

Raman Imaging and
Scanning Electron Microscopy

RISE[®] Microscopy

Correlative Molecular and Ultrastructural Imaging



RISE[®] Microscopy

Molecular and Ultrastructural Imaging

Correlative Raman imaging and scanning electron (RISE) microscopy for comprehensive sample analysis offers a new dimension in imaging: see both the form and substance of your samples at the highest resolution.

RISE[®] microscopy will benefit ...

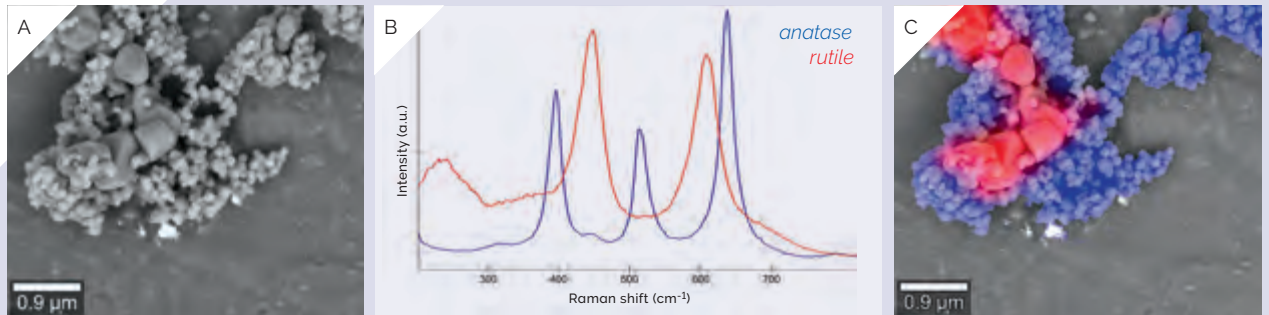
... researchers looking for a deeper understanding of their samples through quick and straightforward measurements controlled with an intuitive user interface.

RISE[®] microscopy is well suited to ...

... investigations in materials science, nanotechnology, forensics, geosciences, life sciences, pharmaceutical research and many other fields of application.



Applications



Titanium dioxide

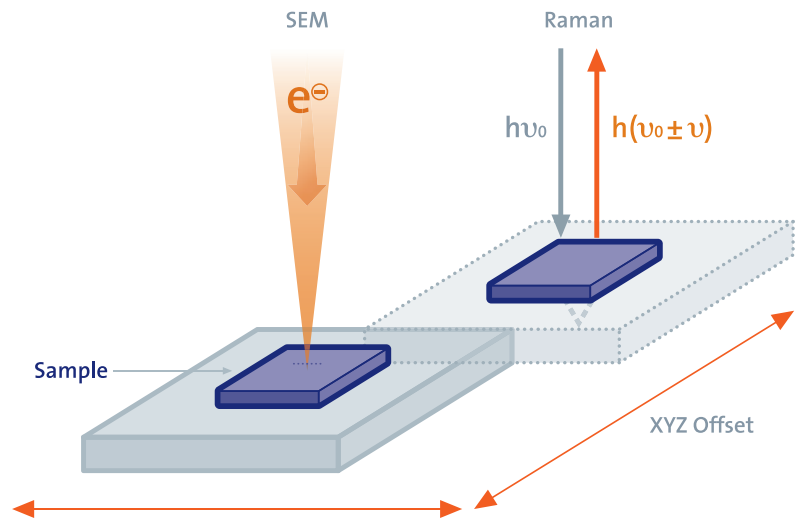
(A) Two modifications of titanium dioxide, anatase and rutile, were mixed and imaged with an SEM. (B) In the Raman spectrum anatase (blue) can be easily distinguished from rutile (red). (C) RISE image derived from Raman spectra and SEM data.



