force microscopy, Review of Scientific Instruments, 77 (2006), 013701
- [2] E. D. Langlois, G. A. Shaw, J. A. Kramar, J. R. Pratt, D. C. Hurley, Spring constant calibration of atomic force microscopy cantilevers with a piezosensor transfer standard, Review of Scientific Instruments 78, 093705 (2007)

SESSION 8:
6th July 2017

"SPECTROSCOPY"

- | | |
|-------------|--|
| 09:00-09:30 | [I-08] M. Targosz-Korecka <i>"The impact of inflammation and some civilization diseases on nanomechanical properties of the endothelium"</i> |
| 09:30-09:50 | [O-22] J. Zemła <i>"Interaction of bladder cancer cells with extracellular matrix proteins measured by AFM"</i> |
| 09:50-10:10 | [O-23] S. Kraszewski <i>"Molecular sharp edge, the antiseptic agents for cell membrane disruption"</i> |
| 10:10-10:30 | [O-24] J. Bobrowska <i>"Spectroscopic search for markers of cancer-related alterations"</i> |
| 10:30-10:50 | [O-25] K. Matuła <i>"Mechanoevolution of Escherichia coli bacteria under nanomechanical stress"</i> |

The impact of inflammation and some civilization diseases on nanomechanical properties of the endothelium

M. TARGOSZ-KORECKA^{1,*}

¹*Institute of Physics, Faculty of Physics, Astronomy and Applied Computer Science,
Jagiellonian University, Krakow, Poland*

*marta.targosz-korecka@uj.edu.pl

Keywords: endothelium, glycocalyx, Atomic Force Microscopy (AFM), inflammation, diabetes.

Endothelium is a tight monolayer of cells that lines the luminal side of each blood vessel [1]. Due to its location, endothelial cells are directly exposed to pro-inflammatory factors as well to variations in the biochemical parameters of blood like hyperglycemia that is accompanying diabetes. Endothelium has a crucial role in the regulation of arterial tone and blood flow. It has a unique ability to convert mechanical stress induced by blood flow into biochemical response, in particular, into the release of NO, which is the main vasodilator. This unique feature of the endothelium is strongly related to the nanomechanical properties of the endothelial cells.

In this talk, we will demonstrate that atomic force microscope (AFM) is a valuable tool for studying nanomechanical properties of the endothelium [2]. First, we will describe the general principle of using AFM as a nanoindenter for probing the Young's modulus of individual endothelial cells and for the determination of the properties of the delicate endothelial glycocalyx surface layer. By means of this method we were able to evaluate the nanomechanical changes of endothelium in both in vitro and ex vivo experiments. In particular, in vitro AFM based nanoindentation was used to reveal a two-step response of the endothelial cells to inflammatory factors (TNF, IL6, LPS) [3] or to observe the stiffening of the endothelial cells in hyperglycemia and the development of the so called stiffness memory effect [4],[5]. Recent, ex vivo study of endothelium from db/db mouse aorta (an animal model of human type 2 diabetes) enable to quantitatively evaluate the changes in glycocalyx degradation in diabetes progression.

Acknowledgments

This research was supported by 1.1.2 PO IG EU project POMOST FNP: "Elasticity parameter and strength of cell to cell interaction as a new marker of endothelial cell dysfunction in hyperglycemia/hypoglycemia"

References

- [1] Szymonski M., Targosz-Korecka M., Malek-Zietek K. Nano-mechanical model of endothelial dysfunction for AFM-based diagnostics at the cellular level. (2015) *Pharmacological Reports* 4: 728-735
- [2] Targosz-Korecka, M., Malek-Zietek, K. E., Brzezinka, G. D. and Jaglarz, M. (2016), Morphological and nanomechanical changes in mechanosensitive endothelial cells induced by colloidal AFM probes. *Scanning*; 38(6): 654–664
- [3] Targosz-Korecka M., Brzezinka G., Danilkiewicz J., Rajfur Z., Szymonski M. Glutaraldehyde fixation preserves the trend of elasticity alterations for endothelial cells exposed to TNF- α . *Cytoskeleton* (2015), 72(3);124-130.

- [4] Targosz-Korecka M, Brzezinka G, Malek K, Stępień E, Szymonski M. Stiffness memory of EA.hy926 endothelial cells in response to chronic hyperglycemia. *Cardiovascular Diabetology* 2013, 12:96
- [5] Malek-Zietek K, Targosz-Korecka M, Szymonski M, (2017) The impact of hyperglycemia on adhesion between endothelial and cancer cells revealed by single-cell force spectroscopy, *Journal of Molecular Recognition*, DOI: 10.1002/jmr.2628

Interaction of bladder cancer cells with extracellular matrix proteins measured by AFM

J. ZEMŁA¹, J. DANILKIEWICZ¹, S. SEWERYN¹,
J. PABIJAN¹, M. LEKKA^{1,*}

¹*Department of Biophysical Microstructures, Institute of Nuclear Physics Address,
Polish Academy of Sciences, PL-31342, Kraków, Poland*

*Malgorzata.Lekka@ifj.edu.pl

Keywords: mechanical and adhesive characteristics of cancer cell, atomic force microscopy, single cell force spectroscopy

The extracellular matrix (ECM) provides not only mechanical scaffold within which cells are embedded but strongly participates in various cellular processes, including morphogenesis, differentiation or migration. It is crucial for maintaining tissue homeostasis which abnormal alterations lead to various diseases including cancer. The ECM alone is not a homogenous structure but, oppositely, its heterogeneous character is defined by distinct fibrous and non-fibrous components that interact with cells through surface receptors called integrins [1]. This interaction underlies all mechanisms playing an important role in cell functioning, such as adhesion, migration, neural development, wound healing and angiogenesis, as it transfers signals from the ECM into the cell. Altered expression and functional status of integrins is also associated with such pathological processes as metastasis. Immunohistochemical studies have shown that normal human urothelium expresses integrins $\alpha3$, αV , $\beta1$, $\beta4$. Their presence in the urothelial basement membrane provides the molecular basis for the impermeability of the bladder wall. The expression of integrins in bladder cancer is aberrant and downregulated, with a progressive loss of its expression from the normal urothelium to invasive bladder cancer [2],[3].

In our studies, by using atomic force microscopy (AFM), both cellular deformability and adhesive properties of two human bladder cancer cells were quantified. Cells originated from non-malignant cell cancer of ureter (HCV29) were significantly stiffer as compared to cells derived from bladder cancer carcinoma (HTB-5). Independently of the cell type, these cells display distinct elasticity in response to ECM protein studied i.e. laminin, collagen and vitronectin. Changes were more prominent for non-malignant cells characterized by well-differentiated actin cytoskeleton while cancerous cells seem to be insensitive to surrounding ECM proteins. Alterations in mechanical properties were accompanied by changes in cellular adhesiveness as revealed through single cell force spectroscopy (SCFS) approach. Here, a single cell, either non-malignant or cancerous one, was used as a force probe to study cell's ability to adhere to surfaces coated with the corresponding ECM protein. In summary, the obtained results enabled to get insight into mechanisms of single cell adhesion – the effective adhesion force showed broad distributions indicating that, despite the presence of single effective bonds, cellular adhesion contains significant contribution originating from unbinding of clusters. It seems that the statistically relevant difference between non-malignant and carcinoma cells was observed as a number of rupture events corresponding to the number of single bonds formed.

Acknowledgments

Research at the Dept. of Biophysical Microstructures IFJ PAN is supported by National Science Centre (Poland) project no UMO-2014/15/B/ST4/04737. The JPK AFM

purchase has been realized under the project co-funded by the Malopolska Regional Operational Program Measure 5.1 Krakow Metropolitan Area as an important hub of the European Research Area for 2007-2013, project no MRPO.05.01.00-12-013/15.

References

- [1] M. Barczyk, S. Carracedo, D. Gullberg, Integrins, *Cell Tissue Res.*, 339 (2010) 269 -280. doi: 10.1007/s00441-009-0834-6.
- [2] L. Seguin, J. S. Desgrosellier, S. M. Weis, D. A. Cheresh, Integrins and cancer: regulators of cancer stemness, metastasis, and drug resistance, *Trends Cell Biol.*, 2015, 25, 234 - 240. doi: 10.1016/j.tcb.2014.12.006.
- [3] K. N. Syrigos, K. J. Harrington, M. Pignatelli, Role of Adhesion Molecules in Bladder Cancer: An Important Part of the Jigsaw, *Urology*, 1999, 53, 428–434. doi: [http://dx.doi.org/10.1016/S0090-4295\(98\)00527-5](http://dx.doi.org/10.1016/S0090-4295(98)00527-5).

Molecular sharp edge, the antiseptic agents for cell membrane disruption

D. DRABIK¹, B. HANUS-LORENZ¹, K. SZOSTAK¹,
S. KRASZEWSKI¹

¹*Department of Biomedical Engineering, Faculty of Fundamental Problems of Technology, Wrocław University of Science and Technology, Wybrzeże Wyspiańskiego 27, Wrocław, 50-370, Poland*

*sebastian.kraszewski@pwr.edu.pl

Keywords: membrane mechanics, antibacterial agents, molecular mechanical properties

Antibacterial agents are undoubtedly one of the greatest inventions of modern medicine. Due to the development in the field of effective elimination of bacteria, it was possible to greatly reduce mortality from bacterial infections. Previously abundant in everyday life and during most of the medical procedures, they are now manageable. The first insights into the inhibition of microorganisms proliferation has started with the work of Paul Ehrlich at the beginning of 20th century. Since then, the mechanism of action of many antibacterial molecules has been uncovered. Because of metabolic tracts modification or changes in molecular processes, therapies are not so effective. **The bacteria are becoming more resistant to antibiotics.** The widespread use of antibiotics in medicine and cultures led to the rapid evolution of the bacteria resistant to many antibiotics, including vancomycin - the antibiotic of last resort, which slowly ceases to be effective for some bacteria strains.[1] The growing usage of naturally occurring antimicrobial peptides,[2] flavonoids[3] or even bacteriophages[4] gives the hope for novel antibacterial treatments. **One of the most promising approaches is to establish a molecule that will change the mechanical parameters of the lipid membrane of bacteria,** leading to the profound destabilization or membrane disruption by changing their mechanical properties. It is a very advantageous solution because the cell has a little possibility of creating resistance to mechanical attack. Molecules able to act as a sharp edge cutting molecularly and selectively the bacterial membranes are hard to be developed. To create this form of attack on the cell, it is necessary to find mechanical parameters that could indicate the effectiveness of used "molecular knife" destabilizing lipid membrane of the bacteria. There are literature indications that certain particle-bilayer interaction or location of particle in bilayer after incorporation could be such a parameter.[5] There is also an original idea that lateral pressure caused by incorporation of particle into lipid bilayer could be such a parameter. This parameter is particularly important in the context of the second proposed mechanism of destabilization of the membrane. The increase in pressure within the membrane and, consequently, an increase of bilayer stress, with a consequent disruption observed on liposomes [6] or micelles[7], is, however, not describing the molecular tool, but only the phenomena on the target sample. This eliminates the possibility of massive direct comparison between different molecules, being the fundament of high throughput screening (HTS) method.

Here we propose the new virtual screening approach focusing on mechanical properties of the used molecule, derived from quantum mechanical (QM) calculations and confirmed numerically by enhanced massive docking (EMD) analysis. Such an approach may eliminate the use of molecular dynamics methods being a high-consuming in time

and in resources. Obtained results on a set of well known antiseptic agents , between them octenidine, chlorhexidine, alexidine and PVP-iodine, confirm the high antibacterial potency of gemini surfactants. We expect, that proposed procedure will simplify the characterization of mechanical properties of biologically active molecules. Not without a significance is also the fact that the staple form of the molecule structure has an impact on the speed of integration into the cell membrane. Only a detailed understanding of the molecular mechanisms governing the selectivity and the destabilization of the cell membrane will allow for the effective development of the optimized molecules with highly specialized antiseptic properties.

Acknowledgments

This work was possible thanks to financial support from National Science Centre (grant number 2015/19/B/NZ7/02380).

References

- [1] Tan, Y.-T., D.J. Tillett, and I.A. McKay, Molecular strategies for overcoming antibiotic resistance in bacteria. *Molecular Medicine Today*, 2000. 6: p. 309-314.
- [2] Bocchinfuso, G., et al., Different mechanisms of action of antimicrobial peptides: insights from fluroescent spectroscopy experiments and molecular dynamics simulations. *Journal of Peptide Science*, 2009. 15(9): p. 550-558.
- [3] Cushnie, T.P. and A.J. Lamb, Recent advances in understanding the antibacterial properties of flavanoids. *International Journal of Antimicrobial Agents*, 2011. 38(2): p. 99-107.
- [4] Fischetti, V.A., Bacteriophage lytic enzymes: novel anti-infectives. *Trends in Microbiology*, 2005. 13(10): p. 491-496.
- [5] Lopez, C.F., et al., Probing Membrane Insertion Activity of Antimicrobial Polymers via Coarse-Grain Molecular Dynamics. *Journal of Theory and Computation*, 2006. 2: p. 649-655.
- [6] Perkins, W.R., et al., Pressure Induced Fusion (Pif) Liposomes: A Solventless Sterilizing Method for Producing Large Phospholipid Vesciles. *Journal of Liposome Research*, 1995. 5(3): p. 605-626.
- [7] Okhi, S., Effects of divalent cations, temperature, osmotic pressure gradient, and vesicle curvature on phosphatidylserine vesicles fusion. *Membrane Biology*, 1984. 77(3): p. 265-275.

Spectroscopic search for markers of cancer-related alterations

J. BOBROWSKA^{1,*}, J. PABIJAN¹ J. RYSZ², K. AWSIUK², A. BUDKOWSKI², M. LEKKA¹

¹*Institute of Nuclear Physics, Polish Academy of Sciences, Radzikowskiego 152, Kraków, 31-342, Poland*

²*Institute of Physics, Jagiellonian University, Łojasiewicza 11, Kraków, 30-348, Poland*
*justyna.gostek@ifj.edu.pl

Keywords: Polyethylene, UV radiation, AFM, Mechanical Properties

Despite continuous efforts in research and outgoing advances in its diagnosis and treatment, cancer still remains one of the most deadly diseases. The development of various biochemical and biological methods increases the chance to detect cancer. In past decades, single cell biomechanics has gained large significance since certain diseases are known to manifest in alterations of mechanical properties of cells. Nowadays, one of the emerging directions is to correlate cellular biomechanics with biochemical and biophysical properties of single cancerous cells. In the presented study we have assumed that changes in the cell surface biochemical composition are correlated with alterations in their mechanical properties. It is suggested that changes in chemical composition, cell morphology and elasticity will depend on the stage of tumor progression [1]. As a model, seven melanoma cell lines derived from various stages of cancer progression were chosen and their properties were compared with the primary human melanocytes.

Atomic Force Microscopy (AFM) has become a well-established method in the research of biological materials ranging from single proteins to living cells as it allows not only for the topography measurements but it enables also the analysis of the elastic properties of various materials, including cells. By means of AFM, the nanomechanical properties of individual melanoma cells were investigated. The differences have been quantified through the Young's modulus. To study biochemical alterations at the single cell level, Time-of-Flight Secondary Ion Mass Spectrometry with its high transmission and mass resolutions was successfully employed [2]. As SIMS experiments have to be carried out in the vacuum conditions, biological samples require appropriate treatment. That is why the sample preparation protocol was developed [3]. Subsequently, high resolution mass spectra were collected for each melanoma cell line and analyzed by means of Principal Component Analysis (PCA).

Acknowledgments

The work has been financed by the NCN project no DEC-2013/11/N/ST4/01860. JB is grateful to Polish National Science Center (NCN) for the financial support of the ETIUDA scholarship no DEC-2015/16/T/ST4/00358.

References

- [1] J. Gostek, K. Awsiuk, J. Pabijan, J. Rysz, A. Budkowski, M. Lekka – „Differentiation between Single Bladder Cancer Cells Using Principal Component Analysis of Time-of-Flight Secondary Ion Mass Spectrometry”; *Analytical Chemistry* (2015) 87(6): 3195–3201
- [2] J. Bobrowska; J. Moffat; K. Awsiuk; J. Pabijan; J. Rysz; A. Budkowski; M. Reading; M. Lekka, – “Comparing surface properties of melanoma cells using time of flight secondary ions mass spectrometry”; *Analyst* (2016) 141: 6217-6225

[3] J. Bobrowska, J. Pabijan, J. Wiltowska-Zuber, B.R. Jany, F. Krok, K. Awsiuk, J. Rysz, A. Budkowski, M. Lekka – “Protocol of single cells preparation for time of flight secondary ion mass spectrometry”; *Analytical Biochemistry* (2016) 511: 52-60

[1] J. Gostek, K. Awsiuk, J. Pabijan, J. Rysz, A. Budkowski, M. Lekka – „Differentiation between Single Bladder Cancer Cells Using Principal Component Analysis of Time-of-Flight Secondary Ion Mass Spectrometry”; *Analytical Chemistry* (2015) 87(6): 3195–3201 [2] J. Bobrowska; J. Moffat; K. Awsiuk; J. Pabijan; J. Rysz; A. Budkowski; M. Reading; M. Lekka, – “Comparing surface properties of melanoma cells using time of flight secondary ions

[3] J. Bobrowska, J. Pabijan, J. Wiltowska-Zuber, B.R. Jany, F. Krok, K. Awsiuk, J. Rysz, A. Budkowski, M. Lekka – “Protocol of single cells preparation for time of flight secondary ion mass spectrometry”; *Analytical Biochemistry* (2016) 511: 52-60

Mechanoevolution of *Escherichia coli* bacteria under nanomechanical stress

K. MATUŁA^{1,*}, Ł. RICHTER¹, A. SILESIAN²,
N. DEREBECKA², J. WESOŁY², M. GRZESZKOWIAK³, B. PEPLIŃSKA³, E.
WYROBA⁴, J. PACZESNY¹, R. HOŁYST¹,

¹ *Institute of Physical Chemistry PAS, 44/52 Kasprzaka, 01-224 Warsaw, Poland*

² *Faculty of Biology, Adam Mickiewicz University, 89 Umultowska, 61-614, Poznań, Poland*

³ *Nanobiomedical Centre, Adam Mickiewicz University, 85 Umultowska, 61-614, Poznań, Poland*

⁴ *Nencki Institute of Experimental Biology PAS, 3 Pasteur Street, 02-093 Warsaw, Poland*

*kmatula@ichf.edu.pl

Keywords: nanomechanical stress, mechanoevolution, *Escherichia coli*, ZnO nanoparticles

Bacterial evolution under antibiotic pressure is currently the topic of great importance, as only recently strains resistant to all known available drugs emerged. Nanoparticles are still believed to be promising remedy. Here we unravel a physical mode of action of nanoparticles. We show that bacteria *Escherichia coli*, exposed to sharp ZnO nanorods, evolve in 72-hour experiment by developing mechanical resistance. The pressure exerted at the bacterium is from 0,3 to 100 MPa. Microscopic studies (TEM, SEM with EDS, cryo-SEM, confocal microscopy) revealed that resistant bacteria change their shape from rod-like to spherical and develop thicker and more dense cell wall (Fig.1). We identified around 30 point mutations in genome and changes in gene expression profile of bacteria exposed to nanorods.

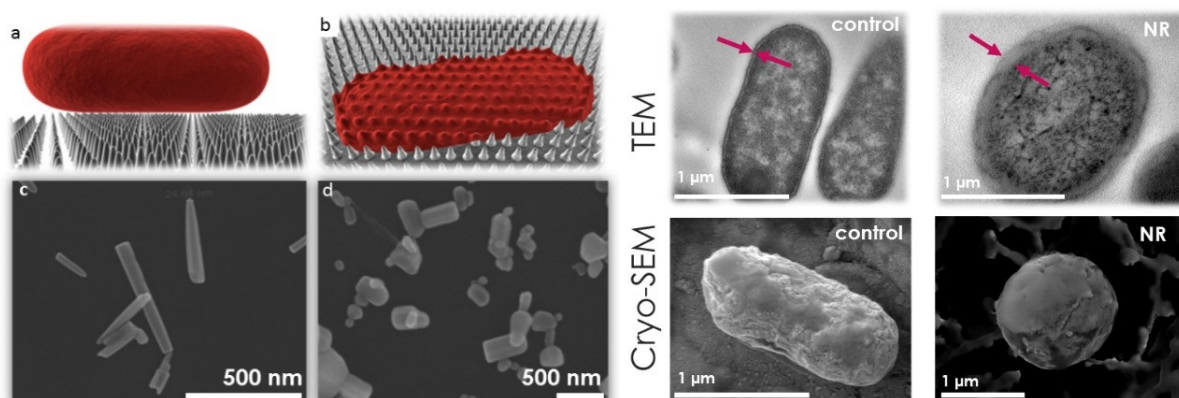


Fig. 1: **a-b)** 3D model of surface of cicada wings; **c)** sharp nanorods of ZnO , **d)** rounded ZnO nanoparticles; and transmission electron microscopy (TEM) and cryo-scanning electron microscopy (Cryo-SEM) of control cells of *E. coli* (control) and cells after exposure to ZnO nanorods (NR).

SESSION 9:
6th July 2017

"NEMS & MEMS TECHNOLOGY I"

- | | |
|-------------|--|
| 11:10-11:40 | [I-09] D. Szmigiel <i>"Submicron sensors fabricated in CMOS based process"</i> |
| 11:40-12:00 | [O-26] J. Chrzanowska <i>"Comparison of tungsten borides layers deposited by laser pulse and magnetron sputtering"</i> |
| 12:00-12:20 | [O-27] M. Hofmann <i>"Selective pattern transfer of nano-scale features generated by FE-SPL in 10 nm resist"</i> |
| 12:20-12:30 | [E-08] A. Sierakowski, K. Piekarska <i>"Electromagnetically actuated microbridges for nanometrology application"</i> |
| 12:30-12:50 | [O-28] A. Korczak <i>"Application of the interval lattice Boltzmann method for a numerical modelling of 2D thin metal films irradiated by ultrashort laser pulses"</i> |
| 12:50-13:10 | [O-29] K. Orłowska <i>"Metrology of the optomechanical interactions with MEMS/NEMS"</i> |

Design of novel type of active test sample for AFM thermal probes characterisation

**P. JANUS^{1,*}, A. SIERAKOWSKI¹, D. SZMIGIEL¹,
M. RUDEK², T. GOTSZALK²**

¹*Instytut Technologii Elektronowej al. Lotników 32/46, Warszawa, Poland*

²*Wroclaw University of Science and Technology, ul. Janiszewskiego 11/17, Wroclaw, Poland*

*janus@ite.waw.pl

Keywords: Atomic Force Microscopy, Scanning Thermal Microscopy, thermal nanoprob-
ing, thermal imaging

One of main issues related with the thermal nanometrology is the quantitative interpretation and reliable calibration of the thermal tip response [1, 2]. Moreover, the quantitative measurement of temperature at nanoscale can be performed only when the propagation of the heat from the tip to the sample and back to the cantilever is known. In this paper we present the updated design and technology of Si/Si₃N₄ micro calibration stage equipped with Pt microheaters [3]. This new design consists of 500 nm thick MEMS structure with 2 independently controlled microheaters with four point connections and improved thermal isolation. It allows for precise control of the temperature dissipation and a contact between surface and tip of cantilever. Localization on thin, low thermally conductive membrane minimizes the heat transfer to the bulk silicon. In order to increase mechanical stability of the structure, the membrane is supported by the tip. Structure stiffness is increased which allows for characterization of relatively stiff (30-70 Nm⁻¹) piezoresistive scanning thermal microscopy probes. The small size and spatial arrangement of independent heaters allows for the controlled heat flow in the membrane and measurements of the temperature distribution. Moreover, dedicated heat sinks located on the membrane decreases the heat transfer to the body of the chip. In the full paper, the manufacturing technology including MEMS surface micromachining is described. The results of electro-themo-mechanical characterization of this new stage and comparison with FEM modeling are also presented. Quantitative analysis of selected SThM probes with developed stage is discussed.

Acknowledgments

This research has been supported by the EU FP7 grant no. 318625, NANOHEAT - Multidomain platform for integrated More-than-Moore/Beyond CMOS systems characterization & diagnostics, MNiSW funding for science in the years 2013 - 2017 granted for co-financed project and ITE statutory funding.

References

- [1] L. Shi and A. Majumdar, "Thermal Transport Mechanisms at Nanoscale Point Contacts," J. Heat Transfer, vol. 124, no. 2, p. 329, 2002.
- [2] G. Hwang et al, Enabling low-noise null-point scanning thermal microscopy by the optimization of scanning thermal microscope probe through a rigorous theory of quantitative measurement, Rev. Sci. Instrum., vol. 85, no. 11, p. 114901, 2014.
- [3] P. Janus et al, Micromachined active test structure for scanning thermal microscopy probes characterization, Microelectronic Engineering, Volume 174, 25 April 2017, Pages 70–73

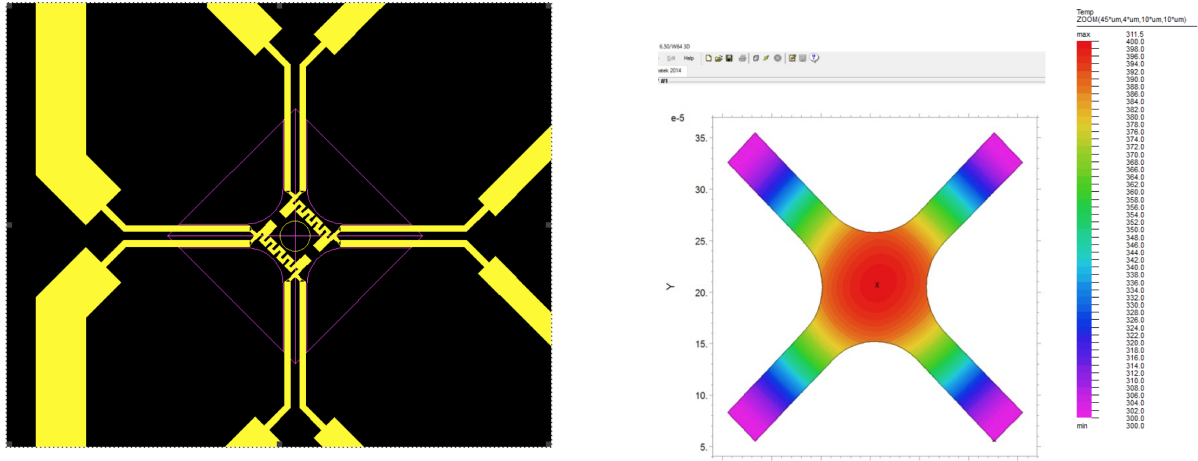


Fig. 1: New layout of the calibration structure (left) results of electrothermal FEM simulations (right)

Comparison of tungsten borides layers deposited by laser pulse and magnetron sputtering

J. CHRZANOWSKA-GIŻYŃSKA^{1,*}, J. HOFFMAN¹, T. MOŚCICKI¹

¹*Institute of Fundamental Technological Research, Pawińskiego 5B, Warszawa, 02-106, Warsaw*

*jchrzan@ippt.pan.pl

Keywords: magnetron sputtering, pulsed laser deposition, superhard materials, tungsten borides

Introduction Tungsten and boron compounds belong to the group of superhard materials and their hardness could exceed 40 GPa [1]. Moreover, these materials could be even harder when they are deposited in the form of a layer [2]. In this study the characterization of microstructure, chemical composition and mechanical properties of the tungsten borides layers deposited by laser pulse and magnetron are presented.

Materials and methods

All layers were deposited from the same target with the boron to tungsten ratio of 2.5. Properties of this target are presented in [3]. During pulsed laser deposition (PLD) the Nd:YAG laser (Quantel YG 981 E10) with a wavelength of 355 nm, repetition rate of 10 Hz and pulse duration of 10 ns was used. The spot area was 3.5 mm² and density of laser radiation (fluence) was 5 J/cm². In the magnetron sputtering (MS) process the power supplied to the magnetron cathode was 60 W and process occurred in argon pressure of $9.8 \cdot 10^{-3}$ mbar (gas flow of argon was 19.2 mL/min).

SEM and TEM images were taken by Hitachi SU-8000 and JEOL JEM-2100, respectively. XRD measurement were made with the use of Bruker D8 Discover (Cu radiation source, 1.5418 Å). Nanoindentation tests were carried out with diamond Vickers tip under the load of 10 mN. Hardness was calculated using the Oliver-Pharr method.

Results and discussion

Pulsed laser deposited layer has droplets on its surface and the droplets size varied from tens nm to several μm . The process of droplets formation was registered by ICCD camera and showed that droplets were ejected after the plasma plume had stopped interacting the target surface (about 2000 ns after laser pulse) and droplets ejection ended after 6 μs . This observation makes it easy to see that the application of the rotary shading could allow to deposit homogenous and smooth PLD layers without any debris on its surface. Further analysis of PLD layer shows that it has nanocrystalline microstructure and hexagonal WB₃ phase with cell parameters $a = 5.200 \text{ Å}$ and $c = 6.313 \text{ Å}$. Nanoindentation test shows that layer has a hardness of 47 GPa.

