Multiscale and Correlative Techniques for Surface Corrosion Analysis

ZEISS

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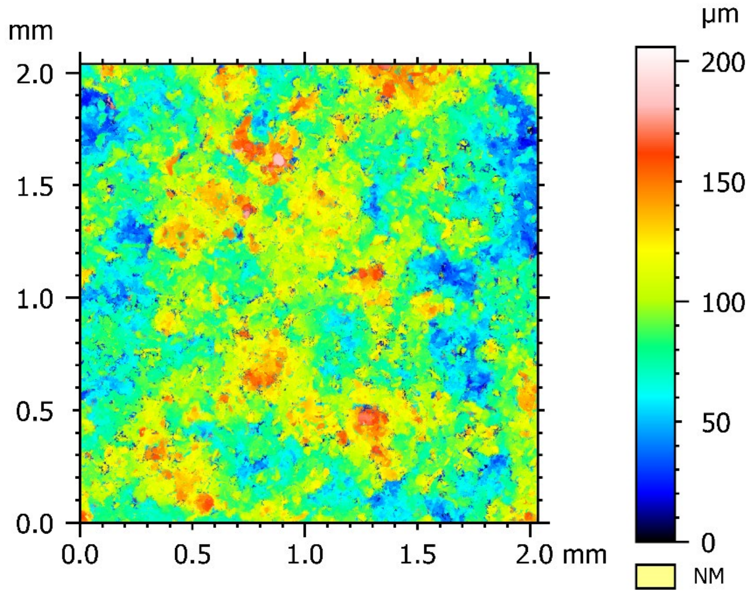
SFEA SMTA

Multiscale and Correlative Techniques for Surface Corrosion Analysis

Abstract: The strength of a bond between a coating and the substrate is significantly affected by factors such as roughness and cleanliness. The ability to non-destructively characterize a surface and understand its topography and roughness is a powerful tool for understanding many aspects of surface behavior; including Corrosion.

Thermal sprayed Aluminum

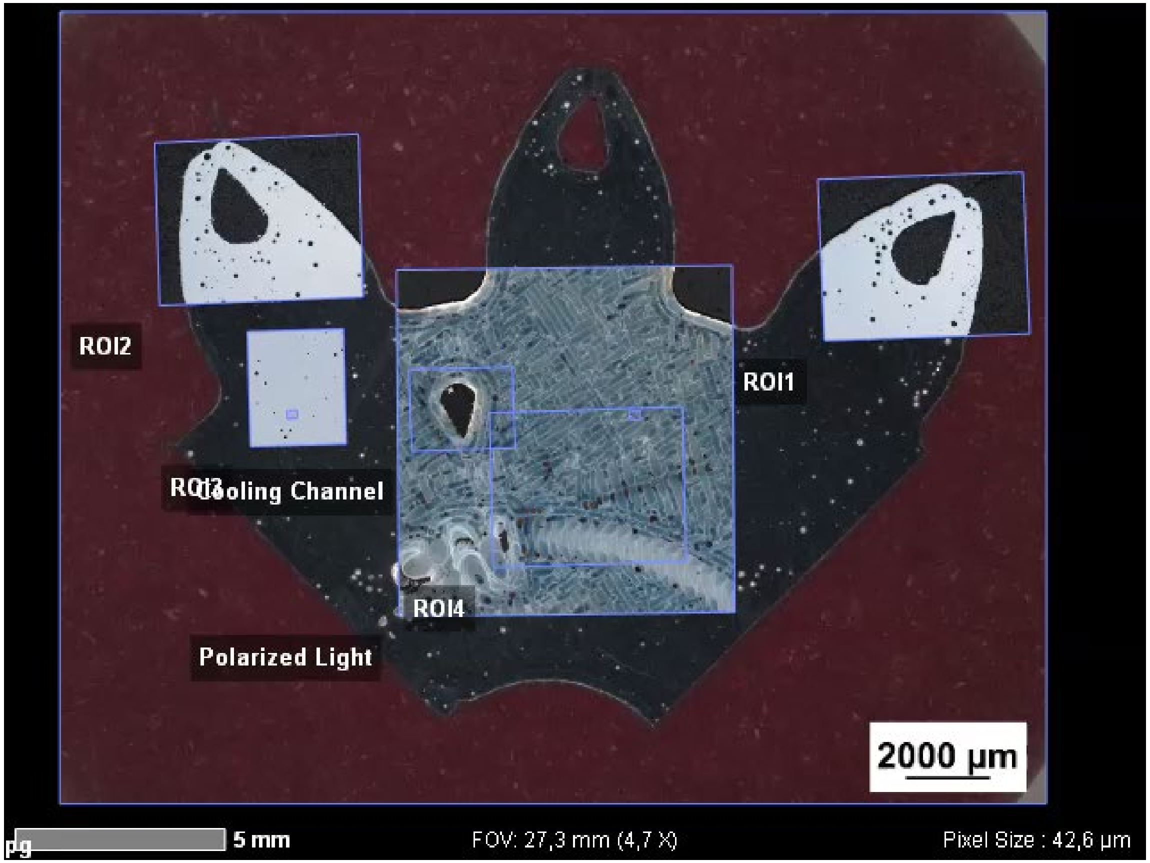


ISO 25178		
Height Parameters		
Sq	0.658	µm
Ssk	-0.091	
Sku	10.247	
Sp	7.664	µm
Sv	10.288	µm
Sz	17.952	µm
Sa	0.362	µm
Spatial Parameters		
Sal	0.108	µm

ISO 4287			
Amplitude parameters - Roughness profile			
Rp	2.65	µm	*Gaussian filter, 0.25 mm*
Rv	3.06	µm	*Gaussian filter, 0.25 mm*
Rz	5.71	µm	*Gaussian filter, 0.25 mm*
Rc	1.50	µm	*Gaussian filter, 0.25 mm, ISO 4287 w/o amendmen...*
Rt	5.71	µm	*Gaussian filter, 0.25 mm*
Ra	0.273	µm	*Gaussian filter, 0.25 mm*
Rq	0.461	µm	*Gaussian filter, 0.25 mm*
Rsk	-0.24		*Gaussian filter, 0.25 mm*
Rku	9.80		*Gaussian filter, 0.25 mm*
Material ratio parameters - Roughness profile			
Rmr	0.445	%	*c = 1 µm under the highest peak, Gaussian filter, 0....*
Rdc	0.450	µm	*p = 20%, q = 80%, Gaussian filter, 0.25 mm*

Multiscale and Correlative Techniques for Surface Corrosion Analysis

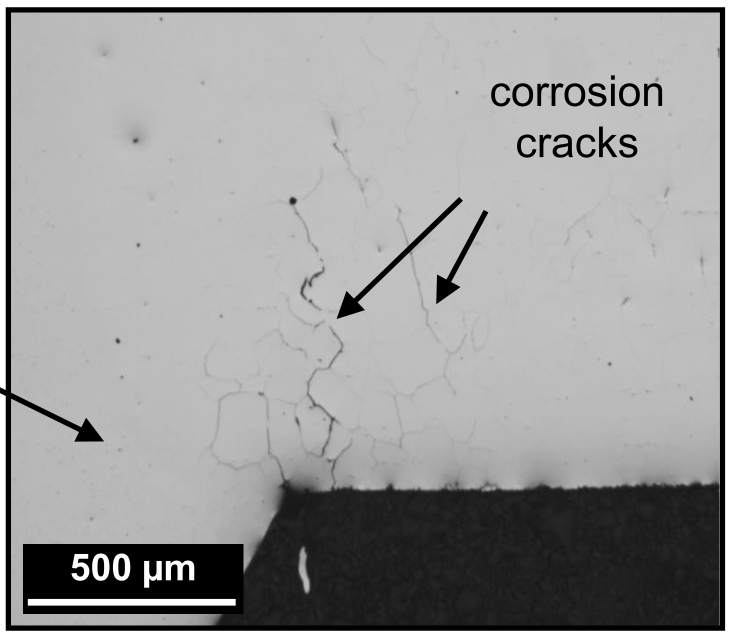
Goal: To look at multi-modal microscopy techniques as tools capable of providing a vast array of analytical data in a non-destructive manner, and show how this can be accomplished in integrated and connected solutions using both hardware and software.



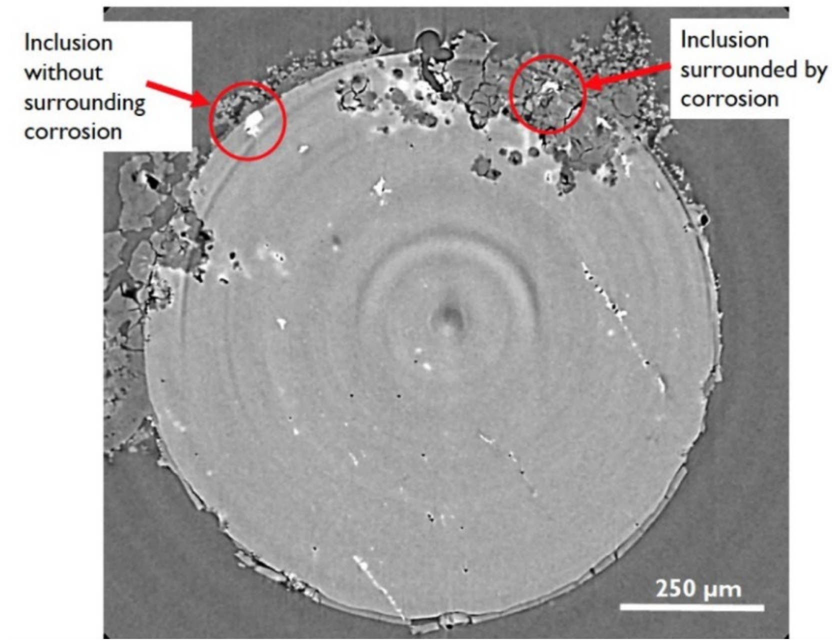
Multiscale and Correlative Techniques for Surface Corrosion Analysis

What does Corrosion look like?

