

THOR

**advanced confocal Raman microscope based
on our patented mirrorless technology**

 **Lightnovo**

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***THOR ideally suited
for most sophisticated
applications in Raman
microscopy***

- ***fast 3D Raman imaging***
- ***SERS imaging***
- ***polarized Raman
imaging***
- ***low frequency Raman
measurements***

- 
- *stokes/antistokes Raman measurements*
 - *provides fast optical shutter technology (<1ms)*

THOR technology covered by 4 international patents licensed by Lightnovo

SPECIFICATIONS

Spectrograph with spectroscopic sensor includes

- deep cooling spectroscopic CCD
- basic optics set

Laser options

Set for excitation at wavelength 532 nm includes

Laser

- power 1500 mW
- spatial mode TEM₀₀, M₂ <1.1
- spectral linewidth (FWHM) <1 MHz
- wavelength stability <2pm over ±2°C and 8 hrs

Optics set for spectral range 90-3700 cm⁻¹

- spectral resolution (FWHM) 4-7 cm⁻¹

Set for excitation at wavelength 785 nm includes

Laser

- power 500 mW
- spatial mode TEM₀₀, M₂ <1.7
- spectral linewidth (FWHM) <10 MHz

Optics set for spectral range 90-2900 cm⁻¹

- spectral resolution (FWHM) 3-5 cm⁻¹

Mapping stage includes

XY scanning table

- travel range 135×85 mm
- velocity 120 mm/s
- resolution 100 nm

Z scanning

- travel range 25 mm
- velocity 6 mm/s
- resolution 1.25 μm

Light microscopy includes

- reflected light microscopy set
- polarized reflected light microscopy set
- set for simultaneous visualization of visible image and laser beam at wavelength 532 nm
- set for simultaneous visualization of visible image and laser beam at wavelength 785 nm

Patented Polarized Raman microscopy set for laser at wavelength 532nm and 785nm includes

- Acousto-Optical Modulator (AOM) based laser shutter and polarization switcher
- motorized half wave plate for laser
- Wollaston Analyzer Unit (WAU),

APPLICATION

Deep volumetric Raman imaging (dVRI) by THOR system

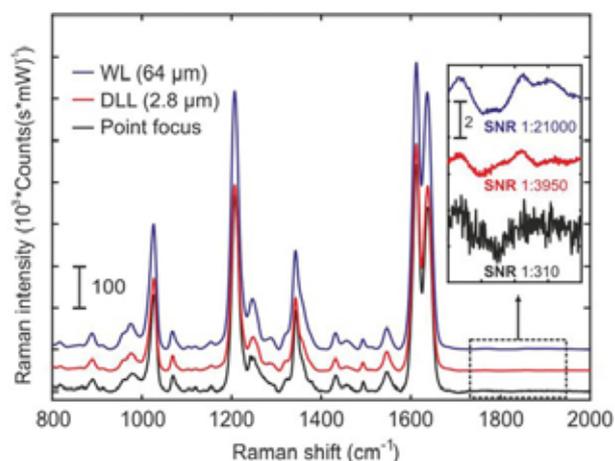
Three-dimensional (3D) confocal Raman mapping is one of the most promising techniques to study the chemical composition of complex organic and inorganic materials. However, quantitative volumetric Raman imaging is still a state-of-the-art technique with few reported applications, mostly due to interference from molecular fluorescence, limited sample transparency at the laser excitation wavelength, complexity of chemometric analysis and low Raman scattering cross section of the chemical components. The molecular fluorescence of samples can be suppressed in several ways. However, the most proper and common techniques like Kerr-gated Raman, ultraviolet Raman and near-infrared (NIR) Fourier transform (FT) Raman are not well applicable to the diffraction limited deep volumetric Raman imaging (dVRI) due to the limitation connected with confocality or penetration depth. The most appropriate solution is the usage of NIR lasers (780, 785, and 830 nm) as excitation sources. For the sole purpose of suppressing sample fluorescence, Nd:YAG lasers with 1,064 nm excitation wavelength are a very attractive solution; however, the Raman scattering cross section becomes usually too low for dVRI.

