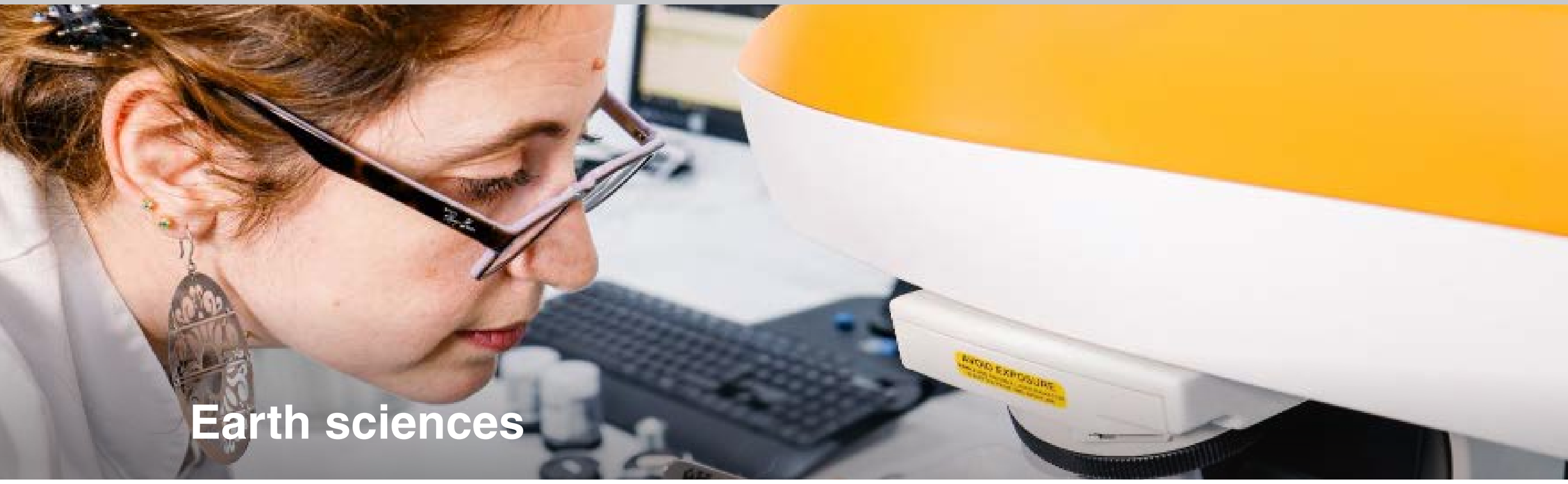
Application note

Analysing gemstones with the inVia™ confocal Raman microscope



Validate the authenticity of gems with Renishaw's inVia confocal Raman microscope

For a long time the human eye and a microscope were the only tools in a gemmological laboratory. However, technological improvements have led to a new range of artificially treated natural stones and synthetic gems that the eye and the microscope cannot distinguish. Today's gemmological laboratories need to have instruments and skills that rival university research centres. Of the many analysis techniques available, Raman and photoluminescence spectroscopies have become the best techniques for gemstone certification and characterisation.

The inVia confocal Raman microscope is the ideal tool for certifying and characterising gemstones

It can easily perform both Raman spectroscopy and photoluminescence spectroscopy. You can unambiguously:

- Quickly identify and authenticate gemstones
- Analyse inclusions to determine geological and geographic origin
- Identify diamond treatments. These include irradiation treatments and high-pressure high-temperature (HPHT) annealing, used to enhance quality or to introduce fancy colours. It can also determine where multiple treatments have been applied
- Identify treatments on coloured stones, such as:
  - Heat treatments (typically on rubies and sapphires), by analysing inclusions
  - Heat treatments based on structural modification (e.g. spinels)
  - Fracture filling-treatments (e.g. emeralds, tourmalines, and glass-filled corundum)
  - Impregnation-treatments (e.g. jades, opals, and opaque stones)
  - Irradiation treatment (e.g. on pearls)
- Differentiate between natural and synthetic gemstones (diamond, spinels, emerald)
- Identify imitations and simulants

The inVia microscope has the further advantage that it can analyse both loose and set stones (in jewellery and watches). It is also a fully capable research instrument, so can be used to conduct scientific research in gemmology.

