Politechnika Wrocławska

Nano 2020 Innovation Conference & Exhibition

Rome, 15-18 September

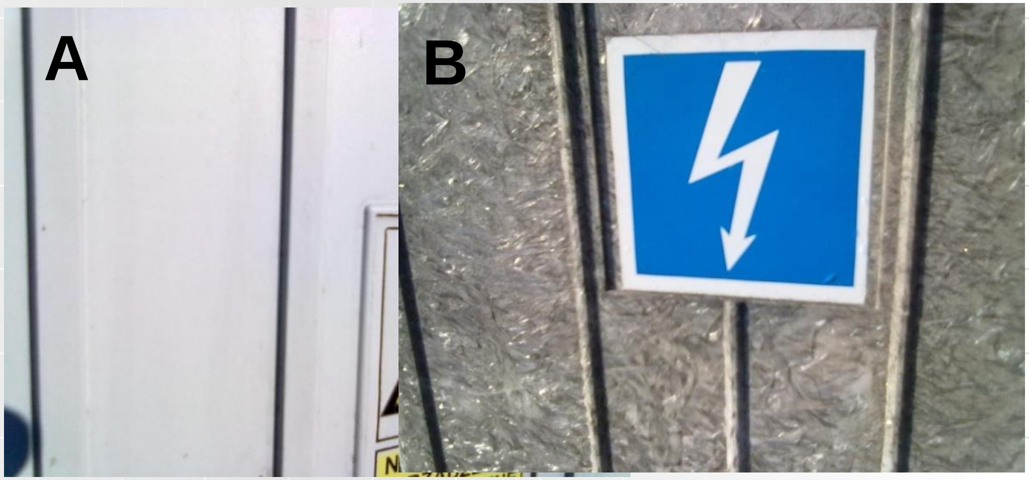
School on Scanning Probe Microscopy

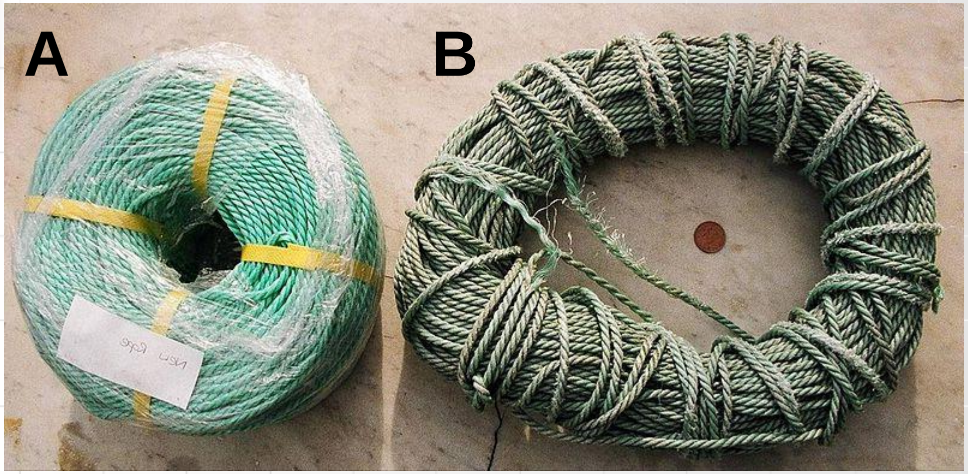
Correlative microscopy – the issue of precise positioning of the sample and its impact on the experiment outcome

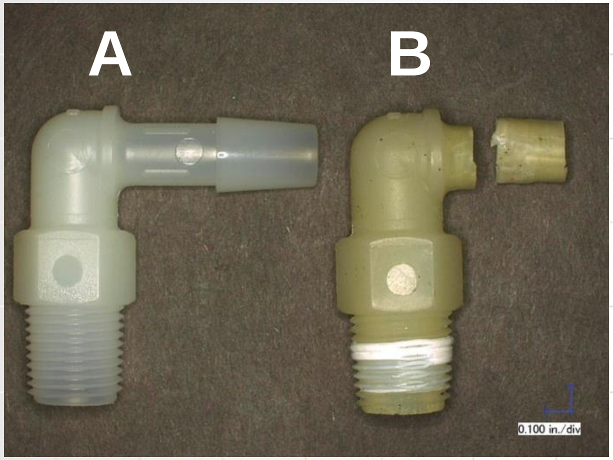
Andrzej Sikora

HR EXCELLENCE IN RESEARCH

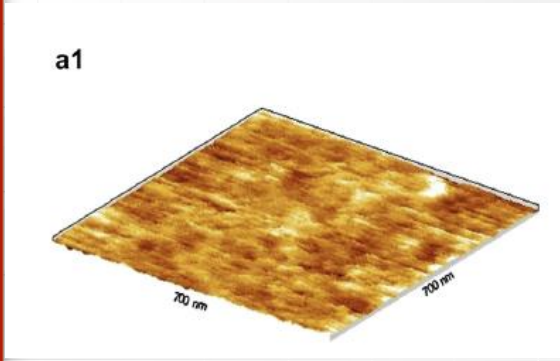
The issue od the polimer deterioration investigation



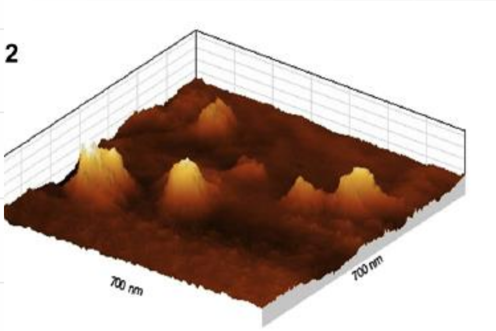




Surface degradation investigation using AFM



Surface roughness	Before immersion		After immersion	
	Neat system (pm)	Nanocomposite (nm)	Neat system (pm)	Nanocomposite (nm)
S_a	448	1.66	571	1.76
S_q	585	2.73	752	2.75



- epoxy nanocomposite ageing observation

- the topography observation after the exposition

- single topography measurements

Ravari F, Omrani A, Rostami AA, Ehsani M, 2012, *Ageing effects on electrical, morphological, and mechanical properties of a low viscosity epoxy nanocomposite*, Polym. Degrad. Stab. **97,** 929–935

Surface degradation investigation using AFM

A summary of the surface roughness (R_a and RMS) measured by the AFM method of the pristine and irradiated PP, PET, PC and PEEK polymers.

Polymer	Impl. fluence (ion/cm^2)	C ions		O ions	
		R_a (nm)	RMS (nm)	R_a (nm)	RMS (nm)
PP	Pristine	1.9	2.4	1.9	2.4
	1.0×10^{10}	2.2	2.8	2.1	2.7
	1.0×10^{11}	2.1	2.7	2.0	2.6
	1.0×10^{12}	2.2	2.6	2.2	2.7
	1.0×10^{13}	–	–	–	–
PET	Pristine	0.5	0.6	0.5	0.6
	1.0×10^{10}	1.8	2.3	1.3	1.6
	1.0×10^{11}	–	–	–	–
	1.0×10^{12}	1.6	2.1	1.1	1.6
	1.0×10^{13}	2.0	2.5	2.7	4.5
PC	Pristine	7.1	8.8	7.1	8.8
	1.0×10^{10}	6.6	8.2	5.0	6.2
	1.0×10^{11}	–	–	5.2	6.4
	1.0×10^{12}	6.3	7.5	4.8	6.1
	1.0×10^{13}	8.0	10.0	–	–
PEEK	Pristine	1.5	1.9	1.5	1.9
	1.0×10^{10}	1.8	2.3	1.9	2.4
	1.0×10^{11}	–	–	–	–
	1.0×10^{12}	1.8	2.2	1.9	2.4
	1.0×10^{13}	2.4	3.0	3.2	4.1

- irradiation of PP, PET, PC with Cn+ and On+ 9.6 MeV heavy ions

- the topography observation after the exposition

- single topography measurements

Mikšová R, Macková A, Malinský P, Slepička P, Švorčík V, 2015, *A study of the degradation of polymers irradiated by C^{n+} and O^{n+} 9.6 MeV heavy ions*, Polym. Degrad. Stab. **122,** 110–121

Surface degradation investigation using AFM

Irradiation time (hours)	Roughness Rq (nm)
0	7 ± 1
25	6 ± 1
50	7 ± 1
75	6 ± 1

- polypropylene photodegradation investigation

- the topography measurement after the exposition

- single topography measurements

Rouillon C, Bussiere P.-O, Desnoux E, Collin S, Vial C, Therias S, Gardette J.-L, 2015, *Is Carbonyl Index a quantitative probe to monitor polypropylene photodegradation?*, Polym. Degrad. Stab., 128, 200–208

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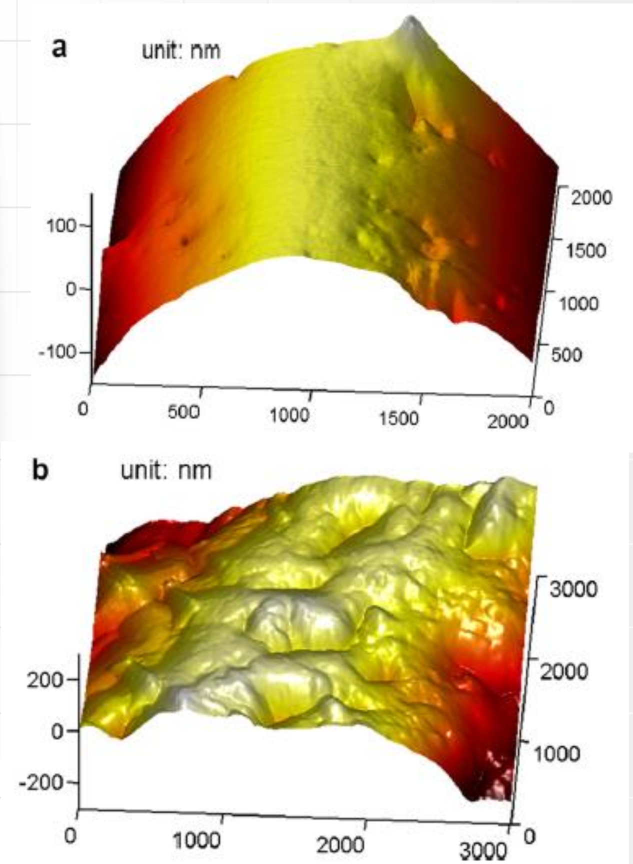
Surface degradation investigation using AFM



- investigation of of UV impact on Kelvar fibers

- the topography measurement after the exposition

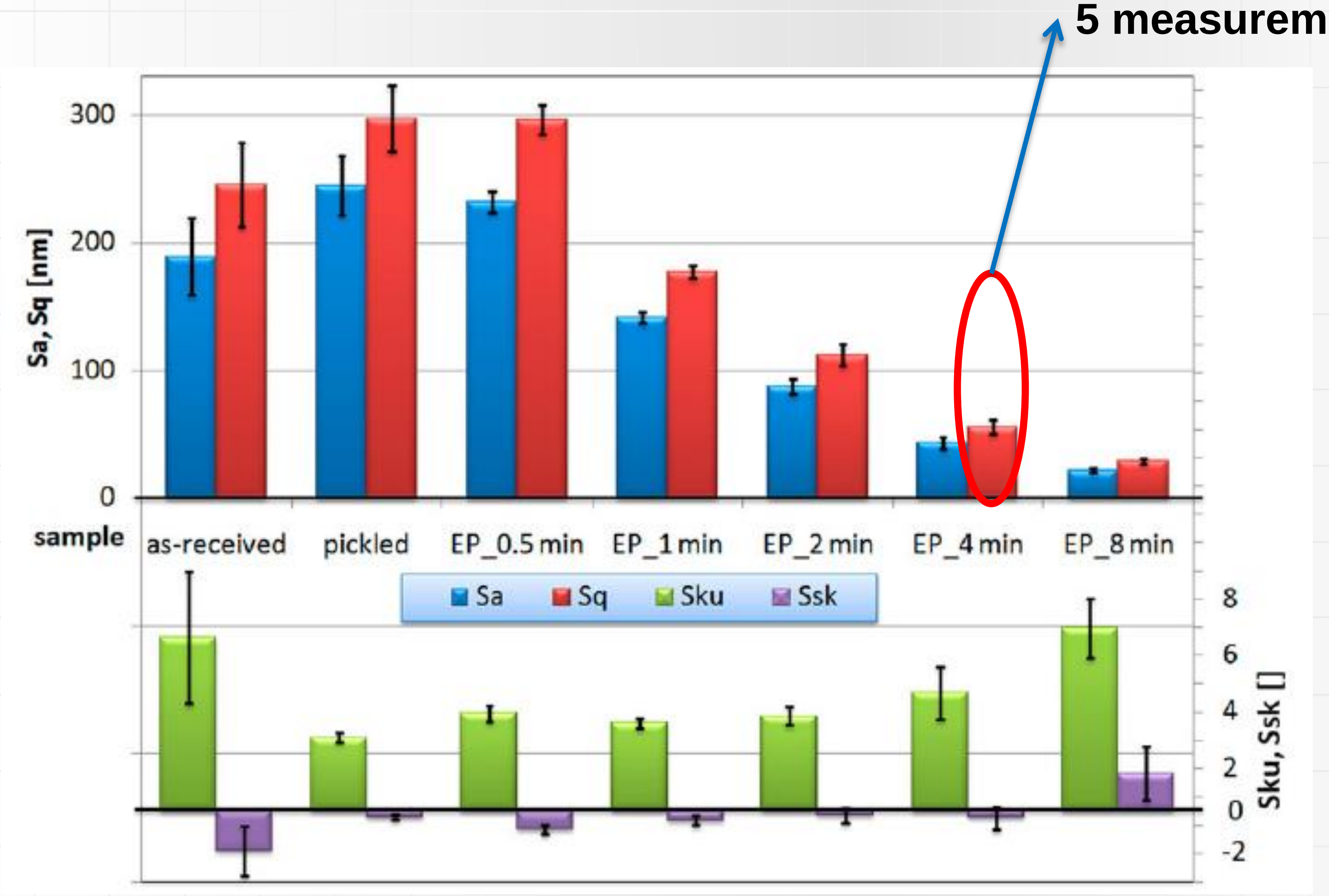
- single topography measurements

The surface roughness under different condition.