In this situation, where the fluorescence of samples takes place, our THOR system operates with NIR laser with wavelength of 785 nm. However, sample transparency is limited, even when using a laser at 785 nm excitation wavelength. It is important to mention that in-depth Raman mapping is also limited by off-axis laser refraction effects leading to Raman signal attenuation and decreased axial resolution. The usual way of addressing this problem is to increase the laser power and/or the exposure time. However, the latter leads to an unrealistic total time of 3D map acquisition (>3 days). Increasing the laser power, instead, overheats and burns the sample.

With THOR system, we present an efficient solution to solve the previously discussed problems of dVRI of samples that manifest fluorescence and low transparency. This result was achieved by the development of a confocal Raman microscope with high Raman signal throughput, optimization of sample mapping method, and further chemometric hyperspectral data analysis based on nonnegative least squares.

Wide Line SERS mapping by THOR system

Surface-enhanced Raman spectroscopy (SERS)-based molecular detection at extremely low concentrations often relies on mapping of a SERS substrate. This yields a large number (>1000) of SERS spectra that can improve the limit of detection; however, the signal collection time is a major constraint. In THOR system, a wide line (WL) laser focusing technique aimed at fast mapping of SERS substrates. The WL technique enables acquisition of thousands of SERS spectra in a few seconds without missing any of the electromagnetic “hot spots” in the illuminated area. In addition, the SERS signal averaging across the line in the WL mode displays extremely high signal-to-noise ratios. The advantages of the WL technique for SERS-based sensing are verified using different analyte molecules, that is, p-coumaric acid and melamine. Results show that the limit of detection can be improved by one order of magnitude compared to results obtained using a common point-focusing Raman microscopes.



see details here: <https://onlinelibrary.wiley.com/doi/full/10.1002/admt.201900999>

Comparison of SERS microscope laser illumination modes. Here we present SERS spectra of BPE (10 μM concentration) measured on Au nanopillars substrate at point (black), DL line (red), and WL (blue) illumination modes; zoomed spectra in the range 1750–1950 cm⁻¹ for SNR demonstration. All presented spectra were acquired at equal conditions: 0.2 mW μm⁻¹ of laser intensity, laser wavelength 785 nm, exposure time 0.1 s, 10× magnification microscope objective. SNR was measured as signal divided on RMS noise.

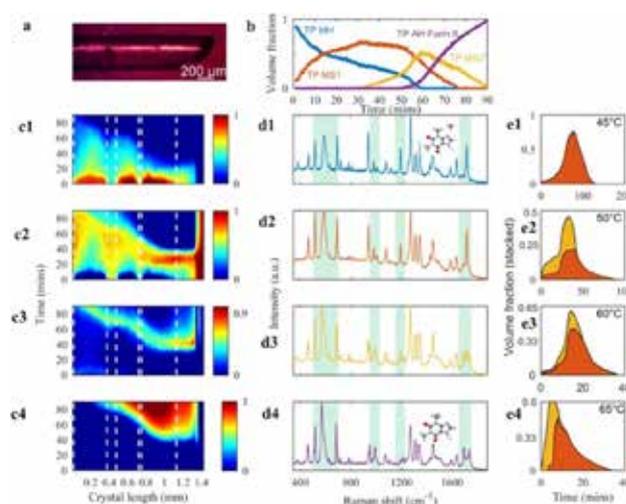


Raman line-focus microscopy with chemical decomposition

A Raman line-focus mapping option was applied for fast simultaneous mapping of differently sized and shaped particles of nitrofurantoin monohydrate, revealing the appearance of multiple solid-state forms and the non-uniformity of this particle system during the complex dehydration process. This method provides an in-depth understanding of phase transformations and can be used to explain practical industrial challenges related to variations in the quality of particulate materials.

Raman experiments resulted in a matrix that has two dimensions $[t, S(v)]$, where t corresponds to the temperature or time points, while $S(v)$ corresponds to Raman spectra. We registered 220 spectra from the laser line at each temperature/time point, and all spectra were grouped as follows: $Mline = [t_1(S(v)_1, S(v)_2, \dots S(v)_{220}), t_2(S(v)_1, S(v)_2, \dots S(v)_{220}), \dots t_n(S(v)_1, S(v)_2, \dots S(v)_{220})]$, where n is the number of temperature/time points. In order to extract information about the concentration and spectral profiles of the studied model compounds, we used multivariate curve resolution (MCR) and non-negative least squares (NNLS) methodology. Both methods realized in THOR data analysis software package.

