| ESRF | **Experiment title:** XEOL and µLaue measurements of Nitride Wires and Microdisplays | **Experiment number**: MA 4182 |
|---|---|---|
| **Beamline**: | **Date of experiment**: from: 7th to: 11th Sept. 2018 | **Date of report**: 24th Feb. 2020 |
| **Shifts:** | **Local contact(s)**: J.-S. Micha | *Received at ESRF:* |
| **Names and affiliations of applicants** (* indicates experimentalists): **O. Ulrich*, O. Robach*** | | |

**Report:**

X-ray excited optical luminescence (XEOL) is an original technique to get information on the intrinsic optical properties of materials. For the first time in the world, we combined XEOL with the µLaue structural analysis on the French CRG-IF BM32@ESRF beamline taking advantages to recording a diffraction pattern without aligning the sample and measuring at the same time the optical emission (a few second counting time is needed for good emitting crystals). The sub-micrometer resolution range provided by the Laue setup is also complementary with ESRF nanobeams (eg. ID16B) usually performing fluorescence-emission mapping on very small areas (a few µm$^2$).

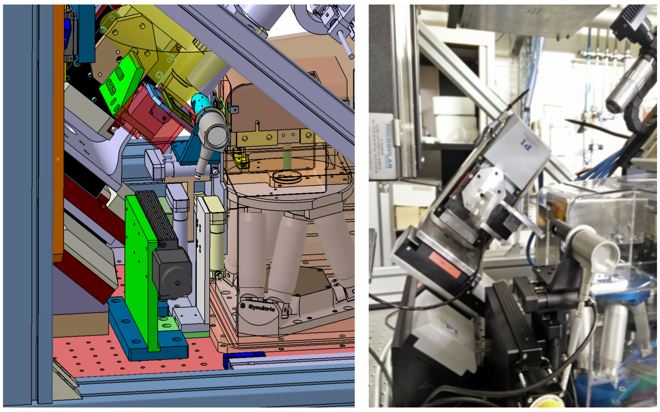

*Fig. 1. Drawing and implementation of the XEOL measurement on the BM32@ESRF µLaue setup.*

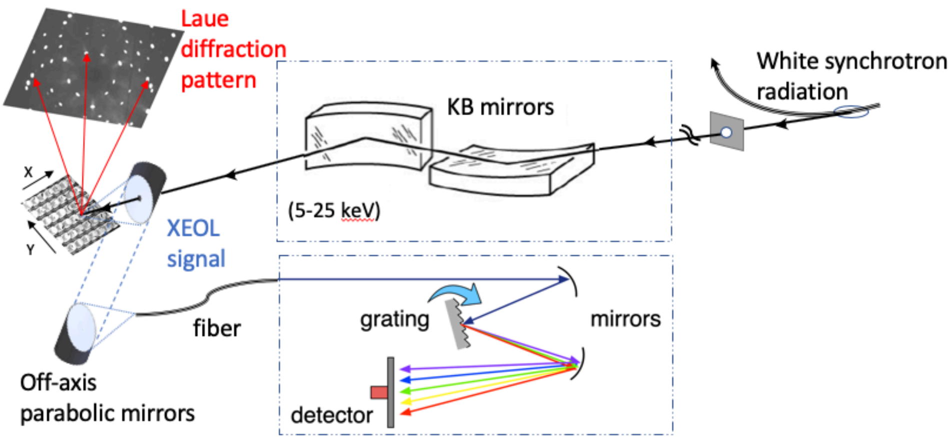


*Fig. 2. Schematics of the XEOL/µLaue setup.*

In the framework of the Bottom-Up project of CEA called LumiX (proposed by the NRS lab.), we developed a new setup on the diffractometer to record the light emission (see the schematics in Fig. 2). The emitted photons are collected by an off-axis parabolic Al-mirror (X-rays are going through the mirror), they are then focalized by a second parabolic mirror to the entrance of an optic fiber going to a QEPRO Oceanview spectrometer. This light is then dispersed by a diffraction grating on a back-illuminated pixels CCD camera cooled with a Peltier device. During the experiment, we have a shutter mechanism to select the acquisition time and to acquire the background. The entire system (computer server, acquisition and analysis software) has been optimized to measure the visible range emission wavelength (see the setup overview in Fig. 1).

Data analysis are still under way and not published, but we performed first experiments with InGaN/GaN core-shell microwires and microdisplays.

