SUMMER 2019

Newsletter

SURFACE

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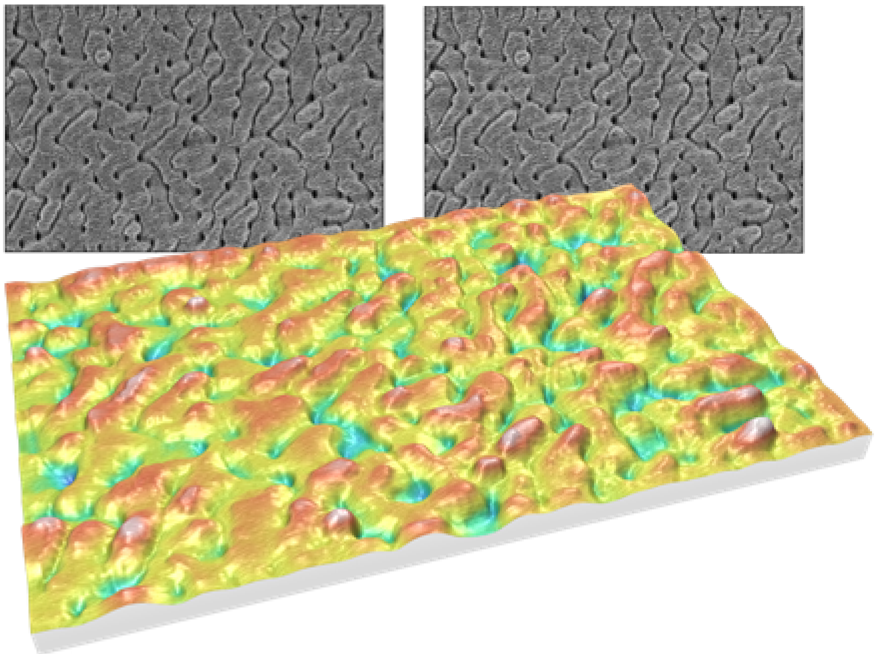
IN THIS ISSUE

3D MODELS OF SEM IMAGES REVEAL PROPERTIES OF LASER-ABLATED SURFACES



3D SEM stereo reconstruction technique used to achieve results where other methods fail

Ultrashort femtosecond lasers are employed in the production of complex devices for a variety of applications. In two recent studies, SEM stereo image reconstruction was crucial in highlighting laser-structured surface properties.

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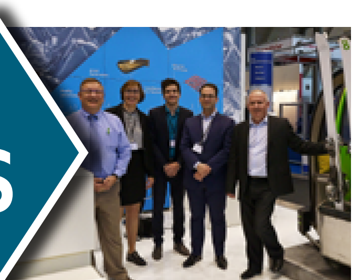
Meet us EVENTS

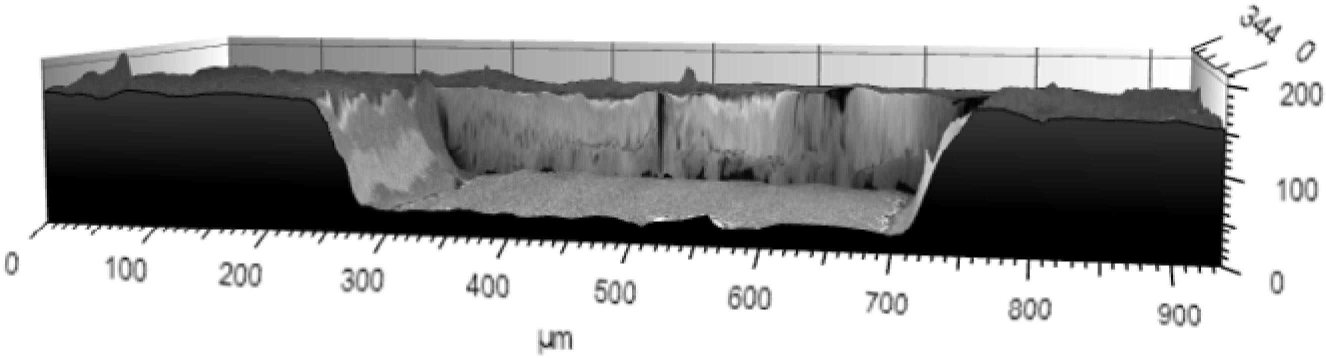


We look forward to seeing you at:

- **Microscopy & Microanalysis 2019 Meeting**
  Booth #1352 - August 5-8, 2019 - Portland, Oregon (USA)
- **Japan Analytical & Scientific Instruments Show (JASIS)**
  Booth #7B-704 - September 4-6, 2019 - Tokyo, Japan

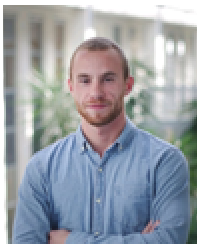
SEM IMAGE 3D RECONSTRUCTION OF LASER-STRUCTURED SURFACES

Ultrashort femtosecond lasers are known for their capacity to efficiently fabricate complex nanostructures and devices for a wide variety of applications: micro-optical components, gratings, biomedical engineering etc. In two recent studies, on both sides of the Atlantic, in which other characterization methods failed to yield valid results, the properties of femtosecond laser-structured surfaces were revealed thanks to a unique SEM image reconstruction technique.