Emphasis will be on highlighting work that has been done using advanced Light and Confocal, Scanning Electron, and X-Ray Microscopy solutions for surface analysis.



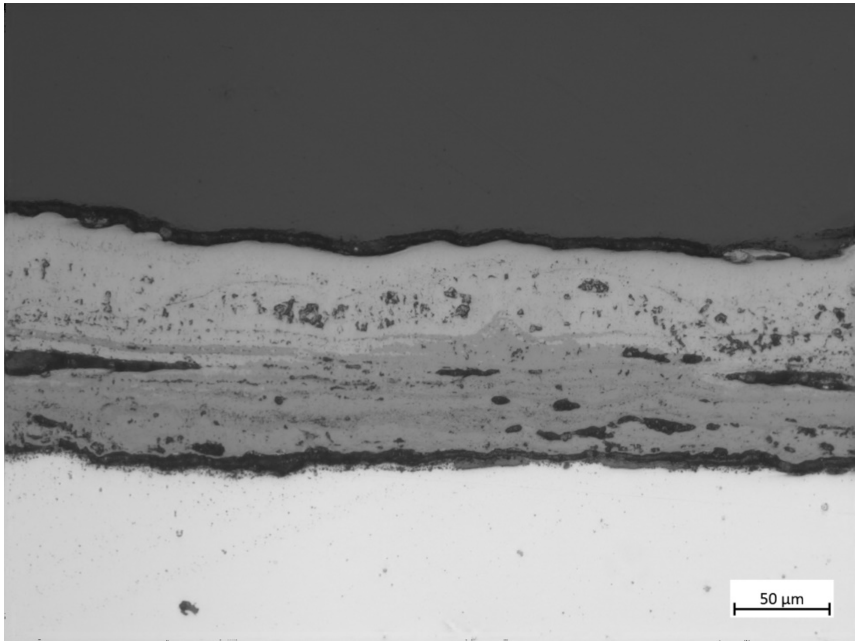
SEM – Chromium Nickel Steel



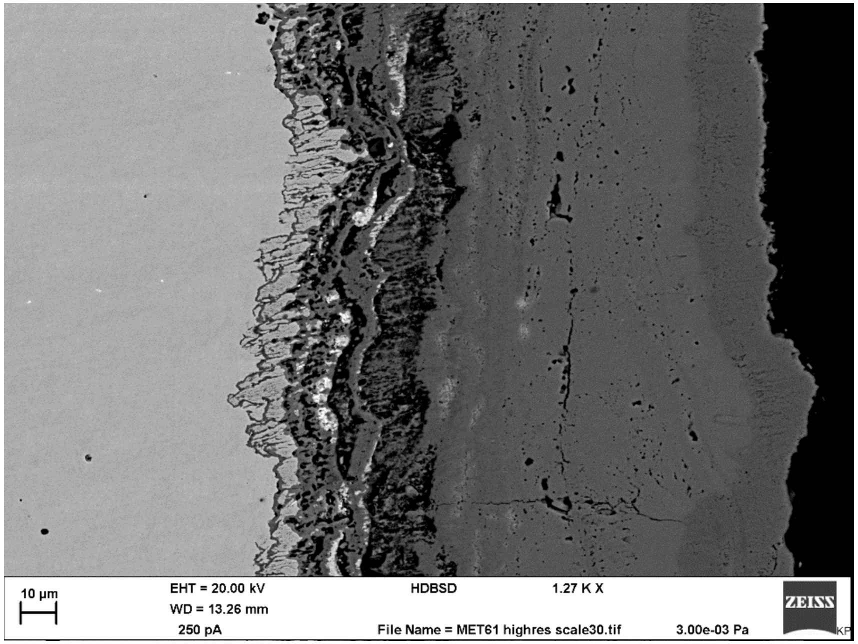
XRM – Aluminum 7475 alloy

Multiscale and Correlative Techniques for Surface Corrosion Analysis

What does Corrosion look like?



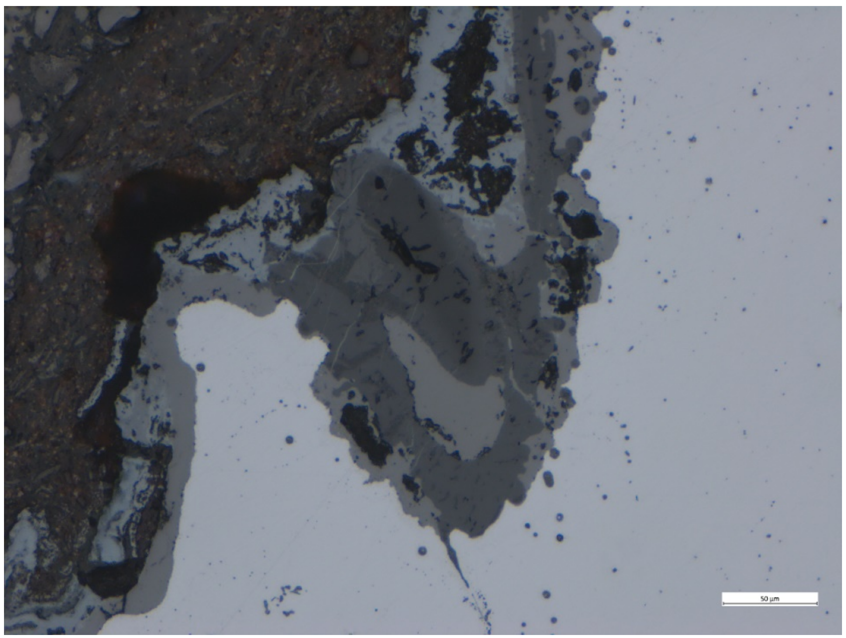
LM – Chromium Steel



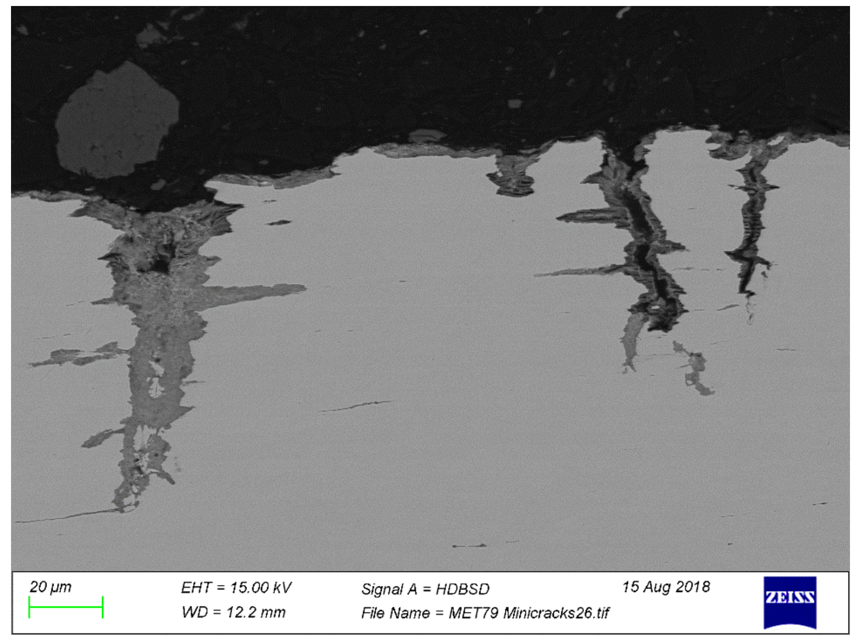
SEM – Chromium Steel

Multiscale and Correlative Techniques for Surface Corrosion Analysis

What does Corrosion look like?



