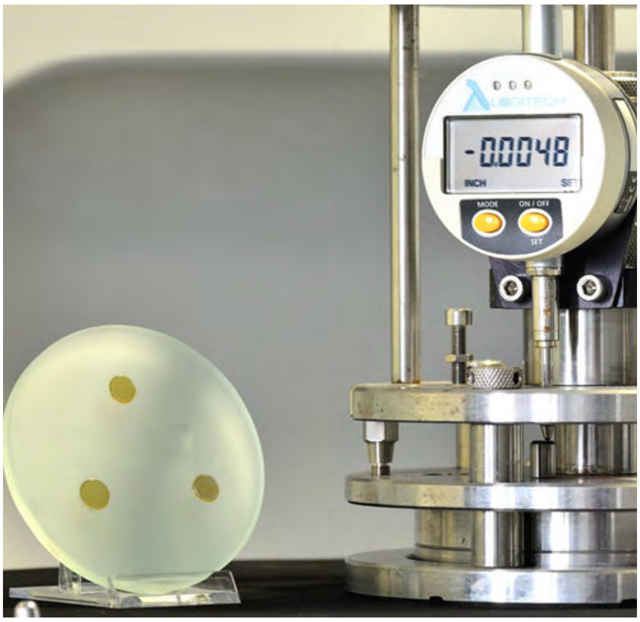
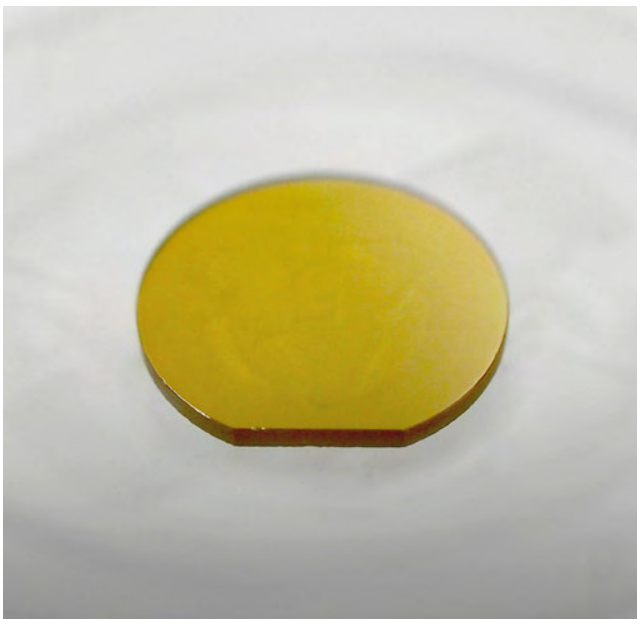
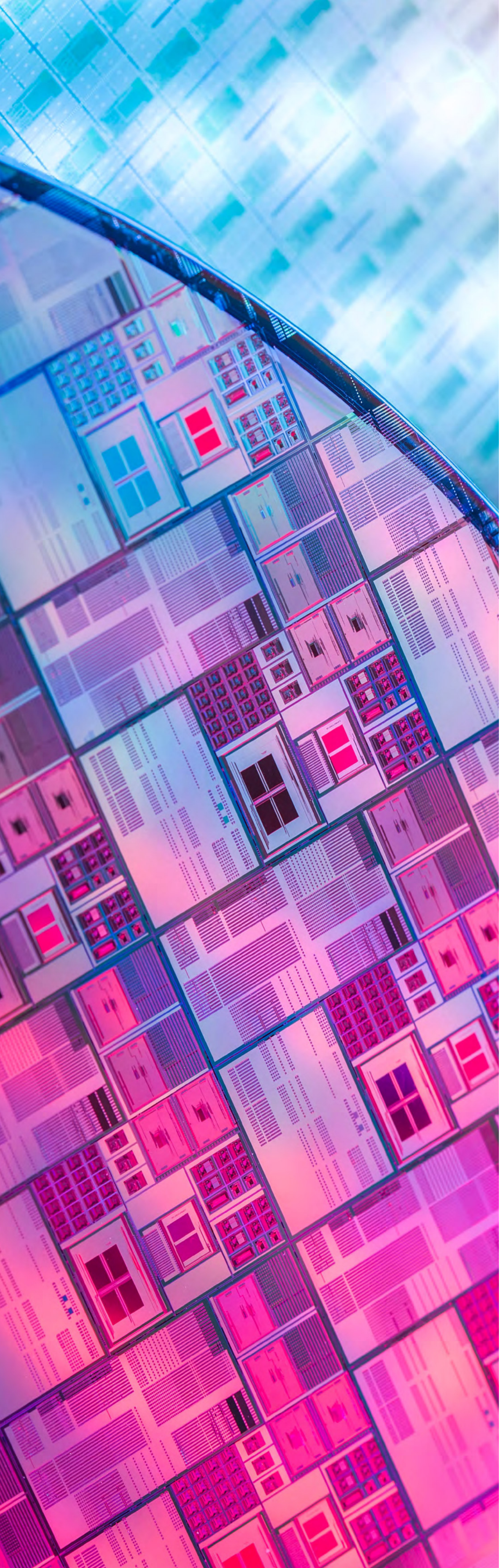
LEIBNIZ-INSTITUT FÜR KRISTALLZÜCHTUNG