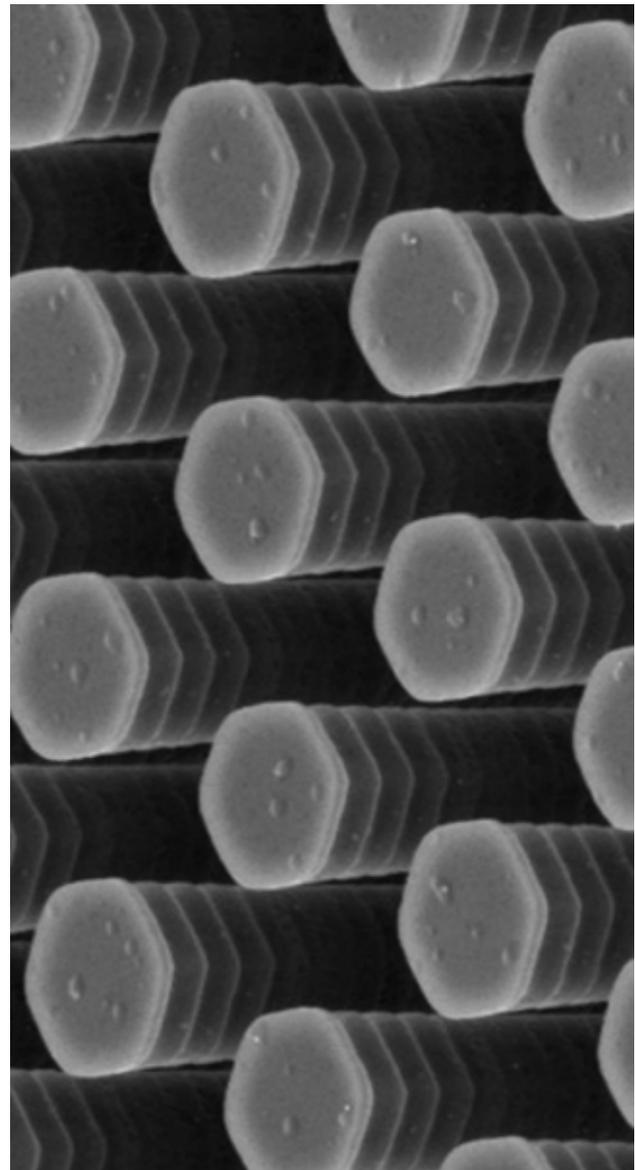
MCR and NNLS decomposed results of hydrate and anhydrous solid-state forms of theophylline. (a) Optical image of TP MH at 25 °C with laser line illumination (white line) from the Raman microscope (b) concentration profile of solid-state forms during isothermal dehydration at 50 °C for 90 minutes starting from TP MH to TP AH form II, via TP MS1 and TP MS2 (c1–4) chemical concentration maps (cut out artefacts from 1.8 mm laser line) of TP MH (c1), TP MS1 (c2), TP MS2 (c3), TP AH form II (c4) in the particle during dehydration where the laser line is illuminated, (d1–4) Raman spectra for TP MH, TP MS1, TP MS2 and TP AH form II respectively,



(e1–e4) area plots showing only the dehydration profiles of TP metastable intermediates at four isothermal conditions (45, 50, 60 and 65 °C). The stable forms of TP were taken into consideration for the area plots.