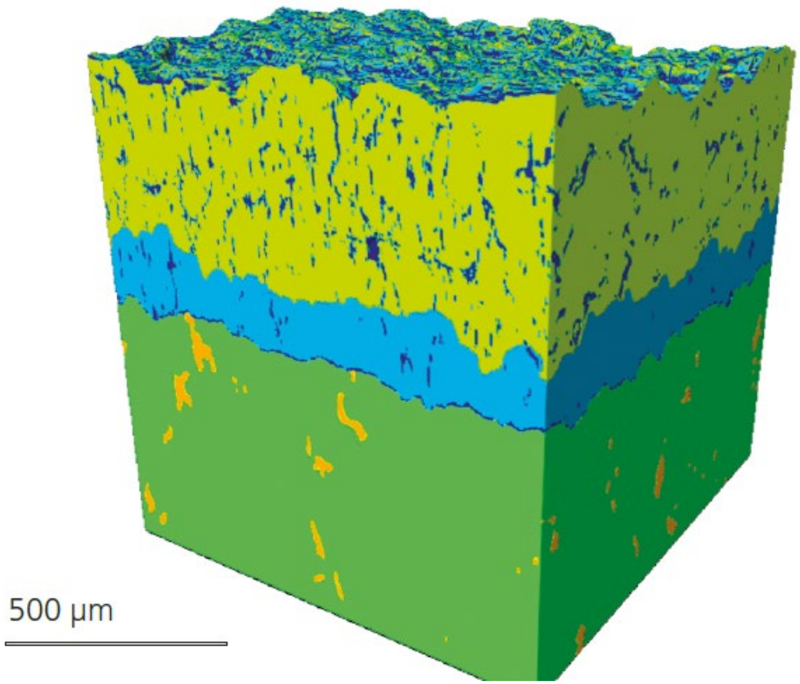
SEM – Corroded Steel

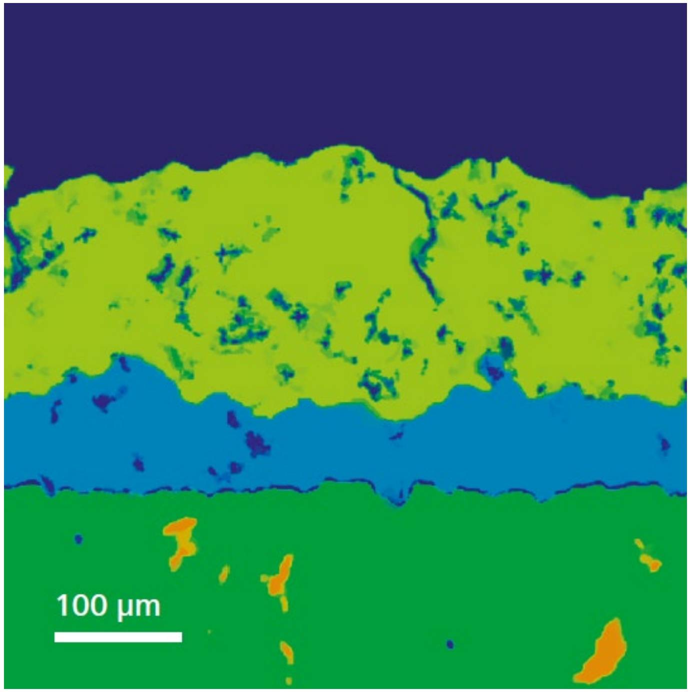


SEM – Cracks in Carbon Steel

Multiscale and Correlative Techniques for Surface Corrosion Analysis

What does Corrosion look like?



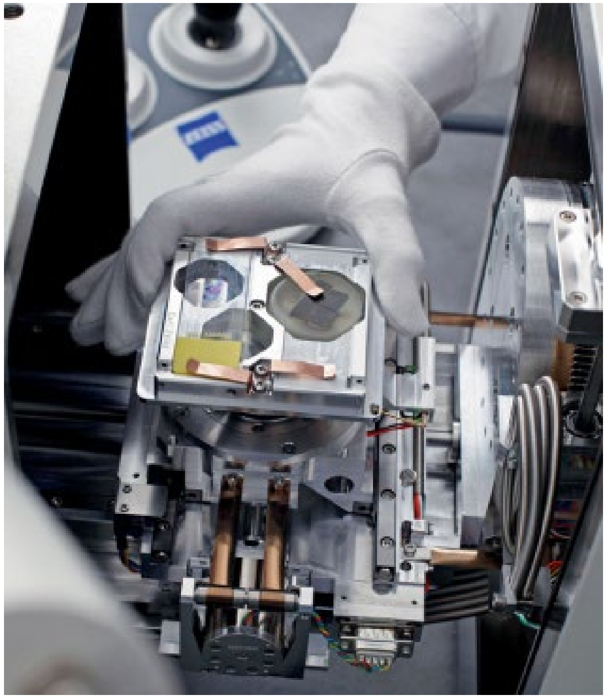


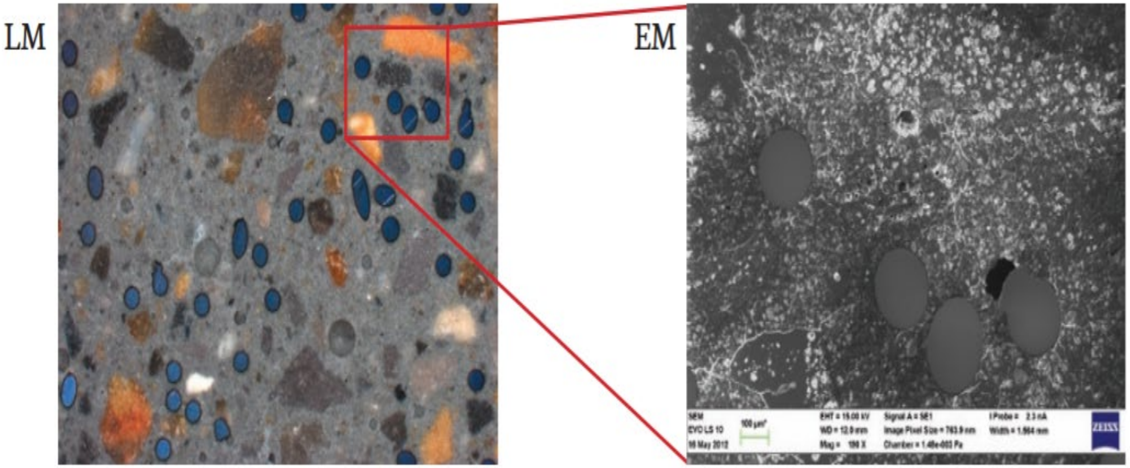
XRM – Thermal Barrier Coating (Nickel alloy base, Aluminum oxide bond coat, yttria-stabilized zirconia (YSZ) top coat

Multiscale and Correlative Techniques for Surface Corrosion Analysis

How do I get images like these?

A coordinated workflow is shown as essential in complex multiscale experiments using Light, Electron, and X-ray microscopy solutions.





Overview of Solutions

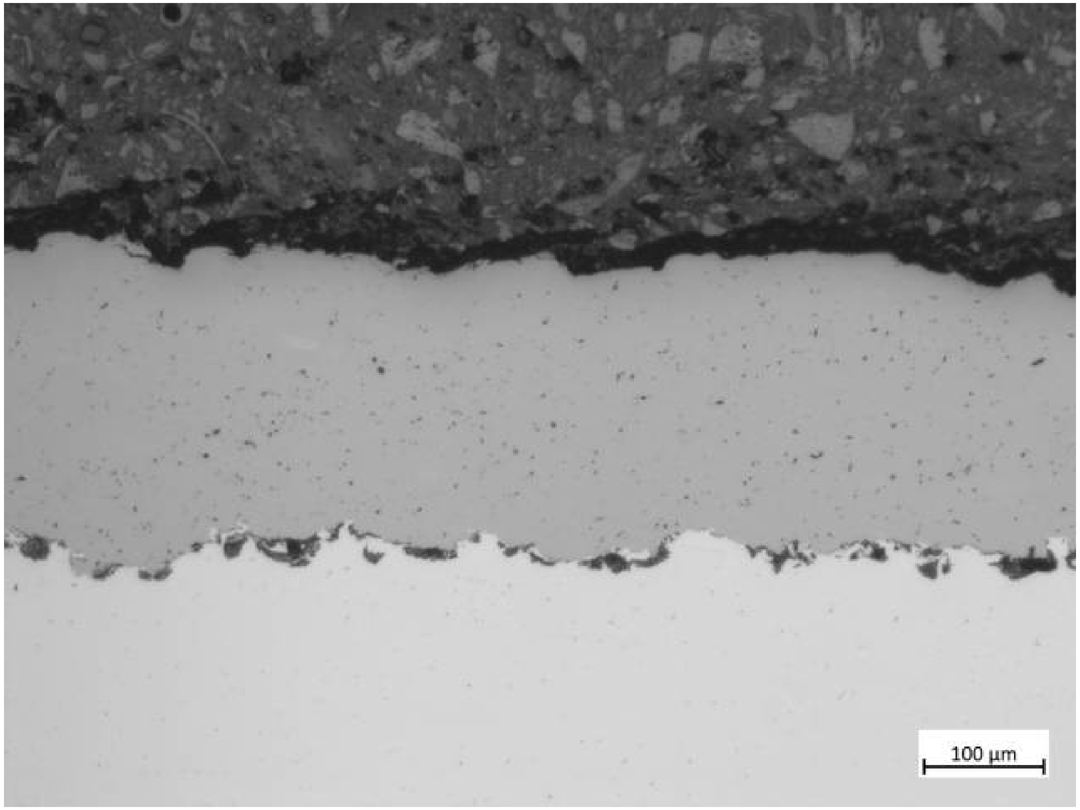
LM – Light Microscopy

CM – Confocal Microscopy

EM – Electron Microscopy (aka Scanning Electron Microscopy)

