**Abstract:** 2D materials are state-of-the-art in nano- and opto-electronics. Characterizing their structural properties with a non-destructive approach at the micron scale is very important. We demonstrate in this paper how LabRAM Soleil™ confocal Raman multimode microscope is the perfect tool for these materials characterization.

**Keywords:** Raman microscopy, Ultra-low frequency, Photoluminescence microscopy, Multimode platform, WS₂ flakes.

**Introduction**

2D materials combine the strong covalent in-plane bonding with weak van der Waals (vdW) out-of-plane interactions. These materials exhibit remarkable thermoelectric, conductive, superconductive, optical, and photovoltaic characteristics, and hold potential for applications in nanoelectronics and optoelectronics devices. Various 2D materials have been explored, including insulators (h-BN), semiconductors (MoS₂, MoSe₂, WSe₂), and others.

The characterization of materials is important to identify and explore their structure. Raman spectroscopy is a powerful and non-destructive characterization tool to study 2D materials. It has the capability to characterize the structural properties of 2D materials, as well as detecting the layer thickness, band structures, strain effects, doping type, concentration, electron–phonon coupling, and interlayer coupling.

In this paper, we present an application of hyperspectral micro-spectroscopy for WS₂ nanostructures characterization by both Photoluminescence and Ultra-low frequency Raman analyses.

**Instruments and methods:**

Our HORIBA LabRAM Soleil™ is a Raman multimode microscope offering the highest throughput in the market with no compromise on resolution. This is a consequence of the unique optical design of this microscope based on dielectric mirrors, with very low signal loss, coupled with high quality gratings, our main expertise. These outstanding characteristics are required to obtain the best quality spectra needed for both Photoluminescence and ultra-low frequency Raman analyses.

By definition, photoluminescence is the most appropriate technique for band structures characterization at the micron scale. Indeed, the luminescence band of a semiconductor informs directly about the bandgap energy. Furthermore, the Raman analysis very close to the laser line allows a precise characterization of the number of layers of a 2D material. Indeed, specific interlayer vibration modes are excited in this spectral range. Being able to have both spectroscopy techniques, photoluminescence and ultra-low frequency Raman on the same instrument, is an important feature to characterize these materials as much as possible. Thanks to the fully automated modules for photoluminescence (PL) and ultra-low frequency (ULF) available on LabRAM Soleil™, it becomes easy to reach alternatively 5 cm⁻¹ frequency and high wavelength range.
Results

First of all, we characterized the band structure of the chosen flake acquiring its PL map. The resulting distribution is presented in Figure 2. Two main areas are observed. They are differentiated by their exciton band positions and intensities. The characteristic spectra shown on Figure 2 for the 2 main areas suggest a monolayer area (purple) and a multilayer area (yellow). Each of them have a specific bandgap energy of respectively 1.918 eV and 1.923 eV.

In order to characterize the exact number of layers and their distribution over the flake, we directly investigated the ULF range of the Raman spectrum. This spectral range is known to present the interlayer modes, expressing the coupling between layers. We acquired a Raman map of the flake focusing on the ULF region, presented in Figure 3. Two interlayer modes are so highlighted. The first one (12-15 cm\(^{-1}\)) shifts from higher to lower frequencies, increasing the number of layers. It corresponds to a stretching mode (S mode). The second mode, a lateral bending mode (LB mode), (20-22 cm\(^{-1}\)) goes on the other way. Based on these changes of position of modes, we can easily differentiate the number of layers applying a multivariate analysis of all spectra (MCR tool of MVAPlus LabSpec 6 software suite). It results in the colors of the ULF Raman map of the flake (Figure 3). The number of layers are so easily differentiated and localized on the sample at the micro-scale. Comparing with PL map, it confirms that the photoluminescence effect is quenched on multilayer areas, and adds the counting step on the number of layers.

Conclusion

Correlated PL and ULF microscopies enable a precise structural characterization of 2D materials, as demonstrated on this WS\(_2\) sample. Being able to switch from one to the other spectroscopy technique in a mouse click, is a very important point to consider for such an application.