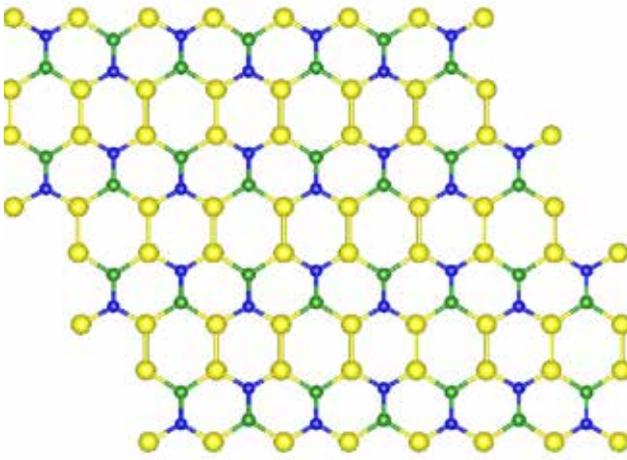
Semiconductors and Microelectronics

In the last years, Raman spectroscopy has shown to be a reliable technique for different skin disease such as cancer and atopic dermatitis. This technique is non-invasive and can provide several information regarding the molecular composition of the surface of the skin and up to several hundred micrometers in depth. However, the instruments are still slow, and bulky. Consequently, there is still the need of an instrument that can be used in the clinical practice and that, in future, patients will use constantly and easily at home in order to evaluate their therapy and to know in advance the triggering of the disease. This will help the diagnosis and the monitoring of a disease reducing time and costs for doctor check-ups. Therefore, the quality of life for patients will improve.



Dislocations and Stress in Electronics

Inhomogeneities and dislocations in heterostructured semiconductors with high levels of local heating in operation conditions can greatly reduce the lifetime of devices like LEDs and laser diodes. Therefore, nano- and microscale orientation mapping of such structures by qRICO provides important information in the development and quality control of devices.

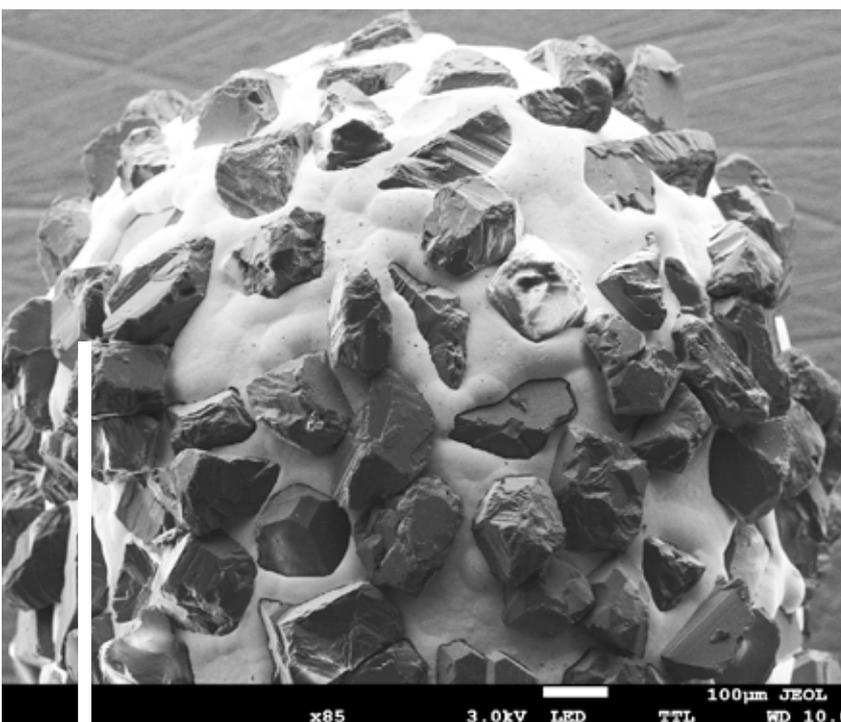
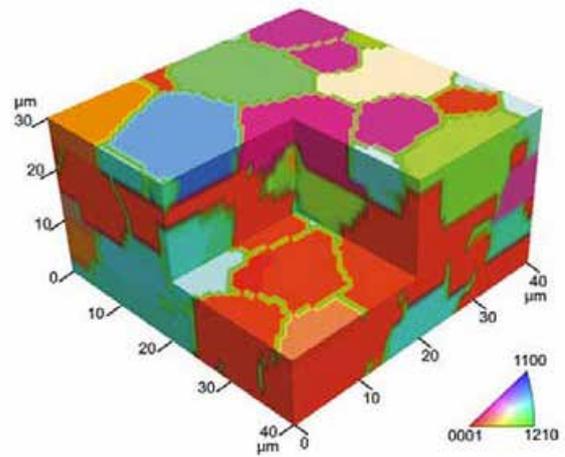


2D Materials

qRICO has a great potential of quantitative orientation mapping in 2D materials. It is used for orientation determination and dislocation search of separate layers in multi layered 2D materials.

