FALL 2020

Newsletter

SURFACE

Surface imaging, analysis & metrology news from Digital Surf

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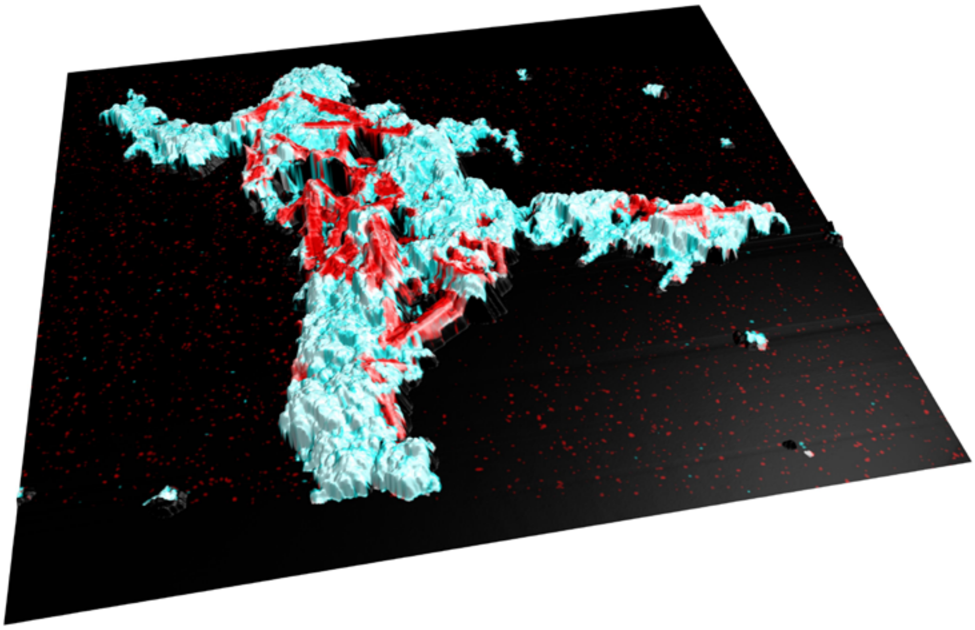
IN THIS ISSUE

CORRELATIVE ANALYSIS
NEW STUDY COMBINES AFM, SEM & EDX DATA



Nanoparticles can be found almost everywhere from foods to cosmetics and pharmaceuticals. Their characterization at the nanometer scale can be particularly challenging.
By combining measurements from several instrument techniques in Mountains®, researchers at the LNE Nanotech Institute (France) found a solution to this problem.

... Turn to page 6 ...

Watch our **WEBINARS**

Digital learning resources

With many still working from home, digital resources are more useful than ever.

Our Mountains® webinars are online, available for you to watch, as many times as you need and from wherever your workspace is at the moment. bit.ly/2Xqqro3

BETTER THAN EVER: THE MOUNTAINS® 8.2 RELEASE

The latest Mountains® software release contains a huge benefit: most users now have access to a range of powerful tools for conducting statistical analysis, including all the latest improvements.

In addition, further developments have been made to bridge the gap with former SPIP™ software for SPM image analysis, making life easier for users familiar with this platform. Improved particle analysis, extra learning resources and color visualization of more types of data (including freeform surfaces) complete this new version.

***Above.** Tools for processing large collections of data and creating comprehensive statistical documents have been made available to most users.*

ACCELERATE ANALYSIS WITH BUILT-IN STATISTICS TOOLS

No need to export results to other programs for statistical analysis!

In modern industrial and research processes, large quantities of measurement data are generated. From version 8.2 onwards, most Mountains® users can access powerful statistical tools to help them handle this ever-growing quantity of data and perform meaningful analysis.

We've also added some cool new features for aggregating series of results in one document.

SPM USERS: THESE ONES ARE FOR YOU!

The past few releases since 8.0 have each seen additional improvements to tools used by those analyzing data from scanning probe microscopy. 8.2 is no exception as we continue to integrate and improve features from SPIP™ software from Image Metrology.

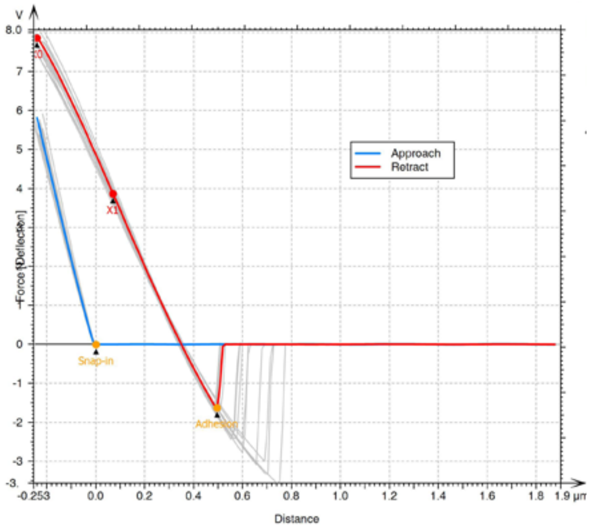
For customers currently making the switch from SPIP™ software following the announcement of the end of technical support and maintenance by the end of 2020, access to essential tools for AFM image analysis has been made quicker and easier.

For those analyzing force curves, the "Correct baseline" tool has been redeveloped to better suit users' needs. Interactive learning tools ("templates") have also been updated.

