

# inLux<sup>™</sup> scanning electron microscope Raman interface

## A universal solution for in situ SEM Raman analysis

Add the inLux<sup>™</sup> SEM Raman interface to your scanning electron microscope (SEM) to provide simultaneous SEM and Raman imaging. Take advantage of the chemical and structural information provided by the inLux interface to better understand your samples and help solve research and material science challenges.

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## Precisely co-localised SEM and Raman measurements

The innovative inLux SEM Raman interface brings high-quality Raman functionality to your SEM chamber. Now you can collect Raman spectra that can produce images in 2D and 3D whilst simultaneously imaging in SEM.

The sample remains static between SEM imaging and Raman data collection locations, so you can be confident of precise co-location when comparing Raman images and SEM images.

## Key benefits

- Information-rich Raman, photoluminescence (PL) and spectral cathodoluminescence (CL) analysis is performed simultaneously and co-located with SEM imaging.
- **Universal** The inLux interface can be mounted on a wide range of SEMs from different manufacturers, with different chamber sizes, and without any SEM modification.
- Non-invasive The inLux probe can be fully retracted with a single click. This ensures that the probe does not interfere with other SEM functions or workflows when not in use.
- Determine distribution Confocal Raman images can be produced as standard, thereby enabling easy measurement of sample heterogeneity.
- **Sample viewing** Large area optical imaging and montaging for visualising your sample and targeting areas of interest.
- **Configurable** Up to two different excitation laser wavelengths, plus an optional CL module.
- **Automated** One-click switching of laser wavelengths for Raman analysis of challenging samples.



## Gain insight using correlative multi-modal analysis

Link surface composition and topography with chemical, structural and electronic information

- Combine SEM, Raman, PL, optical images and spectral CL to deepen your scientific understanding.
- Use true correlative microscopy to better understand and relate topographic, structural and chemical features within your samples.

### Raman image creation and optical montaging made easy

- One-click to switch between Raman collection and optical imaging modes.
- Produce a large area, high-resolution optical image of your sample using optical montaging.
- Detailed Raman images can be obtained over 500 µm in three dimensions with minimum step size of 50 nm using Renishaw's high performance RESOLUTE<sup>™</sup> encoders.

### Move the inLux probe and not your sample

- The inLux probe is rastered over a stationary sample for targeting and Raman data collection in 2D or 3D.
- With a fixed sample you can be confident in the correlation of your mutli-modal measurements.

### Absolute security with encoded collision protection

- Renishaw absolute encoders limit the motion of the inLux probe to a defined safe volume, even after power outages.
- Safe in a multi-user environment because the operator cannot move the inLux probe out of the defined safe volume.



The inLux probe tip showing the electron beam aperture.



RESOLUTE<sup>™</sup> absolute position encoder used in the inLux interface for precise positioning.





# **In-SEM data collection**

The inLux probe contains a parabolic mirror to focus laser light and collect Raman light from your sample in the SEM chamber. The parabolic mirror has a 0.75 mm aperture to enable the electron beam of the SEM to scan the sample during Raman data collection.

Simultaneous SEM imaging during Raman collection, can also confirm the condition of the sample at the time of Raman analysis. Any modifications to the sample during analysis are immediately detected.

### Laser safety

Vacuum sensors in the inLux interface ensure laser light can only be emitted from the inLux probe when the SEM is under vacuum. If the chamber is opened, the laser interlock will be triggered, and the laser is made safe.





# **Connect the inLux interface to different Raman systems**



### Virsa<sup>™</sup> Raman analyser

For dedicated Raman analysis, the inLux interface can be connected to the Virsa Raman analyser. The Virsa analyser offers a compact cost-effective solution to in-SEM Raman analysis, with the high sensitivity and spectral resolution expected from a research-grade Raman system in a rack-mounted body.



