1. Bartosz PRUCHNIK[1], 2. Andrzej SIKORA[1], 3. Krzysztof GAJEWSKI[1], 4. Bartosz ŚWIADKOWSKI[1], 5. Piotr SMAGOWSKI[1], 6. Dominik BADURA[1], 7. Krzysztof KWOKA[1], 8. Tomasz PIASECKI[1], 9. Teodor GOTSZALK[1]

Politechnika Wrocławska, Katedra Nanometrologii (1)
ORCID: 1. 0000-0003-1565-7278; 2. 0000-0001-6649-1963; 3. 0000-0001-6218-0658; 4. 0000-0001-9197-1862; 5. 0000-0002-5146-2868; 6. 0000-0003-1300-6420; 7. 0000-0001-8482-301X; 8. 0000-0002-3187-1488; 9. 0000-0003-4182-9192

doi:10.15199/48.2024.06.41

Educational scanning tunneling microscope – open architecture platform for nanotechnology teaching and nanometrology research

Streszczenie. *W pracy przedstawiamy platformę edukacyjnego skaningowego mikroskopu tunelowego pozwalającego na badania powierzchni w skali nanometrycznej. Zasadniczą zaletą zaprojektowanej konstrukcji jest jej otwarta architektura pozwalająca na prowadzenie różnorodnych eksperymentów zarówno dydaktycznych jak i wysokospecjalizowanych prac naukowych. Przedstawiony system został zaprojektowany w ramach prac dyplomowych i doktorskich w Katedrze Nanometrologii Wydziału Elektroniki, Fotoniki i Mikrosystemów Politechniki Wrocławskiej.* (***Edukacyjny skaningowy mikroskop tunelowy-platforma o otwartej architekturze dla edukacji i badań nanometrologicznych***)

Abstract. *In this paper, we present the in-house hardware and software platform allowing to perform the demonstrations of the design and operation of scanning tunneling microscope (STM) and derivative diagnostic techniques, enabling the determination of the properties of the surface at the nanoscale. The main advantage of the described setup is an open architecture, which is essential in terms of providing full insight into certain aspects of the construction and the ways the measurements are performed. Due to the modular design of the platform, students can excel in their competencies within various forms of learning activity, including basic training classes and diploma works. The described solution is a unique setup that was developed using the experience of the researchers at the Department of Nanometrology, Wrocław University of Science and Technology.*

Słowa kluczowe: mikroskopia bliskich oodziaływań, skaningowa mikroskopia tunelowa, nanometrologia
Keywords: scanning probe microscopy, scanning tunnelling microscopy, nanometrology, control and signal electronics.

Introduction

Since its development back in 1982 [1,2], scanning tunneling microscopy (STM) evolved into an advanced diagnostic technique, enabling while combined with other sample preparation techniques and analysis tools, an insight into the structure of the material at atomic resolution [3–6]. Despite the apparent simplicity of the concept of scanning tunneling microscopy, the complexity of real setups in terms of implementation of specific measurement modes and therefore instrumentation. Yet, the idea behind STM remains simple enough for home-grown constructors to develop their own measurement systems – there are plenty of do-it-yourself (DIY) projects to be found [7]. Also, rapid development in the field of software for control and analysis of measurement is present [8,9]. Developed microscopes are not complicated, open setups, in contrast to the commercially available machines.

Obtaining transparency in the design and operation of STM is an important issue in the education process of future nanotechnology specialists. The training aims to provide the necessary knowledge and experience, on how to prepare and use STM, to obtain atomic-resolution imaging of the sample surface. In particular, the issues of handling the sample, preparing the scanning tip, configuring specific parts of the system, optimizing the measurement parameters, as well as data processing and analysis are essential parts of the training. There are not many laboratories instigating bottom-up courses on scanning probe microscopy (SPM) [10]. In that case, nanotechnology tools for students are needed [11].

To provide the environment enabling the abovementioned training conditions, a specific hardware-software setup was developed in Department of Nanometrology. Unlike commercial STM systems, it provides full transparency in the signals processing and acquisition, including tunneling current, PID signals (in particular Z and error signal), scanning control (X, Y) signals, and output data. System is completed by dedicated software for data acquisition and analysis [12]. While designing this system, the following parameters had to be taken into account:

- open architecture allowing the demonstration of essential parts of the system and their role in the measurement
- access to control and measurement signals, enabling tracing of the way the measurement is performed
- the interface providing flexible configuration of the demonstration setups, which enables observation of every significant module's functionality, in particular, connecting meters or oscilloscopes revealing certain signals presence.

