Newsletter









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USING FIB-SEM TOMOGRAPHY TO ANALYZE THE CHEMICAL COMPOSITION OF A MAGNET



A research team based at JEOL France recently studied the composition of magnet material used in the development of DC motors. A FIB-SEM technique coupled with Mountains[®] 9 software analysis provided accurate, visual results.

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Register for our webinar

The Microscopy & Microanalysis 2021 Conference & Exhibit will be a virtual edition.

Come visit our online booth on August 2-5 and signup for the webinar we'll be giving on "Chemical and morphological analysis in SEM": <u>bit.ly/3kANjN3</u>

INVESTIGATING NEXT-GEN ENGINE COMPONENTS AT THE NANOSCALE

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Currently, one of the major challenges in the automotive industry is the development of direct current (DC) electrical motors. A DC vehicle motor incorporates strong magnetic fields at the rotor location. The higher the magnetic field in a reduced volume, the better the engine efficiency factor. A research team based at JEOL France re-



cently studied the composition of such a magnet using a FIB-SEM technique coupled with a specialized analysis software package.

COMPOSITION OF A CERIUM-ALLOYED Nd-Fe-B MAGNET

The key component of the engine is the permanent magnet located inside the rotor assembly which must have very high efficiency.

The sample studied was a Cerium-alloyed Nd-Fe-B magnet (Ce-NdFeB). The Cerium partly replaces the very expensive Neodymium rareearth element (REE) without compromising the magnetic properties.

The research and development of this technology requires excellent knowledge of the chemical phase tomography of the magnet.

FIB-SEMMACHINING&IMAGERY IN BSE MODE

Mountains[®] 9 software tomography analysis is based on a series of high-resolution images representing successive cross sections taken through the depth of the sample. These different planes are produced by FIB machining and imaged in BSE mode.

BSE images are in shades of gray; the composition of the different gray levels of the image is proportional to the atomic mass of the chemical elements that make up the surface being analyzed.

The FIB/SEM technique was first highlighted by the semiconductor industry, seeking to understand the 3D complex structure and/or internal specific failure of electronic devices. JEOL (Japan Electron Optics Laboratory Company Tokyo, Japan), understood the need for Focused Ion Beam (FIB) systems early on and introduced the JIB-10 instrument in 1983.







Figure 2. One of the series of BSE images with highlighted ROI.

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The experiment involved slicing and imaging the magnet with electrons under a strong magnetic field. This was particularly challenging since the magnetic field deflects the electrons.

The new JIB-4700F multiple-beam system from JEOL was able to overcome this difficulty. The magnet was sequentially milled using the ion beam (red arrow in Figure 1a) and imaged by the electron beam (green arrow)."

IMAGE PROCESSING

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SMILEView[™] Map software, powered by Mountains[®] allowed full 3D imaging and analysis of the chemical composition of the magnet material.

The chemical cube generated from the set of BSE images was segmented according to gray scale ranges corresponding to various chemical elements.

Numerous parameters including volume parameters were then calculated on the differentiated and segmented chemical elements

(see Figure 3).

Once the tomogram is rebuilt, the user can virtually re-section the sample using the software, fully explore the 3D volume and get a full understanding of the magnet inner structure.

In this particular reconstruction we can observe very rich intergranular structure of Neodymium and Cerium with a low concentration of Iron in a Nd2Fe14B magnetic matrix.

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(X	Grain material parameters	Unit	Neodynium	Cerium	Nd2Fe14B matrix
μm	Volume	μm³	278.2	765.5	16528
	Volume ratio	%	1.567	4.311	93.07
	Volume ratio (not empty)	%	1.583	4.357	94.06
Figure 3. Visualization of BSE image stack as a cube (above) and volume	Grains count		4285	7241	550
	Grains mean volume	μm³	0.06493	0.1057	30.05
	Grains volume standard deviation	μm³	1.196	3.494	704.1
parameters (right).	Equivalent diameter of grains mean volume	μm	0.4987	0.5867	3.857

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INTRA-ORAL SCANNERS FOR TOOTH WEAR QUANTIFICATION



Erosive tooth wear is an oral condition most commonly caused by acids in our food and drinks. It involves the etching away of hard dental tissue and if left unmanaged, it can result in complete degradation of enamel and exposure of dentine. A research team at **King's College London** has been working on ways to improve the understanding of the tooth wear process and investigating innovative techniques to monitor and treat it.