Layer deposited by magnetron was very smooth ($R_a = 0.002 \mu\text{m}$) and had columnar microstructure with column diameter of about 40 nm. XRD diffraction pattern of layer deposited by magnetron has only one diffraction line, positioned at $28.8^\circ 2\theta$. TEM analysis (figure 2) allow to determine phase composition and cell parameters of the layer. Magnetron sputtered layer has hexagonal α -WB structure with cell parameters $a = 3.128 \text{ Å}$ and $c = 16.559 \text{ Å}$. Nanoindentation test shows that layer has a hardness of 70 GPa under.

Summary In this work WB₃ layer was deposited by laser pulse and α -WB layer was deposited by magnetron. Both WB₃ and α -WB layers were superhard. In the examined parameters range the magnetron sputtering leaded to the deposition of layer with significantly lower boron content than layer deposited by PLD. Still, magnetron deposited layer

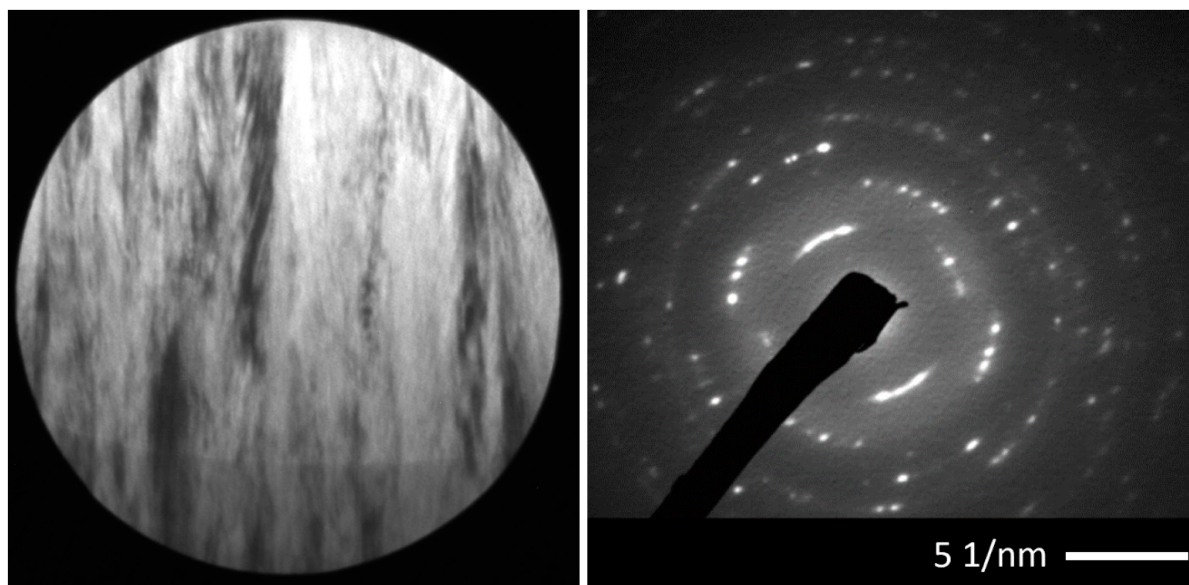


Fig. 1: Selective area electron diffraction of the layer deposited by magnetron with sputtering power of 60 W

had better mechanical properties because it is more homogenous, smooth and harder than layer deposited by laser pulse.

Acknowledgments

This work was supported by the NCN (National Science Centre) Research Project: 2015/19/N/ST8/01

References

- [1] X. -Y. Cheng, X. -Q. Chen, D. -Z. Li, Y. -Y. Li, Computational materials Discovery: the case of the W-B system, *Acta Cryst. C* C70 (2014) 85-103
- [2] J. V. Rau, A. Latini, R. Teghil, A. De Bonis, M. Fosca, R. Caminiti, V. R. Albertini, Superhard tungsten tetraboride films prepared by pulsed laser deposition method, *Appl. Mater. Interfaces* 3 (2011) 3738-3743
- [3] T. Mościcki, J. Radziejewska, J. Hoffman, J. Chrzanowska, N. Levintant- Zayonts, D. Garbiec, Z. Szymański, WB2 to WB3 phase change during reactive spark plasma sintering and pulsed laser ablation/deposition processes, *Ceram Int* 41 (7) (2015) 8273-8281

Selective pattern transfer of nano-scale features generated by FE-SPL in 10 nm resist

M. HOFMANN¹, C. AYDOGAN², B. E. VOLLAND¹, M. KAESTNER¹,
C. LENK¹, B. ERDEM ALACA³, MATHIAS HOLZ⁴,
EBERHARD MANSKE⁵, I. W. RANGELOW¹

¹*Dept. of Micro- and Nanoelectronic Systems, TU Ilmenau, Gustav-Kirchhoff-Str. 1, 98693 Ilmenau, Germany*

²*Electronics and Cryptology Department TUBITAK - YITAL, Gebze, Kocaeli, Turkey*

³*Department of Mechanical Engineering, Koc University, Rumelifeneri Yolu, 34450 Sariyer, Istanbul, Turkey*

⁴*nano analytik GmbH, Ehrenbergstraße 1, 98693 Ilmenau, Germany*

⁵*Institute of Process Measurement and Sensor Technology, TU Ilmenau, Gustav-Kirchhoff-Str. 1, 98693 Ilmenau, Germany*

*hofmann.martin@tu-ilmenau.de

Keywords: Field Emission Scanning Probe Lithography, Cryogenic Plasma Etching, Selective pattern transfer

To keep up with Moore's prophecy in future the critical dimensions of transistors must further shrink in size. In this context, we use field emission scanning probe lithography (FE-SPL) to generate nano-scaled features in 10 nm resist layers. This lithographic method is well explained in a previous publication [1]. Several groups are working on the field of selective pattern transfer of nano-scaled features. With 30 nm resist (ZEP520) selectivities of about 10 were achieved [2]. In terms of FE-SPL we require resist layers in the range of 10 nm in order to be able to generate single nano digit features. Herein, the challenge is to provide a pattern transfer of features into silicon giving practical selectivities, process control and etching depths for device manufacturing. In a cryogenic plasma etching where the sample is cooled down to so called cryogenic temperatures ($< -100^\circ$), a lateral etching is effectively suppressed due to in-situ formation of a SiO_xF_y passivation film. Since the SiO_xF_y film is not stable at room temperature. The generated passivation layer stops the etching process of silicon in both, vertical and horizontal, direction. Low energy ions generated within the plasma are accelerated towards the sample surface and remove the bottom passivation layer. The etching process through fluorine in lateral direction can resume, whilst etching in horizontal direction is still suppressed [3]. The generated patterns were transferred by plasma etching using SF_6/O_2 chemistry at cryogenic temperatures (-120°C). These patterns were analyzed by scanning electron microscopy and atomic force microscopy. Thus, we have fabricated 60 nm deep trenches in silicon. To achieve this, we spun 10 nm calixarene molecular resist on a silicon sample. Lithography was done by FE-SPL, wherein low energy electrons are applied for exposure. Positive-tone, development-less patterning leads to a direct feature formation without the requirement of further development steps. AFM and SEM images, figure 1a) and b), L-shaped test features shown the fabricated nano-features after lithography and cryogenic plasma etching. In figure 1c) the cross-section of the upper part (marked by the red line in a) is shown. As revealed, we were able to produce 60 nm deep trenches resulting in a selectivity (Silicon to resist) of about 6:1. As a result of our study a novel path towards single digit nano fabrication is demonstrated combining field emission scanning probe lithography and cryogenic plasma etching.

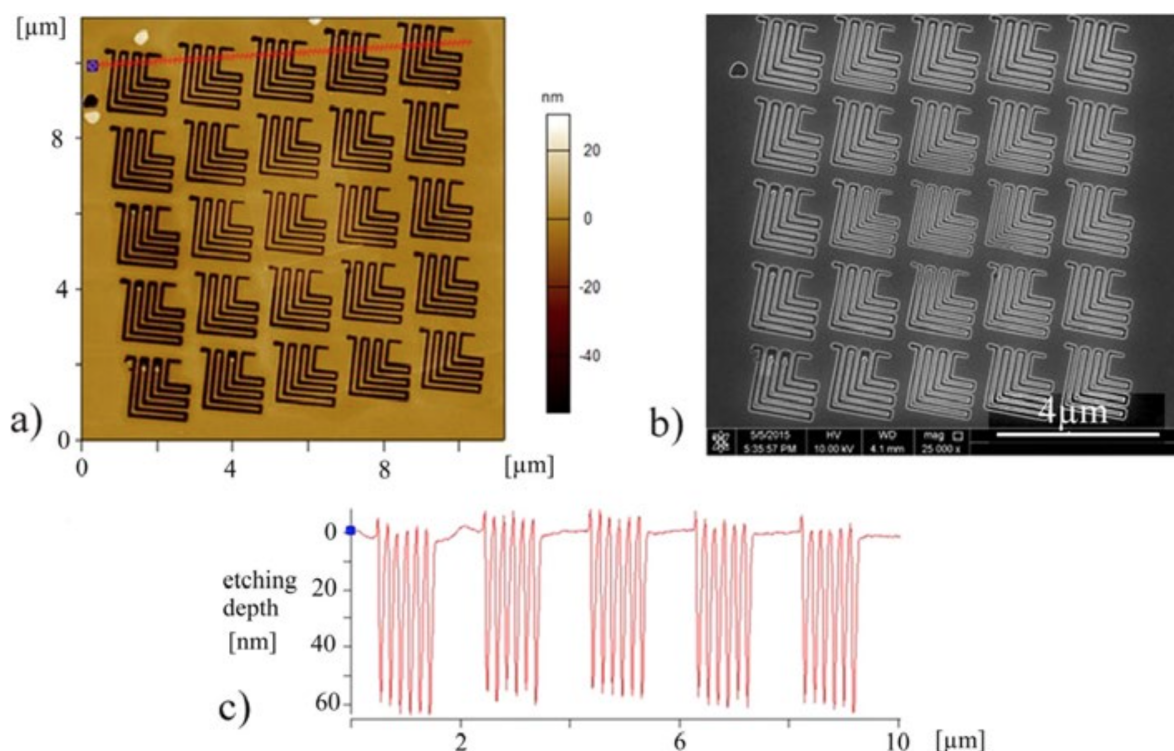


Fig. 1: (a) AFM topography image and (b) SEM images and (c) profile graph of etched in silicon (60 nm deep) test pattern written with FE-SPL in 10-nm thin calixarene molecular resist. [4]

Acknowledgments

The research leading to these results has received funding from the European Union's Seventh Framework Programme FP7/2007-2013 under Grant Agreement No. 318804 (Single Nanometer Manufacturing for beyond CMOS devices – acronym: SNM).

References

- [1] Rangelow, I.W. et. al., Proc. SPIE, 7637 (2010).
- [2] Gu, X. et. al, Advanced Materials 24 (2012).
- [3] Rangelow, I. W., Vacuum 62, 279-291 (2001).
- [4] Rangelow, I. W. et al, J. Vac. Sci. Technol. B 34, 06K202 (2016).

Use of EBL system for automatic control and characterization of micro-mechanical structures

K. PIEKARSKA¹, A. SIKORA²

¹*Prokon Piekarska Katarzyna, ul. Arniki 22, 04-903, Warszawa, Poland*

²*Institute of Electron Technology, al. Lotnikow 32/46, 02-668, Warsaw, Poland*

In presentation the professional Electron Beam Lithography solution developed by Raith company will be briefly described. Experimental results showing the utility of the simplest system (ELPHY Plus family) for automatic control and characterization of micro-mechanical structures will be clearly demonstrated.

SESSION 10:

4th July 2017

"NEMS & MEMS TECHNOLOGY II"

- 14:10-14:40 [I-10] R. Walczak *"Inkjet 3D printing for microsystems"*
- 14:40-15:00 [O-30] P. Korczyk *"Experimental and numerical studies of the effect of inertia on the distribution of flows in microfluidic junctions"*
- 15:00-15:10 [E-09] Neumann *"SPM/SEM integration and new Correlative Probe and Electron Microscopy (CPEM) tool for nanoscience"*
- 15:10-15:30 [O-31] D. Szeliga *"Multiscale model of TRIP steel deformation"*
- 15:30-15:50 [O-32] K. Kwoka *"Nanogranular piezoresistors for deflection measurements of vibrating nanobridges"*
- 15:50-16:10 CLOSING REMARKS

Inkjet 3D printing for microsystems

R. WALCZAK¹

¹*Wrocław University of Science and Technology, Faculty of Microsystem Electronics and Photonics, Janiszewski Str. 11/17, 50-372 Wrocław, Poland*

*rafal.walczak@pwr.edu.pl

Keywords: inkjet 3D printing, microsystems, lab-on-a-chip

Deliberate designing and fabrication of i3Dp microsystems require knowledge on main properties and limits of the i3Dp technique. A comparison of the properties of various models of inkjet 3D printers from the point of view of microstructure fabrication is carried out (Fig. 1(a), 1(b)). Nominal resolution and accuracy as well as the mapping quality of microchannels, deformation of cross-section shape of the microchannels, change of dimension of printed structures, quality of printed surface and ability to fabricate embedded microchannels are investigated first [1]. Designing rules that takes into account real parameters of the printers are next proposed. Important aspects are also mechanical (Fig. 1(c)) and optical properties as well as biocompatibility of the applied building material.

Conclusions from these works are that mechanical properties of printed microstructures are worse than declared by the building material manufacturer (tensile strength is significantly lower) and an "anisotropy" of these properties depending on relation between direction of printing and orientation of applied force is observed. The printed microstructures are semi-transparent-optical detection is possible through thin walls. The autofluorescence of the polymer is not observed for main fluorescence inducing wavelengths. Thus thin layers of the printed structures may be used as parts of the optical detection systems in labs-on-a-chip structures.

According to results of mentioned above research works several labs-on-a-chip were successfully fabricated and tested – spectrophotometric flow-through chip [1], electrophoretic chips (Fig. 2(a)), also as a novel modular approach, and lab-on-disc chip. Application of inkjet 3D printing is also studied from the point of view of development of specialized chip holders as an alternative technique to commonly applied precise mechanics. Knowledge of mechanical properties of the building material enabled development of check microvalve with a microflap sealing the valve seat (Fig. 2(b)) [2]. Last but not least, inkjet 3D printing is used to develop pneumatic and fluidic circuits co-working with labs-on-a-chip and MEMS-type sensors and pumps. As an example a platform for optical and mechanical characterization of oocytes/embryos is presented (Fig. 2(c)). The platform, in spite of the microfluidic chip, contains also components (fluidic/pneumatic channels, miniature pumps, pressure sensor) that enable precise control of the cell flow and pressure-driven deformation (micropipeting) of the cell.

During the presentation results of currently carried out investigations on actuation methods in 3D printed microsystems as well as functionalisation of the printed microstructures will be presented.

Acknowledgments

The works are financed by Polish National Science Centre (NCN) under frame of the

project no 2013/10/E/ST8/0342.

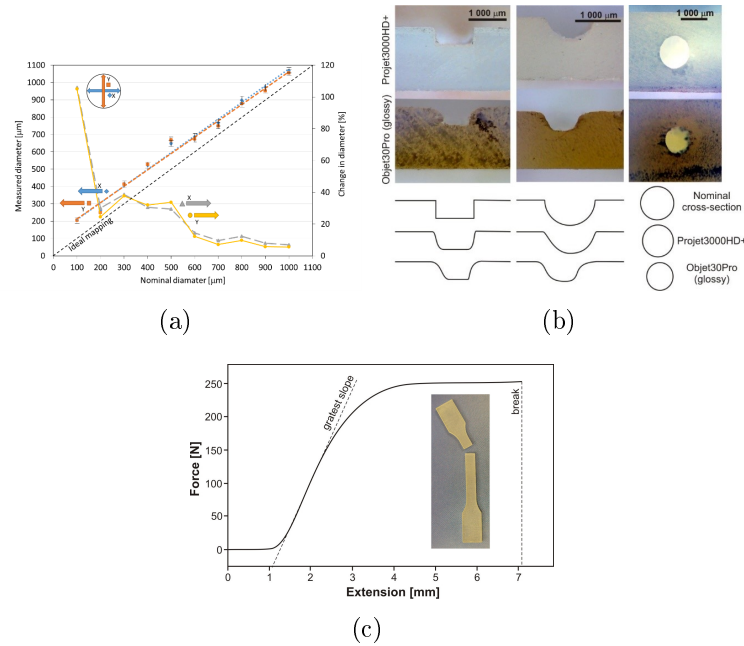


Fig. 1: Exemplary results of geometrical and mechanical characterization of the test structures printed by Projet3000HD+: a) embedded microchannels dimensions, b) optical microscope images and profiles of the cross-sections of open and embedded microchannels; minimal diameter of the embedded microchannels is $250\ \mu\text{m}$, c) example of force-extension curve for printed test structure, inside photo shows the structure after brake; tensile strength is 26 MPa (declared by the manufacturer value is 45 MPa), Young's module is 0.5 GPa.

References

- [1] R. Walczak, K. Adamski, Inkjet 3D printing of microfluidic structures – on the selection of the printer towards printing your own microfluidic chips, *J. Micromech. Microeng.*, 2015, 25, 085013
- [2] R. Walczak, K. Adamski, D. Lizanets, Inkjet 3D printed check microvalve, *J. Micromech. Microeng.*, 2017, 27, 047002

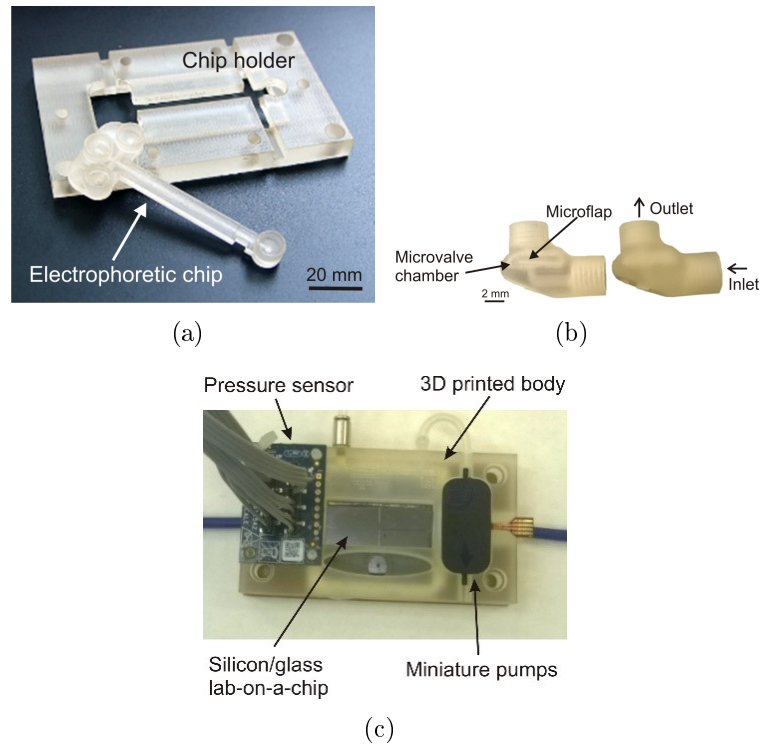


Fig. 2: Examples of microfluidic structures fabricated by us by inkjet 3D printing: a) chip and chip holder for gel electrophoresis of DNA, b) check microvalves, c) integrated platform for oocyte/embryo mechanical and optical characterization.

EXPERIMENTAL AND NUMERICAL STUDIES OF THE EFFECT OF INERTIA ON THE DISTRIBUTION OF FLOWS IN MICROFLUIDIC JUNCTIONS

P. M. KORCZYK^{1,*}, BŁOŃSKI S.¹, M. WIŚNIEWSKA¹

¹ *Institute of Fundamental Technological Research, Address, , Polish Academy of Sciences, Pawinskiego 5B, 02-106 Warsaw, Poland*

*piotr.korczyk@ippt.pan.pl

Keywords: Microfluidic, junction, inertia

The neglect of the inertia simplifies description of the flow. In the ideal non-inertial world the electric analogy [1] can be applied to the analysis and the design of even complex microfluidic structures [2], [3]. This commonly applied approach utilises the fact that stationary, viscous, laminar and incompressible flow through regular pipe satisfies the linear relation between the static pressure drop and volumetric flow rate [4] (analogous to Ohm's law). If the analogy to electric circuit is satisfied, the analytical solutions prescribing fluid flow in microfluidic networks can be derived from equivalent electric circuit equations, which typically reduce to a system of linear algebraic equations [1].

Although the flow through straight regular channel remains laminar for $Re < 2300$, the inertial effects (introducing non-linearities into equations), can appear in all non-regular elements, even for moderate Re [5]. This constitutes limitation for linear electric circuit analogy.

In order to study the limit of the linear approach, we used simple microfluidic device consisting of two identical junctions (Fig.1), which output arms form an internal rectangle. We conducted a series of experiments with devices consisting the same internal rectangle, varying the angle of inlet and outlet channel. Equal flows of a buffer and the indicator were injected into two inlets. The symmetry of the flows due to the symmetry of the device and the measurements of the concentration of the indicator in both outputs allowed us to estimate the distribution of flows in channels with high precision.

The results undoubtedly show that the ratio of flows in two arms of the junction significantly depends on Re , while according to electric circuit analogy should be constant and fixed by the aspect ratio of the rectangle. Importantly our experiments show that the flow through the junction depends strongly on angles between channels. This phenomenon cannot be described or explained by simple electric circuit analogy, which treats a junction as a point. The additional numerical simulations allowed us for efficient investigations of different geometrical configurations.

Finally, we propose the mathematical model – the extension of the electric circuit analogy which includes the effect of inertia and angles between channels. The knowledge about the role of a geometry allows for a design of the microfluidic junction, in which sum of all non-linear elements in equations vanishes and the solution recovers its linear character and independence from Re .

Acknowledgments

Project operated within the grant Sonta-bis (2014/14/E/ST8/00578) of the National Science Centre, Poland.

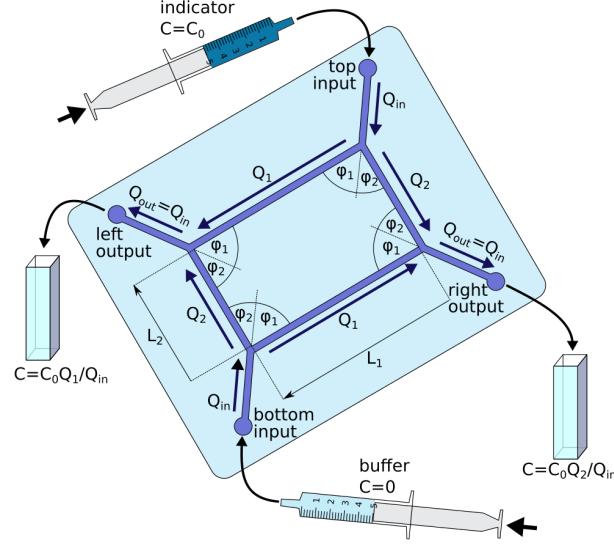


Fig. 1: The idea of the experiment. The microfluidic device consists of two identical joined junctions forming a rectangle of sides: $L_1=16.67\text{mm}$ and $L_2=10\text{mm}$ and with aspect ratio $\beta_0 = L_2/L_1 = 0.6$. The height and width of all channels are $385\text{ }\mu\text{m}$. We fabricated series of different devices keeping all geometrical parameters constant, except the angle of inlet and outlet channels ϕ_1 and ϕ_2 . Syringe pumps were used for setting the same rate of flow of water for both inputs. We used the addition of indicator to one of the input. As the distribution of the indicator on outputs depends on the flows Q_1 and Q_2 , they can be deduced from the measurements of concentration of indicator in outlets.

References

- [1] K. W. Oh, K. Lee, B. Ahn, and E. P. Furlani, "Design of pressure-driven microfluidic networks using electric circuit analogy," *Lab Chip*, vol. 12, no. 3, pp. 515–545, 2012.
- [2] F. Lin, W. Saadi, S. W. Rhee, S.-J. Wang, S. Mittal, and N. L. Jeon, "Generation of dynamic temporal and spatial concentration gradients using microfluidic devices," *Lab Chip*, vol. 4, no. 3, pp. 164–167, 2004.
- [3] B. Lin and A. Levchenko, "Spatial manipulation with microfluidics," *Front. Bioeng. Biotechnol.*, vol. 3, p. 39, 2015.
- [4] N. A. Mortensen, F. Okkels, and H. Bruus, "Reexamination of Hagen-Poiseuille flow: Shape dependence of the hydraulic resistance in microchannels," *Phys. Rev. E*, vol. 71, no. 5, p. 057301, 2005.
- [5] R. I. Zeitoun, S. M. Langelier, and R. T. Gill, "Implications of variable fluid resistance caused by start-up flow in microfluidic networks," *Microfluid Nanofluid*, vol. 16, no. 3, pp. 473–482, 2013.

SPM/SEM integration and new Correlative Probe and Electron Microscopy (CPEM) tool for nanoscience

J. NEUMAN¹,

¹*NenoVision s.r.o. Purkyňova 127, 612 00 Brno, Czech Republic*

*jan.neuman@nenovision.com

Complementarity of the SPM and SEM techniques has been proved in last decades in many different fields of applications. Novel approach of the correlative microscopy will be present utilizing new SPM instrument LiteScope directly integrated into the SEM. NenoVision introduce new tools for the nanoscience which extend the possibilities of nanos-structure characterization. Main advantages of the LiteScope combined with the electron microscope will be shown on several applications. Unique technique of Correlative Probe and Electron Microscopy (CPEM) will be presented and described with the examples of on several cases.

Multiscale model of TRIP steel deformation

K. BZOWSKI¹, D. BACHNIAK¹,
Ł. RAUCH¹, D. SZELIGA^{1,*}, M. PIETRZYK¹

¹*AGH University of Science and Technology, al. Mickiewicza 30, 30-059 Kraków,
Poland*

*szeliga@agh.edu.pl

Keywords: Multiphase materials, TRIP steel, Multiscale models, Deformation

It was observed that heterogeneous materials benefit from the best features due to the mix of phases they are made of [1]. Taking an advantage of heterogeneity is the main strengthening mechanism for modern multiphase steels, which are now developed. As a rule, increasing strength level generally results in decreasing formability. However, transformation induced plasticity (TRIP) steels show a better balance between strength and formability and they are of particular interest for the automotive industry [2]. The excellent mechanical properties of TRIP steels depend on both the multi-phase microstructures and the martensitic transformation of the retained austenite.

A detailed description of the microstructure features of the TRIP steels is required to investigate the correlation between the complex multiphase microstructure and the exploitation properties. A hypothesis was made that description of the heterogeneous microstructure of multiphase steels using RVE with implemented various microstructure features, will allow to build the model with a capability to predict realistically the final TRIP steel properties. Fig. 1 shows schematic illustration of the TRIP microstructure and microstructure from simulation of bainitic transformation (b). The main objective of the paper was formulated to prove this hypothesis. The initial microstructure of the TRIP steels was developed using two approaches. The first was drawing schematic microstructure on the basis of micrographs. The second was obtained using simulations of bainitic transformations using carbon diffusion based model, see [3] for the details of the model.

This paper describes a multiscale simulation method, factoring into a microstructure of processed steel and dynamic structure accounting for phase transformations (TRIP-effect). To optimize the required calculation and computing resources, a SSRVE (Statistically Similar Representative Volume Element) concept [4] was applied. TRIP 700 steel was reproduced in the SSRVE, which was subjected to different deformation schemes. Strain induced transformation of the retained austenite into martensite was simulated. The following experimentally found mathematical relation [2] was selected to reproduce the TRIP-effect:

$$\nu_{RA} = \nu_{RA0} - 26.708\epsilon - 0.003\dot{\epsilon} + 53.516\epsilon^2 - 0.018\epsilon\dot{\epsilon} - 1.06 \times 10^{-5}\dot{\epsilon}^2 \quad (6)$$

where: ν_{RA0} - initial volume of the retained austenite, ϵ , $\dot{\epsilon}$ - strain and strain rate, respectively.

Results of numerical tests are shown in Fig. 2. Initial SSRVE in Fig. 2(a) was subjected to the deformation of 10%. Properties of phases were taken from the literature. Transformation of the retained austenite into martensite was calculated on the basis of equation (1). Distribution of stresses in the SSRVE at the end of deformation is shown

in Fig. 2(b).

After these numerical tests The SSRVE was attached to the FE code, which simulates cold forging process. Simulations were performed and changes of the volume fraction of the retained austenite in various points of the forging were calculated.

Acknowledgments

The paper within NCN project no. NCN 2015/19/B/ST8/01064.

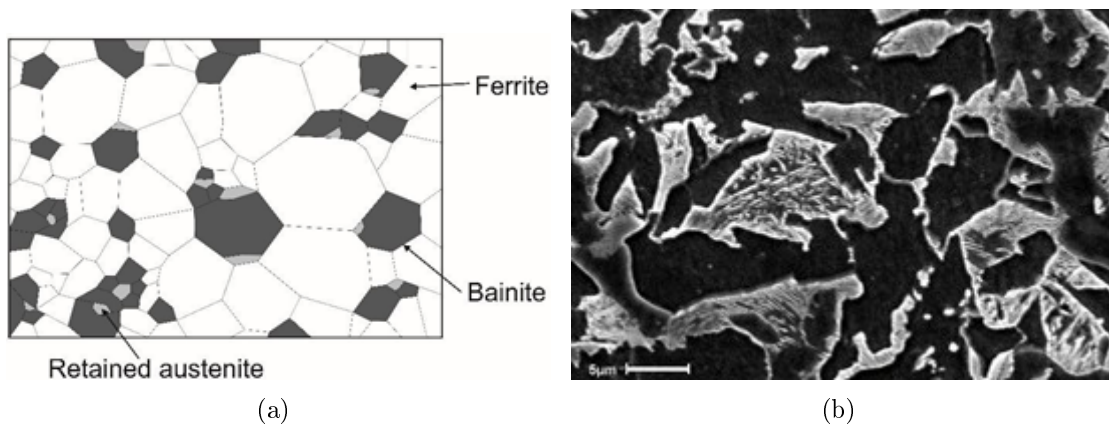


Fig. 1: a) Schematic illustration of the TRIP microstructure (a) and microstructure observed using SEM (b).