Native AlN Substrates for High-Efficient Deep UV Emitters and Next Generation Power Conversion Devices







WHO WE ARE

Single crystalline aluminum nitride (AlN) and the ternary AlGaN alloy is a promising material system for a wide range of advancing (opto-)electronic applications. Its outstanding properties include a wide band gap, high thermal conductivity and robust chemical stability. In addition, the material can withstand at harsh conditions and high temperatures without degradation.

Deep UV light emitting diodes (LEDs) with emission wavelengths between 220-280 nm, could be used for the disinfection of drinking water and waste water treatment, thus providing safe water sources, particulary in remote or disaster-stricken regions. Additionally, deep UV LEDs and laser diodes are important for applications like surface sterilization and bio-chemical sensing.

Beside the deep UV photonics, the strengths of AlN emerge in the field of next generation high-frequency power conversion devices. AlN based high power transistors have already demonstrated the ability to exceed the performance parameters of SiC and GaN transistors. Furthermore, AlN is well suited for high temperature sensing (thermocouples and piezoelectric elements) in the field of temperature or force measurement at high temperature processes.

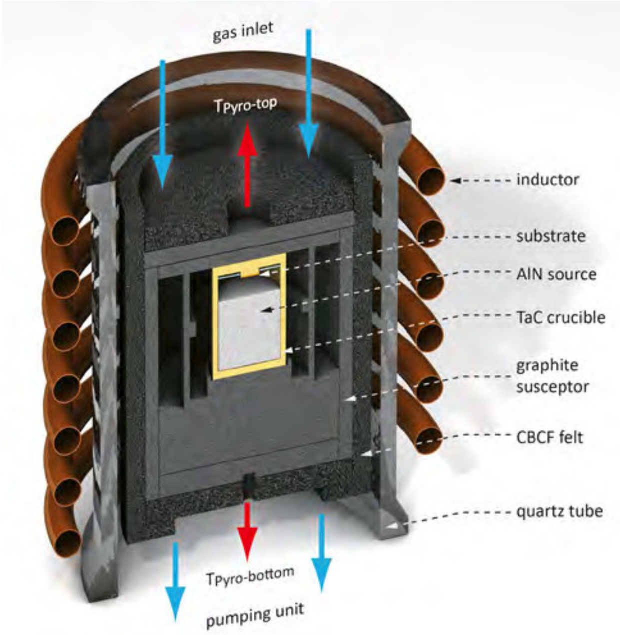
The lattice matching of single crystalline AlN substrates to functional Al-rich AlGaN epi-layers allows the fabrication of high quality AlN / AlGaN heterostructures, which are of central importance for the performance of the devices in the diverse applications.