Other enhancements include: direct access to operators and studies when working with a multilayer dataset and better control of axis settings.

EASIER COLOR PALETTE EDITION

On any data with a color palette, a cursor has been added allowing you to quickly check the uniformity of planes, dispersion of the heights etc.



Information					
Force curve	1 / 15				

Individual cursors	Unit	X0	X1	Snap-in	Adhesion
Force	nN	28.52	14.01	-0.02905	-5.922
Separation	µm	0.3481	0.3595	0.001084	0.3742

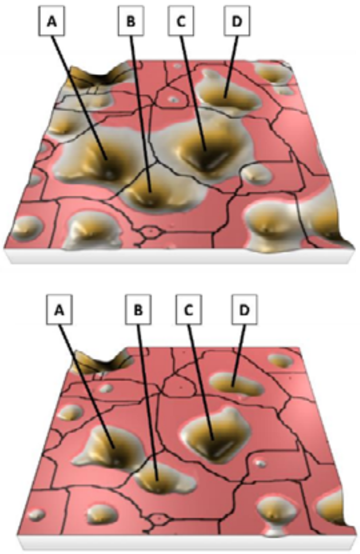
Above. *Series of force curves in a study on adhesion properties (baseline corrected with updated Mountains® 8.2 tool).*

STAY AT THE CUTTING-EDGE OF METROLOGY STANDARDS

8.1 saw the introduction of ISO 21920 profile parameters, before their official publication. In 8.2, users gain access to parameters relative to the revised version of ISO 25178-2, due to be made official in 2021.

In particular, the configuration of Feature parameters has been updated to take into account the upcoming changes, including threshold definition for open/closed motifs.

A new segmentation method for R&W motifs (calculation of upper envelope) is also available.



Top. *All dales are connected (threshold is higher than lowest saddle point).* ***Bottom.*** *With a lower threshold, A and B are connected (open) but C and D are isolated (closed).*

PARTICLE ANALYSIS JUST KEEPS GETTING BETTER

Particle analysis tools are amongst some of the most popular with Mountains® users. That's why it's important to us to keep making them even better and easier to use.

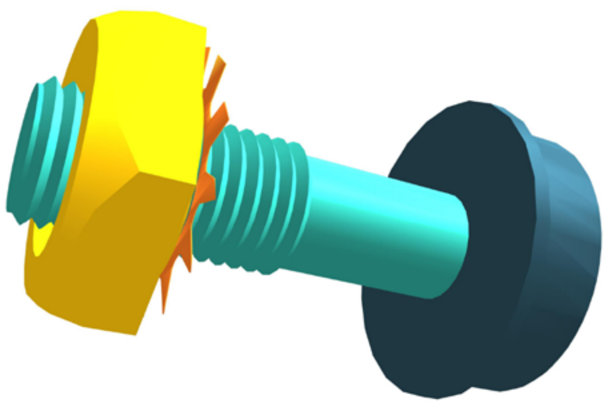
8.2 sees the introduction of new options for sorting and masking particles, including the ability to ignore hidden and non-classified particles in tables and statistical representations.

COLOR MY MOUNTAINS®

Color, as well as making images more aesthetically pleasing, can really help speed up comprehension and interpretation of surface data.

In 8.2, shell (freeform) studiables can now be loaded and displayed using the colors contained in the file itself. Various 3D file formats are supported including the widely used PLY format.

The ability to attribute different colors to Multi-layer profiles also brings an extra level of visualization to users working with this type of data.



Above. *Shell datasets containing color information can now be displayed.*

LEARN MOUNTAINS®... WITH MOUNTAINS®

Whether you're starting to work with Mountains® or whether you're a seasoned user, head over to the Help tab and click on "Home" to access interactive learning materials and get up to speed with the latest tools. 8.2 brings new ready-to-use "templates" for users of SPM (for force curves) and Hyperspectral Imaging techniques.

How to update to Mountains® 8.2
Access to this latest release is included in the Mountains® Software Maintenance Plan (SMP). Please visit www.digitalsurf.com/support/software-updates
To find out more about SMP options, please contact sales@digitalsurf.com

TRANSITION TO MOUNTAINSSPIP®
AN SPIP™ USER'S PERSPECTIVE

With the end of SPIP™ maintenance and support announced for the end of 2020, **Dr Dalia Yablon, founder of SurfaceChar LLC** and longtime SPIP™ user, shares with *Surface Newsletter* her experience on transitioning to MountainsSPIP® for AFM image processing and analysis.

I have been an SPIP™ Image Metrology user since 2003 and quickly became a fan for all of its capabilities including more advanced functionality like particle analysis and force curves that were applicable to all AFM images, regardless of instrument vendor.

So I was curious to see what the differences would be with MountainsSPIP® as I transitioned earlier this year. While the organization, nomenclature, and design of the MountainsSPIP® software is markedly different than SPIP™, I was very pleased to see that the performance and functionality matched and even exceeded SPIP™'s capabilities in several important areas.

AN INTERACTIVE WORKFLOW

One of the most important differentiators in MountainsSPIP® over all other software packages I am familiar with, is the ability to save your workflow for future use and modification. For example, figure 1a shows a 2D representation of some AFM data. With a 3-step workflow, we can create the corresponding 3D image in figure 1b, where the phase is overlaid onto the topography.

