



Project OxyGaN

Project number: 7363

m-era.net 2019 programme

Deliverable D.15 - Report

Y1 public report

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## **Results achieved**

### **WP1. Development of GaN test structures**

The following were designed, executed, and delivered for use in WP2, WP3 and WP4:

- p-GaN/p-GaN/sapphire epitaxial layers.
- monocrystalline GaN substrates with epi-ready surfaces of N polarity.

These materials serve as test substrates for developing technologies related to the production of ZnO:Al (AZO) and ZnMgO:Al (AZMO) oxide layers. Additionally, they are used for investigating the formation of ohmic contacts for GaN-based lasers.

Furthermore, a method for measuring the resistivity of ohmic contacts on bulk n-GaN substrates was implemented using the Terry-Wilson method. It was applied in the measurement of the first batch of AZO-based p-type GaN contacts.

Epitaxial growth and initial processing of simplified laser structures for WP7 was done.

### **WP2. Development of ZnMgO:Al tuned to the desired wavelength and conductivity**

The task focused on the development of ZnMgO:Al thin transparent conducting films. We started with co-sputtering from an AZO and Mg source to confirm the initial assumptions about the possible bulding-in of Mg in the Zn sublattice. The results showed that it was possible to obtain films with 0-10% Mg in the Zn sublattice. Based on these, the first composite AZMO targets were ordered.

To determine the optimal conditions for deposition of AZO-based materials, optimization processes were carried out to find the deposition parameters of AZO targets in two cathode sputtering reactors, with different target to substrate distances. The impact of process parameters such as argon pressure, sputtering power, sputtering mode (DC, pulsed DC, or RF), and the chemical composition of the AZO target was analyzed. It was determined that layers with the lowest resistivity and high optical transmission are obtained during sputtering at the lowest pressure, with minimal influence from power or sputtering mode. Another key parameter, rarely analyzed, was the distance between the target and the substrate. For layers deposited in a reactor where this distance was 30 mm, the resistivity was  $1.5 \times 10^{-3} \Omega\text{cm}$ , while for a reactor with greater distances, resistivity reached as high as  $5 \times 10^{-1} \Omega\text{cm}$ . This effect, not previously observed in the literature, is currently being investigated by the research team. Importantly, the resistivity values at  $1.5 \times 10^{-3} \Omega\text{cm}$  are better than the reported best values for AZO layers, which until now were only achieved with additional substrate heating up to 200°C during deposition. This high temperature process made it impossible to use such layers with lift-off photolithography, which makes the obtained results technologically significant not only from the point of view of the project but also from the point of view of the state of knowledge. A detailed analysis of the parameters influencing the growth of AZO layers on substrates not subjected to additional heating, as well as showing how to obtain high optical transmission and low resistivity were demonstrated at the conference and described and submitted for publication in a Q1 journal.

In parallel to the research on optimizing the composition of AZMO targets, research was carried out on other methods of depositing AZMO layers, in particular by cosputtering, sputtering AZO - Mg composite targets and sputtering AZO - MgO composite targets. Cosputtering was carried out using an AZO target and a Mg target, placed in the sputtering reactor in a way that allows simultaneous deposition of both targets on one substrate. It was observed that the layers created in this way are characterized by a potentially high Mg content (above 5 atomic %), but also by low transmission and limited widening of the energy gap. A miniaturized approach was tested where Mg pieces were attached to an AZO target. Using this approach, layers with Mg content ranging from 1 to 19 at% were obtained within a 2" diameter substrate. As in the two-target approach, there was a darkening of the layers in areas containing more Mg, which means that the growth mechanism and the limitation of mixing of Mg and Zn are analogous. Such phenomena did not occur for composite targets containing MgO instead of Mg, in which case there was no decrease in transmission, but less Mg was incorporated into the material (approx. 1.5-2.2% at. within 2").