Water or air disinfection with UV light

Electric trucks with charging stations

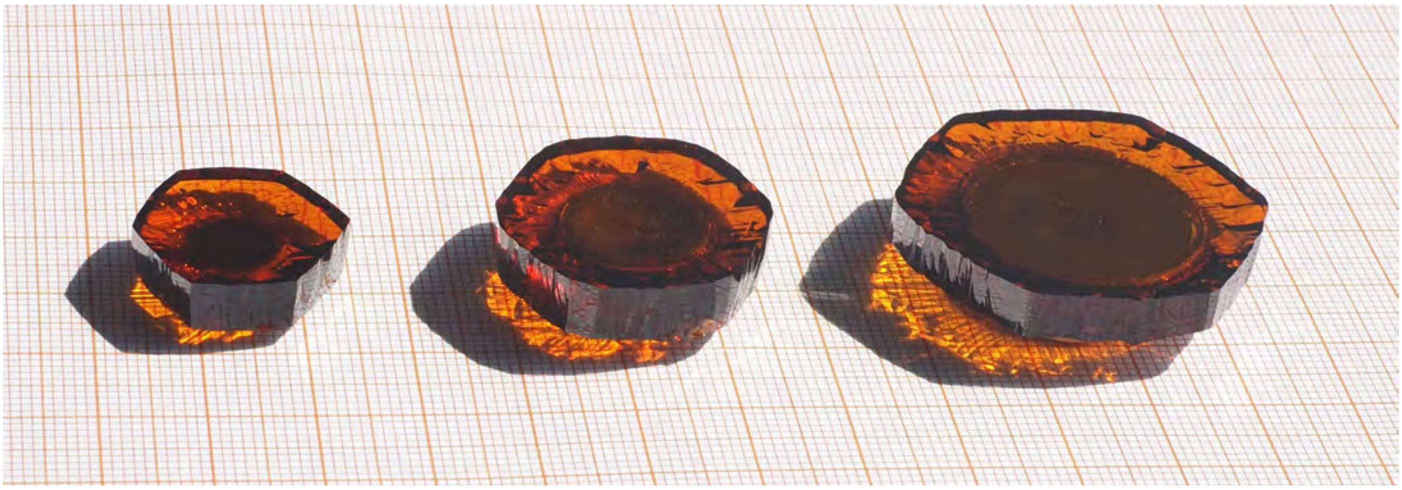
CRYSTAL GROWTH

The Leibniz-Institut für Kristallzüchtung has a longstanding expertise in the growth of aluminium nitride bulk crystals. This also includes the preparation of the wafers into epi-ready substrates. To unlock the full potential of the material and enable high efficient deep UV emitters and high-frequency power conversion devices the demands on the material properties are high. Defects in the crystals reduce the device efficiency and thus it is necessary to keep the dislocation density as low as possible. At the IKZ, we grow bulk aluminium nitride crystals by physical vapour transport (PVT) using native AlN seeds resulting in threading dislocation densities below $10^4/cm^2$. Under suitable AlGaN epitaxial conditions (pseudomorphic growth), this low dislocation density can be maintained throughout the entire device structure. As a consequence, the threading dislocation densities are about 4 to 5 orders of magnitude lower compared to AlGaN devices on sapphire substrates.



The bulk AlN growth takes place in tantalum carbide or tungsten crucibles at growth temperatures between 2100°C and 2300°C at 500-900 mbar ambient pressure. The reactors as well as the thermal insulation felt (carbon-bonded carbon fiber), and large parts of the graphite components can be taken over from the well proven SiC growth technology. Recent breakthroughs in crystal diameter expansion while maintaining the high crystalline quality show the suitability of the IKZ process to achieve industrially relevant crystal diameters ≥ 4 inches in the medium term.

The IKZ researchers developed an AlN growth process with a fast increase of the crystal diameters. The growth rates attain around 200 μm/h in both, the N-polar and in the prismatic m directions resulting in huge expansion angles around 45° leading to in a diameter increase between 10 – 16 mm per run. The full diameter spans over the entire crystal length which means that all cut c-plane wafer have the same (final crystal) diameter. The threading dislocation density is below $10^4/cm^2$. The process outlines a shortcut path to industrially relevant AlN crystal diameters compared to all other published expansion processes for bulk AlN crystals so far.