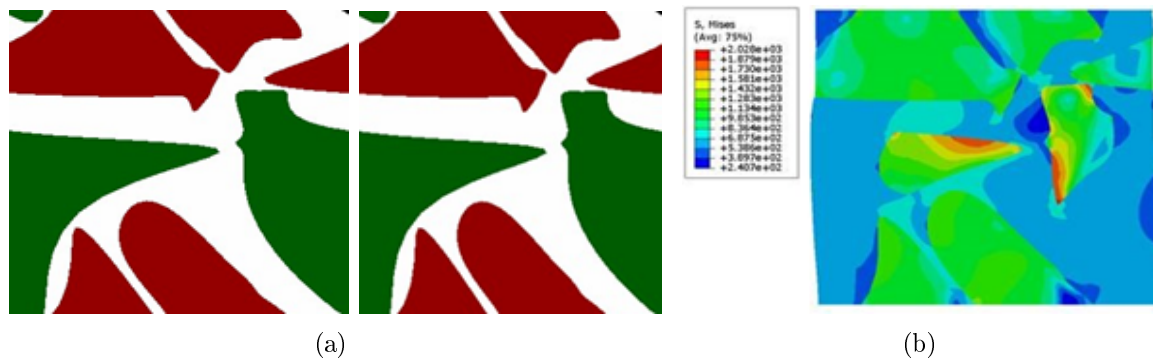


Fig. 2: a) Initial SSRVE – ferrite (white), retained austenite (green), bainite (red), a) Stress distribution in the SSRVE after deformation of 10%.

References

- [1] F.Conde-Rodriguez, J.-C. Torres, A.-L. Garcia-Fernandez, F.-R. Feito-Higueruela, A comprehensive framework for modeling heterogeneous objects, Vis. Comput. 33 (2017) 17-31.
- [2] Grässel O, Krüger L, Frommeyer G, Meyer LW. High strength Fe-Mn-(Al,Si) TRIP/TWIP steels development – properties –application. Int. J. Plast. 16 (2000) 1391–1409.

- [3] Pernach M., Bzowski K., Pietrzyk M., Numerical modelling of phase transformation in DP steel after hot rolling and laminar cooling, *Int. J. Multiscale Comput. Eng.*, 12 (2014) 397–410.
- [4] Rauch Ł., Pernach M., Bzowski K., Pietrzyk M., On application of shape coefficients to creation of the statistically similar representative element of DP steels, *Comp. Meth. Mater. Sci.*, 11, 2011, 531-541.

Nanogranular piezoresistors for deflection measurements of vibrating nanobridges

K. KWOKA^{1,*}, P. KUNICKI¹, I. UTKE²,
T. PIASECKI¹, M. MOCZAŁA¹, T. GOTSZALK¹

¹*Faculty of Microsystem electronics and photonics, Wrocław University of Technology,
Janiszewskiego 11/17, Wrocław, 50-372, Poland*

²*EMPA, Swiss Federal Laboratories for Materials Science and Technology,
Feuerwerkerstr. 39, CH-3602 Thun, Switzerland*

*krzysztof.kwoka@pwr.edu.pl

Keywords: Resonance, FEBID, NEMS

Doubly clamped beams with length lower than 300 μm , width lower than 50 μm and mass on the order of 10 pg were used. To obtain nanobridges Si substrates with Si_3N_4 top layer were etched using RIE (reactive-ion etching) and KOH etching. Additionally modification with focused ion beam (FIB) was performed. Resonance frequencies for such structures were measured using laser vibrometer at over 500 kHz with deflection magnitude on the order of nanometers in the air[1].

Nanogranular Co-C piezoresistors were deposited using focused electron beam induced deposition (FEBID)[1]. Using this technique high control over deposition process and formation of sensing structures on the nanometer scale and in precisely chosen place is possible. Resistors made of nanogranular composites typically exhibit tunneling or variable-range hopping (VRH) conductivity and obtaining high gauge factor (GF) may be possible[2].

During research different external actuation methods were tested, such as piezostacks and piezopills. To determine the best actuator for high frequencies measurements with SIOS SP-S 120 vibrometer were performed. With necessity of sustaining vibration amplitude at over 500 kHz frequency 20 mm ceramic piezopill was chosen as most efficient actuator. For further calculations of sensing properties of the structures vibrations characteristics were measured using laser vibrometer. Electrical measurements of resistance changes of piezoresistors deposited on the edges of nanobridges were performed at resonance frequencies of vibrations and gauge factor around 4 was calculated.

References

- [1] Moczała, M., et al. "Fabrication and characterization of micromechanical bridges with strain sensors deposited using focused electron beam induced technology." *Microelectronic Engineering* 176 (2017): 111-115.
- [2] Huth, M., et al. "Focused electron beam induced deposition: A perspective." *Beilstein journal of nanotechnology* 3.1 (2012): 597-619.

POSTER SESSION:

5th July 2017

15:20 - 16:20

Tunneling vibrometry of MEMS/NEMS devices

**M. BABI^{1,*}, K. GAJEWSKI¹, P. KUNICKI¹,
T. GOTSZALK¹, P. JANUS², A. SIERAKOWSKI² P. GRABIEC²**

¹*Faculty of Microsystem Electronics and Photonics Nanometrology Division, ul. Janiszewskiego 11/17, Wrocław University of Technology, Wrocław 50-372, Poland*

²*Electron Technology Institute, Warsaw, Poland*

*michal.babij@pwr.edu.pl

Vibrometry is a well-known technique for the vibrations measurement of the Micro- and Nano-Electro-Mechanical Systems (MEMS/NEMS). This technique allows to determine the parameters of the vibrating structures, like amplitude and resonance frequency of the vibrations. Nowadays, high mass and force measurement resolution is needed, so more often the Micro and Nano-Electro-Mechanical Systems (MEMS/NEMS) are applicable in laboratory investigations. Development of microelectronic technology and progressive miniaturization of NEMS structures, allows to manufacture sensors, that are capable to measure mass and force in femtograms and femtonewtons range. Despite many advantages of NEMS structures, the NEMS sensors measurements are very troublesome, because of their small dimensions, high resonance frequency (in the range of MHz) and small deflection amplitude (picometer range). It is therefore necessary to apply specially metrology equipment, so this kind of measurements are performed only in a few laboratories in the Europe. The main aim of our investigations is metrology of NEMS devices using Scanning Tunneling Microscopy (STM) methods and techniques. Because of high sensitivity of distance measurements, the tunneling junction will allow to measure such small amplitudes of vibrations. Additionally our STM system will be integrated into Scanning Electron Microscope (SEM). This integration will allow observations of NEMS structures and allow to precise positioning of the STM tip with respect to NEMS devices. In our work we will present the newest vibrometry measurement results using STM. In our investigations we are using custom-made scanning tunneling microscope including scanning head and modular STM controller made by the Nanometrology group at Wrocław University of Science and Technology.

Devices to detect oscillations induced by living organisms

W. CHOMICKI¹, P. STUPAR¹,
R. FOSCHIA¹, S. KASAS², G. DIETLER³

¹*Laboratoire de Physique de la Matière Vivante (LPMV), Ecole Polytechnique Fédérale de Lausanne (EPFL), Lausanne, CH-1015, Switzerland*

Recently our team demonstrated that living organisms attached to an Atomic Force Microscope cantilevers induce oscillations that reflect their viability and their metabolism [1],[2],[3],[4]. Our team demonstrated a possibility that homemade device can fulfill all the requirements to capture oscillations induced by living organisms. The device operates with noise level similar or better than commercially available AFM, accommodates traditional AFM cantilevers and is extremely simple to operate. In addition the price of the instrument is only a fraction of a traditional AFM. We are seeking to implement similar devices in hospitals and research laboratories for rapid drug screening. There is a need for a device that can be operated remotely in closed spaces, hence the development of a motorized version of the device is desirable. This hardware can be easily upgraded with software for automated alignment or drift correction. In this presentation recent progress will be shown.

Acknowledgments

This work has been supported by Gebert Rüf Foundation Project GRS-024/14 and Swiss National Science Foundation Grant Nr. 200021_144321.

References

- [1] G. Longo et al., Nature Nanotechnology 8, 522–526 (2013), doi: 10.1038/nnano.2013.120
- [2] S. Kasas et al., PNAS 112, 378-381 (2015); doi: 10.1073/pnas.1415348112
- [3] P. Stupar et al., Mechanical Sciences 8, 23-28 (2017), doi: 10.5194/ms-8-23-2017
- [4] P. Stupar et al. Clinical Microbiology and Infection 23 (6), 400-405 (2017), doi: 10.1016/j.cmi.2016.12.028

Ł. CZYKIEL¹, M. GRAMALA¹,
M. JAKUBIAK¹, T. GOTSZALK¹

¹*Nanometrology Division, Faculty of Microsystem Electronics and Photonics, Wrocław
University of Science and Technology, ul. Janiszewskiego 11/17, 50-372 Wrocław*

Keywords: Nanometrology, Noise detection, Low frequency

The purpose of the project is to measure the noise of the piezoresistive levers in the low-frequency range. This is important from the point of view of metrology since the knowledge of the noise is significant for interpretation of results. In the case of micro piezoresistive levers, the fluctuations generated in the bridge structure affect the resolution of the lever deflection, and thus the force acting on the lever. The detector is designed to measure low-frequency noise, i.e. the so-called 1/f which occurs during static operation of the lever. The occurrence of these noises on account of to the fact that in each resistor, whose temperature is higher than absolute zero occurs electron movement, resulting in a non-zero, voltage at the terminals of the resistor.

Mechanical deformations of POPC lipid vesicle studied using optical tweezers

D. DRABIK^{*1}, S. DROBCZYŃSKI¹,
S. KRASZEWSKI¹

¹*Department of Biomedical Engineering, Faculty of Fundamental Problems of Technology, Wrocław University of Science and Technology, Wybrzeże Wyspiańskiego 27, Wrocław, 50-370, Poland*

^{*}Dominik.Drabik@pwr.edu.pl

Keywords: membrane mechanics, optical tweezers, POPC, force-extension curve

Biological membrane is the crucial structural element of all living systems. The membrane consists of its building blocks, which are mainly lipids and proteins. This complex structure exhibits variety of behaviors driven by chemical, electrical and/or mechanical phenomena. In order to quantitatively investigate biophysical processes taking place, the artificial model membranes – lipid bilayer – are created composed of only one kind or a few different kinds of lipids. The physicochemical properties of such obtained lipid bilayers depends on molecular composition and their thermodynamic state.

As it was mentioned initially, variety of phenomena, especially the mechanical ones, take place within the lipid bilayers. This mechanical aspect is widely under investigation since it is believed it plays critical role in whole cell functioning. Determination of mechanical properties requires for instance *infinitely thin layer approximation*, due to the parameter being a macroscopic property. Based on Helfrich model [1] mechanically driven deformations of lipid bilayer could help to designate these properties, and, according to the theory, can be divided into three main types: dilation, shear and bending. Dilation deformations occur only as an result of external pressure. Shear deformations depends mainly on composition of lipid bilayer and interactions between lipids and other particles within the bilayer. They are negligible in case of lipids in temperature above their transition phase temperature, otherwise become an huge experimental and computational issue. Finally, bending deformations are driven by energy dissipation of water molecules and are supposed to determine the shape and behavior of vesicle in equilibrium state. Parameters describing mechanical properties are of very small value ranging from 10^{-22} to 10^{-19} J, which makes their measurements challenging and sophisticated. One of the latest approaches to determine mechanical properties of lipid bilayer is to measure external force when deforming bilayer shape. For this purpose silica beads are encapsulated inside liposomes, which are used to stretch bilayer locally with optical tweezers system. It allows to calculate the shape deformations in response to external stretching [2]. Those shape deformations can be then recalculated to external forces on lipid bilayer values.

In our study POPC Giant Unilamellar Vesicles (GUVs) were used to model lipid membrane. Modified electroformation procedure was used to obtain the samples. Specifically, 15 μ l of 1 mg/ml POPC and 3 μ m diameter silica beads (in ratio 15:2) dissolved in methanol was deposited onto platinum electrodes. The organic solvent was evaporated under reduced pressure for 1 h. Next, the electrodes were immersed in 150 mOsm sucrose solution and exposed to the AC 1 Hz electrical field for 4 h in a polytetrafluoroethylene electroformation chamber. Finally 150 mOsm glucose solution was added to induce vesicle descend. The samples were moved to observation chamber and investigated using galvanic

mirrors optical tweezers with stiffness $k_n = (2 \cdot 10^{-5})$ N/m. Samples were illuminated with Nd:YAG 1064 nm 4W laser and visualized on 100x NA 1.3 objective with oil immersion with High Speed CMOS camera Mikrottron MC1362. Obtained results provide a valuable insight into mechanical deformations induced by external force generated at macroscopic level.

Acknowledgments

This work was possible thanks to financial support from National Science Centre (grant number 2016/21/N/NZ1/02767).

References

- [1] W. Helfrich, Elastic Properties of Lipid Bilayers: Theory and Possible Experiments, Z. Naturforsch. 28c (1973), 693-703
- [2] Y. Shitamichi, M. Ichikawa, Y. Kimura, Mechanical properties of a giant liposome studied using optical tweezers, Chemical Physics Letters, 479 (2009), 274-278 doi:10.1016/j.cplett.2009.08.018

The microscale study of mechanical properties degradation of various polyethylene composites exposed to UV radiation

M. BŁASZCZYK¹, M. DURKO¹, Z. IWANICKA¹,
A. LICHOCKI¹, M. NOWICKI², A. SIKORA³

¹*Active Students Association, SEP Branch no. 1 in Wrocław, M. Skłodowskiej-Curie 55/61, Wrocław, 50-369, Poland*

²*Faculty of Technical Physics, Poznań University of Technology, Piotrowo 3, 60-965 Poznań, Poland*

³*Division of Electrotechnology and Materials Science, Electrotechnical Institute, M. Skłodowskiej-Curie 55/61, Wrocław, 50-369, Poland*

*sikora@iel.wroc.pl

Keywords: Polyethylene, UV radiation, AFM, Mechanical Properties

Polymers are the materials thought to be "materials of the future" due to their unique properties. One of the most commonly used polymers is polyethylene which belongs to the polyolefin class. This material is applied in many different areas, both in science and in housework [1], [2]. Ability to use a certain polymer is determined by its stability, including resistance to UV light [3] - [5].

In this work the investigation of UV caused degradation of mechanical properties of various PE composites will be presented. In order to observe said process, polyethylene samples were exposed to UV radiation for a specified time. Changes of the surface of material in a submicrometer scale were observed by force spectroscopy using atomic force microscope (AFM). Nanoscratching and nanoindentation were also performed. Results acquired at nanoscale were compared with data obtained in standardized tensile strength measurements. Achieved values made it possible to determine the validity of the use of polyethylene in conditions with exposure to damaging radiation [6] - [7]. Studies performed at nanoscale enabled the materials characterization with greater sensitivity than those at macroscale, in particular at early stages of degradation. In addition, the impact of the fillers on the results repeatability will be discussed.

Acknowledgments

We thank Electrotechnical Institute for the opportunity to perform research needed for the purposes of this paper. The authors also thank Association of Polish Electrical Engineers SEP, section in Wrocław and branch no. 1 in Wrocław for financial support.

References

- [1] J. F. Rabek, Współczesna wiedza o polimerach (WN PWN, Warszawa, 2008)
- [2] F. Khabbaz, A.C. Albertsson, S. Karlsson, Chemical and morphological changes of environmentally degradable polyethylene films exposed to thermo-oxidation, *Polymer Degradation and Stability* 63 (1999) 127-138
- [3] Degradacja tworzyw polimerowych, Open Access Library Vol. 2 (2011)
- [4] S. Balasubramanian, M. Sundaram, K. Murugasamy, Ch. Angannan, Mechanical and surface properties of low-density polyethylene film modified by photo-oxidation, *Polymer Journal* 43 (2011) 398-406
- [5] F.S. Qureshi, M.B. Amin, A.G. Maadhah, S.H. Hamid, Weather induced degradation of linear low density polyethylene: mechanical properties. *J. Polym. Eng.* 9 (1990) 67-84

- [6] M. Hakkarainen, A.Ch. Albertsson, Environmental Degradation of Polyethylene: Long Term Properties of Polyolefins, Volume 169 of the series Advances in Polymer Science, 177200
- [7] J. Buckley, D. Druminski, A. Halliday, A, Lewis, Mechanical Properties and Degradation of Commercial Biodegradable Plastic Bags, Report for a major qualifying project, Faculty of Worcester Polytechnic Institute In partial fulfillment of the requirements for the Degree of Bachelor of Science (2011)

Fluorescent nanodiamond slurries designed for photonic devices

M. FICEK^{*1}, M. MRÓZEK², M. GŁOWACKI¹,
M. GARDAS¹, W. GAWLIK², R. BOGDANOWICZ¹

¹*Faculty of Electronics, Telecommunications and Informatics, Gdańsk University of Technology, 11/12 Narutowicza St., 80-233 Gdansk, Poland*

²*Institute of Physics, Jagiellonian University, Reymonta 4, 30-059 Kraków*

*matficek@pg.edu.pl

Keywords: detonation nanodiamond, color centers, fluorescence, photonic devices, optical fibres

The color center may couple an electron to become negatively charged (SiV– or NV–) with spin $S=1$. The nonzero spin is responsible for paramagnetic properties of the center. The vacancies may be created in diamond powders or in a bulk CVD or HTHP diamond. Due to photo-conversion between the charged and neutral forms of the center and its light-power dependence, the NV centers are very attractive for applications in various quantum devices. However, there is a serious problem of their coupling with photonic cavities or other devices. These issues are investigated by research teams demonstrated efficient coupling between the NV centers in open Fabry-Perot cavities at 77 K [1]. Recently, the Melbourne team demonstrated coupling of NV spherical micro-resonator of a high Q-factor with nanodiamond integrated in tellurite glass [2]. The main objectives of this study is development of new technological solutions for the creation of photonic structures and quantum devices based on color centers in diamond and to develop novel dedicated spectroscopic methods for studying their optical and microwave properties. Moreover, we intend to investigate the application perspectives of the achieved results and technological potential for development of novel sensors based on nonlinear and quantum optics (biosensors/biomarkers, quantum information processing and quantum cryptography). The proposed method relies on synthesis of diamond with integrated color centers and/or synthesis of photonic fibers with NV centers and has not been used by any other team. It will be developed in two directions: (i) filling the PCF fiber with DND-NV suspensions and (ii) synthesis of PCF fiber precoated with DND-NV suspensions (empty PCF and NVs in the fiber walls) [?]. The sample characterization will be based on optical (absorption, emission, Raman) and microwave spectroscopy with high spatial (confocal, $\approx \mu\text{m}^3$) and spectral (sub-Hz) resolution. The measurements will be performed using a technique of optical detection of magnetic resonance (ODMR) improved by development of the microwave hole burning technique [4]. The development and applications of quantum technologies and nonlinear spectroscopy for studies of color centers in diamonds is very fast. Thanks to the stable crystallographic and electron structure of diamond, NV-color centers exhibit very stable electronic spectra, not much sensitive to various perturbations. Their excellent optical and spin properties (paramagnetism) allow one to use different resonance and spintronic methods of and enable precision metrology.

Acknowledgments

This work was supported by the Polish National Science Centre (NCN) under the Grants No. 2014/14/M/ST5/00715, 2016/21/B/ST7/01430 and 2015/17/D/ST5/02571. The DS funds of Faculty of Electronics, Telecommunications and Informatics of the Gdansk University of Technology are also acknowledged.

References

- [1] R.N. Patel, T. Schröder, N. Wan, L. Li, S.L. Mouradian, E.H. Chen, D.R. Englund, Efficient photon coupling from a diamond nitrogen vacancy center by integration with silica fiber, *Light Sci. Appl.* 5 (2016) 11486.
- [2] [1]. Y. Ruan, H. Ji, B.C. Johnson, T. Ohshima, A.D. Greentree, B.C. Gibson, T.M. Monro, H. Ebendorff-Heidepriem, Nanodiamond in tellurite glass Part II: practical nanodiamond-doped fibers, *Opt. Mater. Express.* 5 (2015) 73.
- [3] R. Bogdanowicz, M. Śmietana, M. Gnyba, M. Ficek, V. Straňák, Ł. Goluński, M. Sobaszek, J. Ryl, Nucleation and growth of CVD diamond on fused silica optical fibres with titanium dioxide interlayer, *Phys. Status Solidi A.* 210 (2013) 1991–1997.
- [4] M. Mrozek, A. Wojciechowski, D.S. Rudnicki, J. Zachorowski, P. Kehayias, D. Budker, W. Gawlik, Coherent population oscillations with nitrogen-vacancy color centers in diamond, *ArXiv151203996 Phys.* (2015). <http://arxiv.org/abs/1512.03996>

FE-SPL tool for 150 mm wafer

M. HOLZ^{*1}, E. GULIYEV¹ A. AHMAD² M. HOFMANN²
M. KAESTNER², A. REUM¹ C. LENK^{1,2} V. ISHCHUK²
N. NIKOLOV³ I. W. RANGELOW¹

¹*nano analytik GmbH, Ehrenbergstraße 1, 98693 Ilmenau, Germany*

²*Dept. of Micro- and Nanoelectronic Systems, TU Ilmenau, Gustav-Kirchhoff-Str. 1,
98693 Ilmenau, Germany*

³*Mikrosistemi LTD, Bul. Vasil Levski 8, Computer Centre, 9010 Varna, Bulgaria*

^{*}sm.holz@nanoanalytik.net

Keywords: Scanning Probe Lithography, Active Cantilever, Nanofabrication

To keep up with Moore's prophecy in future the critical dimensions of transistors must be further scaled down in size. In this context, we developed field emission scanning probe lithography (FE-SPL) to generate single digit nano-features in thin ($>20\text{nm}$) molecular resist layers. This lithographic method was described in previous publication [1]. Here, we are presenting the completion of a FE-SPL-technology platform for 150 mm wafers. In particular, the tool, shown in figure 1, is finished and tested. The system is able to carry out step-and-repeat lithography on 150 mm wafer. Therefore, a 6" X, Y, Theta ($150\times150\text{ mm}^2$ positioning range + 360° rotation for alignment) stage is integrated as bottom positioning stage. The integrated coarse positioning unit ensures a resolution of $<10\text{ nm}$ (X, Y) $|<1.3\text{ }\mu\text{rad}$ (Theta) and a repeatability of 100 nm (X, Y) $|<13\text{ }\mu\text{rad}$ (Theta), respectively. The mechanical setup is designed as an arch type bridge. A natural stone is used as material for mechanical beam in order to minimize the influence of parasitic effects such as thermal drift, mechanical vibration and acoustic noise. The XYZ piezoscanning unit is mounted onto the top unit, which also integrates the approach unit. The piezoscanning unit delivers a scanning/ patterning field of $200\text{ }\mu\text{m} \times 200\text{ }\mu\text{m}^2$, and in Z direction of $25\text{ }\mu\text{m}$. The XYZ position noise of the nano-positioning stage is 0.5 nm for X and Y axis and 0.1 nm for Z axis. To reduce the processing time in step and repeat FE-SPL mode we developed a new approach technique. In particular, the fast approach unit is based on a commercial linear motor with 50 nm minimum incremental motions (repeatability: 100 nm ; backlash: $2\text{ }\mu\text{m}$). In order to increase stability of the approach and to minimize the tilting of the cantilever head, the linear approach motor is supported by linear ball bearings units. The total travel range of the fast tip approach mechanisms is 25 mm . The FE-SPL technology platform is constructed in a modular way. In such a manner the scanning head can be exchanged allowing single cantilever or cantilever array operation. In terms of AFM and lithographic application special active cantilever are applied [1, 2]. For coarse navigation an optical microscope system is implemented delivering a resolution better than $5\text{ }\mu\text{m}$. The previously developed electronic units, 125 Hz FPGA based controller as well as AFM, SPL and pattern generator software were integrated. A full step-and-repeat lithographic operation capability is implemented. Different nano-devices patterned by the FE- SPL tool are shown in figure 2. A dedicated housing of the system is designed and built in order to enable an encapsulated operation minimizing the environmental sources of drift, mechanical noise and instabilities. The tool-specifications are summarized in table 1.

Acknowledgments

The research leading to these results has received funding from the European Union's

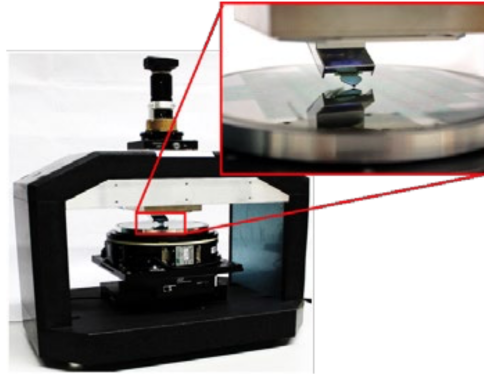


Fig. 1: FE-SPL tool for 150 mm wafers

FE-SPL tool specification:

- Suited for 150 mm wafer
- 6" x, y, Theta stage
- Step & repeat capability
- 8nm stage resolution (S&R)
- Fast cantilever scanner (X-Y-Z):
Range: 200 μ m x 200 μ m x 20 μ m
Pos. noise: 0.5/0.5/0.1 nm
- Granite beam ensures stiffness
- Encapsulated system by housing

Tab. 2: FE-SPL tool specifications

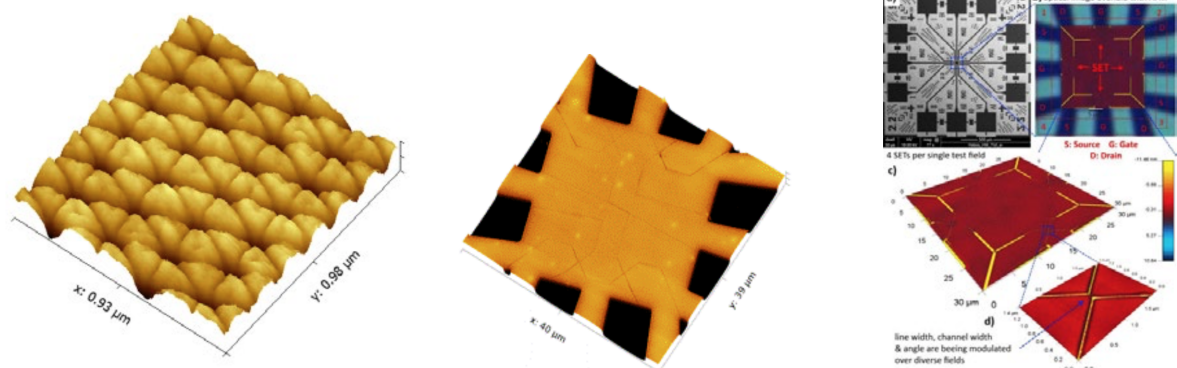


Fig. 3: Left: Plasmonic structures, Middle: SET-Inverter circuit, Right: Mix-and-Match Lithography. Conventional photolithography for large structures (electrodes) and FE-SPL for nano-features defining the SET.

Seventh Framework Programme FP7/2007-2013 under Grant Agreement No. 318804 (Single Nanometer Manufacturing for beyond CMOS devices – acronym: SNM).

References

- [1] Rangelow, I.W. et. al., Proc. SPIE, 7637 (2010).
- [2] Rangelow, I.W. et. al., Journal of Vacuum Science & Technology B34 (6) (2016).
- [3] Ahmad, A. et. al., Journal of Vacuum Science & Technology B34, 06KM03 (2016).

