NewsLetter
Fall 2016

SurFace

Surface imaging, analysis & metrology news from Digital Surf

MRS® 2016 Fall Exhibit
Special edition

In this issue

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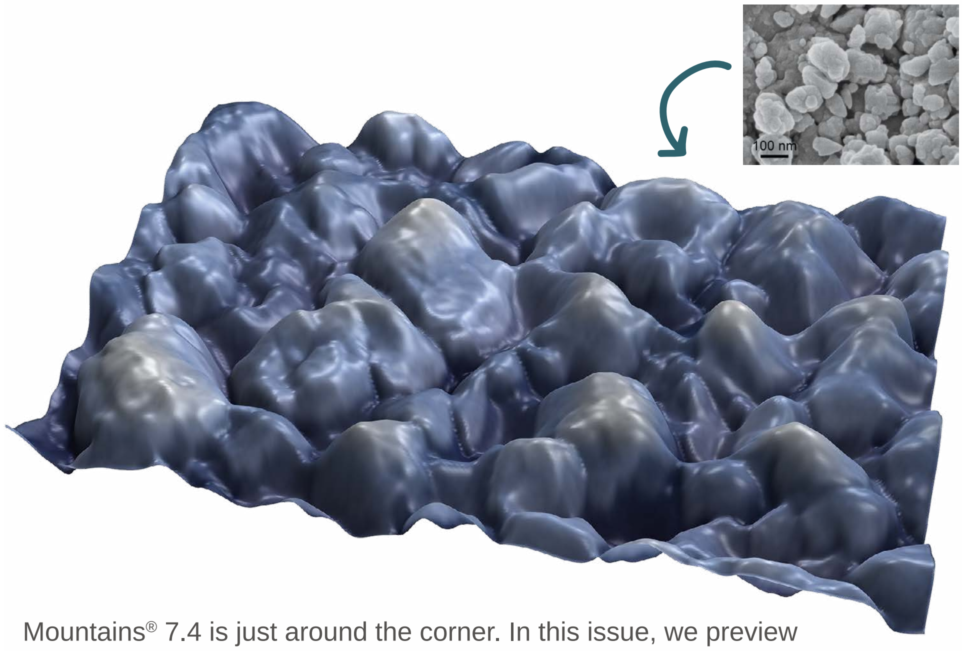
Mountains® 7.4 software preview

Focus on new tools for electron microscopy

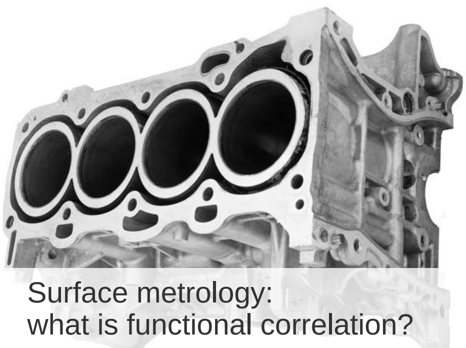


Mountains® 7.4 is just around the corner. In this issue, we preview some of the new features including exciting improvements to the 3D reconstruction tool for single SEM images.

Read more overleaf

Investigating graphene oxide nanostructures

p. 4



Surface metrology: what is functional correlation?

p. 8

PLUS

We look forward to seeing you at

2016 MRS® FALL MEETING & EXHIBIT

in Boston, Massachusetts, USA

November 29 - December 1, 2016

Booth #704

WHAT'S NEW IN MOUNTAINS® 7.4?

A SNEAK PREVIEW OF COMING FEATURES

Mountains® 7.4 is on its way and will be made available to users early 2017.

This new release includes numerous improvements and bug fixes that will interest those using the software to analyze data obtained through profilometry and microscopy.

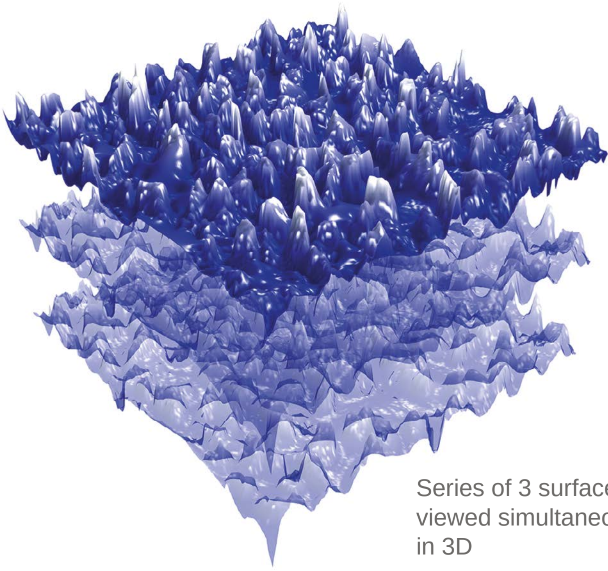
As always, many of the new features included in this version come in response to customer feedback.

Here's just a taste of what to expect:

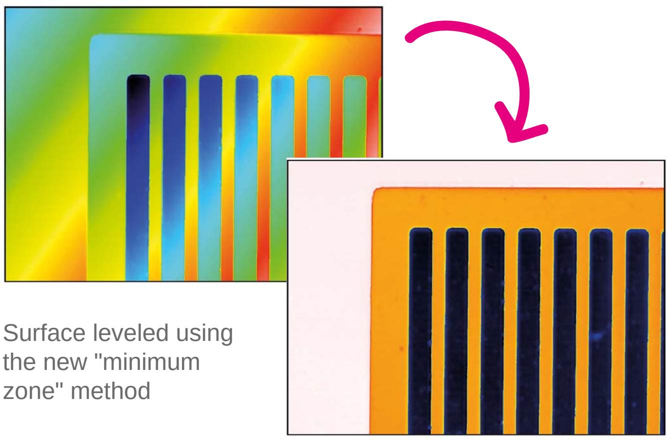
3D views just got even better

Visualize change (wear, deposit etc.) and compare data more easily thanks to the improvement of Mountains® real-time 3D imaging feature which has now been extended to new types of datasets such as:

- series of surfaces
- series of profiles
- hypercubes
- and more



Series of 3 surfaces viewed simultaneously in 3D



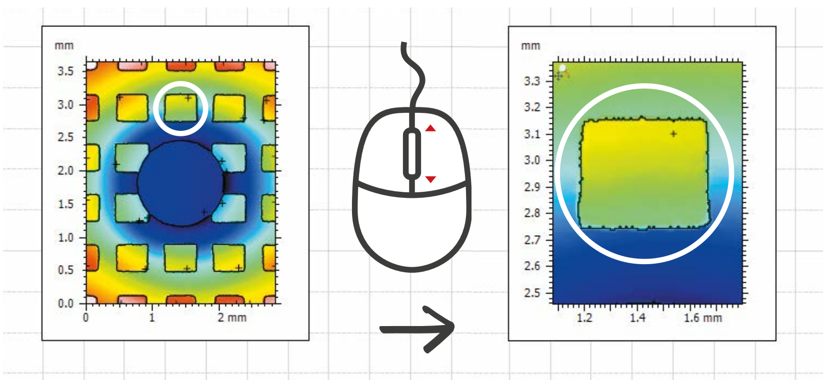
Surface leveled using the new "minimum zone" method

New method for leveling

Leveling is an important operation to perform on any measured data as the result is often not perfectly flat or horizontal.
For users working with multi-plane surfaces, the new "Minimum zone" option for leveling makes eliminating slope quick and easy.

Quick zoom feature

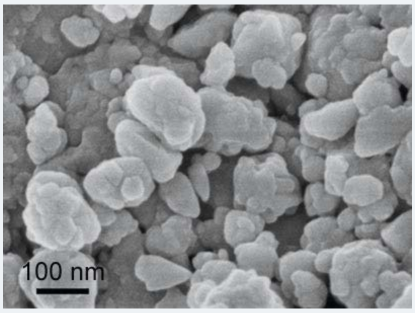
Here's a new feature everyone can use: the mouse wheel zoom now works in all studies performed on surfaces.
This is great for quickly zooming in on a region of interest in your document.
You can also hold down the mouse wheel and move about or zoom in by drawing a rectangular selection.



Focus: new options for single SEM image reconstruction

Ever wanted to get a better look at your SEM sample?

With version 7.2, Mountains® introduced quick and simple 3D enhancement of single SEM images. Based on a unique "shape from shading" technique, this tool is ideal for processing surfaces featuring oblique lighting (ie. those in which the surface is inclined towards the SEM's electron gun.)

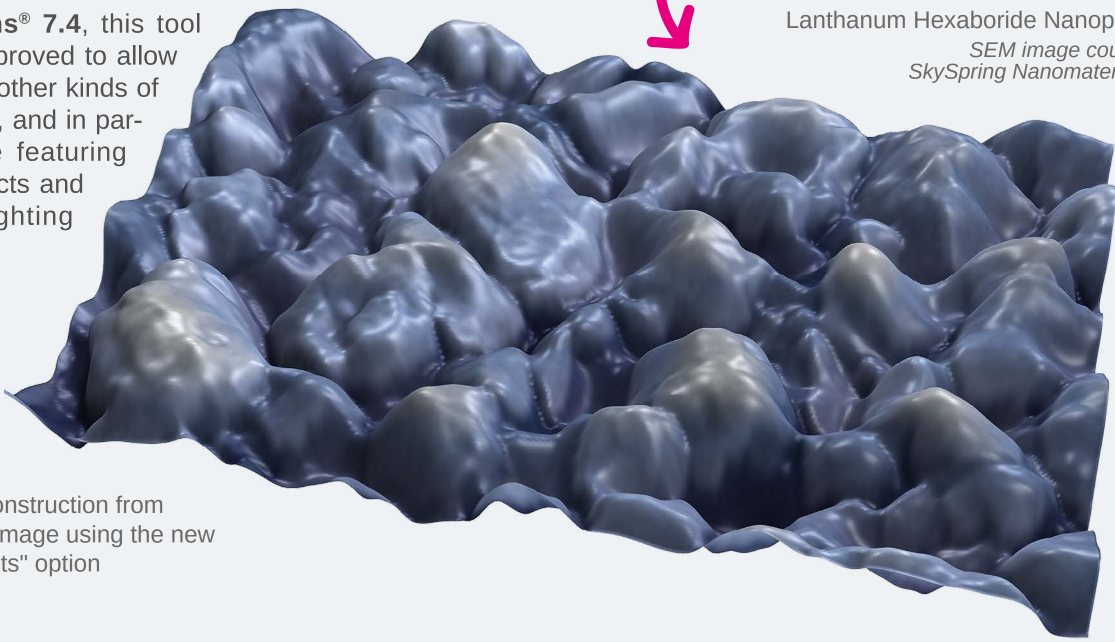


Lanthanum Hexaboride Nanoparticles

SEM image courtesy of SkySpring Nanomaterials Inc.

But what about other types of SEM images?

In Mountains® 7.4, this tool has been improved to allow treatment of other kinds of SEM images, and in particular those featuring multiple objects and balanced lighting conditions.



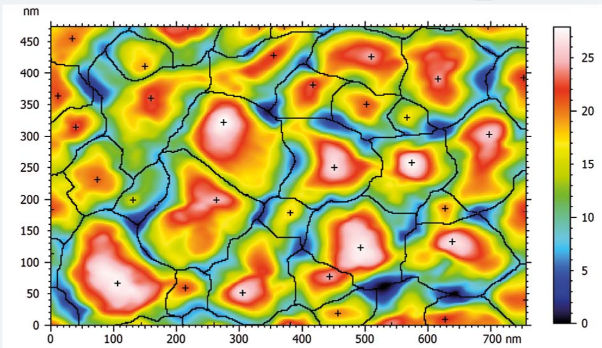
Right. 3D reconstruction from a single SEM image using the new "Multiple objects" option

Why is this useful?