Renishaw plc
Spectroscopy Products Division
New Mills, Wotton-under-Edge,
Gloucestershire GL12 8JR
United Kingdom

T +44 (0) 1453 524524
F +44 (0) 1453 524901
E raman@renishaw.com

www.renishaw.com/raman

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High-pressure high-temperature (HPHT) treatments

HPHT annealing treatments can dramatically improve the colour of stones in the D-Z scale by causing a significant reconfiguration of defects in the mineral lattice.

The first industrial enhancement processes—by General Electric (GE) and Lazare Kaplan International—changed type IIa brown diamonds (N-O on the Diamond Colour scale) to colourless or near colourless (D-H). Photoluminescence (PL) analysis at low temperature (77 K) has proved to be the best tool for detecting this, by monitoring the diamond GR1, N3, H3 and N-V centres.

We illustrate this with low-temperature PL spectra showing spectral differences (taken using 514.5 nm excitation) related to the N-V vacancies in type IIa untreated and treated diamonds (Figure 1).

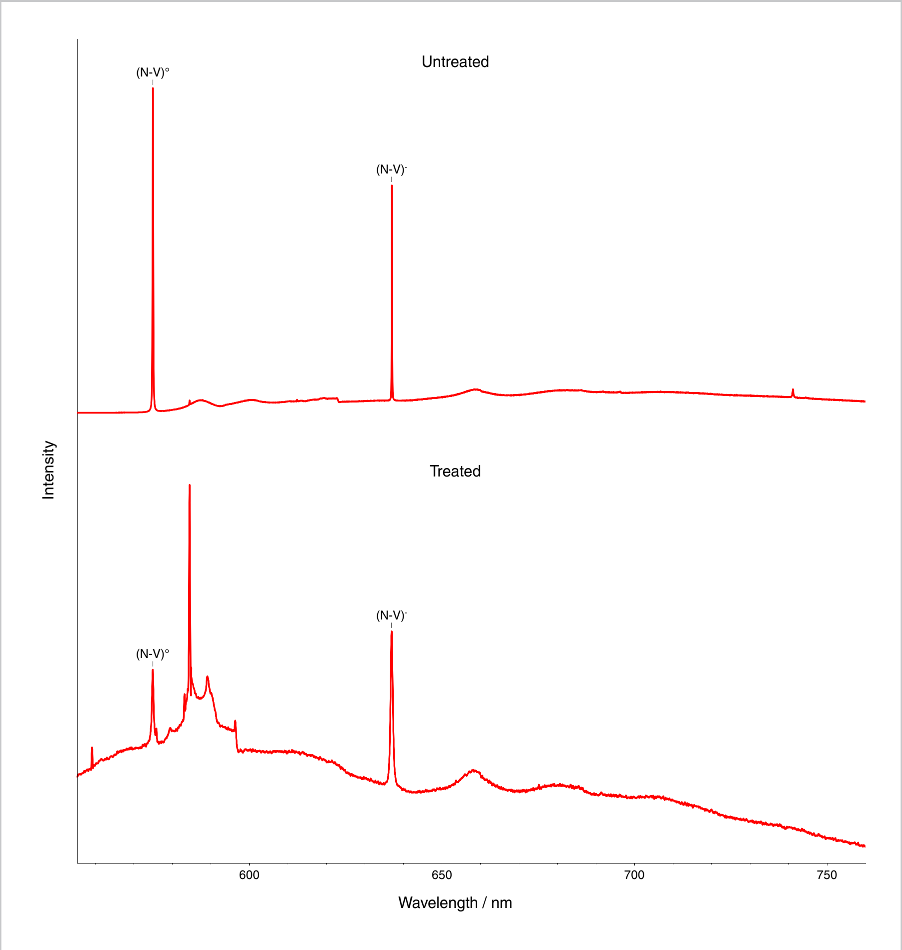

Figure 1 - PL spectra from untreated and HPHT treated Type IIa diamonds.

