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Exploring physical properties of gallium oxide
micro- and nano-structures

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14. ABSTRACT <p>This project aims to study physical properties in undoped and doped Ga₂O₃ micro- and nanostructures through the light-matter interaction, those involved in light generation, absorption, and propagation mechanisms inside the Ga₂O₃. These three features constitute the specific objectives of the project.</p> <p>For the synthesis of undoped and doped Ga₂O₃ and eventually ternary (Al_xGa_{1-x})₂O₃ nanostructures with different architectures, the team used thermal evaporation methods with Chromium, Zinc and Germanium as dopants. The morphological and structural characterization of the obtained samples showed that the method is successful to create arbitrary features that are sufficient to generate the desired properties.</p> <p>Light generation of doped Ga₂O₃ under several injection beams (electrons, UV, visible light, and synchrotron X-ray) included time-resolved, polarized and temperature dependent measurements in some cases. The whole picture of the optical properties includes all recombination paths in the relaxation of excited carriers generated by the injected beams.</p> <p>Theoretical study of the electronic and vibrational states in undoped Ga₂O₃ and (Al_xGa_{1-x})₂O₃ has been carried out by adopting the hybrid-exchange density functional, screened-exchange Heyd-Scuseria-Ernzerhof model. The role of defects, oxygen, and gallium vacancies, in the electronic structure has been assessed in the (Al_xGa_{1-x})₂O₃ compound.</p> <p>Light absorption and transport phenomena. During the project, photoluminescence excitation spectra have been taken to determine the absorption which led to the main emissions of beta- and gamma- Ga₂O₃ nanoparticles. The work has allowed the team to determine the best protocol to achieve the electrical contacts and overcome some issues, such as early detachment of structures from substrates.</p> <p>Light confinement in undoped and Cr doped Ga₂O₃ nanostructures. During the project, DBR-based optical cavities have been built based on Ga₂O₃ microwires with hexagonal shaped cross-section. As a result, the team has produced optical microcavities with sharp Fabry-Perot resonances overlapping the PL spectrum, which agreed with the predictions of the simulations. In addition, temperature dependence studies of the optical resonances and Cr luminescence of the DBR-based cavities have been carried out. The results have shown the feasibility of a wide dynamic range thermometer based on this system. The cavities have been demonstrated to be robust and stable in time, which make them very promising for applications.</p>					
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FINAL PERFORMANCE REPORT

• Grant Number FA8655-20-1-7013

Exploring light-matter interaction in gallium oxide micro- and nanostructures

PI: Dr. Bianchi Méndez

Co-PI: Dr. Emilio Nogales

Period of performance: 1-06-2020 – 31-05-2023.

1. Project summary

• Objectives: This project aims to study physical properties in undoped and doped Ga₂O₃ micro- and nanostructures through the light-matter interaction, in particular those involved in light generation, absorption and propagation mechanisms inside the Ga₂O₃. These three features constitute the specific objectives of the project.

Light emission and absorption mechanisms have been studied in a complementary way by photoluminescence and photoluminescence excitation spectroscopies. On the other hand, light confinement in Ga₂O₃ nanostructures has been assessed both theoretically and experimentally by micro-PL imaging and spectroscopy.

This report summarizes the progress of the work related to the project tasks.

Task 1. Synthesis of undoped and doped Ga₂O₃ and eventually ternary (Al_xGa_{1-x})₂O₃ nanostructures with different architectures by thermal evaporation methods.

Based on the previous experience in single nanowires and plates, we can manage to control the shape of the microstructures somehow. During the project, the impurities of choice have been Cr, as optically active ion, and Zn and Ge impurities, which are used to get intentionally p-type and n-type material, respectively. In addition, first trials on the synthesis of (Al_xGa_{1-x})₂O₃ nanostructures were performed although still no conclusive. Finally, thanks to a collaboration with a chemistry group, we have synthesized beta- and gamma- Ga₂O₃ nanoparticles by a precipitation method.

The morphological and structural characterization of the obtained samples have been carried out by the available techniques in the group. In summary, the task has been accomplished.

