ISSN 1846-6168 (Print), ISSN 1848-5588 (Online)
https://doi.org/10.31803/tg-20230829155921

Original scientific paper
Received: 2023-08-29, Accepted: 2023-08-30

Atomic Force Microscopy: Step Height Measurement Uncertainty Evaluation

Andrej Razumić*, Biserka Runje, Dragutin Lisjak, Davor Kolar, Amalija Horvatić Novak, Branko Štrbac, Borislav Savković

Abstract: The atomic force microscope (AFM) enables the measurement of sample surfaces at the nanoscale. Reference standards with calibration gratings are used for the adjustment and verification of AFM measurement devices. Thus far, there are no guidelines or guides available in the field of atomic force microscopy that analyze the influence of input parameters on the quality of measurement results, nor has the measurement uncertainty of the results been estimated. Given the complex functional relationship between input and output variables, which cannot always be explicitly expressed, one of the primary challenges is how to evaluate the measurement uncertainty of the results. The measurement uncertainty of the calibration grating step height on the AFM reference standard was evaluated using the Monte Carlo simulation method. The measurements within this study were conducted using a commercial, industrial atomic force microscope.

Keywords: atomic force microscope; measurement uncertainty; Monte Carlo simulation

1 INTRODUCTION

It is known that the key indicator of measurement result quality is measurement uncertainty. According to the Guide to the Expression of Uncertainty in Measurement (GUM), measurement uncertainty is defined as a parameter associated with a measurement result that describes the dispersion of values that could reasonably be attributed to the measured quantity with a certain level of probability [1]. The purpose of measurement is to determine the value of the measured quantity. Proper understanding of the data obtained from measurements is crucial for the application of that data [2]. When measurement data are used for decision-making, it is often assumed that the data are accurate to such an extent that information about the measurement uncertainty of the measured result is almost never provided. Generally, a measurement result is only an approximation or estimation of the value of the measured quantity. Therefore, a measurement result is complete only when accompanied by a statement of the uncertainty associated with that estimate [3].

The expression of measurement uncertainty is preceded by establishing a mathematical model that best describes the measured quantity [4]. The mathematical model represents the relationship between output and input quantities and consists of many functionally connected components. According to the GUM, for estimating measurement uncertainty, it is necessary to know all the parameters that influence the measurement process. Additionally, it is necessary to assess the components of each individual parameter that affect the measurement result [1].

Various methods are employed for estimating measurement uncertainty. The widely accepted model for calculating measurement uncertainty in metrology practice is the GUM method [5]. However, due to the large number of influential parameters and the complex and nonlinear functional relationships involved in atomic force microscopy, estimation of measurement uncertainty based solely on the Guide to the Expression of Uncertainty in Measurement is insufficient [6]. Therefore, the Monte Carlo simulation (MCS) method is used. In addition to the Monte Carlo simulation method, the Bayesian method can be used for calculating measurement uncertainty [7]. The Bayesian method for estimating measurement uncertainty is employed when dealing with rare data or complex models. It is also used for quantities that are not normally distributed, have certain constraints, or exhibit skewed density functions [8].

The Monte Carlo simulation (MCS) method is introduced in the Joint Committee for Guides in Metrology (JCGM) 101:2008 Supplement 1 to the Guide to the Expression of Uncertainty in Measurement – Propagation of Distributions Using Monte Carlo Method [9]. The Monte Carlo method involves propagating the distribution of input uncertainty sources using a model to obtain the output distribution. It is a numerical method based on generating a large number of random values and analyzing the obtained data to obtain information about the best estimate of the output quantity [10]. The Monte Carlo simulation method is particularly useful when the underlying probability distributions of the system are not well known or cannot be easily determined analytically [11]. The following are the basic steps involved in using Monte Carlo simulations for estimating measurement uncertainty:

- Identification of relevant sources of measurement uncertainty: The first step is to determine the relevant sources of uncertainty that may affect the measurement result.
- Modeling of uncertainty sources: For each uncertainty source, a specific probability distribution or a set of data from previous measurements should be used. The MCS method is not limited to selecting prior distributions; they can also be asymmetric.
- Generation of random numbers from probability density functions of input quantities: Using the uncertainty sources and their distributions, a large number of M random measurement results are generated.
- Estimation of the output quantity: By generating random numbers from the probability density functions of input quantities, a probability density function describing the measured quantity is obtained.
- Estimation of measurement uncertainty: Based on the output probability density function, the measurement uncertainty interval for a given value is determined.