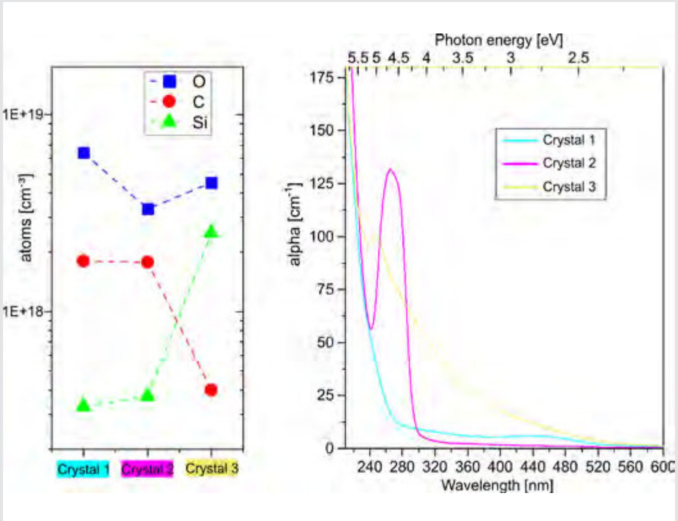


Details can be found here:

C. Hartmann et al., Applied Physics Express 16 (2023) 075502: *"Efficient diameter enlargement of bulk AlN single crystals with high structural quality"* (https://doi.org/10.35848/1882-0786/ace60e)

TAILORING THE OPTICAL AND ELECTRICAL PROPERTIES BY IMPURITY CONTROL

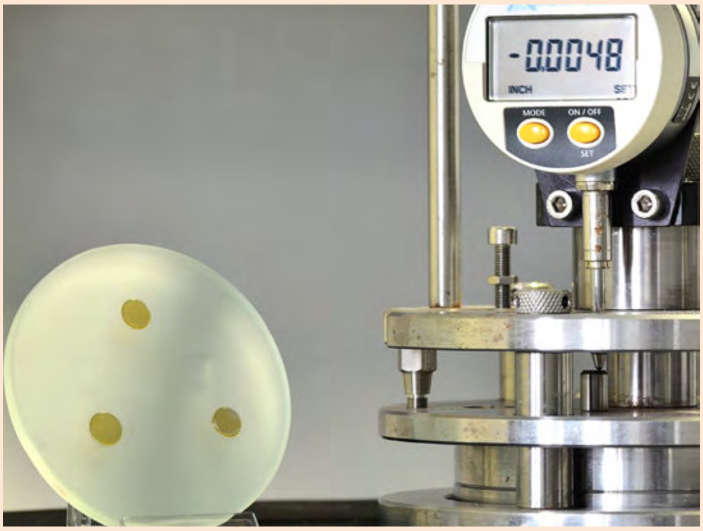
Intensive research is being carried out at the IKZ to understand the effects of the concentration of impurities on the optical and electrical properties. With regard to the use of AlN wafers as native substrates in UVC LEDs the absorption coefficient at the emission wavelength (220–280 nm) has to be as low as possible as the UV light is usually extracted through the substrate in the currently used LED designs. Absorption coefficients α < 30/cm at the emission wavelength are desirable. For use as substrate for lateral power devices, the substrate should be of high impedance. The objectives are the preparation of deep UV transparent as well as semi-insulating substrates.



Impurity conentrations and corresponding absorpion spectra

CRYSTAL PREPARATION

In addition to the growth of as large as possible AlN substrates with tailored optical and electrical properties, the epi-ready surface quality is a further key parameter for the subsequent process steps and the device performance. The team in the preparation lab of the IKZ has developed the complete AlN preparation line from the sawing of c-plane wafers, over lapping and mechanical polishing to the chemo-mechanical polishing (CMP). The CMP finish step using silica under basic conditions leads to a roughness Ra < 0.3 nm. We are able to adjust the substrate off-cut (which mainly determines the step widths / macro step formation) precisely between 0.05 and 0.5 degree. Pseudomorphically grown homoepitaxial layers by MOVPE (by our partners at Ferdinand Braun Institut Berlin und TU Berlin) show the suitable surface quality of the substrate.