XRM – X-ray Microscopy/MicroCT

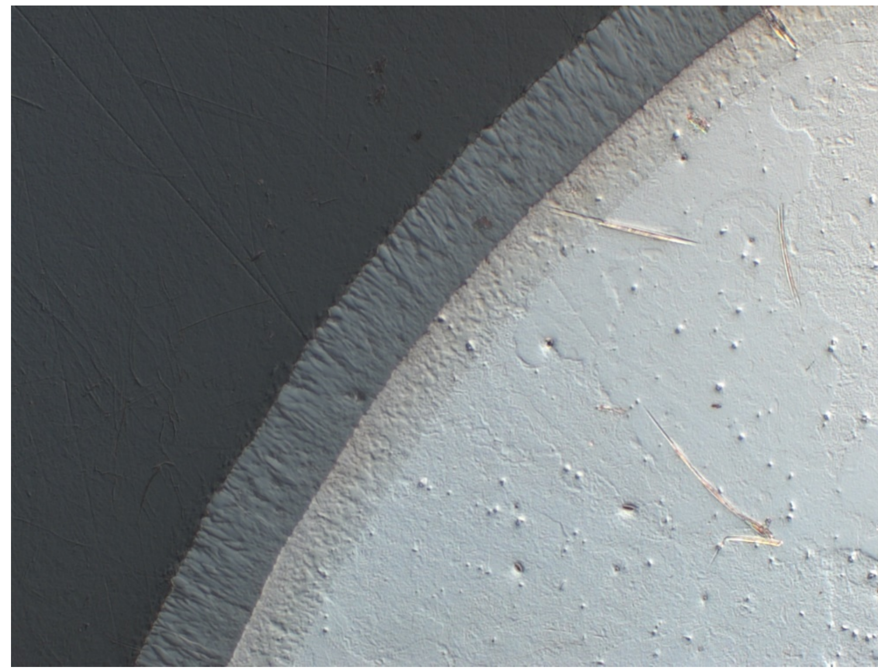
Tantalum coating on Stainless Steel
Light Microscopy



Thermal Spray Coating

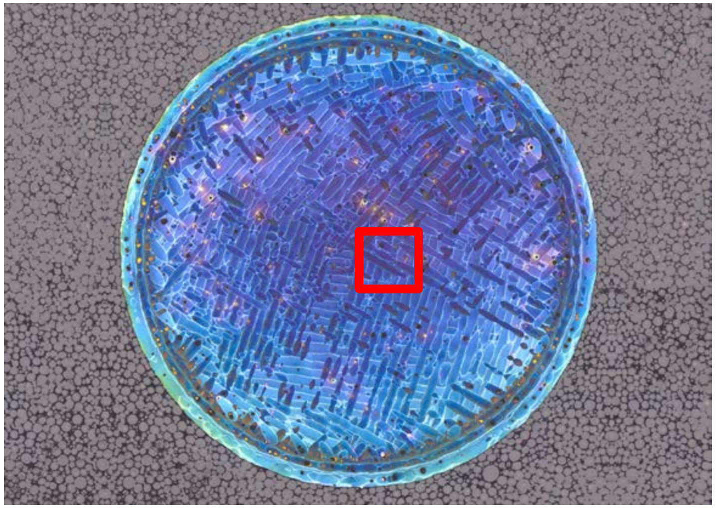
Light Microscopy using Polarized and C-DIC techniques

LM - Polarized light

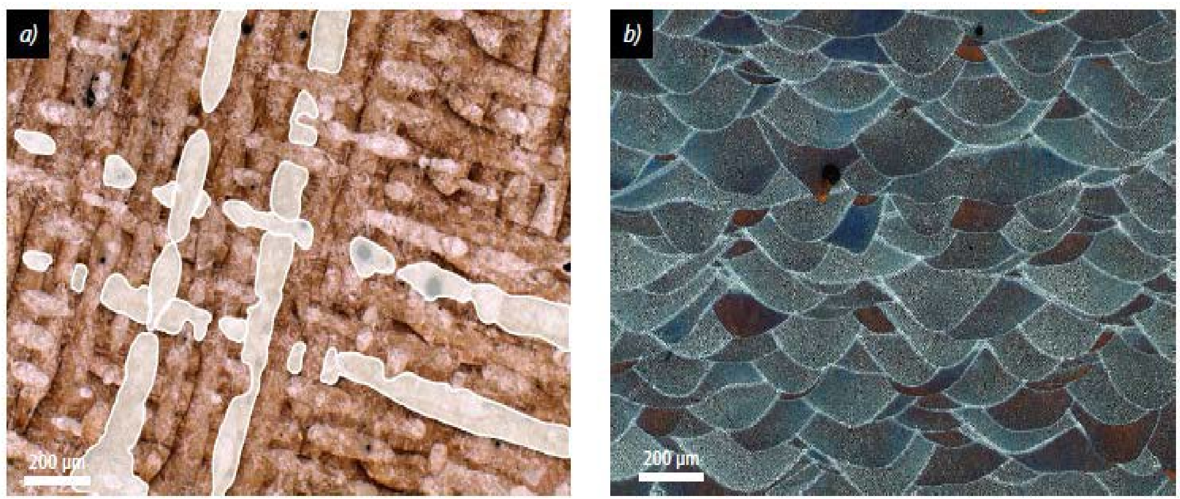


LM – Circular Differential Interference Contrast

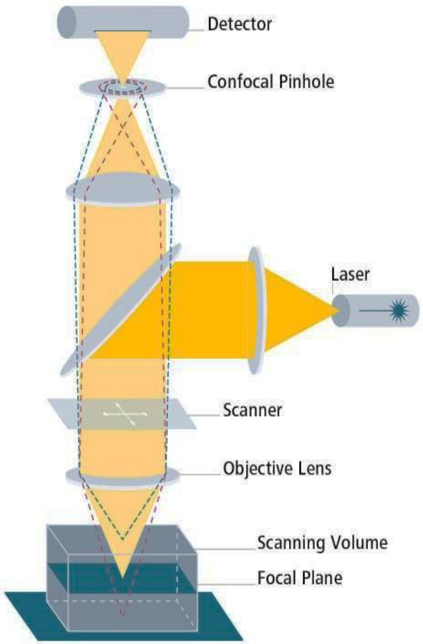
Laser sintered Powdered Metal
Light Microscopy



LM – Cross-section Polarized light



Confocal Microscopy
Overview of Laser Scanning Microscope



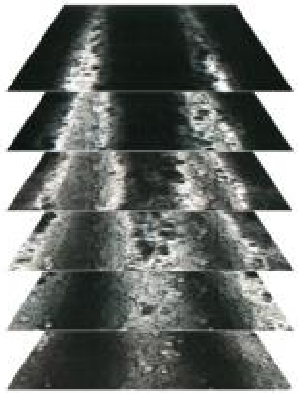
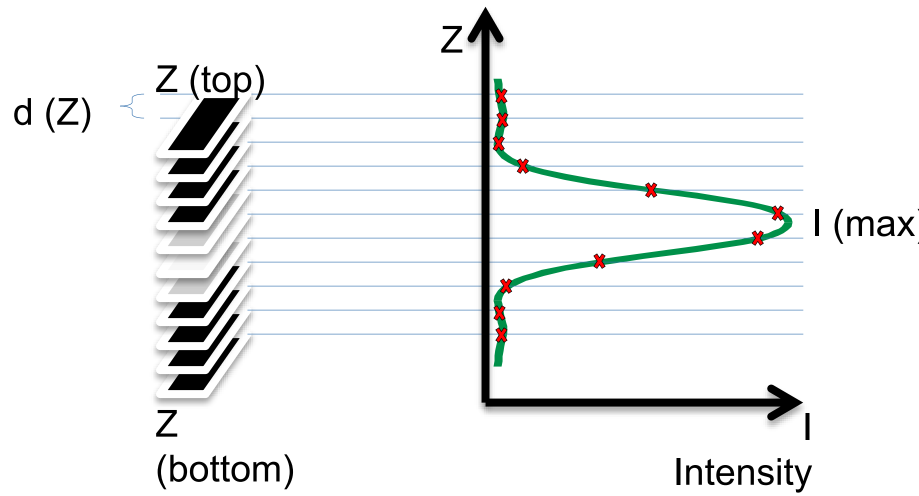
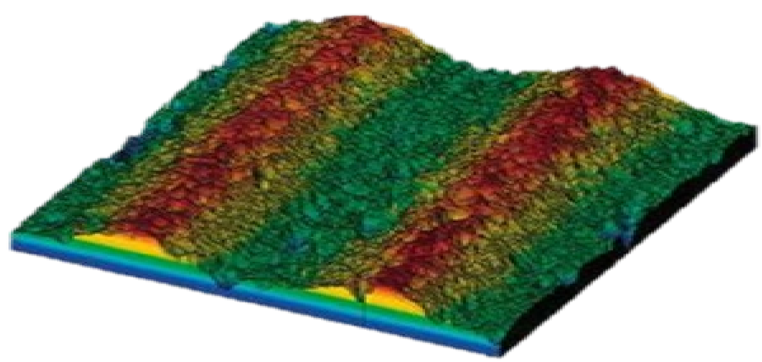