Since the Monte Carlo simulation method allows for considering different uncertainty sources and generating many measurement samples, this method can be considered a more accurate and comprehensive approach to estimating measurement uncertainty than the GUM method [12].

2 AFM AND REFERENCE STANDARD

Research on atomic force microscopy in the field of dimensional nanometrology is relatively unexplored [13]. From the perspective of estimating measurement uncertainty, one of the main challenges in atomic force microscopy is the large number of influential parameters [14]. According to the GUM, it is necessary to know all the parameters that affect the measurement result and their contributions to the measurement uncertainty [15]. Due to the highly complex nature of the measurement system and its various applications in industry and science, which involve analyzing results and measuring samples of different materials and applications, estimating measurement uncertainty in atomic force microscopy according to the GUM is highly complex [16]. Considering the large number of parameters that influence the operation and measurement results of atomic force microscopy, it is necessary to investigate how each parameter affects the measurement process and to what extent [17]. The first step is the identification and classification of influential parameters on the measurement result. In the Ishikawa diagram shown in Fig. 1, the influential parameters are categorized into six main groups: measurand, operator, AFM instrument, image analysis, reference standards, and environmental conditions. It is important to note that certain parameters can belong to more than one group, and there can be overlaps between the groups that include specific influential parameters.

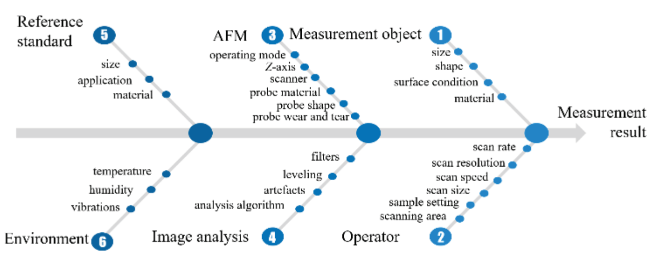


Figure 1 The texts under figures

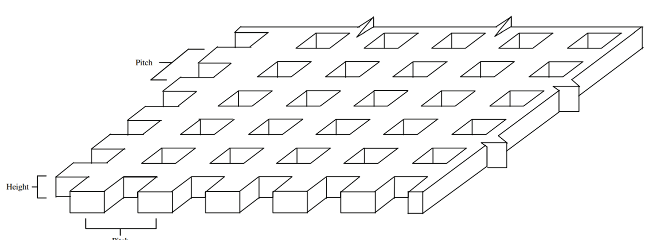


Figure 2 Features on the AFM reference standard [18]

The moderate reference standard employed for conducting measurements is the Surface Topography Standard (STS) developed by the American company VLSI Standards [18], with headquarters in Milpitas. This particular standard is utilized for the calibration of AFM devices. The STS reference standard (Fig. 2) encompasses features defined in all three spatial directions, enabling measurement standardization, calibration, and monitoring of the linearity of the AFM measurement instrument. Additionally, it provides information regarding the condition and wear of the probe tip.

The reference standard consists of a silicone matrix with dimensions of 12 × 8 mm, featuring precisely fabricated silicon dioxide features. The standard contains three groups of features, spaced 100 μm apart. Each grating pattern on which the defined features are located consists of alternating lines and spaces, with a uniform pitch in both the x and y directions. There are three nominal step lengths in the x and y directions: 3 μm, 10 μm, and 20 μm. The nominal height of the features on the standard in the z-axis is 100 nm. The entire matrix, including the features in the calibration area, is coated with a uniform layer of platinum with a nominal thickness of 40 nm (Fig. 3).

