NEWSLETTER
Summer 2017

SURFACE

Surface imaging, analysis & metrology news from Digital Surf

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PLUS

Assessing the role of formulation spreading in sunscreen efficacy

This summer, we take a look behind the scenes at the L'Oréal Advanced Research Division where researchers are working on the constant improvement of sunscreen products

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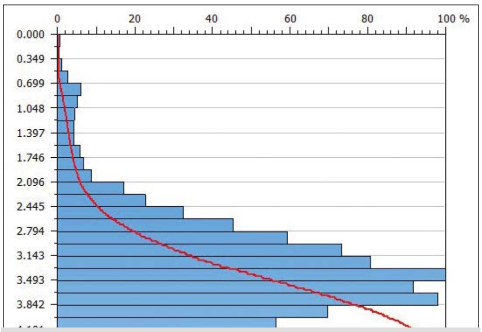

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We look forward to seeing you at:

M&M 2017 MICROSCOPY & MICROANALYSIS August 6-10, 2017 • St. Louis, MO

St Louis, MO, USA
August 6-10, 2017

BOOTH #517

JASIS 2017

Tokyo, Japan
Sept. 6-8, 2017

BOOTH #4A-401

ASSESSING THE ROLE OF FORMULATION SPREADING IN SUNSCREEN EFFICACY

L'ORÉAL Research & Innovation

Beauty giant L'Oréal has gained a reputation over the years for attaching great importance to research and innovation. The Advanced Research Division, located just outside Paris (France), is at the heart of that particular component of the company's mission. One of the many subjects studied by the division's team of specialized researchers is the constant improvement of sunscreens. Florian Formanek, head of the Microscopy & Microanalysis Laboratory, tells us more.

Photoprotection – a key cosmetic segment

SPF, or Sun Protection Factor, is a measure of the efficacy of sunscreens to protect skin against harmful UV radiation. The approved method for SPF determination is through the *in vivo* monitoring of erythema (reddening) caused on human skin by solar-simulated sunlight.

However, alternative *in vitro* measurements exist and are performed by diffuse UV transmission spectroscopy through a sunscreen-covered substrate, such as rough PMMA (polymethyl methacrylate) plates which mimic skin texture and surface free energy.

Cosmetic formulations usually contain several chemical (organic) and physical (inorganic) filters that either reflect, scatter or absorb UVA and UVB radiation to offer an efficient broad-spectrum protection. Other ingredients include water, glycerin (moisturizer), emollients (for smoothing properties), emulsifiers (to mix water with oil), thickeners (to confer viscosity), film formers (water resistancy) and sensory enhancers.

Besides obvious parameters such as film thickness and homogeneity, sunscreen performance can also be affected by the localization of the different filters within the bulk. Depending on the physicochemical properties of the substrate surface, processes like coalescence, dewetting or phase migration occur upon drying and evaporation of volatile compounds, thus impacting the distribution of filters as well as aqueous/oily phases.

We employ Raman imaging in combination with other microscopy techniques to better understand the distribution of UV filters in various formulation bases and compare with *in vitro* SPF assessment.

Experiments and data processing

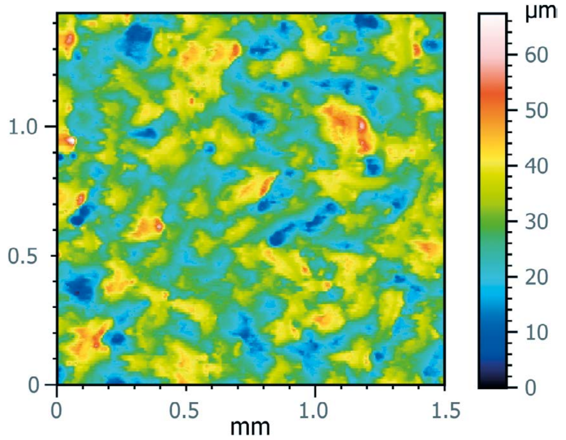
A Renishaw inVia™ Qontor™ Raman microscope equipped with a 532 nm laser is used to acquire 3D or 4D hyperspectral maps containing - at each pixel - a Raman spectrum in the fingerprint region (400-2000 cm^{-1}). A recently introduced real-time focus tracking unit simultaneously provides the topography of the sample surface with micrometric axial resolution.

We use MountainsMap® software firstly to remove outliers (non-measured points obtained during auto-focus), then to compensate the general slope of the sample with the leveling or form removal functions. The next step is to perform morphological filtering, before employing advanced analysis methods.