Above. *Cavity machined with a femtosecond laser.* ***Below right.*** *3D reconstruction of cavity base with roughness parameters.*

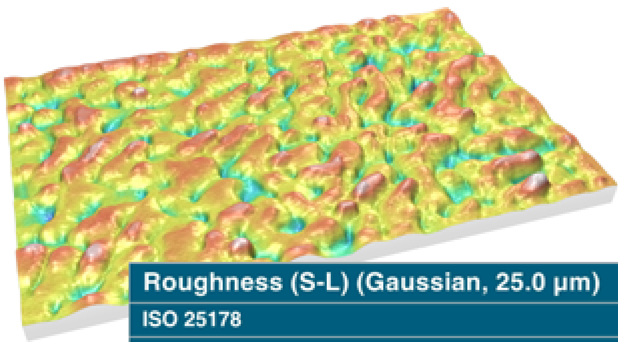
COMPARING ROUGHNESS OF SURFACES



Materials science student Mickaël Le Barh (left) recently completed a study on femtosecond laser micro-machining optimization for the Department of Mechanical Engineering at the University of Ottawa (Canada).

"Deep cavities (500 µm x 500 µm x 100 µm) were machined with a femtosecond laser (see image above) and our goal was to get the smoothest surfaces possible on the bottom and sides of the cavities. To achieve this, we adjusted machining parameters using visual quantification but found it was also useful to be able to perform roughness measurements."

"Initially I attempted to do this using an atomic force microscope (AFM) but the complex geometry of the sample prevented the microscope from accessing the bottom of the cavity. Using a Zeiss scanning electron microscope equipped with



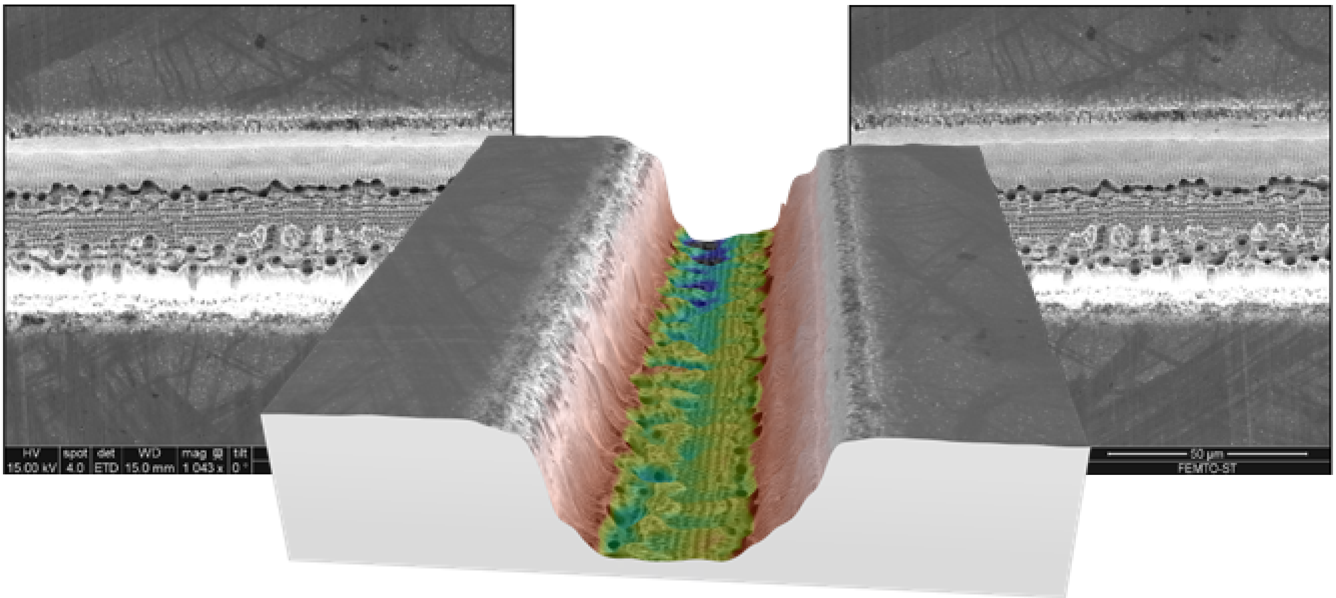
Roughness (S-L) (Gaussian, 25.0 µm)			
ISO 25178			
Height parameters			
Sq	0.373	µm	Root-mean-square height
Sa	0.295	µm	Arithmetic mean height
Sz	3.20	µm	Maximum height

Mountains® software, we were able to obtain the desired result."

"Obtaining smooth surfaces using ultrafast lasers is relevant in many applications where slower processes are currently used such as in sample preparation for electron microscopy, fabrication of microfluidic channels, 3D serial sectioning, micro-electro-mechanical systems and micro-optical elements."

READ MORE

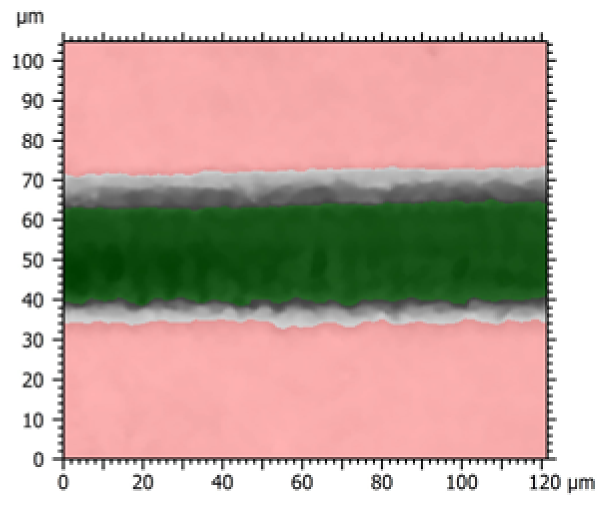
- ***Effect of polarization on ripple formation in deep femtosecond laser machined cavities****. Vincent Racine, Mickaël Le Barh, Graham Killaire, Arnaud Weck. In: Journal of Materials Processing Technology, volume 271, September 2019, pages 162-171. https://doi.org/10.1016/j.jmatprotec.2019.03.034*



