	Experiment title: Grazing incidence XEOL	**Experiment number:** A32-2-849
Beamline: BM32	**Date of experiment:** 1 to 4 March 2022	**Date of report:** 13/02/2023
Shifts: 9	**Local contact(s):** Jean-Sebastien MICHA; Samuel TARDIFF; Olivier ULRICH;	*Received at ESRF:*

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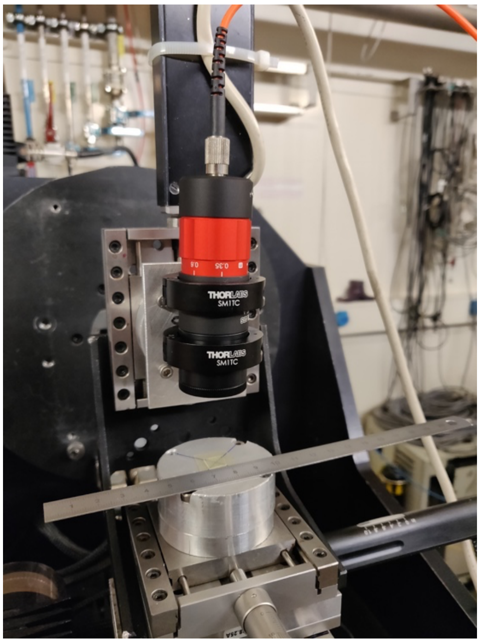
1/ Recall of objectives:

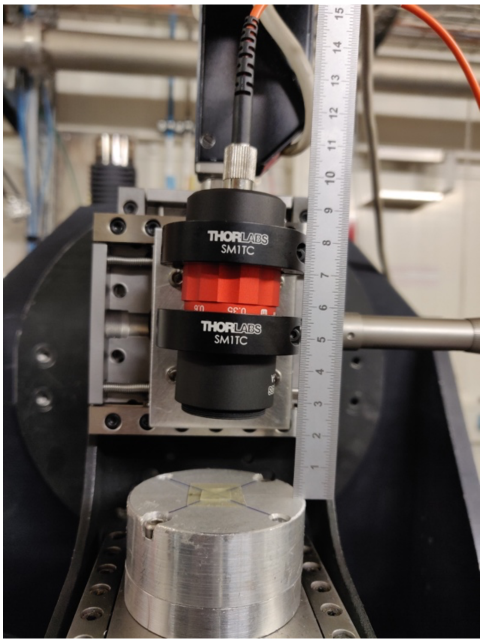
To the best of our knowledge, no XEOL experiments had already been done with the GIXRD geometry. For the first time, this proposal was targeting this to take advantage of the total reflection properties of X-rays.

2/ Experimental method:

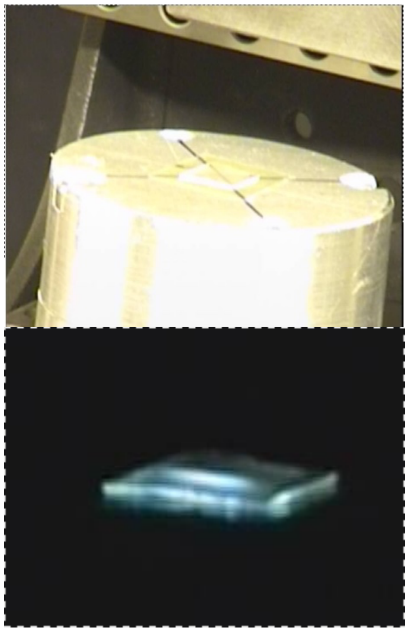
We tested on the GMT diffractometer a new home-made equipment to measure the XEOL signal (i.e., to record the light emission) within the GIXRD geometry. The emitted photons have been collected first by a visible Hastings achromatic triplet lens positioned on top of the surface. In this configuration, the output parallel beam is focalized by an achromatic fiber collimator, and coupled to a multimode optical fiber going to a QEPRO Oceanview spectrometer. In a second step, we removed the Hastings triplet (the first element after the sample) to collect more light from the surface without angular filtering. The fiber output is then dispersed by a diffraction grating (HC1-GE) on a back-illuminated pixels CCD (SCMOS) camera cooled with a Peltier device. This system has been optimized to accommodate a polychromatic light in the 350-650 nm range.



First mounting with achromatic lens.



