# European Synchrotron Radiation Facility

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON



# **Experiment Report Form**

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal: <u>https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do</u>

## **Deadlines for submission of Experimental Reports**

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

## Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a "preliminary report"),

- even for experiments whose scientific area is different form the scientific area of the new proposal,

- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

#### **Deadlines for submitting a report supporting a new proposal**

- > 1<sup>st</sup> March Proposal Round 5<sup>th</sup> March
- > 10<sup>th</sup> September Proposal Round 13<sup>th</sup> September

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

#### Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

#### **Published papers**

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

#### **Instructions for preparing your Report**

- fill in a separate form for <u>each project</u> or series of measurements.
- type your report in English.
- include the experiment number to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	<b>Experiment title:</b> Origin of luminescence bands in Ga <sub>2</sub> O <sub>3</sub> -based nanowire complex architectures and thermal assessment of single nanowire	Experiment number: MA-5727
Beamline: ID16B	<b>Date of experiment</b> : from: 12/07/2023 to: 17/07/2023	Date of report:
<b>Shifts:</b> 15	Local contact(s): Valentina Bonino	Received at ESRF:
Names and affiliations of applicants (* indicates experimentalists): Emilio Nogales <sup>1*</sup> , Daniel Carrasco <sup>1*</sup> , Paula Pérez <sup>1*</sup> , Jaime Dolado <sup>2*</sup> , Bianchi Méndez <sup>1*</sup> <sup>1</sup> Departamento de Física de Materiales, Facultad de Ciencias Físicas, Universidad Complutense de Madrid, Spain; <sup>2</sup> ESRF Grenoble, France		

# **Report:**

Nanoelectronic devices based on semiconductor nanowires have been studied for decades, in order to develop applications in a very wide range of areas, mainly with simple nanowires and, in some cases, with more complex systems, such as crossed nanowires made of two different semiconductors that result in point-like heterostructures [1]. For this work, unique  $Ga_2O_3/SnO_2$  crossed nanowires (NW) heterostructures were developed with a single step thermal treatment [2], which combine different luminescence bands in the same architecture. We report the study of the origin of their luminescence bands using a hard X-ray nanobeam under resonant excitation conditions.

The use of correlative X-ray excited optical luminescence (XEOL) and X-ray fluorescence (XRF) analysis at the nanoscale was led in order to gain knowledge on the luminescence excitation and radiative mechanisms in the Ga<sub>2</sub>O<sub>3</sub>/SnO<sub>2</sub> crossed NW heterostructures. We have studied the XEOL emissions from these hybrid structures (see Figure 1), obtaining strong signals from both kinds of nanowires that form them. Each type of nanowire shows different bands. The XEOL emission from the central, Ga<sub>2</sub>O<sub>3</sub> nanowire (point 2 in figure 1(b), red line in figure 1(c)) is centred in the ultraviolet (UV). It is characteristic of undoped Ga<sub>2</sub>O<sub>3</sub> and presents mainly two components (3.3 and 3.0 eV) related to bound excitons and donor-acceptor pair (DAP) transitions [3]. On the other hand, a red-orange band (1.95 eV) dominates the XEOL spectrum from the  $SnO_2$  wires (point 1 in figure 1(b), green line in figure 1(c)). This band is commonly attributed to oxygen vacancies in SnO<sub>2</sub> bulk material [3].

Besides, a clear dependence of the XEOL-detected XAS measurements with the electric field vector of the X-ray nanobeam was obtained in the Ga<sub>2</sub>O<sub>3</sub> nanowires (see Figure 2). However, a further analysis of these features is needed to



**Figure 1: : (a)** SEM image of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>/SnO<sub>2</sub> crossed nanowires. (b) XEOL map in RGB visualization that depicts the XEOL intensities of visible (green) and UV (red) emissions. (c) XEOL spectra recorded over the SnO<sub>2</sub> (green) and Ga<sub>2</sub>O<sub>3</sub> (red) nanowires.

answer the open questions that remain in relation with the interface and point defects of the materials, which are key to optimize the photonics applications of this crossed-nanowire system in electronics and optoelectronics.



Figure 2: Ga K-edge XAS in fluorescence and luminescence detection modes recorded along the central  $Ga_2O_3$  nanowire with the *c*-axis oriented perpendicular (a), and parallel (b) to the electric field vector of the X-ray nanobeam.

On the other hand, an in-situ test of the increase of  $Ga_2O_3$  nanowires local temperature due to the X-ray nanobeam irradiation is also reported here. Recently, we showed a robust and reliable optical thermometer based on  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>:Cr micro- and nanowires in which tailored Distributed Bragg Reflectors (DBR) were used as optical mirrors to form optical cavities (see Figure 3a) [4]. Due to the fact that there are two different optical features that can be monitored to assess the temperature, one of which is observable from 0 K up to some 340 K and the other from some 100 K up to at least 550 K, a very wide range can be characterized with this system. This optical thermometer combines a micron range spatial resolution with the mentioned wide temperature range applicability. The spectral shift of the Fabry-Perot resonances that form sharp luminescence peaks on the red-NIR band provided by Cr<sup>3+</sup> dopant in Ga<sub>2</sub>O<sub>3</sub>, are the base for the observable quantities used for thermometry in this system. We have successfully studied the variation of the local temperature of the nanowires, through the shift of the Fabry-Perot resonances of the XEOL signal (figures 3(b) and 3(c)). A temperature increase of tens of Kelvin is observed when the higher X-ray fluxes impinge and heat the nanowire's cavity.



**Figure 3.** (a) SEM and micro-photoluminescence images of the DBR-based optical microcavity. (b) spectral shift of one of the XEOLdetected Fabry-Perot resonances obtained in the microcavity when irradiating with different X-ray photon fluxes. (c) XEOL peak shift as a function of the X-ray flux.

It is worth mentioning that further experiments, especially with the Ga<sub>2</sub>O<sub>3</sub>/SnO<sub>2</sub> crossed nanowires heterostructures, would mean a significant step towards having a more profound understanding of their luminescence and electronic mechanisms. These experiments include temperature-dependent and polarization-dependent features of X-ray absorption spectroscopy (XAS) and X-ray excited optical luminescence (XEOL) signals. An in-depth analysis of the X-ray beam-induced current (XBIC) signal generated in contacted Ga<sub>2</sub>O<sub>3</sub>/SnO<sub>2</sub> crossed nanowires will also be of the uppermost importance regarding their electronic properties. Employing a hard X-ray nanobeam, we plan to obtain a correlation between XAS, XEOL and XBIC signals. This multifaceted approach will unveil crucial information regarding their charge carrier dynamics and optical transitions, expected to shed light on the presence of electronic and optically active defects, which are key to potential optoelectronic applications.

#### References

[1] R.J. Hu, L.W. Yu, *Nanotechnology* **33**, 222002 (2022).

- [2] G. Martínez-Criado et al. Nano Letters 14, 5479-5487 (2014).
- [3] M. Alonso-Orts et al. Nano Letters 17, 515–522 (2017).
- [4] M. Alonso-Orts et al. *Small* 18, 2105355 (2022).