#### inVia<sup>™</sup> confocal Raman microscope

Connect the inLux interface to the inVia confocal Raman microscope to add in-SEM analysis to the world's best-selling research grade Raman microscope. The inVia microscope offers world-leading performance and sensitivity in a configurable range of laser excitation wavelengths, detectors and gratings. It is ideal for analysing any Raman active material. The inVia microscope can be used independently for Raman analysis. Your SEM can then be available for other users when *in situ* measurements are not required.



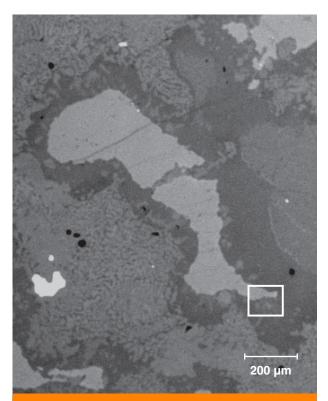
# inLux interface applications

It is possible to access a wealth of complementary information by performing correlative Raman and SEM analysis. Whether you require highly magnified images of your material or more detailed chemical and structural information, together these techniques benefit a huge range of analysis needs.

## Geology

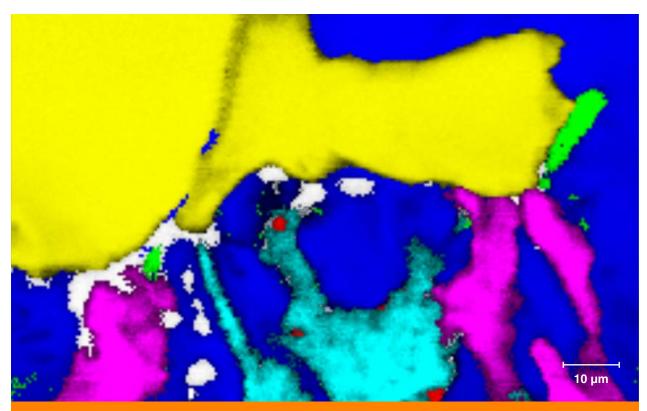
Geological sections are important samples in research areas such as mineralogy, meteoritics, petrology, and mining. Analysing such samples using an SEM is fundamental to understanding morphology and elemental composition. Back-scattered electron images provide contrast based on atomic number and topography. This can provide superior and complementary contrast to optical microscopy. Raman spectroscopy provides important additional information on the chemical composition, crystal structure and polymorphism. The inLux interface can provide all this information on a single platform.

Back-scattered electron images were collected from a geological sample showing features of different contrast. The inLux interface was used to collect Raman data from the same region of the sample enabling the creation of Raman images. The images from the different methods can be analysed using Renishaw's dedicated Correlate<sup>™</sup> software module. This results in detailed chemical information to complement the SEM images, all collected within the SEM. Raman analysis enabled the identification of 10 different minerals and their distribution in the section. Identifying these coexisting minerals enables geologists to study the metasomatism of rocks in the Earth's upper mantle.

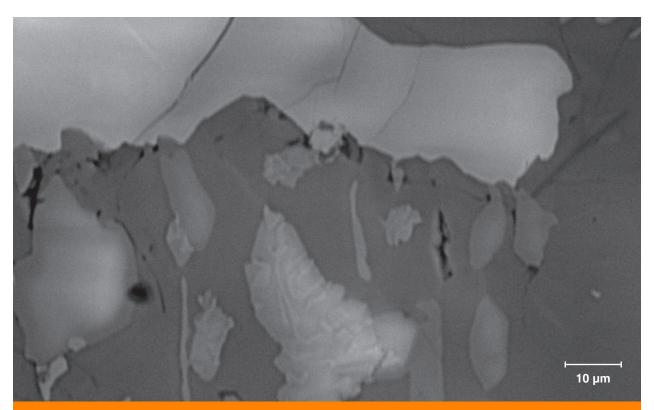


SEM back-scattered electron image with inset box showing region for detailed analysis.





inLux interface Raman image showing the distribution of pyralspite (yellow), pargasite (magenta), orthopyroxene (cyan), labradorite (blue), spinel (red), and phlogopite (green). Minor species including augite, kutnohorite, and enstatite are shown in white.