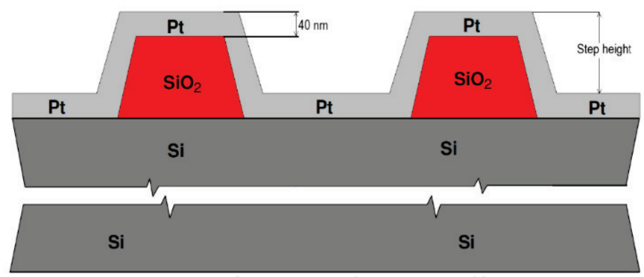


Figure 3 Cross-section of the standard [18]

Given that it is a calibrated AFM standard, it is accompanied by a calibration certificate. The calibration certificate for the STS3-1000P standard [18] states that the depth of the grating groove is (97.6 ± 1.4) nm. The measurements were conducted under the following stable environmental conditions: temperature of (20 ± 1) °C and humidity of (42 ± 2) %. Fig. 4 illustrates the manifestation of the standard, as quantified using the *Oxford MFP-3D Origin* model atomic force microscope, situated at the Faculty of Mechanical Engineering and Naval Architecture in Zagreb.

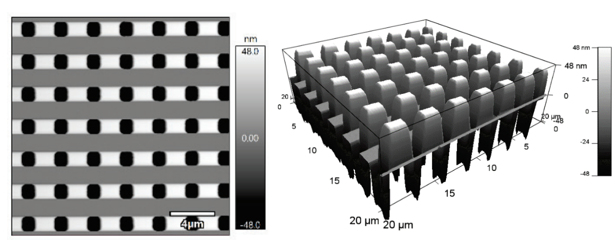


Figure 4 Reference standard in 2D (left) and 3D (right) format

3 FACTORIAL DESIGN OF THE EXPERIMENT

A factorial design of the experiment was conducted as part of this study to demonstrate the significance of input parameters in atomic force microscopy (AFM) measurements. The design of the experiment entails conducting trials across all levels of the designated input parameters. Tab. 1 delineates the input parameters for the experimental framework. Within this experimental setup, each parameter assumes a binary state, thereby constituting a two-factor experimental design. Considering the influential factors (Fig. 1) and the measurement procedure itself, preliminary investigations included measurement input parameters that are selected through software prior to conducting the measurements, as well as the operating mode of the microscope. The design of the experiment was created and analyzed using *Design Expert 13* software.

Table 1 Input factors and experiment leves

Variable identifier	Controlled factor	Variable classification	Metric measure	Inferior extent	Superior extent
A	Operating mode	Nominal	–	Tapping	Contact
B	Scan size	Ratio	µm × µm	10 × 10	20 × 20
C	Scan resolution	Ratio	–	256	512
D	Scan speed	Ratio	µm s^{-1}	12.5	50

As an output, the measured step height of the regular grid on the reference standard was monitored. The measurement results obtained from the two-factor experimental design are shown in Tab. 2.

Table 2 Data from the measurements

Number	Factor *A*	Factor *B*	Factor *C*	Factor *D*	Response
	Operating mode	Scan size	Scan resolution	Scan speed	*h*
	–	µm × µm	–	µm·s^{-1}	nm
1	tapping	10	256	12.5	92.50
2	contact	10	512	50	91.64
3	tapping	10	256	12.5	96.26
4	tapping	20	512	50	95.51
5	tapping	20	256	12.5	98.36
6	contact	20	512	50	85.10
7	contact	20	256	12.5	93.17
8	tapping	10	512	50	82.04
9	contact	10	256	12.5	102.8
10	contact	20	256	50	103.9
11	tapping	10	512	12.5	103.2
12	contact	20	512	50	103.7
13	contact	10	256	12.5	103.4
14	tapping	20	256	50	102.7
15	contact	10	512	12.5	103.6
16	tapping	20	512	50	103.0

Table 3 Analysis of variance for the response

Source	Sum of squares	df	Mean square	*F*-value	*P*-value	Significance
Model	640.7401	5	128.148	17.6565	0.000205	significant
Model members						
A-operating mode	278.6003	1	278.6003	38.38613	0.00016	significant
B-scan size	69.89005	1	69.89005	9.6296	0.012658	significant
D-scan speed	27.29538	1	27.29538	3.760816	0.084406	significant
AD	51.41629	1	51.41629	7.084246	0.025977	significant
BD	49.31872	1	49.31872	6.795238	0.028421	significant
Residual	65.32052	9	7.257836			
Cor total	706.0606	14				

After conducting and analyzing the experimental design, the following results were obtained, Tab. 3.