Treatment time (h)	Roughness (nm)	
	UV irradiation	Moisture
Untreated	38.8	38.8
6	300.6	312.7
12	258.2	304.5
24	213.4	130.8
36	208.5	106.5
48	202.5	110.3

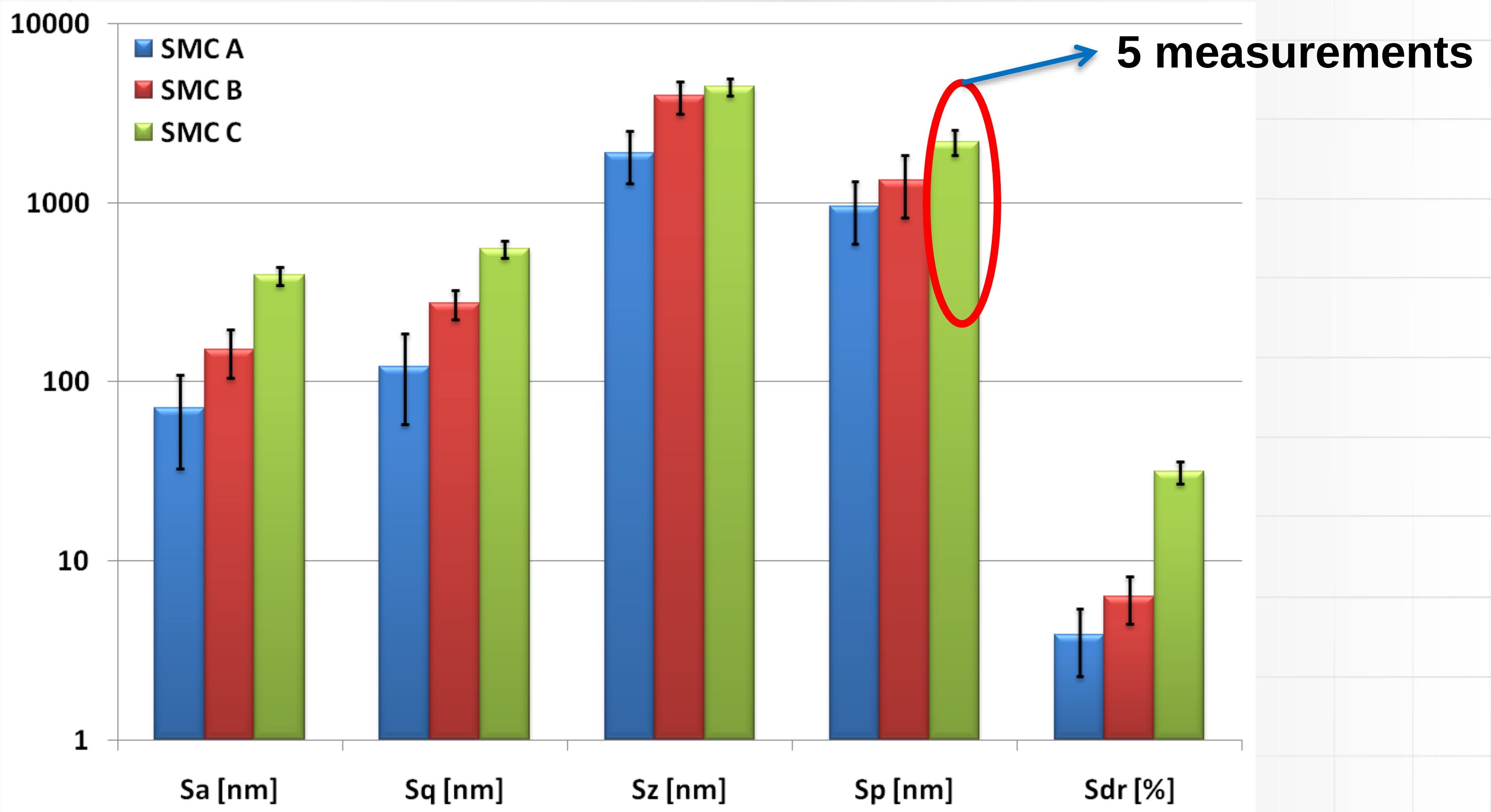
Wang H, Xie H, Hu Z, Wu D, Chen P, 2012, *The influence of UV radiation and moisture on the mechanical properties and micro-structure of single Kevlar fibre using optical methods*, Polym. Degrad. Stab. 97, 1755–1761

Electropolished stainless steel – process dynamic investigation



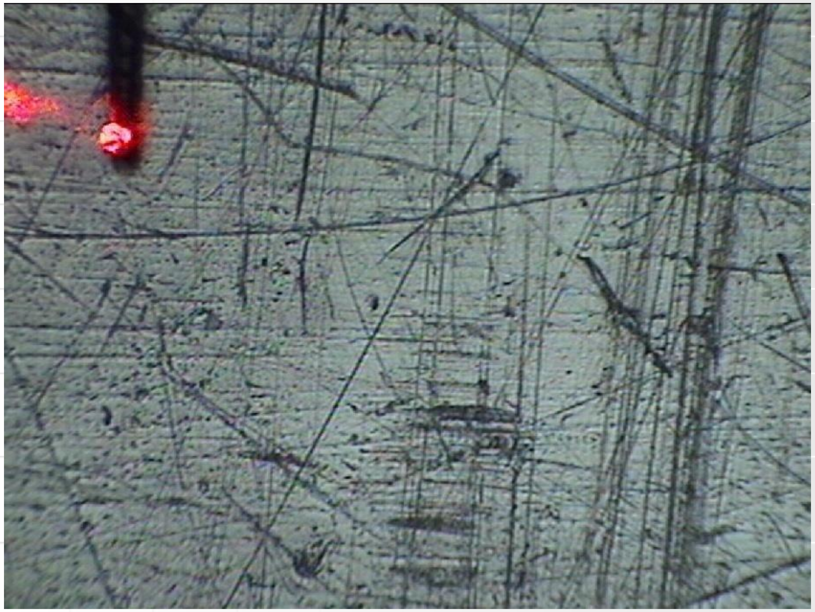
Lochyński P, **Sikora A**, Szczygieł B, 2016, *Surface morphology and passive film composition after pickling and electropolishing*, Surface Engineering 0267-0844, 1-9

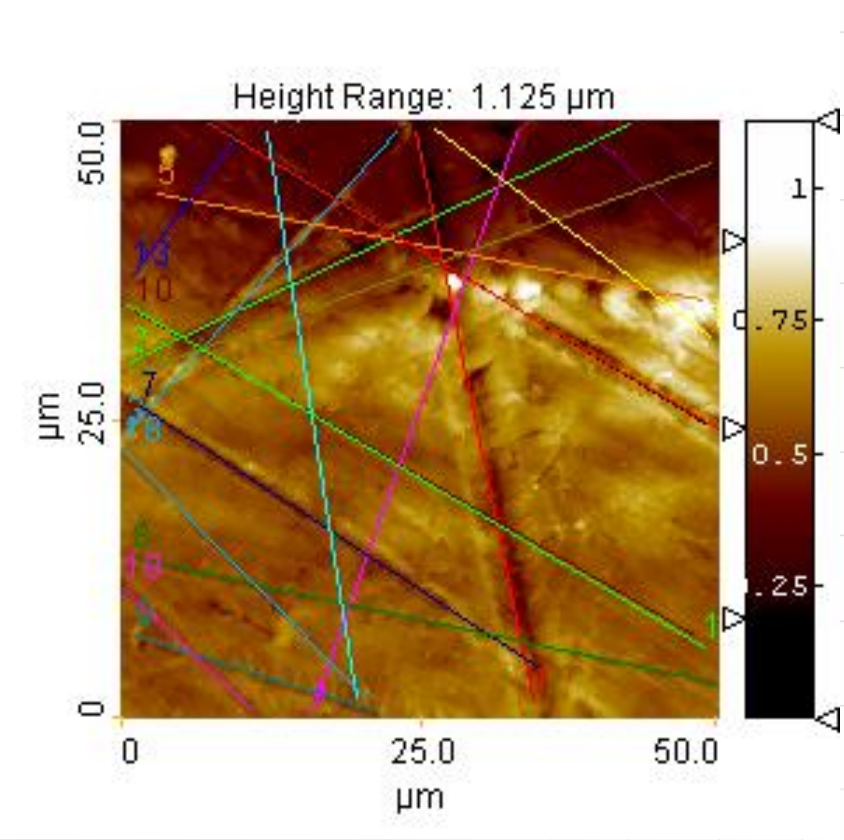
Sheet Moulded Composite – light and temperature induced degradation



Sikora A, Bednarz Ł, Fałat T, Wałecki M, Adamowska M, 2016, *The investigation of the simulated solar radiation impact on the micro- and nanoscale morphology and mechanical properties of the sheet moulded composite surface*, Materials Science-Poland, 34(3), 641-649

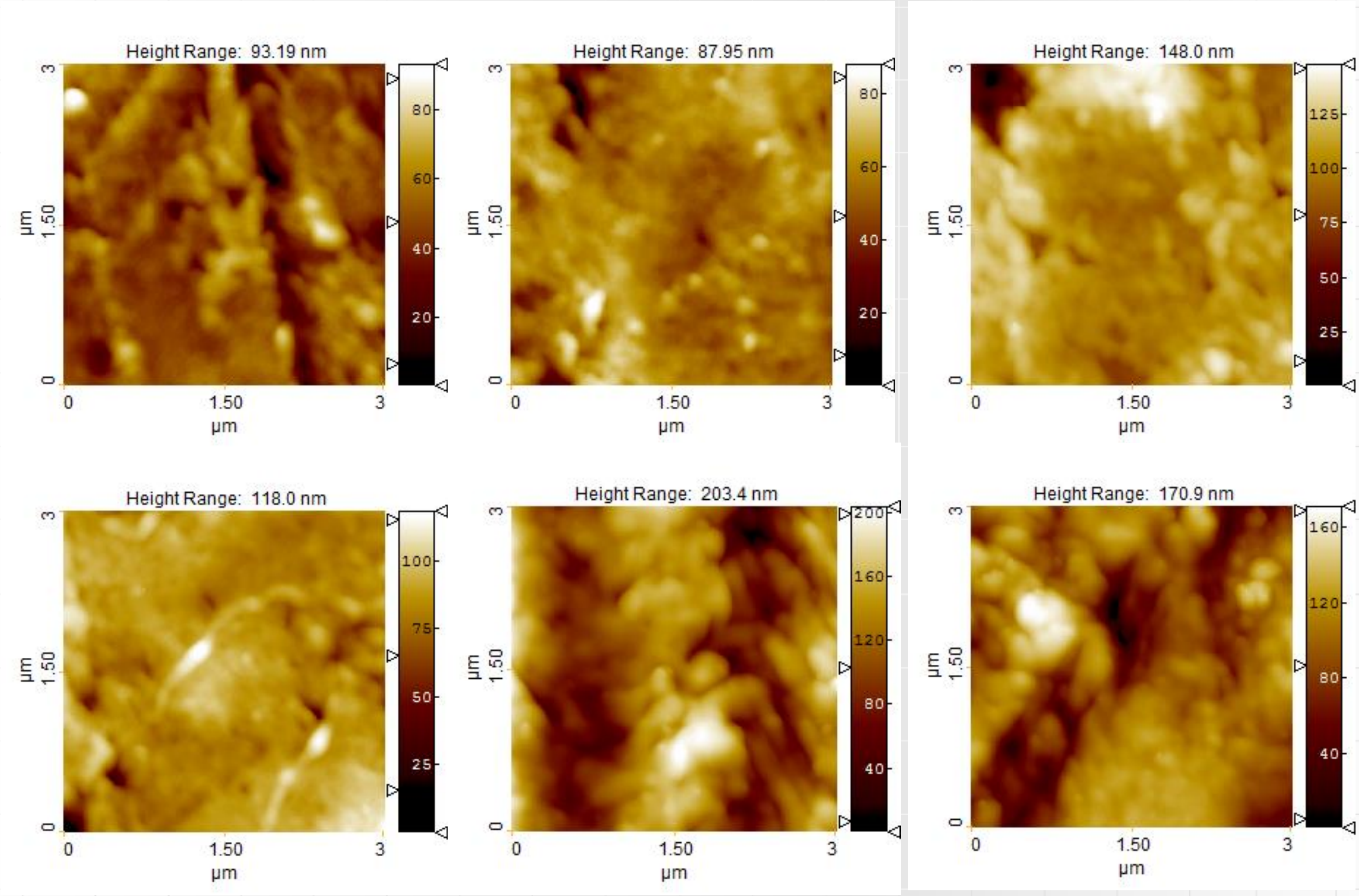
If all the samples were homogenous…





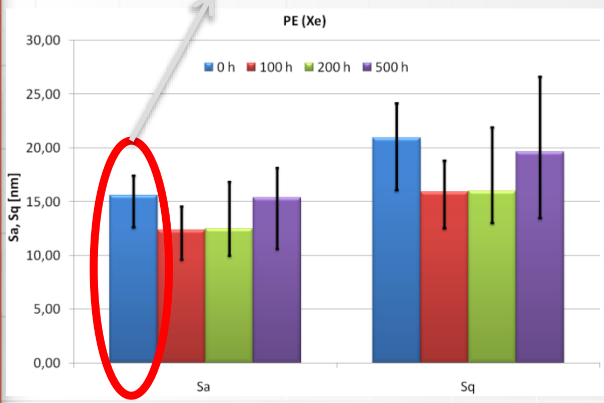
Sikora A, Grabarek A, Moroń L, Wałecki M, Kryla P, 2016, *The investigation of the light radiation caused polyethylene based materials deterioration by means of atomic force microscopy*, IOP Conference Series, IOP Conf. Ser. Mater. Sci. Eng. 113, 012016

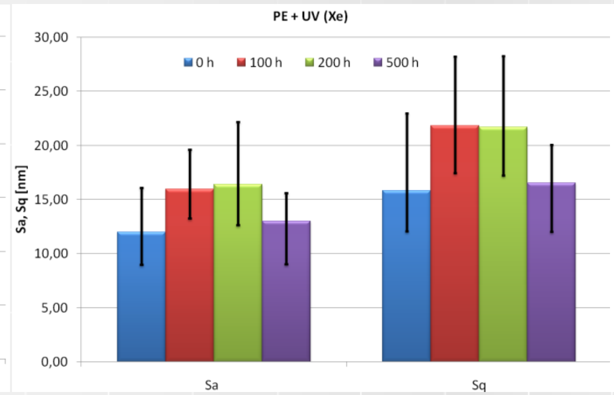
Polyethylene sample non – homogeneity example