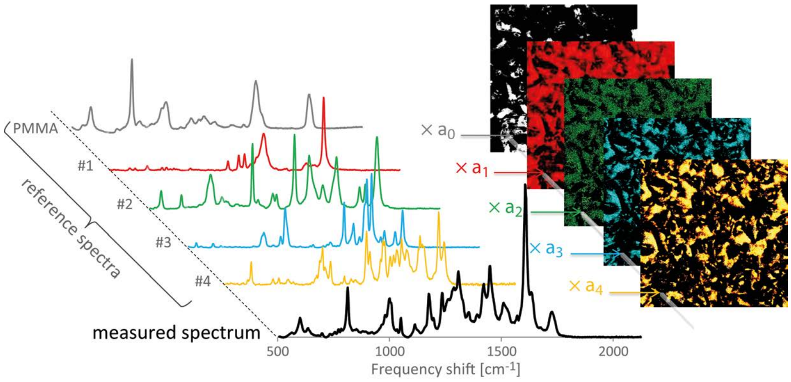
The colocalization module allows us to overlay the relief map with 2D chemical images obtained after chemometric

Photoprotection is the biochemical process that helps organisms cope with molecular damage caused by sunlight.

processing of spectroscopy information. Part of this processing involves the deconvolution of measured spectrum using reference spectra of each individual ingredient, to extract their relative concentration at the laser focus spot.



Above: 3D height profile of a bare PMMA plate with theoretical Sq roughness of 6.6 µm, measured with MountainsMap® at 6.18 µm.



Above: principle of the spectral decomposition processing to retrieve the relative contribution of the different compounds in the formulation.

Left: Reconstructed view of a sunscreen-covered PMMA plate, combining topographic and chemical data.

- 10% height amplification was applied to enhance 3D rendering.
- Gray areas show uncovered PMMA regions, red and green areas reveal the distribution of two different UV filters.
- Calculated Sz parameters (67.4 µm → 50.3 µm) indicate smoothing of the surface through filling of the valleys by the cream.

Other usages of MountainsMap® for our applications include extracting quantitative surface textures from 3D samples (hair, skin, nail, materials) measured by optical profilometry, optical coherence tomography (OCT) or fringe projection, producing stereo views from tilted scanning electron microscopy (SEM) images as well as extracting and processing intensity profiles from energy-dispersive X-ray (EDX) spectroscopic data generated by SEM and TEM.

About the author

Florian Formanek is head of the **Microscopy & Microanalysis Laboratory** within the **L'Oréal Advanced Research Division**, located in the suburbs of Paris, France. His past research in different areas of microscopy has included work on near-field optics, nanophotonics and terahertz imaging.

Read More

Importance of sunscreen products spreading protocol and substrate roughness for *in vitro* sun protection factor assessment, L. Fageon, D. Moyal, J. Coutet and D. Candau. International Journal of Cosmetic Science, 2009, 31, 405–417. dx.doi.org/10.1111/j.1468-2494.2009.00524.x

Repartition of oil miscible and water soluble UV filters in an applied sunscreen film determined by confocal Raman microspectroscopy, M. Sohn, T. Buehler and G. Imanidis. Photochemical & Photobiological Sciences, 2016, 15, 861-71. dx.doi.org/10.1039/c6pp00024j

REFLECTOMETRY OR HOW TO GIVE YOUR SEM IMAGES A 3D UPGRADE

A whole new world of 3D awaits users of scanning electron microscopy (SEM) coupled with Mountains® software.

Christophe Mignot, Digital Surf CEO and specialist in SEM image analysis, runs through one of the coolest techniques available for bringing to life even the tiniest details contained in images.

The Scanning Electron Microscope (SEM) is widely used in various fields of industry and science since it is one of the most versatile imaging and measurement instruments. SEMs allow users to see details 1,000 times smaller than those obtained using conventional microscopy. On the downside, images provided by SEMs are only two-dimensional. Over the last few years, thanks to on-going research carried out by the Mountains® development team, several possibilities have been made available to SEM users for switching from standard 2D images to "topographic" images.

Shape from motion vs shape from shading

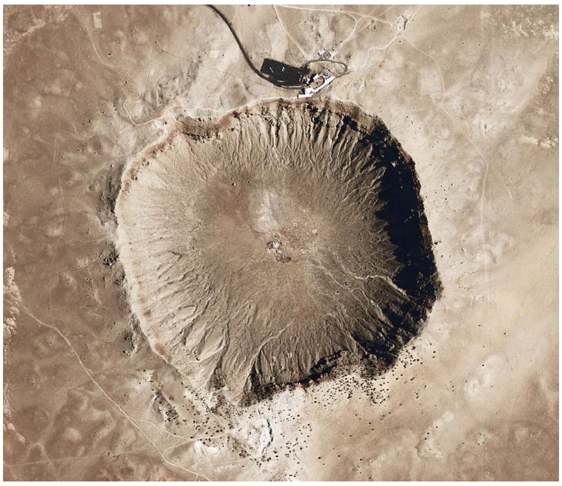
The most metrologically accurate approach to 3D reconstruction of surface features is **stereophotogrammetry**. This technique requires two SEM images of the same object taken from two different angles. Height information is calculated trigonometrically. Subject to good experimental conditions, the method gives accurate values. However, its drawback is obviously that it requires tilting the specimen and taking two successive images of the same area, which may not always be easy, e.g. for a wafer.