Calculating height values from a single SEM image has long been considered impossible. With this improved operator, Mountains® has taken another big step towards reaching this challenging summit.

Of course Z-values of the reconstructed model in this case are only given as an indication. However, as well as providing amazing 3D images of samples, this tool could be used to find correlations between measured heights and production.

In particular, it also opens up possibilities for image segmentation (otherwise difficult in this kind of SEM image). This in turn facilitates further analysis, such as particle counting and characterization (see right).



Number of motifs	43		
Parameters	**Stat.**	**Value**	**Unit**
Area	Mean	8391	nm²
Mean diameter	Mean	90.5	nm
Form factor	Mean	0.550	

New possibilities for particle analysis in SEM images

And also

Many other improvements and new features are included in Mountains® 7.4 for processing data from a wide variety of instrument types:

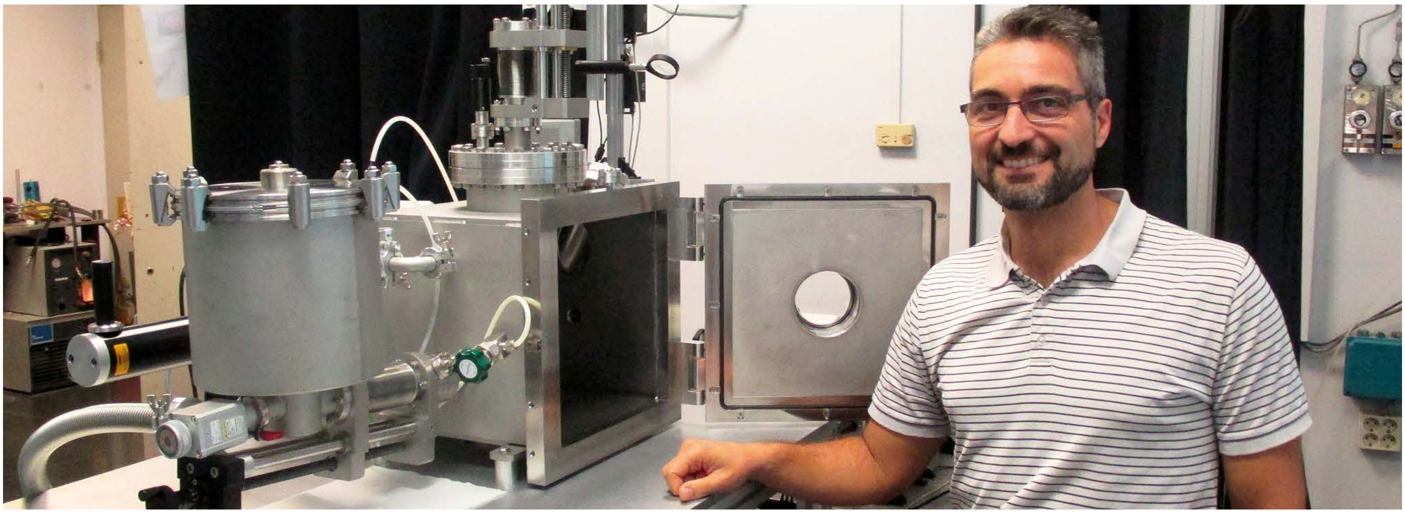
- improved zoom function on profiles & surfaces
- new tools for analyzing spectra
- new features for contour analysis
- more options for text layout
- new parameters etc.

A full list of features will be available as from Jan. 2017 at www.digitalsurf.com

Alternatively, come along to the MRS Exhibit in Boston (Nov 29 - Dec 1) for a preview.

UNCOVERING THE HUGE POTENTIAL OF GRAPHENE OXIDE MEMBRANES

Applications include flexible electronics and water purification



Graphene oxide (GO) is a two-dimensional nanomaterial composed of a graphene monolayer highly functionalized with oxygen-containing chemical groups. Recently, GO nanostructures have attracted great interest due to their exceptional physicochemical properties for many applications. Crucially, GO sheets can be converted into a graphene-like material, named reduced graphene oxide (rGO), by relatively easy, scalable and cost-effective synthesis methods. GO has therefore arisen as a key element in obtaining graphene-like materials at an industrial scale.

Dr Angel Perez del Pino of the Instituto de Ciencia de Materiales de Barcelona (ICMAB)* in Spain, specializes in the transformation of materials by laser processing. With fellow researchers, he recently published findings on the conductivity of such processed GO materials.

"Graphene oxide exhibits very interesting functional properties: it is dispersable in water, biocompatible and its electrical conductivity and optical band gap can be tailored just by modifying its oxidation degree. GO is an electrical insulator whereas rGO is more conductive and can act as a p-type semiconductor. Both GO and rGO have tremendous potential to be used in a variety of electrochemical applications, such as photocatalysts.

Laser processing has arisen as a very promising tool for scalable fabrication of rGO-based devices. In a recent study, we applied nanosecond pulsed ultraviolet laser radiation to GO membranes in gaseous and liquid ammonia-rich conditions. By analyzing structure and composition of the resulting materials, we were able to demonstrate significant differences in the morphology and chemical composition of samples fabricated under analogous laser conditions in these two different environments.

Results and further analysis

Samples irradiated in gaseous conditions undergo a significant deoxygenation process, a slight incorporation of nitrogen species into the reduced GO structure and a large morphological modification. The resulting material is highly conductive whereas the analogous treatment in liquid provokes only a slight decrease in resistance.