With a saved workflow, the user can recall this workflow and go in and adjust individual steps to see what effect they may have without having to redo the entire sequence. This ability is also very instructional for other users: they can see exactly how the image was processed and analyzed without having to take screenshots and explanations. Finally, MountainsSPIP® enables the user to substitute a different image into a given workflow, so that it can be widely applied.

POWERFUL CROSS-SECTIONAL ANALYSIS

Generally, MountainsSPIP® offers advanced, powerful capability in all traditional AFM image processing steps that go beyond the conventional capabilities. For example, cross-sectional analysis is a standard tool offered by most image processing software to draw a cross-sectional profile across the image and measure the distance between two points.

But MountainsSPIP®'s capability of cross-sectional profiling is far more powerful as shown in Figure 2a: one can draw a cross-section profile in any direction including circles, zig-zags, or those that cut through the highest or lowest point (calculated by the software). The software allows up

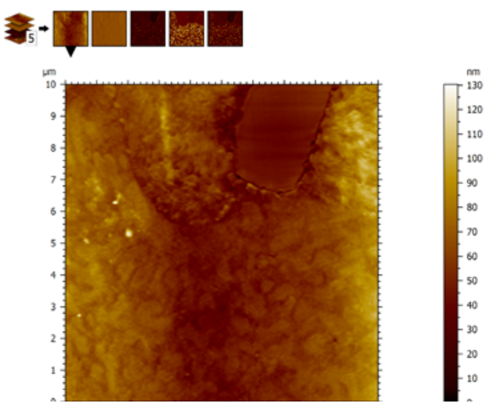


Figure 1a. *Multi-channel AFM data (displayed in 2D), including phase and topography channels.*

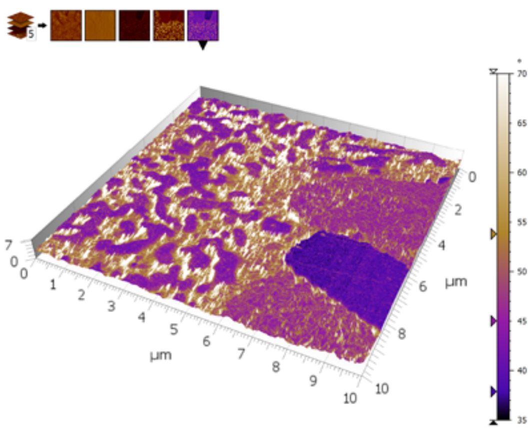
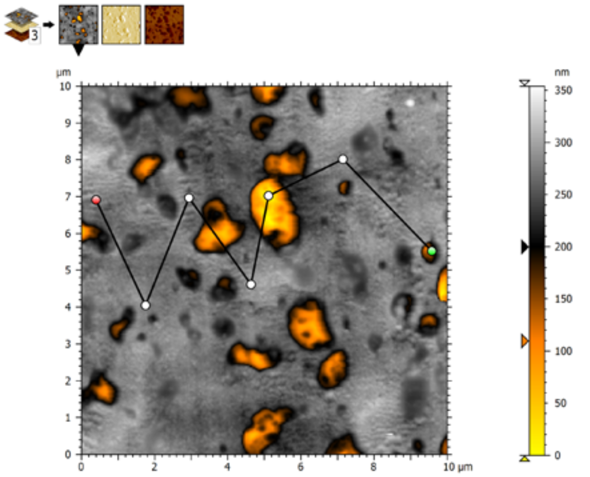


Figure 1b. *3D view of multi-channel surface.*



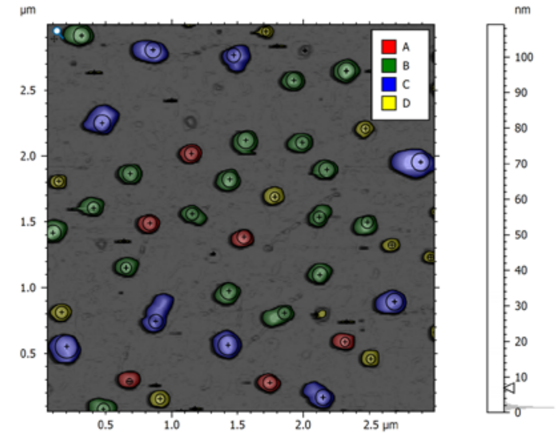
Information	
Layer	a1_00000.0_00000 (Height) (1 / 3)

Figure 2a. *Cross-section profile shown on the surface.*

to 5 cursors simultaneously where a number of parameters can be calculated beyond just difference in x and z as shown in Figure 2b, below.

UNIQUE PARTICLE ANALYSIS FUNCTIONALITIES

Another example of MountainsSPIP® capability lies in the important area of particle detection. All of the conventional options for threshold detection including watershed and edge detection, as well as the ability to merge or split particles are included.