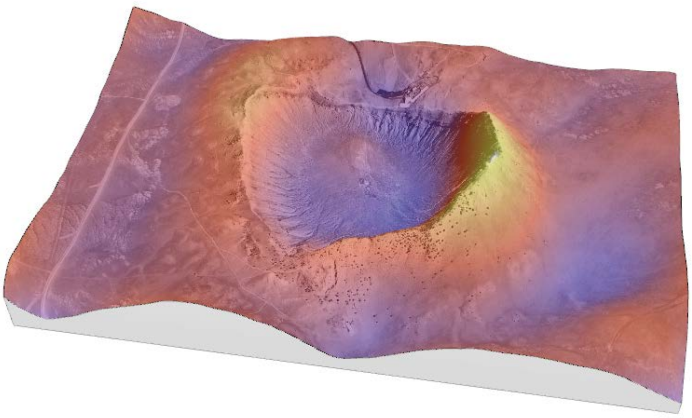
The most common alternative to stereophotogrammetry ("shape from motion") is **reflectometry** ("shape from shading"). The principle uses the intensity of electron reflection on the surface to assess local slope, then compiles local slopes into a complete topography map.

How does shape from shading work?

In the picture below (left) taken from space of Meteor Crater, Arizona (en.wikipedia.org/wiki/Meteor_Crater), we can see that the left part of the crater rim appears light, almost white, whereas the right side is dark. This is obviously because the sunlight is coming from the right. One slope is lighter in color because it is inclined towards the sun, the other is darker because it is inclined away from the sun. This gives us information on slope. Moving in a path from the left of the picture to the right, a light pixel will mean "crater height decreases" and a dark pixel "height increases".

This aerial view, with the sunlight coming in from the side, is equivalent, in terms of illumination, to the electron gun of an SEM operating from above with the electron detector placed to one side of the specimen.





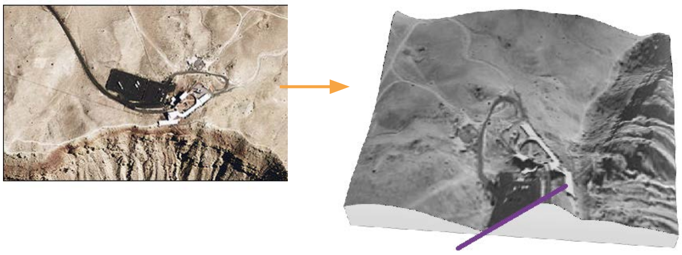
Left. Meteor Crater, Arizona seen from space.

Above. 3D reconstruction from this single image obtained using Mountains® software.

Now, if we compile data from across the entire image, we can build a 3D representation of the meteor crater.

This representation is not perfect however :

1. Rim height is overestimated in one direction (left-right). This is because we can differentiate increase or decrease in slope in relation to the direction light is coming from, but not perpendicular to this. If light is coming from the east, west-east slopes are detected, but north-south slopes are not. This highlights the need to work successively in two orthogonal directions, to make height assessment in both directions possible.
2. At the top of the picture, we can see the visitor center (white) and the road and car park (black). In the 3D reconstruction, these are obviously misinterpreted as slopes (see the slope of the car park in the image below):



So what's the solution?

Using **a four-quadrant detector** will help clear away both of those two issues.

A four-quadrant BSE detector is a ring detector that surrounds the electron beam path. The ring is divided into four quarters. 3D reconstruction in this case, contrary to the stereo method which requires two successive images taken at different tilt angles, can be obtained with only one scan (the four images are acquired simultaneously).

Detectors work in pairs

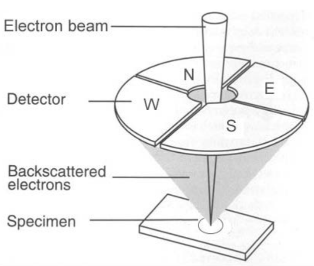
To assess slope in one direction (e.g. west-east), two opposite detectors are used. This allows us to overcome the albedo (car park) issue. If the object is locally darker or brighter, both detectors see the same. It is the differential signal between the two detectors that allows us to distinguish a darker area from a slope and therefore correctly calculate slope.