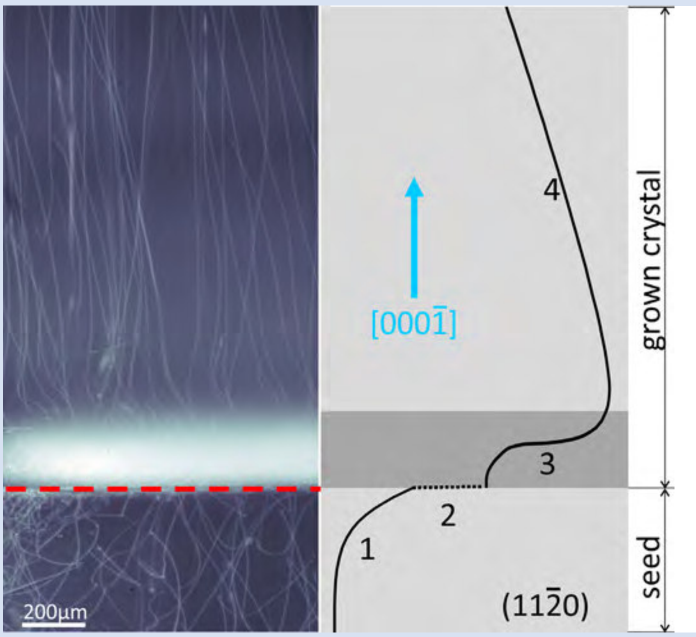


3D X-RAY IMAGING OF DISLOCATIONS

State-of-the-art X-ray diffraction imaging techniques are applied and further developed with our partners from the Karlsruhe Institute of Technology (KIT) to investigate the dislocation distribution and movement in AlN bulk single crystals. Monochromatic beam X-ray topography (MB-XRT) and X-ray diffraction laminography (XDL) measurements provide a substantial enhancement of contrast and additional 3D information. Individual dislocations could thus be separated and tracked in 3D through the crystal at dislocation densities around 10^3/cm². It was possible to show that the dislocations lines are bent in the a-planes that are perpendicular to their Burgers vectors.

More details can be found here:

T. Straubinger et al., Crystal Growth & Design. 23 (2023) 1538: *"Dislocation Climb in AlN Crystals Grown at Low-Temperature Gradients Revealed by 3D X-ray Diffraction Imaging"* (https://doi.org/10.1021/acs.cgd.2c01131)



OUR SERVICES / OUR PRODUCTS

EPI-READY SUBSTRATES WITH TAILORED OPTICAL AND ELECTRICAL PROPERTIES

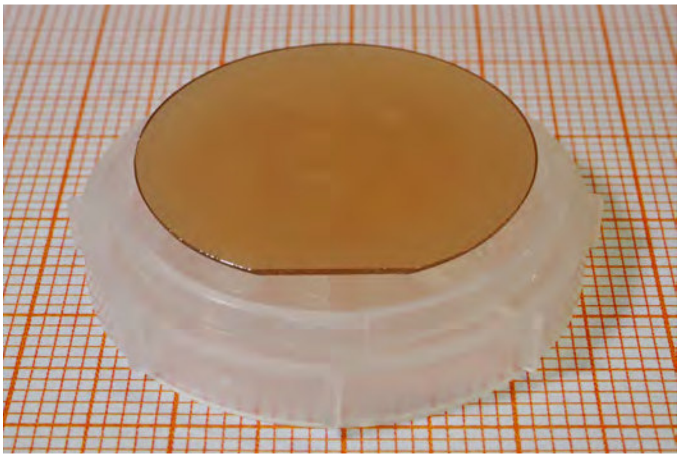
In addition to high quality crystalline materials with tailored properties, reliable access to these materials is essential for the development of new devices or future technologies. We therefore go beyond the academic level by providing small-scale series of aluminium nitride substrates with reproducible properties, which will enable our partners to develop new technologies or devices based on these materials.

The IKZ sells epi-ready c-plane AlN substrates (Ø = 10 mm) with dislocation densities DD < $10^4/cm^2$ (determined bx X-ray topography and defect selective etching). Further details can be found here: **https://www.ikz-berlin.de/en/offer/aluminum-nitride**

Currently, we are in the process of developing the technology to provide 1-inch prototype substrates, with plans to offer also 2-inch demonstrators in the near future. We are open to project ideas for the development of AlN based opto-electronic or high-power devices. Our substrates provide tailored optical and electrical properties (with diameters presently up to 1-inch).