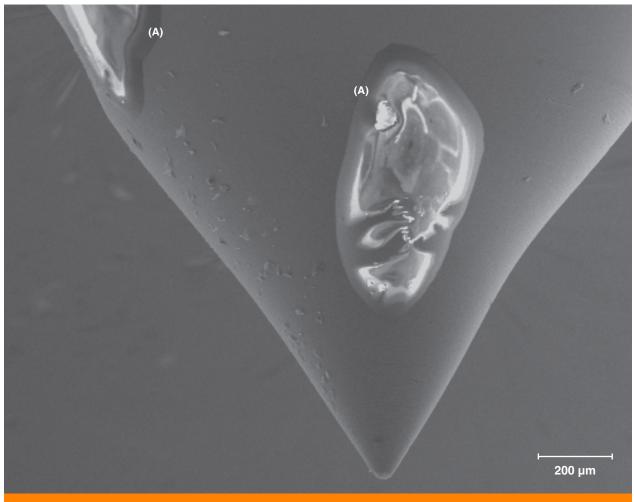
Higher magnification SEM back-scattered electron image showing variation in mineral section. Note the complementary Raman and SEM information between the domains.

## **Identify contaminants**

Raman spectroscopy is a non-contact and non-destructive technique that can provide highly specific chemical information making it ideal for identifying contaminants. Raman spectroscopy is particularly powerful for analysing carbon and organic contaminants that would be difficult to differentiate using elemental analysis. An SEM can be used to locate and study the morphology of small contaminant particles that cannot be resolved by optical microscopy. Next, these particles can be directly targeted for Raman analysis using the inLux interface, without having to move the sample.

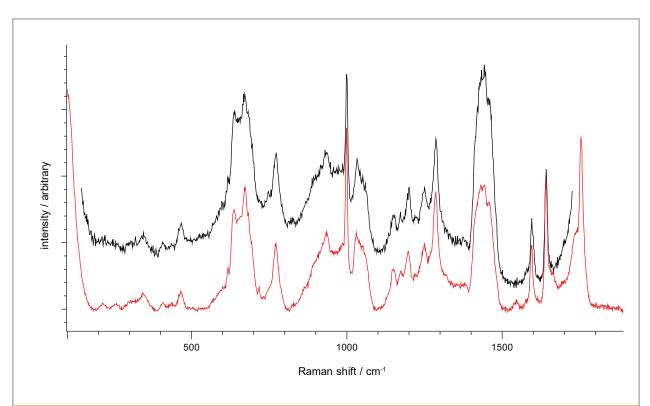
In this example, the inLux interface was used to identify contaminated regions on a carbon coated fuel injector. The Raman spectrum was used to identify the contaminant as a methacrylate UV-cured adhesive, used elsewhere in the production environment. The SEM image was able to confirm the morphology and distribution of the adhesive at high magnification and resolution. The EDS spectrum was consistent with the Raman analysis results, but could only determine that the contaminant was an organic compound, highlighting that in this case the inLux interface was vital. In addition, a Raman depth profile was collected from the sample allowing its thickness to be determined to be  $\sim 5 \ \mu m$ .

The automated multi-laser wavelength capability of the inLux interface was used to easily produce a highquality spectrum in just 5 seconds. One-click laser wavelength changing is crucial for analyses where the contaminant is unknown or may be impure. Renishaw's WiRE<sup>™</sup> software readily compares the Raman spectra from unknown substances against spectral libraries. With software features such as spectrum search that can identify mixtures, identifying contaminants is easy.