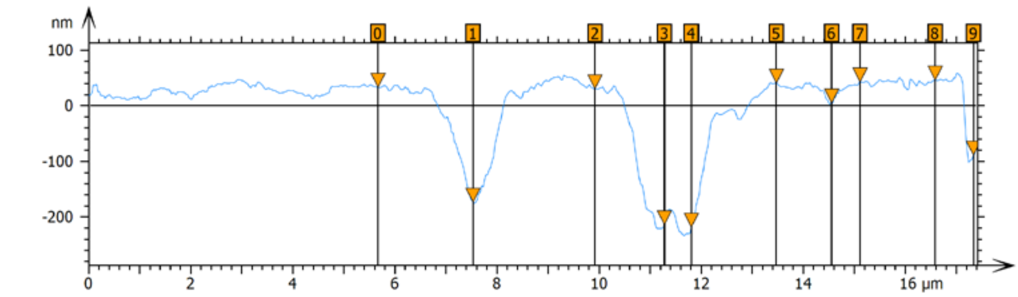


Information		
Method	Threshold detection	
Layer	HeightRetrace (1 / 5)	
Threshold 1	6.918	nm
Number of particles	66	
Coverage	11.27	%
Density	7276154	Particles/mm²

Parameters		Volume of material	Volume of void	Radius (sphere)	Z-max	Volum...
Unit		nm³	nm³	µm	nm	nm³
Particle #14	D	18656	2361	*****	32.29	22853
Particle #15	D	4669	4669	*****	41.62	18893
Particle #16	C	1778108	20534	0.07109	75.88	1831037
Particle #17	D	864.3	900.4	*****	10.46	2275
Particle #18	D	257318	11724	0.05427	45.86	295996
Particle #19	B	1056398	22317	0.06467	81.51	1124908

Figure 3. *Particle analysis study of three different particle varieties.*

In Figure 3, above right, I have created three customized categories based on particle diameter to highlight the various features on the surface. In addition, I have calculated spherical caps for those particles that include them (marked by circles on individual particles) where the radius is calculated as well.



Parameters	Unit	0-1	2-3	4-5	6-7	8-9
Horizontal distance	µm	1.861	1.361	1.666	0.5555	0.7499
Height difference	nm	-206.7	-244.3	259.2	37.78	-134.8
Oblique distance	µm	1.872	1.383	1.686	0.5567	0.7619
Slope	nm/µm	-111.1	-179.5	155.5	68.02	-179.8
Angle	°	-6.338	-10.18	8.840	3.892	-10.19

Figure 2b. *Extraction of a cross-section profile. Height difference, slope, angle and horizontal & oblique distances were calculated.*

But keeping with its tradition of offering more advances, MountainsSPIP® has unique functionalities involving particle classification and visualization that emphasizes ease-of-use, together with the ability to calculate dozens of parameters on particles that we are accustomed with SPIP™.

CONCLUSION

While there is a learning curve with MountainsSPIP®, it is well worth the investment, and one will be rewarded with more advanced and powerful image processing and analysis that crosses all vendor platforms.

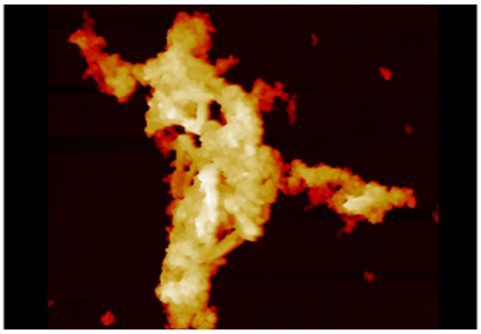
How to switch to MountainsSPIP®
To access more information about MountainsSPIP® software and how to upgrade, please visit www.digitalsurf.com/news/how-to-switch-from-spip-to-mountainsspip/

CORRELATING AFM, SEM & EDX DATA FOR NANOPARTICLE ANALYSIS

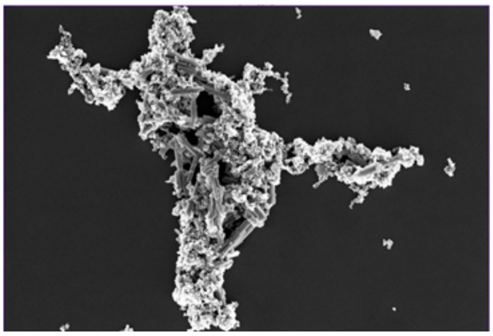
LNE NANOTECH

Nanoparticles are used in many common commercial products like cosmetics, foods, pharmaceuticals and require very specific capabilities for their characterization at the nanometer scale. For this application, a research team at **the LNE Nanotech Institute (France) combined measurements from several instrument techniques** including Atomic Force Microscopy (AFM) and Scanning Electron Microscope (SEM) equipped with a new-generation energy dispersive X-ray detector (EDX).

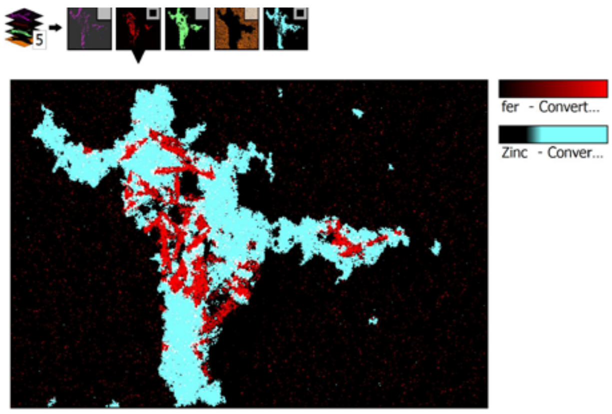
In order to correlate the collected data and extract the relevant information, software with advanced processing capability was required. For this purpose, MountainsLab® developed by Digital Surf is particularly well adapted.