Task 2. Light generation of doped Ga₂O₃ under several injection beams (electrons, UV-vis light and synchrotron X-ray).

The study has included time-resolved, polarized and temperature dependent measurements in some cases. The whole picture of the optical properties includes all recombination paths in the relaxation of excited carriers generated by the injected beams. The luminescence under hard-X ray irradiation has been initiated, with beamtime granted at ESRF (Grenoble) in the next months. Therefore, all subtasks have been accomplished.

Task 3. Theoretical study of the electronic and vibrational states in undoped Ga₂O₃ and (Al_xGa_{1-x})₂O₃.

The study has been carried out by adopting the hybrid-exchange density functional, screened-exchange Heyd-Scuseria-Ernzerhof (HSE06). This functional is well documented and provides a quantitatively correct description of the electronic structure of oxide semiconductors. In particular, the DOS of defect-free Ga₂O₃ and (Al_xGa_{1-x})₂O₃ has been calculated. In addition, the role of defects, oxygen and gallium vacancies, in the electronic structure has been assessed in the (Al_xGa_{1-x})₂O₃ compound.

Task 4. Light absorption and transport phenomena.

During the project, photoluminescence excitation spectra have been taken to determine the absorption which led to the main emissions of beta- and gamma- Ga₂O₃

nanoparticles. Concerning transport properties, we have started with procedures to achieve good electrical contacts, in order to study the carrier transport by Electron Beam Induced Current technique (EBIC) in the SEM and by XBIC in the synchrotron. The work done so far has allowed us to determine the best protocol to achieve the electrical contacts and overcome some issues, such as early detachment of structures from substrates.

Task 5. Light confinement in undoped and Cr doped Ga₂O₃ nanostructures.

During the project, DBR-based optical cavities have been built based on Ga₂O₃ microwires with hexagonal shaped cross-section. To design the cavities, we have simulated the propagating modes through them to determine the reflectivity as a function of the wavelength of DBR, in order to get suitable stop bands in the UV range (undoped wires) or in the NIR (Cr doped wires). The realization of DBR cavities with the dimensions obtained from simulations, has been performed with the aid of a Focused ion beam (FIB) microscope. As a result, we have produced optical microcavities with sharp Fabry-Perot resonances overlapping the PL spectrum, which agreed with the predictions of the simulations.

We have made analytical calculations of the dispersion function $\omega(k)$ in the waveguides. The results have been compared with experimental data from the Fabry-Perot resonances observed in the built DBR-based cavities, designed during the project.

Finally, our last work on this task has been focused on the temperature dependence studies of the optical resonances and Cr luminescence of the DBR-based cavities. The results have shown the feasibility of a wide dynamic range thermometer based on this system. The cavities have been demonstrated to be robust and stable in time, which make them very promising for applications.

2. Overview

In spite of the fact that during the last years the focus of the work on Ga₂O₃ has been placed on high-electronic power applications based on bulk or thin films, this oxide has attracted interest on its optical properties for nearly six decades, since the first works of A. Schawlow [1], H.H. Tippins [2] and others. Its ultrawide bandgap results in a very wide transparency range, down to some 260 nm, well into the ultraviolet, what allows photonics applications unreachable with other transparent conductive oxides.

Following up the previous knowledge on luminescence properties in β -Ga₂O₃ material by the research team, an overview of the achievements during this project have been:

- Structural properties and luminescence behavior of **Ga₂O₃:Zn ribbons** and **β -Ga₂O₃ and γ -Ga₂O₃ nanoparticles** have been analyzed with the aim to study the impact of Zn doping and size/crystalline phase on the optical properties.
- The design and fabrication of **DBR optical cavities on undoped Ga₂O₃** tuned for optical resonances in the UV-blue has been achieved. Experimental reflectivity up to 50% are obtained over the **350-410 nm region** in FIB-assisted fabricated Ga₂O₃ microcavities.
- The demonstration of a wide dynamic range thermometer made of **DBR Ga₂O₃:Cr cavities**, based on luminescent and interferometric transduction mechanisms as proof-of-concept, with temperature precision around 1 K in the 150-550 K range. Besides, the system shows very high stability and spatial resolution in the range of microns.
- Taking advantage of the physical mechanisms behind the temperature-dependent luminescence of resonances positions, the **temperature dependence** of the **anisotropic refractive index** of bulk Ga₂O₃ has been studied

by ellipsometry, in collaboration with CSIC (Spanish Scientific Research Council) and the Washington State University.