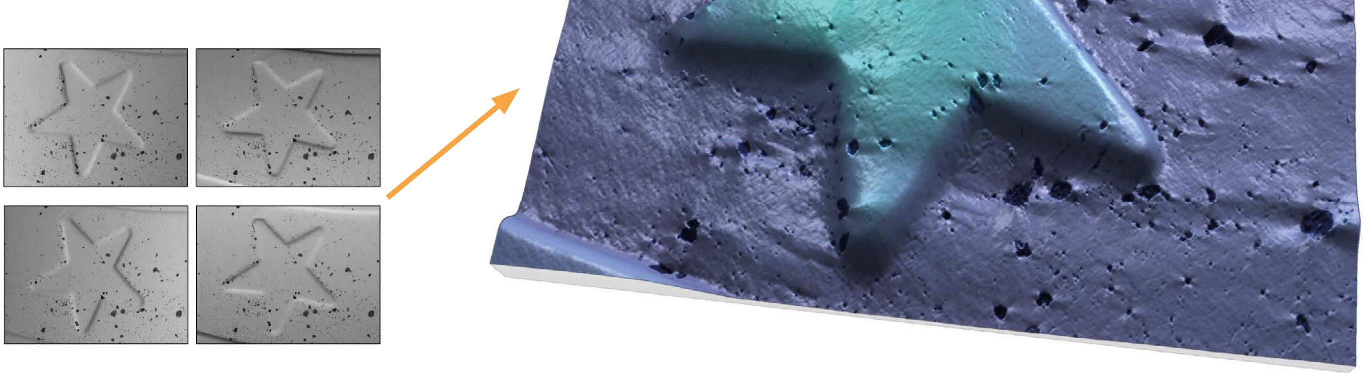
There are two couples of opposite detectors : one specializing in east-west slopes and the other in south-north slopes. Nano-meteor crater topography rebuilt from four four-quadrant images would exhibit homogeneous rim height (not just a high rim on the left and right).

Once slopes in both directions have been correctly detected, they can be integrated into the surface topography and will yield outstanding 3D images. This is something MountainsMap® is very good at doing. And very quick too (calculations achieved in less than a second).

In the image below, a 3D view has been generated using Mountains® from four BSE images obtained simultaneously. We can see that the software is able to reconstruct heights homogeneously regardless of direction. It is also capable of distinguishing different kinds of material inclusions: dark regions and actual holes or even holes with inclusions inside them.

Nowadays most SEMs are delivered with a pre-installed multiple-angle BSE detector. Generally, this is a four-quadrant detector (detectors at 90°). In the case of FEI, it is a three-segment detector (120°). Whatever kind of multiple image detector your SEM possesses, Mountains® has a solution for 3D reconstruction. And seeing the software is now provided by most SEM manufacturers with their instruments, as standard or as an option, why not upgrade your images to 3D now?