Left. *AFM topography of a nanoparticle mixture*



Left. *SEM image of the same surface*



Above. *EDX data. Areas high in zinc are shown in blue; areas high in iron are displayed in red.*

CHARACTERIZING NANOPARTICLES IN MIXTURES

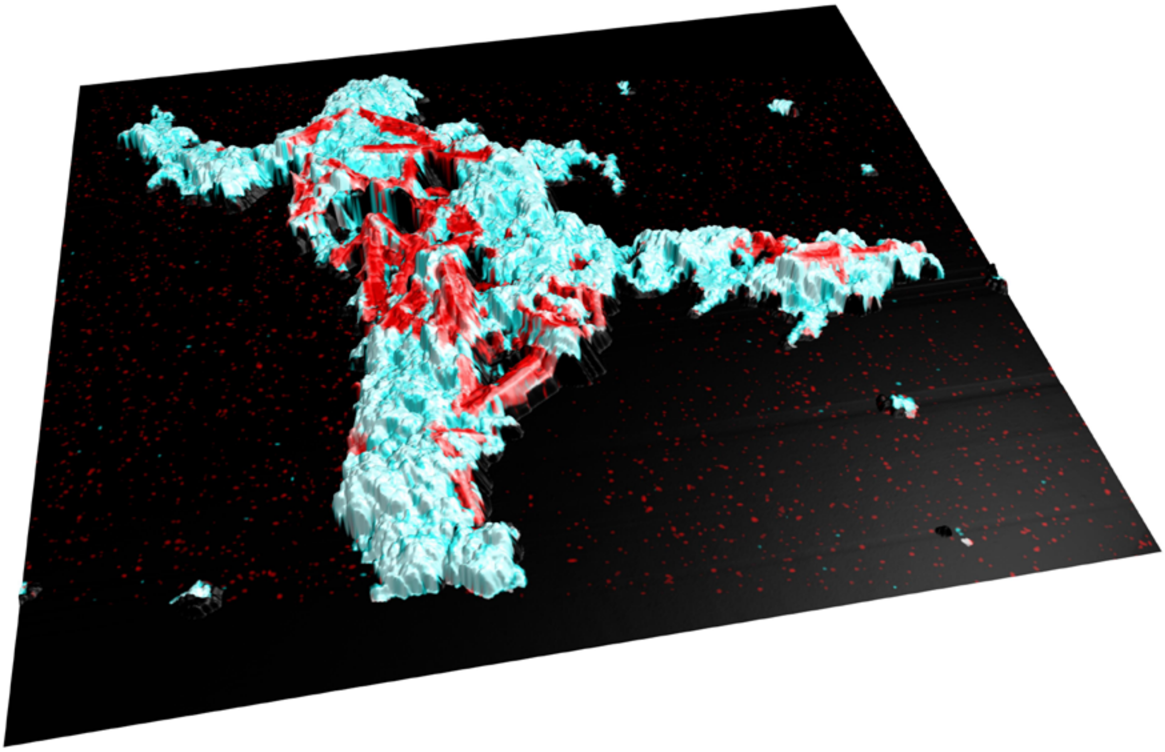
One of the problems regularly encountered in the measurement of nanoparticles are mixtures. Indeed, many products include several populations of nanoparticles to satisfy several needs. For example, to give an orange color to a product, the coloring agents TiO_2 (white pigment) and Fe_2O_3 (red pigment) are mixed.

However, regulatory authorities require producers and suppliers to clearly determine the size and size distribution of each nanoparticle population. This step remains a challenge in the case of mixtures. The correlation of AFM/SEM/EDX measurements is thus a solution to this problem.

AFM and SEM data provide structural information on the particles. Indeed, AFM gives very precise information along the vertical axis, whereas the SEM provides a very high resolution in the plane. EDX is then used as a complementary tool to enable chemical identification and classification of each particle on the microscopy images.

DETERMINING SIZE DISTRIBUTION BY CORRELATION

In a recent study on a mixture of ZnO & Fe_2O_3, we demonstrated that the size distribution of each population is accessible by this combining technique. 3 steps are crucial to achieve the result:



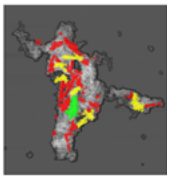
Above. *3D view of colocalized data (AFM topography + SEM image + EDX data).*

1. The sample is correctly prepared to avoid any agglomeration.
2. The region of interest on the substrate is localized in order to image the same particles with different instruments.
3. Correlation of the image data obtained is performed.

As each technique provides images associated with different information and reflecting its own limitations (calibration, dilatations, etc.), correlation can be very difficult to implement without post-processing. MountainsLab® software offers a large range of tools which allowed us to process the various datasets.

Firstly, each image was processed independently. For example, the AFM image was leveled. Then, thanks to the "Colocalization" tool, the AFM and SEM images were combined to obtain an image of the topography of the sample with high resolution in three dimensions. The resulting "image" is then further combined with EDX mapping (Fe and Zn).

Since the correlative image obtained gives us information in 3D, it is possible to obtain an estimation of the volume, size and size distribution of each nanoparticle population imaged.