In this paper, we describe the abovementioned components

of the STM system, their functionalities, and the way the students can benefit from the particular way it was designed. It should be underlined, that the development of this platform engaged the experience and know-how of several scientists at the Department of Nanometrology at Wrocław University of Science and Technology [13,14].

ArmScope-Data acquisition and control electronics

The system architecture of the educational scanning tunneling microscope is shown in fig. 1.The tunneling current It is collected with an I/V converter. The bias current, gain and bandwidth of the I/V converter, based on an OPA 128 operational amplifier (Op), are 1 pA, 100 MΩ and 20 kHz respectively. The output signal of the I/V converter is connected with a signal rectifier and a logarithmic amplifier operating with a LOG100 integrated circuit (IC) by Burr-Brown. The output signal of the logarithmic amplifier is fed to a continuous proportional integrative and differential (PID) controller. The controller gain is set in the range between 10-10 and 1 with the 10 bit resolution. The integrating time constant are defined in two ranges from 100 µs to 1 ms and 1 ms to 10 ms with the resolution of 100 µs and 1 ms respectively. The differential time constant of the PID controller is set from 10 ms to 110 ms with 20 ms resolution.

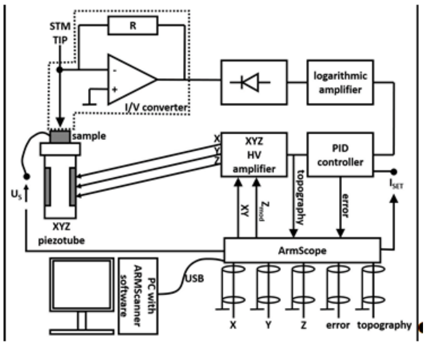


Fig. 1 Architecture of the educational scanning tunneling microscope

As the core of the PID controlled is realized with a group of Ops and the PID settings can be varied in very wide frequency range, the proposed circuitry makes it possible to control the distance between the tip and the investigated surface in the frequency range limited by the resonance frequency of the Z piezoactuator (usually 10 kHz). Operation in such wide bandwidth is often not possible, when the PID controller output is calculated digitally. Moreover, the resolution of the continuous PID controller is limited by the noise of the Ops and not the quantization error of the analog to digital and digital to analog converters (ADCs and DACs). The PID controller parameters are controlled by a microprocessor and PC software. The presented architecture ensures the high precision of the definition of the PID parameters leading to the reliable surface measurements in the constant height and constant current scanning modes. The output of the PID controller is connected with the high voltage (HV) amplifier controlling extension of the piezoactuator, which defines the distance between the tip and the investigated sample. The gain of the HV unit, whose core is built with APEX 340, is 10 V/V and its frequency bandwidth is 10 kHz. The HV amplifier biases also the XY electrodes of the piezoactuator, the bandwidth in these channels is limited to 200 Hz. The scanning, data acquisition and control processes are controlled by the digital controller ArmScope [15]. The controller consists of an analogue to digital converter (ADC) card integrating two of AD7865: 14 bit, 4 channel and 250 kS/s ICs. They enable high resolution and fast acquisition of the topography and error signals. Moreover, it makes it possible to acquire additional signals like head temperature and e. g. recorded when the tip tunneling signal is modulated.

The scanning XY movements are controlled by a scanner processor KOMPAS. It makes it possible to conduct the scanning movement in any angular direction and adjust its size. The maximal scanning frequency is 30 lines/s, which is necessary to acquire surface images with the atomic resolution.

KOMPAS is built around the Xilinx Artix-7 FPGA controlling a set of high resolution DACs: 18-bit 2-channel LTC2758 for scan field generation with a decade analogue attenuator for small scan fields and two 16-bit 4 channel LTC2754-16 DACs providing offset voltages and six auxiliary signals A - F. Scanning raster is generated digitally and filtered with low-pass IIR filters. The scaling within one decade and rotating are performed digitally. The output control voltages are generated using scan field DACs, filtered with reconstruction filters, attenuated if necessary and added with offset voltages.

The most important design goals that KOMPAS 3.0 should meet were the minimization of the noise in the scan field voltages and the reduction of the thermal drift. The goal was achieved with a careful selection of active and passive components. All analogue outputs from the scan field controller are done using differential lines to minimize the noise influence and ground loop problems. As the entire architecture is digital the influence of the signal and mechanical drifts is reduced.