- A **new design** of cavities, based on gallium oxide nanowires where light is confined by Atomic Layer Deposition (**ALD**)-based **DBRs**, as an alternative to FIB, are being also explored in collaboration with the University of Bremen. We have promising, preliminary luminescence results that show light modulation.
- Exploratory work on ternary NiGa₂O₄-based microstructures have been developed from Ga₂O₃ and NiO precursors, via thermal evaporation growth method.

3. Methods

Samples:

β -Ga₂O₃ nanowires were obtained by thermal evaporation, via vapor-solid growth mechanism, using metallic Ga as precursor, at UCM laboratory. The method allows for doping by adding the desired impurities to the precursors.

β - and γ -Ga₂O₃ nanoparticles have been produced by precipitation method in collaboration with the Chemistry Faculty at the UCM.

Unintentionally doped (UID), β -Ga₂O₃ bulk crystal with (100) surface grown by Czochralski method, provided by WSU (J. McCloy).

The DBR cavities were performed by milling with a Focused Ion Beam (FIB) microscope a certain number of holes on an individual Ga₂O₃:Cr wire suspended on a transmission electron microscope grid. The pattern was designed to create an optimized FP optical cavity of length, L, between two DBRs acting as mirrors. FIB facility belongs to University Basque Country (J.M Sanjuan & M.L No).

The alternative DBR system was obtained with ALD multilayers on a Si substrate which sandwiched individual Ga₂O₃:Cr micro- and nanowires deposited on them.

Characterization Methods:

The details of the morphology were assessed by secondary electron (SE) images obtained in a FEI Inspect scanning electron microscope. The DBR patterns were carried out with the aid of a FIB Helios 650 instrument working at 30 kV and 8 pA.

The ellipsometry data have been acquired and analyzed with the WVASE software using the Mueller Matrix formulation in collaboration with the Institute of Optics, CSIC. The fits have been performed using as input data acquired in two different orientations in a wavelength range from 600 to 1200 nm. These measurements were performed at different temperatures between RT and 325°C.

For micro-PL (μ -PL) measurements, performed at RT and above due to laser local heating of the cavity, a 325 nm He-Cd laser in the Horiba LabRAM HR800 optical confocal microscope was used. This equipment allows the separation of excitation and collection points up to several microns, thanks to a custom-made setup [3]. The μ -PL spectra were recorded with a polarizer placed perpendicular to the wire axis, which corresponds to the intrinsic polarization of the main confined optical modes within the cavity [3].

Simulations and Analytical Methods:

The modal analysis of the optical microcavities was assessed by simulations performed with the OptiFDTD commercial software. A short pulse was allowed to propagate from a specific position within the cavity and the power spectrum, as a function of wavelength, was recorded in different areas, within (confined) or outside (scattered) the cavity. In addition, analytical expressions based on Marcatili's approximations for rectangular

waveguides were used in order to obtain a first approximation of the resonance's positions.

First principles calculations:

Density Functional Theory (DFT) calculations have been performed using the CRYSTAL program, in the framework of the hybrid-exchange density functional. The screened-exchange Heyd-Scuseria-Ernzerhof (HSE06) functional is well documented and provides a quantitatively correct description of the electronic structure of oxide semiconductors.

4. Conclusions and future work

- Optical confinement studies

DBR-based optical cavities created within Ga₂O₃:Cr micro- and nanowires have been shown useful as wide temperature range thermometers based on two optical features observed on their luminescence spectra.