Above & right. *Particle analysis performed on correlated data showing areas, volumes etc.*

Information			
Method	Threshold detection		
Threshold 1	9.787	GL	
Number of particles	60		
Coverage	6.647	%	
Density	980720	Particles/mm²	

Parameters	Default classification	Projected area	Perimeter	alent d...
Unit		µm²	µm	µm
Particle #1	A	0.01636	1.016	0.1443
Particle #2	A	0.0658	2.807	0.2894
Particle #3	A	0.04321	2.235	0.2346
Particle #4	A	0.1148	3.824	0.3823
Particle #5	A	0.04262	1.646	0.2329
Particle #6	A	0.02796	0.8127	0.1887
Particle #7	A	0.005284	0.4433	0.08203
Particle #8	A	0.03486	1.370	0.2107
Particle #9	A	0.1383	4.628	0.4197
Particle #10	B	0.1768	5.715	0.4744
Particle #11	A	0.04296	2.034	0.2339
Particle #12	A	0.008609	0.7767	0.1047
Particle #13	A	0.00537	0.4613	0.08269
Particle #14	A	0.04884	1.714	0.2494
Particle #15	A	0.0127	1.109	0.1272
Particle #16	A	0.08148	2.847	0.3221
Particle #17	A	0.05898	2.019	0.2740
Particle #18	A	0.05736	1.938	0.2703
Particle #19	B	0.1371	4.985	0.4179
Mean	*****	0.06778	2.493	0.2652

TiO_2 = chemical formula of Titanium Dioxide.
Fe_2O_3 = chemical formula of Iron Oxide.
ZnO = chemical formula of Zinc Oxide.

INSTRUMENTS AND SOFTWARE USED

*Atomic Force Microscope + Scanning Electron Microscope + Energy Dispersive X ray spectroscopy + **MountainsLab**® software.*

ABOUT THE AUTHORS

Alexandra Delvallée, Sébastien Ducourtieux, Loïc Crouzier and Mouna Oulalite are members of the Nanometrology group at LNE (French National Laboratory of Metrology and Testing). Learn more: www.lne.fr/fr/LNE-Nanotech

IS MY SURFACE **STOCHASTIC OR DETERMINISTIC?**

Stochastic and deterministic are two terms that are used more and more frequently to qualify modern surfaces. What is their meaning? How do they compare with roughness and waviness concepts? What influence do they have on surface function?
Digital Surf's **senior metrology expert François Blateyron** deals with these questions.

Metrologists are used to classifying surfaces based on their roughness, on an axis between rough and smooth. A surface is said to be "rough" when its average wavelength, i.e. the lateral distance between two zero-crossings of the profile is short. Intuitively, a profile with many height variations per millimeter along X is considered as rougher than a profile with fewer variations. The amplitude of the profile variation is also a determining factor. More difficult to understand is the influence of features or structures in the profile. A feature is an identified "motif" that is repeated on the surface. It can be a geometrical feature, such as a cylindrical pillar standing on a flat surface, or it can be shallower, looking more like an irregular bump.

Most classical parameters, such as Rq (or Sq), called "field parameters", only characterize heights or amplitudes of the profile (or the surface). They use all points of the measured surface to calculate the parameter value. Even when they are associated with an L-Filter, they will usually fail to discriminate the influence of the feature shape on the function. In figure 1, **the three profiles have the same Rq value** although they have obviously different functions. The first one is perfectly sinusoidal, the second one has periodical rectangular grooves and the third one is more random although it is pseudo-periodical.

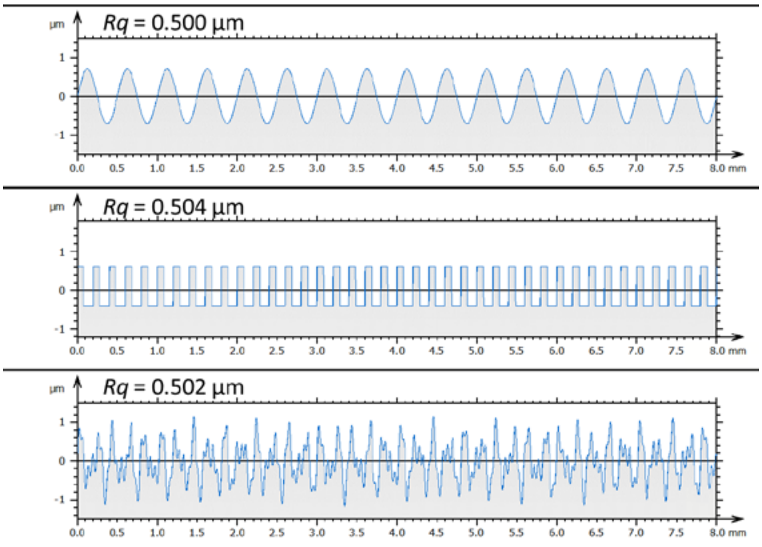


__Figure 1.__ Three profiles with the same roughness value (Rq, L-Filter cut-off = 2.5 mm) but different average wavelengths and different feature shape.

IS "STOCHASTIC" EQUIVALENT TO "ROUGH"?

Obviously, there is an intuitive logical link between "stochastic" and "rough", between "deterministic" and "smooth". However, the "stochastic-deterministic" axis, sometimes referred to as the "Feature spectrum", is not the same as the "rough-smooth" axis. We can even consider that they are two orthogonal axes, forming a 2-dimensional graph in which we can pinpoint materials, machining processes or products (see figure 2).