Mechanical setup

The designed scanning tunneling microscope frame is presented in fig. 2. This is the so called scanning sample design, which means that the sample is moved in XYZ directions over the fixed tunneling tip.

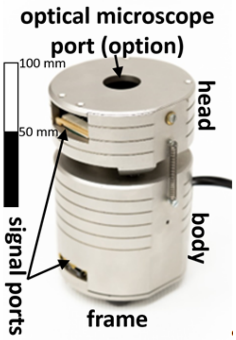


Fig. 2 Mechanical setup of the educational scanning tunneling microscope

The microscope frame consist of two main mechanical parts: a body and a measurement head – fig. 2. In order to ensure the highest mechanical stiffness the microscope body was machined as a stainless steel cylinder with diameter and height of 60 mm and 90 mm respectively. The symmetrical, cohesive one block architecture makes the setup less susceptible to the thermal expansion and external mechanical vibrations. The body houses a piezoelectrical stepper motor 8301F by Picomotor, whose spindle, defining the vertical position of the measurement head, can be moved in the range up to 12,7 mm with resolution below 30 nm. In the center of the microscope body a piezoectrical scanner is mounted. It consists of two piezotubes: the inner one is responsible for the XY scanning movements, whereas the outer controls the distance between the scanning tip and the investigated sample. In this setup it is possible to move the sample in X and Y directions of up to 3,2x3,2 µm and in Z axis up to 900 nm, with the scanning frequency of 30 lines/s and bandwidth of 2 kHz. The sample holder is grounded in order to ensure the proper shielding of the electrostatic field generated, when the piezotube electrodes are biased. The scanner is connected to the control electronics through an FFC ribbon. Typical biasing voltage varies from +/- 60 up to +/- 120 V in dependence of scanner configuration. Principles of working of piezoelectric scanners can be found in [16,17].

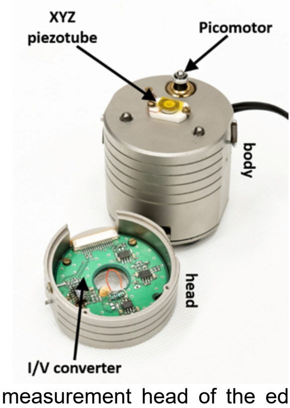


Fig. 3 Body and measurement head of the educational scanning tunneling microscope

The microscope head integrates a tip holder and a measurement electronics – fig. 3. The measurement electronics integrates an I/V converter with the gain of 100 MΩ for currents bigger than 100 fA operating in the bandwidth of up to 10 kHz and a circuitry biasing the sample. The entire system is supplied with voltage of +/-5 V in order to reduce the power dissipated in the head electronics and ensuring the best thermal stability. The tip of the tunneling microscope is placed in a syringe needle and contacted with a short wire with the I/V converter in order to reduce the input capacitance and improve the signal to noise ratio (SNR)-fig. 4.

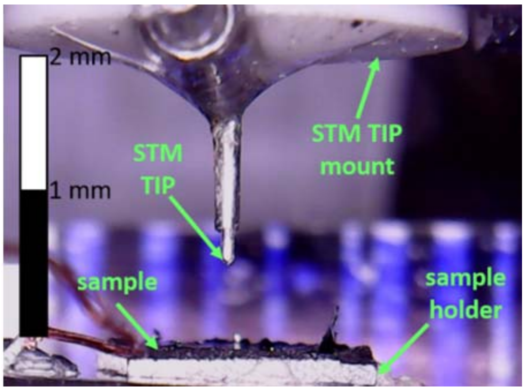


Fig. 4 Scanning tip over the investigated samples

ArmScanner - Control and data acqusition and processing software

The presented system is controlled by the ArmScanner software [15]. The data acquisition functions make it possible to configure the measurement channels (up to 8 channels) for the signals from the microscope measurement head – fig. 5. The corresponding windows present visually the result of the signal acquisition. The data acquisition panel enables also to define speed and resolution of the mechanism controlling the approach of the tip towards the investigated sample. This can be done in two modes: the manual one when the microscope operator defines the moment when the approach procedure is interrupted and the automatic one, when the output of the tunneling current defines the stop of the approach. Such a solution ensures both the speed and reliability of the approach process, which is preserves the tip quality and protect the surface against damage.

The scan option software routines define the size and angular position of the scanning field. Moreover, the basic scan options can be determined, which involves the definition of the scanning algorithm (linear or sine movement if the so called fast axis) the speed of the tip – fig. 5. The flexible and precise definition of the scanning movements is crucial as it influences the tip-surface distance control and the quality of recorded measurements. The scan movements must be controlled so as not to excite resonance vibrations of the piezotube and simultaneously to reduce the time of the image acquisition and avoid measurement disturbances. In order to tune the PID controller settings special routines are implemented making it possible to modulate the control setpoint and/or the position of the XYZ piezotube – fig. 6a.