- Monolayer
- 2L: AB
- 2L: Rotation >20°
- 2L: Rotation 3-8°
- 2L: Rotation 0-3°
- 2L: Rotation 12°
- More than 2L

Twisted bilayer graphene

CVD-grown, bilayer graphene (here on Si/SiO₂) is often twisted and folded. These structural properties cannot be differentiated by SEM or EDS. However, variation in peak intensities of graphene's Raman bands and changes in the FWHM (full width at half maximum) of the bands allow for the determination of the stacking order and the twist angles of the layers. These features can be correlated with the structural features by RISE microscopy.

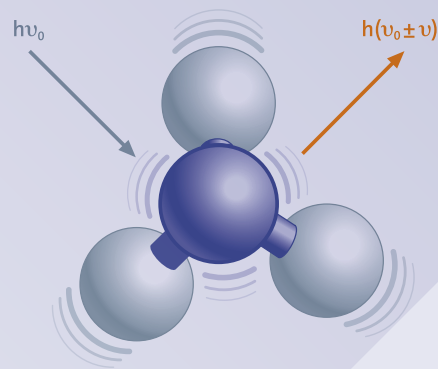


Principle of RISE[®] Microscopy

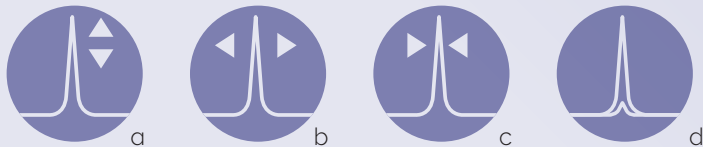
Samples are automatically transferred from one measuring position to the other within the vacuum chamber of the combined Raman-SEM instrument, streamlining the workflow and drastically improving ease of use.

The Raman Principle

- A Raman spectrum describes the energy shift of the excitation light (laser) as a result of inelastic scattering by the molecular bonds in a sample.
- Each molecule and chemical compound produces a particular Raman spectrum when excited and can be easily identified by this unique 'fingerprint'.
- Raman spectroscopy is a well-established and nondestructive method for analyzing the molecular composition of a sample.