"Currently, there is a pressing requirement for the development of quantitative in vivo methods to measure tooth wear directly on patients" explains Dr Polyvios Charalambous, member of the research team. "Conventional methods for clinically monitoring tooth wear rely on subjective qualitative visual feedback".

ASSESSING A NEW TOOL

"Intra-oral scanners (IOS) are handheld clinical devices that can capture the 3D geometry of teeth inside a patient's mouth with the aim of replacing the need for conventional dental impressions. They are becoming more common in the clinical setting and have a potential for in vivo quantification of surface changes on teeth and dental materials using surface metrology principles."

"Our research consists of investigating the performance of IOS in quantifying tooth wear. Our first goal was to determine the IOS's measurement limitations by using a controlled and standardized wear model in order to reduce measurement errors and comparing it to a highly accurate 3D profilometer. Our aim was to measure wear changes on polished human enamel (depths of 21.8–269.0µm) as well as changes on surfaces with increasing roughness. The datasets were analyzed with Mountains[®] software."

"Our study revealed for the first time that IOS can only detect Sq surface roughness of silicon carbide particle sizes above 68µm. This highlights limitations in measuring short-wavelength surface features compared with profilometry.

In terms of step height detection, an automated lesion localization algorithm revealed the measurement threshold of IOS to be 44.02µm, showing for the first time that enamel lesions can be reliably quantified using automated detection at the sub-50µm level. This is comparable to the level of annual wear of human enamel, estimated to be around 38 µm (Lambrechts et al. 1989).



This finding shows promise for IOS as a diagnostic and research tool for quantifying tooth or material wear."

Above. Measurements (µm) of lesion depth using a non-contact laser profilometer (NCLP) and an intra-oral scanner (IOS).

AUTOMATING ANALYSIS

"The unique functionalities of Mountains® software helped us develop an automated lesion detection and analysis workflow. To investigate the measurement limitations of IOS, measurements of Sq roughness, 3D surface step heights (ISO 5436-1) and XY wear lesion areas were compared with those of the non-contact laser profilometer (NCLP), corroborated by Gaussian skewness (Ssk) and kurtosis (Sku) analysis.





Above. Top. Step height analysis of the automated lesion surface using an intra-oral scanner. **Bottom.** Step height analysis of the same surface using a non-contact laser profilometer.

Firstly, the 3D step height (in µm) of the central lesion was calculated using a predefined selection of the polished enamel reference areas compared to the lesion, based on ISO 5436-1. Using Mountains[®], these predefined areas were programmed to automatically have the same size and location on each NCLP and IOS digital surface."

"Secondly, an automated lesion localization

algorithmwas used to determine the measurement threshold of IOS by localizing and measuring the XY lesion area (in mm²) on each scan. We used the Particle analysis feature to detect surface points of measurement (POM) with Z-amplitudes below the mean plane of the surface heights according to the histogram, on each 3x1mm dataset. The Z-heights of surface points inside a given lesion would be below the mean plane of surface heights in an otherwise planar surface, resulting in lesion localization. The surface area (in mm²) of these points was calculated for each IOS scan and compared to the NCLP."

"Finally, the Gaussian surface height distribution was analyzed on each 3x1mm dataset using surface skewness (Ssk) and kurtosis (Sku) parameters. The surface distribution curve of a nominally flat surface superimposed with a random roughness has a Gaussian distribution symmetrical about the height of the nominal plane which is quantified by Ssk equal to zero and Sku equal to three. In contrast a surface with a central lesion will display negative skewness and kurtosis less than three."



Above. Particle analysis of lesion using an IOS scan (top) and NCPL scan (bottom).



INSTRUMENTS AND SOFTWARE USED

True Definition intra-oral scanner by Midmark, TaiCaan Technologies XYRIS 2000 CL profilometer + MountainsMap® software (Step height study & particle analysis).