High Speed AFM Imaging and Nanolithography with Parallel Scanning Probes

M. HOLZ^{1,*}, A. AHMAD¹, A. REUM^{1,2},
T. ANGELOV¹, T. IVANOV², I. ATANASOV², E. GULYIEV², V. ISHCHUK², M.
KAESTNER², Y. KRIVOSHAPKINA², C. LENK², N. NIKOLOV³, I. W.
RANGELOW²

¹*Nano Analytik GmbH, Ehrenbergstraße 11, 98693 Ilmenau, Germany*

²*Department of Micro- and Nanoelectronic Systems (MNES), Institute of Micro and Nanoelectronics, Ilmenau University of Technology, Gustav-Kirchhoff-Str.1, 98693 Ilmenau, Germany* ³*Mikrosistemi, 9010-Varna, Bulgaria*

*m.holz@nanoanalytik.net

Keywords: AFM, Active Cantilever Array, Metrology, Inspection, Parallel Imaging, Nanofabrication

A crucial limitation of Scanning Probe Microscopy (SPM) and Scanning Probe Lithography (SPL) is the size of the measurement area and the speed with which this area can be imaged or patterned. The array-systems of an autonomous working cantilever have the capability to significantly increase the measurement area and speed compared to single cantilevers [1]. As previously presented, the productivity of this kind of imaging and metrology systems can be increased due to massively parallel scanning probes with a very large scale of integration. In the frame of the PRONANO-Project, we built an array of 128 cantilevers having a pitch of 200 μ m, which resulted in huge data acquisition processing to form the composite image using a DSP controlling unit for every cantilever [2]. This paper presents the fabrication and characterization of an active parallel cantilever device integrating four self-sensing and self-actuating probes in an array controlled by a multi-channel FPGA controller. The cantilevers are actuated thermomechanically and their bending is measured piezoresistively [3]. The piezoresistive read-out routinely ensures atomic resolution and a high imaging speed [4],[5]. The thermo-mechanical actuation allows a static and dynamic actuation of every cantilever individually and enables a simultaneous AFM operation of all cantilevers in an array [6]. The static actuation has a hub of $\pm 1.6\mu$ m and the dynamic actuation is used to drive the cantilever at his resonance frequency. The active cantilever array is moved over a surface to be imaged by a piezoelectric, nanopositioning scanner, which enables 200 μ m $\times$ 200 μ m scanning with 0.2 nm resolution. The pitch between cantilever tips is 125 μ m and allows in case of parallel imaging with one scan of the stage to acquire an image with a maximum size of 0.5 mm $\times$ 0.2 mm. The integrated cantilever devices are fabricated from a silicon-on-insulator (SOI) wafer using surface micromachining and a gas chopping plasma-etching process [6]. The SPL technology is based on the use of lowenergy electrons for direct writing or mask-less lithography [7]. The electron emission current is in the range of 1 to 50 pA and undergoes Fowler-Nordheim (FN) law [4]. Every cantilever has an 8 μ m-high conical silicon tip [8], which is used as an AFM scanning probe and as an electron field emitter. The multichannel, scalable controller architecture allows four FPGA boards to scan and collect data simultaneously. The scanning time of 4096 $\times$ 1024 px frame is 10:40 mins, the pixel resolution is 122 nm, and the required memory is 128 Mbits, assuming 32 bits topography data for 1 pixel. This throughput of the parallel set-up is more than 6 orders of magnitude larger than SPM working with a single cantilever. In this paper, we will

present investigation on throughput, reproducibility, resolution, positioning accuracy in case of AFM Imaging and FNNanolithography.

Acknowledgments

The research leading to these results has received funding from the European Union's Seventh Framework Programme FP7/2007-2013 under Grant No. 318804 "SNM".

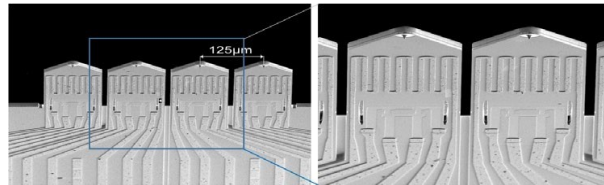


Fig. 1: "Quattro" cantilever array based on self-sensing and self-transduced cantilever.

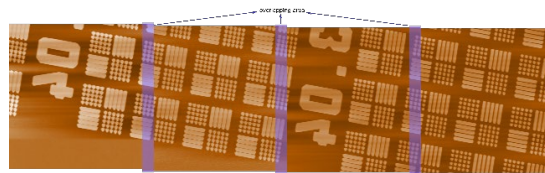


Fig. 2: AFM image obtained with array of four self-transduced und self-sensing cantilever. The imaged field is with size of $500\text{ }\mu\text{m} \times 135\text{ }\mu\text{m}$ where every sub-field is $135\text{ }\mu\text{m} \times 135\text{ }\mu\text{m}$ large. The overlap between the fields is $10\text{ }\mu\text{m}$. The composite AFM image consists 1.048.576 pixels attained in 126 seconds with four cantilever in non-contact-mode simultaneously.

References

- [1] K. Ivanova et al., J. Vac. Sci. Technol. B 26, 2367–2373 (2008).
- [2] I. W. Rangelow et al., Microelectron. Eng. 84, 1260-1264 (2007).
- [3] I. W. Rangelow, S. Skocki, and P. Dumania, Microelectron. Eng. 23, 365 (1994).
- [4] M. Kaestner et al., J. Vac. Sci. Technol. B 32, 06F101 (2014).
- [5] A. Ahmad et al, J. Micro/Nanolith. MEMS MOEMS 14, 031209 (2015).
- [6] I.W. Rangelow, Microelectron. Eng. 83,1449–1455 (2006).
- [7] I.W. Rangelow et al., Proc. SPIE 7637, 76370V (2010).
- [8] I.W. Rangelow and S. Biehl, J. Vac. Sci. Technology B 19, 916-919 (2001).

Six - axes AFM in SEM with self-sensing and self-transduced cantilever for high speed analysis and nano-lithography

M. HOLZ^{1,*}, T. ANGELOV¹, E. GULYIEV²,
A. AHMAD², A. REUM^{1,2}, I. ATANASOV², T. IVANOV², V. ISHCHUK², M.
KAESTNER², Y. KRIVOSHAPKINA², I. W. RANGELOW², N. NIKOLOV³

¹*Nano Analytik GmbH, Ehrenbergstraße 11, 98693 Ilmenau, Germany*

²*Department of Micro- and Nanoelectronic Systems (MNES), Ilmenau University of Technology, Gustav-Kirchhoff-Str.1, 98693 Ilmenau, Germany*

³*Mikrosistemi, 9010-Varna, Bulgaria*

*m.holz@nanoanalytik.net

Keywords: AFM, SEM, Active Cantilever

Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM) are routinely used techniques for high-resolution surface research. The self-sensing and self-transduced cantilever [1]-[4] allows an easier system integration and substantial decrease of the mass of the scanning head. The AFM microscope provides better controllability, significant higher imaging speeds with the potential for CD-metrology automation, and an in-situ inspection capability combined with the SEM. In this paper, the concept and first results of novel AFM in SEM for nanoscale characterization are presented. A six-axes AFM system is being developed and integrated into a high resolution SEM/FIB system allowing nanoscale analysis, nano-manipulation and scanning probe lithography. The compact and modular AFM setup enables probe- as well as samplescanning and uses self-sensing AFM cantilevers. The top-probe-scanner allows positioning of the cantilever in a space of 20 mm × 20 mm × 10 mm with precision of 1 nm. The sample-scanner is used for metrology purposes securing 0.2 nm resolution in x-, and y-axes and 0.1 nm in z-axes. Simultaneous control imaging software was developed to merge SEM and AFM information for mixed imaging of nanoscale objects. A FPGA AFM controller is embedded into a special control system architecture that allows for automation of nano-manipulation imaging and SPLsequences. The operation modes of the AFM include contact, non-contact, mass-detection and scanning-probe lithography [5]. The controller is offering amplitude-, frequency-, and phasesensitive detection. With respect to resolution and robustness, this technique is directly dependent on tip quality (material, aspect ratio, shape and operation mode of the AFM). Therefore, a carefully calibration of CD-AFM includes a probe geometry deconvolution and can be used as metrological tool where a key parameter is the shape of the tip. The active cantilever includes a conical shaped tip with 15 +/- 3 nm radius which is formed in highly doped, single crystal silicon, and offers long life in non-contact operation and is favorable for top-CD measurement of lithographical features and long-life operation as Fowler-Nordheim emitter for Scanning Probe Lithography [6]. These work goals at providing a new eloquent representation of detailed results from combined examinations, by applying fast AFM-methods and SEM-image fusion, AFMSEM combined metrology verification and 3D-visualization. Combined AFM and SEM capabilities provide a view of sample topography due to a large number of hybrid imaging and sub-10 nm measurement techniques. Metrology application scenarios from a combined AFM and SEM study will be discussed.

Acknowledgments:

The research leading to these results has received funding from the European Union's Seventh Framework Programme FP7/2007-2013 under Grant No. 318804 "SNM".

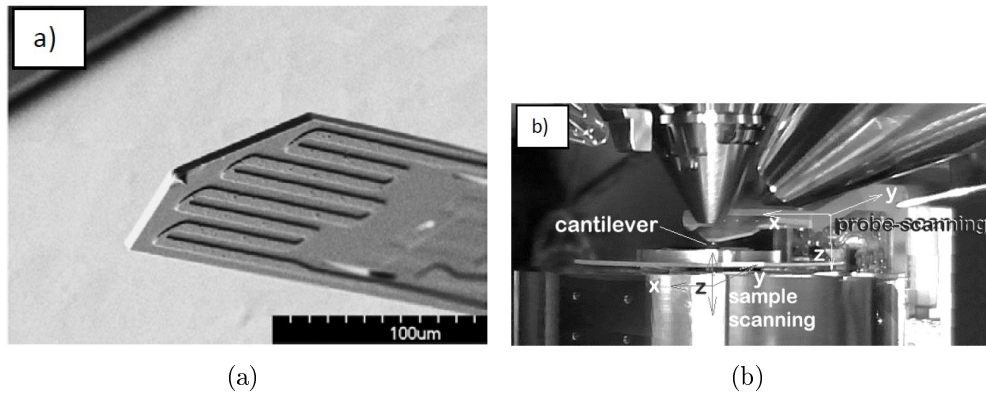


Fig. 1: 1(a) Self-sensing and self-transduced cantilever used for AFM in SEM; 1(b) Picture of the SEM-CCD camera shows the AFM in SEM set-up. The design allows probe- as well as sample-scanning, AFM tilting from -10 to + 60 and the SEM working distance of 4mm (high resolution).

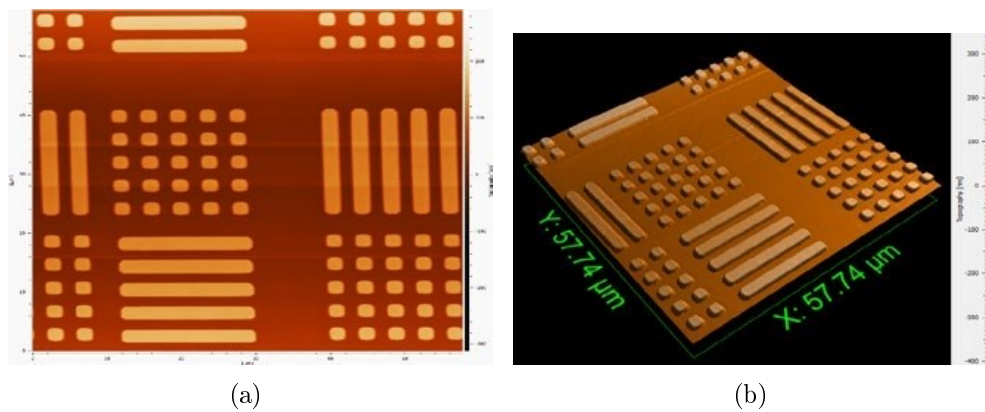


Fig. 2: 2D and 3D AFM images obtained with the AFM in SEM.

References

- [1] I. W. Rangelow, S. Skocki, and P. Dumania, *Microelectron. Eng.* 23, 365 (1994).
- [2] I.W. Rangelow, *Microelectron. Eng.* 83,1449–1455 (2006).
- [3] M. Kaestner et al., *J. Vac. Sci. Technol. B* 32, 06F101 (2014).
- [4] A. Ahmad et al., *J. Micro/Nanolith. MEMS MOEMS* 14(3), 031209 (2015).
- [5] M. Kaestner et al., *J. Vac. Sci. Technol. B* 32, 06F101 (2014).
- [6] M. Kaestner et al., *J. Micro/Nanolith. MEMS MOEMS* 14, 031202 (2015).

Investigation of PMMA surface stress development caused by the photooxidation following UV radiation exposition.

M. BŁASZCZYK^{*1}, M. DURKO¹, Z. IWANICKA¹,
A. LICHOCKI¹, A. PIOTROWIAK¹, M. MOCZAŁA², A. SIKORA^{2,*}

¹*Active Students Association, SEP Branch no. 1 in Wrocław, M. Skłodowskiej-Curie 55/61, Wrocław, 50-369, Poland*

²*Division of Electrotechnology and Materials Science, Electrotechnical Institute, M. Skłodowskiej-Curie 55/61, Wrocław, 50-369, Poland*

^{*}sikora@iel.wroc.pl

Keywords: Poly(methyl methacrylate), UV radiation, material stress, volume changes, AFM

Poly(methyl methacrylate) is commonly known polymer, which is applied in many different areas. Besides typical behavior of the material due to irradiation [1]-[4], changes in the volume of this polymer under the influence of ultraviolet radiation can determine its application. These changes can be observed not only by infrared analysis [5], but also by the use of atomic force microscope, which is characterized with high sensitivity [6]-[7].

In the paper, the investigation results obtained on PMMA samples exposed to the UV radiation using specially designed masks will be presented. The purpose of this procedure was to expose only the selected sample areas to UV radiation and enable the photooxidation process, which continued without the light access. The volume increase of tested material was measured using atomic force microscopy and other optical techniques. As the consequence, the stress appearance between the outer and inner layer of the material could be noticed. Next to the morphological changes (roughness), the presence of sub-micron scale cracks was observed (Fig. 1), and their further evolution to centimeter size in length features. In addition, the force spectroscopy technique was utilized in order to observe the stiffness changes in time. These measurement methods also allowed to observe the transparency changes and yellowing of the material that was exposed to radiation.

Acknowledgments

We thank Electrotechnical Institute for the opportunity to perform research needed for the purposes of this paper. The authors also thank Association of Polish Electrical Engineers SEP, section in Wrocław branch no. 1 in Wrocław for financial support.

References

- [1] D. Sobków, K. Czaja, Wpływ warunków przyspieszonego starzenia na proces degradacji poliolefin, *Polimery* 48 (2003) 627-632
- [2] H. Kaczmarek, A. Kamińska, A. van Herk, Photooxidative degradation of poly(alkyl methacrylate)s, *European Polymer Journal* 36 (2000), 767-777
- [3] Zs. Geretovszky, B. Hopp, I. Bertóti, I.W. Boyd, Photodegradation of polycarbonate under narrow band irradiation at 172 nm, *Applied Surface Science* 182(2002) 85-90
- [4] G.F. Tjandraatmadja, L.S. Burn, M.J. Jollands, The effects of ultraviolet radiation on polycarbonate glazing, *Durability of Building Materials and Components* 8 (1999) 884-898
- [5] N. Nagai, T. Matsunobe, T. Imai, Infrared analysis of depth profiles in UV-photochemical degradation of polymers, *Polymer Degradation and Stability* 88 (2005) 224-233

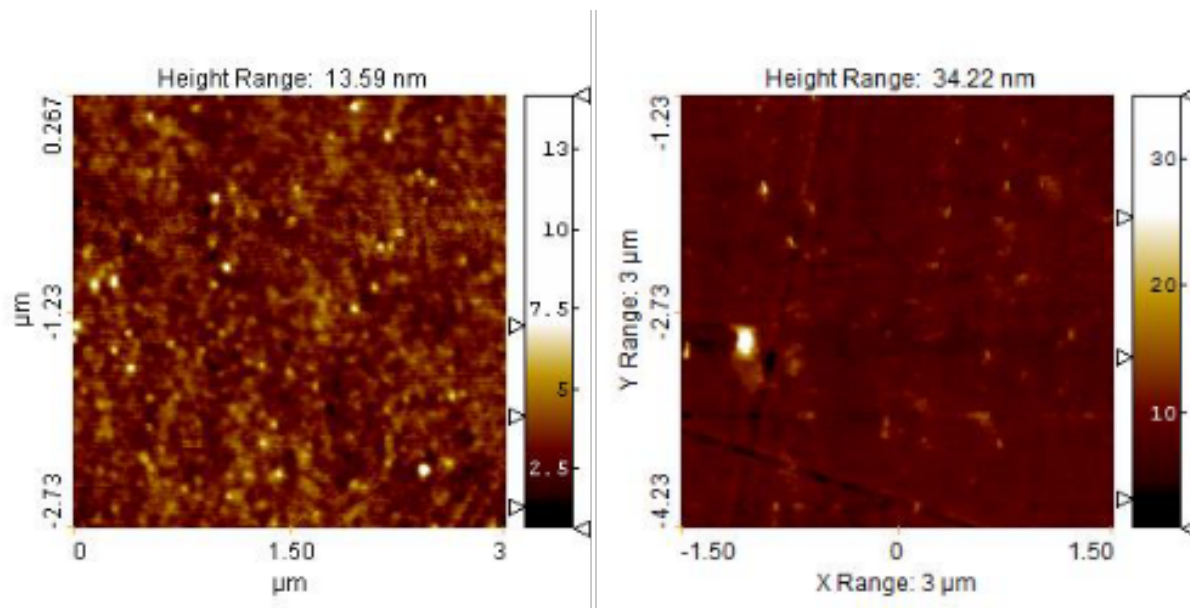


Fig. 1: The AFM images of the PMMA surface before and after UV light exposition (72 hours) and further degradation development due to photooxidation.

- [6] A. Sikora, Rozwój i zastosowanie zaawansowanych technik mikroskopii sił atomowych w diagnostyce materiałów elektrotechnicznych, Wybrane zagadnienia, Zeszyt 257, Prace Instytutu Elektrotechniki (2012) 17-58, 60-66
- [7] A. Sikora, The new approach to the investigation of the roughness changes of the non-uniform materials irradiated with UV light and imaged by means of atomic force microscopy supported with precise repetitive scanning area positioning, Measurement Science and Technology 28 (3) (2017), 034016

Design of novel type of active test sample for AFM thermal probes characterisation

P. JANUS^{1,*}, A. SIERAKOWSKI¹, D. SZMIGIEL¹,
M. RUDEK², T. GOTSZALK²

¹*Instytut Technologii Elektronowej al. Lotników 32/46, Warszawa, Poland*

²*Wroclaw University of Science and Technology, ul. Janiszewskiego 11/17, Wroclaw, Poland*

*janus@ite.waw.pl

Keywords: Atomic Force Microscopy, Scanning Thermal Microscopy, thermal nanoprob-
ing, thermal imaging

One of main issues related with the thermal nanometrology is the quantitative interpretation and reliable calibration of the thermal tip response [1, 2]. Moreover, the quantitative measurement of temperature at nanoscale can be performed only when the propagation of the heat from the tip to the sample and back to the cantilever is known. In this paper we present the updated design and technology of Si/Si₃N₄ micro calibration stage equipped with Pt microheaters [3]. This new design consists of 500 nm thick MEMS structure with 2 independently controlled microheaters with four point connections and improved thermal isolation. It allows for precise control of the temperature dissipation and a contact between surface and tip of cantilever. Localization on thin, low thermally conductive membrane minimizes the heat transfer to the bulk silicon. In order to increase mechanical stability of the structure, the membrane is supported by the tip. Structure stiffness is increased which allows for characterization of relatively stiff (30-70 Nm⁻¹) piezoresistive scanning thermal microscopy probes. The small size and spatial arrangement of independent heaters allows for the controlled heat flow in the membrane and measurements of the temperature distribution. Moreover, dedicated heat sinks located on the membrane decreases the heat transfer to the body of the chip. In the full paper, the manufacturing technology including MEMS surface micromachining is described. The results of electro-themo-mechanical characterization of this new stage and comparison with FEM modeling are also presented. Quantitative analysis of selected SThM probes with developed stage is discussed.

Acknowledgments

This research has been supported by the EU FP7 grant no. 318625, NANOHEAT - Multidomain platform for integrated More-than-Moore/Beyond CMOS systems characterization & diagnostics, MNiSW funding for science in the years 2013 - 2017 granted for co-financed project and ITE statutory funding.

References

- [1] L. Shi and A. Majumdar, "Thermal Transport Mechanisms at Nanoscale Point Contacts," J. Heat Transfer, vol. 124, no. 2, p. 329, 2002.
- [2] G. Hwang et al, Enabling low-noise null-point scanning thermal microscopy by the optimization of scanning thermal microscope probe through a rigorous theory of quantitative measurement, Rev. Sci. Instrum., vol. 85, no. 11, p. 114901, 2014.
- [3] P. Janus et al, Micromachined active test structure for scanning thermal microscopy probes characterization, Microelectronic Engineering, Volume 174, 25 April 2017, Pages 70–73

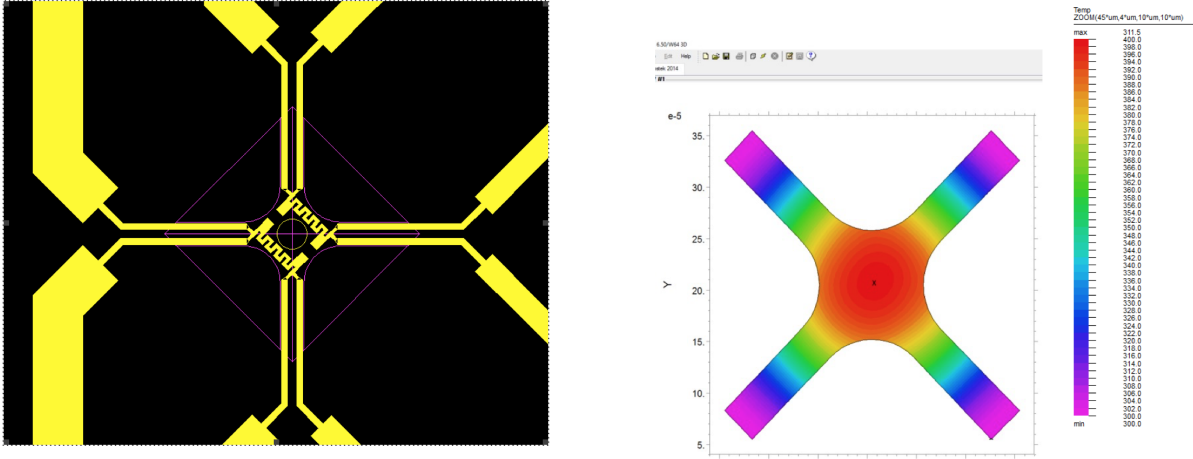


Fig. 1: New layout of the calibration structure (left) results of electrothermal FEM simulations (right)

Multi-frequency Kelvin Probe Force Microscopy in ambient condition and quantitative assessment of images quality

D. KOPIEC¹, G. JÓŹWIAK^{1,*}, M. MOCZAŁA¹,
A. SIERAKOWSKI², T. GOTSZALK¹,

¹*Faculty of Microsystem Electronics and Photonics, Wrocław University of Science and Technology, Janiszewskiego 11/17, Wrocław 50-372, Poland*

²*Division of Silicon Microsystem and Nanostructure Technology, Institute of Electron Technology, Lotników 32/46, Warsaw 02-668, Poland*

*grzegorz.jozwiak@pwr.edu.pl

Keywords: KPFM, multi-resonance, electrostatic force, image quality, information channel capacity

Kelvin Probe Force Microscopy (KPFM) [1] is a scanning probe microscopy (SPM) technique for investigation of such electrical properties as contact potential difference (CPD), charge and work function of various heterogeneous materials (semiconductor, organic or polymer structures, optical layers and self-assembled monolayers (SAM)) [2]. In recent years KPFM has been receiving special attention due to the fact that it is used for studying electrical properties of graphene. It should be noted that it is a non-contact and non-destructive method used not only in ultra-high-vacuum conditions, but also in gaseous and liquid environment. There are two techniques used in KPFM measurements. The first one called lift mode requires two separate scans. The surface topography is measured in intermittent contact mode during the first scan. Next, electrostatic force is measured in the second scan, when the tip is moving a constant distance above the surface. The distance is constant provided that XYZ position of the probe is stable and precisely controlled. So the stability of environmental factors like temperature, humidity, tip wear are critical in this KPFM mode. In many cases, the tip-sample distance is approximately several dozen nanometers and this requires to use large amplitude of electrostatic excitation signal. As a consequence, the spatial resolution becomes relatively poor due to long range electrostatic force acting on KPFM tip. These requirements can be relaxed if the measurements of topography and electrostatic force are done simultaneously. It can be achieved by multifrequency operation, where the cantilever is simultaneously excited in two or more cantilever eigenfrequencies. We present a single-pass and multifrequency KPFM technique with amplitude detection of mechanical vibration and electrostatic force. It was implemented as a homemade setup with optical beam deflection (OBD) detection in ambient conditions, which allows to measure a wide range of materials. In the proposed solution the cantilever is simultaneously excited into oscillations at first two eigenmodes. The first one is used for electrostatic force measurement and the second for topography measurements. The results of different actuation schemes are shown. The response time of the first two cantilever flexural modes and sensitivity of electrostatic force measurement are discussed. The properties of the proposed system was investigated by measurements of topography and CPD of self-assembled monolayers in ambient conditions. The registered images were quantitatively compared by estimation of the maximum capacity of information channel of the imaging system as introduced by Shannon in [3].

Acknowledgments

The research was cofounded by the Foundation for Polish Science (FNP) within the TEAM

Programme FoMaMet (Grant No. TEAM/2012-9/3) co-financed by the European Regional Development Fund resources within the framework of Operational Program Innovative Economy and Faculty of Microsystem Electronics and Photonics Wroclaw University of Science and Technology statutory founding.

References

- [1] M. Nonnenmacher, M. O'Boyle, H. Wickramasinghe, Kelvin probe force microscopy, *Applied Physics Letters* 58 (1991) 2921–2923
- [2] W. Melitz, J. Shen, A. C. Kummel, S. Lee, Kelvin probe force microscopy and its application, *Surface Science Reports* 66 (2011) 1–27
- [3] C. E. Shannon, Communication in the presence of noise, *Proceedings of the IEEE* 86 (1998) 447–457

The analysis of the collagen fibers orientation in the cranial dura mater tissue in relations to its mechanical properties

A. SIKORA^{1,*}, M. KARPIŃSKA², A. KĘDZIA³

¹*Electrotechnical Institute, Division of Electrotechnology and Materials Science, M. Skłodowskiej-Curie 55/61, 50-369 Wrocław, Poland*

²*Active Students Association, Association of Polish Electrical Engineers SEP (Branch No 1 in Wrocław), M. Skłodowskiej-Curie 55/61, 50-369 Wrocław, Poland* ³*Department of Anatomy, Wrocław Medical University, T. Chałubińskiego 6a, 50-368 Wrocław, Poland*

*sikora@iel.wroc.pl

Keywords: dura mater, atomic force microscopy, collagen fibers, tissue mechanical properties, neuroanatomy

Dura mater [1], [2], the outermost membrane of connective tissue surrounding brain and spinal cord is often susceptible to mechanical injuries. It is indispensable to properly determine how mechanical energy propagates throughout each type of tissue after mechanical impact is applied. The attempts of assessing mechanical properties of dura mater were already performed [3], [4], but so far no research aimed at the analysis of the microscale structural ordering of this tissue with its mechanical properties was presented. In particular, specific orientation of the collagen fibers bundles (Fig. 1) can be considered in terms of providing directional strengthening of the cranial dura mater's structure.

The aim of the presented work was to measure topography of the upper part of cranial dura mater with the use of atomic force microscopy (AFM), as it has already proved to be a reliable tool [5]-[7], while estimating properties of biological samples of small dimensions. In addition, optical microscope enabled studying the morphology of bigger parts of the investigated tissue, providing the insight into the millimetre scale structure of the tissue. The investigated sample was taken from 6-month-old human embryo. The analysis of obtained data revealed that collagen fibres tend to arrange themselves in roughly specified directions throughout dura mater (Fig. 1). As was shown in works aimed at the investigation of mechanical properties of anisotropic materials [8], the composites containing fibres that are parallel immersed in a matrix, exhibit significantly increased resistance to mechanical strain.

It proves that collagen fibres play reinforcing role in dura mater with the emphasis of certain directions considered as the critical in terms of the brain safety.

Acknowledgements

We thank Electrotechnical Institute for the opportunity to perform research needed for the purposes of this paper. The authors also thank Association of Polish Electrical Engineers SEP, Wrocław section and branch no. 1 in Wrocław for financial support.

References

- [1] R. Daroff, M. J. Aminoff, *Encyclopedia of the Neurological Sciences* (Academic Press, 2014)
- [2] <http://www.encyclopedia.com/medicine/anatomy-and-physiology/anatomy-and-physiology/meninges> (accessed 15.05.2017)

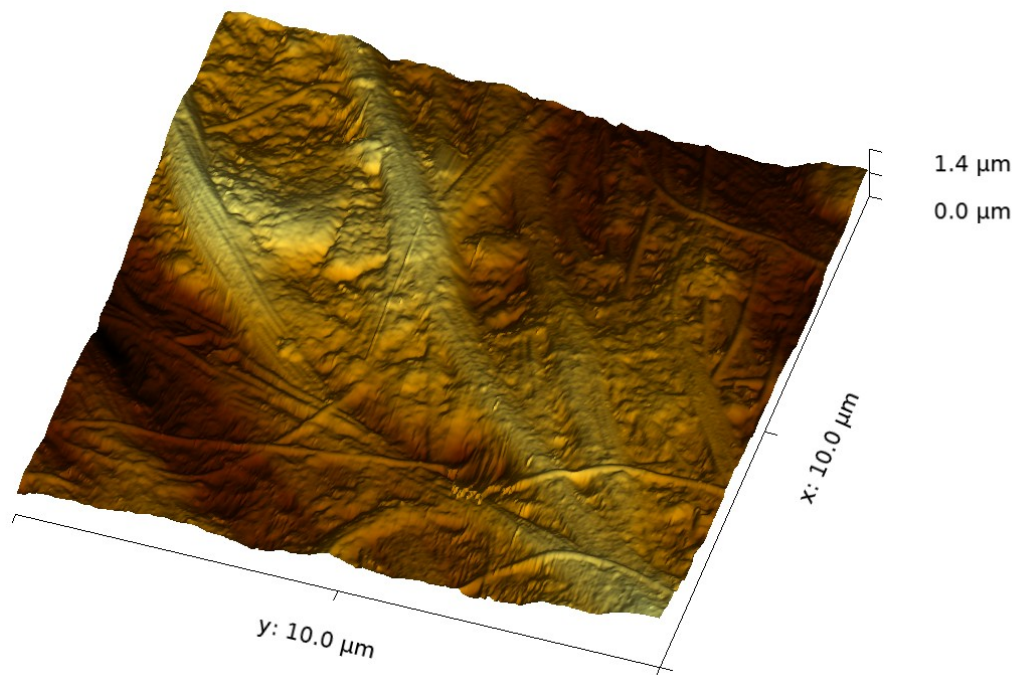


Fig. 1: The AFM image of the dura mater tissue. The collagen fibers bundles are visible.