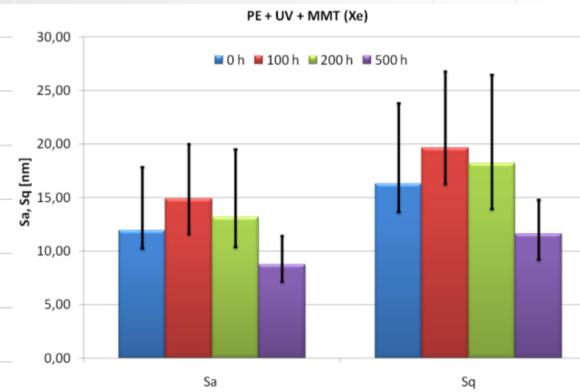


Polyethylene light induced degradation

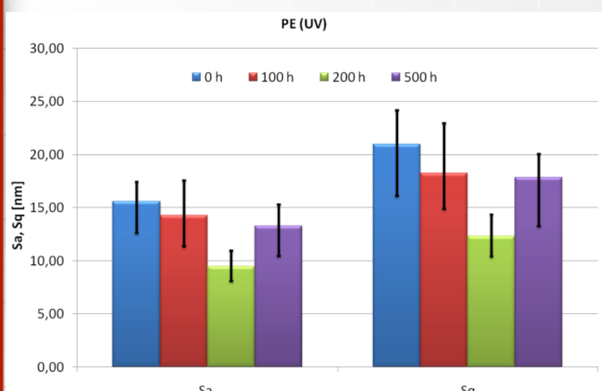
20-30 measurements Xenon

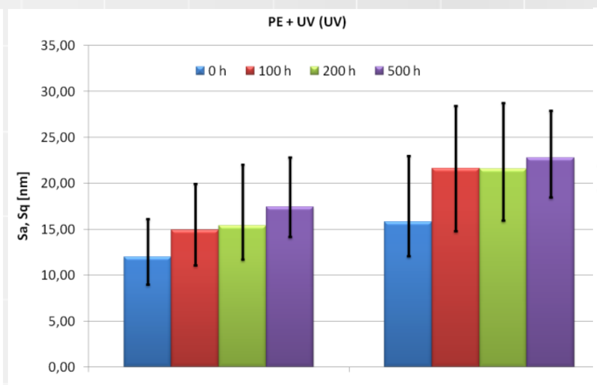


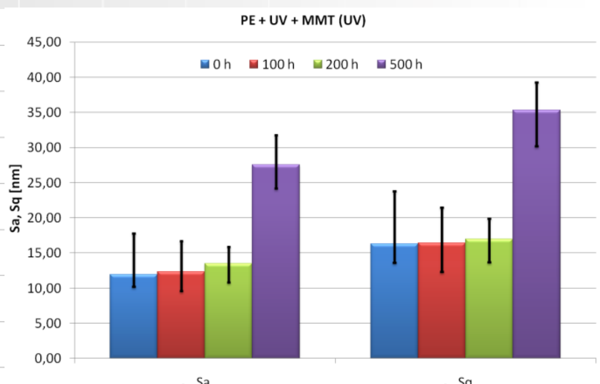




UV



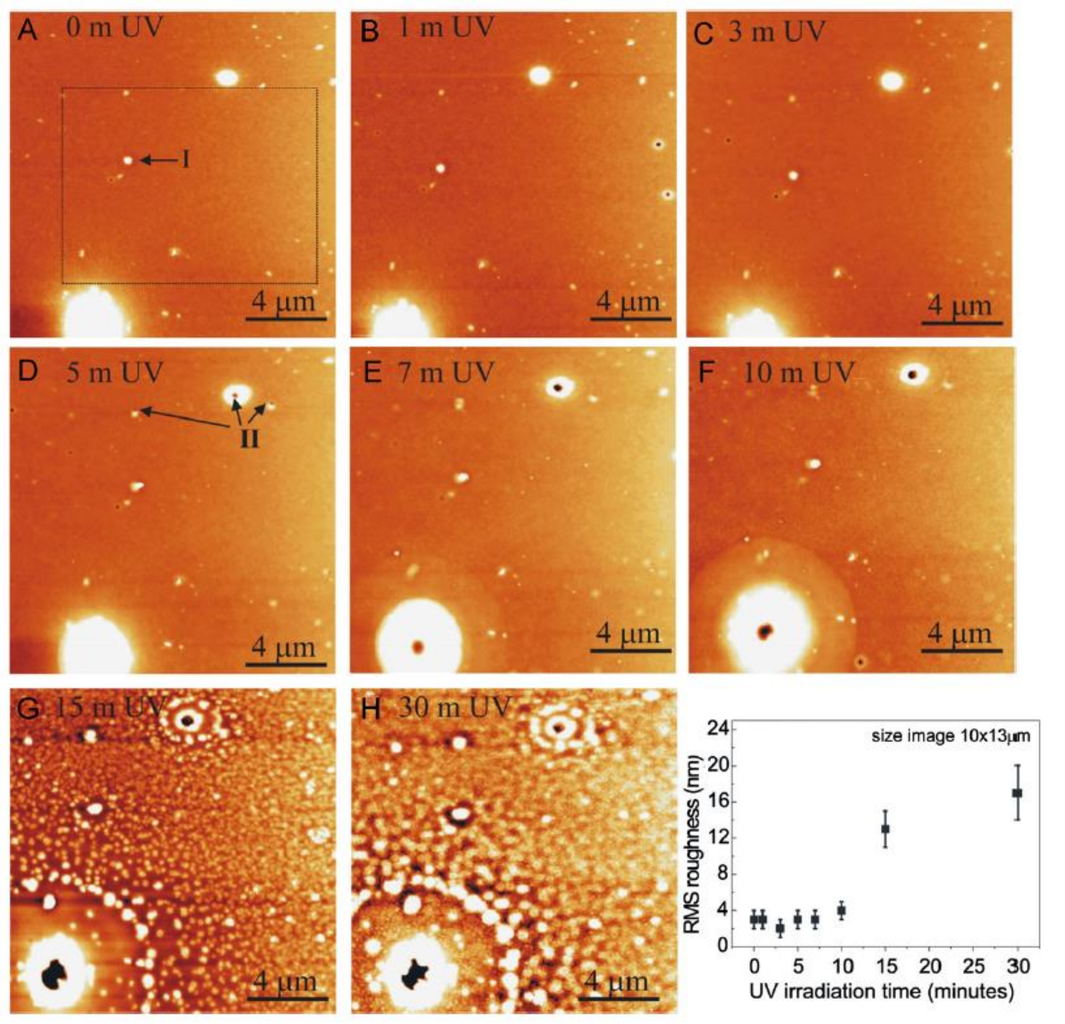




Sa Sq Sa Sq Sa Sq

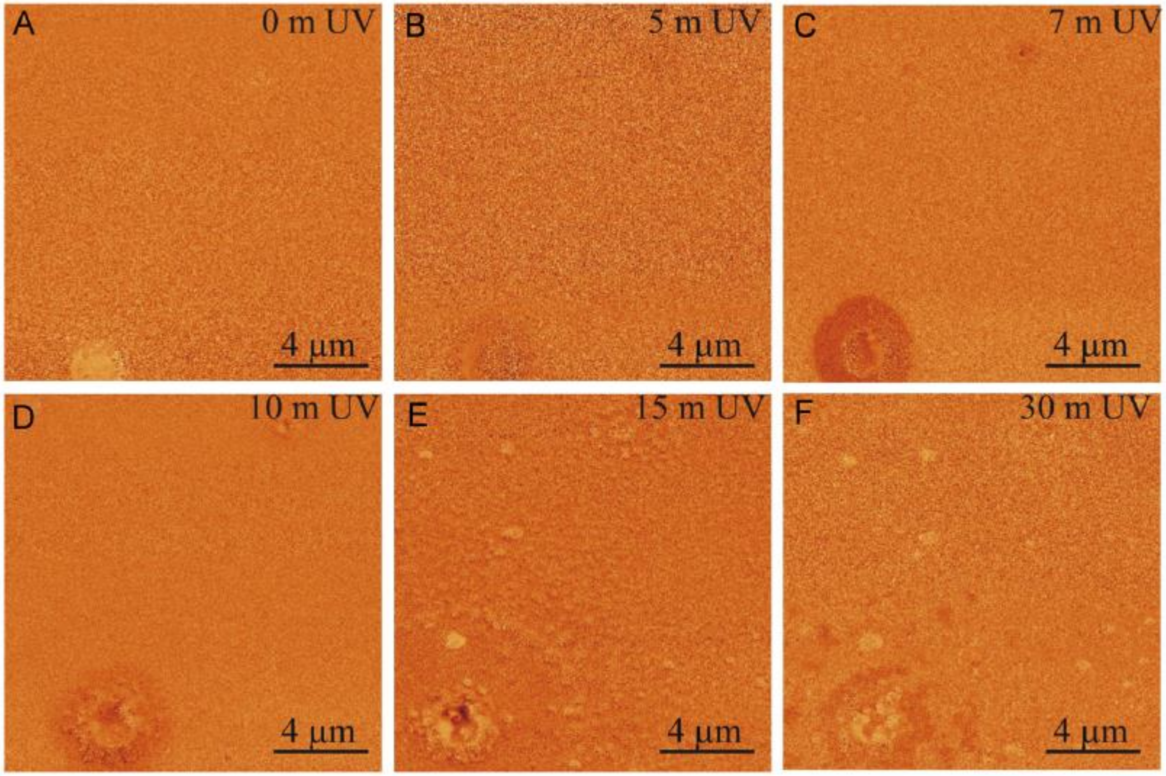
PE PE+UV PE+UV+MMT

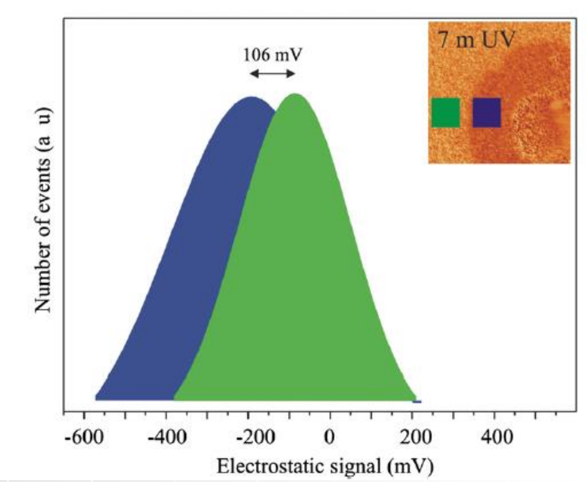
Surface degradation investigation using AFM

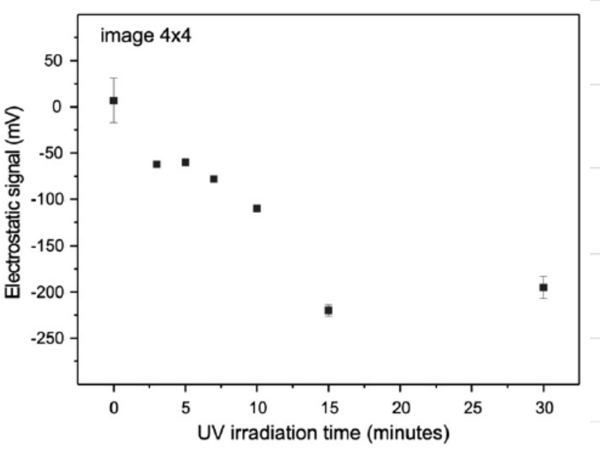


Jose Abad, Nieves Espinosa, Rafael Garcia-Valverde, Jaime Colchero, Antonio Urbina, *The influence of UV radiation and ozone exposure on the electronic properties of poly-3-octyl-thiophene thin films*, Solar Energy Materials & Solar Cells 95 (2011) 1326–1332

Surface degradation investigation using AFM

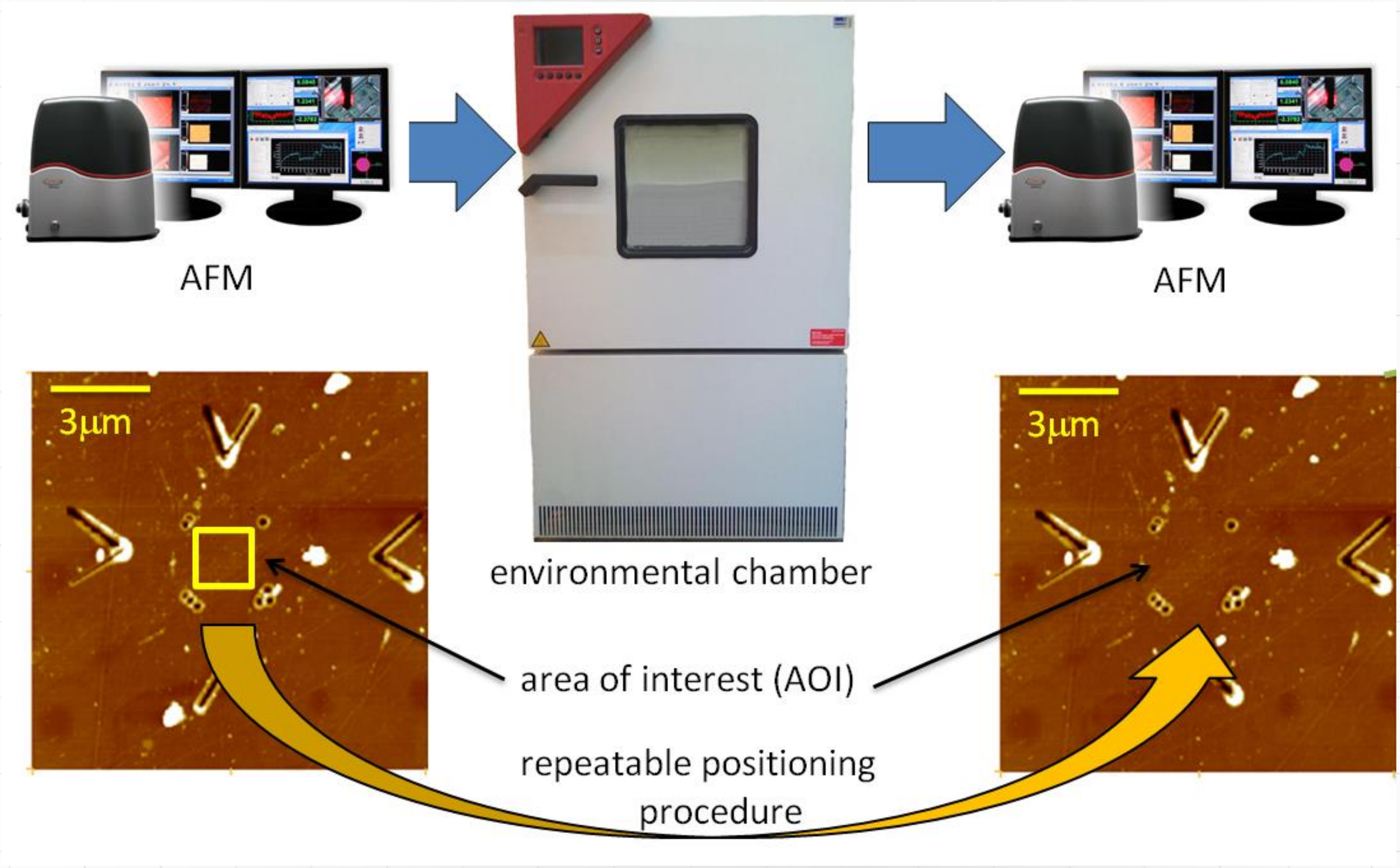






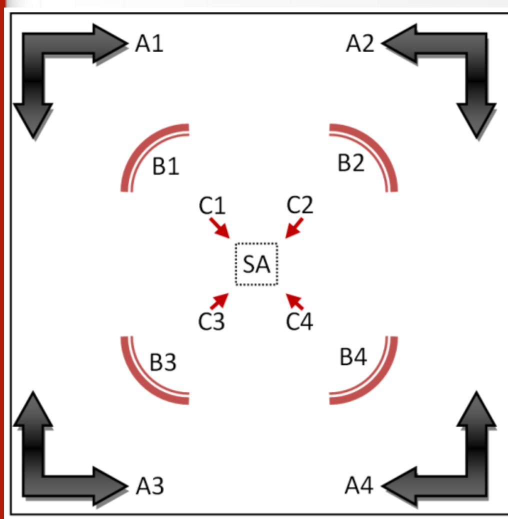
Jose Abad, Nieves Espinosa, Rafael Garcia-Valverde, Jaime Colchero, Antonio Urbina, *The influence of UV radiation and ozone exposure on the electronic properties of poly-3-octyl-thiophene thin films*, Solar Energy Materials & Solar Cells 95 (2011) 1326–1332

…and finding a certain spot while moving the sample between devices

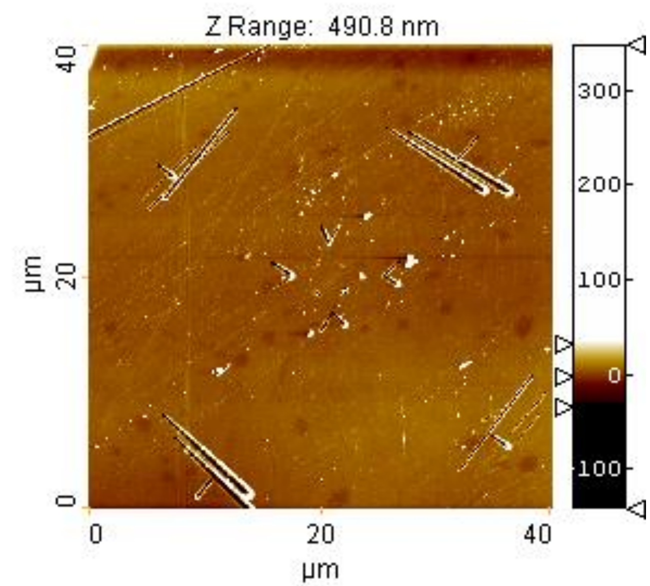


Nanomarkers - quick and easy sample positioning

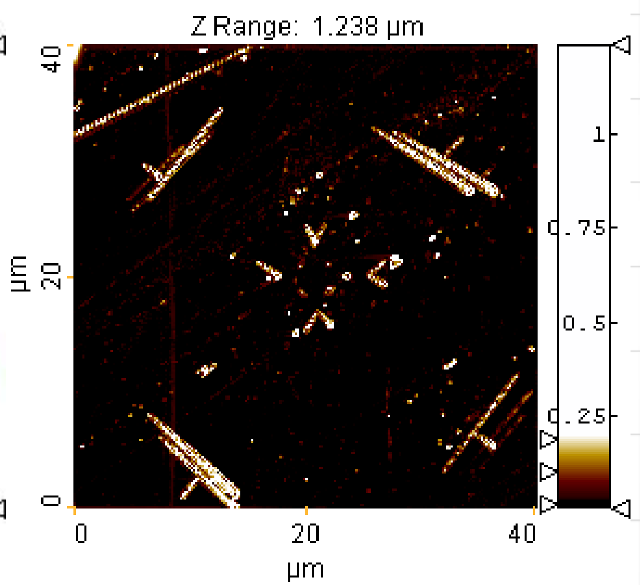
polycarbonate



diagram



topography



inclination transform of topography

Andrzej Sikora, *Development and utilization of the nanomarkers for precise AFM tip positioning in the investigation of the surface morphology change*, Optica Applicata Vol. 43, No. 1, 2013, 163-171