Above. *3D model of a laser ablated micro-line reconstructed from two SEM images obtained at different tilt angles.*

SURFACE WETTABILITY IN BIOMEDICAL APPLICATIONS

In a similar application, Vahan Malkhasyan, PhD Researcher in Laser Material Processing at the FEMTO-ST Institute in Besançon, France has been studying femtosecond laser-matter interaction of metals and transparent materials for fabricating super-hydrophobic (water-repellent) and super-hydrophilic (super-wetting) surfaces for biomedical devices. Once again, 3D reconstruction of SEM images was paramount to getting reliable results.



Differential parameters	P2 - P1	Unit
Zmean(higher) - Zmean(lower)	25.1	µm

Above. *Automatic step height measurement performed on reconstructed SEM data.*

"In this study, it was important to measure the depth of crater impacts & lines ablated on different surfaces. The depths to be quantified were situated in the 5-60µm range" explains Vahan. "We compared topographies obtained by stereo reconstruction of SEM images and by optical and mechanical measurements."

"Using our 3D profilometer, it was difficult to obtain profile depth values of the impacts. In some cases, the values found were inaccurate. However using our FEI SEM to obtain two images of the sample which were then built into a 3D model using Mountains® software gave much more credible values (see image below left)."

Vahan's PhD on "Femtosecond laser structuration of surfaces and replication on polymers" was defended last April.

HOW TO PERFORM STEREO RECONSTRUCTION

Building a measurable 3D model of SEM data explained in video on our YouTube channel: https://youtu.be/V8yK65Jctyg

MEASURE ANY OBJECT IN A MICROSCOPE IMAGE

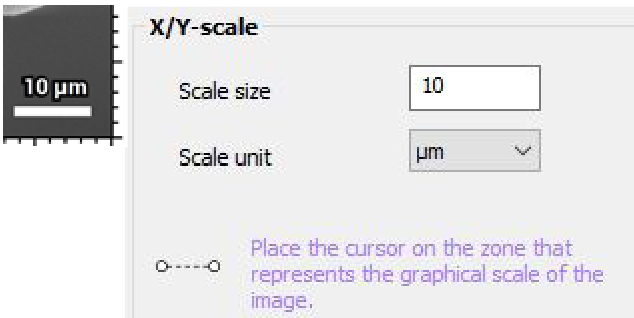
With Mountains® 8 comes an extended set of possibilities for accurately measuring any feature directly on image data from any instrument (SEM, SPM, profilometer etc.)
Sonja Braun, in charge of technical writing at Digital Surf, highlights the new options available.

Performing distance measurements on collected data can be useful for a wide range of applications, in particular when working with microscope images including those from scanning electron microscopy (SEM) or scanning probe microscopy (SPM).

In Mountains® 8 the "Manual measurements" study replaces the previous "Distance measurement" and now includes more measurement tools. It calculates many more parameters, similarly to the Particle analysis study, including Area and Perimeter parameters. Let's have a closer look with this scanning electron microscope image of pollen.

1. IMAGE SCALE

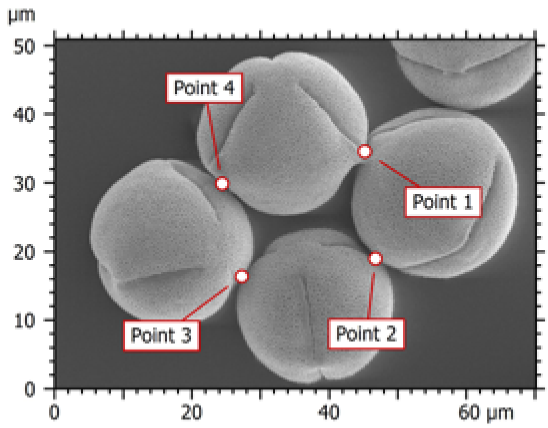
One crucial step to look out for is setting your image to scale before making any measurements. In the Operators tab, choose "Scale the image" and use the graphical scale bar (dimension block) or the known length of any feature in the image to set correct dimensions.



Set scale

2. COORDINATES OF A POINT

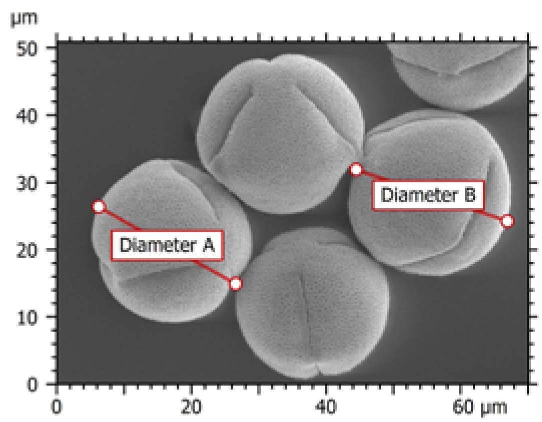
In the Studies tab, choose "Manual measurements". To show the coordinates of a point, simply click on the "Point" tool then on any place in the image. You can select and measure multiple points.