READ MORE

The measurement threshold and limitations of an intra-oral scanner on polished human enamel. P. Charalambous, S. O'Toole, T. Bull, D. Bartlett, R. Austin. In: Dental Materials: <u>doi.org/10.1016/j.den-tal.2021.01.006</u>

ACKNOWLEDGEMENTS

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66 ANALYZING DATA FROM A LARGE-SCALE MULTI-INSTRUMENT PROJECT

The Institute of Electronics, Microelectronics and Nanotechnology (IEMN) in France, a research center with large-scale facilities dedicated to micro and nano fabrication processes, recently completed the "Dirac III-V" project investigating ways of producing Dirac electrons (electrons without any mass). This high-level project called for the use of many different fabrication and characterization methods as well as a software program capable of bringing together and processing the different kinds of datasets generated.

MANUFACTURING A HONEYCOMB ARRAY IN AN InGaAs QUANTUM WELL

Molecular beam epitaxy (MBE) was used to grow a semiconductor quantum well, the main challenge being to turn this quantum well into an artificial honeycomb lattice by nanoperforating an array of pores at the limit of conventional lithographic techniques.

For that purpose, block copolymer lithography was used to produce a mask with a periodicity of 36nm. The pores were etched in the mask and into the SiO₂ protecting layer.

After etching, **scanning electron microscopy** (SEM) was used to precisely check and quantify the geometry of the lattice and the degree of disorder which arises from the transfer of the microdomains which exist in the block copolymer film.

The sample was then transferred into another etching machine to transfer the pores created in the mask to the InGaAs layer.

Thus, a lattice of pores was created in the quantum well.



Above. The nanoperforated quantum well imaged using SEM to check the quality of the etching processes and examine the transfer of the grain boundaries of the block copolymer mask into the SiO₂ protecting layer.

MULTI-PHYSICAL SAMPLE CHARACTERIZATION

While the SEM produces high-resolution 2D (x,y) images allowing analysis of the lateral disorder in this two-dimensional structure, **atomic force mi-croscopy (AFM)** was used to validate the etching processes by extracting vertical topographical information.

At this stage of the study, the surface of the sample was oxidized and transferred back to the MBE lab for cleaning and As capping which ensures a protection of the sample against air-exposure. **X-Ray and UV photoelectron spectroscopy** were used to characterize surface chemistry and investigate the bond structure of the sample.



Above. AFM was used to obtain height information on the sample and check the depth of pores.

Left. The Nanoprobe instrument combines several technologies in itself: SEM, STM, optical spectroscopy etc.



The **Nanoprobe**, a unique multi-physical instrument housed at the IEMN combining SEM, STM and spectroscopic techniques, was further used to measure the electrical resistivity of the native quantum well and the nanoperforated quantum well.

"On this sample we wanted to probe the conductivity of a quantum well to ensure that the sample was compatible with lower temperature



Above. Scanning tunneling spectroscopy (STS) data processing was performed using Mountains®.

measurements" explains Maxime Berthe, SPM development engineer, "All the output measurements were analyzed together in one single software; Mountains[®] software allowed us to correlate our results."

Finally, **Scanning tunneling microscopy (STM)**, a surface-sensitive technique, was used to get accurate information about morphology and to resolve the outstanding electronic properties of the nanoperforated quantum well (in scanning tunneling spectroscopy mode).

"Once the measurements were completed and the data acquired, the next step was to extract some useful information and that's where Mountains[®] software is very useful because we can process both topography and spectroscopy data in the same place." reports Nemanja Peric, PhD researcher at IEMN.

RESULTS

"The project was very challenging but rewarding when we were able to show the existence of specific electron states and finally the state we are looking for" concludes Bruno Grandidier, project leader. "Mountains" was key to processing the different nanofabrication steps involving multi-physical analysis and finally to prove the existence of electrons with very light mass."

READ MORE

Engineering a Robust Flat Band in III–V Semiconductor Heterostructures, N. Franchina Vergel et al., Nano Lett. 2021, 21, 1, 680–685, Dec. 2020. doi.org/10.1021/acs.nanolett.0c04268

SEE THE STORY IN VIDEO: bit.ly/3wSQClk

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WHAT ARE MULTI-CHANNEL CUBES?