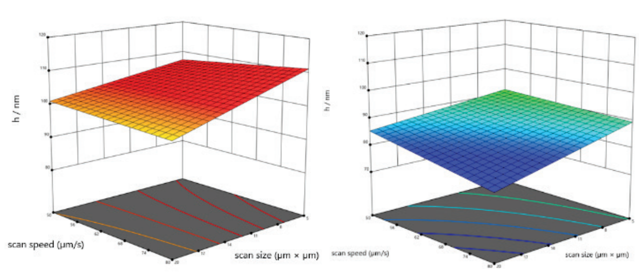
The *F*-value of the model, amounting to 17.6565, and the *P*-value of 0.000205, indicate that the selected model is suitable for determining the significant input variables on step height *h*. *P*-values of model components less than 0.05 indicate the significance of individual components in the model. In this model, factors A, B, D, as well as their interactions AD and BD, are significant.

Tab. 4 provides an overview of the metrics delineating the caliber of the formulated mathematical model with respect to the variable h. The computed value of the adjusted coefficient of determination, R_{adj}^2, stands at 0.8561. This signifies that the input parameters have been well chosen, i.e., 85.61% of the model can be described by the selected input parameters and their interactions. The coefficient of determination, R_{pre}^2, is 0.6864. This indicates that 68.64% of the data obtained from the experiments can be explained by the predictive model, which is satisfactory. The disparity between the predicted coefficient of determination, R_{pre}^2 (0.6864), and the adjusted coefficient of determination, R_{adj}^2 (0.8561), registers at less than 0.2. This suggests that the anticipated and calibrated determination coefficients exhibit a rational concordance. The value of the determination coefficient, R^2, is 0.9075. The attained level of precision is 10.8403, surpassing the stipulated minimum threshold of 4.

Table 4 Statistical characteristics of the model

Standard deviation	2.69
Arithmetic mean	97.15
Coefficient of variation, %	2.77
Coefficient of determination R^2	0.9075
Adjusted coefficient of determination R_{adj}^2	0.8561
Predicted coefficient of determination R_{pre}^2	0.6864
Adequate precision	12.9829

Fig. 5 depicts the output variable h in a three-dimensional representation as a function of scanning speed and scanned area size. The conducted design of the experiment has demonstrated that the input variables that significantly influence the step height of the reference etalon are the operating mode, scanned area size, and scanning speed. All subsequent measurements carried out in this study were performed in the contact mode, with a scanning speed of 12.5 µm·s^{-1}, and a scanning resolution of 256 over a scanned area size of 20 µm × 20 µm.



a) b)

Figure 5 The three-dimensional representation of the output variable h depending on scanning speed and scanned area size is shown for (a) the contact mode and (b) the tapping mode.

4 EVALUATIONOF MEASUREMENT UNCERTAINTY

The selected method for estimating the measurement uncertainty of the step height of the calibrated AFM reference sample is the Monte Carlo simulation method. After identifying the influential variables affecting the measurement result of the step height, a mathematical model of the output variable is established, represented by Eq. (1). The Monte Carlo simulation method for evaluating the measurement uncertainty of the step height of the calibrated AFM reference sample was implemented using the *Python* programming language based on Eq. (1) and the input parameters shown in Tab. 5.

$$h = h_x + r \cdot t \cdot l + d \cdot \alpha \cdot \Delta\vartheta + \delta r + \delta R + \delta probe \qquad (1)$$

Table 5 Input variables and probability density function

Input variable x_i		Probability density function $g(x_i)$
Measured value	h_x	Normal distribution (M, 98.9 nm, 0.9 nm)
Scan rate	r	Uniform distribution (M, –0.005 s^{-1}, 0.005 s^{-1})
Scanning time	t	Uniform distribution (M, –0.5 s, 0.5 s)
Scan length	l	Uniform distribution (M, –0.005 nm, 0.005 nm)
Nominal step height	d	100 nm
Temperature expansion coefficient	α	Normal distribution (M, 2.57 × 10^{-6} K^{-1}, 0.019 × 10^{-6} K^{-1})
Temperature difference	$\Delta\vartheta$	Uniform distribution (M, –2 K, 2 K)
Repeatability	δr	Normal distribution (M, 0 nm, 0.104 nm)
Reproducibility	δR	Normal distribution (M, 0 nm, 0.823 nm)
Probe	$\delta probe$	Normal distribution (M, 0 nm, 0.86 nm)