Points	Unit	Point 1	Point 2	Point 3	Point 4
X	µm	45.2	46.8	27.3	24.4
Y	µm	34.6	19.0	16.4	29.9

Coordinates of a point

3. DISTANCE MEASUREMENTS



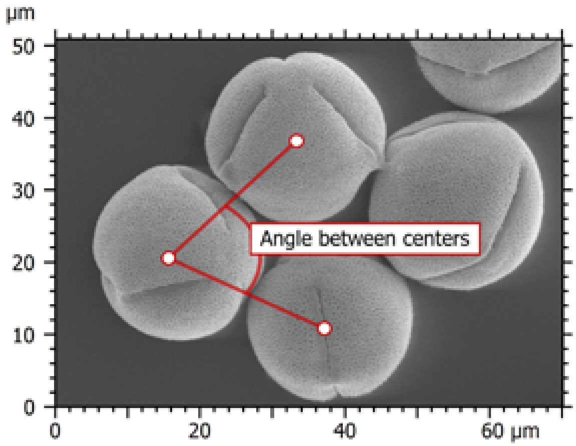
Distances	Unit	Diameter A	Diameter B
HDist	µm	23.3	23.8

Distance between two points

Click on "Distance" and then click on two points in the image to measure the distance between them. Again, you may measure several distances simultaneously. Each movement of one of the points immediately results in an update of the coordinates in the table below.

4. ANGLES

Click on "Angle" then draw the angle by clicking on three points on the image.

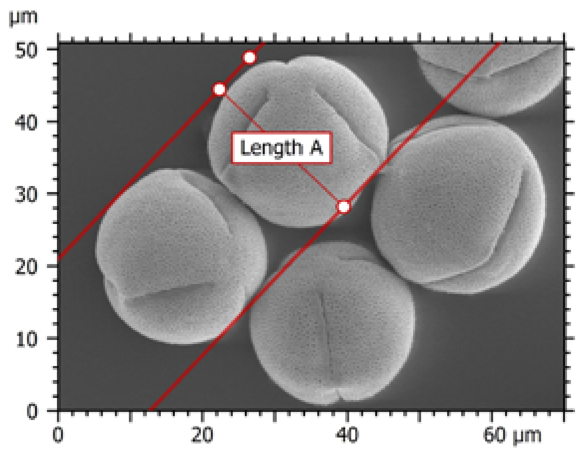


Angles	Angle between centers	Unit	
Angle	66.9	°	

Angle measurement

5. DISTANCE BETWEEN PARALLEL LINES

Click on "Parallel lines" then click twice in order to define the position of the two parallel lines. You can move and rotate the lines.



Parallel lines	Length A	Unit	
Distance	23.6	µm	

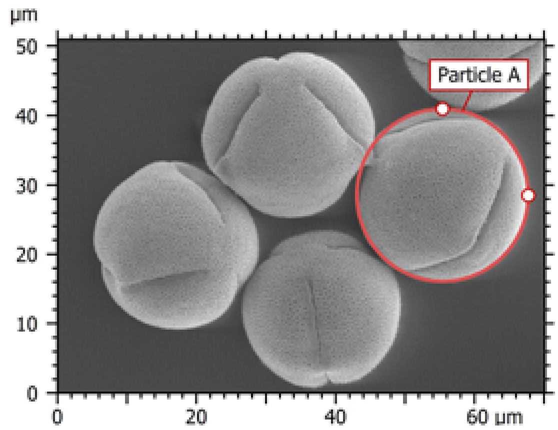
Distance between parallel lines

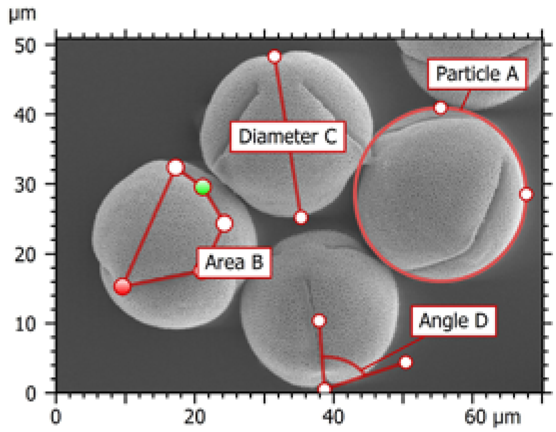
6. SHAPE MEASUREMENTS

Lastly, you can measure any shape you wish on your image data (rectangle, square, circle, ellipse, custom shape etc.).

Click "Shape" then click to define the start point of the shape, and click again to define the end point. Use the handles to change the size of the shape.

Tip: To draw a square, hold down Shift while using the Rectangle tool. To draw a circle, use the Ellipse tool, without using the handles.





Plane	Unit	Particle A	Area B	
Area	µm²	481	132	
Perimeter	µm	77.8	48.8	

Distances	Unit	Diameter C	
HDist	µm	23.5	

Angles	Unit	Angle D	
Angle	°	76.1	

Custom shape measurement (note that several measurements can be made on one image)

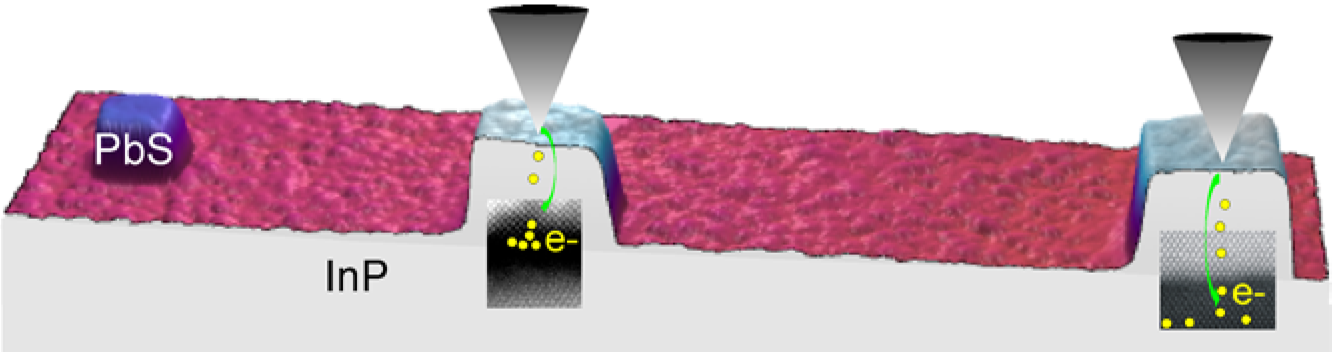
NB. The Particle Analysis feature in Mountains® also provides automatic measurement tools for this type of data.