- [3] E. Mazgajczyk, K. Ścigała, M. Czyż, W. Jarmundowicz, R. Będziński, Mechanical properties of cervical dura mater. *Acta of Bioengineering and Biomechanics*. 14 1(2012) 51-58
- [4] R. van Noort, T. R. P. Martin, M. M. Black, A. T. Barker, C. G. Montero, The mechanical properties of human dura mater and the effects of storage data. *Clin. Phys. Physiol. Meas.* 2 3 (1981) 197-203
- [5] M.-C. Giocondi, D. Yamamoto, E. Lesniewska, P.-E. Milhiet, T. Ando, C. Le Grimellec, Surface topography of membrane domains. *Biochimica et Biophysica Acta* 1798 (2010) 703-718 doi: 10.1016/j.bbame.2009.09.015
- [6] A. Sikora, A. Kędzia, A Quantitative Comparison of Dura Mater Tissue Structures Measured with Atomic Force Microscopy. *Adv Clin Exp Med* 21 4 (2012) 487-493
- [7] D. Erts, B. Plyakov, H. Olin, E. Tuite, Spatial and Mechanical Properties of Dilute DNA Monolayers on Gold Imaged by AFM. *J. Phys. Chem. B*, 107 15(2003) 3591-3597 doi: 10.1021/jp0261657
- [8] M. Kumaresan, S. Sathish, N. Karthi, Effect of Fiber Orientation on Mechanical Properties of Sisal Fiber Reinforced Epoxy Composites. *J. App. Sc. Eng.* 18 3(2015) 289-294 doi: 10.1007/s12633-015-9314-7

Dip-coating process optimization for insulating layers fabrication on quartz tuning forks

BARTOSZ KAWA^{1,*}, TOMASZ PIASECKI¹

¹*Faculty of Microsystem electronics and photonics, Wrocław University of Technology,
Janiszewskiego 11/17, Wrocław, 50-372, Poland*

*205685@student.pwr.edu.pl

Keywords: Dip-coating process, quartz tuning fork, insulating coatings, ring-down technique

The aim of the work was to optimize the process of coating the insulating layers on the quartz tuning forks (QTF) sensors used in biological application. Due to the nanometric deflections of QTFs prongs, they can be used in the density and viscosity measurements. QTFs biological measurements are difficult because bacteria liquid environment is conductive due to the dissociated ions. Additional insulating layers are necessary. In this case the electro insulating PMR-F lacquer was used. The requirements for the insulating layers were: small impact on mechanical and electrical properties of QTF; accurate, reliable and repeatable process of preparing; compatibility with the QTFs ring-down measurement method developed by Nanometrology Group. The insulation layer quality depended from concentration of lacquer, velocity of drawing QTFs from lacquer and curing temperature. Following experiments were conducted due to obtain the best coating parameters: capacitance measurements which later gave the information about thickness of deposited layers, 24 hours tests in 0.9% saline which simulated bacterial environment in order to know long term behaviour of QTFs coated with lacquer, dielectric strength measurements which gave the information of insulating PMR-F layer endurance on higher exciting voltage. Concluding all results, the best parameters for coating were obtained. The reliable, fully controlled and automated process of coating were developed. Perfect compatibility of coated insulating layers with the ring-down QTF measuring method has been found.

References

- [1] T. Piasecki, G. Guła, K. Waszczuk, Z. Drulis-Kawa and T. Gotszalk, „Quartz tuning fork as in situ sensor of bacterial biofilm”, *Sensors and Actuators, B: Chemical*, nr 210, page 825-829, 2015.
- [2] K. Waszczuk, “Metrologia i sterowanie kamertonów piezoelektrycznych w roli czujników mikrobiologicznych i biochemicznych” PhD thesis, PWR, WEMiF, Wrocław 2016.

The influence of thin obstacle geometry on the flow in microchannels

M. KMIOTEK^{*1}, A. KUCABA-PIĘTAL¹

¹*Department of Fluid Mechanics and Aerodynamics, The Faculty of Mechanical Engineering and Aeronautics Rzeszow University of Technology, al. Powstańców Warszawy 12, Rzeszów 35-959, Poland*

^{*}kmimal@prz.edu.pl

Keywords: microchannels, obstacle, Finite Element Method

Due to the miniaturization process, in many technical devices microchannels are used in cooling systems. Because of the small size of microchannels, the flow inside is laminar, which caused a slow heat exchange. In order to intensify the heat exchange, the flow must be disturbed, for example by introducing obstacles. Performed studies have shown that not only the spacing and relative height of the obstacle affects the flow disturbance intensification, but also their shape.

In this paper we will present comparison how the obstacles of various shape, triangular and perpendicular disturb the flow. Water flows in two dimensional channels of 400 micrometer height is studied. Corresponding Reynolds numbers are equal to: 20, 50, 100 and 200 - such flows occur in various technical micro devices cooling systems[2].

We report experimental and numerical results on heat and mass transfer in the micro channel where thin obstacles are immersed on the wall, to disturb the flow. Thin obstacle we call the one for which the parameter $o=w/h$ (w - width, h - height of the obstacle) satisfies inequality: $o < 0.5$.

For the numerical investigation, a Navier –Stokes computer code has been solved by use Adina CFD & FSI solver [2]. Experiments were performed by use micro Particle Image Velocimetry (μ PIV) [3].

Water flows distributions in the microchannel with triangular and rectangular thin obstacles were compared to visualize how their shape influences the fluid field. For instance, in Fig 1 we demonstrate differences in streamlines for flows past a single obstacles of triangular and perpendicular shape for parameter values: $Re=20$, $h/H=0.5$. It can be observed that the rectangular obstacle caused strongest vortex formation in water flow. In Fig 2 the numerical results against experimental for rectangular obstacle for $Re=20$ for $h/H=0.5$ are presented. The agreement is below 3%.

Performed investigations have shown that the placement of a thin obstacles with a rectangular shape on the wall increases the heat exchange in the microchannel from 15 to 74% relative to the smooth microchannel for temperature difference fluid/wall equal to 40°C.

Acknowledgments

We thank Electrotechnical Institute for the opportunity to perform research needed for the purposes of this paper. The authors also thank Association of Polish Electrical Engineers SEP, section in Wrocław and branch no. 1 in Wrocław for financial support.

References

- [1] ADINA R & D, Inc.[2013], ADINA Theory and Modeling Guide, Watertown, USA, Volume III:ADINA CFD & FSI,

- [2] O. A. Akbari, D. Toghraie, A. Karimipour, M. R. Safaei, M. Goodarzi, H. Alipour, M. Dahari, Investigation of rib's height effect on heat transfer and flow parameters of laminar water-Al₂O₃ nanofluid in a rib-microchannel, *Applied Mathematics and Computation* 290 (2016) 135–153
- [3] S. Błoński, P. Domagalski, M. Dziubiński, T. A. Kowalewski, Hydrodynamically modified seeding for micro-PIV, *Arch. Mech.*, 63, 2 (2011) 163–182,

Nanovortex dynamics in quartz nanocavity

A. KORDOS^{*1}, A. KUCABA-PIĘTAL¹,

¹*Department of Fluid Mechanics and Aerodynamics, Rzeszow University of Technology,
35-959 Rzeszow, Poland*

**a-kordos@prz.edu.pl*

Keywords: Nanofluidics, Nanovortices, Molecular Dynamics, Flow in nanochannel

The investigation of fluid flow inside a long, an open type nanocavities helps to gain understanding of many complex phenomena. This paper is continuation of our previous works [1] devoted to nanovortex investigations in the open type long nanocavities. Aim of the present paper is to study nanovortex dynamics in quartz nanocavity. Molecular dynamics method is used to study the problem and LAMMPS program [4] to perform the simulation. The computer representation of molecules of real materials were used [1, 2]. Simulation details can be found in [3]. The nanocavity is 7 nm width, 25.2 nm long and situated perpendicular to the main nanochannel of 22 nm height. The water flow in the nanocavity is induced by 2D Poiseuille water nanoflow in the main nanochannel. Water flow is simulated by adding constant force to molecules in the main channel. The walls of the main channel and the nanocavity are made from quartz.

The performed simulation corresponds to the 19 ns of real water flow. To visualize the nanovortex formation, a sequence of images of water velocity vector fields in the nanocavity was plotted using MD simulation data. The average velocity vector fields were obtained by averaging MD data with the bin method [5]. Averaging data from MD simulation at equal time intervals of the flow allowed to plot images of the averaged vector velocity fields at these intervals. The obtained sequence of images representing vector velocity distributions at successive time intervals, shows the nanovortex evolution depicting the formation mechanism.

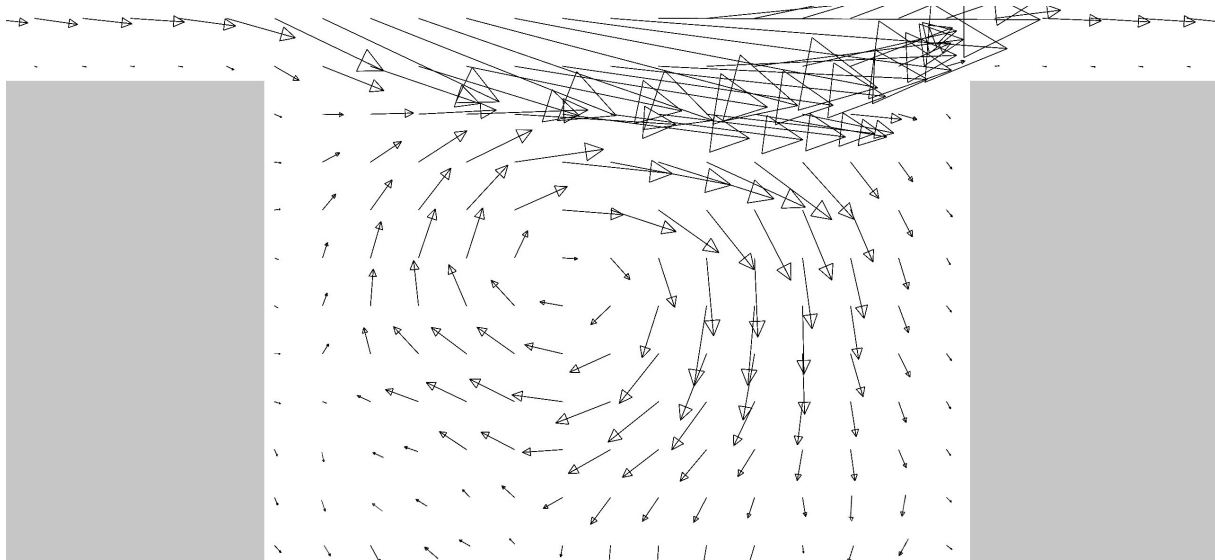


Fig. 1: Fully formed nanovortex in 2D open type nanocavity (A portion of the velocity vectors field)

Acknowledgments

Acknowledgments to ICM Warsaw University for access to high power computers (Research Project No. G45-8)

References

- [1] J. L. F. Abascal, C. Vega, . J. Chem. Phys., 123 (2005)
- [2] R. T. Cygan, J.J. Liang, A. G. Kaliniche, Journal of Physical Chemistry B, 108 (2004) 1255-1266
- [3] A. Kucaba-Piętal, A. Kordos, Nanovortices formation in 2D open type long nanocavities. Molecular dynamics study, Microfluidics and Nanofluidics, (in review) (2017)
- [4] LAMMPS, Sandia National Laboratories, <http://lammps.sandia.gov/>
- [5] M. P. Allen, D. J. Tildeslaey, Computer simulation of Liquids, Clarendon press, Oxford, 1991

Linear and Logarithmic Transducers Modules Used in Scanning Probe Microscopy

**C. WAWRZYNIAK¹, P. KULA¹, M. RUDEK¹,
K. ORŁOWSKA¹, M. BABIŃ¹, T. PIASECKI¹, T. GOTSZALK¹**

¹*Nanometrology Division, Wrocław University of Science and Technology, Wrocław,
Poland*

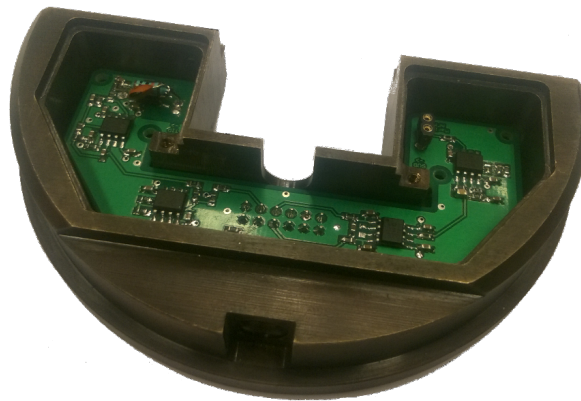
Current-voltage transducers are used in scanning probe microscopy. They are one of the most important microscope subassemblies used to measure the electrical properties of the tested surface. I-U Transducers in atomic force microscopes should have a low level of native noise (due to low measured currents). Due to the processing characteristics we can divide them into: linear and logarithmic transducers, due to polarization: of the substrate or blade.

The main aim of the work was to implement the Conductive Atomic Force Microscopy mode (C-AFM) in own design – ARMScope. I-U transducers are implemented directly in the SPM head, eliminating the need for long interconnections between the probe and the processing circuit, which in turn reduces the negative influence of the lead resistance and parasitic capacitance.

The modules developed by the authors are used in Scanning Tunneling Microscope (STM) and AFM:

- the linear module was used for measurements using C-AFM mode for measuring high-oriented pyrolytic graphite,
- the thermally stabilized logarithmic module is particularly useful in Scanning Spreading Resistance Microscopy (SSRM). In SSRM mode, the measured current can be characterized by high dynamics of changes. Stabilization eliminates drift of current that increases the measurement error.

The systems were characterized for their own noise and thermal stability.



FIB modifications for magnetically driven cantilever actuation efficiency increase

P. KUNICKI¹, K. ORŁOWSKA¹,
W. MAJSTRZYK¹, T. GOTSZALK¹

¹Wrocław University of Science and Technology, Janiszewskiego 11/17, Wrocław, 50-372, Poland

*piotr.kunicki@pwr.edu.pl

Keywords: FIB modifications, MEMS, cantilever, mechanical characterization

We present the results of experiment performed on MEMS cantilever using SEM/FIB microscope. The unique double loop cantilever design allows us for easy actuation investigation at first and second fundamental frequency modes. Various methods of actuation were applied. For magnetic actuation well position defined permanent magnet was used to get the same actuation force during experiment. The special dual current source was fabricated. The characterization of cantilevers were done with SIOS Nanovibration Analyzer – the high resolution interferometric system. The modifications of cantilevers were performed with SEM/FIB FEI Helios NanoLab 600i microscope. Two undercuts were performed in desired places. The actuation efficiency increased about 75 % for first and about 90 % for second fundamental mode. The motivation of this work was to decrease dissipated power on cantilever to reduce thermal effects. By authors modifications, the power can be decreased about 4 times.

Acknowledgments

The research was founded with statutory grant of Nanometrology group, 0401/0234/16, at Wrocław University of Science and Technology.

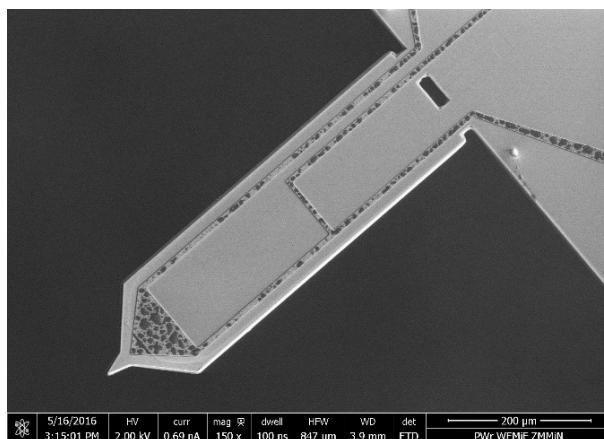


Fig. 1: SEM image of double loop cantilever

Metrology of MEMS based femtocalorimeter

M. LACHAWIEC^{1,*}, T. GOTSZALK¹,
M. RUDEK¹

¹*Wrocław University of Science and Technology, Wybrzeże Wyspiańskiego 27, Wrocław,
50-370, Poland*

*213961@student.pwr.edu.pl

Keywords: calorimeter, MEMS membrane

Calorimeters in microscale make quantitative measurements on very small samples. It can be adopted for small-scale measurements. Design of MEMS membrane used in research is shown in Figure 1. A membrane serves as the sample holder and provides thermal isolation. It consist of platinum heater and platinum temperature sensor. The membrane is made from silicon nitride.

The calorimeter is designed to perform micro/nanoscale thermal analysis with high sensitivity on extremely small amounts of sample and can be operated at very fast rates. Reduced sample size means reduced sample consumption. It also means higher resolution and better thermal isolation. Suspending the thermal sensor and heater on MEMS membrane provides excellent isolation. Temperature can be measured using integrated resistors by measuring changes in the resistivity of the sensing element. The overall resolution and sensitivity depends on the thermal mass of the sensor, sample mass, and the evaluation and minimization of heat losses during measurements. Other calorimeters designs have been used to measure cell metabolism, interfacial reactions, phase transition, nanoscale biological reactions.

To evaluate the ability to measure using the MEMS membrane it was made a research. It was measured changes of temperature during heating. It was determined thermal time constant, sensitivity and resolution of femtocalorimeter.

References

- [1] WIREs Nanomed Nanobiotechnol 2012, 4:31-41. doi: 10.1002/wnan.155

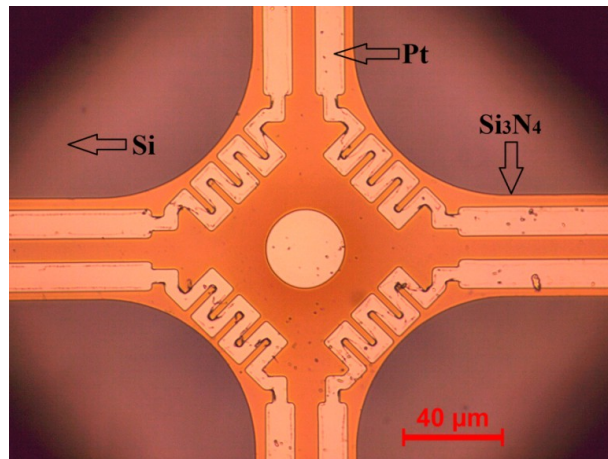


Fig. 1: MEMS membrane

Investigations on Plasma Durability of Resists for Field-Emission Scanning Probe Lithography (FE-SPL)

C. LENK^{1,*}, Y. KRIVOSHAPKIN¹, M. HOFMANN¹, B. VOLLAND¹,
M. KAESTNER¹, T. IVANOV¹, S. LENK¹, M. HOLZ²,
A. AHMAD¹, A. REUM², I. W. RANGELOW¹

¹*Department of Micro- and Nanoelectronic Systems (MNES), Ilmenau University of Technology, Gustav- Kirchhoff-Str. 1, 98693 Ilmenau, Germany*

²*nano analytik GmbH, Ehrenbergstraße 1, 98693 Ilmenau, Germany*

*claudia.lenk@tu-ilmenau.de

Keywords: plasma etching, resist, plasma durability, FE-SPL

Field-Emission scanning probe lithography (FE-SPL) is a high-resolution nanopatterning technique [1, 2] using low-energy electrons emitted from a scanning probe. It allows patterning resist with structures having sub-10nm resolution in ambient conditions. However, in order to enable highest resolution ultrathin resist layers (5-15nm) are applied. Linked with that, the pattern transfer by plasma etching becomes a difficult challenge with respect to selectivity, etching rates and process control.

To secure the high resolution during pattern transfer for device fabrication, anisotropic etching processes are required. These can be achieved by cryogenic plasma etching [?] using SF₆ and O₂ plasma at temperatures below -60°C. Thus, resist materials are required, which exhibit high plasma durability.

In this work we have studied and compared different resist materials according to their plasma durability and their selectivity for silicon etching. In particular, monomolecular resists as calixarene are compared with polymeric resist films consisting of Poly(3-hexylthiophen-2,5-diyl) (P3HT), AZ® Barli®, Polystyrene (PS), Polyhydroxystyrene (PHS) or Polymethylmethacrylat (PMMA) [6]. Therefore, these resists are spin-coated onto silicon substrates with layer thicknesses ranging from 7 to 10 nm. Standard patterns are generated into the resist films via FE- SPL with different size and resolution. Samples are plasma etched at cryogenic temperatures.

Before and after etching as well as after resist strip the patterns are analyzed by AFM. Therefrom derived the selectivity and etching rates are determined and compared for different resist materials. While selectivities for calixarene, AZ Barli, P3HT and PMMA are comparable (in the range 3-4:1), PHS exhibits an extraordinary plasma durability yielding a selectivity in the range of 20:1. SPL resolution with respect to pitch between parallel lines is in the range of 10nm half pitch for PHS, calixarene, PS and AZ Barli. Thereby, the smallest values for critical dimensions were obtained for calixarene and AZ Barli, i.e. 5 nm line width.

Acknowledgments

The research leading to these results has received funding from the European Union's Seventh Framework Programme FP7/2007-2013 under Grant Agreement No. 318804 (Single Nanometer Manufacturing for beyond CMOS devices – acronym: SNM).

References

- [1] Durrani, Zahid et.al: Scanning probe lithography approach for beyond CMOS devices. In: SPIE Advanced Lithography, 2013

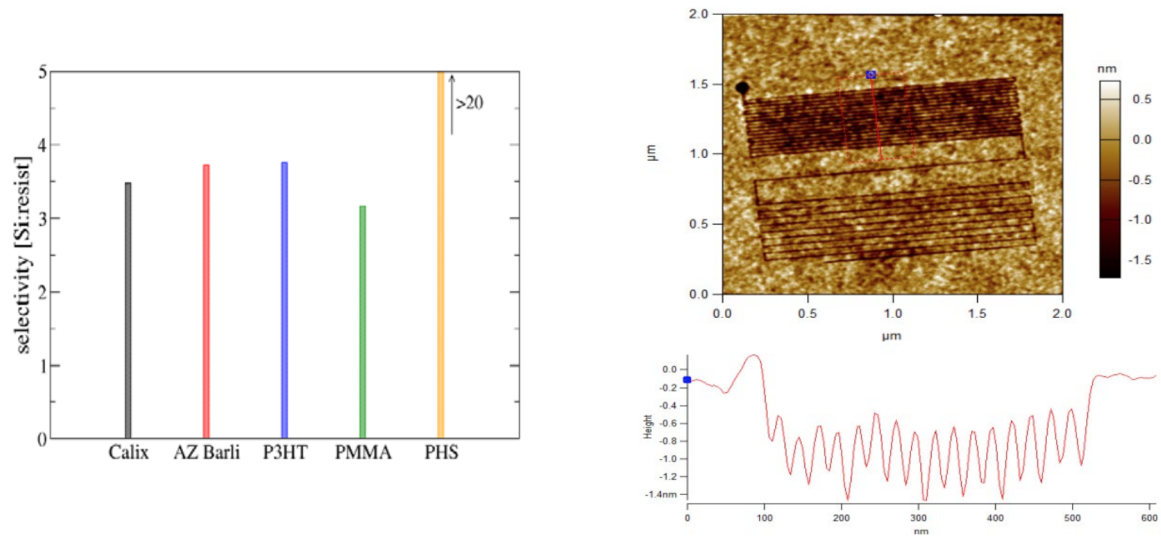


Fig. 1: (left) Comparison of etch selectivities for different resist materials, (right) example of 10nm half pitch written on AZ Barli.

- [2] M. Kaestner et al: Advanced electric-field scanning probe lithography on molecular resist using active cantilever. In: J. Micro/Nanolith. MEMS MOEMS, 2015
- [3] Liu, Z. et.al: Super-selective cryogenic etching for sub-10 nm features. In: Nanotechnology 24, 2012
- [4] Gu, X. et.al: High Aspect Ratio Sub-15nm Silicon Trenches from Block Copolymer Templates. In: Advanced Materials 24, 2012
- [5] I.W. Rangelow et.al: Pattern-generation and pattern-transfer for single-digit nano devices
- [6] Y. Krivoshapkina et.al: Low-energy electron exposure of ultrathin polymer films with scanning probe lithography. In: Microelectronic Engineering, 2017

Synthesis and mechanical properties of sol-gel oxide materials

B. BABIARCZUK¹, M. SZALKOWSK¹,
A. SZCZUREK¹, D. LEWANDOWSKI¹, J. KRZAK^{1,*}

¹*Department of Mechanics, Materials Science and Engineering, Faculty of Mechanical Engineering, Wrocław University of Technology, Smoluchowskiego 25, 50-372 Wrocław, Poland*

*justyna.krzak@pwr.edu.pl

Keywords: sol-gel methods, thin films, aerogels, mechanical properties

There are a lot of effort to measure mechanical properties of thin films and aerogels obtained by sol-gel method. Regarding the thin films the tools used for such measurement gives us a parameters correlated and comparable with other measurement done by this tool. In case of native silica aerogels, measurements of mechanical properties are very limited. There are a lack of literature data regarding mechanical properties sol-gel oxides in nano and submicro scale. This properties are very important from the bioapplication point of view [1], [2]. The mechanical properties of surface of metallic implant materials are still far from the tissue that is to be replaced. In direct contact of individual cells colonizing an implant, an important role is played by the properties of the surface of the implanted material.

The paper presents an attempt to measure the properties of the surface of SiO₂ multi-layer coatings and native SiO₂ aerogels obtained by the sol-gel process. Based on flexural strength tests and the cracking resistance, the quality of adhesion of the coatings was described. Moreover, tension tests for coatings and compression tests for aerogels were performed. The results indicate that the obtained coatings approach of surface properties of metallic implants materials to appropriate for the tissue environment.

Acknowledgments

The work has been supported by Grant no. 0401/0029/17 at Wrocław University of Science and Technology.

References

- [1] E.J. Lee, F. Kurtis Kasper, A.G. Mikos, Biomaterials for Tissue Engineering, Annals of Biomedical Engineering, 2013, DOI: 10.1007/s10439-013-0859-6
- [2] B.-H. Lee, Ch. Lee, D.-G. Kim, K. Choi, K.H. Lee, Y. D. Kim, Effect of surface structure on biomechanical properties and osseointegration, Materials Science and Engineering C 28 (2008)

Electromagnetically actuated silicon microcantilever arrays for multicantilever force-distance spectroscopy

W. MAJSTRZYK ^{*1}, K. ORŁOWSKA ¹, T. GOTSZALK ¹,
A. SIERAKOWSKI ², P. GRABIEC ², I. RANGELOW ³

¹ *Wroclaw University of Science and Technology, Janiszewskiego 11/17, 50-372 Wroclaw, Poland*

² *Institute of Electron Technology, Al. Lotników 32/46, 02-668 Warsaw, Poland*

³ *Ilmenau University of Technology, Gustav-Kirchhoff-Str. 1, D-98684 Ilmenau, Germany*

*wojciech.majstrzyk@pwr.edu.pl

Keywords: AFM Cantilever, Lorentz Force, Piezoresistive Bridge, Virtual Reference

Force Spectroscopy is one of the most commonly applied methods and techniques in scanning probe microscopy (SPM). One of its drawback is relatively low throughput when investigations are performed with single cantilever. Additional limitation is application of bulky piezoelectric actuators with limited bandwidth and with many parasitic resonances. We present a concept of an array with heavily Boron Doped Silicon microcantilevers where each and every one of the cantilevers is actuated electromagnetically. The application of the array of silicon microcantilevers working simultaneously will enhance throughput and credibility of characterization for multicantilever force spectroscopy. In our experiments we have focused on possibility of actuating arrays of four cantilevers. In the electromagnetic actuation scheme, force is exerted on a conducting path in which current flows immersed in uniform magnetic field [1]. Force is the directly proportional to both magnetic induction, and current flowing through conducting path

$$\vec{F}_L(t) = \vec{I} \times \vec{B} \cdot L \quad (7)$$

The basic advantage of utilized method is that we control deflection in both directions. In arrays we applied each cantilever has its own Lorentz conductive loop thus we can control them independently and simultaneously. In our experiments we have applied SIOS Nano Vibration Analyzer to calibrate sensitivity of the cantilever deflections at low frequencies (pseudo-static mode) and at resonance. The application of heavily Boron Doped Silicon technology ensures that the cantilevers are built from uniform type material which results in obtained deflections ranging up to 1 μm without bimaterial effect.