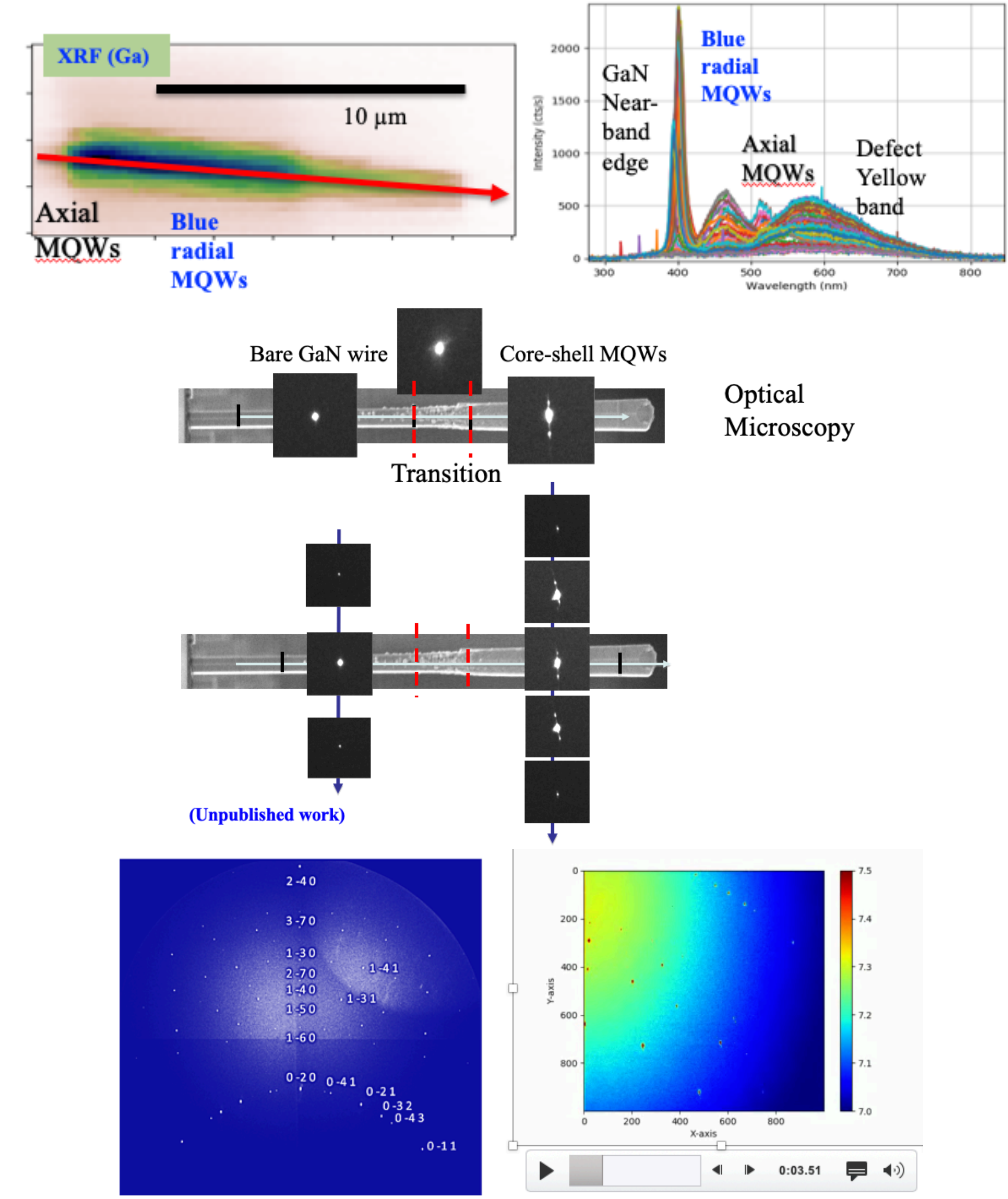


*Figure 3: Top: XRF mapping of Ga element and example of a line-scan showing 4 components of the XEOL emission: the GaN near-band edge, Blue radial MQW, axial MQW and the defect yellow-band. Middle: examples of the evolution of a single Bragg Laue peak related to the MQW. Bottom: example of a full Laue pattern and a zoom on a quadrant.*

In a systematic way, as illustrated in Fig. 3 for wires, we performed in parallel XRF, XEOL and µLaue mappings and related videos. It provides a link between the emission and the spatial position along the wires, in particular we visualize the existence of radial (sidewall m-plane) MQW emission but also longitudinal one at the to of the wires. The µLaue mapping provides the evolution of the Bragg peaks related to the GaN stem, but also the existence of MQW-peak satellites in the core-shell part. Interestingly we have seen a rotation of the crystal along its length. The quantitative analysis of this rotation and of the strain value in the mapping is under way.

µLEDs (micro-light-emitting diodes) are an emerging display technology that uses very small LEDs for pixels..