Ceramics

3D grain mapping is very important in ceramics technology, because properties such as fracture strength is strongly influenced by the statistical distribution of grain orientation and the grain boundary topology. Thus qRICO provides unique information in piezo-, magneto-, and ferroelectrics.



Superhard Materials

Abrasives, drilling tools, superhard transparent windows – all mentioned examples require the knowledge of crystallographic orientation of polycrystalline surfaces.

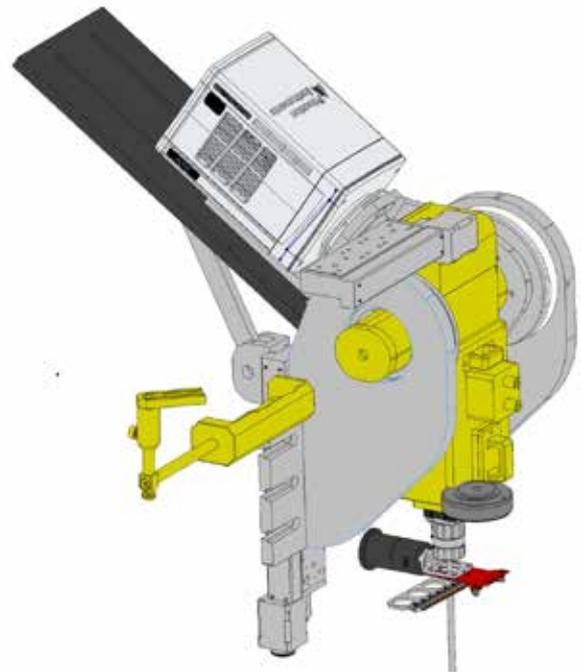
TECHNOLOGY

Working principles and advantages

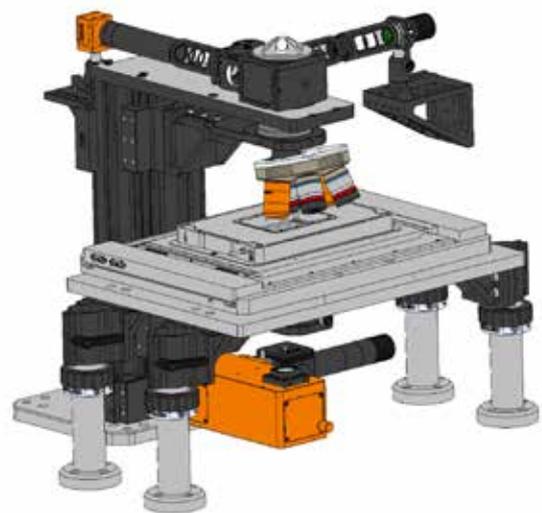
Our advanced Raman microscopes THOR based on mirrorless Raman beam delivery technology ideally suited for most sophisticated applications in Raman microscopy:

- fast 3D Raman imaging;
- SERS imaging;
- polarised Raman imaging;
- low frequency Raman measurements;
- stokes/antistokes Raman measurements;
- fast optical shutter technology (< 1ms).

THOR can be upgraded for quantitative Raman based crystallographic orientation mapping (qRICO technology). We develop qRICO technology in collaboration with our partner Xnovo Technology ApS. THOR and qRICO technology covered by 4 international patents licensed by Lightnovo. Based on our collaboration setup, Lightnovo develops and manufactures qRICO devices, support it with polarized Raman data acquisition and data analysis software. Xnovo Technology develops crystallographic data reconstruction and crystallographic data analysis software. Xnovo Technology also responsible for commercialization and sales of qRICO solution.



THOR spectrograph with patented technology for spectroscopic camera tilt around fused silica transmitting grating. Technology allows spectral range tuning.