Second mounting without achromatic lens.



A sample showing the beam fingerprint of light.

It also allows large angle Bragg diffraction with about 40° incidence and emergence.
The Bliss server to synchronize the exposure and counting time (also acquiring detector background) has been fully implemented for usual XEOL experiment.
To align the beam footprint with the optical collection axis, we decide to mount the detection on X,Z translations and the sample on X,Y translations. We have checked that these alignments must be done very accurately with the Hastings lens, and that this constraint is relaxed without.
The nominal height of the beam was about 44 µm. Three samples with InGaN/GaN multiple quantum wells have been measured with different QW number (i.e. thickness of the active emitting layer) and In composition (i.e. wavelength emission): T2080 (30QW 18%In); T2133 (100QW); T1408 (15 QWs In 10%)

3/ Results (for illustration purpose, only without the Hastings lens):
The experiment was really successful and we learn a lot of things, in agreement with expectations:
- *Demonstration of the advantage of the grazing incidence geometry to improve exciton generation by several order of magnitude compared to the usual geometry.*

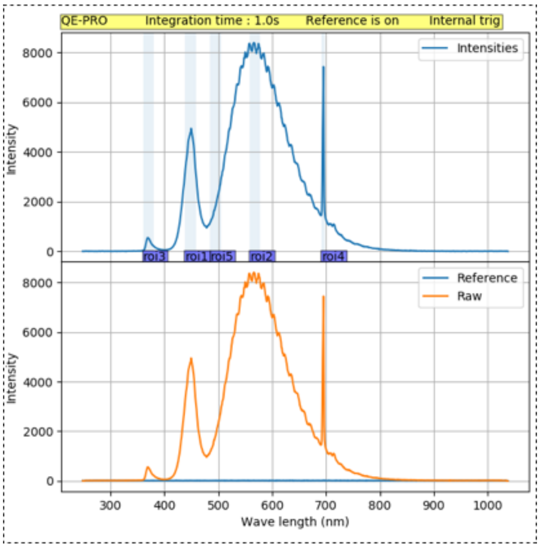


Fig. 1. Example of XEOL signal (T2080).

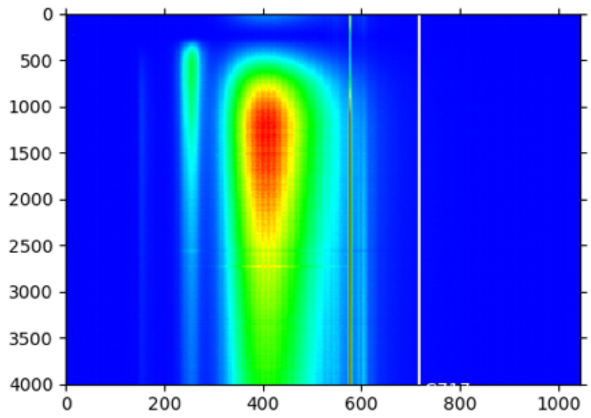


Fig. 2. XEOL Mapping changing the incidence angle (T2080).

Four signals are recorded:
- the small near-band edge emission at 380 nm.
- the MQW emission at 425 nm
- the defect-band (yellow at 560 nm)
- a sharp peak coming from the sapphire substrate.

As shown in the figure below, the MQW emission is strongly enhanced close to the grazing incidence angle. It is explained by the position of the MQW close to the surface. On the contrary, the yellow band, coming from defects is distributed in the depth of the sample, e.g. in the thick GaN buffer on top of the sapphire substrate. The maximum of intensity is therefore at much higher angle.

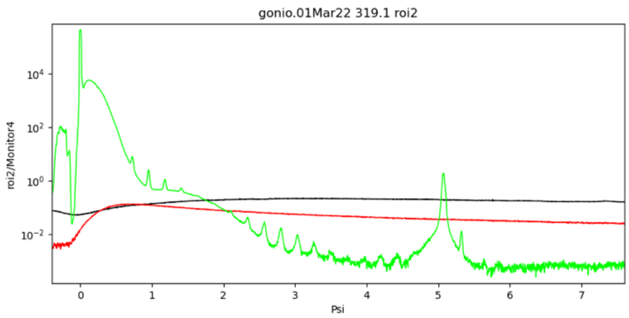


Fig. 3. Diffuse X-ray reflectivity signal with XEOL MQW (red) and defect band (black). Phi is the emergence angle (deg.).