In order to ensure the reliable tunneling contact between the tip and the parameters of the PID controller must be tuned with high resolution and repeatability. This is done with a dedicated routine of the ArmScope software, where the entire control, including the HV amplifier gain and bandwidth, are defined – fig. 6b. The developed software operates on the PC and microcontroller platforms is flexibly composed. This which in combination with open architecture control and data acquisition enables simple adaptation to the experimental requirements. To the technologies, which can be built up in this way belong nanolithography and all multi pass measurement modes including Kelvin probe force microscopy (KPFM) and magnetic force microscopy (MFM).

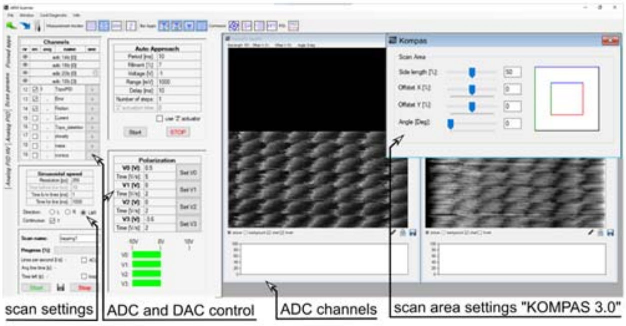


Fig. 5 ArmScope software-Data acquisition and tip-surface approach panel

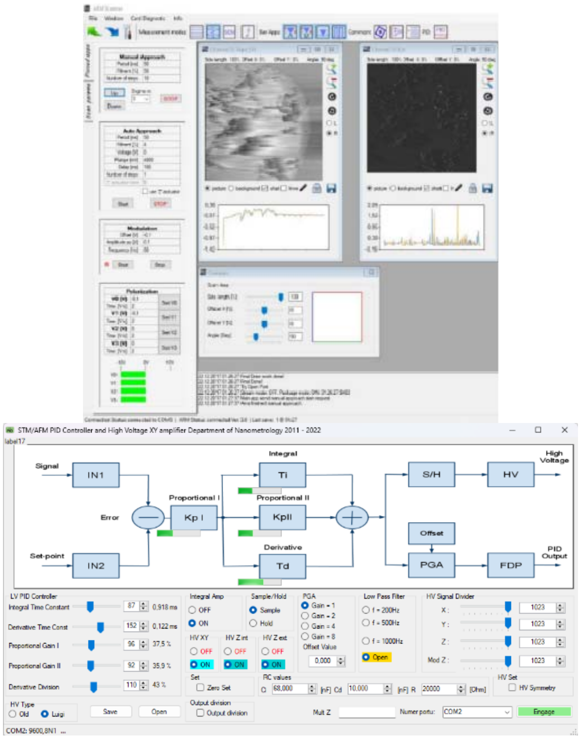
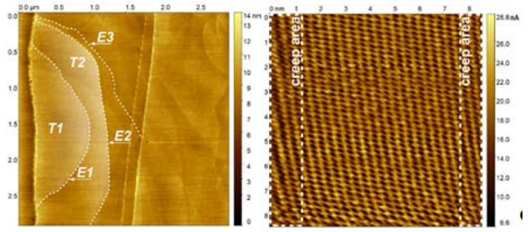


Fig. 6 ArmScanner software above: ArmScope software-Scan and modulation control panel below: ArmScope software-PID controller settings of the controller parameters

Results

In our experiments we use samples of highly ordered pyrolytic graphite (HOPG), which are available in various crystal orientations. The HOPG surface can be easily prepared for the further experiments by exfoliation done with a sticky tape. The HOPG crystals are also used as the source for the graphene investigations, which is usually the first step in the nanometrology of two dimensional (2D) materials.