Now we have a tool to help us categorize surfaces. In the upper-left quadrant of figure 2, rough-stochastic surfaces are obtained by traditional machining processes. In the lower-left quadrant, smooth-stochastic surfaces are obtained by super-finishing or polishing, or by fluid-coating. In the lower-right quadrant, the smooth-deterministic surfaces are produced by molding or lithography to create micro-patterns. Last, in the upper-right quadrant, rough-deterministic surfaces contain features in a more natural or organic distribution. Many other surfaces or machining processes can be displayed in such a graph.

A third axis could even be implemented, to form a 3-dimensional graph where surfaces are a point in space, like a point cloud, leading to 8 categories. This third axis would be "scale" to highlight the multi-scale character of some surfaces that may have, for example, a smooth and stochastic behavior at large scales and a rough & deterministic behavior at small scales.

WHY CATEGORIZE?

These categories can help users choose the right tools to analyze their surfaces. Stochastic surfaces can be characterized using statistical parameters taking into account all points (field parameters). They can usually also be correctly characterized by profilometry as they are isotropic surfaces. At the other end of the spectrum, deterministic surfaces have structures or features that must be characterized individually and then averaged over all features. They also require areal measurements.

The table below summarizes some good practices (of course, these recommendations may vary with the application).

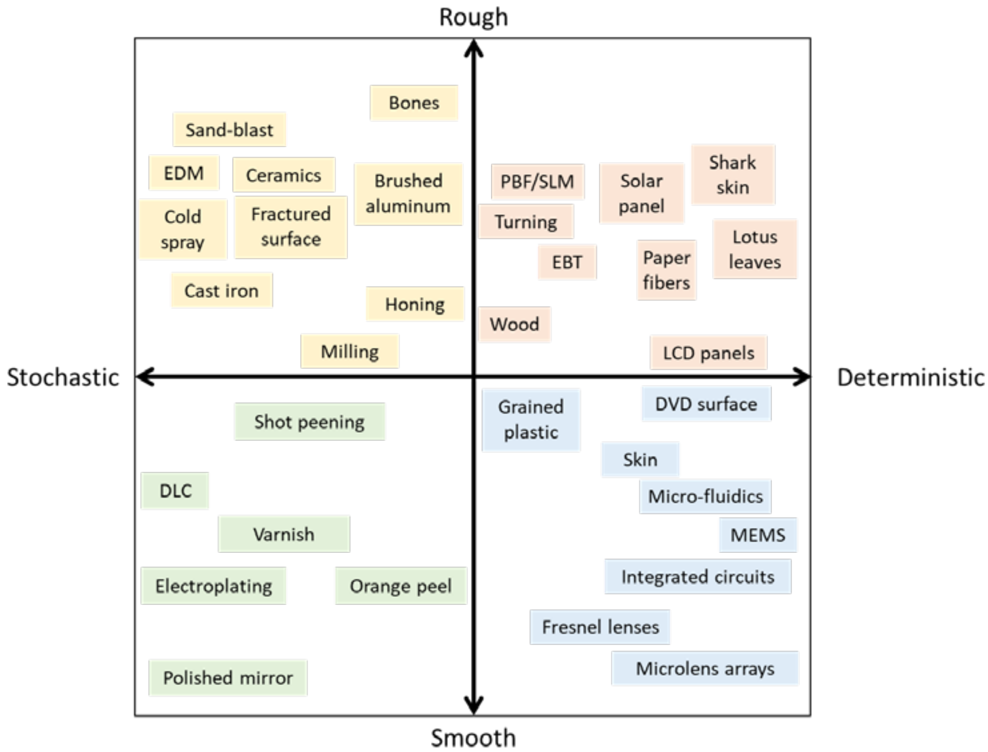
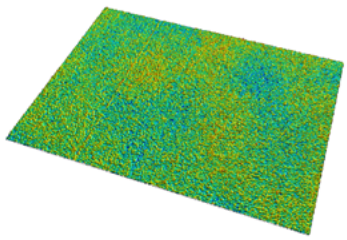
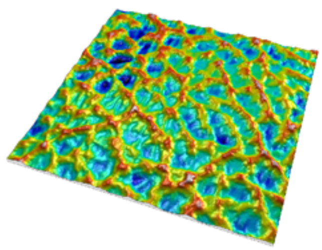
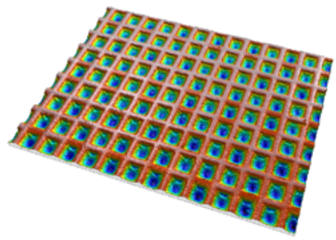


Figure 2. *Categorization of machining processes & materials using both "stochastic-deterministic" and "rough-smooth" approaches.*







	Stochastic surfaces	Stochastic-Deterministic surfaces	Deterministic surfaces
Rough	• Profile analysis is suitable • Use "Field" parameters • Rq, Rsk, Rku, Rmr, Rdq • Surface is usually isotropic • Reproducibility should be good regardless of measurement position • Waviness can be used if function is related to large wavelengths (vibration, sealing, contact)	• Profile or areal analysis is possible • Use roughness parameters • Surface may not be isotropic • Profile analysis may be possible perpendicular to the lay • "Feature-based" characterization is sometimes possible • Results may differ depending on measurement position	• Use only areal analysis • Prefilter the surface (denoise) • Use "Feature-based" characterization • Watershed segmentation • "Field" parameters may be used, with caution
Smooth		• Areal analysis is preferable • Use waviness parameters • Sz, Sdq, Sdr • Select cut-off < 5 x dominant wavelength • Requires many analyses for statistical significance	• Use only areal analysis • Use "Feature-based" characterization • Threshold or Watershed segmentation • "Field" parameters are useless • Report statistics on parameters calculated on each feature