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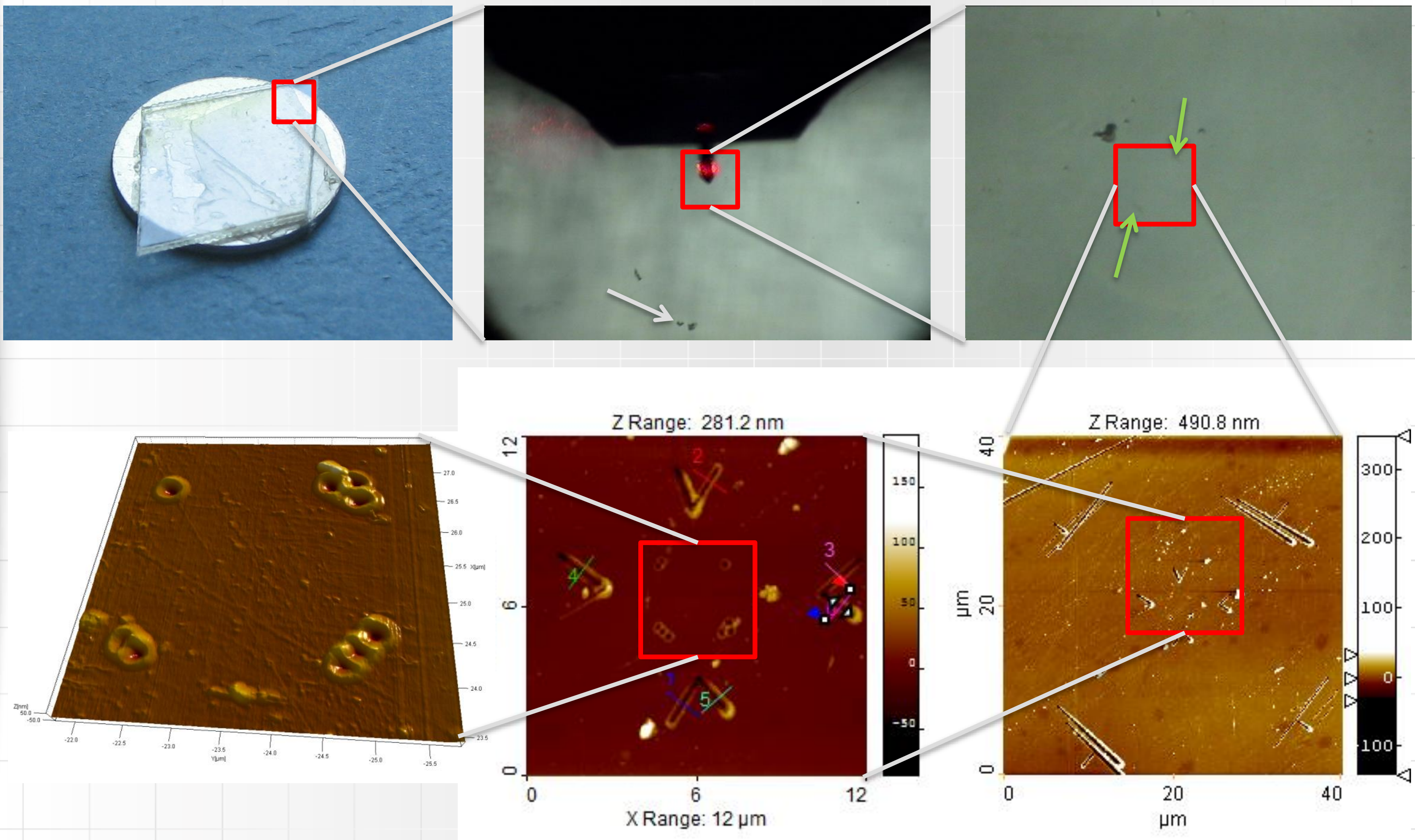
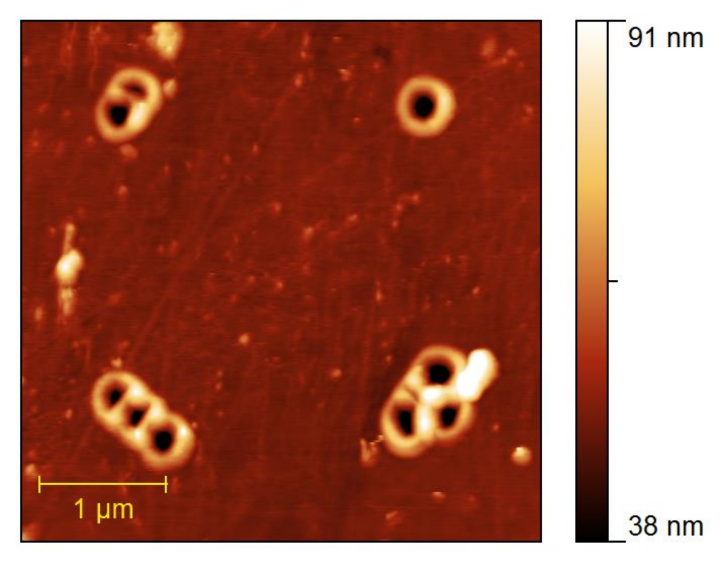
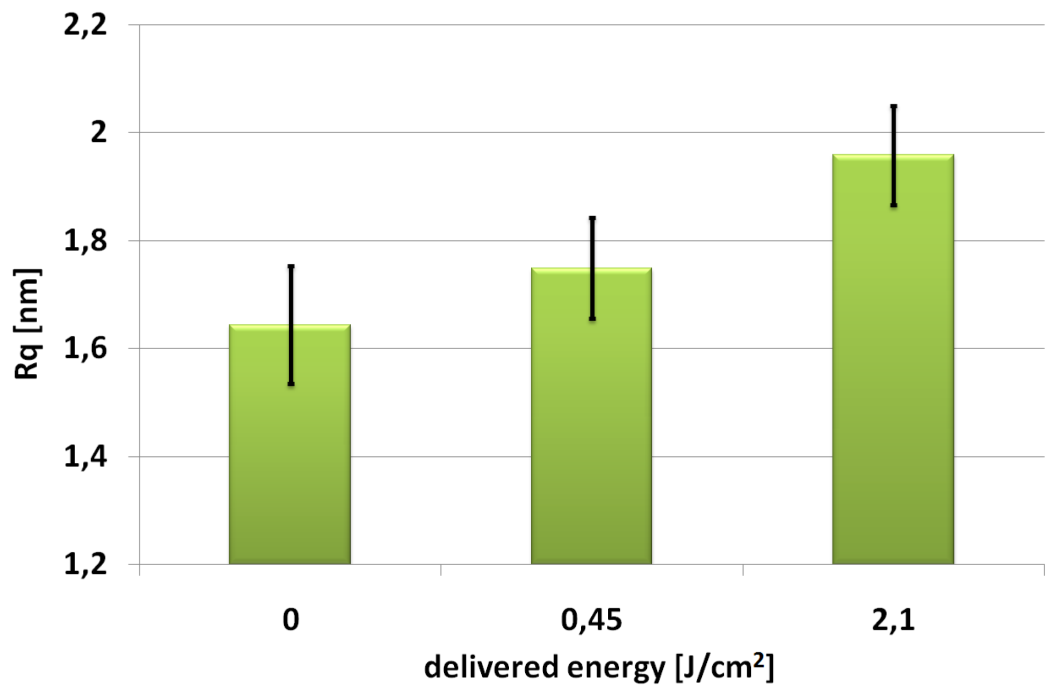
Nanomarkers – quick and easy sample positioning method



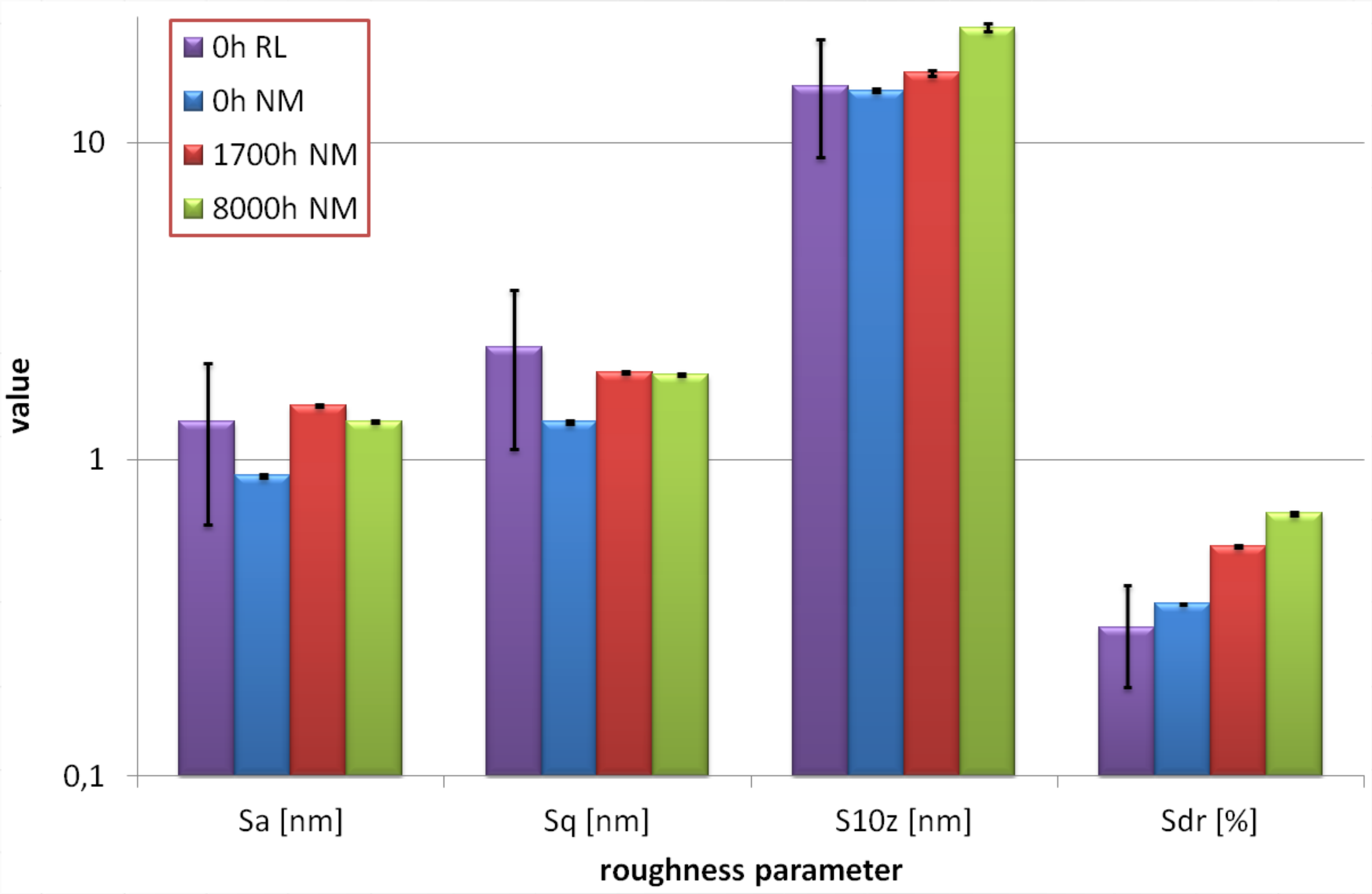
Repetitive and precise sample positioning





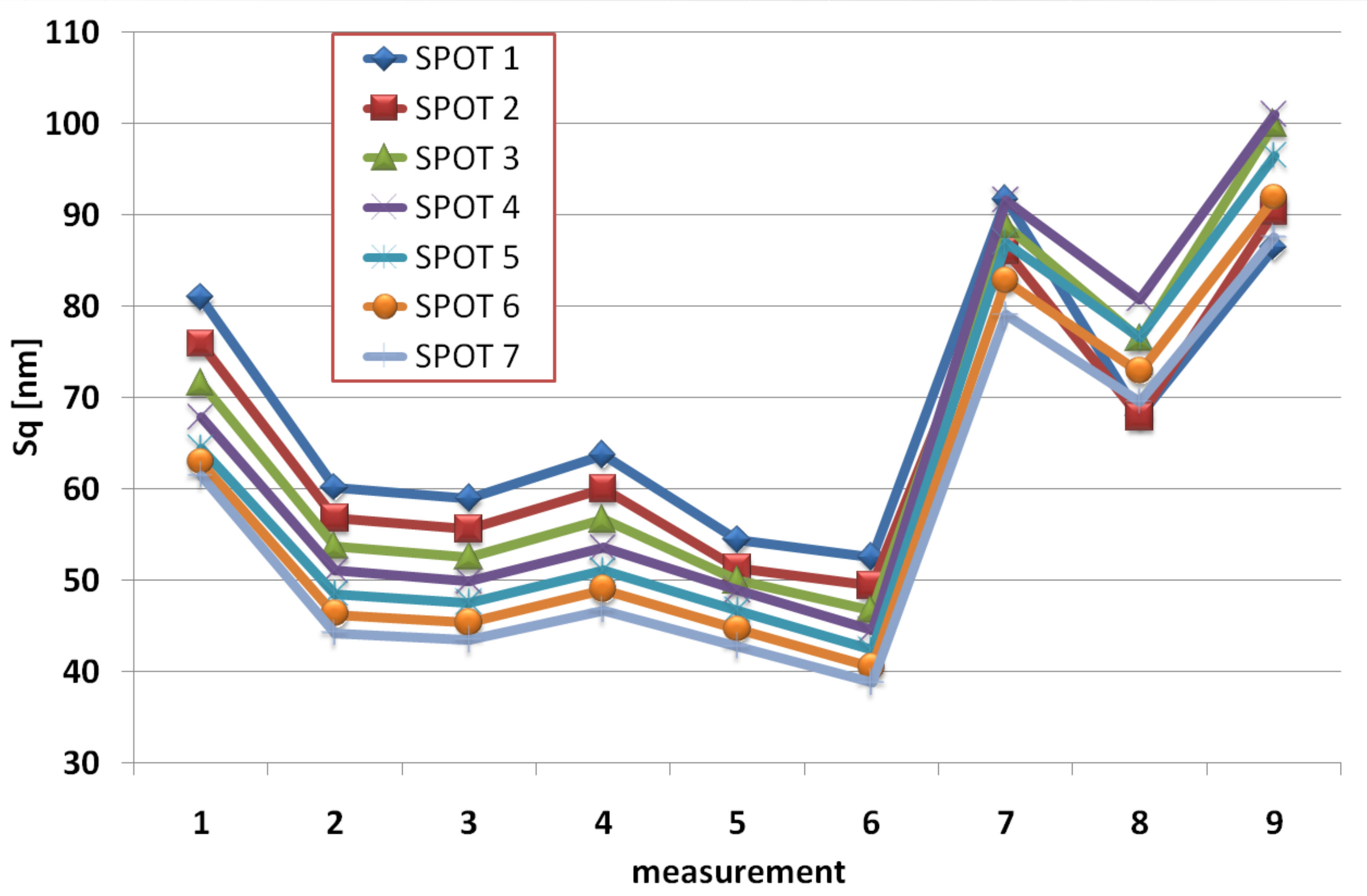
A. Sikora, *Micro- and nanoscale evaluation of the materials deterioration. Towards faster and cheaper tests and development of the materials for electrical applications*, Numerical Modelling and Simulation, ed. J. Sikora, E. Usak, Electrical Engineering Institute printing office, 7-10