Sounds good? Ready to try for yourself?
=> Get a free trial: digitalsurf.com/free-trial

ANALYZING PHOTOELECTRON SPECTROSCOPIC MEASUREMENTS

Indium phosphide (InP) is a binary semiconductor used for instance in the fabrication of high-performance devices such as photodetectors and solar cells.

In a recent study led by an international research group, scientists from the **Institut Supérieur de l'Électronique et du Numérique (ISEN)** in Lille, France, investigated a new method for growing PbS nanoplatelets on sulfur-treated InP (001) surfaces. Scanning tunneling spectroscopy (STS), an extension of scanning tunneling microscopy (STM), and photoelectron spectroscopy were used to analyze results. **Bruno Grandidier** of the ISEN, tells us more.



Above. *Scanning tunneling spectroscopy demonstrated the difference in chemical composition and structure with and without chemical passivation of the InP surface (AFM image processed with Mountains® software).*

GROWING PbS NANOPLATELETS ON InP

"These nanoplatelets are usually made by immersing an InP substrate in a solution containing lead and selenium precursors." explains Bruno Grandidier of the ISEN. "This is a low cost technique, easy to implement, but which has the disadvantage of generating interface defects between the nanocrystals and the semiconductor substrate. These defects greatly degrade the performance of optoelectronic components made from these heterostructures, such as photodetectors."

A NEW FABRICATION METHOD

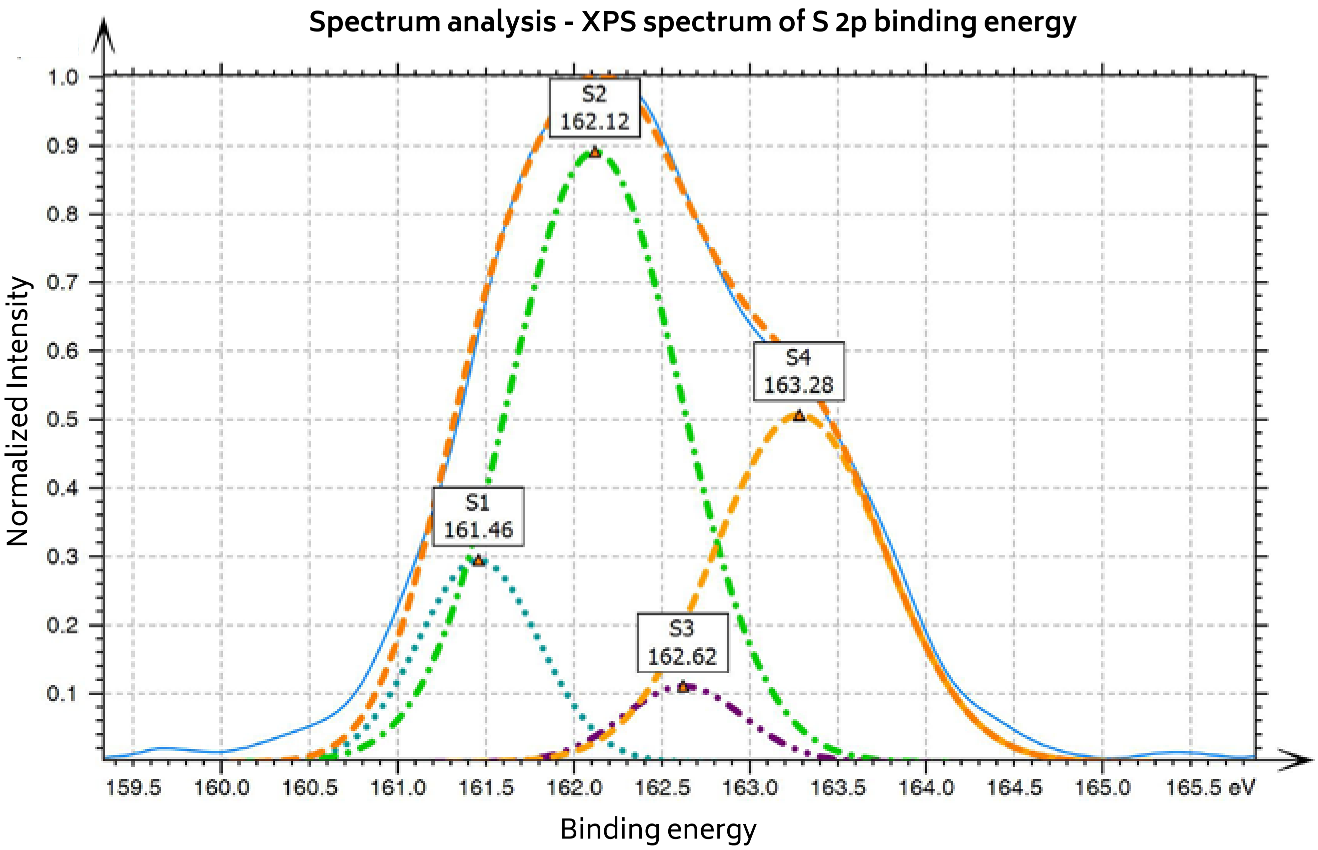
"In our study, we were able to show that by chemically passivating the surface prior to the nanocrystalline growth, it is possible to produce non-degraded interfaces. The results of this can be seen directly on measurements made with scanning tunneling spectroscopy. Chemical and structural analyzes of the interfaces also show that their quality is comparable to the heterostructures produced in ultrahigh vacuum by molecular beam epitaxy (MBE)."