Image stack

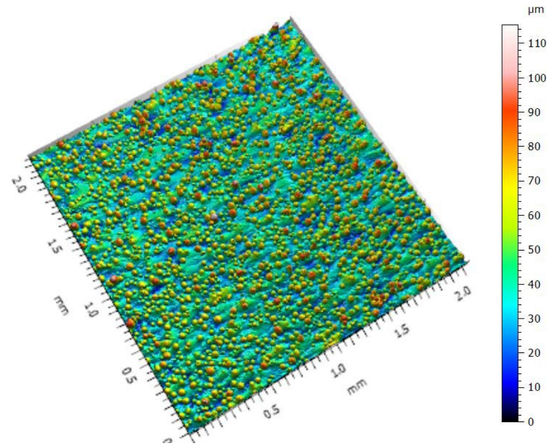




Height map

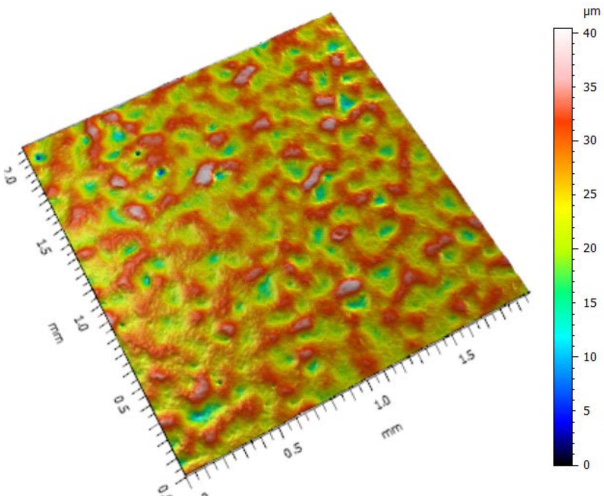
Initial Surface Roughness Evaluation of Ti-6Al-4V
Grit Blasting

- Confocal microscopy shows level of roughness



As built

ISO 25178		
Height Parameters		
Sq	15.1	µm
Ssk	0.689	
Sku	3.15	
Sp	70.3	µm
Sv	45.1	µm
Sz	115	µm
Sa	12.2	µm



After Grit blasting

ISO 25178		
Height Parameters		
Sq	3.87	µm
Ssk	-0.228	
Sku	3.30	
Sp	14.2	µm
Sv	26.3	µm
Sz	40.5	µm
Sa	3.07	µm

Electron Microscopy
Scanning Electron Microscope (SEM)

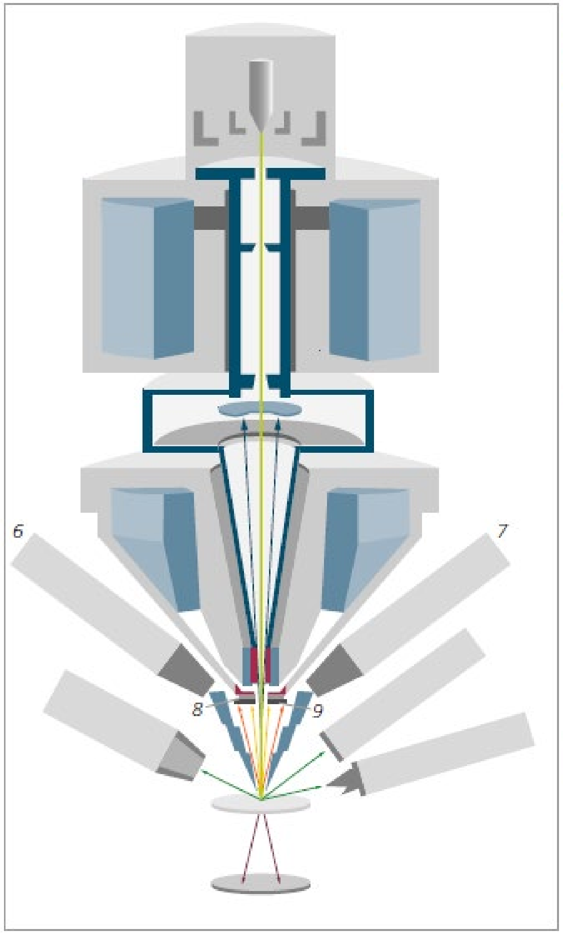
1) SEM is an essential tool for identifying, locating and characterizing corroded regions, elemental depletion, and corrosion products.

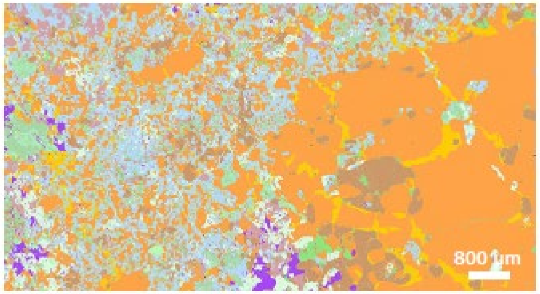
2) Particularly for parts that are exposed to high temperature or unusual chemical environments in the course of their normal performance.

3) Assessment of cross sections by SEM allows a whole new mode of imaging: grain boundaries which are not easily visible under the light microscope may show under SEM, with corroded/depleted layers likely revealed as well.

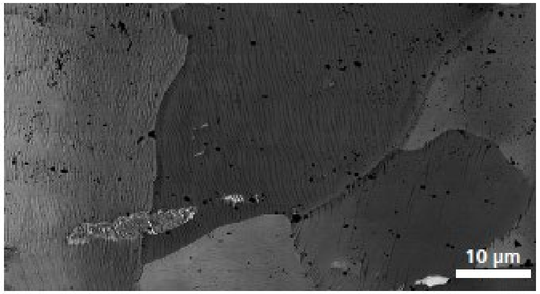
4) Routine assessment of surfaces of welds, brazes, joints, and coatings is also made stronger by SEM - particularly because EDS (Energy Dispersive X-ray Spectroscopy) gives information about elemental dilution and any unusual variations in composition.

Scanning Electron Microscopy
Overview

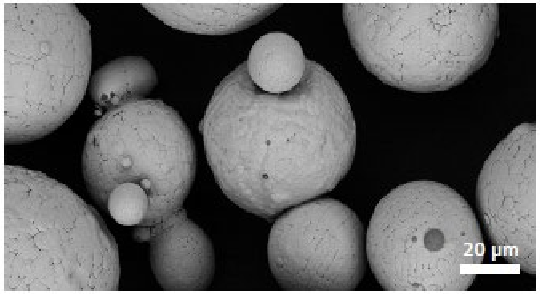




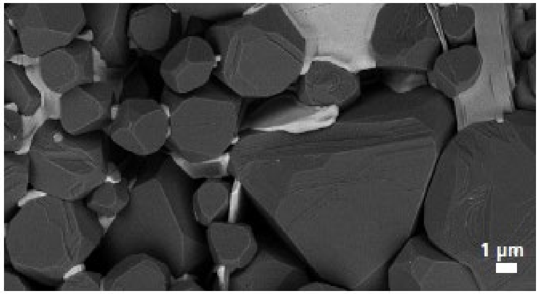
6 / 7 Advanced EDS Detection
Advanced EDS analysis geometry of 8.5 mm working distance and 35° take-off angle for delivering data at twice the speed or half the probe current, Sample: courtesy of University of Leicester.



8 AsB Detector
Angular selective BSE detector for crystallographic and channeling contrast imaging of metals and minerals.



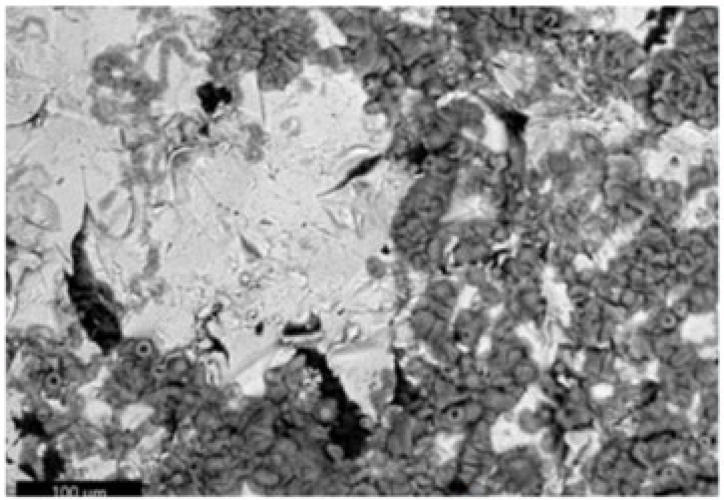
9 HDBSD
High definition BSE detector for excellent low kV compositional imaging of all samples in all vacuum modes.