The roughness changes observation using positioning solution

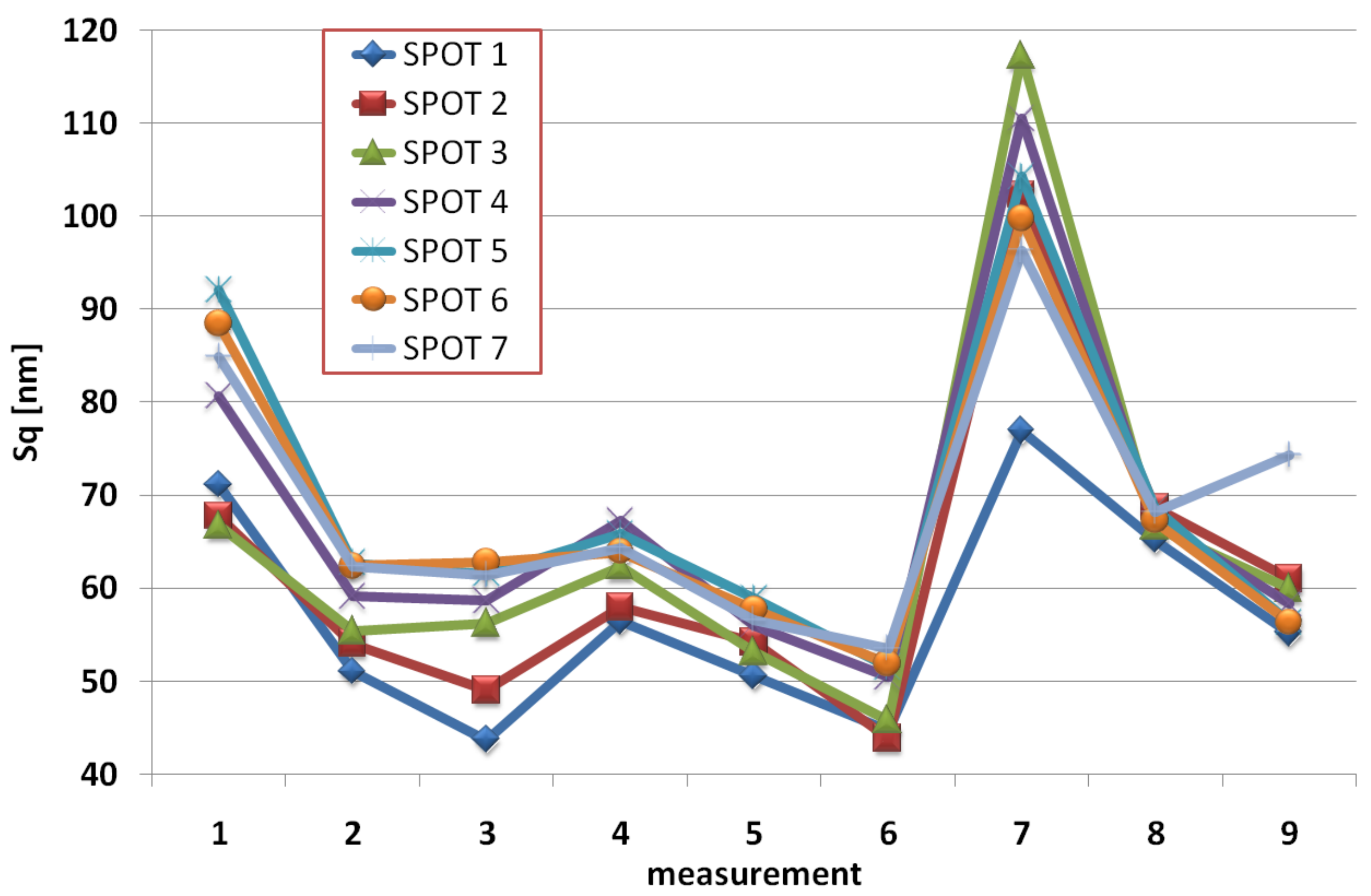


Andrzej Sikora, *Improvement of the scanning area positioning repeatability using the nanomarkers developed with nanoscratching method*, Measurement Science and Technology 25 (2014) 055401

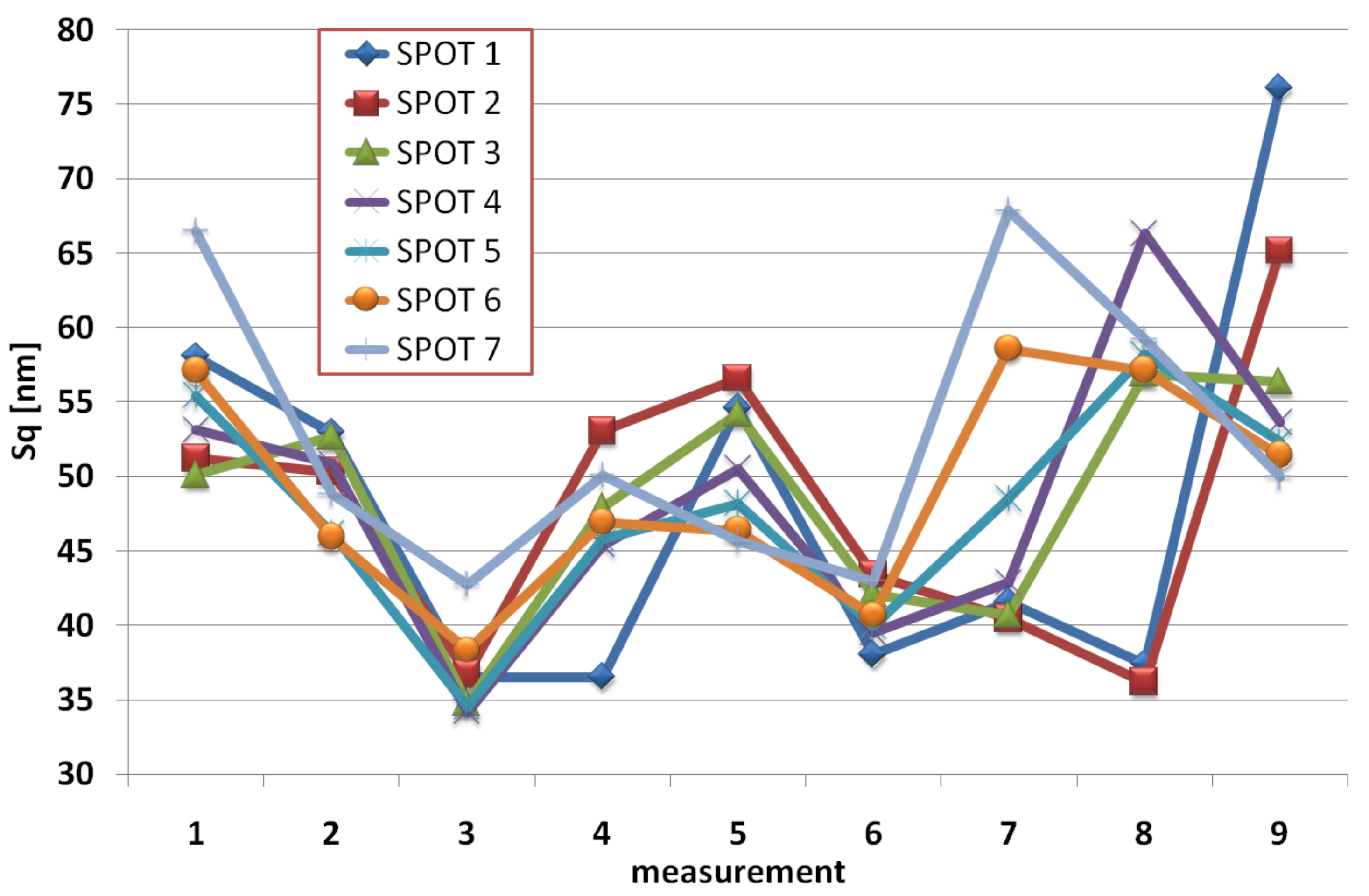
Polycarbonate degradation profile



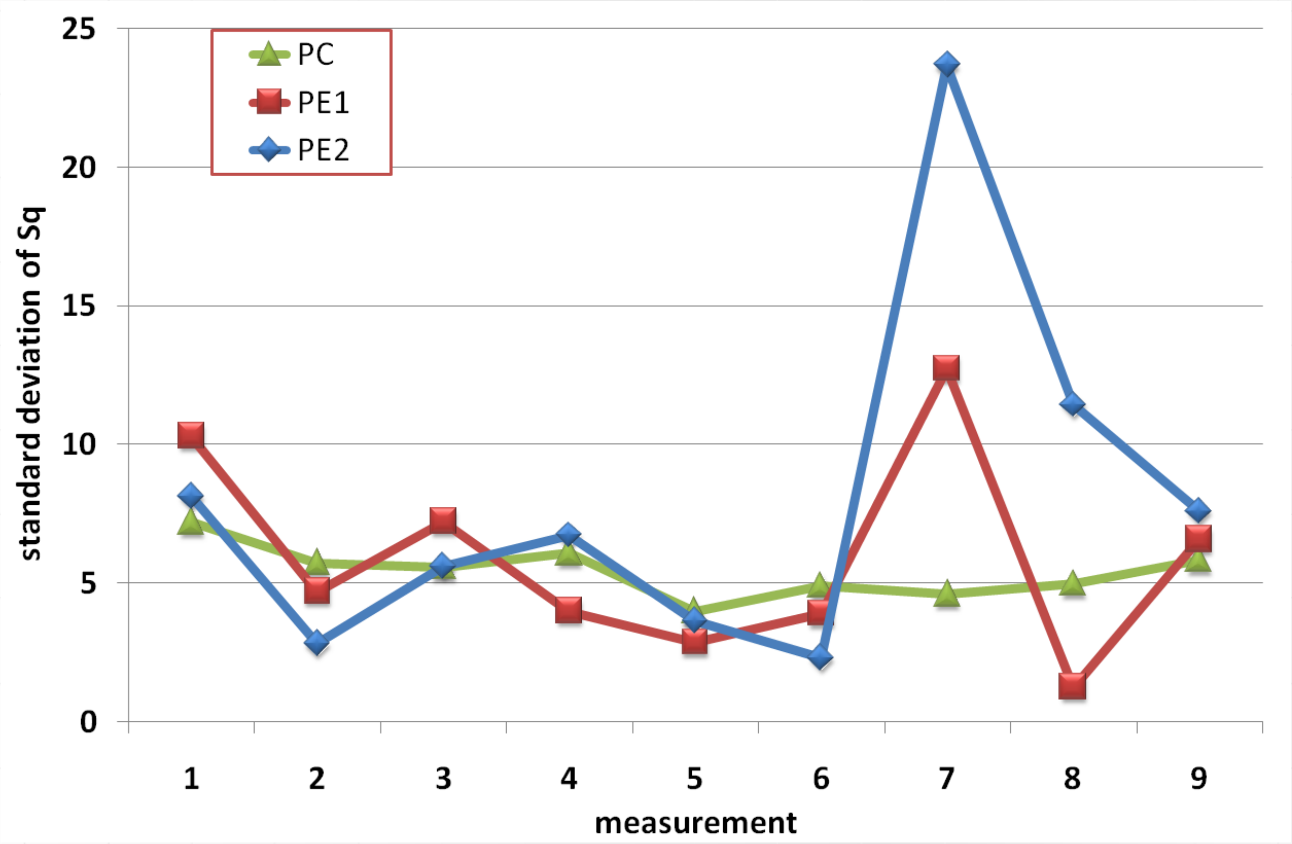
Polyethylene degradation profile



Polyethylene with soot degradation profile



Roughness distribution of investigated materials



Sikora A, *The improvement of the roughness changes analysis of the non-uniform surfaces investigated by means of atomic force microscopy with precise repetitive scanning area positioning*, Measurement Science and Technology, 2017, 28 034016

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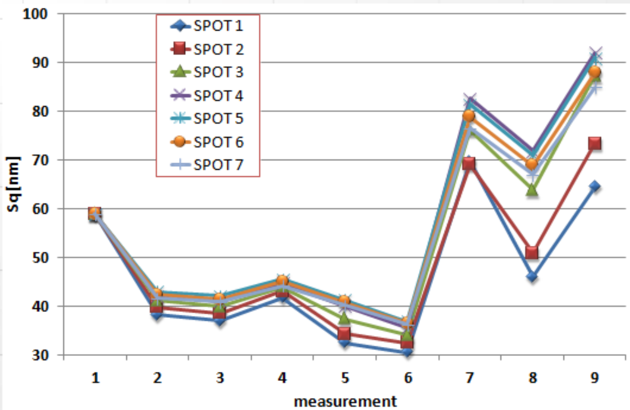
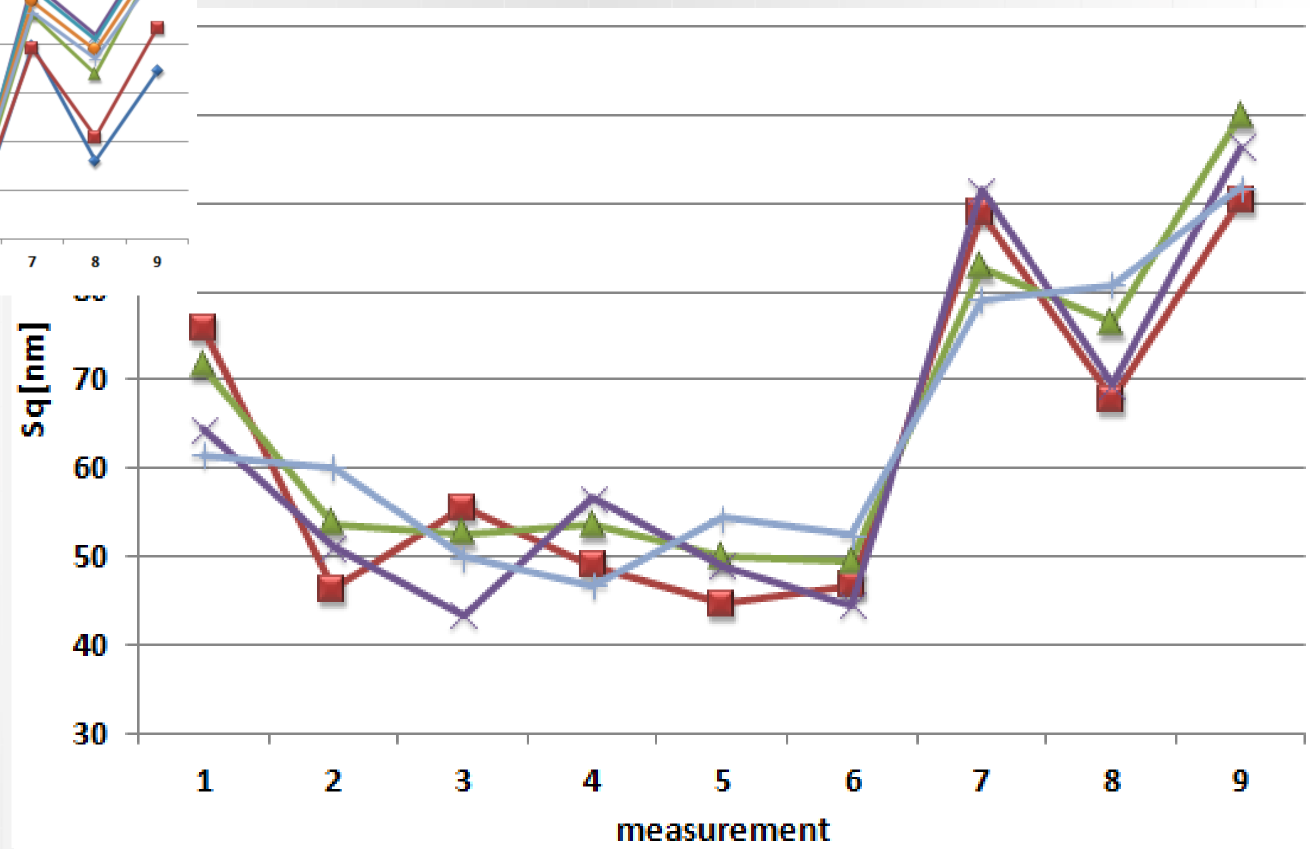
Modeling of random accessing of investigated spots on PC surface