ANALYZING PHOTOELECTRON SPECTROSCOPIC MEASUREMENTS

"Several complementary methods were used to characterize the InP substrates in this study.

In particular we used photoelectron spectroscopy to highlight the quality of the interfaces. By analyzing spectra produced we were able to confirm surface passivation thanks to the existence of two components in the S 2p core level, where the strongest component is caused by the In-S bonds and the smallest one being related to the Pb-S bonds in the nanoplatelets."

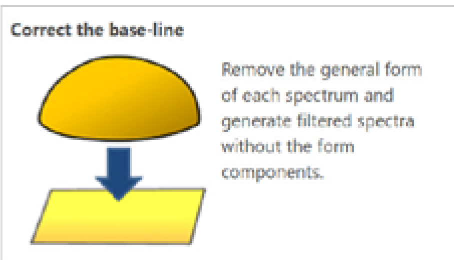
=> SEE ILLUSTRATION ON FOLLOWING PAGE

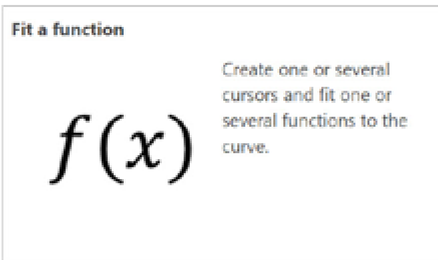


Cursor parameters	Axis name	Unit	S1	S2	S3	S4
Amplitude	Intensity	points	0.29	0.89	0.11	0.51
Energy	Energy	eV	161.46	162.12	162.62	163.28
FWHM	Energy	eV	0.80	1.14	0.80	1.14
Model			Gaussian	Gaussian	Gaussian	Gaussian

Above. *XPS spectrum measured on PbS nanoplatelets grown on passivated InP substrates. The surface passivation is supported by the existence of two components in the S 2p core level, where the strongest component (S2) is caused by the In–S bonds and the smallest one (S1) being related to the Pb–S bonds in the nanoplatelets.*

In the above example, the spectrum was analyzed using the following MountainsSPIP® software tools:





READ MORE

- ***Trap-Free Heterostructure of PbS Nanoplatelets on InP(001) by Chemical Epitaxy.*** *L. Biadala, W.Peng, Y. Lambert, J. H. Kim, D. Canneson, A. Houppe, M. Berthe, D. Troadec, D. Deresmes, G. Patriarche, T. Xu, X. Pi, X. Wallart, C. Delerue , M. Bayer, J. Xu and B. Grandidier. ACS Nano, 2019, 13 (2), pp 1961–1967. DOI: 10.1021/acsnano.8b08413#*

CONTACT

- *Bruno Grandidier - bruno.grandidier@isen.iemn.univ-lille1.fr*

WHAT'S NEXT FOR MOTIFS PARAMETERS ON PROFILES?

R&W motifs (ISO 12085) have been widely used for over 40 years, mainly in France. Now part of an improved method using watershed segmentation, they are soon to be included in the revision of profile standards (ISO 21290). Following his paper on the subject at the recent Met & Props 2019 conference*, **François Blateyron, Digital Surf's surface metrology expert** explains.

R&W MOTIFS METHOD REBORN

The watershed segmentation method, applied to surfaces, is becoming increasingly successful thanks to its ability to automatically identify structures and texture cells on surfaces, called "motifs". The resulting motifs can be considered as "significant" due to a discrimination method, called "Wolf-pruning" which reduces over-segmentation and merges non-significant motifs with larger adjacent ones, by using a threshold based on a percentage of the total height Sz.

The threshold is compared to the height between the highest saddle point and the peak in the case of a hill (or between the pit and the lowest saddle point in the case of a dale). Feature parameters described in ISO 25178-2 allow the characterization of these significant motifs, and they can be used in the tolerancing of many modern surfaces that contain periodical or identified structures.

PROFILE SEGMENTATION

Watershed segmentation can also be applied on profiles, in a simplified form, to identify profile motifs. It is thus being considered as a candidate for replacing the old R&W motifs method (ISO 12085:1996) that was built on a complex algorithm with many special cases. The new method is expected to stabilize motif detection and provide compatible parameters. The profile watershed segmentation is described in ISO/WD 16610-45 and the associated parameters are included ISO/CD 21920-2, both in development.

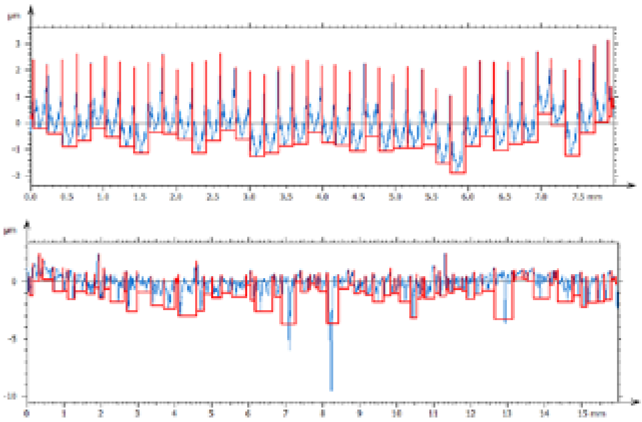
It is important to ensure continuity and compatibility with the former method so that users can update their drawings and tolerances to the new method without too many changes. Therefore, it is important to compare parameter values given by these two methods and define adequate default values.