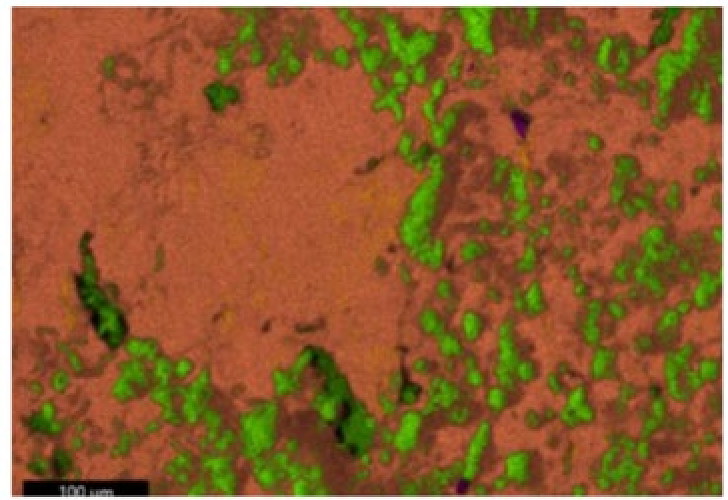
9 BSD4*
Four parallel outputs of the BSE detector for real-time 3D imaging and surface metrology. Example of a compositional

Corroded Mild Steel

Electron Microscopy using BSE and EDS



Backscattered electron image of corroded mild steel, revealing backscattered electron densities that point to compositional heterogeneities as a result of corrosion



Iron (red) and Oxygen (green) elemental map confirming the location of oxidized regions on the sample

EM – BSE (Back Scattered Electron) image

EM – EDS/EDX (Energy Dispersive X-ray Spectroscopy) image

Organic Coating Corrosion

Electron Microscopy using Variable Pressure and Voltage

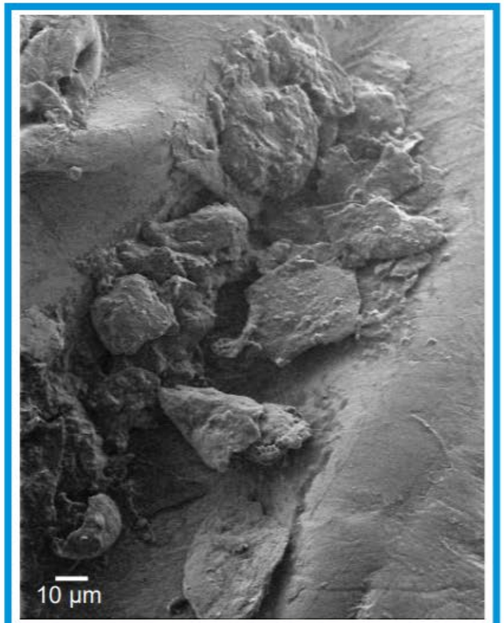


Image @ 3 kV
Signal A = C2D
Extended Pressure Mode
Chamber = 40 Pa

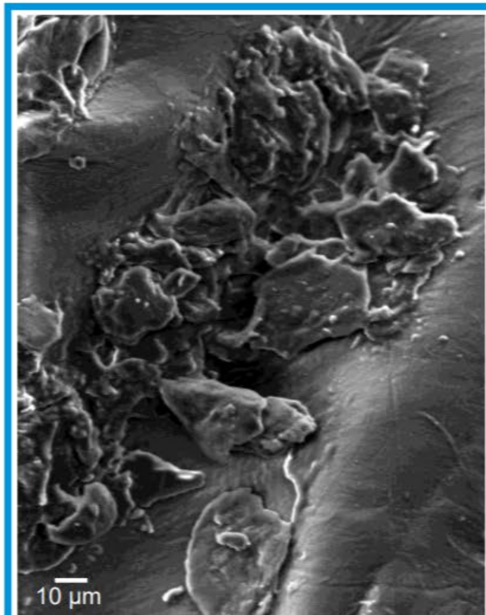


Image @ 10 kV
Signal A = C2D
Extended Pressure Mode
Chamber = 50 Pa

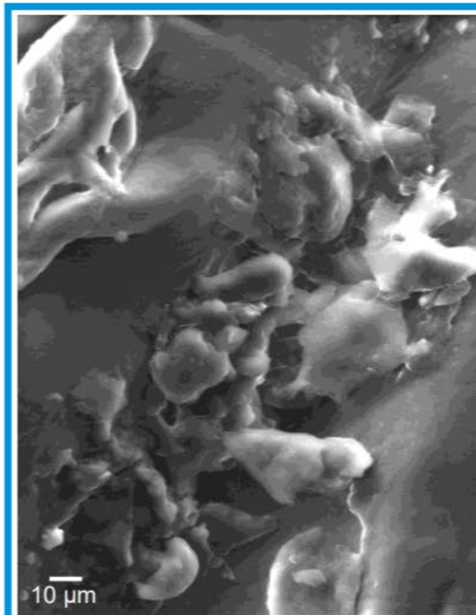


Image @ 20 kV
Signal A = C2D
Extended Pressure Mode
Chamber = 60 Pa

Surface detail of a GORE-TEX organic coating on a copper guitar string, imaged at 5, 10 and 20 kV. Note the progressive “edginess” of surface detail at lower primary beam acceleration voltages — the sweet spot for LaB_6 operation.

X-ray Microscopy

1) X-ray microscopy (XRM), a nondestructive 3D imaging technique, is used to understand the time-dependent evolution and overall extent of corrosion damage in the surface and sub-surface regions.

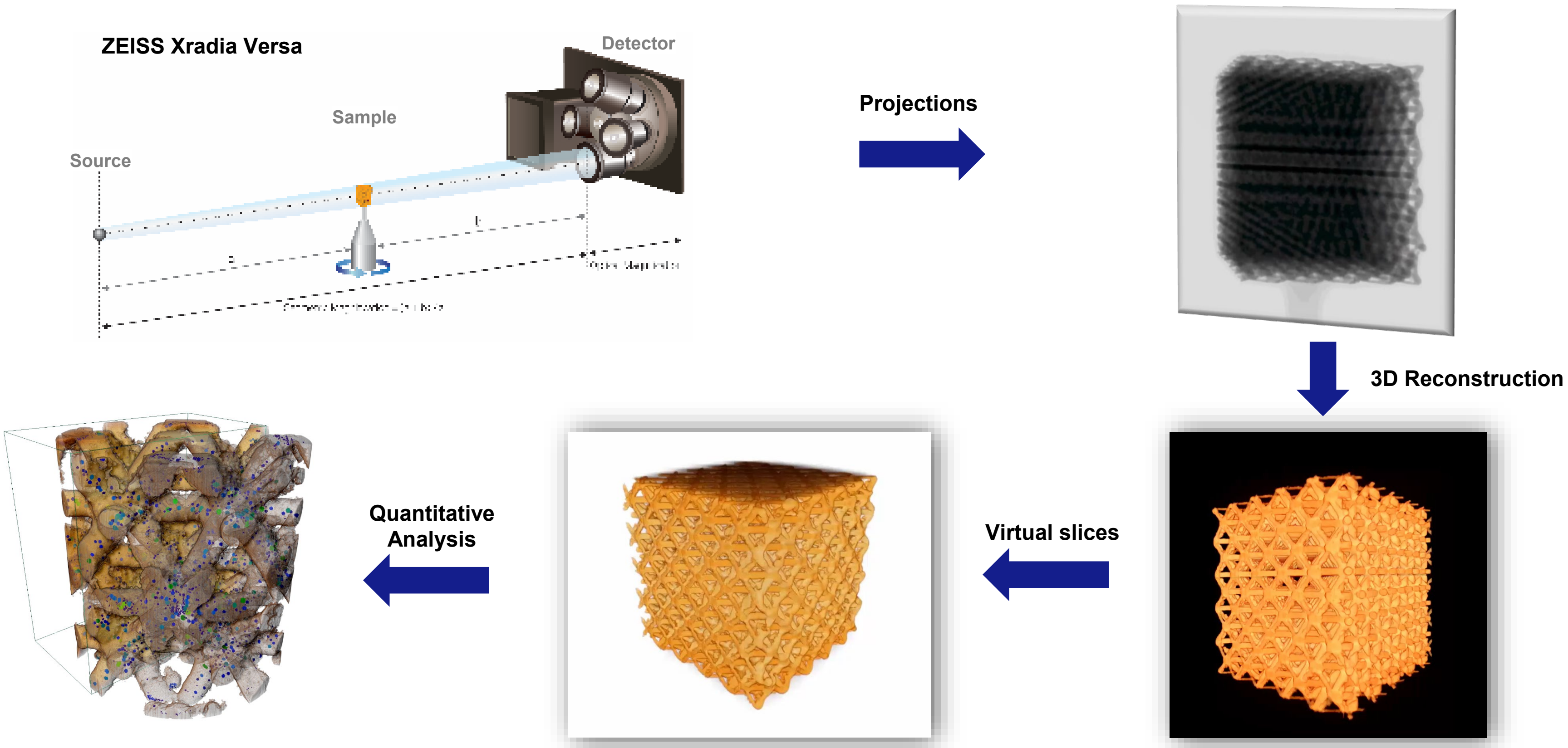
2) Probing the internal structure without the need for complex sample preparation and physical sectioning techniques

3) Moving from sub-micron XRM to nanoscale XRM, finally to FIB (Focused Ion Beam) - SEM

4) Utilizes 3D imaging techniques in a coordinated Correlative workflow (via hardware and software) at the micro and nanoscale to investigate corrosion damage