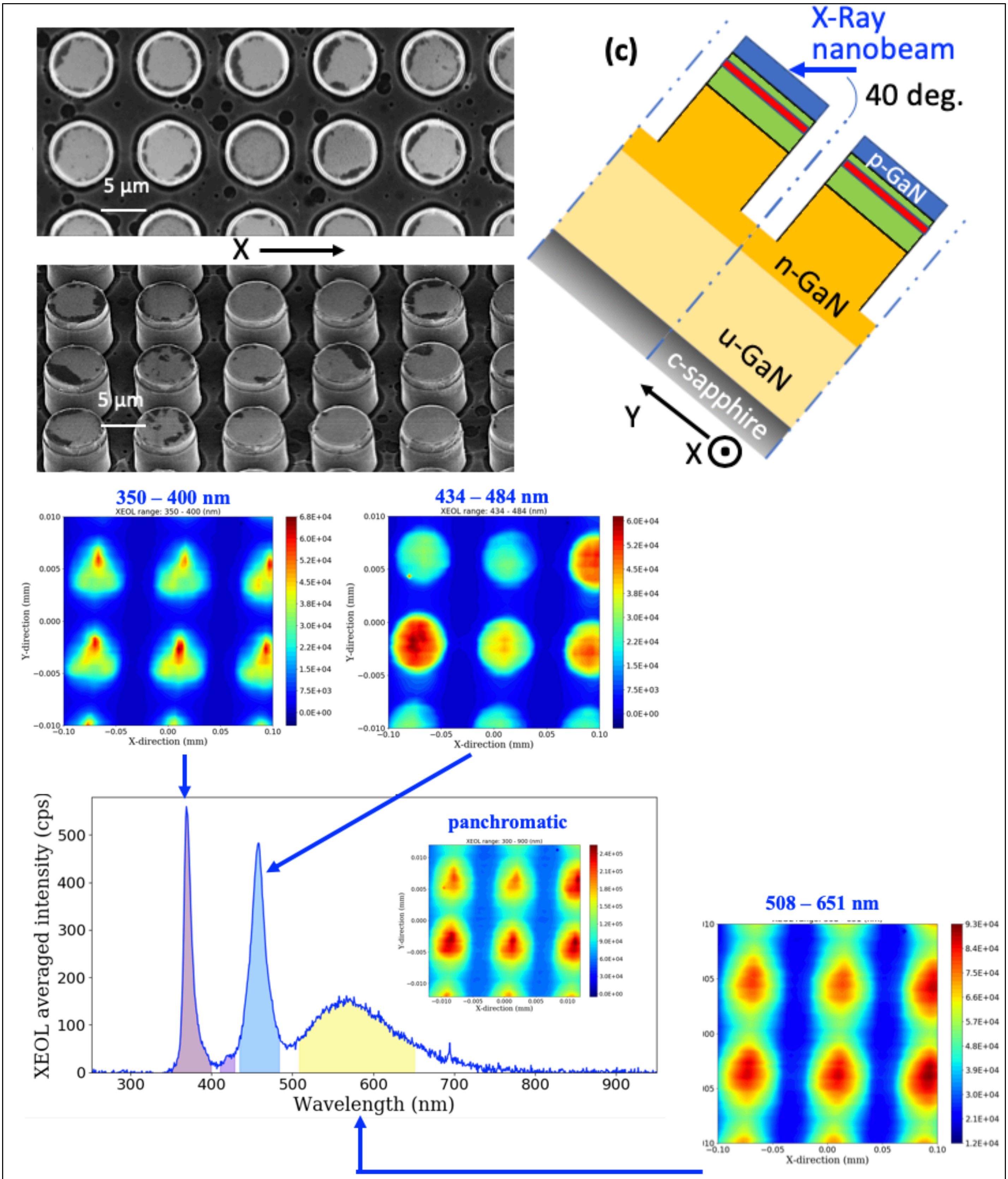


*Figure 4: Left: XEOL/µLaue measurement schematics. SEM-view of µLED samples and schematics. Right: XEOL hyperspectral mappings of the µLEDs showing the panchromatic spectrum and image, and the individual mappings of near-band edge, blue and defect-band emission.*

Much the same as any other current display technology, it combines red, green and blue sub-pixels to reproduce colour. Although there are currently no µLED displays in mass production today, there is a massive motivation for the technology to penetrate major display markets and replace incumbent LCD and OLED technologies in a wide range of applications (HDTVs, smartphones, smart watches, and head-up displays, virtual reality and augmented reality headsets). However, many development challenges remain before µLEDs can fully realize their potential (greater efficiency and longevity at low current density). Nitride materials are well-positioned to fulfil this We studied 6 µm and 7 µm LEDs processed by the CEA/Leti as a benchmark of their state-of-the-art technology.