A total of M = 100,000 simulations were performed. The component of the measured value was based on 15 repeated measurements of the step height, from which the mean and standard deviation were calculated. The components of scanning speed, scanning time, and scanning length followed rectangular distributions according to the resolutions of the respective variables. The coefficient of thermal expansion of silicon was taken as 2.57 × 10^{-6} K^{-1} with an expanded uncertainty of 0.038 × 10^{-6} K^{-1}. The repeatability and reproducibility of the results were calculated in accordance with ISO 5725-2:2019 [19], which defines the basic method for determining the repeatability and reproducibility of a standard measurement method. A total of 15 repeated measurements were performed in two measurement series. Within each measurement series, the measurements were conducted under repeatability conditions, with the same metrology, measuring instrument, and measurement conditions, and the measurements were repeated in a short period of time. Between the measurements performed in the first and second measurement series (conducted on two different days), the repeatability conditions were not fully met, but the reproducibility conditions were considered. The component of the tip influence on the measurement result was taken from the literature [20]. An analysis of the tip influence on the measurement result was performed, resulting in a value of 0.86 nm.

After performing the Monte Carlo simulation method, the results are shown in Fig. 6 and Tab. 6. The estimated standard deviation of the step height h of the calibrated AFM reference sample is 1.5 nm. The measurement result reads: h = (98.9 ± 2.9) nm with a coverage probability P of 95 %. Therefore, it can be stated with 95 % confidence that the measured step heights on the AFM reference sample will fall within the interval of 96.0 nm to 101.8 nm.

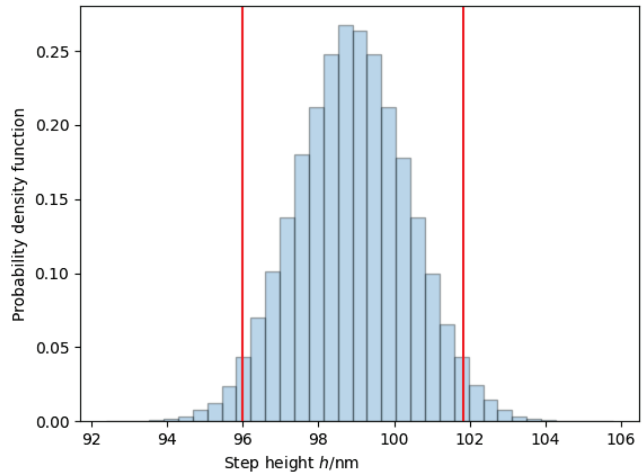


Figure 6 Probability density function of AFM reference standard step height

Table 6 Statistical parameters of the output function

Parameter	Value
Lower limit of the interval (2.5 %)	96.0 nm
Upper limit of the interval (97.5 %)	101.8 nm
Expanded measurement uncertainty U	2.9 nm
Arithmetic mean	98.9 nm
Standard deviation	1.5 nm

Furthermore, a comparison was conducted between the results obtained by AFM from FMENA in Zagreb and VLSI Standards in Milpitas [18]. The comparison of results is performed by calculating the agreement factor En, as given by Eq. (2).

$$En = \frac{|\bar{x}_1 - \bar{x}_2|}{\sqrt{U_1^2 + U_2^2}} \leq 1 \qquad (2)$$

where is: $\bar{x}_1$ - arithmetic mean of the step height obtained by VLSI Standards, nm; $\bar{x}_2$ - arithmetic mean of the step height obtained by FMENA, nm; U_1 - expanded measurement uncertainty specified by VLSI Standards, nm; U_2 - expanded measurement uncertainty specified by FMENA, nm.