SPM-based electric characterization was carried out to further investigate conductivity and the resulting series of spectra processed in MountainsMap® showed the distinct electrical properties of each sample (figure 1 to the right).

When measured with scanning probe microscopy, laser-induced structural defects appear in the gaseous environment sample as tiny filament-like features. Using the multilayer feature in MountainsMap® (figure 2), we were able to create three-dimensional topography-resistance maps which confirm that these filamentary structures mostly show higher resistance at their topmost sites (crests)."

* The ICMAB is a research institute of the Consejo Superior de Investigaciones Científicas (CSIC), the largest research organization in Spain focusing on basic and applied research. Find out more: www.icmab.es

MountainsMap®, an all-in-one solution for multiple types of analysis

As in many research projects, several different kinds of instruments were used to characterize samples in this study (atomic force microscopy, scanning electron microscopy, X-ray photoelectron spectroscopy, resistance measurement instrument). MountainsMap® software provides multiple instrument compatibility and is able to deal with a wide variety of scientific analysis procedures. Here are just two examples.

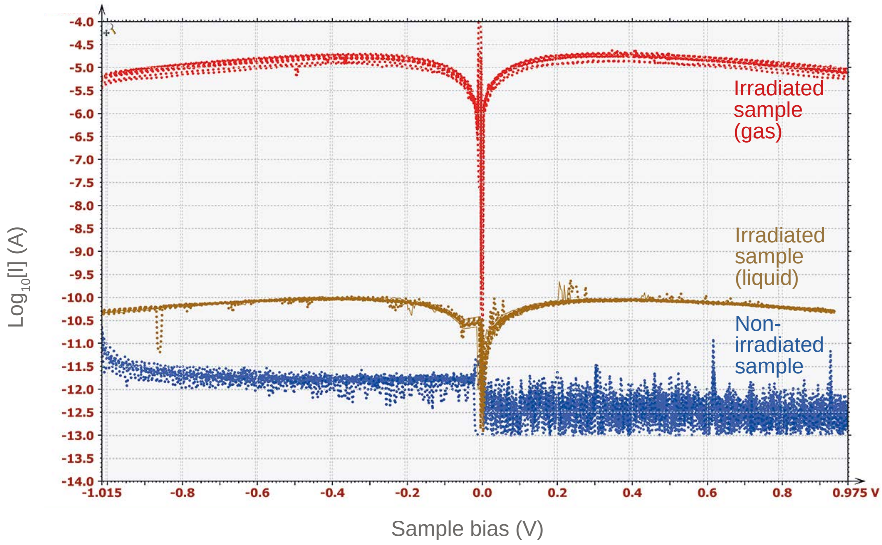
1 Math functions (operator)

Math functions can be applied to data using MountainsMap®.

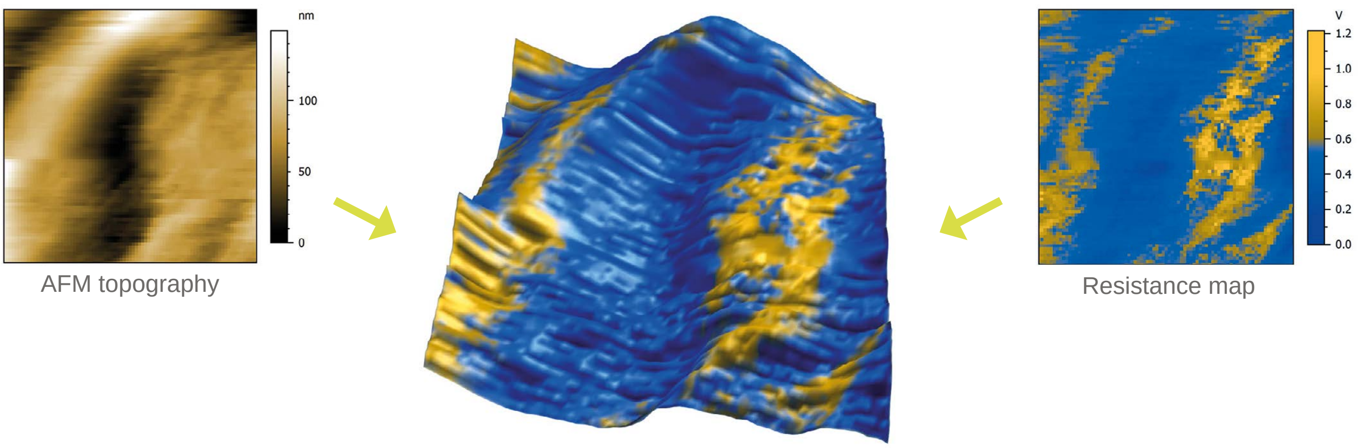
In this case, the math function **abs(A)-13** is applied to a series of spectra in order to convert the raw signal into electrical resistivity.

This makes it possible to observe asymmetric resistance in the non-irradiated GO membrane (in blue).

The irradiated samples (gaseous conditions in red, liquid conditions in gold) reveal symmetric behavior and disclose the ohmic nature of the material.



2 Build multilayer surface (operator) + 3D view (study)



Superimposition of the two layers shows a high correspondence between resistance-related structural defects and topography in the GO sample irradiated in gaseous conditions.

About the author

Research scientist at the Instituto de Ciencia de Materiales de Barcelona (ICMAB) in Spain, Angel Perez del Pino's work focuses on laser processing of nanomaterials (laser surface irradiation, laser direct write, MAPLE). He is also a specialist of SPM-based characterization of functional materials.

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Read more:

Laser-induced Chemical Transformation of Free-standing Graphene Oxide Membranes in Liquid and Gas Ammonia Environments, A. Perez del Pino, E. György, C. Cotet, L. Baia, C. Logofatu, RSC Advances, 2016, 6, 50034-50042.
http://dx.doi.org/10.1039/C6RA07109K

CHARACTERIZING THE SILICON DANGLING BOND

Researchers use combined STM & spectroscopy technique

Semiconductor designers are constantly pushing back the limits of materials science. In the current age of the atomic-level device, one of the main challenges for researchers is to characterize the performance and properties of structures at the nanoscale.