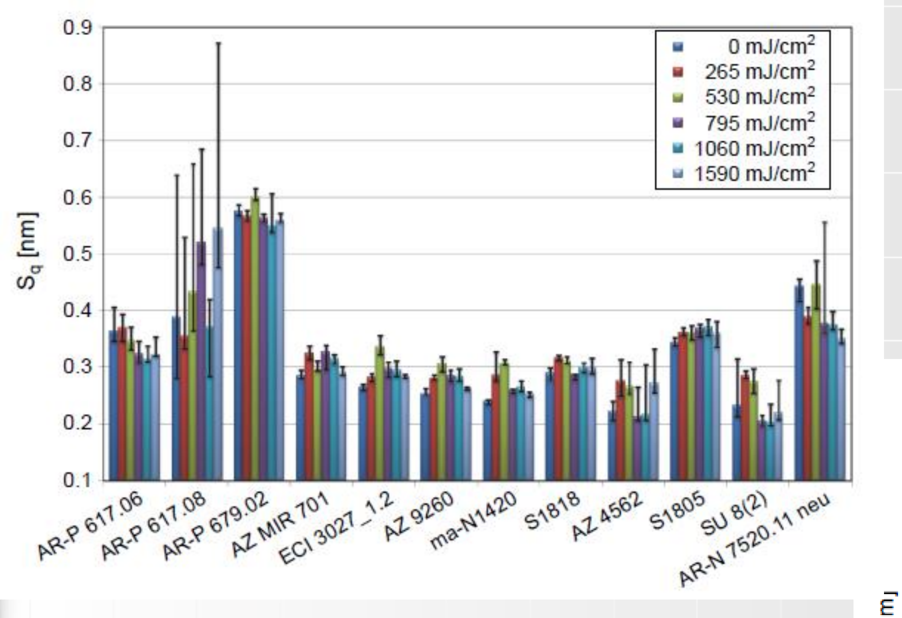
ordered measured areas

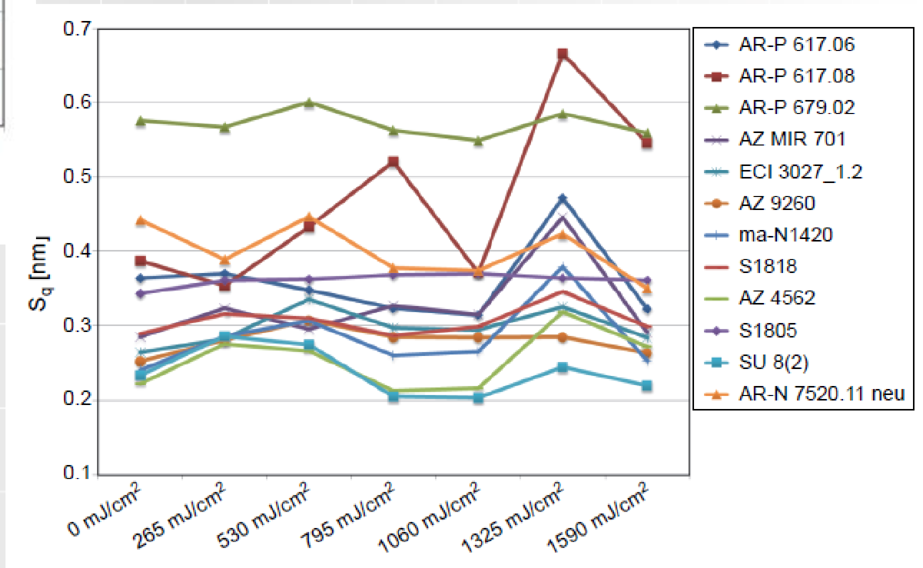
mixed measured areas



Andrzej Sikora, *The improved accuracy and sensitivity of the observation of the surface's deterioration by means of atomic force microscopy supported with repetitive spatial high-accuracy sample positioning*, Nanoscience and Nanometrology, Vol. 3, No. 1, 2017, pp. 6-11

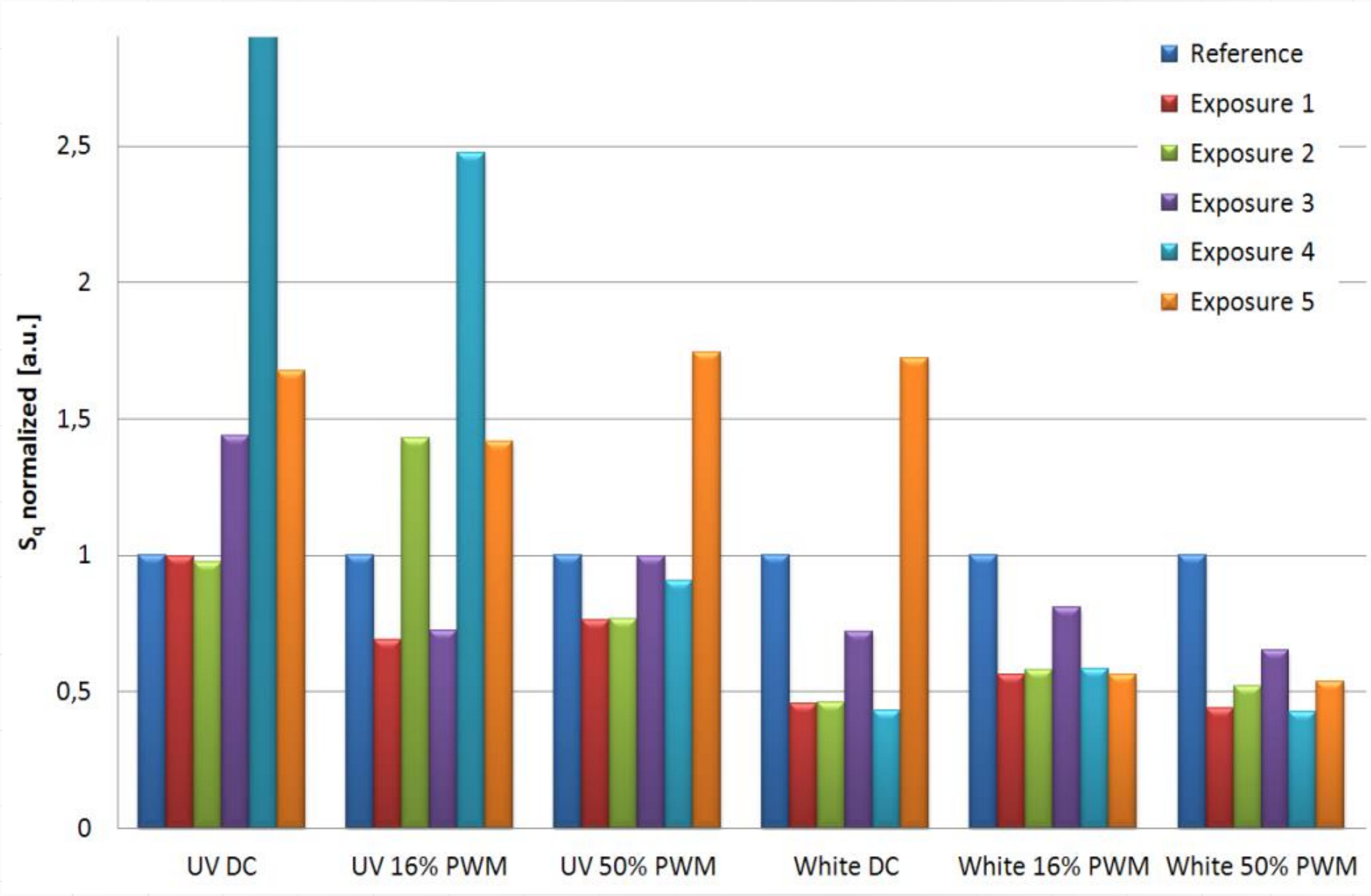
The roughness changes of photoresists after the exposition to UV light





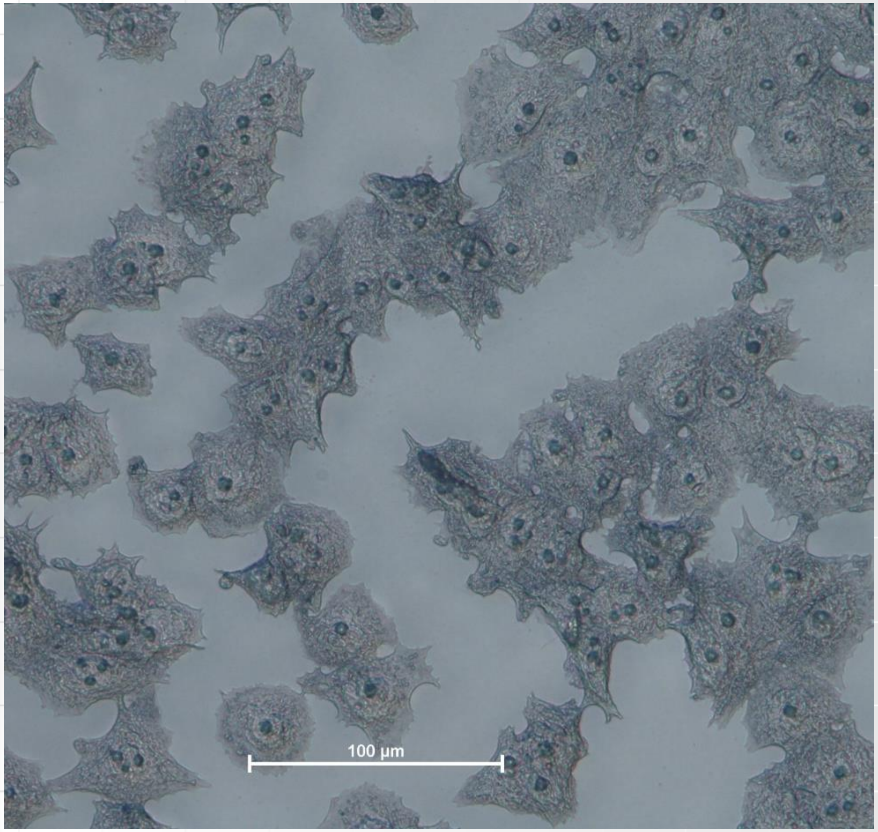
A. Sikora, P. Janus, A. Sierakowski, *The impact of the light exposure on the morphological properties of selected photoresists*, Optica Applicata, Vol. XLIX, No. 1, 177-185, 2019

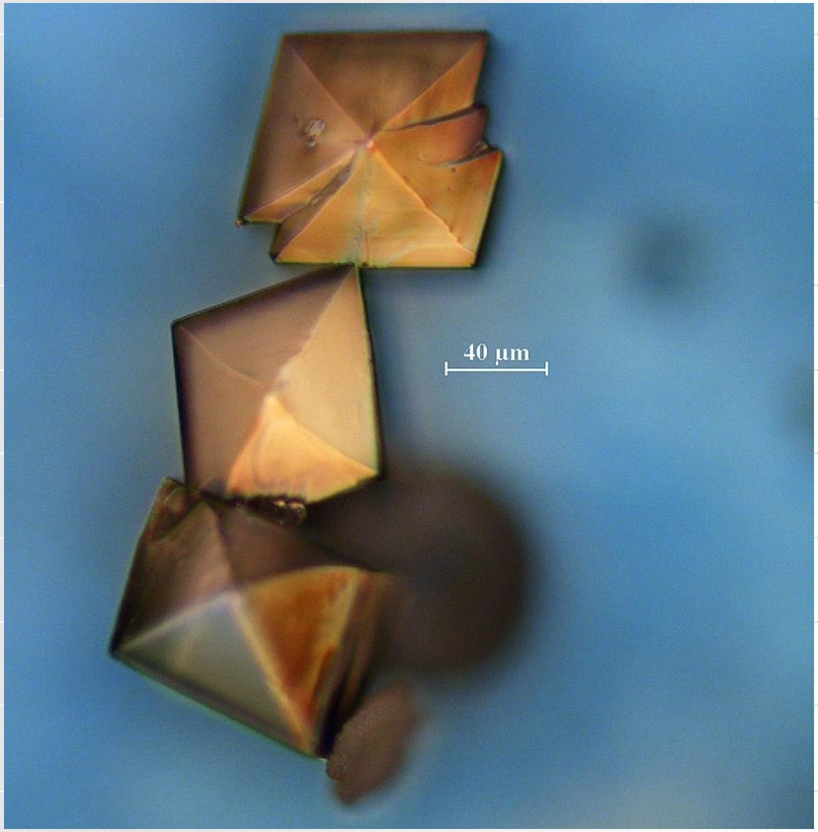
The observation of deterioration dynamics of PMMA for various modes of LED system powering



A. Sikora, K. Tomczuk, *The impact of the LED-based light source working regime on the degradation of polymethyl methacrylate*, Lighting Research & Technology, 0, 2019, 1-12

Nanoscratching-based markers have limited application area





M. Poznar, J. Stolarski, **A. Sik**ora, M. Mazur, J. Olesiak-Bańska, K. Brach, A. Ożyhar, P. Dobryszycki, *Fish Otolith Matrix Macromolecule-64 (OMM-64) and Its Role in Calcium Carbonate Biomineralization*, Cryst. Growth Des. 2020, 20, 9, 5808–5819

M. Moczała, M. Karpińska, M. Poznar, P. Dobryszycki, **A. Sikora**, *Application of argon plasma sheet in the etching process of calcium carbonate crystals for AFM tests*, Materials Science – Poland 36(1), 2018, 75-79 10.1515/msp-2018-0016

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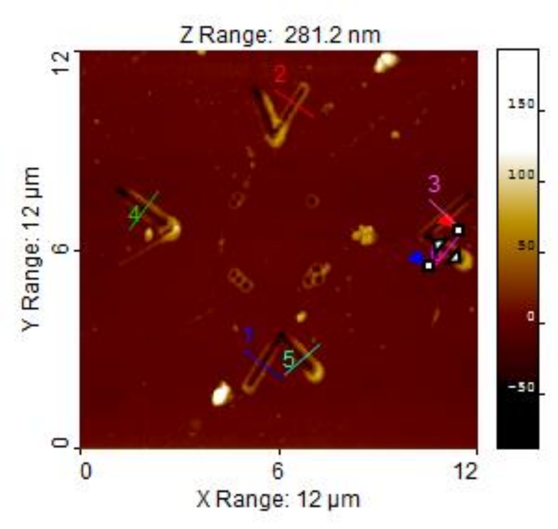
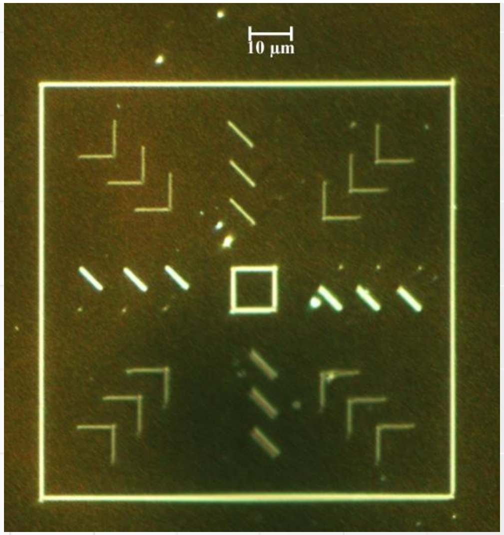
Various solutions enabling easy sample positioning