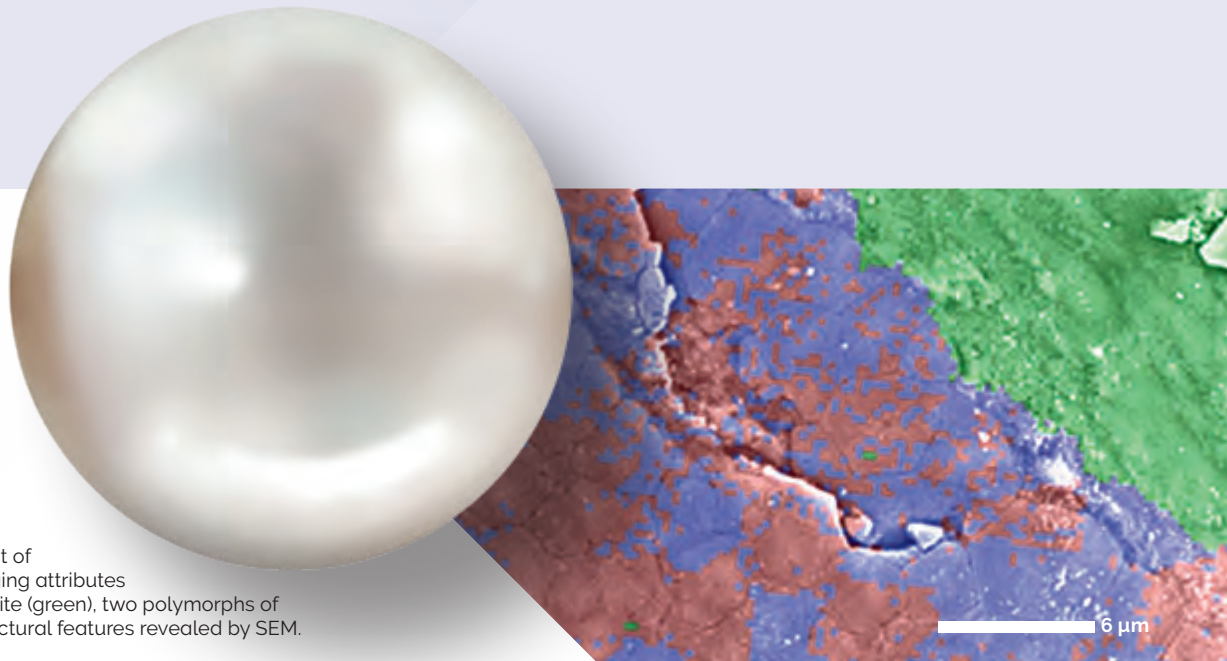


Inelastic scattering of light by a molecule



Additional sample information from Raman spectra:

- a. Peak intensity: Quantity of a specific compound
- b. Peak shift: Identification of stress and strain states
- c. Peak width: Degree of crystallinity
- d. Polarization state: Crystal symmetry and orientation



RISE microscopy measurement of a pearl's surface: Raman imaging attributes aragonite (blue, red) and vaterite (green), two polymorphs of calcium carbonate, to the structural features revealed by SEM.

RISE[®] Microscopy

RISE systems combine all features of a stand-alone SEM and a research-grade Oxford Instruments witec360 Raman microscope within one instrument to provide:

- Quick and convenient switching between Raman and SEM modes.
- Automated sample transfer from one measuring position to the other within the vacuum chamber.
- An integrated software interface for user-friendly measurement control.
- Easy correlation of the experimental results and image overlay.
- SEM and Raman imaging capabilities without compromise.
- A truly confocal optical path.
- Research-grade optical imaging.

Common vacuum chamber

Fully integrated chemical and structural analyses accelerate correlative measurements.



Confocal Raman Imaging

Oxford Instruments' witec360 Raman imaging systems combine Raman spectroscopy with confocal microscopy to offer:

- Complete Raman spectral acquisition at every image pixel with diffraction-limited resolution (~300 nm).
- Unprecedented performance in speed, sensitivity and resolution.
- Outstanding depth resolution ideally suited to 3D image generation and depth profiles.
- Ultrahigh-throughput spectroscopic capability for the highest sensitivity.
- Nondestructive imaging: No staining or other specialized sample preparation is required.



Scan table

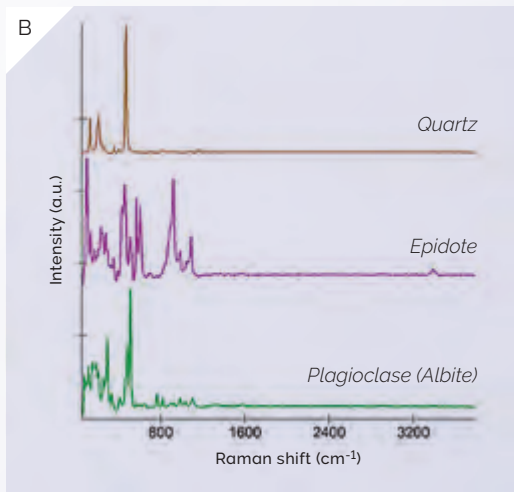
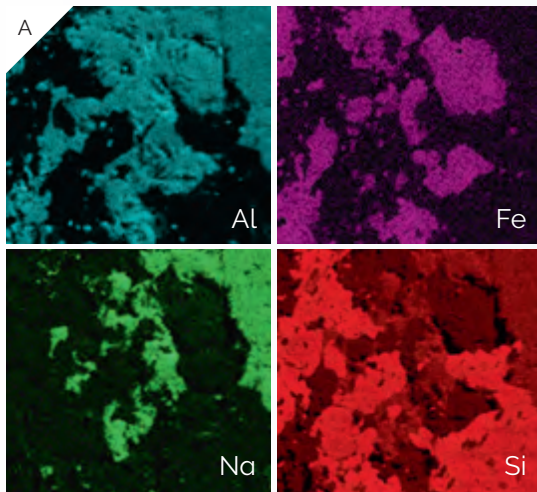
The sample remains inside the vacuum chamber during both the SEM and Raman measurements to ensure a streamlined workflow.