The IKZ is able to provide:

- epi-ready c-plane AlN substrates with absorption coefficients α(265 nm) < 30 /cm (for UVC emitters)
- semi-insulating epi-ready c-plane AlN substrates with the Fermi-level at mid-bandgap (for high power devices)



25 mm epi ready Al-polar AlN substrate with dislocation density DD < $10^4/cm^2$

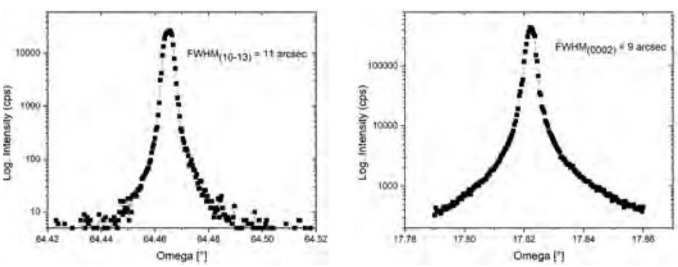


Fig. 7: Typical double crystal rocking curves measured with open detector aperture in the 0002 and 10-13 reflection on the Al-polar substrates

CHARACTERIZATION

IKZ section "Material Science" provides profound understanding in fundamental science combined with a wide range of state-of-the-art measurement tools.

STRUCTURAL CHARACTERIZATION

IKZ operates several high-resolution diffractometers that are capable of detecting strain in crystals smaller than 10^{-5}, which may be due to composition variation or stress. The spatial resolution by this technique is 200 × 200 μm^2. Maps of strain can be measured as for sample area of several cm^2.

Alternative imaging techniques are available, such as laser scattering tomography (LST), X-ray fluorescence and diffraction mapping, electron channeling contrast imaging, cathodoluminescence mapping, Raman microscopy and electron microscopy. Each technique exhibits different probing depth length scales.

Using imaging techniques, low densities of (individual) dislocation can be measured. LST can yield 3D networks of dislocations if they are decorated. Electron-based techniques provide ultimate resolution on the nanometer-level.

Defect selective etching (DSE) based on alkaline etchants are developed for bulk AlN as well as for Al(Ga)N epilayers at IKZ. Dislocation densities are locally resolvable over wide range up to $5x10^9/cm^2$.

OPTICAL CHARACTERIZATION

The IKZ provides absorption spectroscopy measurements from the UV down to NIR range, Raman spectroscopy as well as high resolution cathodoluminescence. These experiments can be performed in a wide temperature range from 4 K up to 1300 K.

ELECTRICAL CHARACTERIZATION

To assess the electrical properties of AlN crystals and layers at IKZ, we use current transport and capacitive measurements. In particular, conductivity and Hall measurements are carried out in a wide temperature range from 1.4 K up to 1100 K in different atmospheres like N_2, O_2, Ar, forming gas or vacuum to determine charge carrier mobility, doping density, compensation ratio and dopant activation energies as a function of growth conditions. Additionally, deep-level transient spectroscopy, thermal admittance spectroscopy and photo-assisted capacitance spectroscopy give insights in defect properties and their influence on the material properties.

THE LEIBNIZ-INSTITUT FÜR KRISTALLZÜCHTUNG IKZ

The Leibniz-Institut für Kristallzüchtung (IKZ) in Berlin-Adlershof is an international state-of-the-art competence center for science & technology as well as service & transfer for innovations in and by crystalline materials. The R&D spectrum thereby ranges from basic over applied research activities up to pre-industrial research tasks.

Crystalline materials are key technology enabling components to provide electronic and photonic solutions to today's and future challenges in society such as artificial intelligence (communication, mobility etc.), energy (renewable energies, power conversion etc.) and health (medical diagnosis, modern surgical instruments etc.).

The IKZ provides innovations in crystalline materials on account of its combined in-house expertise on plant engineering, numerical simulation and crystal growth, enabling it to achieve highest-quality crystalline materials with tailored properties.

Nanostructures, thin films and volume crystals are investigated, the latter being the unique selling point of the institute. Cutting-edge theoretical and experimental materials science know-how is a strong asset for IKZ's R&D activities.

Together with partners from institutes with technology platforms as well as industry companies, the institute will in future also drive innovation by crystalline materials, namely the reliable evaluation and benchmarking of innovative crystalline prototype materials for disruptive technology approaches.

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