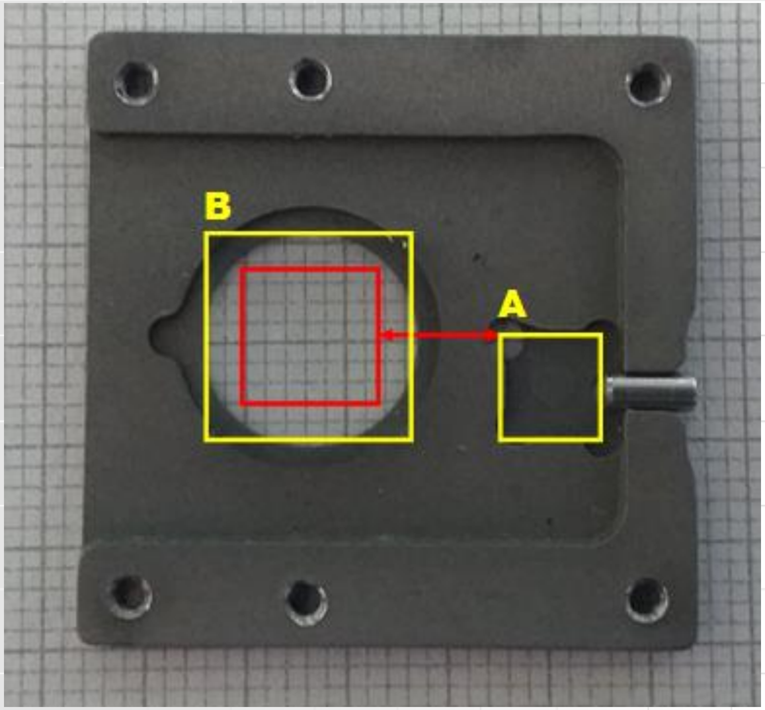
nanoscratching



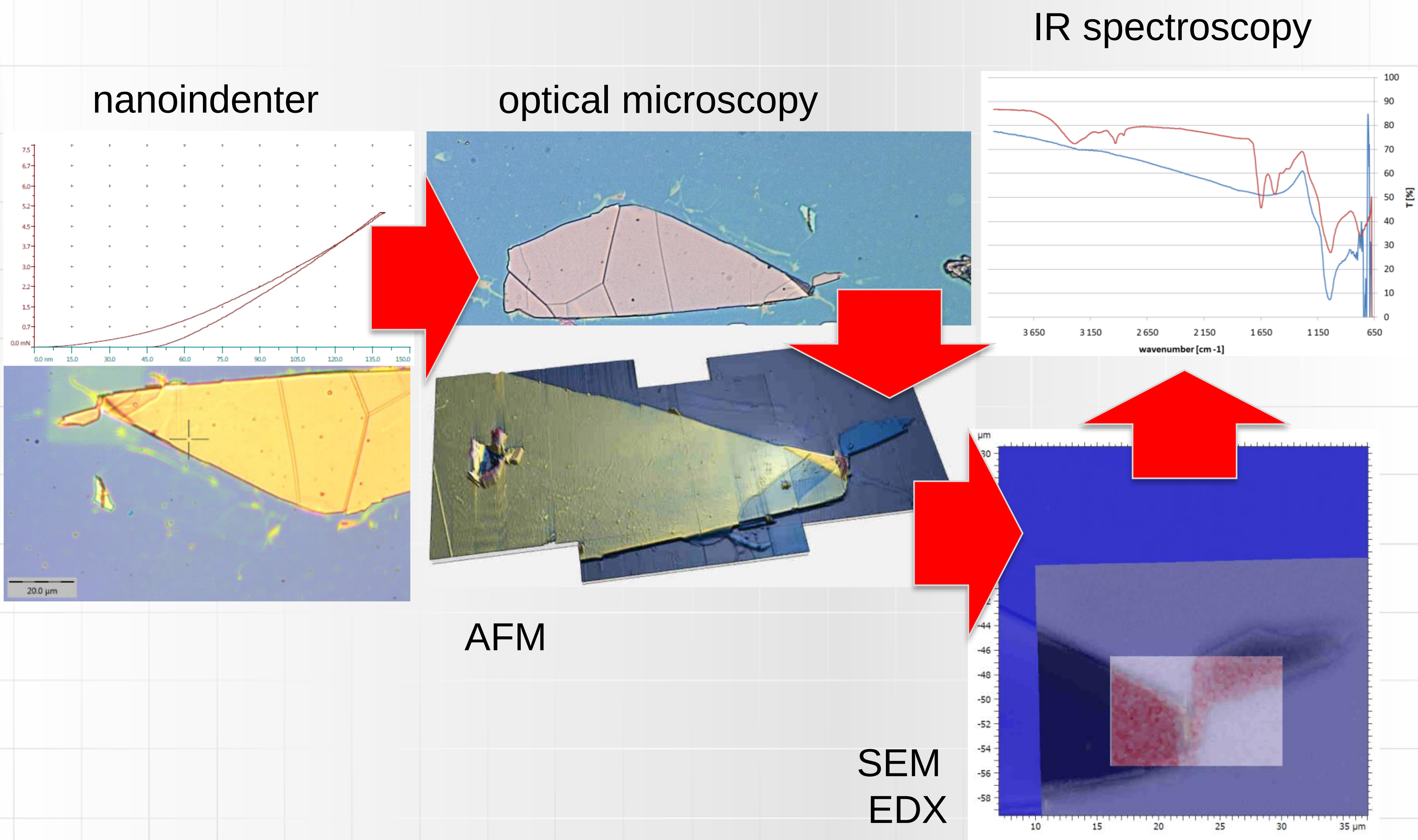
FIB



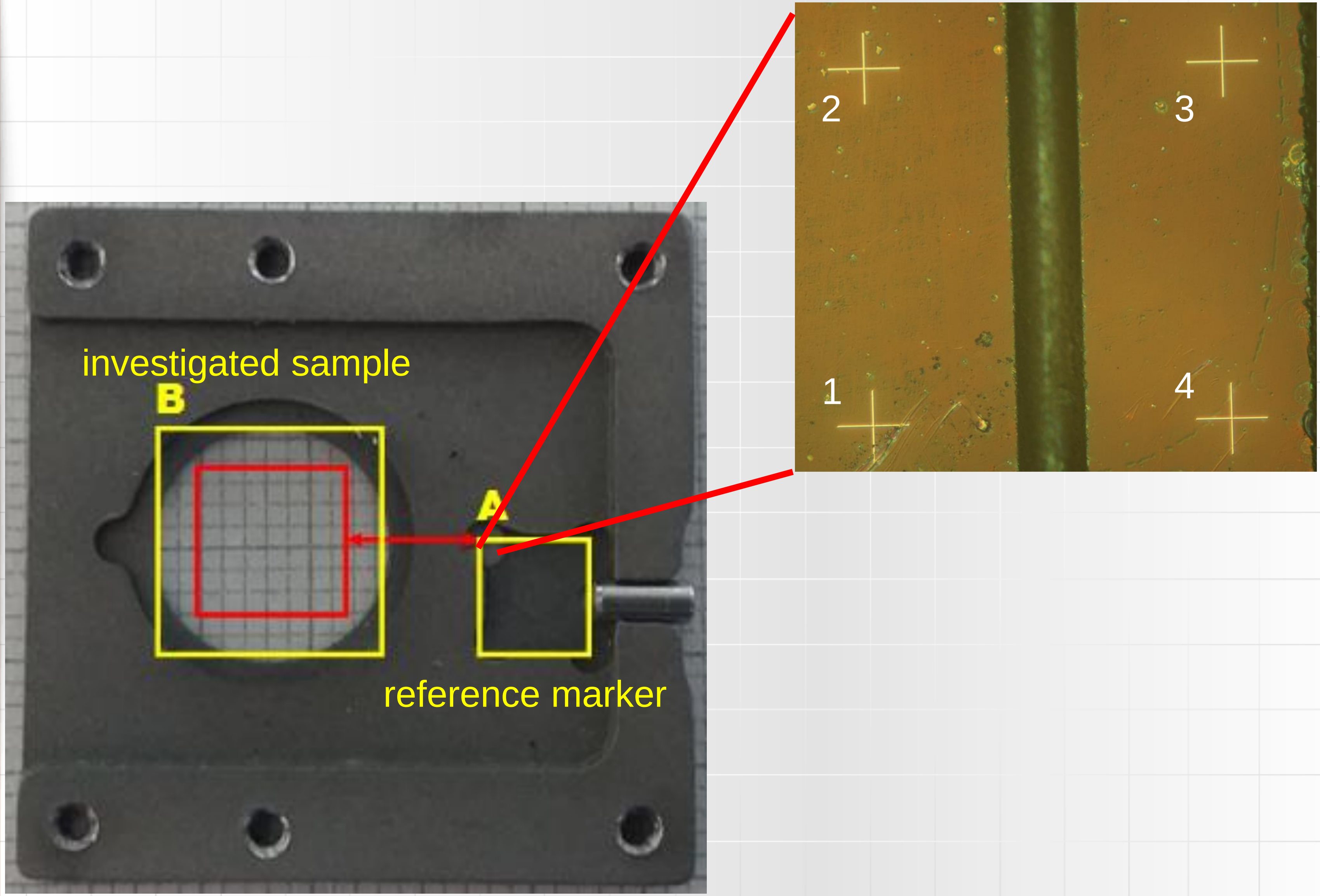
CORRESCOPY
CORRELATIVE IMAGING



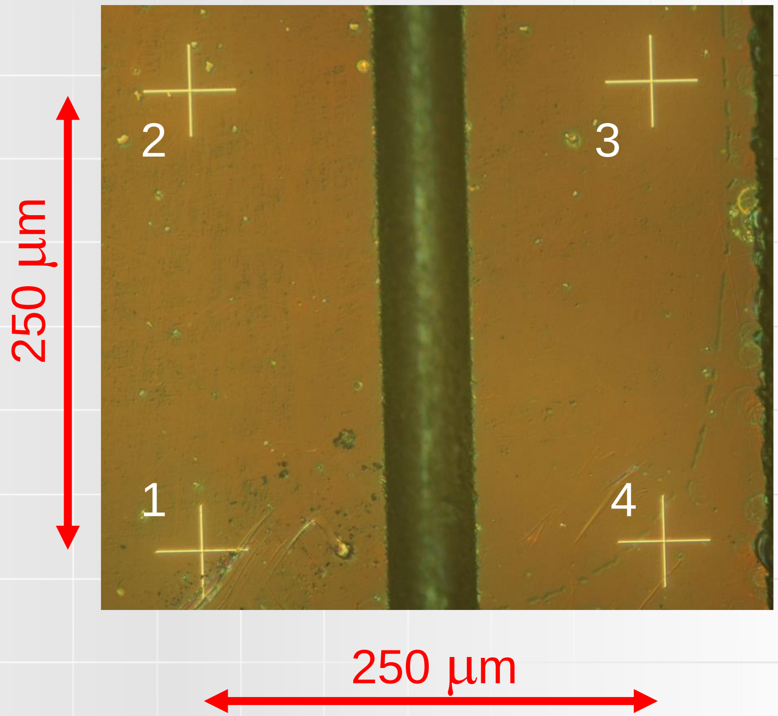
Correlative microscopy - examples of investigation with various devices



Correlative microscopy sample holder - how accurate the positioning is possible?



Correlative microscopy sample holder - how accurate the positioning is possible?

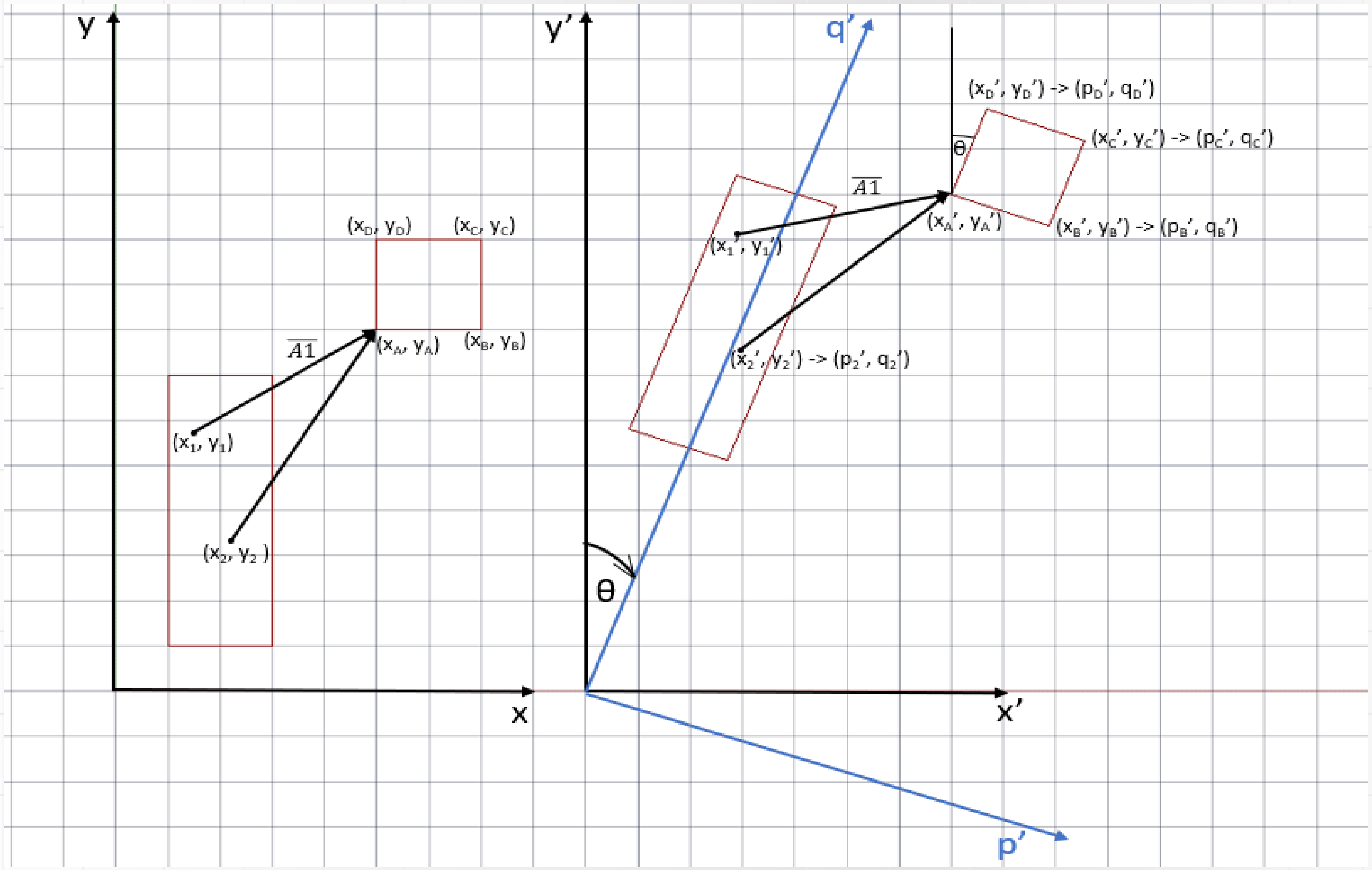


2-points based reference (1 rotation angle can be determined)

4-points based reference (4 rotation angles can be determined)

Does the effort provide better positioning?