Acknowledgments

This research has been supported by the NCN OPUS 9 Grant - "Metrology of molecular interactions using electromagnetically actuated MEMS force sensors-MetMolMEMS" (Grant No. 2015/17/B/ST7/03876).

References

- [1] K. Nieradka et. al, Fabrication and characterization of electromagnetically actuated microcantilevers for biochemical sensing, parallel AFM and nanomanipulation, *Microelectronic Engineering*, 98 (2012) 676–679

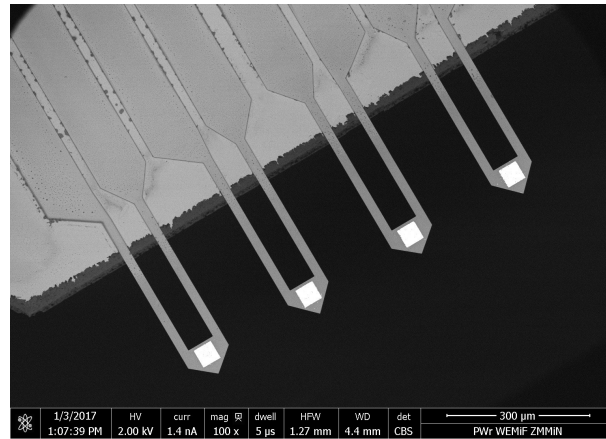


Fig. 1:) SEM image of the electromagnetic BDS cantilever arrays

Review of the EU FP7-ICT Project "Single Nanometer Manufacturing for beyond CMOS devices (SNM)"

M. KAESTNER^{1,*}, S. LENK¹, I. W. RANGELOW¹

¹*Department of Micro- and Nanoelectronic Systems (MNES), Ilmenau University of Technology, Gustav-Kirchhoff-Str. 1, 98693 Ilmenau, Germany*

*marcus.kaestner@tu-ilmenau.de

Keywords: Nanofabrication, Nanoelectronics

From 2013 to 2017 the European FP7 project "Single Nanometer Manufacturing for beyond CMOS devices (SNM)" (coordinated by Prof. Ivo W. Rangelow, TU Ilmenau) targeted the development of the manufacturing of semiconductor chips with critical dimension in sub-10 nm range. The sixteen project partners from eight European countries worked successfully together in order to shape a future technology for the production of highly-functionalized nanoelectronic devices in single nanometer range.

The project was divided into four technical work package groups, which addressed (WPG1: WP1-WP4) the development of different lithographic methods; (WPG2: WP5-WP7) pattern transfer, metrology and resist development; (WPG3: WP8-WP9) design and characterization of devices and (WPG4: WP10-WP11) the high-throughput manufacturing.

One project outcome is the development of several novel nanofabrication technologies including lithography (Fig. 1, [1]-[3]), plasma etching [4] and metrology [2] at single-digit nanoscale. For example, the group at TU Ilmenau (TUIL) investigated a cost-efficient single-digit nanometer lithography process using low energetic electrons emitted from one or several scanning probe microscope (SPM) tips [1]-[4]. Since its invention in 1986, SPM technology became one of the most important surface analysis and fabrication methods for the nanometer scale. Despite of this remarkable progress of the SPM technology, insufficient imaging or writing throughput often hinders broad industrial application. The extension of the SPM-technique to cantilever array operation could overcome the fundamental slowness and increase the throughput by parallelization of imaging and patterning. The new generation of active single-crystal silicon cantilever arrays with integrated individual cantilever actuation and sensing was developed and fabricated by TU Ilmenau. This technique allows high-resolution inspection and precise generation of the nano-features due to the use of the AFM writing or imaging mode. In 2003, we discovered the so called "positive tone" development-less lithography in molecular resists using low energy electrons emitted from a SPM tip. This was the ignition of the idea for lithography at the single-digit nanometer level.

Acknowledgments

The research leading to these results has received funding from the European Union's Seventh Framework Programme FP7/2007-2013 under Grant Agreement No. 318804 (Single Nanometer Manufacturing for beyond CMOS devices – acronym: SNM).

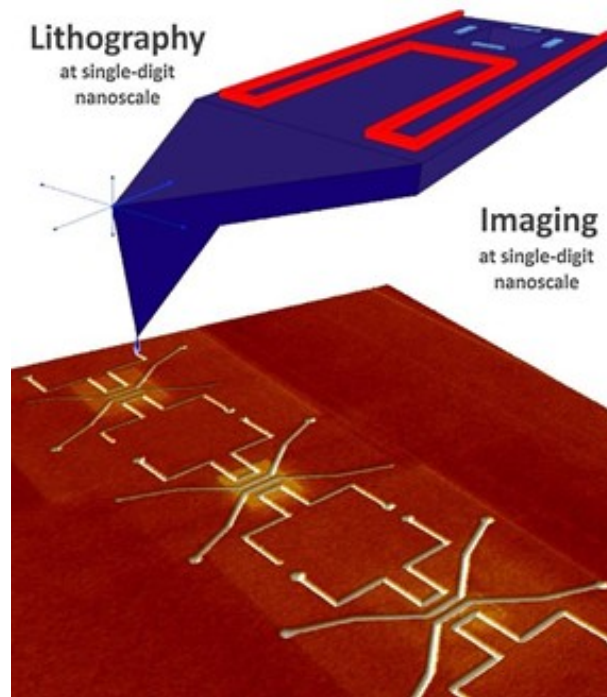


Fig. 1: Field Emission Scanning Probe Lithography (FE-SPL) incorporating imaging and lithography at single-digit nanoscale. Due to the closed loop lithographic capability feature inspection, overlay alignment as well as stitching is integrated allowing step-and-repeat and mix-and-match procedures for large scale patterning.

References

- [1] M. Kaestner et al., J. Micro/Nanolith. MEMS MOEMS 12, 031111 (2013).
- [2] M. Kaestner et al., J. Vac. Sci. Technol. B 32, 06F101 (2014).
- [3] M. Kaestner et al., J. Micro/Nanolith. MEMS MOEMS 14, 031202 (2015).
- [4] I. W. Rangelow et al., J. Vac. Sci. Technol. B 34, 06K202 (2016).

Effect of different micronozzle geometry on heat transfer characteristics of impingement cooling surface with inhomogeneous temperature distribution

K. MARZEC^{*1}, A. KUCABA-PIĘTAL²

¹*Rzeszów University of Technology, ul. Powstańców Warszawy 12, 35-959, Poland*

²*Rzeszów University of Technology, ul. Powstańców Warszawy 12, 35-959, Poland*

^{*}k_marzec@prz.edu.pl

Keywords: impingement cooling, heat transfer, Nusselt number

Introduction

An impingement cooling system is an array of microjets with high velocity fluid which is made to strike a target surface. Many experimental and numerical investigations of arrays of impingement cooling microjets are focused on high Nusselt number delivery [1]. The majority of the studies are experimental ones. However many simulations of the impingement cooling systems are numerical [2]. Only a few are devoted to the problem of cooling a surface with an inhomogeneous distribution of temperature. Such situation occurs in many technical applications such as photovoltaic cells. The main goal of the numerical investigation was to examine an inline array of ten impingement microjets which is heated with various heat fluxes $q_w(x)$.

Problem Formulation

The impingement cooling system consists of an array of ten impingement micronozzles directed normally to the flat surface. Cylindrical, convergent and divergent micronozzles with taper angle $\alpha=30^\circ$ are taken into consideration. In this study diameter $D=800\ \mu\text{m}$ of the micro-jet was considered. The micronozzle exit-plate distance $X/D=8$ and the micronozzle pitch-diameter ratio $S/D=8$ were constant for all simulations. The distribution tube inlet diameter was $D_{\text{in}}=5000\ \mu\text{m}$. Air was supplied to the system by the supply-tube only from the left side. The right side of the tube was closed. At the first analysis the wall, the microjet impinged onto was represented by the constant heat flux $q_w(x)=5000\ \text{W/m}^2$ and later on by the three increasing and decreasing linear functions $q_w(x)$.

Solution method and numerical setup

The simulations were performed using Computational Fluid Dynamics (CFD) code Ansys CFX. In the present investigation the $k-\omega$ shear stress transport (SST) turbulence model was used. SST model is recommended for such applications [3]. The investigation was limited to the steady state assumption and dynamic features of the impinging microjets were ignored. To validate the numerical technique and the solution procedure the comparison of numerical results of an impingement cooling system of surface with uniform temperature distribution with experimental were performed which showed satisfying agreement [4]. The numerical calculations were carried out using unstructured tetrahedral grids with 1,79 mln elements and 323119 nodes generated by the Ansys CFX mesher. The influence of the numerical grid density on the results of the heat transfer coefficient and the Nusselt number in the stagnation region was taken into consideration. As to investigate the sensitivity of the results of the numerical analysis, the Grid Convergence Index GCI was calculated [5] and it might be concluded that numerical results

on the fine grid were grid independent [6].

Solution method and numerical setup

Flow of an array of ten impinging micronozzles had a very complex behavior for each type of the microjets and inlet flow parameters. It is a result of the deflection at the stagnation area (especially at the first microjets) and because of the jet to jet interaction, which is called “fountain effect”. The sudden change in the flow direction in the micronozzles before striking onto the target plate resulted in the deflection angle in the direction of the flow inside the distribution tube. This was also observed in [7]. The highest flow deflection angle was observed at the first micronozzle. On the last one, the flow direction was perpendicular to the cooled surface. Therefore an eccentricity of the velocity distribution at the micronozzle area was noticed. The optimal system configuration for a given heat flux gradient $q_w(x)$ will be determined by the constant mass flow rate and the uniform Nusselt number distribution on a cooled surface. The uniform Nusselt number distribution plays a significant role as it might reduce thermal stresses on a cooled surface. The usage of a constant heat flux $q_w=5000\text{ W/m}^2$ resulted in the highest averaged Nusselt number $N_u=4,59$ for the cylindrical micronozzles. In addition, it was seen that the line of an averaged Nusselt number increased slightly in the x direction (along the flow of the distribution tube). Also it was noticeable that the line of the averaged Nusselt number decreased in the direction of the flow in the distribution tube for the heat flux function $q_w(x)=5000-1000\text{ W/m}^2$ and $q_w(x)=5000-0\text{ W/m}^2$. The heat flux $q_w(x)=5000-25000\text{ W/m}^2$ represented most uniform values of the Nusselt number N_{uo} in the stagnation region for all of the microjets. Presented conclusions are significant for the designers to handle with the temperature gradient on the cooled surface. Especially during the design of the photovoltaic cells or turbine cooling systems.

References

- [1] J. San, W. Shiao, Effect of jet plate size and plate spacing on the stagnation Nusselt number for a confined circular air jet impinging on a flat surface, *International Journal of Heat and Mass transfer* 49 pp. 3477-3486 2006.
- [2] M. Zukowski, Heat transfer performance of a confined single slot jet if air impinging on a flat surface *International Journal of Heat and Mass Transfer* 57 pp 484-490 2013.
- [3] N. Zuckerman, N. Lior, Jet impingement heat Transfer: Physics, Correlations and Numerical Modeling *Advanced in Heat Transfer* Vol. 39 2006.
- [4] K. Marzec, A. Kucaba-Piętal, Applications of computer science in impingement cooling system design *Pre-Proceed. of 9th International Conf. on Applied Mathematics Baia Mare, Romania*, 2013
- [5] S. Błoński, 2009 PhD Thesis: Laminar-turbulent flow analysis in micro-channels *Polish Academy of Science, Institute of Fund. Techn. Research*.
- [6] K. Marzec, A. Kucaba-Piętal, Heat transfer characteristic of an impingement cooling system with different nozzle geometry, *Journal of Physics Conference Series* Ser. 530, 2014.
- [7] K. Marzec, A. Kucaba-Piętal, Numerical investigation of a heat transfer characteristics of an impingement cooling system with non-uniform temperature on a cooled surface, *Journal of Physics Conference Series* Ser. 745, 2016.

The influence of calcination temperature on the optical and structural properties of TiO₂ thin films prepared by sol-gel and spin coating

T. TAŃSKI^{1,2}, W. MATYSIAK^{1,*}, D. KOSMALSKA¹,
A. LUBOS¹

¹*Department of Materials Processing Technology, Management and Technology in Materials, Institute of Engineering Materials and Biomaterials, Silesian University of Technology, Konarskiego 18a Str., 44-100 Gliwice, Poland*

²*Center for Nanotechnology, Silesian University of Technology, Konarskiego 18a Str., 44-100 Gliwice, Poland*

*wiktormatysiak@polsl.p

Keywords: spin coating, sol-gel, TiO₂, UV-Vis

The significant development of nanotechnology in recent years has been associated with production of oxide nanomaterials such as nanoparticles, nanotubes, nanofibres, thin films and nanoparticles, which have unprecedented physical and chemical properties. Oxides Al₂O₃, TiO₂, SiO₂, ZnO, Bi₂O₃ are the most interesting material for scientists because these materials are available in the world and easy of application. In the last two decades, one of the most commonly studied and used oxide is titanium dioxide. High interest in industry and research by TiO₂ is due to the potential application of titanium dioxide in various industries, such as the cosmetic, construction, medical, electrical and chemical industries. TiO₂ has high photocatalytic activity, relatively low price, high physical and chemical stability and low toxicity. These properties making titanium dioxide one of the most interesting photochemical materials [1]. In the word, scientists used many different methods to produce thin films, for example chemical vapor deposition, sol-gel method, ultrasonic spray pyrolysis, anodic electrophoretic deposition. Sol-gel technique combined with spin coating has found its greatest use in making thin films. The reason for this is the easy application of the coating on large surfaces, the low cost of gel production and the control of the microstructure films [2]. Titanium (IV) butoxide (TNBT, 97% Sigma Aldrich) was used as a precursor of titania. Solution was prepared by the 2 ml TNBT in 2 ml acetic acid (AcOH, Sigma Aldrich). As prepared solution was subjected to magnetic stirrer for 30 minutes. In order to prepare H₂O/ EtOH solution the following were used: 1,5 ml deionized water and 10 ml ethanol (EtOH, purity of 99.8%, Sigma Aldrich) and was stirred on magnetic stirrer for 30 minutes. Next, both prepared mixtures: TNBT/AcOH and EtOH/H₂O were mixed and stirred at magnetic stirrer for 1 hour. Thin films were deposited on the glass by sol-gel spin coating method. Samples have been calcination in the three temperature: 400°C, 500°C and 600°C [3]. The structure and morphology of TiO₂ sol-gel coatings applied to the glass substrate were analyzed by scanning electron microscopy and atomic force microscopy. It has been noted that as the calcination temperature increases, the thickness of the films decreases and has a value: 360 nm for films before calcination and 260 nm, 180 nm, 150 nm for samples calcination in 400°C, 500°C, 600°C. From the spectra, it was observed that the films were transparent in visible region and showed characteristic absorption in UV region at a shorter wavelength range. As the temperature of the heating temperature increases, the absorption line to move towards the Vis wavelength range. In this study, a systematic preparation of photocatalytic TiO₂ thin films using a simple and cost effective method was obtained. The films with different

solution can be reliably fabricated by sol–gel spin coating process. The results are of value to deepen the understanding of the effect of film thickness on the photocatalytic activity of TiO₂ thin films. On the basis of the studies, it can be concluded that the calcination temperature affects the optical and structural properties. Titanium dioxide thin films can be used for in catalysis, antibacterium, anticorrosion, photovoltaic cells, self-cleaning surface and water purification [1]-[3].

Acknowledgments

The research presented in this article was financed by the National Science Centre, Poland based on the decision number 2014/15/B/ST8/04767.

References

- [1] Ulrike Diebold, The surface science of titanium dioxide, Surface Science Reports 48 (2003) 53 - 229
- [2] Hosseini, K.Ç. Içli, M. Özenbaş, Ç. Erçelebi, Fabrication and Characterization of Spin-coated TiO₂ Film, Energy Procedia Volume 60 (2014)
- [3] G. Kenanakis, D. Vernardou, A. Dalamagkas, N. Katsarakis, Photocatalytic and electrooxidation properties of TiO₂ thin films deposited by sol–gel, Catalysis Today Volume 240 Part A (2015) 146 – 152

Penetration of carbon nanostructures into lipid bilayer cell membranes: A molecular dynamics study

A. MONTAZERI^{*1,2}, **B. MEHRAFROOZ**³, **A. VASEGH**¹ nbcvbn

¹*Faculty of Materials Science and Engineering, K.N. Toosi University of Technology, Tehran, Iran*

²*School of Nano-Science, Institute for Research in Fundamental Sciences (IPM), Tehran, Iran*

³*Faculty of Mechanical Engineering, K.N. Toosi University of Technology, Tehran, Iran*

*a_ montazeri@kntu.ac.ir

With the emergence of engineered carbon nanomaterials such as carbon nanotubes (CNTs) and fullerene into the modern drug delivery systems, treatment of disease through cell membrane injection is now a reality. The extraordinary ability of these engineered carbon nanomaterials to cross the cell membrane acting as a drug carrier leads to a plethora of application in this scope. Therefore, investigating the interaction of engineered carbon nanomaterials with cell membrane has become a pressing challenge. Here, we address this issue through a series of molecular dynamics (MD) simulations. In this work, a synergy between biological concepts and MD simulation is employed to study and compare the penetration of a CNT and fullerene into a pure cell membrane under various injection velocities, with a more emphasis on the minimization of disruption in the lipid bilayer. The main aim is to derive guidelines for optimum design of the engineered carbon nanomaterials dedicated to penetrate the cell membrane. To this end, from mechanical point of view, deformation of the lipid bilayer, force and stress exerted on the nanocarrier, and its corresponding deformation are examined during the penetration.

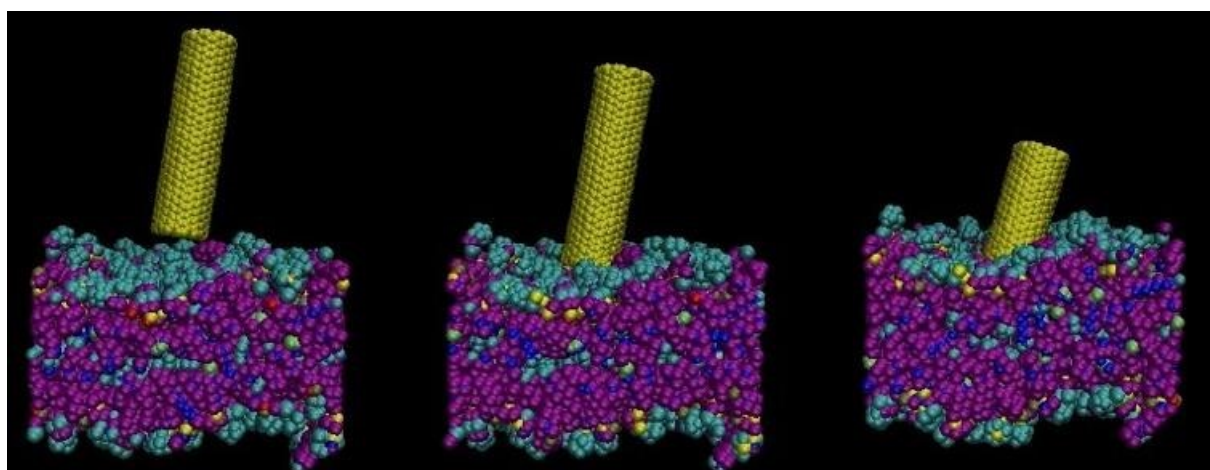


Fig. 1: Snapshots of penetration of SWCNT into a cell membrane

ARMSCOPE - The versatile platform for custom SPM systems

T. PIASECKI^{*1}, M. RUDEK¹, M. ŚWIĄTKOWSKI¹,
K. GAJEWSKI¹, W. MAJSTRZYK¹, M. BABI¹, A. DZIERKA¹, T. GOTSZALK¹

¹*Faculty of Microsystem Electronics and Photonics, Wrocław University of Science and Technology, Janiszewskiego 11-17, 50-372 Wrocław, Poland.*

^{*}tomasz.piasecki@pwr.edu.pl

Keywords: SPM, AFM, SThM, STM, nanometrology

ARMSCOPE is being developed at Nanometrology Group at the Faculty of Microsystem Electronics and Photonics, Wrocław University of Technology, Poland. The main goal is to build a versatile, modular system for SPM measurements. Its backbone is an ARM-microprocessor based main unit with universal system bus used to communicate with other components of the system: programmable arbitrary scanner controller „Compass”, digital or analogue PID controllers with high voltage amplifiers, precise 18-bit ADC module, DC motors controller, and others, depending on the specific needs. ARMSCOPE is controlled and the measurement results are visualised by dedicated Microsoft Windows - based application which communicates with the custom firmware in the system controller.

The versatility of the ARMSCOPE system allows for using it in various measurement modes. In conjunction with precise current-to-voltage converter it may work as an Scanning Tunnelling Microscope (STM) and Scanning Tunnelling Potentiometry (STP) microscope able to reach atomic scale resolution (Figure 1). It may also work as an Atomic Force Microscope (AFM) with various scanners and using both optical and piezoresistive deflection readout [1]. It may work in various measuring modes, for example Kelvin Probe Force Microscopy mode (KPFM), as a Conductive AFM (C-AFM), Scanning Thermal Microscope (SThM - Figure 2) [2], Piezoresponse Force Microscopy (PFM) and others. Using multi-channel analogue-to-digital converter allows for simultaneous multicantilever scanning and/or sensing [3].

The results of the system characterization and exemplary measurements are presented.

Acknowledgements: Authors would like to acknowledge the former Nanometrology Group members: Michał Zielony, Paweł Zawierucha, Grzegorz Wielgoszewski and Daniel Kopiec. Their efforts contributed significantly in the SPM systems development.

References

- [1] Gotszalk, T., et al., *Shear force microscopy using piezoresistive cantilevers in surface metrology*, Proceedings of SPIE - The International Society for Optical Engineering 9236 (2014) 92360A
- [2] Wielgoszewski, G. et al., *Carrier density distribution in silicon nanowires investigated by scanning thermal microscopy and Kelvin probe force microscopy*, Micron 79 (2015) 93-100
- [3] Nieradka K., et al., *Expanded beam deflection method for simultaneous measurement of displacement and vibrations of multiple microcantilevers*, Review of Scientific Instruments 82 (2011) 105112

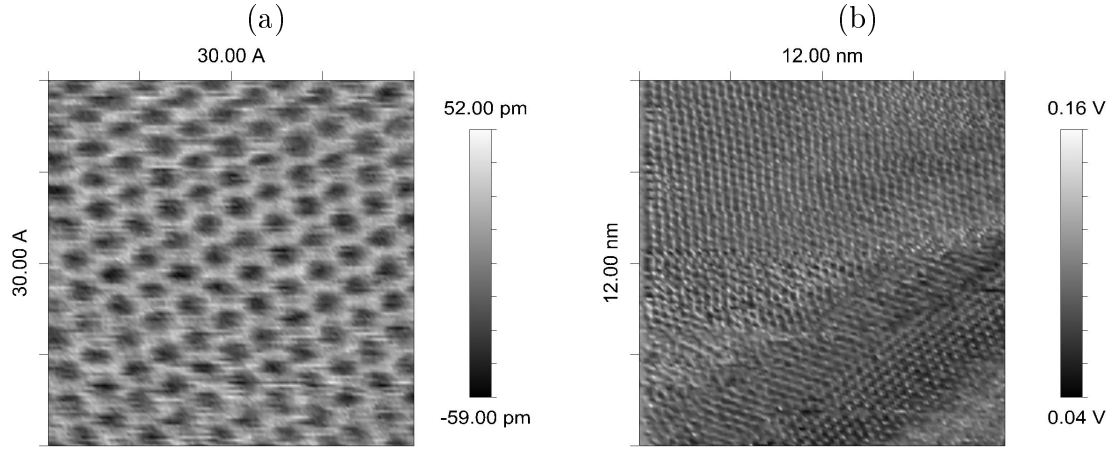


Fig. 1: Exemplary scan of HOPG surface at room conditions obtained using the ARM-SCOPE system working in STM mode showing the atomic scale resolution: topography signal at 3×3 nm scan (a) and tunnelling current signal at 12×12 nm scan showing the grain boundaries (b).

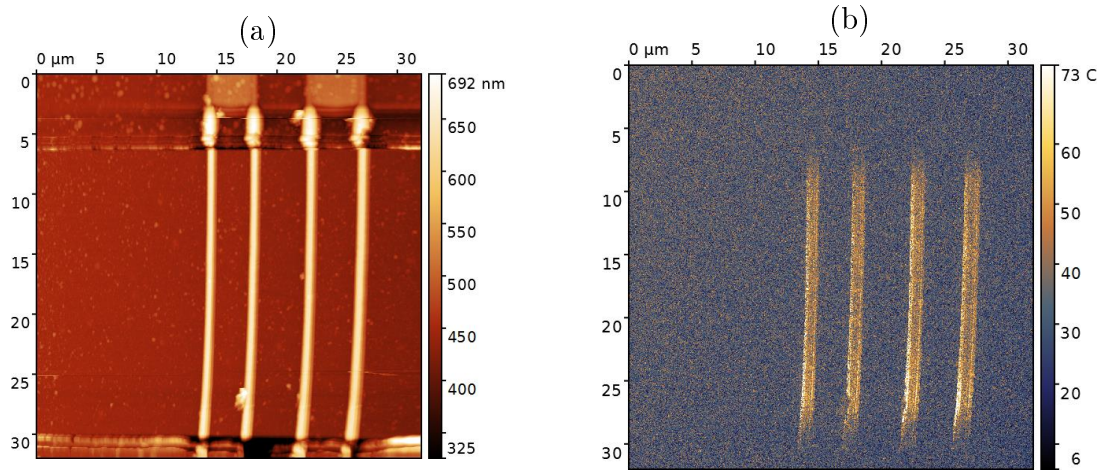


Fig. 2: Topography (a) and temperature distribution (b) on the FinFET devices obtained using ARMSCOPE system showing the temperature rise at the bottom of the channels due to the pinch-off effect [2]

Study of electrostatic effects on the surface of UV treated polyethylene

M. BŁASZCZYK¹, M. DURKO¹, Z. IWANICKA¹,
A. PIOTROWIAK¹, A. SIKORA^{2,*}

¹*Active Students Association, SEP Branch no. 1 in Wrocław, M. Skłodowskiej-Curie 55/61, Wrocław, 50-369, Poland*

²*Division of Electrotechnology and Materials Science, Electrotechnical Institute, M. Skłodowskiej-Curie 55/61, Wrocław, 50-369, Poland Poland*

*sikora@iel.wroc.pl

Keywords: Polyethylene, UV radiation, AFM, electrostatic effects

In this work a study of occurrence of electrostatic effects on the surface of UV irradiated polyethylene will be presented. In order to observe said process, the polyethylene samples were irradiated with ultraviolet light, then the studies were performed using an atomic force microscope (AFM), which enables the surface-scanning tip detection with high sensitivity, and provides detection a set of complex and subtle interactions of the material with the surrounding. Moreover, a set of standard methods was used in order to measure the electrical properties of investigated samples and to observe the impact of the phenomena observed at nanoscale. On the basis of the research conducted, it was found that material degradation is a more complex process than the hitherto adopted model. Obtained data were compared in order to select the most appropriate approach for further investigation of observed phenomena.

Acknowledgments

We thank Electrotechnical Institute for the opportunity to perform research needed for the purposes of this paper. The authors also thank Association of Polish Electrical Engineers SEP, Wrocław section and branch no. 1 in Wrocław for financial support.