Version 9 of the Mountains[®] software platform sees the introduction of support for several new data types, including multi-channel cubes. **Christophe Mignot**, Digital Surf CEO, explains the nature of this new kind of "studiable" and highlights the main applications and advantages for users.

FROM 1D TO 3D (CUBES)

The first software program created by Digital Surf back in 1990 was called "DigiProfil"; it enabled the study of profiles, i.e. a variation along one axis, **z** = **f**(**x**).

Shortly afterwards, in 1991, the company also launched software for analyzing surface topography and became one of the pioneers in this field. A second dimension was thus added: **z** = **f** (**x**, **y**)

The two software programs were merged in 1996 and the Mountains[®] platform was born. A countless number of variations have since been released to cover a large number of applications, from profilometry to microscopy.

The next step was inevitable. Instrument manufacturers and users who have multi-instrument analysis laboratories require versatile platforms, in order to switch easily from one type of data to another.

Mountains[®] 9 therefore adds a 3rd dimension making it possible to open, visualize and study tomographic data, where **i** = **f** (**x**, **y**, **z**).

WHY "MULTI-CHANNEL"?

Nanometric objects and organisms are nowadays called upon to perform increasingly technical and intelligent functions in life sciences, mechanics, microelectronics etc. These functions must be observed and characterized. A basic description of relief (topography) is no longer sufficient: we must be able to map the chemical composition of a surface and study its local physical properties.

Used by thousands of engineers and metrologists worldwide for analyzing topography, Mountains[®] has also become over the years a powerful tool for correlative microscopy. The software can manage "multi-channel" (or "multi-layer") images, i.e. those made up of several physical signals. Associating a fluorescence image, conductivity or chemical composition of a sample with a 3D topographic map has become a routine operation.

This "multi-channel" ability applies regardless of dimension: extract a vertical section from a multi-channel image **i1**,**i2**, ..., **i**_N = **f** (**x**, **y**), and you'll obtain a multi-channel profile **i1**,**i2**, ..., **i**_N = **f** (**x**).

Now Mountains[®] allows processing not only of mono-channel cubes, corresponding to the tomographic density of a single material **i** = **f** (**x**, **y**, **z**), but also multi-channel cubes enabling the simultaneous observation and quantification of the density of N mixed materials **i1,i2**, ..., **i**_N = **f** (**x**, **y**, **z**).

FROM SPECTROMETER TO HYPERSPECTRAL CUBE

A multi-channel cube is initially obtained from the measurement of a complete spectrum for each voxel. Each horizontal section of a tomographic cube is considered a hyperspectral image.

FROM HYPERSPECTRAL CUBE TO MULTI-CHANNEL CUBE

However, even if certain instruments such as confocal Raman adequately measure one spectrum per voxel, storing a complete spectrum for each voxel is not very realistic.



Above. Segmentation of a multi-channel cube: for each voxel, the main element (or zero) is retained.

Instead, it is possible to compare this spectrum (as early as possible in the process) with the N reference spectra of the N known materials, the density of which we wish to map in the cube, and retain only the result.

The information is thus reduced to N intensity cubes, one per material analyzed. In other words we consider that we have N channels for each voxel: **i1,i2, ..., i**_N = **f** (**x**, **y**, **z**).

FROM MIXED CUBE TO SEGMENTED CUBE

Each voxel therefore represents the intensity (or abundance) of several mixed materials. The simplest way to represent this mixture is to assign one color per material and display the mixture of colors thus formed (for example a green channel and a red channel mixed together will give a large number of shades from red to green as an indication of the proportion of each material).

Since the density of a material can vary continuously from zero (no material at all) to a maximum value, there is not always a notion of border between matter and void.

However, to make the study of material distribution easier, we may want to segment the cube, in other words artificially make such a border appear between the presence and absence of a material. We can then create grains of material whose number, size, shape, position, density etc. can be studied.

Segmentation consists in choosing, for each voxel, the principal material number and in ignoring the others at one particular point. A significance threshold must be defined below which the density of a material is considered to be zero. And if the intensity of all materials is below the threshold, the voxel is deemed empty.

Each voxel in the cube will only represent either a given material or the void (material number or zero).