The applications of the described STM system span from microscopic to nanoscopic surface investigations – fig. 7a and fig. 7b. The recorded images show surface features in a scale corresponding to the scan area. In fig. 7a many monoatomic terraces of the width of up to hundreds of nanometers (details in fig. 7a: T1, T2), and crystal edges of height of several monoatomic layers (details in fig. 7a: E1, E2, E3) are visible. The orientation of the HOPG crystal along the monoatomic terrace defines the surface electrical conductivity [18], thus it influences the conductivity of the structures containing few terraces, whose size is of hundreds of nanometers. At scales small enough, when the image of the crystal terrace is zoomed in ca. 300 times, atomic lattice is visualized – fig. 7b. It should be noticed that in both pictures the image resolution of 512x512 points is still maintained, which results from the KOMPAS scanning controller architecture, where the image size is defined independently on the numbers of the recorded points. The creep area depicted in fig. 7b indicates the creep of the XY piezotube, which can be recorded when surface is investigated with higher scanning frequency (bigger than 20 lines/s). The image fidelity can be proved, when the same image is recorded at the being modified scan size – fig. 8a and fig. 8b. Thus, in the error control and height domains the proportional numbers of the observed atomic details are identified.



a) b)

Fig. 7 STM images recorded on HOPG surface

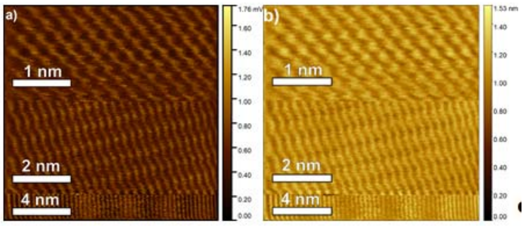


Fig. 8 Scan field change during measurement on the HOPG surface sample with constant speed. Pictures in tunneling current and height domain presented. Feature size (atomic lattice) changes dimensions appropriately to the scan area. Scan width changes from unitary to half and quarter, with dimensions of details changing appropriately. In the largest scale, Moire pattern may be observed

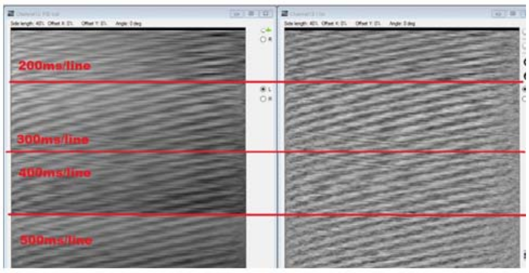


Fig. 9 Atomic lattice imaged with scan speed from 500 ms/line (2 Hz) to 200 ms/line (5 Hz). Feature size (atomic lattice) doesn't change dimensions appropriately to the scan speed, proving it's surface origination. Picture is taken directly out of the ARMScann

Another method relies on the modification of the scanning speed while scanning the same image. In this case the same number of the atomic details, again in the control error and height domains, must be seen – fig. 9. The analysis of the HOPG atomic images makes it possible to calibrate the deflections of the XY actuator. The atomic images can be analyzed using various algorithms, e.g. Gwyddion lattice analysis tool [19,20], which adjusts rhomboidal lattice over the hexagonal HOPG one-fig. 10. Hence, the acquired lattice dimensions - a1 and a2 - correspond with the lattice dimensions of 0.283 nm (double distance between two nearest atoms) and 0.245 nm (distance between every second lattice atom) - as described in [21] – fig. . By that, total scan area may be derived based on atomic lattice dimensions – table 1. Similarly, the vertical sensitivity may be calculated based on the height of a singular HOPG atomic layer. Assuming interlayer height of 0.67 nm, the vertical sensitivity equals 24 nm/V

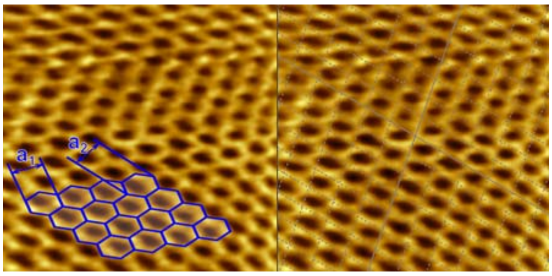


Fig. 10 STM images of the atomic HOPG structure. Atomic lattice dimensions determined by sample scan. Idealized hexagonal lattice inscribed into picture – (left); Gwyddion lattice fitting tool, by which values lateral distances were determined - a1 and a2 – (right)

The observed lattice asymmetry is the result of a tension present in the surface material (e.g. stemming from the sample mounting on the actuator holder). Therefore the lattice deformation is linked directly with the strain induced in the substrate. Hence, measurement of force, tension and load may be conducted by lattice shape observation. It may be further described by the ratio of the measured lattice vectors. The STM measurements are fundamentally electrical and as such serve the purpose of the electrical sample nanocharacterization. Apart from clear material distinction in form of the conductivity or the material work function, the electronic structure of the surface may be also visualized. The surface electronic structure is also determined by the buried atomic layers, which results in imaging of the so called Moire patterns. Their presence proves angular disparity between the subsequent crystal layers. Thus, the shape of the recorded pattern may be used to investigate the spatial structure of multilayered 2D materials.