The 574.8 nm and 637.0 nm PL bands are related to the uncharged (N-V)° and negatively charged $(N:V)^-$ vacancies. The ratio of their integrated intensities and the width of the 637.0 nm band (full-width half-maximum, FWHM) give indications about the treatment, with the widths being broader for treated stones (Table 1).

The PL bands at 740.9 nm and 744.4 nm can also be monitored. These correspond to the general radiation centre (GR1), the neutral vacancy defect V°. This is commonly present in type II diamonds and is strongly reduced in intensity after annealing.

Spectral measure	Untreated	Treated
574.8 nm / 637.0 nm bands	1.7 – 7.7	0.3 – 0.7
637 nm band width (FWHM)	0.47 – 0.80	0.64 – 1.00

Table 1 – Typical parameters from PL spectra of type IIa untreated and treated diamonds showing the spectral differences related to the N-V vacancies.

Renishaw plc
Spectroscopy Products Division
New Mills, Wotton-under-Edge,
Gloucestershire GL12 8JR
United Kingdom

T +44 (0) 1453 524524
F +44 (0) 1453 524901
E raman@renishaw.com

www.renishaw.com/raman

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Chemical vapour deposited (CVD) type IIa synthetic diamonds

It is of major importance for the jewellery industry to distinguish between natural and laboratory grown diamonds. This can be done when bands from silicon-related lattice defects (Si Centre) are present in the low temperature PL spectrum. Silicon is usually an impurity introduced during the CVD process and is not a common feature in natural diamonds.

These defects correspond to a doublet of peaks between 736.6 nm and 736.9 nm in the PL spectrum.

Moreover, bands at 740.9 nm and 744.4 nm are related to the GR1 feature and are typically absent in CVD grown diamonds.

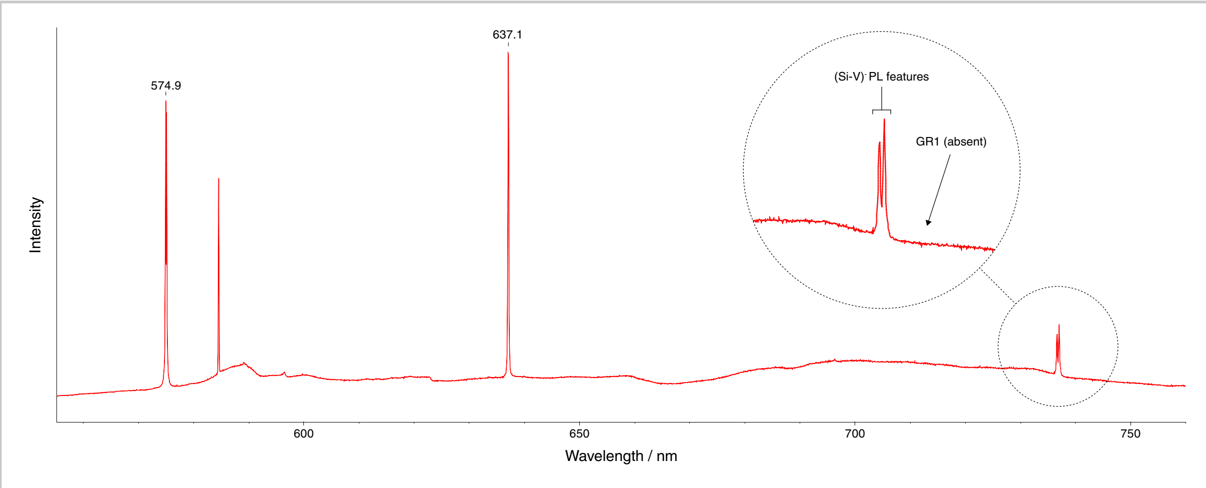

Figure 2 - Type IIa CVD laboratory-grown diamond, showing silicon related lattice defects at 736.6 nm and 736.9 nm, and absence of the GR1 band (position indicated by the arrow).