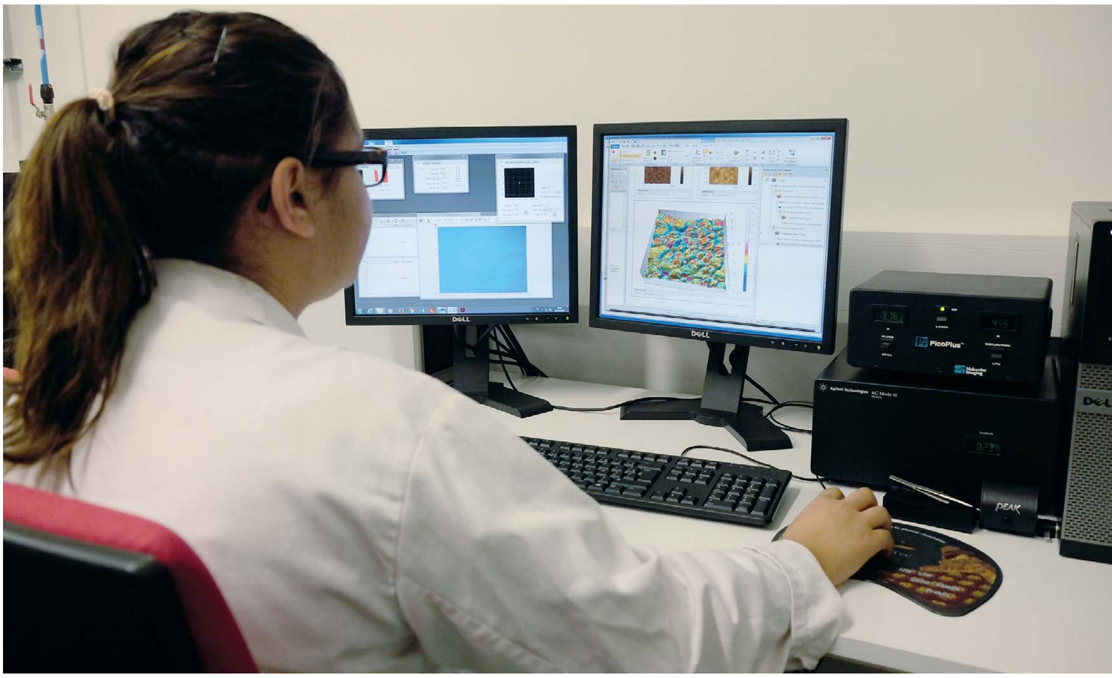


Above left. Diagram representing four-quadrant BSE detector. **Below left.** A detail of a 1 euro coin scanned using a Hitachi scanning electron microscope equipped with a four-quadrant detector, resulting in four images of the same area. **Right.** 3D reconstruction using Mountains® software.

TOWARD IMPROVED INTEGRATION OF BIOCERAMIC IMPLANTS

Nestled in the heart of west-central France, the city of Limoges is famous for its high quality porcelain factories which employed up to 10,000 workers at the height of their golden age during the second half of the 19th century.

Fast forward to the present day, and a group of researchers at the University of Limoges are investigating other, more modern and innovative kinds of ceramic material, known as bioceramics.



The group is part of the "Science of ceramic processing and surface treatments" laboratory* which studies materials and applications relative to the energy, information technology and health sectors.

"Bioceramics are an important subset of biomaterials. They are particularly useful in the repair and reconstruction of bone" explains Dr Chantal Damia, member of the research group.

“Our team’s main focus is the development of devices based on calcium phosphate bioceramics for applications in bone tissue engineering. The composition of these materials is very close to the mineral part of bones and they thus exhibit osteoconductive properties (i.e. they allow bone ingrowth into porous implants).

The development of advanced bioceramics for bone remodeling requires better knowledge of their interactions with the biological environment (i.e. the bone cells and proteins involved in cell adhesion). We have recently been working on setting up characterization tools to monitor these interactions.

This research is particularly useful for improving the efficiency of medical devices such as bone substitutes. It also opens up possibilities for extending their use to large bone defects (>20 cm^2) for which natural bone regeneration only occurs at the peripheral region between the bone and the substitute.

AFM force mapping of protein on calcium phosphate bioceramics

One current focus is the impact of bioceramic surface topography and composition on protein adhesion forces. We investigated this using a technique known as **atomic force spectroscopy** coupled with **PicoImage data analysis software** (based on Mountains® technology).

We chose to concentrate on one protein in particular, fibronectin (Fn), which promotes cell adhesion allowing the differentiation and survival of osteoblasts (cells involved in the process of bone formation).

To measure the adhesive force between fibronectin and bioceramic material, the protein was grafted onto the cantilever tip of our Keysight atomic force microscope (AFM). This tip was put into contact with a calcium phosphate bioceramic substrate made from silicated hydroxyapatite powder synthesized by an acqueous precipitation method.

Once contact between the functionalized tip and the substrate was obtained, the cantilever was removed and the adhesive force of fibronectin measured. Force mapping was performed with spectroscopy mode in air. Results of the study are soon to be published.”

* Research unit operated by the University of Limoges in association with the French National Center for Scientific Research (UMR CNRS 7315).

Université de Limoges

cnrs dépasser les frontières

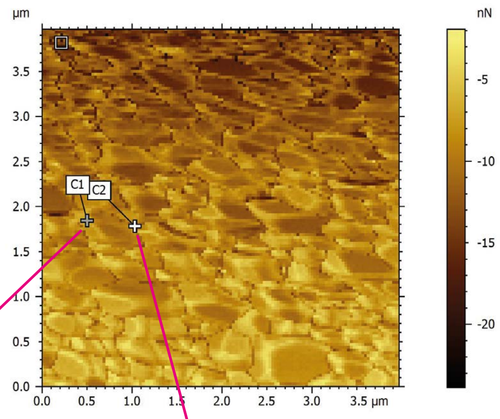
Force curve analysis tools used in this study

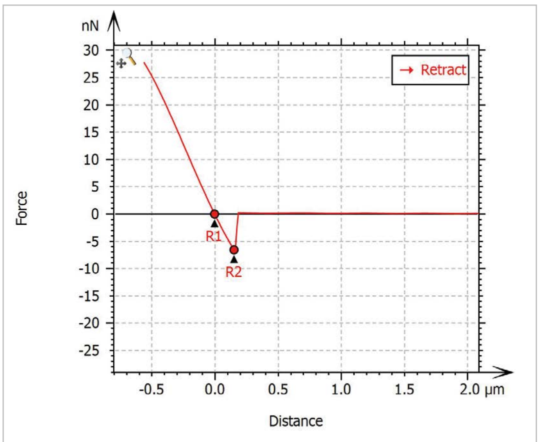
1 Interactive parameter maps

The force value is calculated for each point of the surface at the adhesion event cursor (a histogram of values can be displayed.) Force values are displayed in a parameter map.

