ESRF	**Experiment title:** **Optoelectronic properties of individual InGaN nanowires studied by simultaneous X-ray fluorescence and X-ray excited optical luminescence spectroscopy**	**Experiment number**: MA-4089
Beamline: ID16B	**Date of experiment**: from: 30/03/2018 to: 02/04/2018	**Date of report**: 21/02/2020
Shifts: 9	**Local contact(s)**: D. Salomon	*Received at ESRF:*

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Report:

The aim of this experiment was to study the composition-luminescence relationship of $In_xGa_{1-x}N$ nanowires (NWs) by simultaneous X-ray fluorescence (XRF) and X-ray excited optical luminescence (XEOL) mapping of single NWs. For this purpose, InGaN NWs were grown by hydride vapor phase epitaxy (HVPE). For the X-ray excited optical luminescence and X-ray fluorescence measurements, the NWs were transferred from their growth substrate onto a SiN membrane. The NWs were then located by optical microscopy and more precisely by X-ray fluorescence mapping employing a 29.6 keV pink X-ray beam which was focused down to 70 x 70 nm^2 using KB mirrors.

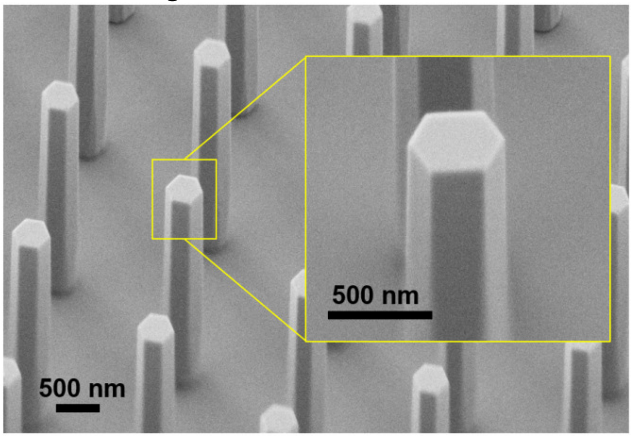

Fig. 1: Tilted-view SEM image of InN NWs.

While XRF mapping of the InGaN NWs did not show any compositional gradients, X-ray excited optical luminescence spectroscopy was not successful. The fact that no luminescence spectra could be recorded may be caused either by the small NW diameter and, thus a very small volume of matter that is excited by the focused X-ray beam leading to luminescence intensity below the detection threshold or by a luminescence wavelength that is out of the detectable range of the spectrometer.

On the other hand, we investigated pure InN NWs with a diameter of 500 nm (see Fig. 1), which were synthetized by hydride vapor phase epitaxy using the selective areea growth approach, by XRF mapping. For this purpose, the as-grown NWs were mapped on the top and NWs transferred on a SiN membrane were mapped along their growth axis. XRF elemental mapping of In-Kα of the top of InN nanorods is displayed in Fig. 2(a). It does not reveal any InN formation on the SiN_x mask. This indicates that perfect selective area growth conditions are achieved in HVPE growth environment. The growth temperature, which is high enough in our growth conditions, is the main parameter to achieve a perfect selectivity by taking advantage of the adatom sticking coefficient difference of the chloride precursors on the SiN_x mask and the GaN/c-Al_2O_3 template. Additionally, a low indium intensity in the core of the nanorods compared to their edges can be seen. XRF In-Kα mapping recorded along individual dispersed InN NWs revealed a lower In content at the base (except edges) than at the top (Fig. 2(b)). These findings indicate that the NWs actually start to grow as a tube and only after reaching a length of ~500 nm, the growth of a compact NW starts. These findings were confirmed by transmission electron microscopy of a vertical cross-section of an InN NW as demonstrated in Fig. 2(c).

These results were recently published in CrystEngComm [1] and presented in different conferences [2,3,4].

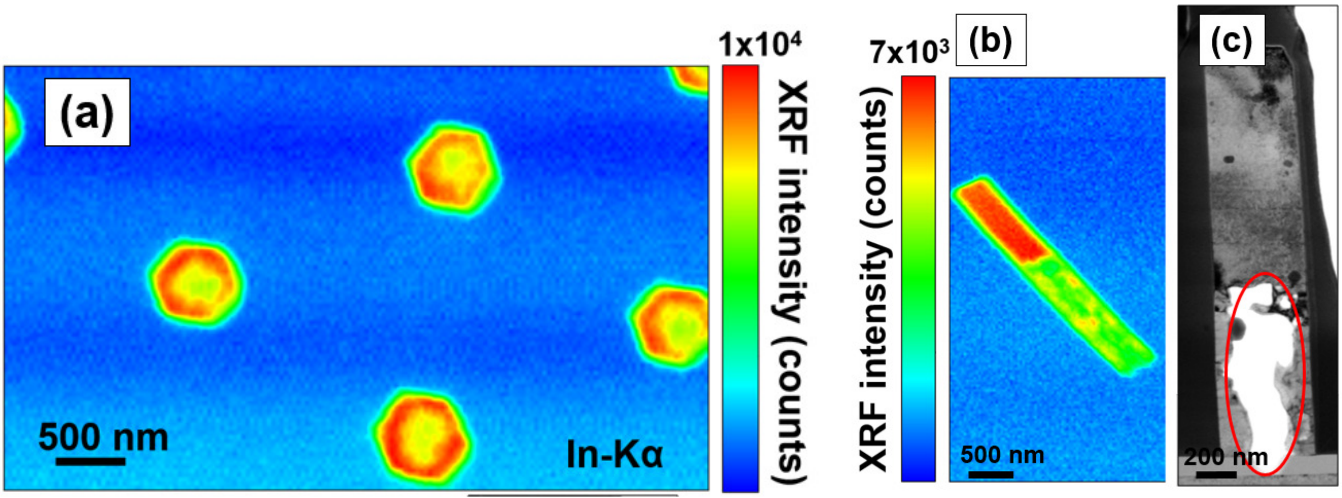

Fig. 2: (a) XRF map of In-Kα distribution measured from the top of as grown InN nanorods: there is no InN nucleation on the SiN_x mask. (b) XRF map of In-Kα distribution measured on dispersed InN nanorod indicating that the indium distribution is different between the top and the base of the nanorod. (c) Bright-field TEM image along the [11-20] zone axis of InN nanorods: the red circle evidences the presence of a void.

References

[1] M. Zeghouane, G. Avit, T.W. Cornelius, D. Salomon, Y. André, C. Bougerol, T. Taliercio, A. Meguekam-Sado, P. Ferret, D. Castelluci, E. Gil, E. Tournié, O. Thomas, A. Trassoudaine, *Selective growth of ordered hexagonal InN nanorods*, CrystEngComm 21 (2019) 2702 – 2708

[2] M. Zeghouane, Y. Andre, G. Avit, T.W. Cornelius, D. Salomon, C. Bougerol, D. Castelluci, E, Gil, P. Ferret, O. Thomas, V. G. Dubrovskii and A. Trassoudaine. HVPE growth of InGaN nanowires with a controlled indium mole fraction and selective area growth of well-ordered InN nano- and microwires. Les Journées Nano, Micro et Optoélectronique (JNMO), Agay, Juin 2018. *Poster presentation*

[3] M. Zeghouane, G. Avit, Y. André, T.W. Cornelius, D. Salomon, C. Bougerol, P. Ferret, D. Castelluci, E. Gil, O. Thomas, A. Trassoudaine. Selective growth of (In,Ga)N nanowires by HVPE. Session plénière du GDR PULSE, Clermont-Ferrand, 1-4 Juillet 2019. *Oral presentation*

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