Benefits

of combined RISE[®] microscopy and energy-dispersive X-ray spectroscopy (EDS)

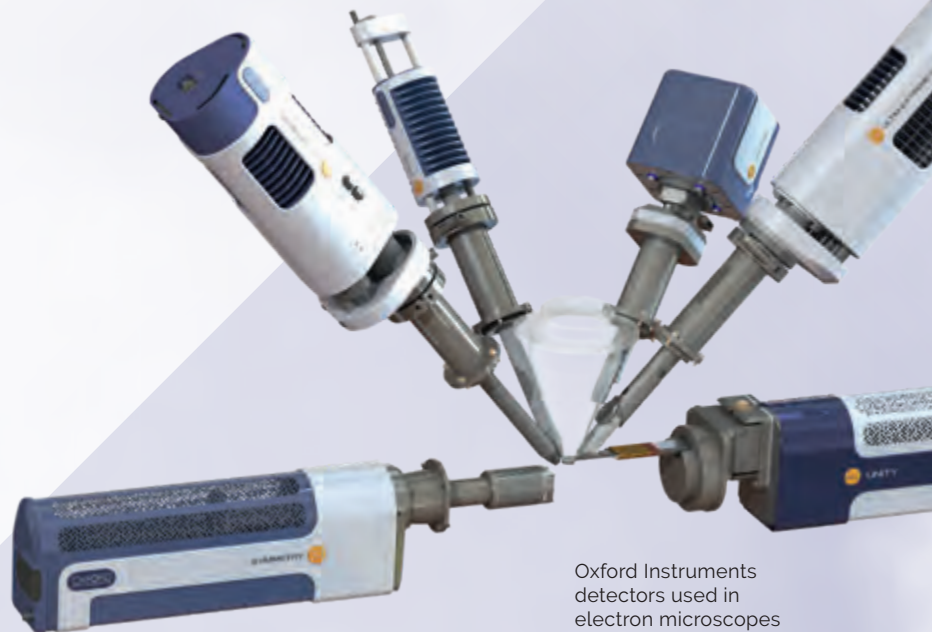
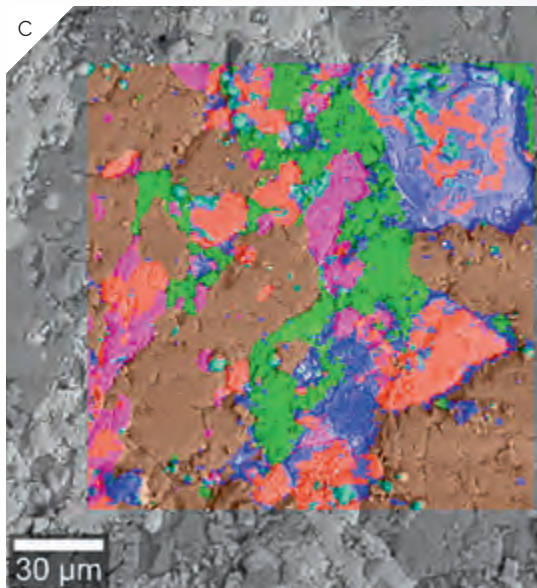
Take full advantage of Oxford Instruments' analytical tools for SEM systems to characterize the chemical properties of a sample. EDS can detect elemental composition and Raman spectroscopy can identify molecules and their bonding characteristics.