Two cursors were created on the parameter map (right) to display and compare individual force curves in neighboring zones.

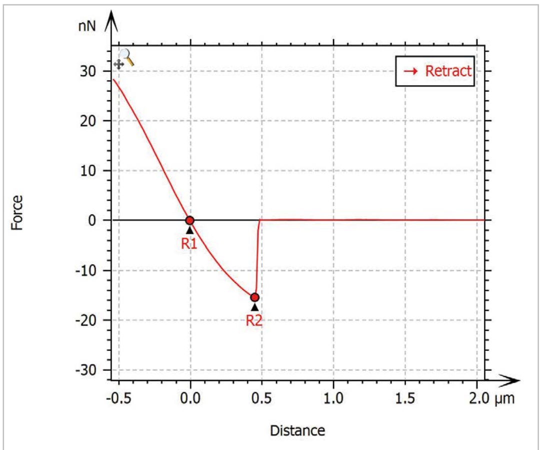
This feature is interactive: the displayed force curve is updated when the cursor is moved.





Force curve parameters	Value	Unit
Spring constant	0.0485	N/m

Parameters for individual cursors	Unit	R2
Distance	µm	0.150
Force	nN	-6.56



Force curve parameters	Value	Unit
Spring constant	0.0485	N/m

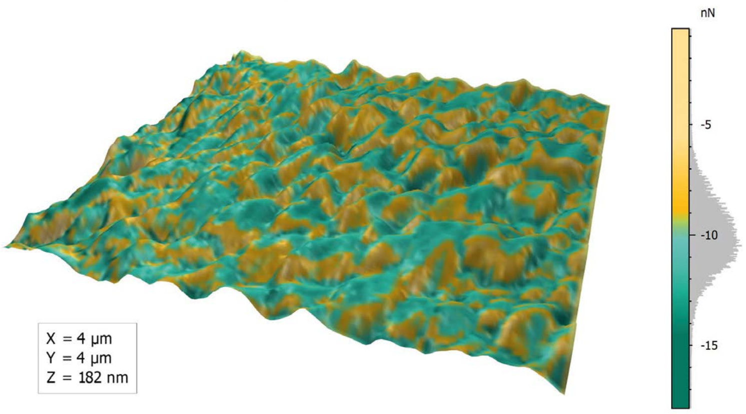
Parameters for individual cursors	Unit	R2
Distance	µm	0.449
Force	nN	-15.4

2 Multilayer 3D view

Force value was applied as texture to the topography of the sample.

In the resulting 3D model, the granular structure of the surface is clearly visible (the bioceramic material is obtained from a powder).

It is possible to study correlations between topography and force.



Authors (members of the Science of ceramic processing and surface treatments laboratory - SPCTS):
Chantal Damia, senior lecturer, Nadia El Felss, PhD student, Valérie Coudert, assistant engineer
Contact: spcts@unilim.fr

Read more:
Laboratory website: www.unilim.fr/spcts/-Presentation-.html
Video presentation: www.lesceramiquesdufutur.com/home

SURFACE METROLOGY QUIZ: TEST YOUR KNOWLEDGE

How much do you know about surface metrology?

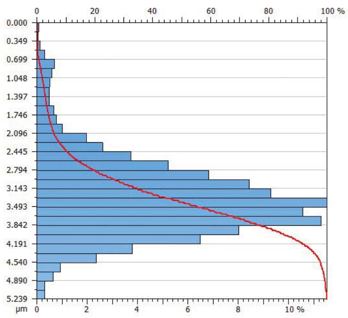
In this edition of Surface Metrology Q&A, François Blateyron, Digital Surf's ISO surface metrology expert, is the one asking the questions. This quiz covers topics such as: areal surface texture, areal field parameters and profile parameters.

Help is at hand if you get stuck as all the answers are to be found in the pages of our online metrology guide: www.digitalsurf.com/en/guide.html

Please bear in mind that for some questions, there are several correct answers.

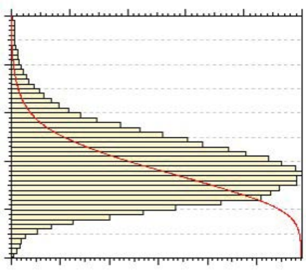
Question 1: Which of the following parameters are related to height distribution?

A. Rz
B. Rq
C. Rsk
D. Rmr



Question 2: Which of the below height distribution graphs corresponds to a negative skewness?