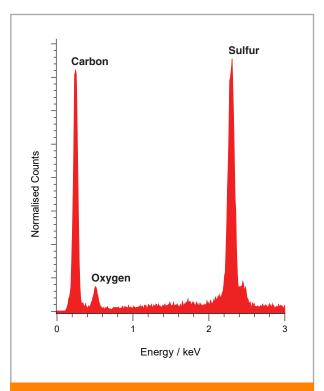


SEM secondary electron image of contaminant regions (A) on the tip of a carbon coated fuel injector.

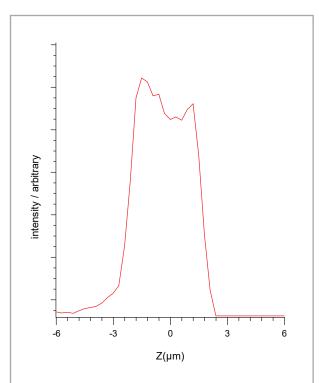




Raman spectrum of an unknown contaminant (black) with a library spectrum (red) of a UV adhesive. The clear match between the spectra enables identification of the unknown and highlights the strength of the inLux interface for contamination analysis. Data collected with 532 nm laser excitation.



The EDS spectrum of the contaminant provides elemental information, but does not allow for full identification of the contamination.

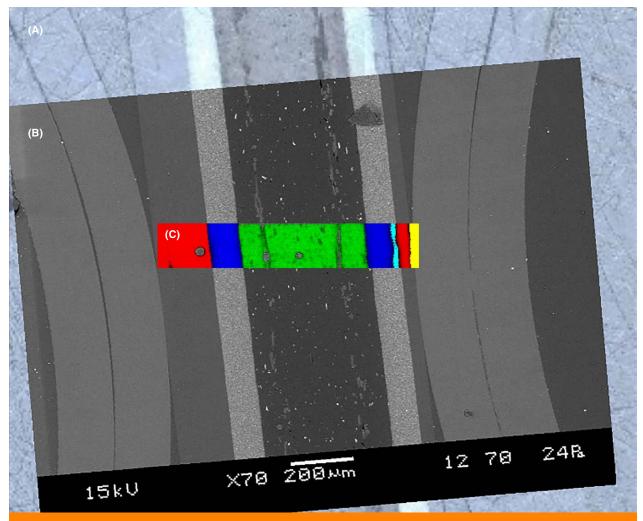




## **Revealing subtle organic components**

Scanning electron microscopy is a powerful tool for resolving material distribution far below the diffraction limit of optical microscopy. It is routinely used to image either sub-micrometre features, or the morphology of larger features. However, the ability of EDS to characterise materials is limited to the generation of elemental information. This is often challenging when differentiating between organic polymer materials. The inLux interface enables the generation of Raman images which can easily differentiate between similar organic and inorganic compounds.

An identity card cross-section was prepared within a resin mount. The cross-section was analysed with Raman spectroscopy to determine the different materials and layers present. Spectral identification was made using Renishaw's polymer and inorganic Raman libraries. The resulting optical and chemical images were then correlated directly to the back-scattered electron image using the WiRE software. The Raman images detected an additional thin layer (~12.5 micrometres thick), containing copper phthalocyanine dye. This layer is not visible in the back-scattered electron image or the optical image. All images are available to view, on the same coordinate system, in the WiRE software, allowing detailed multi-modal, correlative analysis.



Overlaid correlative image showing the optical image (A), the SEM back-scattered electron image (B), and the Raman image (C). The Raman image illustrates the chemical composition of the various layers of the polymer laminate as polycarbonate (red), rutile  $TiO_2$  (blue), poly[4,4'-methylenebis(phenyl isocyanate)alt-1,4- butanediol/poly(butylene adipate) (green), copper phthalocyanine (cyan) and polyethylene terephthalate (yellow). There is a clear correlation between all images. Note the cyan layer which is not visible in the SEM or optical image, which highlights the benefits provided by Raman analysis.