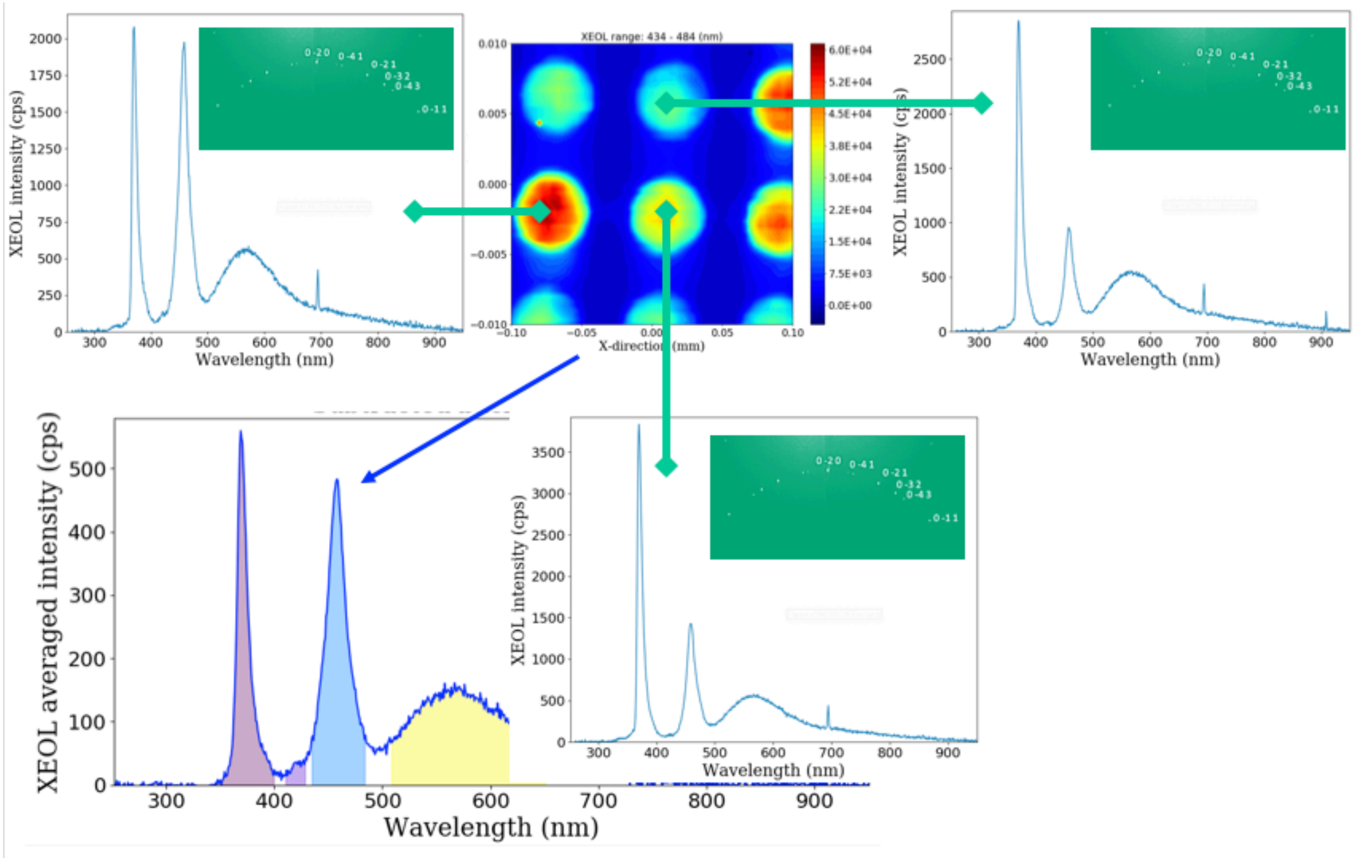


*Figure 5. Examples of XEOL spectra and µLaue diffraction pattern extracted from the positions at the center of 3 µLED pixels. The mapping image is obtained for the 434-484 nm range.*

During the first experiment, we developed the full acquisition chain and hyperspectral analysis with linux servers and home-made Python codes as demonstrated in Figures 4 and 5. It allowed a precise mapping of the pixel and the correlation between light emission and strain/defect is under way. We changed of detector and data corrections are still under way.
An interesting point corresponds to the fact that a statistical analysis can be done with this method. The fast acquisition allows measuring hundred of µLEDs and have a simultaneous view on the dispersion of emission wavelength and strain.