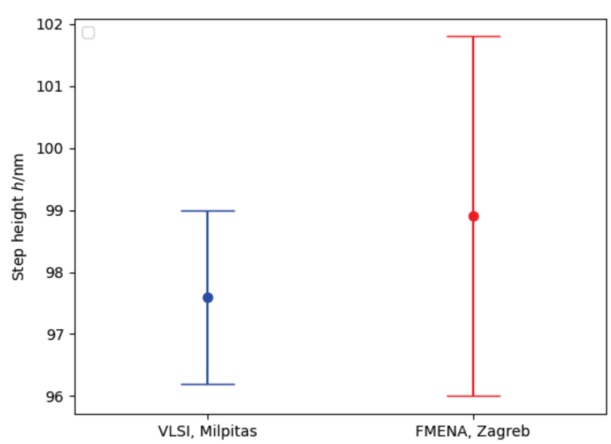


Figure 7 Comparison of the values obtained by VLSI Standards and FMENA

The obtained value of the conformity factor En is 0.40. Since the conformity factor is less than 1, it can be concluded that the results are compatible. Additionally, the results can be presented graphically, as shown in Fig. 7. It can be observed that the mean value of the simulated data lies within the measurement uncertainty specified by the Calibration Certificate.

5 CONCLUSION

The study encompassed the assessment of measurement uncertainty in atomic force microscopy. The measurements were conducted on an AFM reference standard utilized for the instrument calibration purposes. These reference specimens incorporate a calibration grating featuring a precisely defined step height, accompanied by information pertaining to the expanded measurement uncertainty.

In the field of metrology, diverse methodologies are employed for the estimation of measurement uncertainty. Despite the predominant usage of the GUM method, it becomes imperative to employ alternative techniques when dealing with AFM measurements. This is primarily attributed to the presence of numerous influential factors that impact the measurement outcomes, as well as the complex and nonlinear functional relationships between the input and output variables. Consequently, the utilization of additional uncertainty estimation methods becomes indispensable.

To systematically identify the influential variables and establish a comprehensive understanding, an Ishikawa diagram was employed, effectively categorizing these variables into six principal groups. Furthermore, a two-factor design of the experiment was implemented to ascertain the significance of the operational mode, scan speed, and scan size on the step height—a critical parameter of interest.

In light of the above, the Monte Carlo simulation method was applied to estimate the measurement uncertainty associated with the step height of the AFM reference standard. By leveraging the Monte Carlo simulation approach, crucial insights regarding the expanded measurement uncertainty were acquired, revealing a step height value of (98.9 ± 2.9) nm with a confidence level of 95 %. Additionally, a conformity factor was computed to facilitate the comparison between the obtained results and the information outlined in the calibration certificate of the AFM reference standard. Remarkably, the conformity factor indicated compatibility between the results obtained by company VLSI Standards and Faculty of Mechanical Engineering and Naval Architecture, substantiating the reliability and accuracy of the measurements.

Acknowledgement

The Article Processing Charges (APCs) is funded by the European Regional Development Fund, grant number KK.01.1.1.07.0031.

6 REFERENCES

[1] See https://www.iso.org/sites/JCGM/GUM/JCGM100/C045315e-html/C045315e.html?csnumber=50461

[2] See https://www.researchgate.net/publication/361909628_Good_practice_in_evaluating_measurement_uncertainty_--_Compendium_of_examples

[3] Coskun, A., Theodorsson, E., Oosterhuis, W. P. & S. Sandberg (2022). Measurement uncertainty for practical use. *Clinica Chimica Acta, 531*, 352-360. https://doi.org/10.1016/j.cca.2022.04.1003

[4] Castro, H. F. F. (2021). Validation of the GUM using the Monte Carlo method when applied in the calculation of the measurement uncertainty of a compact prover calibration. *Flow Measurement and Instrumentation, 77*, 101877. https://doi.org/10.1016/j.flowmeasinst.2020.101877

[5] Kessel, W. (2022). Measurement uncertainty according to ISO/BIPM-GUM. *Thermochimica Acta, 382*(1), 1-16. https://doi.org/10.1016/S0040-6031(01)00729-8.