READ MORE IN THE SURFACE METROLOGY GUIDE

- *How to characterize lateral features* *guide.digitalsurf.com/en/guide-lateral-features.html*
- *Areal Feature parameters* *guide.digitalsurf.com/en/guide-areal-feature-parameters.html*
- *About surface function* *guide.digitalsurf.com/en/guide-surface-function.html*

EVENTS & PRODUCT HIGHLIGHTS

JASIS 2020 EDITION: OUR FIRST IN-PERSON EVENT OF THE YEAR

Despite the current challenging health situation, we were very excited to be exhibiting at the JASIS trade show in Tokyo from November 11 to 13 for our first in-person event of the year.

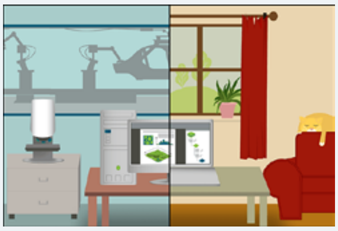
JASIS is known as the largest Asian Analytical & Scientific Instruments show and gathers every year the major instrument manufacturers and thousands of visitors from the scientific community.

This edition was a bit different from usual and the event was organized in a simplified format with measures to prevent the spread of Covid-19 infection. Over 330 companies were present all the same to exhibit their latest analytical solutions & innovations.

Damien and our interpreter Sato-san were on-site to welcome visitors to the Digital Surf booth and to provide them with a live demo of Mountains® software.

Would you like to continue the Jasis 2020 experience? Visit the Jasis Webexpo and find out more about Mountains® software solutions (in Japanese): www.jasis.jp/webexpo/

CURRENT MOUNTAINS® LICENSE OFFERS



"MOUNTAINS® FROM HOME" OFFER

If you currently use a valid Mountains® software license at your office or lab, you can request a time-limited license that you can use from anywhere (or request to extend your time-limited license if you have already obtained one).

The good news is: regardless of the version you currently hold, we'll give you a license for our most up-to-date version 8 release with access to full features, free of charge.

Offer available until December 31, 2020.

Visit: bit.ly/36jV2rR

SWITCHING FROM SPIP™ TO MOUNTAINSSPIP®

If you are an SPIP™ customer with a recent maintenance contract, you can benefit from a free MountainsSPIP® 8 license valid until the end of 2020. Don't hesitate to get in touch with us so that we can activate it for you.

You can continue using your SPIP™ license as well, however, SPIP™ will only be maintained until December 31, 2020.

Visit: bit.ly/32afPel

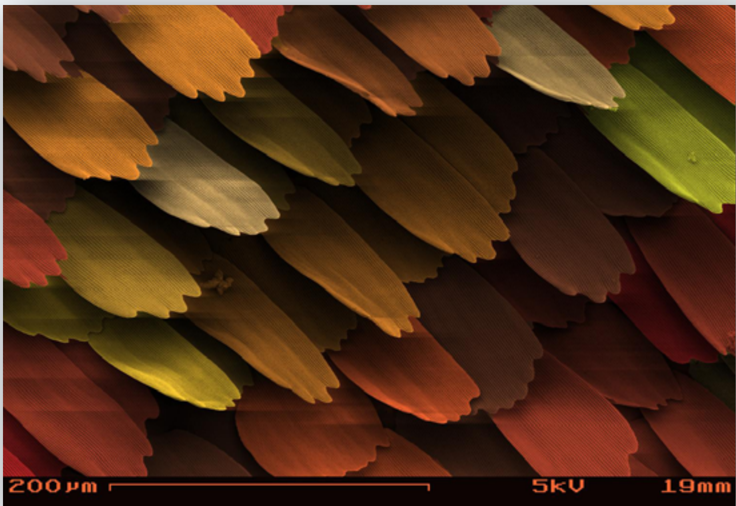
WHAT'S HOT ONLINE

POPULAR ON FACEBOOK

Oct 22, 2020: The Digital Surf staff got together for a first aid training session.

The team got to learn how to examine a casualty, use a defibrillator and perform chest compression. We hear there was even some singing along to the Bee Gees "Stayin' Alive" song.

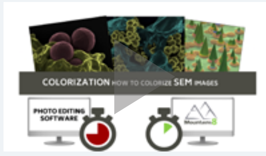
bit.ly/352tt5a



NEW ON INSTAGRAM

Sep 22, 2020: Digital Surf's recently debuted Instagram channel is a new favorite. This channel is dedicated to sharing wonderful images from the microscopy world. bit.ly/2JHgNZj

Have you visited our YouTube channel recently?



Check out our channel for new tutorial videos on SEM & SPM image analysis, as well as other Mountains® software advanced features!

bit.ly/2U2I2za

Surface Newsletter

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MEET DIGITAL SURF

- **2020 Virtual MRS Spring/Fall Meeting & Exhibit**
  Exhibit dates: Nov 27 -Dec 3, 2020
  www.mrs.org/meetings-events/fall-meetings-exhibits/2020-mrs-spring-and-fall-meeting

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