Tomography in 3D X-ray Microscopy

How it works – Inconel 3D Printed lattice

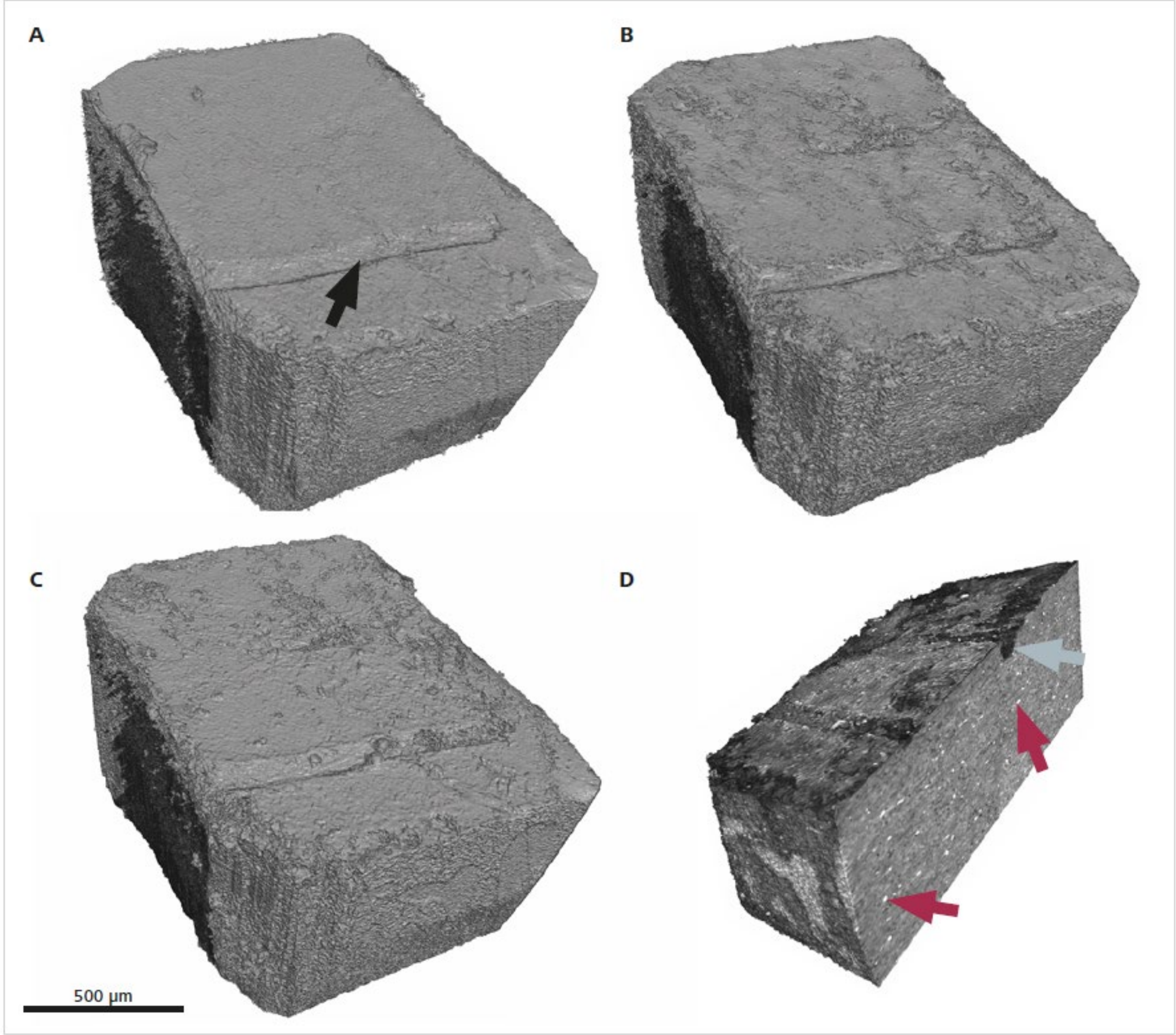


Multiscale Corrosion Damage in Magnesium Alloy

In situ - Correlative Workflow using XRM and EM

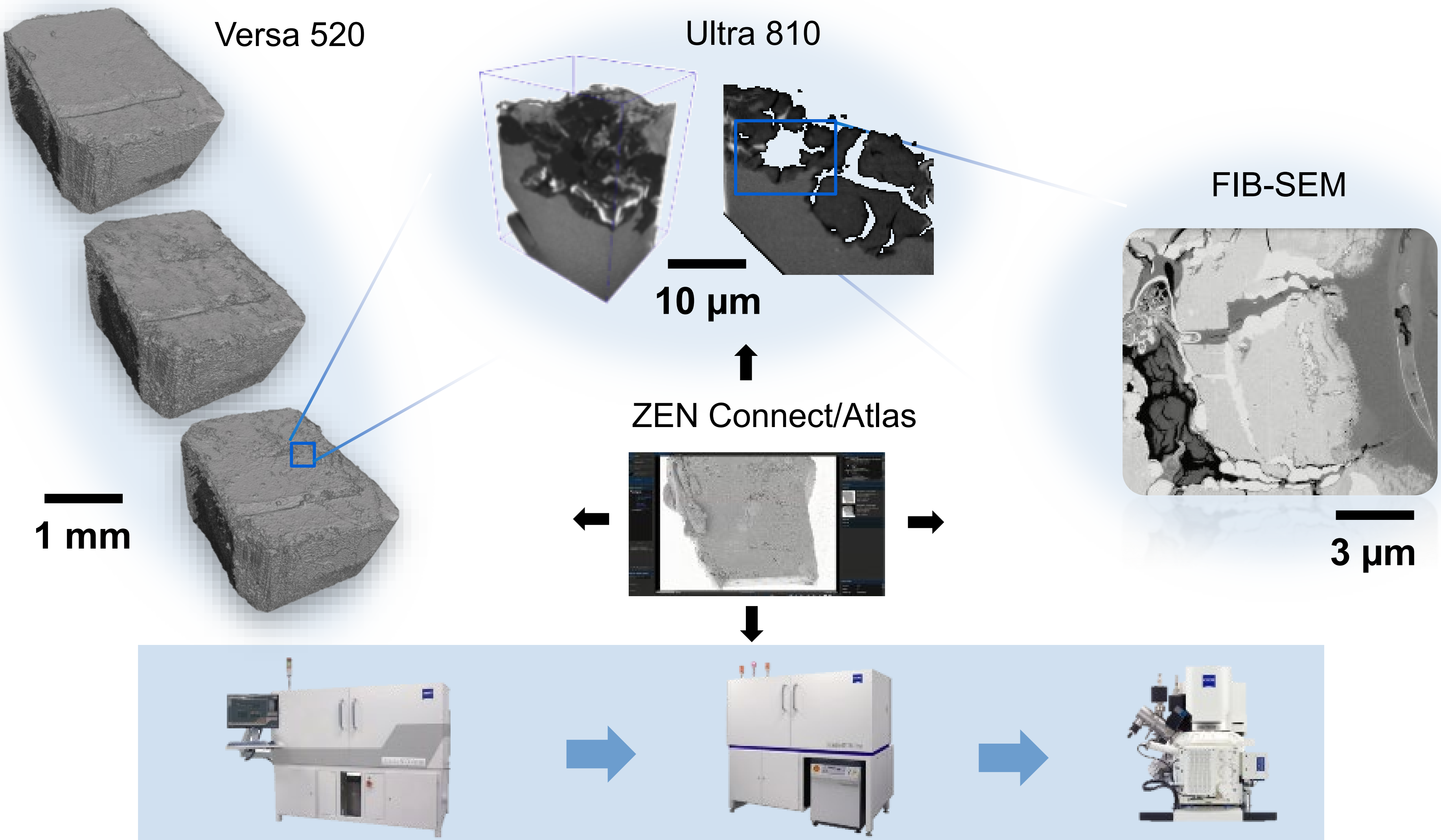
2 hrs

8 hrs



14 hrs

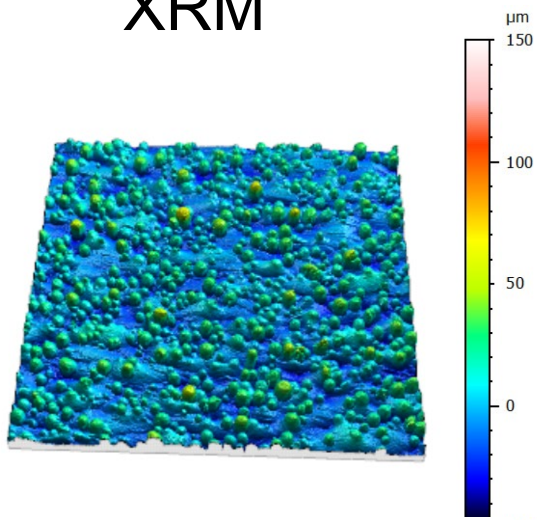
Multiscale Corrosion Damage in Magnesium Alloy
Correlative Workflow using XRM and EM