Analysis of inclusions

The inVia microscope's EasyConfocal™ technology provides high spatial resolution and enables you to rapidly study small gem inclusions. The inVia microscope supports transmitted, reflected, and darkfield illumination, giving you a range of options for locating the inclusions. Figure 3 illustrates an inclusion in natural corundum, pink sapphire variety.

Inclusions can be easily and quickly identified using Renishaw's inorganic materials and minerals spectral library, which contains over 1000 spectra of relevant species. Figure 4 shows one of the inclusions in the corundum being identified as a spinel.

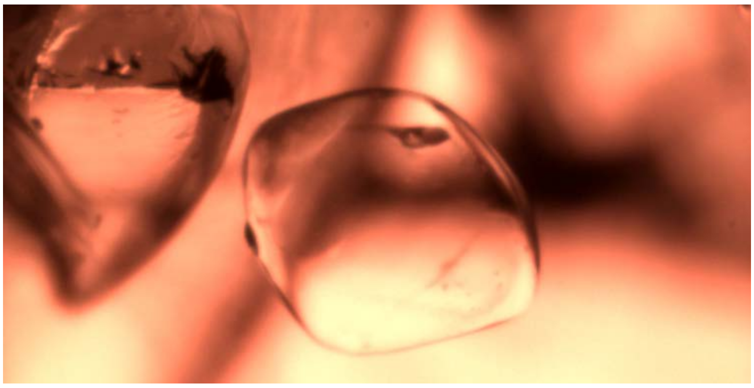

Figure 3 – a spinel inclusion in Natural Corundum, Pink Sapphire variety.

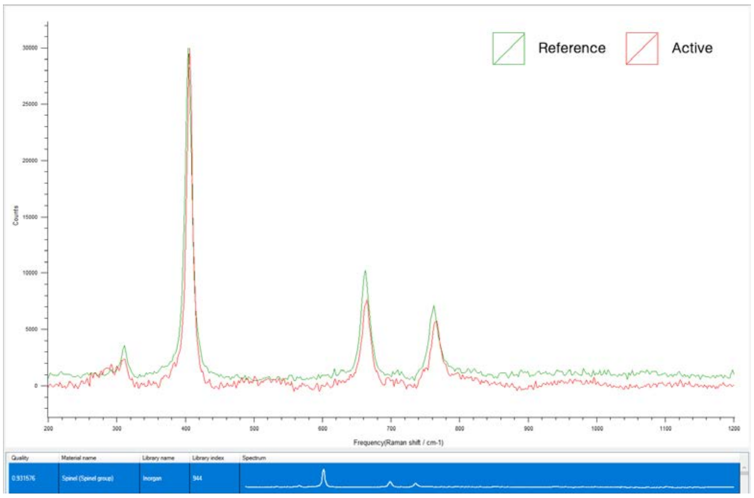

Figure 4 – using the Renishaw library search to identify the corundum inclusion as being a spinel.

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www.renishaw.com/raman

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inVia™ is a comprehensive analysis tool:

These examples illustrate the ease with which you can use the inVia Raman microscope to certify authenticity and detect treatments of gemstones, as part of the daily routine in your gemmological laboratory. The inVia microscope enables you to exploit the advantages of a top-grade Raman microscope and those inherent in Raman spectroscopy to:

1. Unambiguously identify materials using a dedicated gemmological spectral library
2. Analyse rapidly and non-destructively
3. Make measurements without the need for sample preparation
4. Analyse inclusions with high spatial-resolution and high sensitivity

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Renishaw. The Raman innovators

Renishaw manufactures a wide range of high performance optical spectroscopy products, including confocal Raman microscopes with high speed chemical imaging technology, dedicated Raman analysers, interfaces for scanning electron and atomic force microscopes, solid state lasers for spectroscopy and state-of-the-art cooled CCD detectors.

Offering the highest levels of performance, sensitivity and reliability across a diverse range of fields and applications, the instruments are designed to meet your needs, so you can tackle even the most challenging analytical problems with confidence.

A worldwide network of subsidiary companies and distributors provides exceptional service and support for its customers.

Please visit www.renishaw.com/raman for more information.