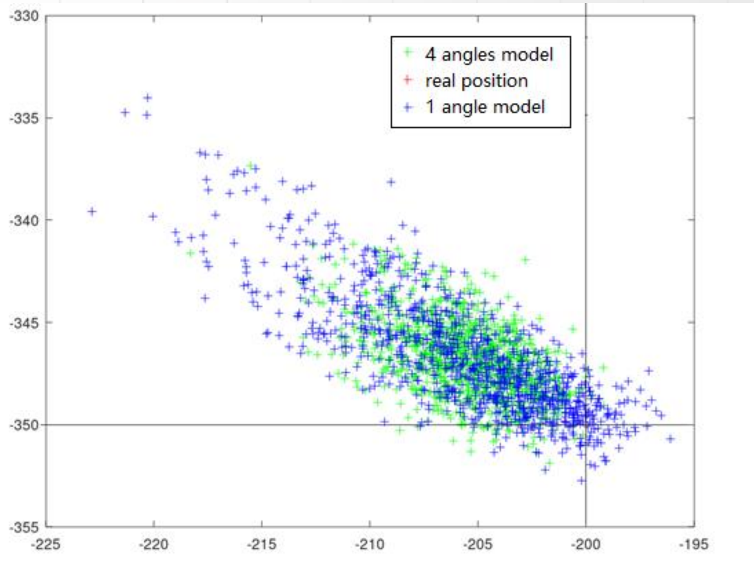
Extending solution to four reference points



acquisition of the coordinates set (1st device)

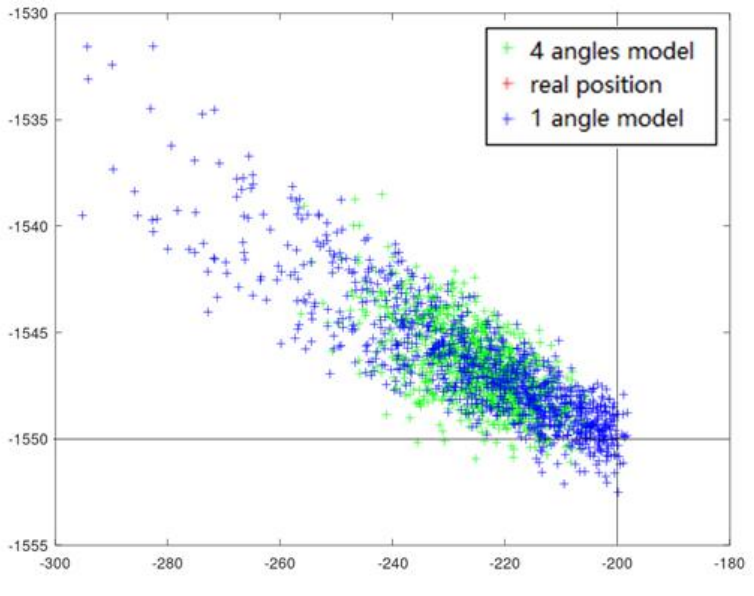
determination of new coordinates after the sample repositioning (following devices)

Positioning accuracy simulations

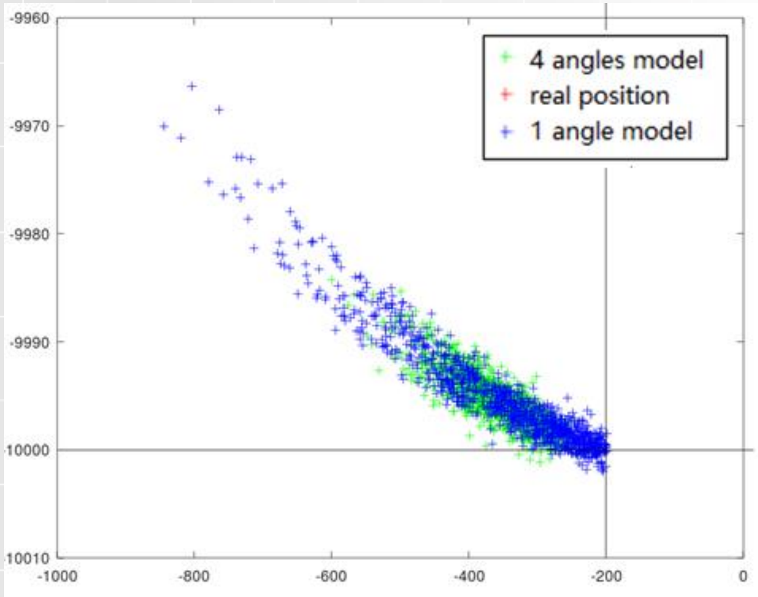


Statistical dispersion around point (-200, -350), σ = 2

Coordinates of investigated spots are provided as the input parameter as well as the position readout accuracy of certain device (sigma).
Coordinates of the reference point is (0,0).

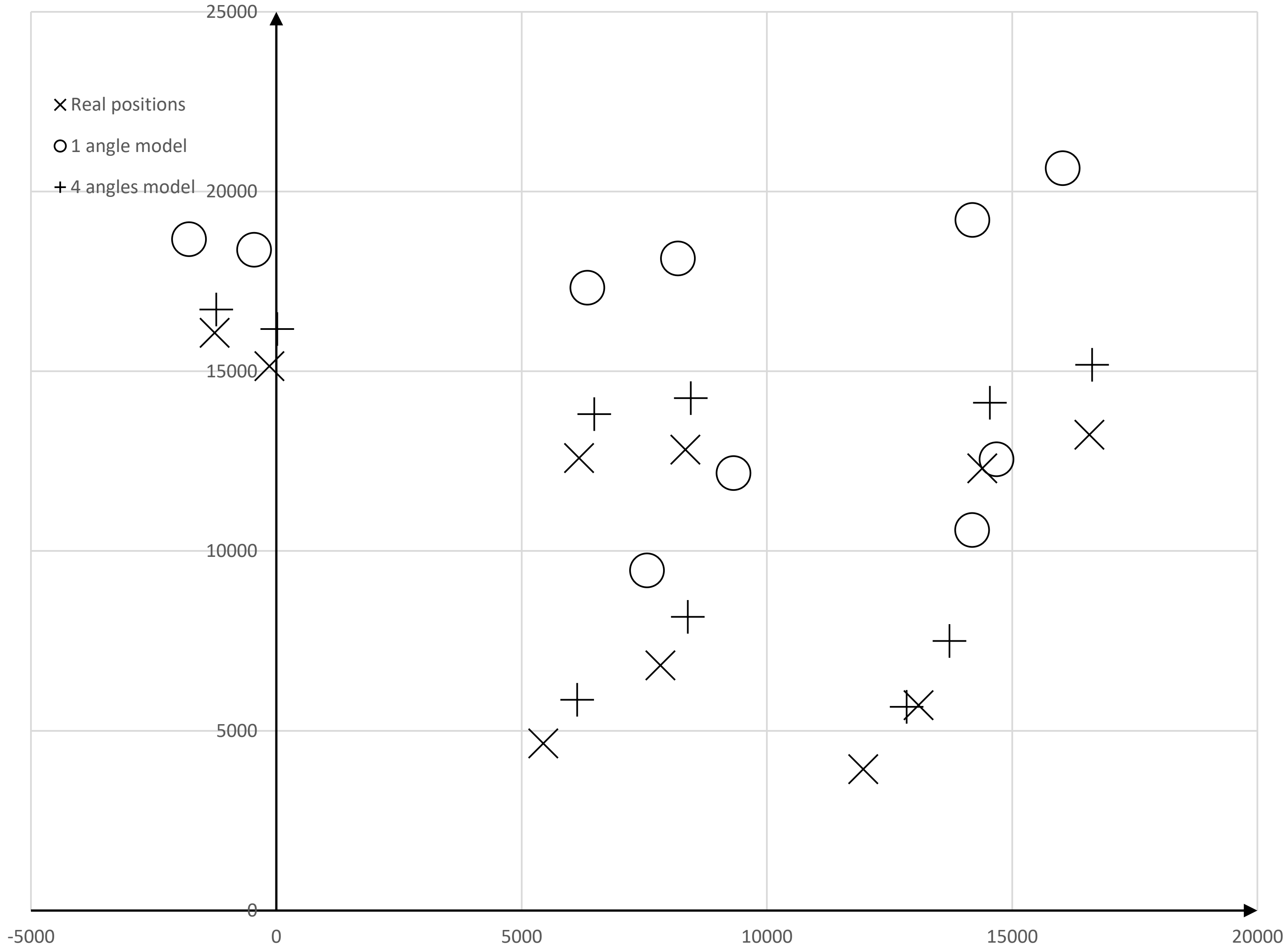


Statistical dispersion around point (-200, -1550), σ=2



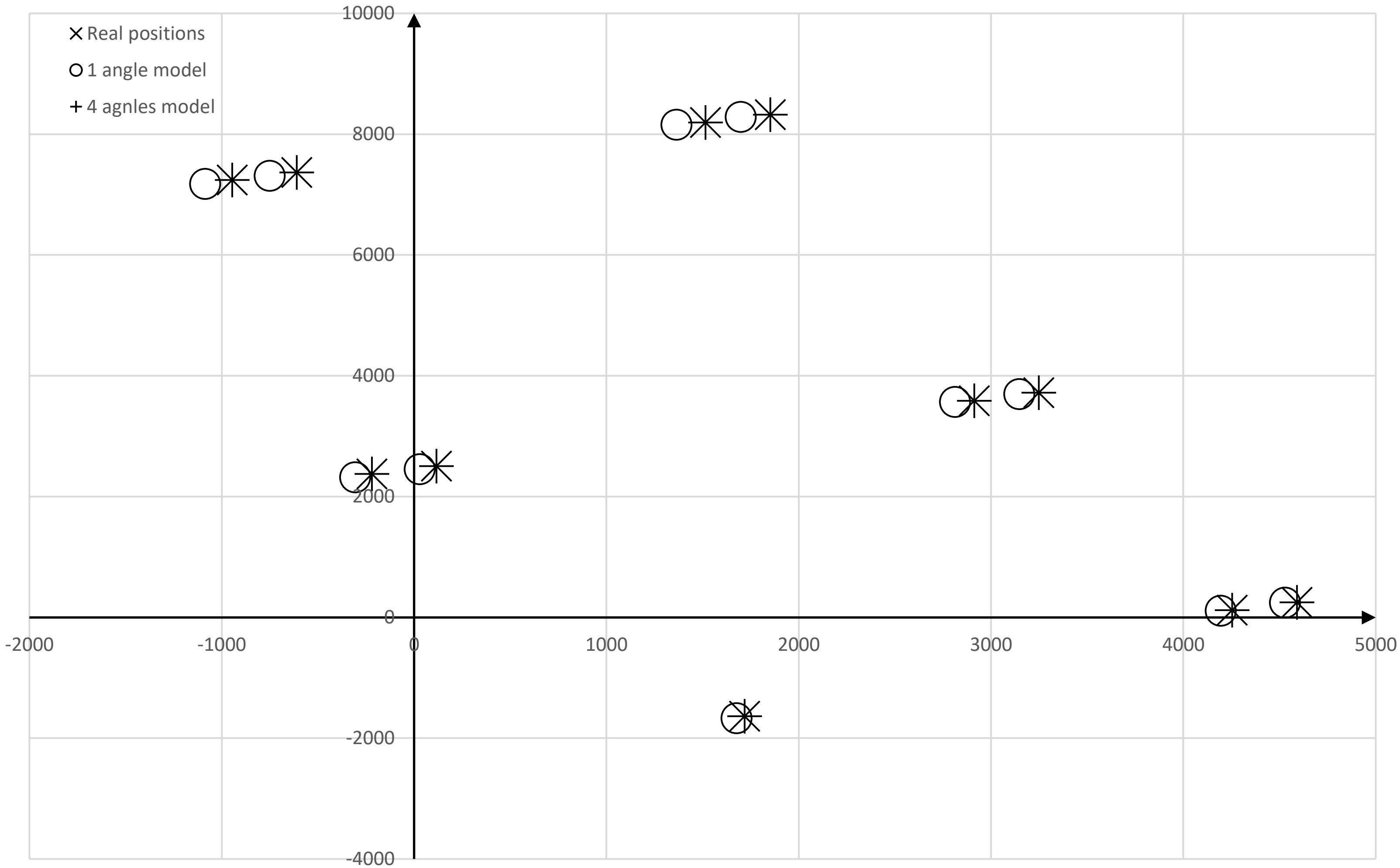
Statistical dispersion around point (-200, -10 000), σ=2

Experimental verification
AFM with automated stage



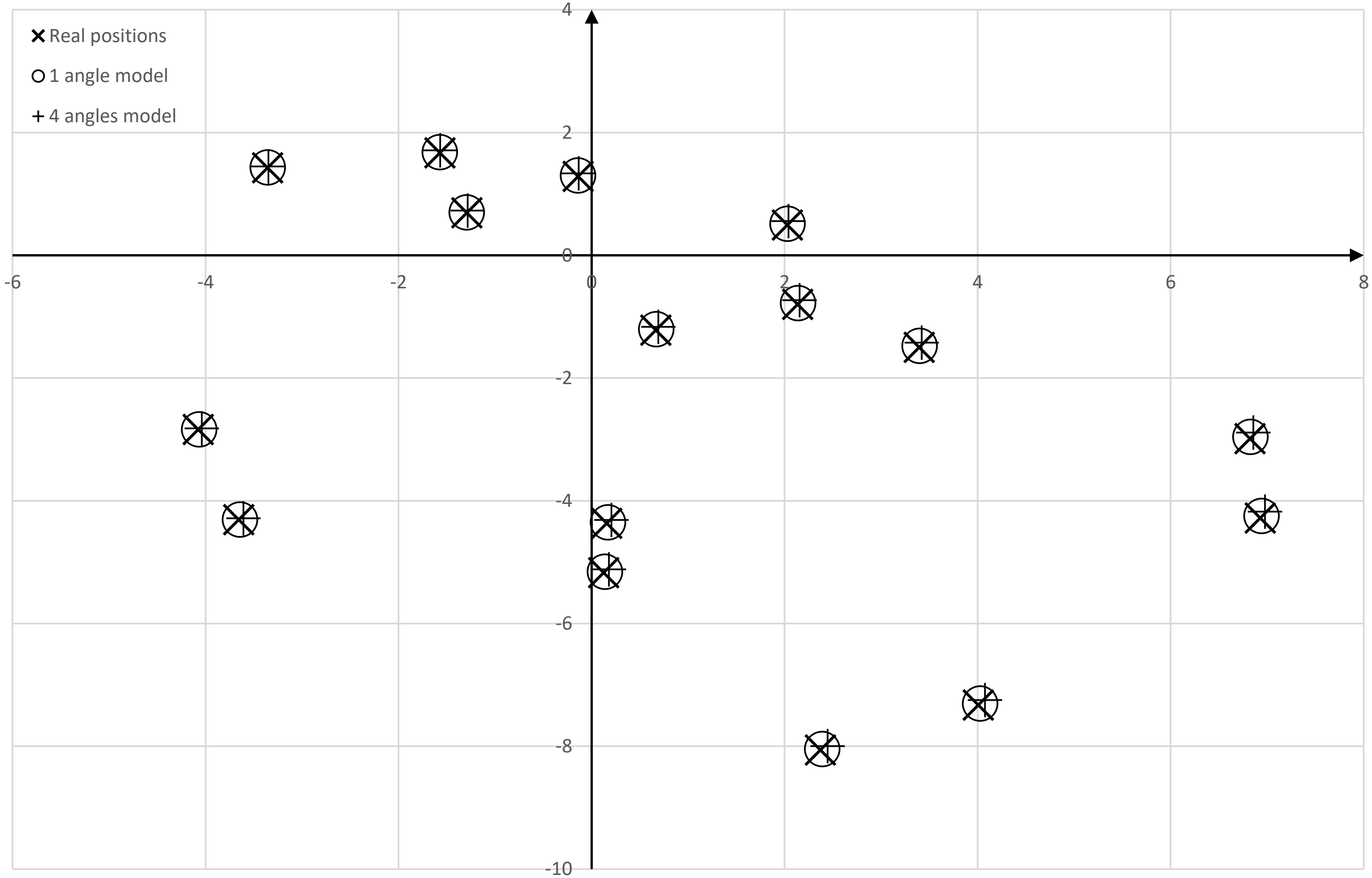
Position readout accuracy of was determined at level of 50 μm.

Experimental verification
optical microscope with automated stage



Position readout accuracy of was determined at level of 1 μm.

Experimental verification
SEM with automated stage



Position readout accuracy of was determined at level of 50 μm.

Summary

- recently performed research require complex, multi-method investigation of certain submicron features, therefore universal solutions enabling easy and repetitive sample positoning are desired

- some solutions providing submicron sample positioning were presented, and the application examples supported their usability

- correlative microscopy features enables precise samples positioning in diagnostic devices, providing reliable observation of surface properties changes, as the impact of the spatial non-homogeneity of the samples can be significantly reduced

- it was shown, that utilization of 2-points and 4-points reference feature in correlative microscopy sample holder is effective, while 4-points reference increases the positioning repeatability, in particular in case of the devices with low coordinates readout accuracy

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Thank you for your attention