[6] Nguyen L. T. P. & Liu, B. H. (2022). Machine learning approach for reducing uncertainty in AFM nanomechanical measurements through selection of appropriate contact model. *European Journal of Mechanics – A/Solids, 94*, 104579. https://doi.org/10.1016/j.euromechsol.2022.104579

[7] Lira, I. (22016). The GUM revision: the Bayesian view toward the expression of measurement uncertainty. *European Journal of Physics, 37*(2), 025803. https://doi.org/10.1088/0143-0807/37/2/025803

[8] See https://opg.optica.org/ao/abstract.cfm?uri=ao-51-25-6196

[9] See https://www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf

[10] Harris, P. M. & Cox, M. G. (2014). On a Monte Carlo method for measurement uncertainty evaluation and its implementation. *Metrologia, 51*(4), S176. https://doi.org/10.1088/0026-1394/51/4/S176

[11] Witkovský, V., Wimmer, G., Ďurišová, Z., Ďuriš, S., & Palenčár, R. (2017). Brief overview of methods for measurement uncertainty analysis: GUM uncertainty framework, Monte Carlo method, characteristic function approach. *The 11th International Conference on Measurement*, 35-38. https://doi.org/10.23919/MEASUREMENT.2017.7983530

[12] Mahmoud, G. M. & Hegazy, *R. S.* (2017). Comparison of GUM and Monte Carlo methods for the uncertainty estimation in hardness measurements. *International Journal of Metrology and Quality Engineering, 8*(14). https://doi.org/10.1051/ijmqe/2017014

[13] Rao, A. et al. (2007). Characterization of nanoparticles using Atomic Force Microscopy. *Journal of Physics: Conference Series 61*, 971-976. https://doi.org/10.1088/1742-6596/61/1/192

[14] Kumari, C. & and Chak, S. K. (2019). Study on influential parameters of hybrid AFM processes: a review. *Manufacturing Review. 6*, 23. https://doi.org/10.1051/mfreview/2019022

[15] Razumić, A., Horvatić Novak, A., Štrbac, B., Alar, V. & Runje, B. (2021). The influence of filtering on surface topography parameters obtained by the AFM. *The 21st International Conference Materials, Tribology, Recycling. MATRIB 2021*, 418.

[16] Wagner, R., Moon, R., Pratt, J., Shaw, G. & Raman, A. (2011). Uncertainty quantification in nanomechanical measurements using the atomic force microscope. *Nanotechnology 22*(45), 455703. https://doi.org/10.1088/0957-4484/22/45/455703

[17] Razumić, A., Horvatić Novak, A, Štrbac, B. & Runje B. (2011). Influence of scan parameters on surface topography obtained via AFM. *Etikum 2021*, 17.

[18] VLSL Standards (2022). Certification of calibration: Surface topography standard

[19] See https://www.iso.org/standard/69419.html

[20] Pétry, J., De Boeck, B., Sebaihi, N., Coenegrachts, M., Caebergs, T. & Dobre, M. (2021) Uncertainty evaluation in Atomic Force Microscopy measurement of nanoparticles based on statistical mixed model in a Bayesian framework. *Measurement Science and Technology, 32*(8). https://doi.org/10.1088/1361-6501/abe47f

Authors' contacts:

Andrej Razumić, assistant
(Corresponding author)
University of Zagreb, Faculty of Mechanical Engineering and Naval Architecture,
Ivana Lučića 5, 10000 Zagreb, Croatia
andrej.razumic@fsb.hr

Biserka Runje, PhD, Prof.
University of Zagreb, Faculty of Mechanical Engineering and Naval Architecture,
Ivana Lučića 5, 10000 Zagreb, Croatia
biserka.runje@fsb.hr

Dragutin Lisjak, PhD, Prof.
University of Zagreb, Faculty of Mechanical Engineering and Naval Architecture,
Ivana Lučića 5, 10000 Zagreb, Croatia
dragutin.lisjak@fsb.hr

Davor Kolar, PhD
University of Zagreb, Faculty of Mechanical Engineering and Naval Architecture,
Ivana Lučića 5, 10000 Zagreb, Croatia
davor.kolar@fsb.hr

Amalija Horvatić Novak, PhD
University of Zagreb, Faculty of Mechanical Engineering and Naval Architecture,
Ivana Lučića 5, 10000 Zagreb, Croatia
amalija.horvatic@fsb.hr

Branko Štrbac, PhD, Assoc. Prof.
University of Novi Sad, Faculty of Technical Sciences,
Trg Dositeja Obradovića 6, Novi Sad, Serbia
strbacb@uns.ac.rs

Borislav Savković, PhD, Assoc. Prof.
University of Novi Sad, Faculty of Technical Sciences,
Trg Dositeja Obradovića 6, Novi Sad, Serbia
savkovic@uns.ac.rs