During this project, the temperature-dependent anisotropic refractive index and thermo-optic coefficients for gallium oxide have been further characterized by comparison of two independent experimental techniques, i.e. ellipsometry and interferometry in an optical cavity, as well as FDTD simulations from such cavities. The comparison allows a better knowledge of these important parameters for Ga₂O₃-based photonics and optoelectronics.

Reflectivity of DBRs for the UV-blue region can be notably enhanced as compared to our previous results, as learned from FDTD simulations. Further steps have been taken by designing and creating new, improved cavities. The improvement has been confirmed with the simulations and should be soon confirmed experimentally.

A different strategy to confine light within gallium oxide nanowires, based on ALD films, is under development and shows highly promising results for Cr-doped nanowires. To this purpose, we count with the collaboration of our egressed doctor Manuel Alonso-Orts, who defended his thesis in October 2020 and is currently appointed at the University of Bremen as a postdoctoral researcher.

Future work will be focused on the new strategy for the cavities, both in characterization, optimization of the design and actual fabrication of the optimized cavities, along with the finding of further applications of UV-blue oriented microcavities.

- Other Ga₂O₃ phases and compounds

i) We have experimental results on synthesis, structural and luminescence characterization of nanoparticles of β-Ga₂O₃ and γ-Ga₂O₃ and two papers have been published (Material Letters 2020, Nanomaterials 2023). In fact, the use of such nanoparticles was shown to be beneficial for hybrid solar cells (Materials Letters 2020).

ii) The line on the achievement of (Al_xGa_{1-x})₂O₃ nanostructures and exploration of the fabrication of nano-heterostructures based on β-Ga₂O₃ micro- and nanowires has started in a limited way, and hopefully we could further develop it in the near future.

iii) Future developments, which are now starting to be explored, include the development of Ga₂O₃ nanomembranes (quasi bidimensional). Our thermal evaporation method produces freestanding nanostructures that meet the requirements of this morphology, and some trials are ongoing to define the hypothesis. Besides, we have already obtained successful mechanically exfoliated, freestanding nanomembranes from bulk beta-Ga₂O₃ single crystals. Furthermore, doping with some optically active ions has also been performed on these nanomembranes by thermal diffusion. Further photonic structures will be designed from these nanostructures. These features are subject of the new started project.

- i) the study of the feasibility of Ga₂O₃ as quantum material by suitable doping and defects engineering, as well as coupling with photonic structures, made of Ga₂O₃ or other materials.

5. Impact

The results obtained in the current period, along with the previous ones, are believed to make an impact on the basic knowledge of the optical properties of gallium oxide, which will be important mainly for photonics applications of this material. In fact, one interesting application has been already shown, the thermometer based on the optical cavities, published in Small. Other journals where the results obtained during this project have been published are Journal of Materials Chemistry C, Journal of Alloys and Compounds or Nanomaterials. Besides, two invited talks have been given in international conferences on the results obtained during this project. These applications are now overcome by the high-power electronics applications, but in the future are likely to be also of great importance, given the very special physical features of gallium oxide.

6. Publications

- Publications of PIs directly related to the topic of AFOSR project, Ga₂O₃ nanomaterials. Since the project started in June 2020, FA-8655-20-1-7013 grant is mentioned in Acknowledgments in the 6 first ones.