The 3D visualization tool provided in Mountains® 9 makes it possible

Right. FIB-SEM series of a helical structure loaded as a multi-channel cube in Mountains® 9.

to blend these two approaches: "mixed" representation and segmented representation. Animations allowing clear visualization of the distribution of each material in the cube can be generated.

THE SPECIFIC CASE OF FIB-SEM

In FIB-SEM (focused ion beam scanning electron microscopy) the ion beam cuts through the sample, layer by layer, while the electron beam compositionally images each layer. EDX/EDS analysis will yield a multi-channel cube as described above. On the other hand, use of a FIB-SEM in BSE mode is different.

Backscattered electron images give us the atomic number of materials observed in the cross section: the larger the nuclei, the more electrons are returned and the lighter the pixels are in the image.

We can thus build single channel cubes (each voxel contains a gray level). The representation in "mixed" mode shows these gray levels.

Each gray level range is representative of a material; the cube can be segmented by designating grayscale ranges and assigning them to a material using a thresholding tool. Once the cube is segmented, it behaves like a multi-channel cube (like EDX).

With multi-channel cubes, Mountains[®] users can now analyze materials in all dimensions. It's also easier to switch from one type of data to another, with numerous visualization and analysis tools available for each type.



REVISION OF ISO 25178-2: WHAT'S COMING?



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Since its publication in 2012, the ISO 25178-2 standard defines parameters and specifications for areal surface texture analysis. The next few months will see the validation of a revised version of the standard. Digital Surf's senior surface metrology expert **François Blateyron** outlines the changes to come.

The ISO 25178-2 standard, published in 2012, was an important milestone in the evolution of surface texture. For the first time, an ISO standard defined a set of areal surface texture parameters. This was significant progress compared to classic profilometry.

Now, a major evolution of that standard is heading for publication, probably by the end of 2021.

One important change brought by this revision is located in the **Feature parameters** chapter, with a corrected definition of open and closed motifs. With the previous definition, it was not possible to properly implement and use these concepts which are useful for tribology.

In the same chapter, a new set of Feature parameters has been added, to allow the characterization of the motifs' shape in the horizontal plane, a clever complement to height, area and slope evaluation. Two other parameters have also been added to quantify the density of pits and the mean pit curvature, the same way they were defined for peaks.

The revised text of the standard was improved and homogenized with the new ISO 21920 standard for profiles, in order to define concepts common to both standards in the same way.

It also allowed the addition of a new parameter, **Ssw** (dominant spatial wavelength) that was defined for profiles. Several adjustments or name changes have been made: the **Sxp** parameter (extreme peak height) is now called **Sdc** (section height difference) and default material ratio values have been updated.

Smr now has its threshold plane referenced from the highest peak instead of the mean plane, as defined for profiles in ISO 4287.

Several parameters of the **Sk** family have been renamed with an additional k suffix, for consistency (**Smrk1**, **Smrk2**, **Sak1**, **Sak2**). This is reflected in Figure 1.



Information			
Filter settings	Unfiltered.		
Parameters	Value	Unit	
Sk	4.927010	μm	
Spk	1.098019	μm	
Svk	2.210534	μm	
Smrk1	7.183146	%	
Smrk2	88.837048	%	
Sak1	0.039436	µm³/µm²	
Sak2	0.123380	µm³/µm²	

Figure 1. Renamed Sk parameters.

The chapter on fractal parameters was rewritten for clarity and some parameters have also been renamed for consistency with the other **Field parameters**.

New annexes have been added, in particular Annex G that provides a useful analysis workflow (see Figure 2).



Figure 2. The specification analysis workflow, added in Annex G.

The Spring 2021 meetings of ISO TC213/WG16 finalized the work on four documents, ISO 25178-2 and ISO 21920-1, -2 and -3.

These documents are now ready to be sent to FDIS vote, the last formal vote before publication. They should then be sent to the ISO secretariat in Geneva by Fall for publication before Christmas. After that, the next task will be to start the revision of ISO 25178-3 to reflect the changes brought by the new revision of Part 2. The active participation of Digital Surf in international standardization allows us to bring our experienced vision as a software editor to the definition of new tools and concepts, and makes it possible to develop these new tools in MountainsMap[®] so that early users can experiment and generate feedback.