Bruno Grandidier, research scientist with the French National Center for Scientific Research (CNRS), reports on his recent work, focused on understanding the electronic properties of silicon dangling bonds.

"Silicon adopts the diamond crystal structure displayed by carbon and thus forms tetrahedral sp^3 bonds.

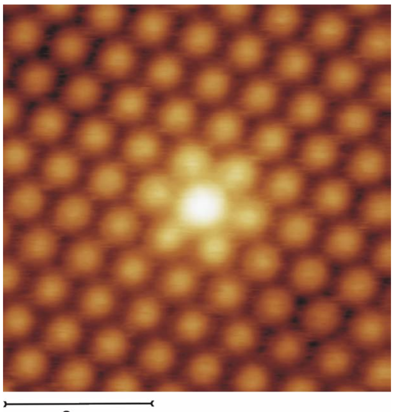
At the surface of a Si crystal, the periodicity of the crystal is broken and the surface atoms rearrange themselves to diminish the number of unsatisfied bonds. These non-bonding *sp* orbitals, which are localized on three-fold-coordinated Si atoms, are known as **dangling bonds**. While they had an undesirable effect on electronic devices in the past, they are now regarded as the ultimate quantum dot for engineering quantum devices on silicon surfaces[1].

A flower-like shape on the crystal surface

Non-passivated dangling bonds can be created on a the surface of a Si(111) crystal that is highly doped with boron atoms to yield a B-doped Si(111)-√3x√3R30° surface.

They give rise to localized states in the band gap of silicon (1.2 eV) that can be characterized with scanning tunneling microscopy (STM) and spectroscopy at a single level.

In the atomically-resolved STM image below, each rounded protrusion corresponds to a single Si adatom (atom lying on the crystal surface). A non-passivated dangling bond appears as a very bright adatom, surrounded by six bright adatom neighbors, forming a flower-like shape.



By acquiring spatial maps of the current and the differential conductivity along with the topography (using a method called Current Imaging Tunneling Spectroscopy or CITS), it is possible to measure the extent of the electron wave function of each quantum level of the non-passivated dangling bond with picometer resolution[2&3]."

References

1. Schofield, S. R., et al. "Quantum engineering at the silicon surface using dangling bonds." Nature communications 4 (2013): 1649.
2. Berthe, M., et al. "Probing the carrier capture rate of a single quantum level." Science 319.5862 (2008): 436-438.
3. Nguyen, T. H., et al. "Coulomb energy determination of a single Si dangling bond." Physical review letters 105.22 (2010): 226404.

About the author

Bruno Grandidier is a research scientist with the French National Center for Scientific Research (CNRS). He works at the Institut d'Électronique, de Microélectronique et de Nanotechnologies (IEMN), in Lille, France, his main areas of research being semi-conductor nanomaterials and near-field microscopy. He also lectures on quantum mechanics, solid state physics, nanosciences and nanotechnologies at the Institut Supérieur de l'Électronique et du Numérique (ISEN) in Lille.

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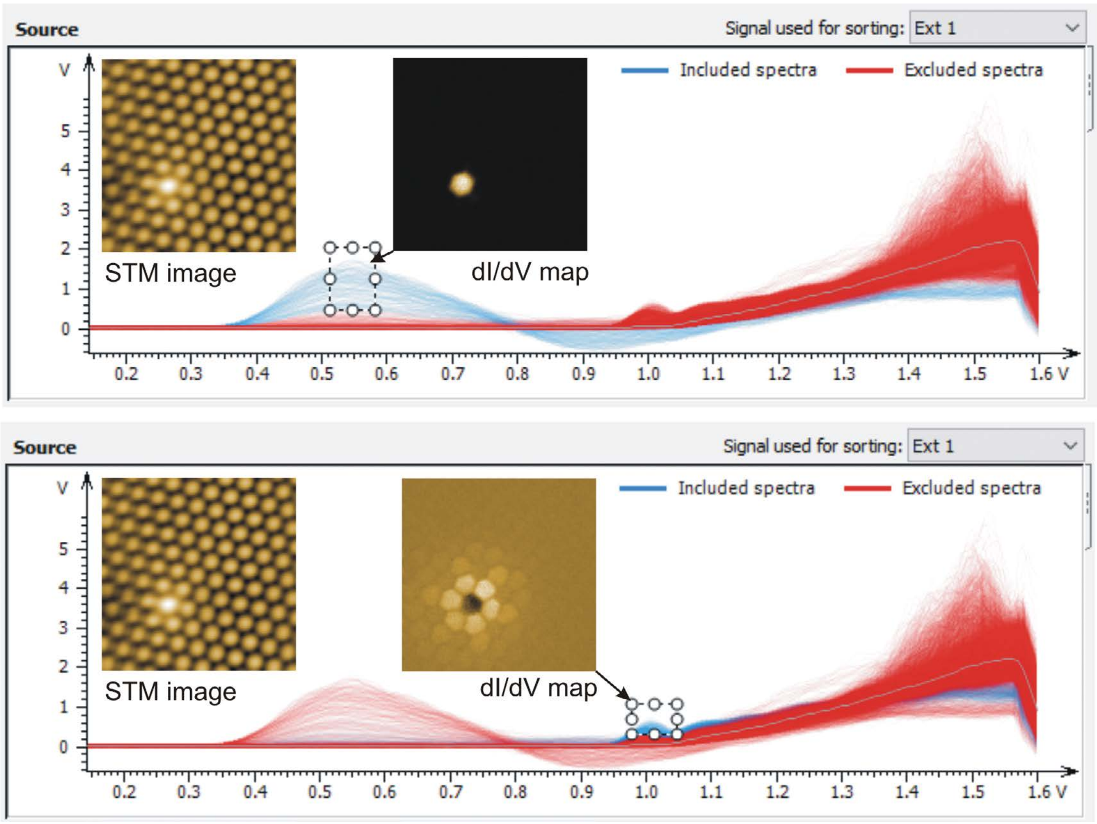
New tools in MountainsMap® for spectral analysis

MountainsMap® software was used to view and analyze CITS cubes containing 14,400 spectra.