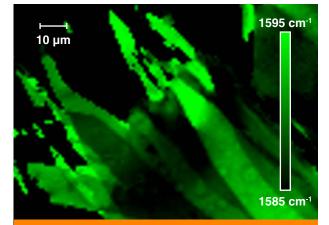
## Raman imaging of materials and semiconductors

Many of the novel properties exhibited by materials arise from their size, shape or thickness. Graphene, nanorods and nanotubes are examples of where the high magnification of scanning electron microscopy is vital to visualise the sample. As well as revealing the chemical and structural nature of the material, Raman analysis can also provide information on physical properties. The inLux interface can produce Raman images illustrating crystallinity, strain, and electronic properties that can be correlated to those from the SEM.

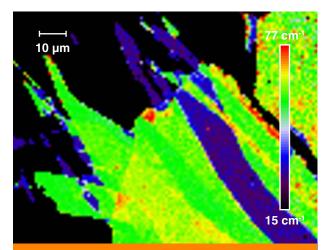
The identification of single layer graphene is fundamental to its research and production. Here we have used the inLux interface to produce Raman images of single and multi-layered graphene. Over 11,000 spectra were collected by moving the inLux probe, while the sample remains stationary inside the SEM. The WiRE software was used to determine the band characteristics of the G band and the 2D band. This produces results of high accuracy and precision, allowing layer thickness and strain to be visualised.



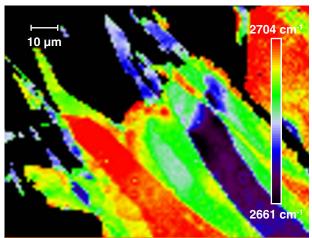
SEM secondary electron image of graphene corresponding to the same area as the Raman images shown.



Raman image of G band position, an indicator of local strain and doping in the graphene layers.



Raman image of the graphene 2D band width, an indicator of the number of graphene layers. Single layer material (blue), few layer material (green), and thicker material in (yellow/red).



Raman image of 2D band position, this can be used to understand the local electronic properties of the graphene.

### Technical highlights: inLux SEM Raman interface

General	
Dimensions	W 804 mm x H 257 mm x D 215 mm
Mass	< 20 kg
Fibre optic cable length	4.6 m
Compatible Raman spectrometers	Renishaw inVia confocal Raman microscope, Renishaw Virsa analyser
Compatible SEM models	Compatible with models from all major SEM suppliers
SEM port requirement	Requires free SEM side or rear port
SEM performance	The inLux interface does not require any SEM modification and can be fully retracted when not in use so it does not interfere with SEM performance or that of other accessories
Movement control	Trackpad, WiRE software
Contact protection	Touch sensor, safe working volume monitored using absolute encoders
Laser safety	Laser interlocked to chamber vacuum
Raman mapping/imaging	Supplied as standard
Fibre optic module selection	Up to two different laser excitation wavelengths + optional cathodoluminescence module
Available laser excitation wavelengths	405 nm, 532 nm, 660 nm, 785 nm (others available on request)
Laser switching	Automated, motorised and software controlled
Lateral spatial resolution	< 1 µm @ 532 nm
Confocal performance	< 6 µm @ 532 nm
Spectral resolution	See spectrometer specification sheet
Raman imaging and optical imaging	
Raman imaging modes	Point mapping, StreamHR Discrete point, line, depth profile, area, depth slice, volume
Raman imaging area	> 500 μm × 500 μm × 500 μm
Raman imaging step size	50 nm encoded
Optical field of view	300 μm x 300 μm
Maximum optical imaging area	> 2 mm x 2 mm
System	
Power requirements (external supply)	Input: 100 Vac to 240 Vac, 50 Hz to 60 Hz, 90 W.
Operating humidity	Maximum 80% RH up to +31°C, decreasing linearly to a maximum 50% at +40°C
Ambient temperature	Recommended: 20°C to 30°C stable to $\pm$ 1°C
	Operating: 5°C to 40°C
Standards	CE, UKCA
Warranty	12 months standard; extended warranty and service packages available
Connection to system computer	USB 3.0

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