THOR mapping and white light microscopy system. Mapping system include long travel range XY stage (close loop, resolution 100nm) and nanopositioning XYZ stage (close loop, 1nm resolution).

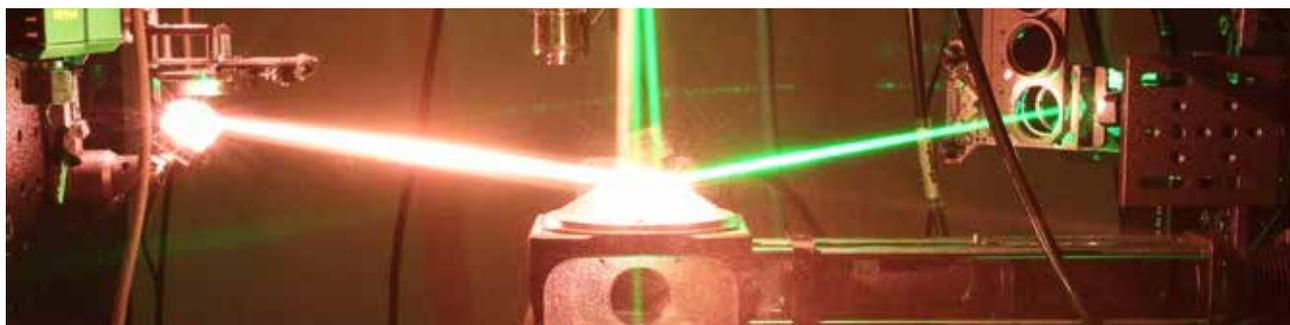
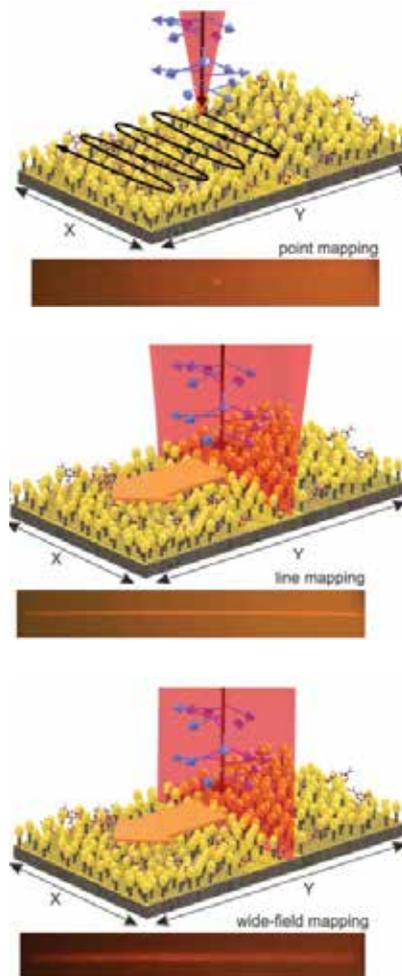
Wide line surface-enhanced raman scattering (WL-SERS) mapping