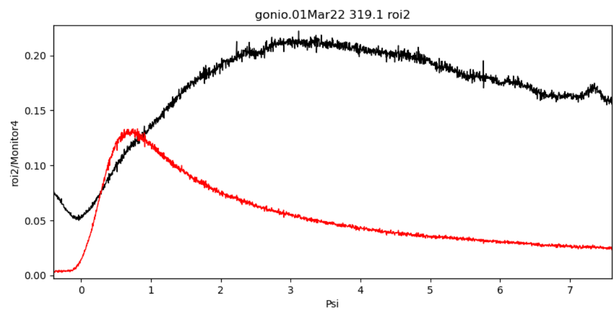


Fig. 4. Zoom of XEOL signals with MQW (red) emission and defect band (black). Phi is the emergence angle (deg.).

- Quantitative measurements of the influence of the incidence angle. Comparison with a theory similar to the de Boer treatment for glancing-incidence x-ray fluorescence of layered materials. For this proposal, we will not change the energy to cross the Ga absorption edge, it could be an extension for next studies.

As show in Fig. 3, the shape of the MQW XEOL intensity has a shape similar to what is obtained with the glancing-incidence fluorescence of thin layer. This shape will be quantitatively fitted.

- Measurements of light emission efficiency while scanning the Bragg conditions under GIXRD and symmetrical geometries. Do diffraction conditions play a role in XEOL intensity?

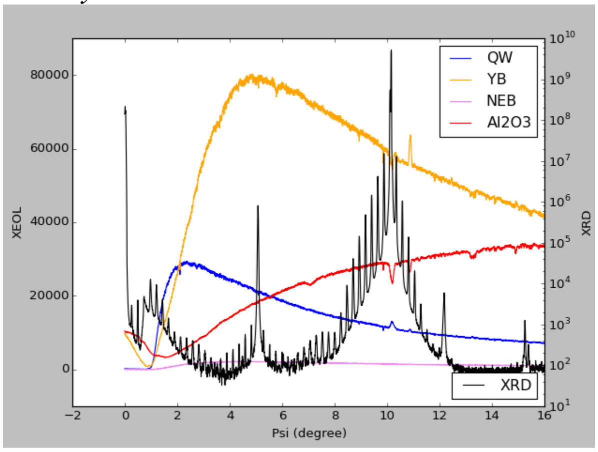


Fig. 5. X-ray reflectivity signal and XEOL signals. Psi (deg.) is the emergence angle. (T2080)

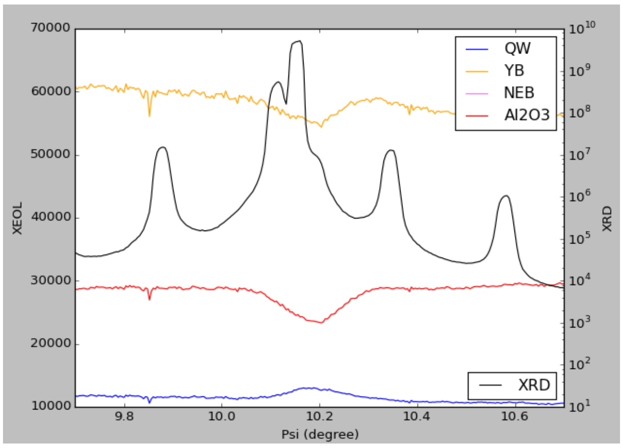


Fig. 6. Zoom of XEOL intensity changes close to the second order peak. Psi is the emergence angle.

- A new route for XEOL measurements that could be used for measuring surface emitters. And in particular low emitters that can directly benefit from this approach.

The analysis will be carried out to analyse quantitatively the differences resulting from the sample features (thickness and composition), and the grazing incidence effect that is very clear (for example in Fig. 4). The lost of intensity evidenced in Fig. 6 has to be explained. An assumption is that the 0-order peak of the superlattice correspond to a high reflection, that increase the number of photons responsible of XEOL intensity in the MQW.

As expected, this effect (6-times intensity in Fig. 4) can be used to have XEOL signal on low-emitter materials.

We are really happy that all the expected results of the initial proposal have been demonstrated. It corresponds also to a significant improvement of the BM32 possibilities. A publication is under way to communicate these results in a letter.