	Authors	Title	Publication
1	D. Carrasco, E. Nieto, Manuel Alonso-Orts, R. Serna, J. M. San Juan, M. Luisa Nó, J. Jesenovec, J. S. McCloy, E. Nogales, and B. Méndez	Temperature dependent refractive index and FDTD simulations for thermometers in β -Ga ₂ O ₃ nanowires	Nanomaterials, 13, 1126 (2023).
2	M. García-Carrión, J. Ramírez-Castellanos, E. Nogales and B. Méndez	Temperature-Dependent and Time-Resolved Luminescence Characterization of γ -Ga ₂ O ₃ Nanoparticles	Nanomaterials, 13, 1445 (2023)
3	M. Alonso-Orts, D.Carrasco, J. M. San Juan, M. L. N., A. de Andrés, E. Nogales, and B. Méndez	Wide Dynamic Range Thermometer Based on Luminescent Optical Cavities in Ga ₂ O ₃ :Cr Nanowires	Small, 18, 2105355 (2022).
4	M. Alonso-Orts, D.Carrasco, J. M. San Juan, M. L. N., A. de Andrés, E. Nogales, and B. Méndez	Ga ₂ O ₃ microwires as wide dynamical range temperature sensors	Proc. SPIE 12002, Oxide-based Materials and Devices XIII, 120020A (2022).
5	M. Alonso-Orts, G. Chilla, R. Hotzel, E. Nogales, J. M. San Juan, M. L. No, M. Eickhoff, B. Mendez	Near-UV optical cavities in Ga ₂ O ₃ nanowires	Optics Letters 46, 278 (2021).
6	J García-Alonso, D Maestre, E Nogales, A Cremades, B Méndez	Study of NiGa ₂ O ₄ microneedles grown by a thermal-evaporation method	J. Alloys and Comp. 919, 165718 (2022).
7	M. B. Barbosa, J. G. Correia, K. Lorenz, A. M. L. Lopes, G. N. P. Oliveira, A. S. Fenta, J. Schell, R. Teixeira, E. Nogales, B. Méndez, A. Stroppa & J. P. Araújo	Contactless doping characterization of Ga ₂ O ₃ using acceptor Cd probes	Scientific Reports 12, 14584 (2022)
8	M. Alonso-Orts, Ana M. Sánchez, E. Nogales and B. Méndez	Ge incorporation in gallium oxide nanostructures grown by thermal treatment	J. Mater. Sci., 55, 111431–11438 (2020)

9	J. García-Fernández, M. García-Carrión, A. Torres-Pardo, R. Martínez-Casado, J. Ramírez-Castellanos, E. Nogales, J. M. González-Calbet and B. Méndez	New insights into the luminescence properties of a Na stabilized Ga-Ti oxide homologous series	J. Mat. Chem. C, 8, 2725-2731 (2020)
10	M. García-Carrión, J. Ramírez-Castellanos, E. Nogales, B. Méndez, C. C. You, S. Karazhanov, E. S. Marstein	Hybrid solar cells with b- and c-gallium oxide nanoparticles	Materials Letters, 261, 127088 (2020)

- Invited talks in conferences:

- “Ga₂O₃ microwires as wide dynamical range temperature sensors”, Manuel Alonso-Orts, Daniel Carrasco, José M. San Juan, María L. Nó, Alicia de Andrés, Emilio Nogales, and Bianchi Méndez. **SPIE OPTO 22-28 January 2022**, San Francisco.

- “Thermometry based on luminescence spectroscopy from cavities in Ga₂O₃:Cr micro- and nanowires” Manuel Alonso-Orts, Daniel Carrasco, José M. San Juan, María Luisa Nó, Alicia de Andrés, Emilio Nogales, and Bianchi Méndez, **ICOSIMS 2023 20-22 June 2023** (online, organized by University of Aveiro, Portugal).

- Oral presentations in conferences

- “Bragg reflector micro- and nanowire optical cavities based on gallium oxide: exploring light confinement by Atomic Layer Deposition as an alternative to Focused Ion Beam patterning”, Manuel Alonso-Orts, Ruben Neelissen, Daniel Carrasco, Emilio Nogales, Bianchi Méndez, Martin Eickhoff. **MRS Spring Meeting**, 8-13 May 2022, Honolulu.

- “Thermometry with optical cavities made of luminescent Ga₂O₃:Cr nanowires”, Manuel Alonso-Orts, Daniel Carrasco, José M. San Juan, María Luisa Nó, Alicia de Andrés, Emilio Nogales, and Bianchi Méndez. **Nanospain Conf**, 17-20 May 2022, Madrid (Spain).

- “Light confinement in gallium oxide based microcavities”, Manuel Alonso-Orts, Daniel Carrasco, José María San Juan, María Luisa Nó, Alicia de Andrés, Emilio Nogales, Bianchi Méndez. **Nano 2022** 6-10 June 2022. Seville (Spain).