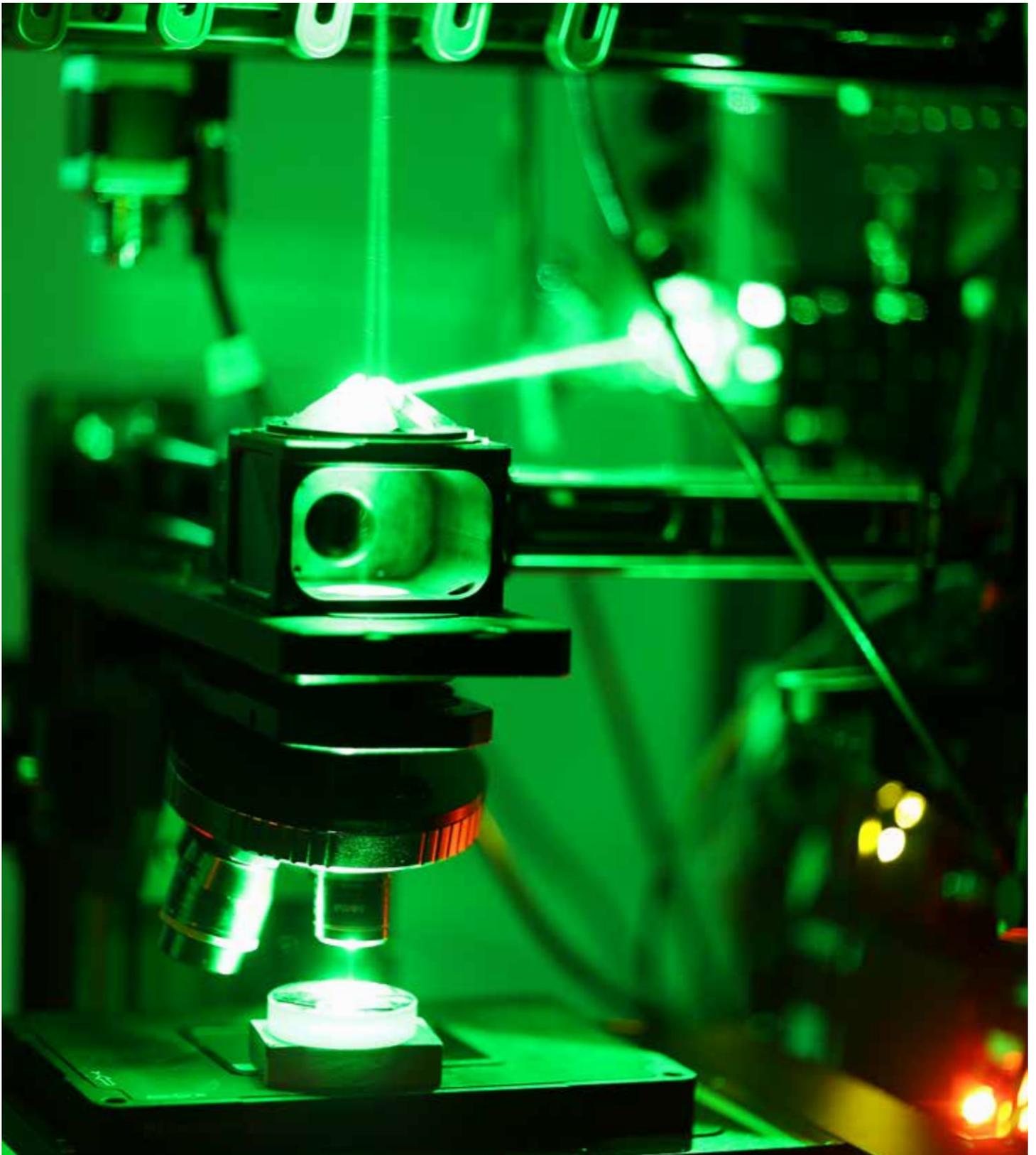
The main blocks for WL-SERS are the following:

- (i) Laser beam delivery system where the laser point focus, diffraction limited line and wide line laser illumination modes are implemented.
- (ii) Epi-detection-based microscope for simultaneously obtaining an optical image and a laser beam illumination profile on the surface of the SERS substrate.
- (iii) Raman beam delivery system capable of projecting an aberration-corrected laser line image onto a spectroscopic imaging sensor.

Laser beam delivery

The laser beam delivery layout consists of two geometries: the first geometry is a point illumination mode and the second geometry is for producing the DLL and WL sample illumination modes. The DLL-focus mode is achieved when a sample is illuminated by a diffraction limited laser line, that is, $d_{DLL} = 2.8 \mu\text{m}$ in width using a 785 nm laser excitation wavelength and 10 \times magnification lens. The WL mode is obtained by expanding the width of the laser line, that is, $d_{WL} > 2.8 \mu\text{m}$ and up to 64 μm , which is achieved by adjusting the position of a cylindrical lens in the laser delivery path. The laser beam profile is modified using a set of cylindrical lenses including laser line generator lens. In order to image the entire ≈ 2 mm long DLL/WL laser profile on a sample, the aperture for the laser beam was designed to fit the field of view (FOV = 2.2 mm) of the 10 \times near-infrared (NIR) microscope objective.





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