### **WP3. Development of ZnMgO:Al-based contacts to N-face n-GaN**

The behavior of AZO and AZO layers on ultrathin interfacial metal layers in contact with nitrogen-polar n-GaN was investigated. n-GaN substrates with an electron concentration of approximately  $1 \cdot 10^{18} \text{cm}^{-3}$  were transferred from IWCPAN in the form of a 2" wafer cut into squares with sides of 9 mm. Photolithographically shaped c-TLM patterns with lift-off technique were used for testing ohmic contacts. Before deposition, the substrate was deoxidized by bathing in a 2:1 mixture of H<sub>2</sub>O:HCl for

1', then rinsed in deionized water, dried with nitrogen and immediately placed in a vacuum chamber. It was found that layers made of AZO alone did not create ohmic contact with n-GaN, despite heating in nitrogen at temperatures up to 700°C. X-ray diffraction tests did not show any differences in the material after annealing at different temperatures, which shows that the essence of the junction is the interface between GaN and AZO, which may be possible to modify, as assumed in the project. Interface studies are currently being carried out using high-resolution TEM microscopy at Hungarian partners.

For reference purposes, a conventional Ti/Al/TiN/Au ohmic contact was realized and formed after annealing in nitrogen at 750°C. Very high currents of 0.5A at voltages of 0.25V were recorded for this contact, which shows that the structure works properly.

Several metallic interlayers were studied with some showing promising results with high currents and symmetrical, although non-linear I-V characteristics.

**WP4. Development of ZnMgO:Al-based contacts to Ga-face p-GaN**

Research on contacts based on AZO as well as AZO with interfacial films was started in order to determine appropriate structures for creating ohmic contacts. Work was carried out on samples with p-GaN epitaxial layers produced on Al<sub>2</sub>O<sub>3</sub> substrates by IWCPAN. The concentration of holes in the p layers was approximately 4·10<sup>17</sup> cm<sup>-3</sup>. For reference purposes, ohmic contacts were manufactured containing a standard Ni/Au bilayer, formed at a temperature of 500°C in synthetic air flow. In this way, high-quality ohmic contacts with a contact resistance of 4.3·10<sup>-4</sup> Ωcm<sup>2</sup> were obtained, which, similarly to the case of WP3, allowed to confirm the good quality of the supplied structure. Before depositing all layers in WP4, surface deoxidation was performed in a solution of HCl in H<sub>2</sub>O, similar to that in WP3.

In the case of p-type GaN layers, contacts with AZO showed an ohmic character after annealing at 800°C. These contacts were therefore tested in WP7 in the structure of a laser diode. The use of selected metallic interlayers allowed the contact forming temperatures to be reduced to 600 and 650 °C, respectively, while increasing the current intensity by more than an order of magnitude. The material characterization results have not yet given the answer as to why the ohmic contacts form and thus the research on this matter will be carried out further in the next year of the project.

**WP5. Advanced characterization**

TEM and TOF-SIMS characterization of AZO and AZMO films as well as selected contacts from WP3 and WP4 was carried out.

**WP6. Contact validation in diode structures**

According to the plan of the project, WP6 will start in M20, beyond the scope of this report.

**WP7. Demonstrating an operational packaged LD**

WP 7 is implemented in close cooperation with tasks WP1 and, in the future, WP6, so that the results obtained in them can be taken into account and integrated in the production process of laser diode structures and the demonstrator can be tested in accordance with the internal standard operating procedures applicable at TOPGAN.

Research in WP 7 is carried out on laser structures grown for this purpose, using the MOVPE technique on single-crystal n-GaN substrates as part of WP1, carried out by IWCPAN. A simplified processing scheme was developed for the top laser contacts based on AZO.

No changes were observed in the position and width of the electroluminescence spectra after deposition of AZO oxide layers for contact formation at 800°C, which may indicate that this process does not cause degradation of the active layers of the laser structure, produced epitaxially at lower temperatures.

**Dissemination of results**

Work and conference name	Presentation type
None presented in the first year of the project	

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