	RISE [®]	EDS
Detailed insight	Molecular and bonding information, crystallinity, material stress and strain, crystal orientation	Elemental quantitation and distribution, microstructure and crystal structure
Wide-ranging analysis	Sub-micron and large-area molecular information, 3D chemical imaging	Point analysis, large area mapping, particle analysis
Dynamic vision	Analysis of chemical processes possible	Live Chemical Imaging in real time with AZtecLive
Multimode operation	Independent Raman operation, Raman-SEM image correlation	Simultaneous SEM and EDS data acquisition



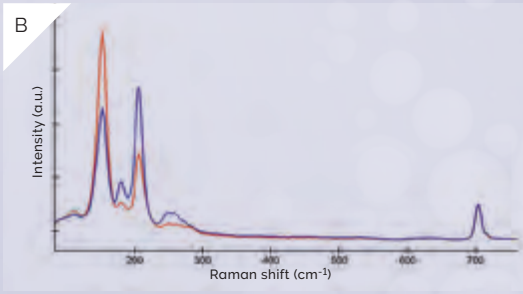
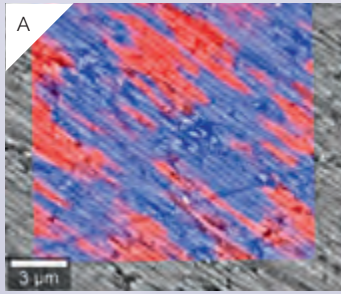
Geoscience sample

(A) Distribution of elements, measured in EDS mode of the SEM.
(B) Raman spectra of the same sample area: Quartz (brown), epidote (pink), plagioclase (green).
(C) RISE image showing the distribution of the molecular compounds in the sample. Raman imaging parameters: 22,500 spectra, 80 ms integration time per spectrum



Oxford Instruments detectors used in electron microscopes

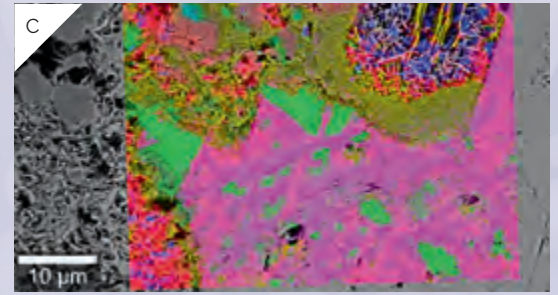
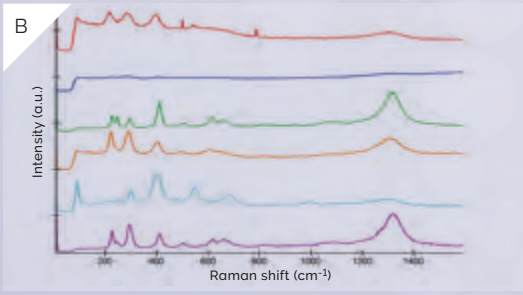
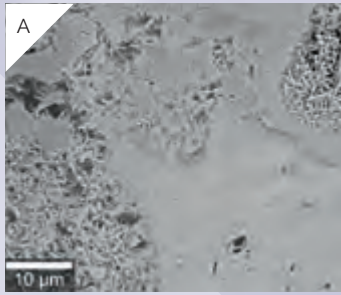
Applications



Abalone shell

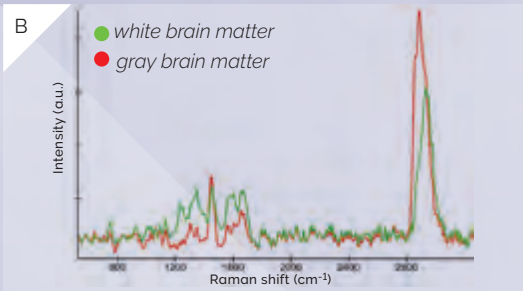
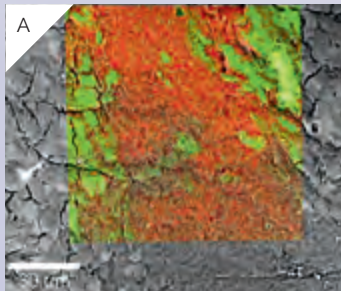
(A) RISE image of a polished cross section reveals the layered structure of the nacre (mother-of-pearl). It consists of aragonite, a crystal form of calcium carbonate.