Tab. 1 Characteristic dimensions of the inscribed rhomboidal lattice and derived HOPG lattice with the lateral amplification rate of the scanning head

Coordinate	Rhomboidal lattice dimension (%)	Corresponding HOPG lattice dimension (nm)	Ratio (nm/area)
a_1	7.13e-5	0.245	3964
a_2	6.17e-5	0.283	3969

Summary

In this article we presented the architecture of the educational scanning tunneling microscope. The developed system can be used in teaching the nanotechnology and nanometrology basics for students at engineering and natural science university faculties. The open architecture of the microscope hardware (ArmScope) and software (ArmScanner) makes it possible to apply this machine in versatile nanoscopic solid state investigations. Not only the surface topography can be recorded in the scale varying from to 3x3 micrometers down to 2x2 nanometers (using the same XYZ piezoactuator) but also the electronical properties along the crystal edges. The presented measurement system is also the perfect tool in the investigations of 2D materials. It makes it possible to observe the Moire patterns, which correspond with the twist between the subsequent crystal planes. We also presented the routine of the reliable nanometrology of a HOPG sample, identifying the terrace and atomic surface composition. It opens new ways to perform nanometrology with crystal lattice-based dimensional standard [22,23].

Operation in air makes it not only more available for educational work. There are specimen classes not suitable for vacuum systems, including biological samples. STM examination of organic matter is a promising direction in modern nanometrology [24,25].

The setup of the machine was designed also to enable future nanolithography experiments. In this way nanoprototyping of the nanolectronical devices (e. g. single electron devices (SEDs) and/or quantum contacts) using tips of various shape and properties (e.g. gallium nitride and/or diamond probes) will be possible [26]. STM still remains one of only few techniques able to resolve single ion implantation, what becomes more important in the age of SED development [27,28]. The presented system can be integrated with an optical microscope, making it possible to observe the position of the tunnelling tip over the investigated sample. Moreover, it is the efficient solution for the fast surface scanning experiments and related nanometrology experiments.

This project (20IND08 MetExSPM) has received funding from the EMPiR programme co-financed by the Participating States and from the European Union's Horizon 2020 research and innovation programme.

***Autorzy**: mgr inż. Bartosz Pruchnik, Politechnika Wrocławska, Katedra Nanometrologii, C-2, Janiszewskiego, 52-372 Wrocław, E-mail: bartosz.pruchnik@pwr.edu.pl,*
dr hab. inż. prof. uczelni Andrzej Sikora, Politechnika Wrocławska, Katedra Nanometrologii, C-2, Janiszewskiego, 52-372 Wrocław, E-mail: andrzej.sikora@pwr.edu.pl,
dr inż. Krzysztof Gajewski, Politechnika Wrocławska, Katedra Nanometrologii, C-2, Janiszewskiego, 52-372 Wrocław, E-mail: krzysztof.gajewski@pwr.edu.pl,
dr inż. Bartosz Świadkowski, Politechnika Wrocławska, Katedra Nanometrologii, C-2, Janiszewskiego, 52-372 Wrocław, E-mail: bartosz.świadkowski @pwr.edu.pl,
dr inż. Piotr Smagowski, Politechnika Wrocławska, Katedra Nanometrologii, C-2, Janiszewskiego, 52-372 Wrocław, E-mail: piotr.smagowski@pwr.edu.pl,
mgr inż. Dominik Badura, Politechnika Wrocławska, Katedra Nanometrologii, C-2, Janiszewskiego, 52-372 Wrocław, E-mail: dominik.badura@pwr.edu.pl,
dr inż. Krzysztof Kwoka, Politechnika Wrocławska, Katedra Nanometrologii, C-2, Janiszewskiego, 52-372 Wrocław, E-mail: krzysztof.kwoka@pwr.edu.p,l
dr hab. inż. prof. uczelni Tomasz Piasecki, Politechnika Wrocławska, Katedra Nanometrologii, C-2, Janiszewskiego, 52-372 Wrocław, E-mail: tomasz.piasecki@pwr.edu.pl,
prof. dr hab. inż. Teodor Gotszalk, Politechnika Wrocławska, Katedra Nanometrologii, C-2, Janiszewskiego, 52-372 Wrocław, E-mail: teodor.gotszalk@pwr.edu.pl

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