* *Paper co-authored with Bertrand Leroy, Peugeot-Citroën SA*

DOMAIN OF APPLICATION

The method identifies motifs made of a succession of peak-pit-peak triplets. Motifs identify tool marks on a machined surface and make it possible to identify height and width of this machining signature, in order to verify process settings and compliance. It is assumed that the measured profile exhibits motifs-like features. Usually, machining methods that leave a periodical or semi-periodical texture signature, such as turning or grinding, can easily be verified using the motifs method.

The main parameters of ISO 12085 are R (mean height of motifs) and AR (mean width of motifs). When these parameters are calculated on a profile with clearly identified motifs, the mean value is significant and associated with a small standard deviation. Interestingly, the original CNOMO method defined SR and SAR parameters as the standard deviation for R and AR, but the publication in an ISO standard saw the loss of these important parameters.

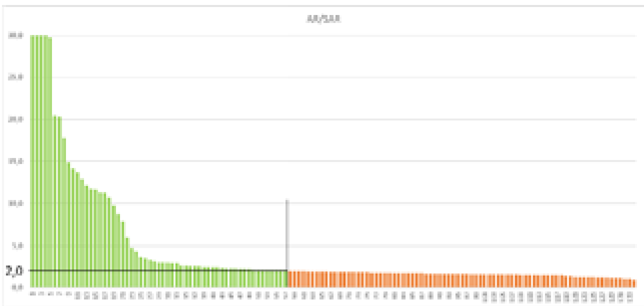


Top. *Motifs on a profile with periodical marks. Mean height R and mean width AR are significant.*
Bottom. *Motifs on a irregular profile. R and AR are just a statistical mean of small and large motifs averaged together.*

What do we get if we calculate these parameters on irregular or stochastic profiles? Well, we can always calculate a mean value, but we average narrow and wide-motif widths, short and tall-motif heights etc. Moreover, motifs are unstable when reversing or shifting the profile. Some say that the method is inherently not stable, but the truth is that it should be used only on profiles that exhibit a minimum of texture motifs, i.e. periodical or semi-periodical profiles. And unfortunately, the practice is not in line with that principle; many users continue to calculate R and AR on irregular profiles, instead of using classic Rq, Rsk parameters etc. which are better suited to irregular profiles. This observation also applies to RSm and Rc from ISO 4287 that are associated with a segmentation method too.

DEFAULT VALUES

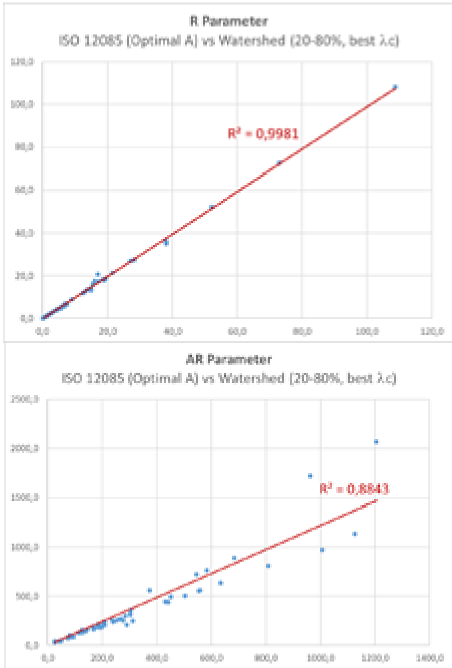
ISO 12085 uses a limit A to separate roughness from waviness motifs. But it also controls the segmentation itself. Its default value of 0,5mm for a profile length of 16mm is usually used as-is without any question. But the reality shows that a better value can often be found if we consider that the motif segmentation should detect regular motifs with the smallest standard deviation. The simple ratio AR/SAR can be calculated, and if a profile is periodical, the standard deviation will be small compared to the mean and the ratio will be high. On the contrary, the ratio will be small on irregular profiles. This can be used to test several values for the limit A in order to define the "optimal limit A", i.e. the one with the highest value for the ratio AR/SAR.



Above. *The graph of AR/SAR values is split in two categories with a threshold = 2,0 which means that the standard deviation is not higher than 50% of the mean value. The 57 green profiles on the left can be considered as suitable for the motifs method. A finer selection can be obtained with a combination of R/SR and AR/SAR ratios.*

COMPARISONS

The comparison of values on R and AR between the old method (with optimal limit A) and the watershed segmentation, with an adequate pruning shows very good correlation which means that the new method is a safe replacement, when used with the correct settings.



Above. *Comparison of the ISO 12085 (optimal A) and watershed methods shows good correlation.*

FURTHER WORK

More investigations must be done in order to adjust the method and answer any remaining questions. For example, the upper envelope used to calculate waviness parameters in ISO 12085 can be replaced by a morphological closing filter using a disc as a structuring element. But with what radius?

Another unsolved question relates to the removal of deep valleys or high peaks, which was done in the old method using a threshold on motifs height when exceeding 1,65σ of the height distribution. With the watershed segmentation, a simpler statistical method could be used. This work will then help to define adequate default values in the new ISO standards and define good practice.