The search for localized states and the visualization of the spatial distribution of the electron on each state was achieved either manually or automatically, thanks to two recently developed tools.



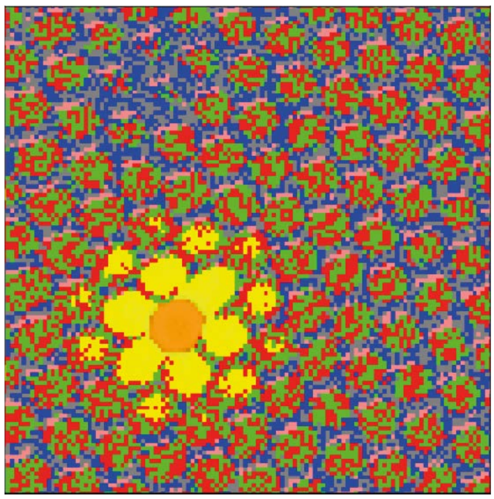
Above. Flattened spectra view helps determine energy level properties

Sort spectra

The first is based on the selection of a set of spectra from a flattened spectra view. As shown to the right, the flattened spectra view allows direct observation of peaks and of their spatial occurrence using the Sort spectra operator. The results are then compared to the topography and the extracted slices of the dI/dV cube (see insets) at a particular energy peak in order to determine the energy level properties.

Extract component

The second method relies on powerful algorithms for independent component analysis (ICA), to demix a set of statistically independent sources (orthogonal quantum states). As shown in the figure to the right, the Extract component operator (soon to be available in MountainsMap®) provides robust data mining capabilities for spatial mapping of both quantum levels. Moreover, for each component detected, the operator indicates the related spectrum, thus simplifying the screening of large numbers of spectra.



Above. Result of component extraction

Results

The study reveals the existence of two states, one level positioned deep in the band gap of silicon and strongly localized on the Si adatom and a second level below the edge of the conduction band with a spatial extent over a few Si adatom neighbors.

Such analysis is of great importance in understanding electron transfer dynamics and the Coulomb repulsion into a single Si orbital as well as the interaction of the electron with the vibrational modes of the Si adatom.

DOES THE FUNCTION OF A SURFACE DETERMINE HOW I SHOULD CHARACTERIZE IT?

Depending on the functional role of an engineered surface, all or part of the surface may be taken into consideration when calculating parameters.

François Blateyron, Digital Surf's ISO surface metrology expert, sheds light on this issue and details methods for dealing with it.

Many functions of engineered surfaces involve only part of the surface but not all of it.

For example, when two surfaces are in contact - as in the case of mechanical seals - only portions of these surfaces (the highest peaks and hills) interact with each other. Deep valleys or dales do not have any influence in this case.

Conversely, the ability to retain lubricant in the internal surface of a cylinder bore (to ensure correct sliding of the piston) only depends on grooves and pits.

Despite this, most of specifications on drawings indicate field parameters such as Ra or Wa. Field parameters are calculated using all points of a profile or a surface. Traditional surface texture parameters are usually based on sums of heights. But did you know that another family of parameters, known as feature parameters, focuses only on particular points or portions of the surface?

Feature parameters focus only on particular points or portions of the surface

French motifs

Thirty years ago, a French consortium of automotive manufacturers developed a graphical method, called “R&W motifs” aiming to identify structures (or motifs) on profiles that play an active role in the function of surfaces.

Later standardized under the reference ISO 12085, this method defines motifs as the triplet “peak-valley-peak”. The algorithm described in the standard is based on the identification of local extrema followed by the selection of significant motifs.

When a motif is found to be insignificant, it is merged with one of its neighbors to form a larger one. After selection, only significant motifs remain. Parameters are then calculated to provide the mean height and mean width of these motifs and they can be used in specifications.

This method proved very efficient, especially after a large-scale campaign to qualify more than 20,000 mechanical components using these parameters with respect to different functions. This then made it possible to derive guidelines for designers to help them correctly specify the surface texture of components on drawings according to the expected function.

Areal features

More recently, a new feature detection method was introduced in the ISO 25178-2 standard. It is based on watershed segmentation of the surface followed by a pruning method aimed at selecting significant features on the surface.

Areal features can be points (peaks, pits or saddle points), lines (ridge or course) or areas (hills or dales) which can then be characterized with numerical values such as heights, areas, volumes, orientation, form factor, etc. They are called feature parameters.

When detecting dales (which are the micro-scale equivalent of catchment basins), the ridge lines around them correspond to contours of the texture cells. Contours around hills are called course lines. This segmentation and the parameters associated with it make it possible to characterize how the surface interacts with its environment through its features. Crucially, they only take into account certain parts of the surface.

Watershed segmentation can also be used to detect shapes. Pre-filtering highlights steep slopes, and the result is a kind of contour detection allowing automatic partition of the surface.