MountainsMap[®] version 9.0, published last June, already incorporates the changes made in the revision of ISO 25178-2. It also includes the new parameters of ISO 21920.

ADDITIONAL RESSOURCES

- MountainsMap® 9.0 features: www.digitalsurf.com/software-solutions/profilometry/
- ▶ ISO/FDIS 25178-2: <u>www.iso.org/standard/74591.html</u>
- Proposed corrections for the ISO 25178 standard series on areal surface texture. Blateyron, François. (2015). www.researchgate.net/publication/279185907_Proposed_corrections_for_the_ISO_25178_standard_series_ on_areal_surface_texture
- Surface Metrology Guide: <u>www.digitalsurf.com/guide</u>

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PARTNERSHIP NEWS

COVALENT METROLOGY PARTNERS WITH DIGITAL SURF TO BRING CUTTING-EDGE ANALYTICAL SOLUTIONS TO INSTRUMENT USERS

Covalent Metrology and Digital Surf will collaborate to advance software-enabled analysis, data visualization and more for research and industry.

Covalent Metrology, a leading analytical services provider offering one of the largest portfolios of characterization techniques in North America, is working with Digital Surf, a global leader in analytical software development, to offer software tools for data processing and visualization to its range of clients. The collaboration will also allow both companies to pool customer insights in the aim of providing better services and more powerful analysis tools.

Together, the two organizations possess unmatched experience and breadth of understanding in the analytical services business. Digital Surf has partnered with world renown surface profiler and microscope manufacturers for more than 30 years, supplying them with software tools that accompany their instruments to empower robust imaging analysis. To complement Digital Surf's extensive software expertise, Covalent Metrology brings deep and diverse metrology and material characterization experience across an expanding portfolio of cutting-edge instruments and industry application areas. Active feedback and collaboration between the two organizations will fuel faster development of more effective solutions for analytical software and services.

"This partnership will help bring tailored, automated analytical software solutions for a wide range of techniques and applications," says



Christophe Mignot, CEO of Digital Surf, "We are also excited to work with the Covalent team to identify new features that could help scientists and engineers access improved analytical insight and guide more effective decision making."

Digital Surf's Mountains® analysis software package, a recognized industry-standard for surface characterization, recently deployed its 9th version and now supports specialized solutions for several imaging techniques as well as multi-instrument data confluence. Covalent is implementing Mountains® 9 software with its scanning probe microscopy (SPM) team and will soon expand the software across other advanced imaging analysis groups.

"We are thrilled to partner with Digital Surf and are excited about the innovative software and service enhancements the relationship will inspire. Powerful analytical software is absolutely necessary for engineers and scientists to produce the most impactful results possible. It complements cutting-edge instrumentation and expertise with the tools needed to transform raw data into meaningful conclusions," says Craig Hunter, Chief Executive Officer of Covalent, "By expanding the use of Mountains® analysis software alongside our range of instrument technologies, we can help clients access substantially deeper insights."

As the Mountains[®] 9 suite deploys across select Covalent groups, clients will benefit from more robust statistical analysis, presentation-ready reports, and advanced visualization features. Mountains[®] 9 enables efficient display, manipulation and study of 2D images, profiles, 3D surface topographies including freeform surfaces, spectroscopic maps and more. These can then be investigated with tools that isolate features of interest and extract quantitative measurements of key properties. For customer projects encompassing multiple characterization methods, data can be automatically aggregated and correlated between instruments and techniques.



WHAT'S HOT ONLINE



POPULAR ON FACEBOOK

We threw it way back with an old-timey image of one of Mountains® software predecessors, DigiSurface. Can we please take a moment to appreciate that squared mouse?: <u>bit.ly/2VDsIgR</u>





Check out our channel for tutorial videos on surface analysis and SEM & SPM image analysis, with Mountains[®] software!

bit.ly/2U2I2za



LOVED ON INSTAGRAM

The Mountains[®] team was thrilled to release version 9. To celebrate the occasion we designed these beautiful coasters using surfaces analyzed with Mountains[®] software. Get a closer look at each one of them: <u>bit.ly/2U0W7Ru</u>



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