Segmentation of motifs on profiles is still a useful method. Watershed segmentation, together with a pruning and adequate configuration make it possible to solve the stability problems of ISO 12085 but keep the benefit of 40 years of experience in qualifying mechanical components.

RESOURCES

- *ISO 12085:1996 GPS – Profile method – Motifs parameters*
- *ISO/WD 16610-45 – Filtration – Profile morphological: Segmentation*
- *ISO/CD 21920-2 – Surface texture: Profile – Terms, definitions and surface texture parameters*
- *Blateyron F, Adam M (2004), Application of image segmentation to motifs evaluation on 2D profiles, Proc. XI Colloq. Surfaces, Chemnitz, 56-64.*

EVENTS HIGHLIGHTS

REVIEW OF ANOTHER SUCCESSFUL TRADESHOW SEASON

CONTROL 2019

How exciting it was for Digital Surf to present its brand-new booth at the **Control trade fair** in Stuttgart, Germany on May 7-10! With over 870 exhibitors from 33 countries and more than 27,000 visitors to this new edition, Control is possibly the biggest, most impressive show on quality control anywhere in the world.

On a totally redesigned booth with the goal of presenting the release of version 8 software, visitors were welcomed by the Digital Surf team to the world of Mountains®, complete with a ski-lift transformed into a meeting room especially for the occasion.

Mountains® software solutions were also visible throughout the six exhibition halls on our partners' stands. Fully integrated with a number of surface measuring instruments, the benefits in terms of accuracy and productivity were clear for all to see.

Above. *The Control 2019 show was a highlight on the Digital Surf events calendar*

E-MRS SPRING

Digital Surf also attended the **Spring Meeting of the European Materials Research Society (E-MRS)** that was held in Nice, France from May 28-30. Christine and Nicolas were on hand to meet the European materials science community and discuss their data analysis applications, including particle analysis and correlative analysis.

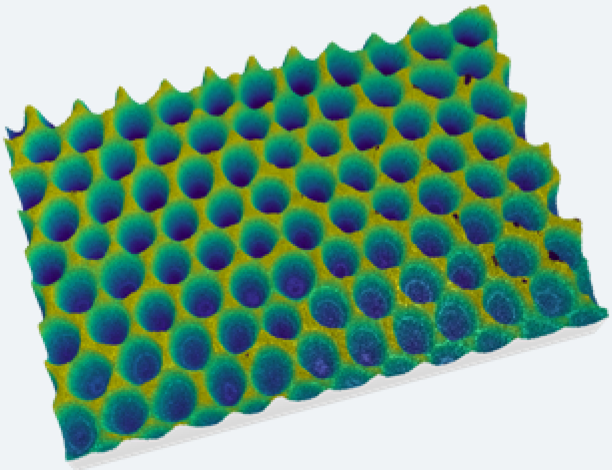
MET & PROPS 2019

The **22nd International Conference on Metrology and Properties of Surfaces** was held in Lyon, France from June 3-5. Anne and François B. were pleased to represent Digital Surf who has been a partner of this event for years. They were pleased to meet with over 80 international surface metrology professionals to discuss the Mountains® latest analysis methods and filtration techniques.

Above. *Digital Surf COO François Blateyron with keynote speaker Dr Gert Wolf at Met & Props 2019.*

Laure Bepoix

WHAT'S HOT ONLINE

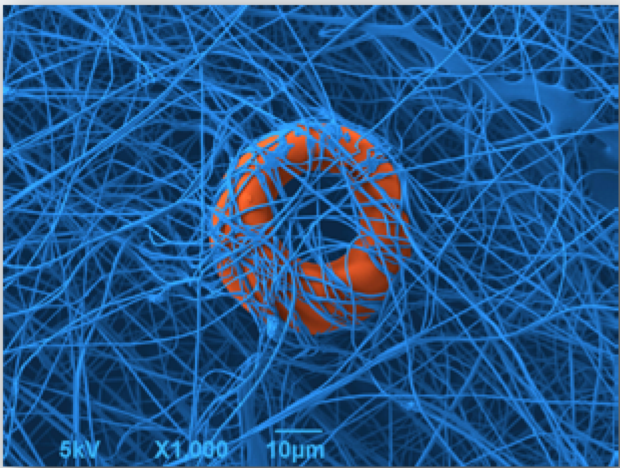


POPULAR ON FACEBOOK

July 12, 2019: Digital Surf organized a contest for the attendees of the **1D2D Nanomat international summer school** who had been learning how to use MountainsSPIP® 8 software to process their microscopy image data.

Hundreds of visitors cast their vote for their favorite images and three lucky winners won their sample printed in 3D (winning image above by Nathali Franchina).

bit.ly/2XKOK13



SEEN ON LINKEDIN

July 3, 2019: JEOL USA published this amazing image, the winner of the company's monthly image contest.

Submitted by **Simone Lauciello of the Fondazione Istituto Italiano di Tecnologia**, it shows a blood platelet trapped among silk fibers. Taken on a JEOL JSM-6490LV SEM, it was colorized using Mountains® software.

bit.ly/2GaihX9

Have you visited our YouTube channel recently?

We just posted six new Mountains® 8 features tutorials to help you get started using the new version.

Check it out!

https://youtu.be/UXZ0EnK8hxc

Surface Newsletter

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MEET DIGITAL SURF

- **Microscopy & Microanalysis 2019 Meeting** - booth #1352
  August 5-8, 2019 - Portland, Oregon, USA
- **Japan Analytical & Scientific Instruments Show (JASIS)** - booth #7B-704
  September 4-6, 2019 - Tokyo, Japan

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Surface Newsletter, July 2019

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