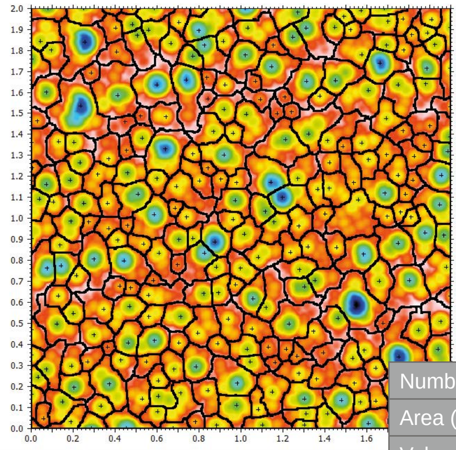
Many applications can benefit from the use of feature parameters. Designers and metrologists must now work together in order to establish function correlation between these parameters and their process parameters. As was the case for profiles with French motifs, it is anticipated that controlling functions using feature parameters may lead to better correlations compared to the widely used (but somewhat meaningless) Ra!

What is functional correlation?

This expression refers to the intended function of a surface on a component as imagined by the designer. For example, the top surface of a cylinder block is supposed to have a sealing function with the opposite surface formed by the cylinder head via a gasket. The sealing efficiency is correlated with the flatness deviation of these surfaces, the height of large hills and the depth of large dales.

A function on steel sheets is the ability to aid paint adhesion while still being smooth enough to allow press forming tools to slide on the surface without cracks appearing. This function is achieved by texturing small scale patterns onto the surface using rollers (thus creating adequate roughness) but also by leaving enough flat plateaus so that the surface appears smooth at larger scales.

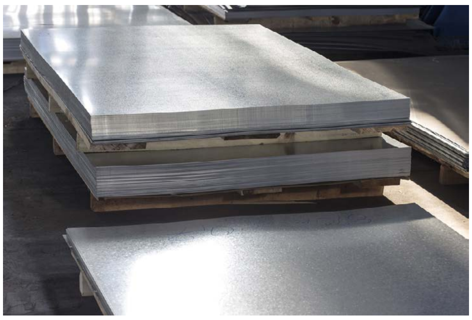
Checking the sealing efficiency of a cylinder block



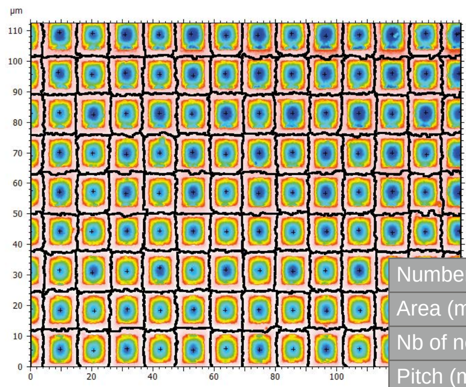
Identification of texture cups and qualification of:

- mean size
- mean volume
- density etc.

Number of motifs	276
Area (mean)	0.01244mm²
Volume (mean)	11227µm³



Steel sheets: obtaining optimum paint adhesion and large-scale surface smoothness



- Automatic segmentation of texture cells on the structured surface
- Detection of missing cells.

Number of motifs	126
Area (mean)	138.2µm²
Nb of neighbors (mean)	5.976
Pitch (mean)	13.34µm
Coflatness (mean)	0.2115µm

Resources

- **ISO 16610-85**: GPS – Filtration – Areal morphological – Segmentation
- **Feature parameters**, chapter 3 in “Characterisation of areal surface texture”, published by Springer
- **Feature parameters**, in the Digital Surf Surface Metrology Guide: www.digitalsurf.com/en/guidearealfeatureparameters.html
- **Segmentation and feature parameters - A corrected definition of watershed segmentation and feature parameters defined in ISO 25178-2**, F. Blateyron, Conference proceedings of the International Conference on Surface Metrology, April 2016, www.researchgate.net/publication/303390269

EVENTS & PRODUCT HIGHLIGHTS

Mountains® on the road
Impressions from the summer shows

It was indeed a busy summer for our team of surface metrology and analysis experts. Mountains® software was on show at no less than three exhibits on three different continents.

Christophe, Cyrille and Damien kicked off proceedings at the end of July with Digital Surf's first participation at the **Microscopy & Microanalysis** conference and exhibit in Colombus, Ohio (USA).

Hosted by the Microscopy Society of America (MSA), this event is one of the highlights in the international microscopy calendar, showcasing the latest advances in science and microscopy analysis techniques.

A few weeks later, at the end of August, the team was able to attend an event a lot closer to home. The 16th **European Microscopy Congress** took place in Lyon (France) only a couple of hours drive from the Digital Surf headquarters in Besançon.

As part of Europe's largest exhibition dedicated to microscopy, Digital Surf was proud to present all the latest Mountains® capabilities to our visitors. In all, the congress gathered over 2500 attendees from over 50 countries.

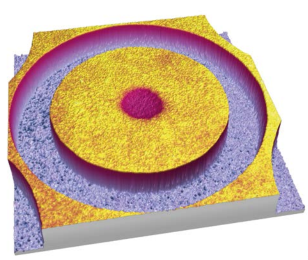
Last, but certainly not least, was the **Japan Analytical & Scientific instruments Show (JASIS)** in Tokyo (Japan) at the beginning of September, with its staggering 24 000 visitors!

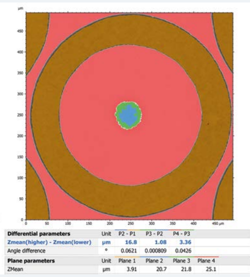
Arnaud, Damien and our Japanese friend and interpreter Sato-san were on hand to meet our partners and clients from the fields of electron microscopy, near-field microscopy and spectroscopy. Thanks to all who came by our booth! We look forward to seeing you next year.

Product news

▶ July 2016

Together with partner Nanosystem, Digital Surf is proud to launch NanoMap Alpha, a new surface imaging & metrology software product based on industry-standard Mountains® Technology.