(B) Raman spectra can enable the differentiation of crystal orientations (blue, red), revealing the anisotropy of the aragonite phase.



Iron mineralogy

(A) In the SEM image a piece of hematite (Fe_2O_3) shows some structural characteristics. **(B)** Hematite, goethite and vonsenite were identified from their Raman spectra. The spectra of crystal forms of hematite are depicted in green, orange and purple, those of goethite ($\text{FeO}(\text{OH})$) in red and cyan. The spectrum of vonsenite ($\text{Fe}^{2+}_2\text{Fe}^{3+}(\text{BO}_3)_2\text{O}_2$) is shown in blue. **(C)** Correlation of Raman and SEM data resulted in the RISE image.

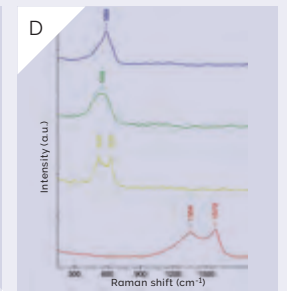
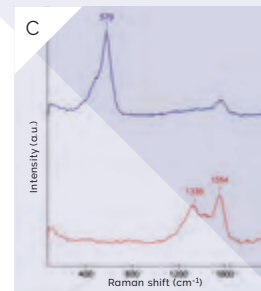
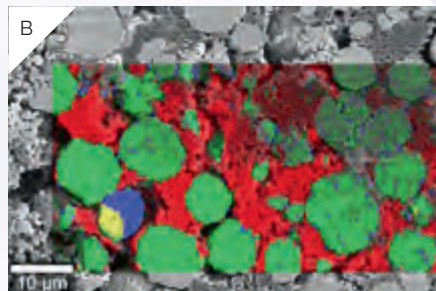
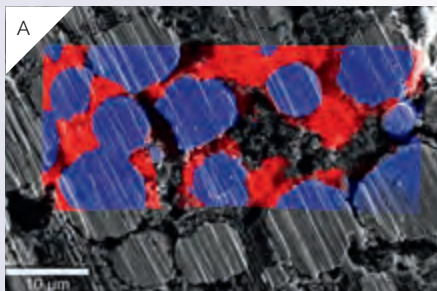


Brain tissue

(A) RISE image of a hamster brain tissue sample. In the color-coded Raman image the white brain matter is shown in green and the gray matter in red. **(B)** The corresponding Raman spectra reveal the different spectral characteristics of the white and gray brain matter.

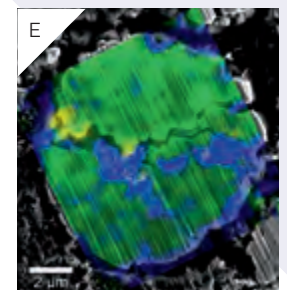
LT GaAs sample

The color-coded Raman image, showing the gold substrate (yellow) that can be clearly distinguished from GaAs (red), is correlated with the ultrastructure of the sample.



Cathode of a fast-cycled Li-ion battery

(A) Cross section of an uncycled cathode. Particles embedded in amorphous carbon (red) contain uniformly-distributed lithium nickel cobalt manganese oxide (Li-NMC, blue). **(B)** Cross section of a cycled and rapidly charged cathode. Compared to the uncycled cathode the particles appear inhomogeneous. One particle (bottom left) shows a completely different composition, including a spectrum (blue) identical to the Li-NMC spectrum of the uncycled electrode. **(C)** Typical Raman spectra of Li-NMC (blue) and amorphous carbon (red) of the uncycled cathode. **(D)** Raman spectra of molecules detected in the cycled cathode. Changes in peak positions and peak width indicate variance of Li-NMC composition. **(E)** RISE image of a particle of a cycled cathode that reveals changes in Li-NMC composition and substantial structural degradation.



Sample courtesy of Dean Miller (Tescan USA)

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