A. Graph 1
B. Graph 2
C. Neither



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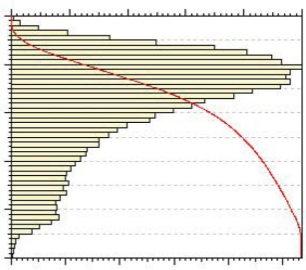


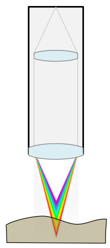
2

Question 3: Which of the following statements are true?

A. It is mandatory to use a Gaussian filter for roughness parameters
B. Some surface texture parameters can be calculated without any filter
C. Without any specific indication, the Gaussian filter should be used
D. The Gaussian filter is best used on structured and stratified surfaces

Question 4: Which of the following instrument technologies have a dedicated document in the new areal standard?

A. White-light interferometry
B. Fringe projection
C. Areal stylus profilometry
D. Chromatic confocal probe
E. Scanning electron microscopy
F. Confocal microscopy

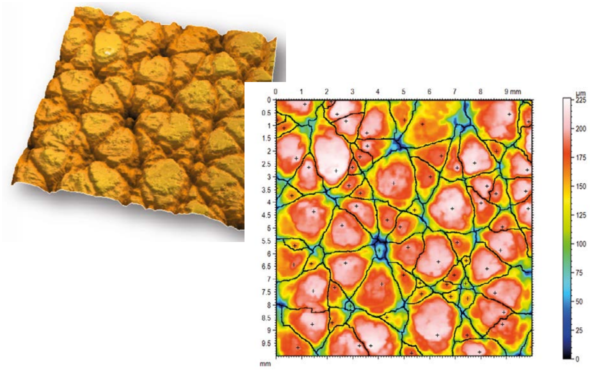


Question 5: In which of the following cases it is necessary to evaluate surface texture on a surface instead of on a profile?

A. When the surface is isotropic
B. When the surface is structured
C. When the surface contains scratches in various directions
D. When the surface has very small grains equally distributed
E. When the surface has big grains/holes unequally distributed
F. When roughness is above 10 µm

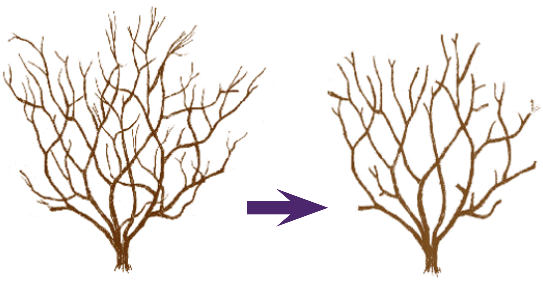
Question 6: Feature parameters are areal parameters…

A. that are not related to surface heights
B. that quantify topological features
C. that do not take into account all surface points
D. that are calculated after a height discrimination



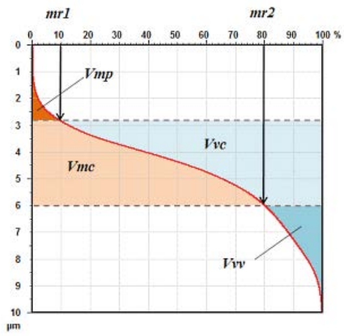
Question 7: Wolf pruning is…

A. used in the calculation of spacing parameters
B. used in segmentation, to reduce the number of features
C. based on a percentage of Sz
D. a special type of nesting index



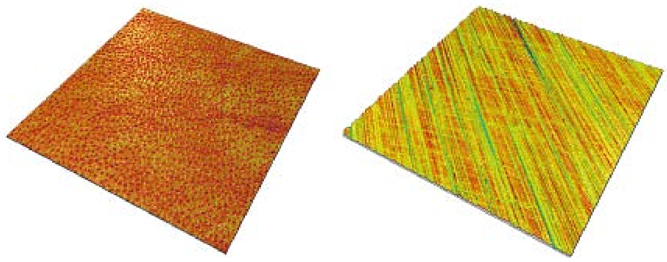
Question 8: Which of the following statements about volume parameters are true?

A. Vmp could be used in replacement of Spk
B. Volume parameters contain material and void volume
C. The value of Vmc is always greater than the value of Vmp
D. Volume parameters are calculated from the Abbott curve



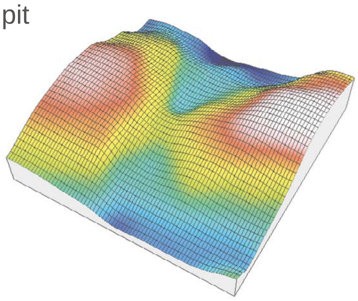
Question 9: Which surface characteristics are assessed with Str?

A. Surface complexity
B. Surface isotropy
C. Surface flatness
D. Surface smoothness



Question 10: Which of the following statements about saddle points are true?