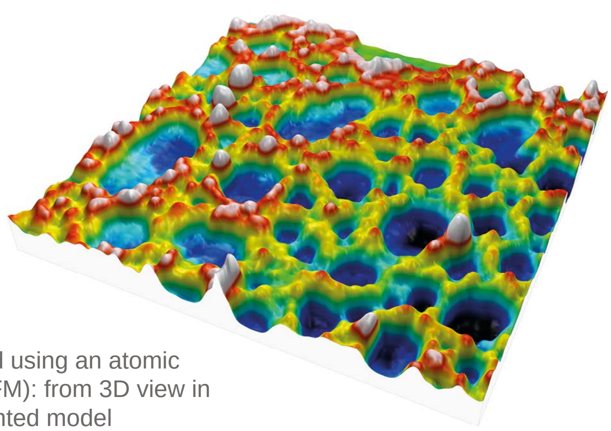


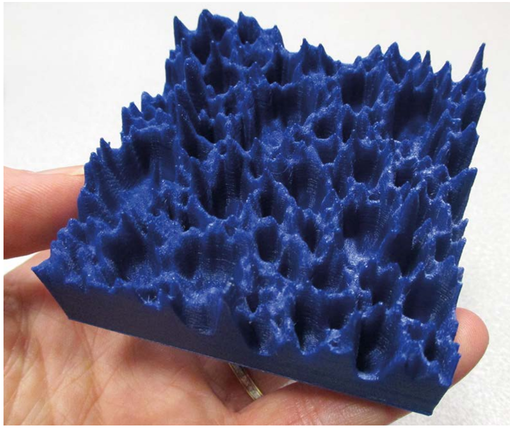


If you are analyzing semiconductors, PCBs, displays, engineered parts, chemical materials or optical parts check out the complete list of features: https://goo.gl/OC31rP

Find out more by visiting: www.nanosystemz.com/en/

BRING YOUR SAMPLES TO LIFE
NEW OPTIONS FOR 3D PRINTING





Ink deposit measured using an atomic force microscope (AFM): from 3D view in Mountains® to 3D printed model

In recent years 3D printing has taken the world by storm and with the latest advances this technology is becoming more and more accessible to the general public.

As far as the world of microscopy is concerned, there are many benefits of using 3D printing to produce scaled-up replicas of samples.

Two-dimensional (2D) images can often prove insufficient for detailed analysis of organization and morphology of micro-structures.

However, being able to generate a highly accurate, solid scale replica of the sample that you are studying with a microscope, being able to hold it in your hand, examine it and show it to students or colleagues, opens up new perspectives and better understanding of complex micro-systems.

Another interesting use of 3D printing is the fabrication of micrometer-scale devices for the medical and electronic sectors.

Improved export for 3D printing

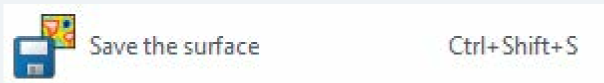
Up until now, users of Mountains® and MountainsMap® software products could already export 3D models of their microscopy and profiler datasets in STL, VRML and X3D formats for 3D printing.

However, new 3D printing file formats have become increasingly popular in recent times. In particular the 3MF format has the advantage of allowing users to manage color and/or texture, something not possible with STL. This file format is set to become a new standard in 3D printing.

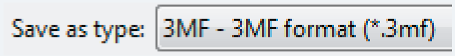
With the new 7.4 release of Mountains®, exporting 3D models in 3MF for direct 3D printing is now possible. Let's take a look at the new options available.

Print a Mountains® 3D model in 4 easy steps

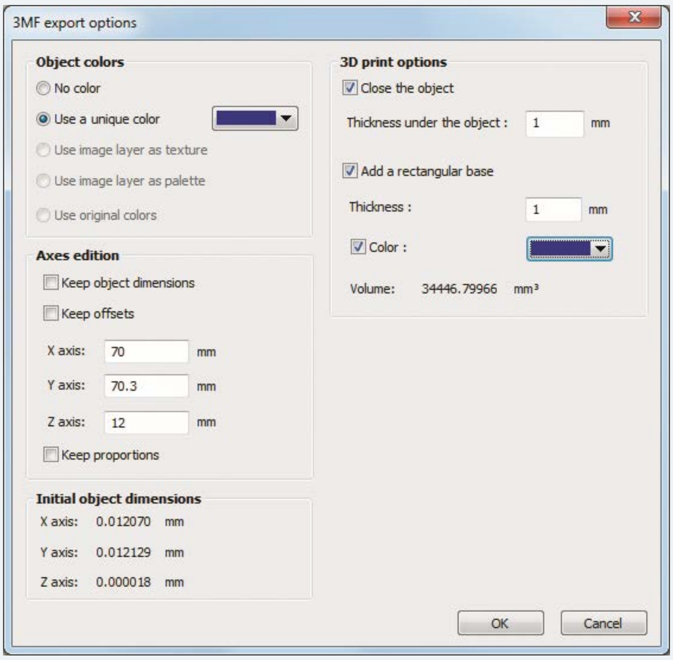
1. Go to the File menu and save your surface



2. Choose to save as a 3MF file



3. Choose export options:
   - apply a color or use colors contained in an image file (if available)
   - edit axes (choose scale factors for X, Y and/or Z)
   - select options for 3D printing (close the object, add a base etc.)



4. Your sample is ready for printing (no post-processing required)!

USEFUL LINKS

FREE TRIAL

TRY MOUNTAINS MAP®

MountainsMap® Premium Software with all the options, free for 30 days!

CONTACT US FOR AN UPDATE

Contact us for information about updating MountainsMap® 6 or earlier software to the latest version of MountainsMap® 7

WATCH A MOUNTAINS® 7 TUTORIAL

Get the most out of Mountains® 7 by watching one of our tutorial videos

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Learn more about Mountains® 7 software by downloading a brochure in English, French, German or Japanese

MEET DIGITAL SURF

MRS Fall Exhibit

Hynes Convention Center, Boston, Massachusetts, USA
November 29 - December 1, 2016
www.mrs.org/fall2016

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