References

- [1] J.F. Rabek, Współczesna wiedza o polimerach (WN PWN, Warszawa, 2008)
- [2] R. Sharma, R. A. Sims, M. K. Mazumder, Modification of Surface Properties of Polymeric Materials, Journal of the Arkansas Academy of Science Vol. 56(2006)
- [3] Ch.-Y. Liu, A. J. Bard, Electrostatic electrochemistry: Nylon and polyethylene systems, Chemical Physics Letters 485 (2010) 231–234
- [4] D. Briggs, Surface treatments for polyolefins in ‘Surface analysis and pretreatment of plastics and metals’(MacMillan Publishing, New York, 1982)
- [5] P. J. Zilinkas, T. Lozovski, J. Jurksus, Electrostatic Properties and Characterization of Specific Polymeric Materials for Building Purposes, Materials Science Vol. 16 (2010), 1392–1320

Detection of chemical agents, volatile organic compounds, and explosives with nano-functionalized, self-sensing cantilever

Mathias Holz a), Tzvetan Ivanovb), Ahmad Ahmada), Alexander Reum a,b), Nikolay Nikolovc), and Ivo W. Rangelow b)*

M. HOLZ¹ A), T. IVANOV¹ B), A. AHMAD¹A), A. REUM^{1,2} A,B), N. NIKOLOV³, I. W. RANGELOW²)

¹Nano Analytik GmbH, Ehrenbergstraße 11, 98693 Ilmenau, Germany

²Department of Micro- and Nanoelectronic Systems (MNES), Institute of Micro and Nanoelectronics, Ilmenau University of Technology, Gustav-Kirchhoff-Str.1, 98693 Ilmenau, Germany ³ Mikrosistemi, 9010-Varna, Bulgaria

*ivo.rangelow@tu-ilmenau.de

Keywords: Self-sensing cantilever-resonators, Active Nanocantilever, Masss-Metrology

Explosives detection has been improved thanks to the development of new self-sensing nano-cantilever techniques capable of performing very sensitive detection and quantifying certain chemical-affinity parameters. In this paper, we will present the progress in detection of chemical agents, volatile organic compounds, and explosives for civil security. The amplitude and the phase shift of the cantilever oscillation can be correlated with the presence of specific molecules and their quantity. Most of the enthusiasm comes from new possibilities and properties of nano-mechanics. Sensors (simple, robust, sensitive, cost-effective) combined with nanotubes, also called nano-functionalised cantilever-sensors, are serving as bridge between advanced detection/diagnostics and daily safety uses. Here, we present some of the latest developments of nano-factionalized nano-dimension cantilevers. Nanoelectromechanical cantilevers as NEMS devices, oscillating at high frequencies, are skilled to be of levels better of mass sensitivity compared to many larger commercially available cantilevers. The goal of this development was to realize sensors capable to achieve a mass resolution comparable to the mass resolution of a mass spectrometer. Around 60 years ago from now the measurements of small masses started by using Quartz Crystal Microbalance sensors having a mass sensitivity (S) in the order of $S \approx 1010 \text{ Hz/g}$ -- which are still wide spread in many Physical Vapor Deposition tools. Furthermore, knowing the sensitivity of the sensor the recalculation of the actual mass change is possible. However, during the resent years different approaches have been made from different research groups by using different materials with different shaped mass sensors. In many cases the mass detection requires sophisticated electronics- and experimental setup and often they are limited to ultra-high vacuum environment. Here, we present a new approach of a nano sized silicon based mass cantilever using the piezoresistive quantum-size effect readout to measure its resonance frequency, which is in the order of 5 MHz to 120 MHz (Fig.1). The small cantilever is driven externally by a thermal-bimorph or piezo-electrical method. A special developed by nano analytik GmbH, Ilmenau, 125-MHz FPGA based electronic with ultra-fast Phase-Locked Loop (PLL) and software with wide bandwidth and digital signal filtering allows a continuous (almost real time) measurement of the sensors resonance frequency. This electronic is capable to detect the self-thermo-mechanical oscillations of the cantilever. We achieve thermal noise-limited detection of mechanical resonances in these devices at room conditions. A change of the sensors mass is immediately highlighted in the analyses software, where the actual mass change is calculated from the amount of change in frequency and knowing the mass sensitivity. The calibration

of the mass sensor showed a mass sensitivity of $S = 11.31 \times 10^{-18} \text{ g/Hz}$. We used a new fabrication step which includes a 3D micro- and nano structuring process to form Oxide-Nano-Tubes (ONT) of the surface of the mass sensor. The 3D-NT structures are employed as highly selective absorber of a certain kind of molecules. The mass sensitive sensors are very promising to be used in many fields of application. Although being an emerging discipline, self-sensing nano- cantilevers has raised huge interest and expectations.

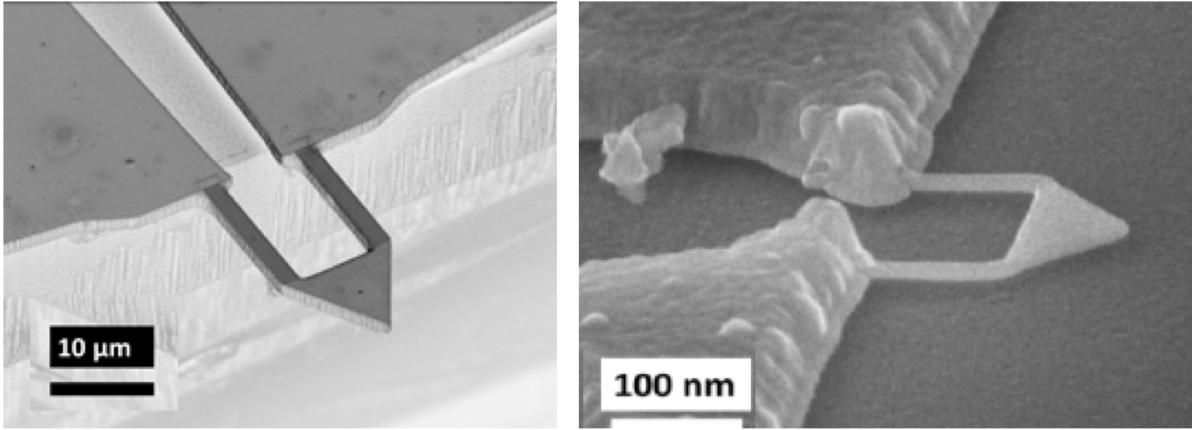


Fig. 1: Scanning electron micrograph of diverse cantilevers with dimensions from 30μm to 300 nm.

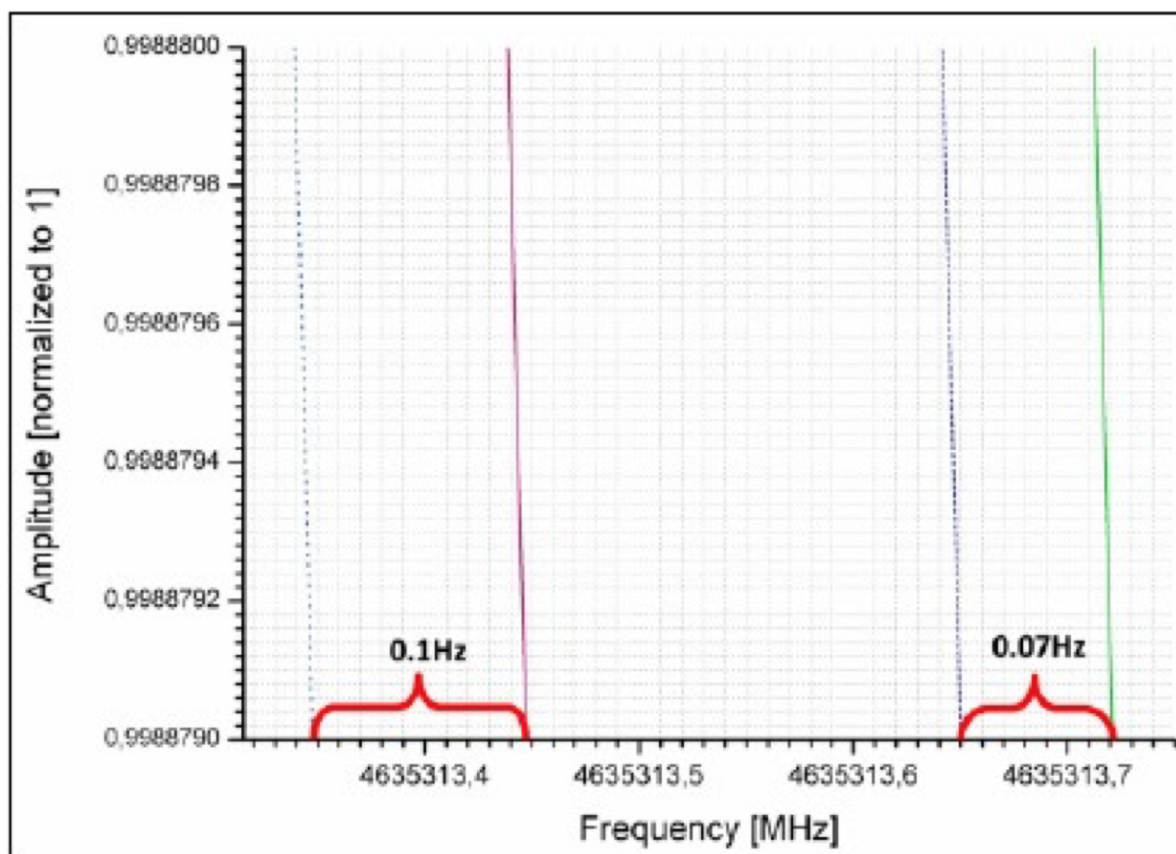


Fig. 2: SMass induced resonance frequency shift due to absorption of volatile organic compound. The measured frequency shift of 7 mHz is equal to 791zg mass change.

Electrostatic Force Interactions in Field-Emission Scanning Probe Lithography with Micromechanical Cantilevers

P. MARINUSHKIN¹, M. HOLZ², A. AHMAD¹, A. REUM^{1,2}, T. IVANOV¹, E. GULYIEV¹, V. ISHCHUK¹, M. KAESTNER¹, M. HOFMANN¹, C. LENK¹, S. LENK¹, I. W. RANGELOW¹

¹*Department of Micro- and Nanoelectronic Systems (MNES), Institute of Micro and Nanoelectronics, Ilmenau University of Technology, Gustav-Kirchhoff-Str.1, 98693 Ilmenau, Germany*

²*Nano Analytik GmbH, Ehrenbergstraße 11, 98693 Ilmenau, Germany*

*ivo.rangelow@tu-ilmenau.de

Keywords: Scanning probe lithography with cantilevers, active nano-cantilever

The proposed system of Scanning Probe Lithography (SPL) [3] based on active micro-mechanical cantilevers in this connection is of great interest. In this method, a cantilever is held near the surface on which the resist is applied, and its local exposure is carried out by the current flowing between the tip of the cantilever and the wafer. Key features of the SPL method are combining a reading/writing functions and high positional accuracy (atomic force microscope determines the coordinates of points with an accuracy of 1 nm), which allows in a single scanning cycle to plot the topography of the sample surface, to select the site of interest and then expose it. In SPL lithography mode flexural deformation of the cantilever occurs under the influence of electrostatic forces. Since the intensity of this electrostatic load increases with deflection, it has a destabilizing influence on the cantilever. Consequently, electrostatically induced bending affects the pattern formation process and is one of the contributors to the instability of SPL. In particular, it can result in shortening the circuit and can lead to unexpected probe behaviour or damaging the tip. Therefore it is important to reduce the influence of the electrostatically induced bending. For this reason accurate estimations of voltage and deflection of the cantilever at this state are of high importance for designing the SPL. Electric potential applied between the substrate surface and the cantilever gives rise to electrostatic forces acting on the cantilever. The elastic restoring force induced in the cantilever balances the electrostatic force and restricts cantilever's motion. With decreasing the gap, the electric charges redistribute on the deflected cantilever's surface which entails the increase of the electrostatic force and further deflection of the cantilever until the electrostatic force balances the elastic restoring force. The corresponding values of the voltage and the peak deflection of the cantilever are called the pull-in voltage and the critical gap respectively. Due to the fact that as the cantilever beam deforms, the electrostatic charges redistribute causing an increasingly non-uniform distribution of electrostatic forces and, thus altering the mechanical loads, it would be very difficult to compose the analytical model which explains the behavior of the cantilever in electrostatic field, especially in all three spatial dimensions. Therefore finite-element analysis is appropriate to use to explain the unstable scenery and quantify the pull-in parameters [5]. The practical realization of the finite element method was performed using the commercial finite element package COMSOL Multiphysics [4]. Figure 2, shows the example of the results obtained for the electric potential and deflection of the cantilever with an applied DC bias. As the color legend indicates, the bending of the cantilever reaches about 5 nm onto a distance of 25 nm from the surface under a +45 V bias voltage. From the high density of equipotential surfaces beneath the cantilever

it is also clear that electric field is mainly concentrated around this region. Enlarged images of the potential and electrostatic field distribution maps show how the electric field is distributed in the gap between the tip and the surface. It can be seen from Figure , that high concentration of electric field appears around the tip apex which is due to the tiny tip surface area. The electric field strength reaches a value of about $4.5 \times 10^9 \text{V/m}$.

In this article we will present the combination of parameters of the cantilever, providing a trade-off between the imaging and lithography capabilities. The advantage of using cantilevers with longer tips and larger inclination angle in terms of decreasing the susceptibility of the cantilever to electrostatic field will be demonstrated. Moreover, it will be shown that the 2D approximation is sufficient to provide an estimate of the parameters describing the electrostatically induced bending what is important in the light of the need to balance discretization quality and computational costs due to large difference in dimensions between the cantilever and tip. Results also highlight the fact that the tip does not play a relevant role for the modeling of the cantilever static deflection under electrostatic force.

Acknowledgments

We thank Electrotechnical Institute for the opportunity to perform research needed for the purposes of this paper. The authors also thank Association of Polish Electrical Engineers SEP, section in Wrocław and branch no. 1 in Wrocław for financial support.

References

- [1] M. Kaestner, T. Ivanov, A. Schuh, A. Ahmad, T. Angelov, Y. Krivoschapkina, M. Budden, M. Hofer, S. Lenk, J.-P. Zoellner, I. W. Rangelow, Scanning probes in nanostructure fabrication, *Journal of Vacuum Science & Technology B, Nanotechnology and Microelectronics: Materials, Processing, Measurement, and Phenomena* 32 (2014). doi: 10.1116/1.4897500
- [2] T. Michels, I. W. Rangelow, Review of scanning probe micromachining and its applications within nanoscience, *Microelectronic Engineering*, Volume 126, 25 (2014) 191–203. doi: 10.1016/j.mee.2014.02.011.
- [3] M. Kaestner, K. Nieradka, T. Ivanov, S. Lenk, Y. Krivoschapkina, A. Ahmad, T. Angelov, E. Guliyev, A. Reum, M. Budden, T. Hrasok, M. Hofer, C. Neuber, I. W. Rangelow, Electric field scanning probe lithography on molecular glass resists using self-actuating, self-sensing cantilever, *Proc. SPIE 9049, Alternative Lithographic Technologies VI* (2014). doi: 10.1117/12.2046973
- [4] COMSOL Multiphysics Reference Guide
- [5] Wen-Ming Zhang, Han Yan, Zhi-Ke Peng, Guang Meng, Electrostatic pull-in instability in MEMS/NEMS: A review, *Sensors and Actuators A: Physical*, Volume 214 (2014) 187–218. doi: 10.1016/j.sna.2014.04.025.

Fig. 1: Example of the simulation results (dimensions are indicted in microns). a) Surface plot showing electric potential distribution within probe–surface gap; b) Amplitude of the electric field underneath the tip (the localized field enhancement can be clearly seen around the tip).

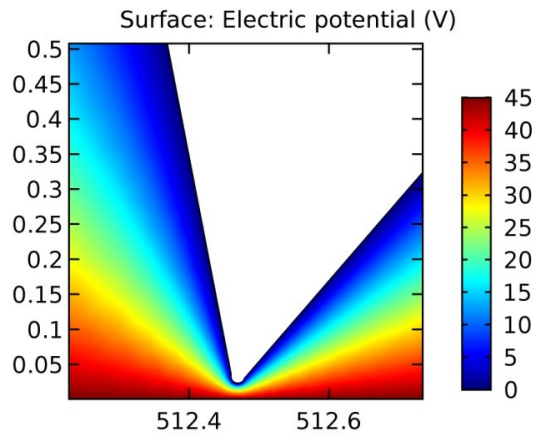


Fig. 2: a)

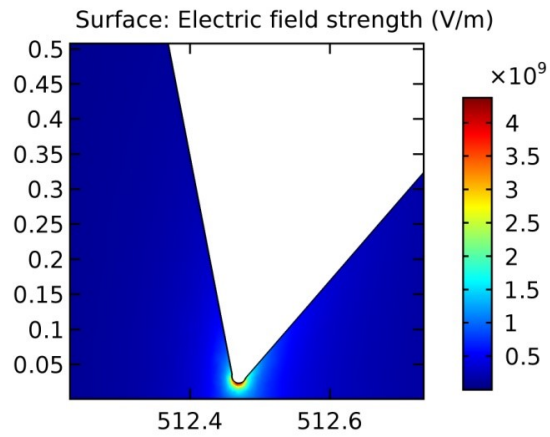


Fig. 3: b)

Microfabricated stage for scanning thermal microscopy calibration

M. RUDEK^{1,*}, **A. ZIĘBOWICZ**², **B. ZIĘBOWICZ**³,
P. JANUS⁴, **A. SIERAKOWSKI**⁴, **T. GOTSZALK**¹ **D. SZMIGIEL**⁴

¹ *Wrocław University of Science and Technology / Faculty of Microsystem Electronics and Photonics, 50-372 Wrocław, Poland* ² *Department of Biomaterials and Medical Devices Engineering, 44-800 Zabrze, Poland* ³ *Institute of Engineering Materials and Biomaterials, 44-100 Gliwice, Poland* ⁴ *Institute of Electron Technology, 02-668 Warszawa, Poland*

*maciej.rudek@pwr.edu.pl

Keywords: zirconium dioxide, AFM, SThM, scanning thermal microscopy, thermal calibration

Specialized measurement technique called Scanning Thermal Microscopy (SThM) requires calibration process of the thermal sensor located on the end of the probe. Mostly there are resistive made of gold, platinum or platinum-iridium coating. The dimension of this sensor is smaller than $5\mu m$ in this reason it should be made calibration temperature stage in micro regime. Repeatedly the thermal stage are larger than cantilever or even holder. In this reason should be made active stage with active area smaller than $50nm$ and separate parasitic heat from active element to the cantilever. The case plate was made using CAD/CAM technology preparation process from zirconium dioxide. This is a white crystalline used for multifunctional applications. Its most naturally occurring form, with a monoclinic crystalline structure, but ZrO_2 is often more useful in its phase 'stabilized' state. Upon heating, zirconia undergoes disruptive phase changes. By adding small percentages of yttria (Y_2O_3), the structure transitions from tetragonal to monoclinic phase are eliminated, and the resulting material has superior thermal, mechanical, and electrical properties [1][2]. In prepared plate with $500\mu m$ holes was glued micro copper rods which got very high conductivity ($401W/(m \cdot K)$) in the polymer with low thermal conductivity ($< 5W/(m \cdot K)$).

In this paper we present the results of investigation active sample. The measurements was made using the SThM which stems directly from AFM. Report technique, permissive high resolution topography and thermal investigations, utilizes novel piezoresistive SThM probes with piezoresistive detector of deflection. Which are manufactured in standard technology: MEMS, CMOS and Focus Ion Beam in post-processing fabrication steps[3]. Moreover the probes allow to obtain high resolution thermal structure measurements. Presented methodology and techniques allow to obtain quantitative temperature and thermal conductivity of investigated samples in nanoscale. In the work was performed the necessary tests to establish that the proposed solution may be an alternative to monolithic heaters.

Acknowledgments

The work has been supported by Grant no. 0401/0029/17 at Wrocław University of Science and Technology.

References

- [1] Chevalier J., Gremillard L, Virkar A.V., Clarke D.R., The tetragonal-monoclinic transformation in zirconia: lessons learned and future trends, *J. Am. Ceram. Soc.*, 2009, vol. 92, no 9, pp. 1901–1920.
- [2] Kelly J.R., Denry I., Stabilized zirconia as a structural ceramic: an overview, *Dent. Mater.*, 2008, 24, pp. 289–298.
- [3] P. Janus, P. Grabiec, A. Sierakowski, T. Gotszalk, M. Rudek, D. Kopiec, W. Majstrzyk, G. Boetsch, B. Koehler, Design, technology, and application of integrated piezoresistive Scanning Thermal Microscopy (SThM) microcantilever, *Scanning Microscopies 2014, Proc. of SPIE Vol. 9236, 92360R*
- [4] Janus, P., Sierakowski, A., Grabiec, P., Rudek, M., Majstrzyk, W., Gotszalk, T. Micromachined active test structure for scanning thermal microscopy probes characterization, (2017) *Microelectronic Engineering*, 174, pp. 70-73.

Realization of sub-micron photolithography using GCA-4800 wafer stepper

L. RUTA^{*1}, A. KUBIAK¹, Z. LISIK¹,

¹*Lodz University of Technology, Department of Semiconductor and Optoelectronic Devices, Wolczanska 211/215, 90-924 Lodz, Poland*

^{*}lukasz.ruta@p.lodz.pl

Keywords: silicon technology, photolithography, photoresist, wafer stepper

Nowadays the silicon technology is very mature and well-known. Various structures are manufactured, from simple discrete elements to advanced and sophisticated integrated circuits and sensors containing thin layers. The various processes are in everyday use for the purpose of manufacturing of such a circuits. These are: wet cleaning processes for the preparation of the substrates for production, wet and dry etching, ion implantation, oxidation, thermal processing, thin layers growth as well as photolithography, considered to be the most crucial part of the production process in microelectronics. Photolithography can be performed with the use of mask aligner or wafer stepper. Modern IC's manufactured today are mostly processed using the most advanced "photo" methods, which allow to obtain the resolution of tens of nm. However, many laboratories are still equipped with photolithographic tools that offer much worse resolution, typically in the range of 1 μm .

The goal of this work was to verify the capabilities of the GCA-4800 stepper, whether the standard resolution declared by the manufacturer as ca. 1 μm can be lowered so to obtain structures of good, repeatable quality with resolution in the range of hundreds on nanometers. For this purpose the series of experiments were done with the use of various photosensitive emulsions (photoresists). Moreover, the influence of the process parameters on the final lithography effect were investigated. The verification of photoresists, provided by two various suppliers: Microresist and Microchemicals, was conducted. Fig. 1 presents the photos of the samples obtained after photolithography process done with the use of GCA-4800 wafer stepper.

Obtained samples were observed using optical and SEM microscopes. Our experiment proved, that providing proper selection of the photosensitive emulsion together with optimized process parameters, GCA-4800 wafer stepper allows to fabricate structures with as low resolution as 700 nm.

Acknowledgments

This work was also supported by National Centre for Research and Development "Ultra-fast Photodetector based on graphene" GRAF-TECH/NCBR/13/20/2013.

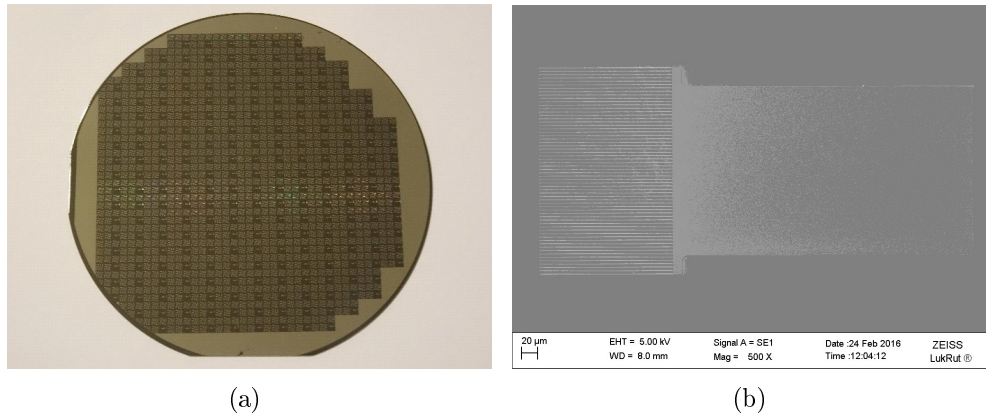


Fig. 1: Samples fabricated using GCA-4800 wafer stepper:
a) structures fabricated on Si wafer;
b) 700nm width metalization realized using GCA-4800.

Utilization of plasma sheet in the quasi-tomography of the polymer-based nanocomposites

A. SIKORA^{*1}, M. MOCZAŁA¹

¹*Division of Electrotechnology and Materials Science, Electrotechnical Institute, M. Skłodowskiej-Curie 55/61, Wrocław, 50-369, Poland*

^{*}sikora@iel.wroc.pl

Keywords: nanocomposites, polymers, AFM, plasma sheet

The growing market of nanomaterials and nanocomposites is due their unique mechanical, electrical, optical and thermal properties. In order to develop the technology, appropriate diagnostic methods providing essential information about the fabricated materials are requested. One of the most important issues in the process optimization is providing the nanofiller's homogenous distribution. This information can be obtained by using 3D mapping of the mechanical properties of the material. Although the microtomography [?] became very popular micrometer-resolution imaging tool in terms of analysis of volume related data, the detection resolution of such device may be insufficient when fine, nanometer size feature are created in the material. On the other hand, the best introduced diagnostic solution of the submicron mechanical properties imaging is the group of atomic force microscopy measurement modes such as: phase imaging, force volume, force modulation mode, Harmonix [4], NanoSwing [5], PeakForce QNM and others. In order to enable the access inside of the material, one needs a specific method of the matter removal. Combined AFM and ultramicrotome solution was successfully introduced [6], however its application range is limited to the range of samples of specific size and stiffness. Also so called Nanojet [7] system was developed, which enabled the local plasma etching and AFM imaging in one instrument. In this paper we present the solution of much wider application range, as the surface etching bases on the plasma blade [8, 9], which allows to perform the imaging of mechanical properties of the material. This tool enables processing the samples up to 60 mm in width in a single sweep. By cyclic procedure of removing material and surface imaging, one is able to obtain the quasi-tomographical data, providing and insight into the 3D composition of the material. The example of the sample surface's etching results and revealing the nanoparticles embedded in the polymer matrix is shown in figure 1.

Acknowledgments

This work was partially supported by a statutory activity subsidy from the Polish Ministry of Science and Higher Education for the Electrotechnical Institute and National Science Centre – grant number DEC-2015/19/B/ST8/02123.

References

- [1] P. Soltani, M. S. Johari, M. Zarrebini, Effect of 3D fiber orientation on permeability of realistic fibrous porous networks, *Powder Technol.*, 254 (2014) 44-56 doi: 10.1016/j.powtec.2014.01.001
- [2] T. Fałat, P. Matkowski, B. Płatek, C. Zanden, J. Felba, L.-L. Ye, J. Liu, Investigation on interaction between indium based thermal interface material and copper and silicon substrates, *Proc. of European Microelectronics Packaging Conference, EMPC2013* (2013),1.
- [3] A. Sikora, L. Bednarz, T. Fałat, M. Walecki, M. Adamowska, The investigation of the simulated solar radiation impact on the micro- and nanoscale morphology and mechanical proper-

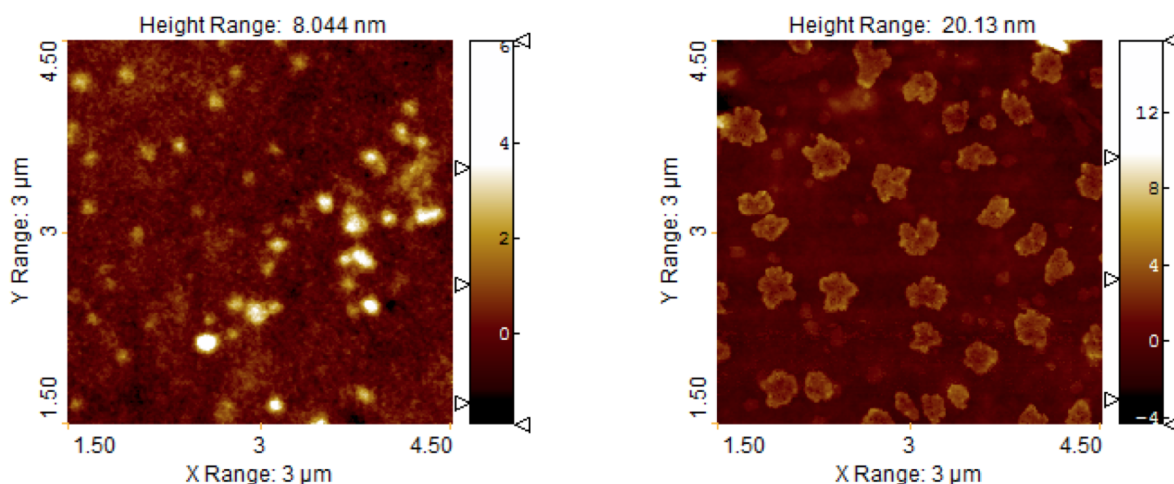


Fig. 1: The AFM topography of the polyazomethine with PCBM nanofiller before (left) and after (right) plasma etching.

ties of the sheet moulded composite surface, *Materials Science-Poland*, 34(3) (2016) 641-649 doi: 10.1515/msp-2016-0080

- [4] O. Sahin, S. Magonov, C. Su, C. F. Quate, O. Solgaard, An atomic force microscope tip designed to measure time-varying nanomechanical forces, *Nature Nanotechnol.* 2(8) (2007) 507–14 doi:10.1038/nnano.2007.226
- [5] A. Sikora, Ł. Bednarz, Mapping of mechanical properties of the surface by utilization of torsional oscillation of the cantilever in atomic force microscopy, *Central European Journal of Physics*, 9(2), (2011) 372-379 doi: 10.2478/s11534-010-0127-4
- [6] A. E. Efimov, A. G. Tonevitsky, M. Dittrich, N. B. Matsko, Atomic force microscope (AFM) combined with the ultramicrotome: a novel device for the serial section tomography and AFM/TEM complementary structural analysis of biological and polymer samples, *J Microsc.* 226 (2007) 207-17 doi:10.1111/j.1365-2818.2007.01773.x
- [7] J. Voigt, F. Shi, P. Hudek, I. W. Rangelow, Progress on nanostructuring with Nanojet, *Journal of Vacuum Science & Technology B*, 18, 3525 (2000); doi: 10.1116/1.1319823
- [8] M. Jasiński, M. Goch, J. Mizeraczyk, Urządzenie mikrofalowe do generacji płaszczyzny plazmowej, *Prz. Elektrotechniczny* 86 (2010) 112–114
- [9] M. Jasiński, J. Mizeraczyk, Plasma sheet generated by microwave discharge at atmospheric pressure, *IEEE Trans. Plasma Sci.* 39(11) (2011) 2136–2137 doi: 10.1109/TPS.2011.2162006

Integrated electrochemical sensors in biochemical diagnostics

P. SZYMANOWSKA^{1,*}, D. NOWAK^{1,*}, T. PIASECKI^{1,*}, T. GOTSZALK^{1,*}

¹*Faculty of Microsystem, Electronics and Photonics, Wrocław University of Science and Technology ul. Janiszewskiego 1/17 50-372 Wrocław, Poland*

*paulina.szymanowska@pwr.edu.pl, damian.nowak@pwr.edu.pl,
tomasz.piasecki@pwr.edu.pl, teodor.gotszalk@pwr.edu.pl

Keywords: electrochemical impedance spectroscopy, integrated electrochemical sensor, LTCC ceramic

Miniaturized and integrated analytical devices, including chemical sensors, are at the forefront of modern analytical chemistry. The construction of novel analytical tools takes advantage of contemporary micro- and nanotechnologies, as well as materials science and technology. Typically the integrated electrochemical sensors are fabricated on silicon [1],[2] and LTCC ceramic [3],[4] surface.

