X-ray excited optical luminescence coupled with high-resolution X-ray diffraction on individual monolayer WSe_2 flakes

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Application Note

Introduction

For in-situ and/or in-operando studies on highly localized and anisotropic phenomena the correlation of functional response to structural changes remains a challenge. A combined investigation of novel materials functional properties, such as photoluminescence, together with their structural properties, e.g. by X-ray diffraction (XRD) analysis is therefore of high interest.

As an example for the performance of the system we measured monolayers of transition-metal-dichalcogenides (TMDCs), here WSe_2. The TMDCs family presents a native bandgap ranging from the ultraviolet to the infrared , a rather large in-plane electrical carrier mobility (up to 200 $cm^2\ V^{-1}\ s^{-1}$), strong light-matter interaction, and strong exciton-binding energies up to 1 eV at room temperature, which makes them ideal candidates for optoelectronic and photonic applications [1]. In addition, the band structure in WSe_2 shows a transition from an indirect band-gap in bulk to direct one for a single monolayer. As a consequence, WSe_2 monolayer flakes exhibit bright luminescence properties [2].

Experimental configuration

In the center of the system stands a six-circle diffractometer that allows a full strain characterization of crystalline specimen by recoding three dimensional reciprocal space maps for a set of Bragg reflections. Therefor a collimated highly brilliant X-ray beam impinges on the sample. By rocking the sample around the bragg condition, the strain state of the sample is investigated. Simultaneously the incident X-ray beam excites optical luminescence that is emitted by the sample, as depicted in Fig. 1. The optical collection system aims to increase the photon yield and minimize signal losses by making use of a collimating optics and a glass fiber to transport the light into the spectrometer.

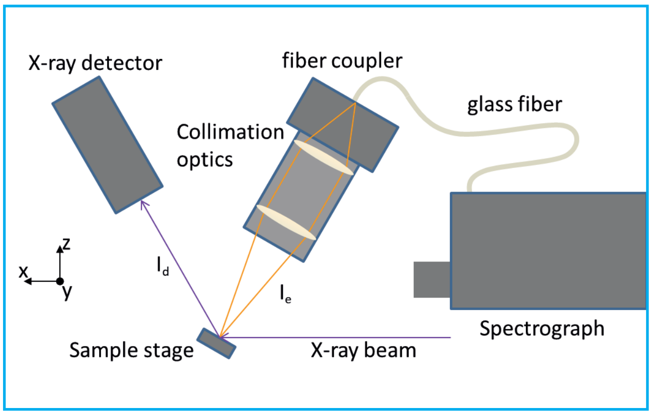

Figure 1: Schematic of the XEOL combined XRD configuration, indicating the emitted signal (I_e) and diffracted signal (I_d) while the sample is mounted onto a six-circle diffractometer, and inclined by ~30 deg.

With a protective silver coating on all spectrograph mirrors highest reflectivity is achieved for a wavelength range from 400 nm to 1000 nm. A turret holding three different gratings, 300 l/mm, 600 l/mm and 1200 l/mm allows sufficient flexibility from wide range, overview spectra, to highly resolved spectra, respectively. While the Andor multicore quartz glass fiber offers a core diameter of 200 µm (NA = 0.22) with an operation window from UV to VIS.

Sample preparation and first measurements

First measurements with the described collection system were carried out at beamline P08 of the German Electron Synchrotron (DESY), in Hamburg. The portable set-up of the optical collection system allows for simple adaptation to different experimental end stations and a quick exchange. The 2D material system was excited with a monochromatic 15 keV x-ray beam.

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The sample was produced by mechanical exfoliation to obtain flakes of WSe_2, with sizes in the order of (5 x 10) μm². Such flakes were transferred to patterned Si (111) substrates with Au markers to identify individual flakes. An x-ray beam focused down to a size of (3 x 30) μm² allowed to excite optical luminescence from a single WSe_2 Monolayer, as seen in the inlay of Fig. 2. A full spectrum was recorded with an integration time of 100 s in single photon counting mode and applying background correction (Fig. 2.)

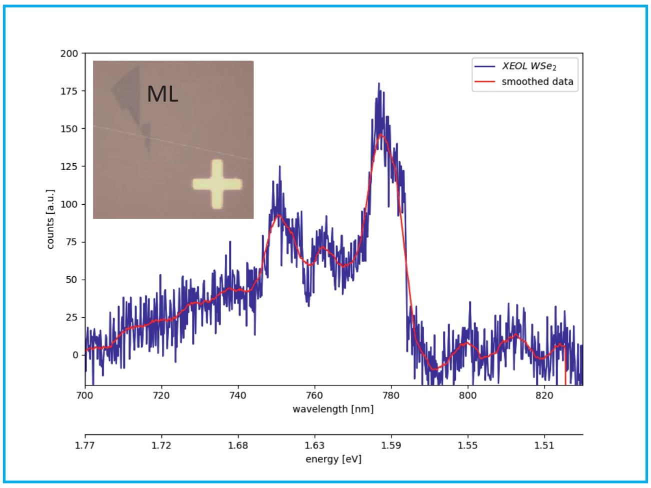

Figure 2: XEOL spectra from a single WSe_2 Monolayer at RT. The inlay shows the specimen next to a neighboring Au marker, with a length of 20 μm, under an optical microscope.

Conclusion

XRD provides a model free insight to structural configurations with an exceptional flexibility for in-situ and in-operando measurements. At the same time the specimens' functional response can be probed at the very same location and state.

The Andor iVac DR316B-LDC-DD CCD detector mounted on a Shamrock SR-303i-B-SIL spectrograph allows detecting full spectra from a single monolayer of WSe_2, excited by highly brilliant X-rays. The combination of the individual components makes this system highly flexible for user-operation in a wavelength range from 400 nm to 1000 nm and can be used for the characterization of different material systems.

References

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