- “Optical cavity modes in luminescent β-Ga₂O₃:Cr nanowires for thermometry”, Manuel Alonso-Orts, Daniel Carrasco, José María San Juan, María Luisa Nó, Alicia de Andrés, Bianchi Méndez. **Quantum Matter 2022** (21-23 June 2022. Barcelona (Spain).

- “Optical microcavities in luminescent beta-Ga₂O₃ nanowires: tunability from near-UV to near-IR and temperature sensors”, Manuel Alonso-Orts, Daniel Carrasco, Gerwin Chilla, Rudolfo Hötzel, Jose M. San Juan, Maria L. No, Martin Eickhoff, Alicia de Andrés, Emilio Nogales, Bianchi Mendez. **MRS Fall Meeting**, Boston, 2022.

- Luminescence of doped Ga₂O₃ nanowires” International Workshop on **Ultraviolet Materials and Devices** (IWUMD 2023) 5-8 June 2023.

- “Gallium oxide temperature-dependent refractive index and Cr doped β-Ga₂O₃ nanowires as thermometers” International Workshop on **Ultraviolet Materials and Devices** (IWUMD 2023) 5-8 June 2023.

- PhD thesis and Master thesis

Manuel Alonso Orts (2016 - 2020). “Architectures based on Ga₂O₃ micro- and nanowires for photonic applications”. Supervisors: E. Nogales and B. Méndez (Oct 2020)

Marina García Carrión (2018 - 2021). "Synthesis and characterization of micro- and nanomaterials of gallium oxide-based compounds". Supervisors: E. Nogales and B. Méndez (Dec 2021).

Daniel Carrasco Madrigal (2021 -) has started the PhD thesis on "Nanostructures gallium oxide for applications in photonics" supervised by B. Méndez and E. Nogales. Previously Daniel Carrasco defended his Master thesis in July 2021 and currently is hired under this Project.

Paula Perez Peinado. Master thesis on "Novel photonic structures based on crossed nanowires" September 2023. Supervisors: E. Nogales, B. Méndez and J Dolado. She will go on with the PhD thesis starting on September 2023.

Nuno Muñoz Sánchez, Master thesis on "Study of electronic states of defects in wide bandgap semiconductors", July 2022. Supervisors: B. Méndez and R. Martinez.

- Training capacity

Project: "International Research Experiences for Students" IRES

Track I: STEM Advancement through International Learning (SAIL)

Participants: Universidad Interamericana de Puerto Rico and Departamento Física de Materiales UCM (2023 - 2025). Funded by NSF (Award ID 2153108).

The project will train 6 undergraduate students every year, through a 9-weeks research program mentored by some team members of the UCM group.

In addition to that, every academic year one or two undergraduate students have been working in the framework of this project for their Final Degree thesis.

- Dissemination

- Science week, outreach in other universities and different cities for general public (Emilio Nogales)

- Conference organizers: B. Méndez, E. Nogales and R. Martínez were part of the Organizing Committee of the Nano 2022 – 16th International Conference on Nanostructured Materials, Seville, Spain, 6 – 10 June 2022.

- Symposium organizer: E. Nogales organized the symposium "Defects, doping and processing of semiconductor nanostructures" in the Nano 2022 – 16th International Conference on Nanostructured Materials, Seville, Spain, 6 – 10 June 2022.

- Program committee: B. Méndez, Oxide-based Materials and Devices International Conference, which is part of the SPIE Photonics West

- "Making wide bandgap oxides ready for the next materials generation". Seminar talk by Bianchi Méndez at University Bremen, January 2023.

- " β -Ga₂O₃ Nano- and Microwires for Widely Tunable Optical Microcavities". Invited talk by Emilio Nogales (Webinar) for the King Abdullah University of Science and Technology, KAUST, Saudi Arabia. 18 March 2021.

- " β -Ga₂O₃ Nano- and Microwires for Widely Tunable Optical Microcavities". Invited talk by Emilio Nogales (Webinar) for INESC MN, Microsistemas a Nanotecnologias, Portugal. 9 July 2021.