The design of the integrated electrochemical sensor for Electrochemical Impedance Spectroscopy (EIS) and cyclic voltammetry measurements was presented in this work. The diameter of sensor was 5.1 mm. This electrochemical sensor contains a gold (Au) working microelectrode (WE), silver/silver chloride (Ag/AgCl) reference microelectrode and platinum (Pt) counter microelectrode (CE). Working electrode, reference electrode and counter electrode was fabricated on LTCC ceramic surface. Portable EIS system consisting of the impedance analyser and prototype single channel potentiostat was built and tested. The preliminary results of the Ag/AgCl reference electrode evaluation were presented.

References

- [1] Baraket, A., Lee, M., Zine, N., Sigaud, M., Bausells, J., Errachid, A. (2017). A fully integrated electrochemical biosensor platform fabrication process for cytokines detection. *Biosensors and Bioelectronics*, 93, 170-175.
- [2] Ziółkowski, R., Górski, Ł., Prokaryn, P., Zaborowski, M., Kutyla-Olesiuk, A., Ciosek, P., Wróblewski W., Malinowska, E. (2013). Development of silicon-based electrochemical transducers. *Analytical Methods*, 5(20), 5464-5470.
- [3] Pesquero, N. C., Gongora-Rubio, M. R., Yamanaka, H. (2013). A novel LTCC electrochemical cell construction and characterization: a detection compartment for portable devices. *Analyst*, 138(15), 4298-4304.
- [4] Manjakkal, L., Synkiewicz, B., Zaraska, K., Cvejic, K., Kulawik, J., Szwagierczak, D. (2016). Development and characterization of miniaturized LTCC pH sensors with RuO₂ based sensing electrodes. *Sensors and Actuators B: Chemical*, 223, 641-649.

MEMS-based setup for nanocalorimetric monitoring of living cell activity

M. Świątkowski a), W. Chomicki b), M. Rudek a), K. Kapczyńska c),
Giovanni Dietler b), Teodor Gotszalk a)

**M. ŚWIĄTKOWSKI^{1,*}, W. CHOMICKI², M. RUDEK¹,
K. KAPCZYŃSKA³, G. DIETLER², T. GOTSZALK¹**

¹*Nanometrology Division, Wrocław University of Science and Technology, Wrocław, Poland*

²*Laboratory of Physics of Living Matter, EPFL, Lausanne, Switzerland*

³*Ludwik Hirszfeld Institute of Immunology and Experimental Therapy PAS, Wrocław, Poland*

*michal.swiatkowski@pwr.edu.pl

Keywords: nanocalorimetry, living cell heat, MEMS

Nanocalorimetric monitoring of living cells bacteria activity is a method that allows detection of heat produced by metabolic processes. Single bacterium can produce about 2 pW in a logarithmic growth phase [1]. In order to detect such a small amount of heat, MEMS-based sensors should be used. We have estimated resistance change of the sensor covered by bacteria colony to be in order of few miliohms.

In our work we have developed experimental setup for living cell activity monitoring. It is based on a MEMS doubly-clamped bridge with platinum resistor as a temperature sensor. Resistance change of single miliohm can be measured in a Wheatstone bridge which can be precisely tuned and set in the AC operating mode. This mode allows usage of a lock-in amplifier and resolution of the setup is further enhanced. Data acquisition was performed with 14-bit ADC converter from National Instruments NI-PCI 6251 and experiment was controlled by LabVIEW software.

For the experiments we have used *E. Coli PCM 2057* species, which is a standard for antibiotic activity tests, cultured in lysogeny broth. We have used standardized procedure for cell adhesion onto MEMS sensor surface developed at the EPFL. Sensor covered with adhered bacteria and dipped into few μL volume of broth was enclosed in a 3-D printed liquid cell. Recorded signal was processed to extract signal variance which carries information about bacteria heat production fluctuation and metabolic activities.

In our presentation experimental setup will be described. We will also present results of living bacteria monitoring performed with doubly-clamped bridge MEMS sensors.

Acknowledgments

This work was partially funded with Nanometrology Division statutory funds - 0401/0234/16.

References

- [1] J. Lerchner, Chip-calorimetric evaluation of the efficacy of antibiotics and bacteriophages against bacteria on a minute-timescale, *J Therm Anal Calorim* (2011) 104:31–36

Galvanically isolated system for scanning thermal microscopy resistive probes based on a transformer ratio-arm bridge

M. ŚWIĄTKOWSKI ^{1,*}, M. RUDEK¹, G. WIELGOSZEWSKI ¹,
A. WOJTUŚ ², T. PIASECKI ¹, T. GOTSZALK ¹

¹*Nanometrology Division, Wrocław University of Science and Technology, Wrocław, Poland*

*michal.swiatkowski@pwr.edu.pl

Keywords: AFM, SThM, transformer bridge, Drobnowidz AFM

Atomic force microscopy is a widely used technology for investigation and characterization of the nanomaterials. Its functionality can be easily expanded by applying dedicated extension modules, which can measure electrical conductivity or temperature of the sample. In this paper we introduce a transformer ratio-arm bridge setup dedicated for AFM-based thermal imaging. The key features of the thermal module are: low-power driving signal that prevents from undesired tip heating while performing resistance measurement and sensor location between transformers that ensures galvanic isolation of the tip from the electronic circuit while scanning in contact mode. The proposed expansion module is compact and can be integrated onto AFM head close to the cantilever. Calibration process and resolution of 11 mK of the proposed setup is shown.

Acknowledgments

The described research was supported by the statutory grant of the Faculty of Microsystem Electronics and Photonics at the Wrocław University of Science and Technology.

Mechanical exfoliation of the van der Waals crystals

**M. TAMULEWICZ¹, J. SERAFIŃCZUK^{1,*},
K. GAJEWSKI¹, R. KUDRAWIEC², T. GOTSZALK¹**

¹) *Faculty of Microsystem Electronics and Photonics, Wrocław University of Science and Technology, Janiszewskiego 11/17, 50-372 Wrocław, Poland*

²*Institute of Physics, Wrocław University of Science and Technology, Wybrzeże Wyspiańskiego 27, 50-370 Wrocław, Poland*

*jaroslaw.serafinczuk@pwr.edu.pl

Keywords: van der Waals crystals, 2D materials, mechanical exfoliation, transition metal dichalcogenides (TMD)

Nowadays two-dimensional materials have gained particular interest due to their unique electrical, optical, mechanical and thermal properties. Transition metal dichalcogenides (TMD) is a group of materials which in contrast to the zero band gap semiconductor graphene exhibit a wide band-gap (e.g. 1.58 eV for MoS₂). TMD have the general formula MX₂, wherein M is a metal atom: Mo, W, Ti, Ta, Nb or Zr, and X is: S, Se or Te. MX₂ layers are held together by the weak van der Waals forces, causing that thin flakes can be easily exfoliated from bulk crystal [1]. Single layer TMD exhibits many unique properties, for example: surprisingly high photoluminescence (PL) efficiency [2], a clear spin splitting of valence bands due to strong spin-orbit coupling [3] and a novel spin-valley coupling [4] etc. In spite of its structural similarity to graphene, the layered TMD structure exhibits some advantages over it. Originally, TMDs are a semiconductor and has intrinsic band gap. In presented work we want to show some basic works on the optimization of the mechanical exfoliation process. As a research object ZrS₂ was taken into account. We show results of the basic research – optical microscopy and AFM investigations of the obtained flakes. These investigations allow to optimize exfoliation process and show influence of the exfoliation parameters e.g. size of the TMD sample, force of the tearing of the adhesive tape, number of tearing. Optimization of these parameters made the exfoliation process less random.

Acknowledgments

This work was financed from WrUST statutory grant no. 0401/0234/16. KG work was partially supported by the National Science Centre, Poland by the PRELUDIUM 9 programme (grant Nr 2015/17/N/ST7/03850).

References

- [1] K. S. Novoselov, D. Jiang, F. Schedin, T. J. Booth, V. V. Khotkevich, S. V. Morozov, and A. K. Geim, Proc. Natl. Acad. Sci. U.S.A. 102 (2005), pp. 10451.
- [2] R. S. Sundaram, M. Engel, A. Lombardo, R. Krupke, A. C. Ferrari, Ph. Avouris, and M. Steiner, Nano Lett. 13 (2013) , pp. 1416.
- [3] E. S. Kadantsev and P. Hawrylak, Solid State Commun. 152 (2012), pp. 909.
- [4] A. M. Jones, H. Yu, N. J. Ghimire, S. Wu, G. Aivazian, J. S. Ross, B. Zhao, J. Yan, D. G. Mandrus, D. Xiao, W. Yao, and X. Xu, e-printarXiv:1303.5318 (2013).

Experimental analysis of modular microfluidic geometries for passive manipulations on droplets

**D. ZAREMBA¹, S. BLONSKI¹,
P. M. KORCZYK^{*1},**

¹*Institute of Fundamental Technological Research, Address, , Polish Academy of Sciences, Pawinskiego 5B, 02-106 Warsaw, Poland*

^{*}piotr.korczyk@ippt.pan.pl

Keywords: Microfluidics, Droplets, Microfluidic traps, Channels

Passive techniques for manipulation on droplets are an interesting and promising approach for the design of microfluidic devices which on one hand are easy-to-use and on the another hand, execute complex laboratory procedures [?]. We present the comprehensive study of basic microfluidic components allowing for encoding different operations on droplets into the structure of the device. The understanding of hydrodynamic interactions between the continuous flow and the movement of a droplet in confined space of different microfluidic geometries is crucial for a rational and effective design of new generation of modular microfluidic processors with embedded instructions.

In the previous publication, we showed examples of passive microfluidic modules called microfluidic [?]. Traps were built with two basic geometries: obstacles and slit-bypasses (Fig.1). Modifications of these elements and their combination in different arrangements created traps with different functions such as: i) metering prescribed volume of a droplet (Fig.2); ii) immobilisation of a droplet; iii) combining consecutive drops.

The previous publication demonstrated the potential of the new approach, however, it lacked a comprehensive analysis of principal mechanisms. Potential applications of this technology require detailed investigations on mechanisms ruling movement of droplets. One of the most important parameters is the hydraulic resistance. In presented procedure, it is estimated in specially designed microfluidic networks. Two inputs of the device are fed with equal flows of: i) a buffer and ii) a buffer with an indicator. As the distribution of the initial amount of the indicator between both outputs corresponds to the ratio of resistances of the straight channel and the channel with investigated modules, the latter one can be estimated by direct measurement of the concentration of indicator. This method is noninvasive and offers high precision ensured by the use of the spectrophotometer. The results showed the intriguing fact that the addition of slit-bypasses to the channel does not change significantly the resistance although the increase of the cross-section. In the case of the obstacle, on the contrary, even short obstacle, introduces the significant rise of resistance.

The other investigations consisted in observations of the kinetics of traps. We observed both – the movement of a droplet and the flow of continuous phase by a microscope and a camera. The image analysis of the contours of a droplet and the movement of tracer particles provided

information on the interaction between both flows and its dependence on the local geometry (Fig.3). As we expected, during the flow of droplet through traps-like structures the bypasses are activated or deactivated depending on the actual position of a droplet. The gathered knowledge is very important for the understanding of the principle of passive structures. As we will show, the appropriate arrangements of base elements allowed as for successful design and fabrication of new passive elements.

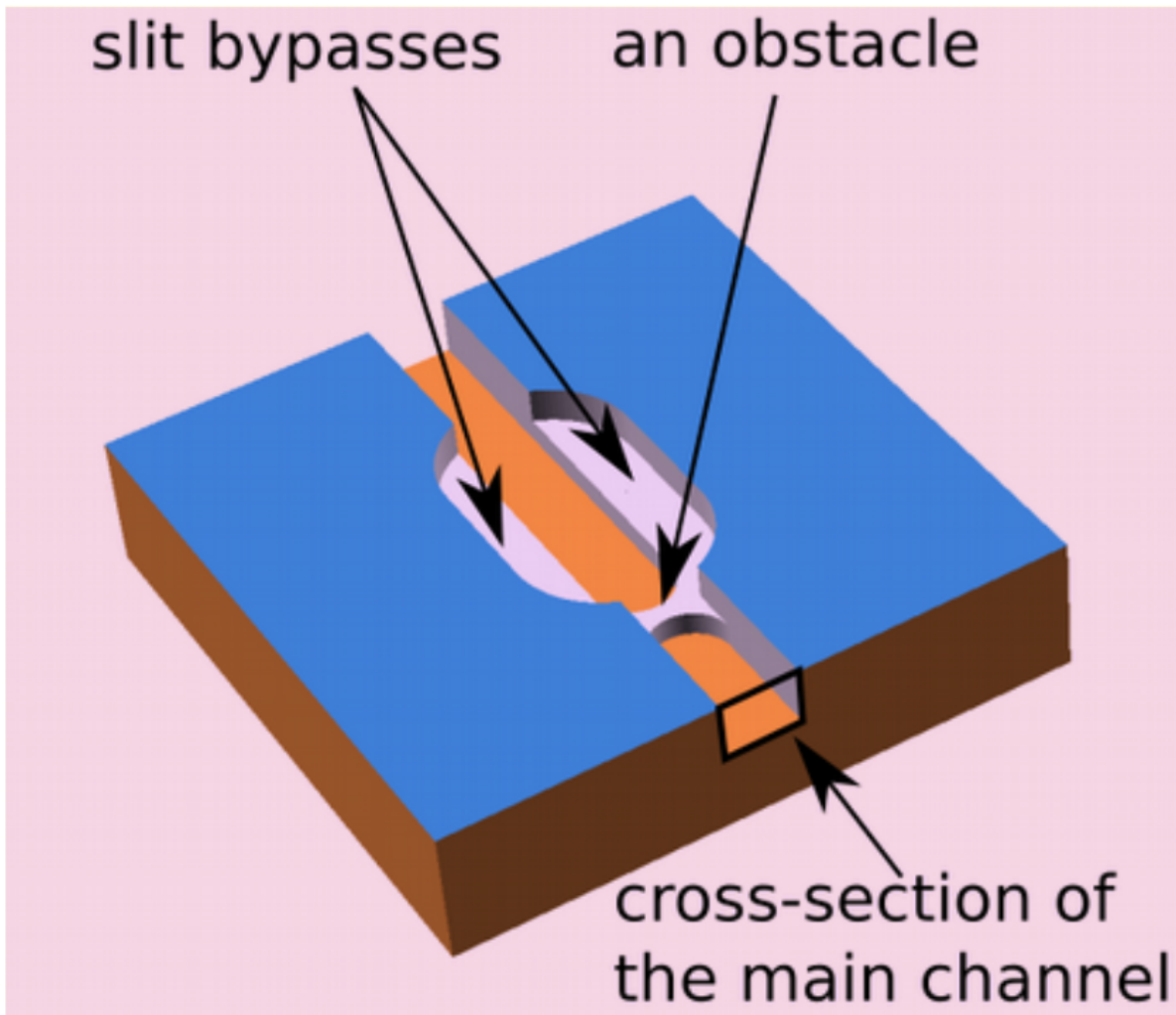


Fig. 1: 3-D presentation of the metering trap. The base components such as slit bypasses and an obstacle are shown.

Acknowledgments

2014/14/E/ST8/00578, National Science Centre, Poland

References

- [1] P. Zhu and L. Wang, "Passive and active droplet generation with micro uidics: a review," *Lab Chip*, Nov. 2016.
- [2] "A Laplace pressure based micro uidic trap for passive droplet trapping and controlled release," *Biomicro uidics*, vol. 6, no. 1, p. 014110, Feb. 2012.
- [3] A. M. Pit, M. H. G. Duits, and F. Mugele, "Droplet Manipulations in Two Phase Flow Micro uidics," *Micromachines*, vol. 6, no. 11, pp. 1768–1793, Nov. 2015.
- [4] P. M. Korczyk, L. Derzsi, S. Jakiela, and P. Garstecki, "Micro uidic traps for hard-wired operations on droplets," *Lab Chip*, Aug. 2013.

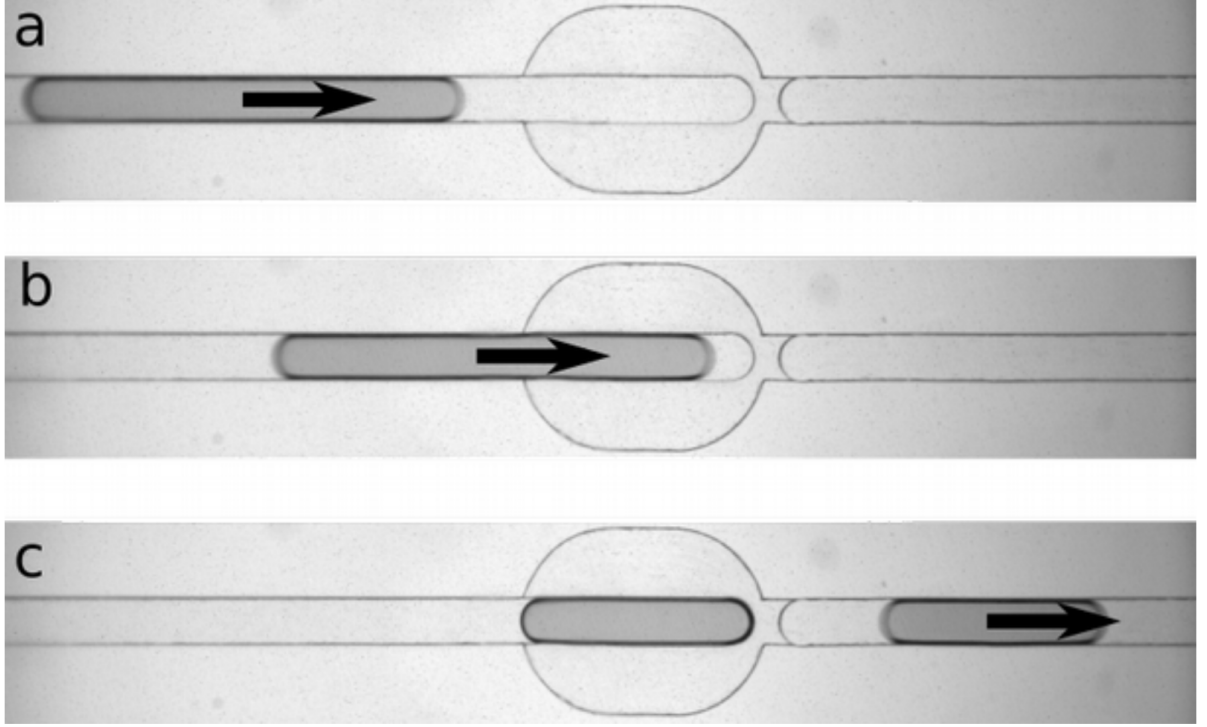


Fig. 2: The consecutive snapshots presenting a droplet traveling through a metering trap: a) droplet approaching to the trap, b) droplet entering into the trap, c) droplet after decomposition into two parts.

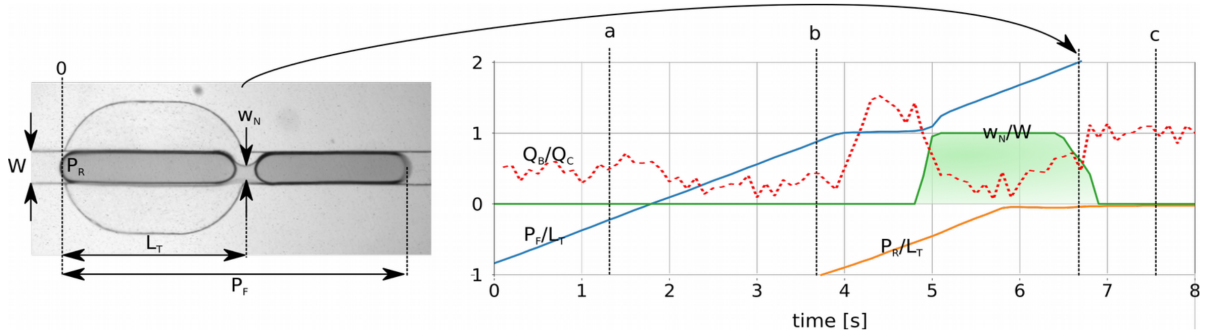


Fig. 3: The example of the analysis of kinetics. Left – the snapshot taken just before decomposition of a droplet. Right – plots of measured kinetic parameters: Q_B/Q_C , P_F/L_T , P_R/L_T , w_N/W . Where: Q_B – rate of flow through bypasses, Q_C – total rate of flow of continuous phase, L_T – length of the trap, P_F – position of the front of a droplet, P_R – position of the rear of a droplet, W – width of the channel, w_N – width of the neck.

Index

- Świątkowski M., 156, 172, 173
- Adabi M., 46
- Ahmad A., 59, 63, 120, 123, 125, 144, 159, 162
- Alaca B., 94
- Angelov T., 63, 123, 125
- Atanasov I., 63, 123, 125
- Awskiuk K., 85
- Aydogan C., 63, 94
- Błaszczuk M., 116, 127, 158
- Błoński S., 102
- Babiarczuk B., 146
- Babij M., 111, 140, 156
- Bachniak D., 105
- Badur J., 34
- Bartosz Kawa, 135
- Blonski S., 175
- Bobrowska J., 85
- Bogdanowicz R., 57, 65, 118
- Budkowski A., 85
- Budzioch J., 72
- Busch I., 39
- Bzowski K., 105
- Chomicki W., 20, 112, 172
- Chrzanowska-Giżyńska J., 92
- Ciceri F., 41
- Cuniberti G., 50
- Czykiel Ł., 113
- Danilkiewicz J., 73, 81
- Dannberg O., 42
- Darowicki K., 65
- Dazzi A., 16
- Dec B., 65
- Deniset-Besseau A., 16
- Derebecka N., 87
- Dianat A., 50
- Dietler G., 20, 112, 172
- Diez S., 25
- Dillon E., 16
- Drabik D., 83, 114
- Drobczyński S., 114
- Dunlop I., 46
- Durko M., 116, 127, 158
- Durrani Z., 63
- Dzierka A., 156
- Dziomba T., 39
- Erdem Alaca B., 11
- Felgner A., 39
- Ficek M., 57, 65, 118
- Foschia R., 20, 112
- Fröhlich T., 42
- Głowacki M., 118
- Gajewski K., 53, 111, 156, 174
- Gall M., 50
- Gardas M., 118
- Gawlik W., 118
- Gluch J., 50
- Gnatek D., 52
- Gołuński Ł., 57
- Goniszewski S., 46
- Gotszalk T., 21, 53, 57, 75, 90, 108, 111, 113, 129, 131, 140–142, 147, 156, 165, 171–174
- Grabiec P., 53, 75, 111, 147
- Gramala M., 75, 113
- Grzeszkowiak M., 87
- Guliyev E., 59, 120
- Gulyiev, 123
- Gulyiev E., 125, 162
- Gutierrez R., 50
- Hanham S., 46
- Hanus-Lorenz B., 83
- Hao L., 53
- Hemmleb M., 39
- Herman K., 73
- HERMANN P., 18
- Hierold C., 9
- Holyst R., 25, 87
- HOEHL A., 18
- Hoffman J., 92
- Hofmann M., 63, 94, 120, 144, 162
- Holz M., 59, 63, 94, 120, 123, 125, 144, 159, 162
- HU X. K., 18
- Ionescu A. M., 53

Ischuk V., 123
 Ishchuk V., 120, 125, 162
 Ivanov T., 42, 59, 63, 123, 125, 144, 159, 162
 Iwanicka Z., 116, 127, 158

 Józwiak G., 131
 Jakubiak M., 113
 Janus P., 21, 90, 111, 129, 165
 Jenni L., 9
 Jones M., 63
 Jordan R., 50

 KÄSTNER B., 18
 Kühnel M., 42
 Kędzia A., 133
 Kaestner M., 59, 63, 94, 120, 123, 125, 144, 149, 162
 Kapczyńska K., 172
 Kardani A., 71
 Karpińska M., 133
 Kasas S., 20, 112
 Kasprzycki P., 19
 Klein N., 46
 Kmiotek M., 136
 Koenders L., 39
 Kopiec D., 57, 131
 Korczyk P., 175
 Korczyk P. M., 102
 Kordos A., 138
 Kosmalska D., 153
 Kowalczyk-Gajewska K., 31
 Kowalewski T. A., 27
 Kranzmann A., 11
 Kraszewski S., 83, 114
 Krivoshapkina Y., 59, 63, 123, 125, 144
 Krzak J., 146
 KRZYSTECZKO P., 18
 Kubiak A., 167
 Kucaba-Piętal A., 47, 136, 138, 151
 Kudrawiec R., 174
 Kula P., 140
 Kulik A., 16
 Kumar L., 9
 Kunicki P., 21, 108, 111, 141
 Kwoka K., 108
 Kwong Hong Tsang D., 46

 Leblebici Y., 11
 Lekka M., 73, 81, 85
 Lenck O., 39
 Lenk C., 59, 63, 94, 120, 123, 144, 162
 Lenk S., 63, 144, 149, 162
 Lewandowski D., 146
 Liao Z., 50
 Lichocki A., 116
 Lichocki Z., 127
 Lischner J., 46
 Lisik Z., 167
 Lubos A., 153

 Mühle U., 50
 Majstrzyk W., 21, 63, 75, 141, 147, 156
 Manske E., 38, 94
 Marinushkin P., 162
 Marzec K., 151
 Mathurin J., 16
 Matuła K., 87
 Matysiak W., 153
 Medrano Sandonas L., 50
 Mehrafrooz B., 155
 Mościcki I., 49
 Mościcki T., 92
 Moczala M., 57, 108, 127, 131, 169
 Montazeri A., 71, 155
 Mrózek M., 118

 Nakielski P., 27
 Nechepurenko D., 63
 Neuman J., 104
 Niedzielski P., 53
 Nikolov N., 120, 123, 125, 159
 Nowak D., 171
 Nowicki M., 116

 Orłowska K., 57, 140, 141, 147

 Pabijan J., 73, 81, 85
 Paczesny J., 87
 Pawłowska S., 27
 Peplińska B., 87
 Petrov P., 46
 Piasecki T., 108, 135, 140, 156, 171, 173
 Piechocka I., 27
 Piekarska K., 96
 Pierini F., 27
 Pietrzyk M., 105
 Piotrowiak A., 127, 158

Ptak A., 69, 73
 Rangelo I., 144
 Rangelow I., 63, 75, 94, 120, 147, 159
 Rangelow I. W., 42, 59, 123, 125, 149, 162
 Rauch Ł., 105
 Reum A., 59, 63, 120, 123, 125, 144, 159, 162
 Richter Ł., 87
 Roman C., 9
 Rudek M., 21, 75, 90, 129, 140, 142, 156, 165, 172, 173
 Ruhnow F., 25
 Ruta L., 167
 Ryl J., 65
 Rysz J., 85

 Sadowski P., 31
 Sawczak M., 57, 65
 SCHUAMCHER H. W., 18
 Serafińczuk J., 174
 Seweryn S., 81
 Shaforost O., 46
 Sierakowski A., 21, 75, 90, 96, 111, 129, 131, 147, 165
 Sikora A., 116, 127, 133, 158, 169
 Silesian A., 87
 Siuzdak K., 65
 Sobaszek M., 57, 65
 Sozański K., 25
 Strupiński W., 53
 Stupar P., 20, 112
 Stupkiewicz S., 31
 Szalkowska M., 146
 Szczurek A., 146
 Szeliga D., 105
 Szmigiel D., 21, 90, 129, 165
 Szostak K., 83
 Szymanowska P., 171

 Tański T., 153
 Taşdemir Z., 11
 Tabaka M., 25
 Tamulewicz M., 174
 Targosz-Korecka M., 79

 ULM G., 18
 ULRICH G., 18
 Unger M., 16
 Utke I., 108

 Vasegh A., 155
 Volland B., 63, 94, 144

 Władowski M., 62
 Walczak R., 99
 Wawrzyniak C., 140
 Wesoly J., 87
 Wiśniewska A., 25
 Wiśniewska M., 102
 Wielgoszewski G., 173
 Wojtuś A., 173
 Wollschläger N., 11
 Wyroba E., 87

 Yilmaz M., 11
 Yacoot A., 40

 Zaremba D., 175
 Zarodkiewicz Ł., 30
 Zemła J., 81
 Zembrzycki K., 27
 Zhang T., 50
 Ziółkowski P., 34
 Ziębowicz B., 165
 Ziebowicz A., 165
 Zschech E., 50