A. They are located at the intersection of a course line and a ridge line
B. They are points where all downward paths converge
C. They are points used in the pruning process
D. They are points located exactly between a peak and a pit



Resources & answers

- You will find help on the questions in this quiz (as well as a wealth of other information on surface metrology) within the pages of the Digital Surf Surface Metrology Guide: www.digitalsurf.com/en/guide.html
- To see the answers, fill in the online quiz: goo.gl/forms/Ncme0coxjcczKaeA3

Good luck!

EVENTS & PRODUCT HIGHLIGHTS

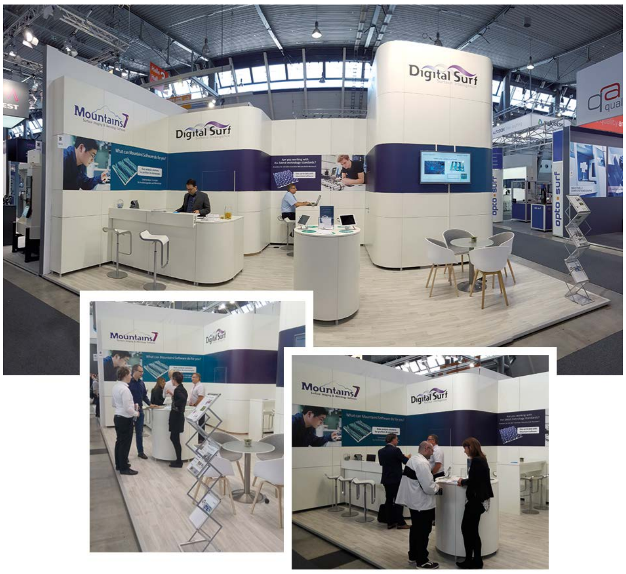
A look back at Control 2017

Control 2017, the world's leading trade fair for quality assurance took place in Stuttgart, Germany on May 9-12.

Alongside over 940 exhibitors from over 30 countries, Digital Surf was proud to participate and present the latest 7.4 release of Mountains® software for profilometry and microscopy. New functions on show included improved zoom, 3D printing and 3D imaging options.

Great to see so many of our partners demonstrate Mountains®-based software products on their own stands too.

A big thank you to one and all and we look forward to seeing you again in 2018!



Met & Props 2017

The 16th International Conference on Metrology and Properties of Engineering Surfaces was held June 26-29 in Gothenburg, Sweden.

Four successful and very busy days saw the presence of 121 delegates and 9 exhibitors from 17 different countries. Anne and François represented Digital Surf and were very happy to meet with many members of the international surface metrology community.

As the meeting came to a close, the organizers announced two future events not to be missed:

- the Met & Props summer school in 2018
- the 17th International Conference on Metrology and Properties of Engineering Surfaces in Lyon, France in 2019.

3D printing contest

▶ Win your sample in 3D!

Following their success at the MRS Exhibit last fall in Boston, our 3D printed models of data processed using Mountains® software will once more be on show to visitors to the Digital Surf booth at the Microscopy & Microanalysis Exhibit in St Louis, Missouri (August 6-10) and the Japan Analytical & Scientific Instruments Show in Tokyo, Japan (Sept. 6-8).

And what's more if you'd like to see your data printed in 3D, all you have to do is send us your files! You have **until September 30, 2017** to submit them to applications@digitalsurf.com (be sure to send us a Mountains® file (.mnt) containing a 3D view of the data you wish to be printed).

We will then select a winner who will receive a 3D printed model by post. This model will also feature on our booth at this year's MRS Exhibit.

WHAT'S HOT ONLINE

Popular on Facebook

Staff at Digital Surf's Besançon office took part in a one-day photo shoot. Watch out for the photos on the brand new website coming soon!

World Metrology day - May 20, 2017

This year's theme: measurement for transport. Did you know surface metrology plays a crucial role in making our modes of transport safer, more reliable and more fuel-efficient?

Have you visited our YouTube channel recently?

Discover tips for getting started with Mountains®, tutorials for SEM image reconstruction & colorization and much more!

www.youtube.com/channel/UC5cyEQHs-9IWZdn0p-cJcJA

Surface newsletter

Know a friend or colleague who would be interested in receiving the *Surface Newsletter*? Let us know: contact@digitalsurf.com

The newsletter is available for download on our website www.digitalsurf.com

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Microscopy & Microanalysis Booth 517
America's Center Convention Complex, St. Louis, Missouri, USA
August 6-10, 2017
www.microscopy.org/MandM/2017

Japan Analytical & Scientific Instruments Show
Makuhari Messe, Tokyo, Japan
September 6-8, 2017
www.jasis.jp/en

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