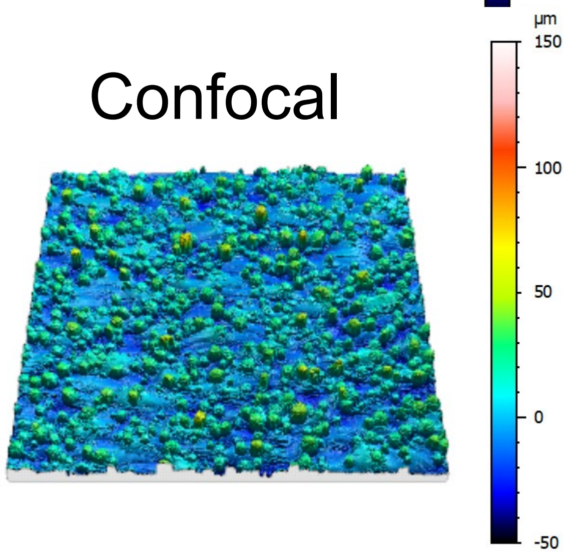
Surface Roughness Evaluation

XRM vs Confocal – Sa Comparison

XRM



Confocal

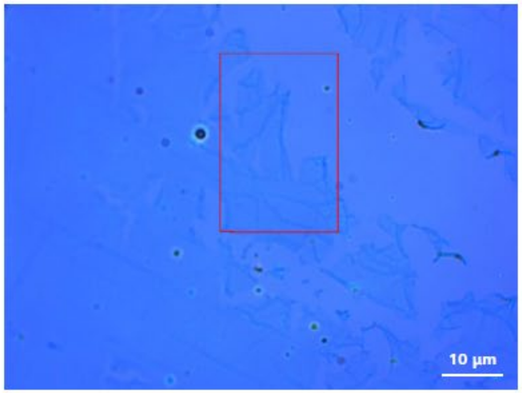


Sa results between XRM and Confocal are very favorable

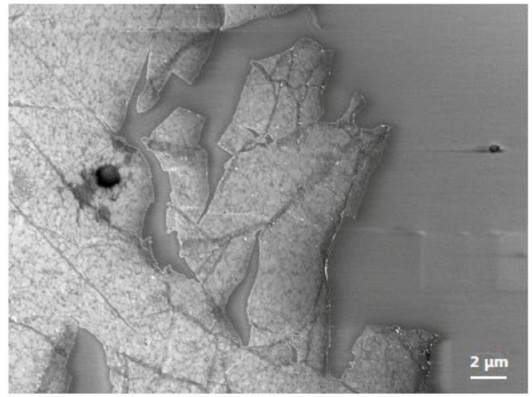
ISO 25178			
Height Parameters			
	520 Versa	LSM 800	
Sq	15.1	14.8	µm
Ssk	0.700	0.776	
Sku	3.11	3.29	µm
Sp	66.9	71.3	µm
Sv	40.7	86.9	µm
Sz	108	158	µm
Sa	12.2	12.0	µm

Graphene and Green Arsenic
Correlative Microscopy from LM to EM to RAMAN

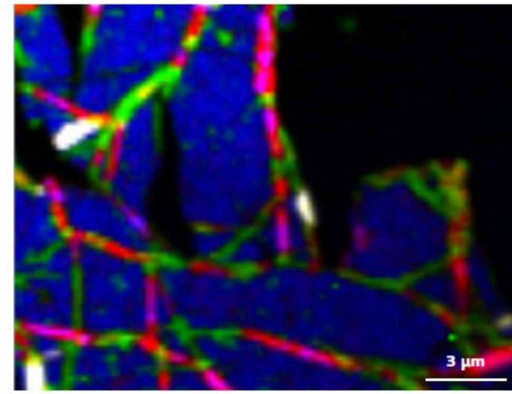
Chemical Vapor Deposition (CVD) Graphene



Light Microscope, transmitted light

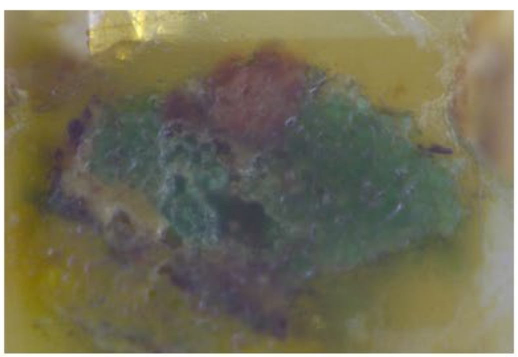


Scanning Electron Microscope, Inlens detector

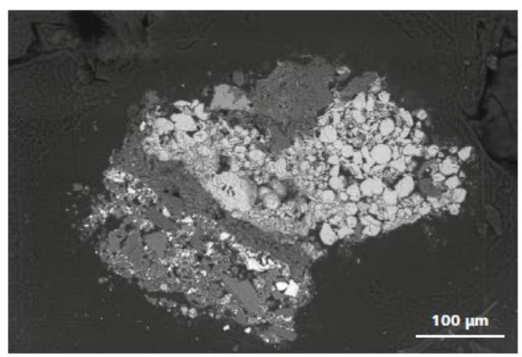


Light Microscope, Raman image

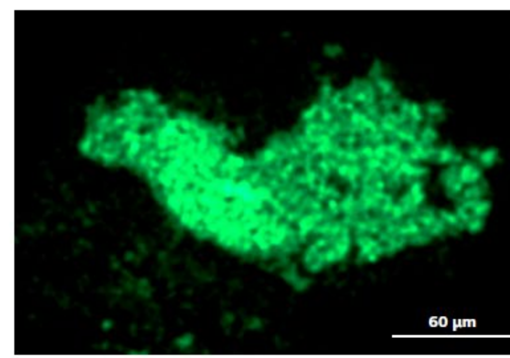
Green Arsenic Particle



Light Microscope, reflected light



Scanning Electron Microscope, BSD detector



Scanning Electron Microscope, EDX

Multiscale and Correlative Techniques for Surface Corrosion Analysis

Summary

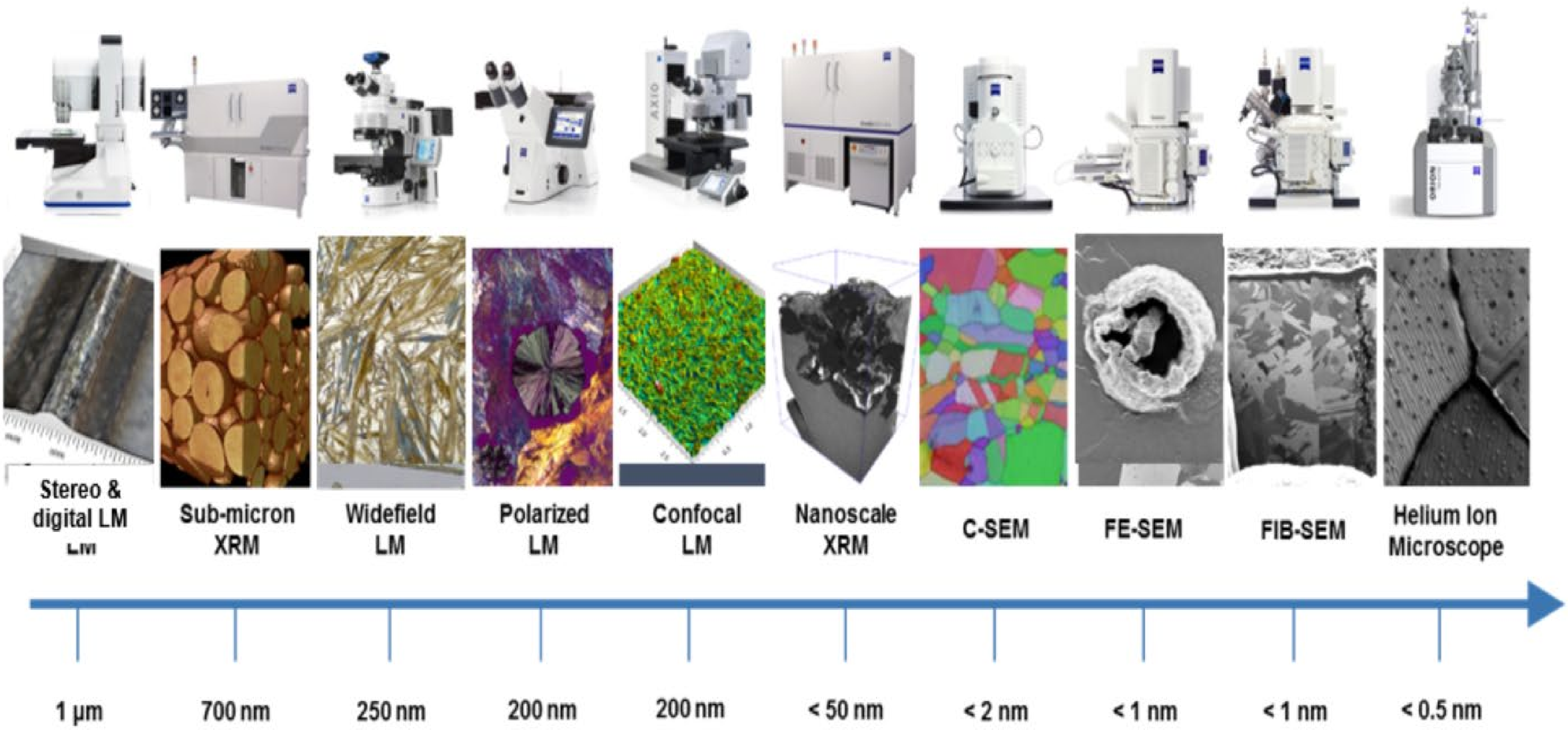
- Combining data results from multiple solutions can identify complex crack and corrosion geometries which can lead to a more complete understanding of the underlying mechanisms for Corrosion.

- Techniques exist to create a Correlative Workflow that can simplify the acquisition of data across multiple scales – from Nano to Micro to Millimeter.

- The use of a semi-automated Correlative Workflow speeds up the process of searching the same ROI across multiple instruments allowing us to quickly identify areas of potential failure.

- Software and Hardware options can be combined to create a single project combining all of the data collected via Light, Confocal, Electron, and X-ray Microscopy.

Zeiss Product Solutions



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