Structural, optical, and electrical properties of bulk AlN crystals grown by PVT

M. Bickermann*[1], B.M. Epelbaum[1,2], and A. Winnacker[1]. *1. Department of Materials Science 6, University of Erlangen-Nürnberg, Martensstr. 7, D-91058 Erlangen, Germany. 2. SiCrystal AG, Paul-Gossen-Str. 100, D-91052 Erlangen, Germany.*
* Corresponding author: Phone: +49 9131 8527730, Fax: +49 9131 8528495, *e-mail: matthias.bickermann@ww.uni-erlangen.de*

Homoepitaxy of semiconductor nitrides like GaN, AlN, and their alloys is considered a very promising way to improve structural quality of the epilayers. In the present study, structural, electrical and optical properties of AlN substrates grown in our laboratory have been investigated. AlN substrates containing single-crystalline areas up to 5 x 5 mm were fabricated out of dense, high-purity AlN boules with up to 2 inch in diameter and 15 mm in height. Structural quality of the grains was investigated with electron microscopy, X-ray diffraction, and Raman spectroscopy. The grains are mostly oriented along the c-axis direction. While the structural order of the grains is rather high, several defects including sub-grain boundaries, voids and Al precipitates have been identified. Optical properties measured by optical absorption as well as electrical and thermal properties are presented and discussed in terms of structural peculiarities of PVT-grown AlN crystals. The results serve as a basis for further optimization of bulk crystal growth of AlN.

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* Corresponding author: Phone: +49 9131 8527730, Fax: +49 9131 8528495, *e-mail: matthias.bickermann@ww.uni-erlangen.de*

Homoepitaxy of semiconductor nitrides like GaN, AlN, and the ternary GaAlN alloys is considered a very promising way to improve structural quality of the epilayers and thus device operation. Single-crystalline AlN substrates offer better chemical and thermal stability as well as higher thermal conductivity in comparison to GaN. However, up to now only small single crystals of AlN grown by the PVT method are available [1,2]. Structural quality of PVT grown AlN is very little investigated [1]. Similar to the case of SiC some years ago, single-crystalline yield as well as structural and electrical properties have to be significantly improved prior to industrial application. In this study some structural, optical, and electrical properties of AlN crystals grown in our laboratory have been investigated. The results are discussed in terms of further optimization of bulk crystal growth of AlN.

AlN substrates containing single-crystalline areas up to 5 x 5 mm were fabricated out of dense, high-purity AlN boules with up to 2 inch in diameter and 15 mm in height (see abstract of Epelbaum et al.). Chemical analysis (GDMS) shows that the grown AlN boule contains 80 ppm wt oxygen (equal to a concentration of about 1×10^{19} cm^{-3}) as well as some ppm wt tungsten (originating from the growth set-up) and carbon (presumably due to sample preparation procedures). Other elements were found only in the sub-ppm range. The oxygen concentration is the lowest reported up to date for bulk AlN [3].

Samples cut from the boule were investigated using X-ray (Laue patterns, diffraction data, and texture analysis) and electron beam techniques (scanning microscopy, back-scattering electron and EDX analysis). Grown samples are strongly textured with single crystallites mostly oriented along the c-axis direction. Structural defects include sub-grain boundaries, voids, and Al precipitates. No traces of oxygen-induced structural defects were found. Density of 3D defects such as voids and Al inclusions depends strongly on crystal orientation and polarity (see Fig. 1). The crystalline structure in single-crystalline areas is rather high, as FWHM values of phonon peaks in Raman micro-spectroscopy are as low as 5 cm^{-1}, and diffraction peaks of single crystallites with a FWHM of about 90 arcsec could be resolved.

Optical absorption (see Fig. 2) shows peaks in the visible blue and UV range, respectively, that are related to nitrogen vacancies and oxygen according to literature [3]. Analysis of the oxygen related peak confirms that the oxygen content is in the range of 10^{19} cm^{-3}. Electrical measurements at elevated temperatures show a resistivity of about 5×10^7 Ωcm at 500 K and a thermal activation energy of 0.85 ± 0.5 eV in the temperature range of 500...700 K. The thermal conductivity of a wafer with 12.5 mm in diameter and 500 µm in thickness was measured to be up to 186 W/mK at room temperature. In the range of 300...1500 K a T^{-1} dependence of the thermal conductivity was found. The obtained values are significantly lower than the theoretical limit [4]. This is attributed to structural defects (voids and sub-grain boundaries) rather than oxygen content.

As a result, the grown AlN boules contain single-crystalline areas of high structural quality. However, structural defects present in the AlN substrates may negatively influence the quality of epilayers in subsequent epitaxial growth. Also deep levels, probably oxygen and vacancy-type defects, influence strongly the optical and electrical properties of the substrates. To improve substrate quality, growth conditions have to be optimized to reduce structural defects as well as impurity incorporation.

References

[1] B. Raghothamachar, M. Dudley, J. C. Rojo, K. Morgan, L. J. Schowalter, J. Cryst. Growth 250 (2003), p. 244

[2] R. Schlesser, Z. Sitar, J. Cryst. Growth 234 (2002), p. 349

[3] G. A. Slack, L. J. Schowalter, D. Morelli, J. A. Freitas Jr, J. Cryst. Growth 246 (2002), p. 287

[4] G. A. Slack, R. A. Tanzilli, R. O. Pohl, J. W. Vandersande, J. Phys. Chem. Solids 48 (1987) p. 641

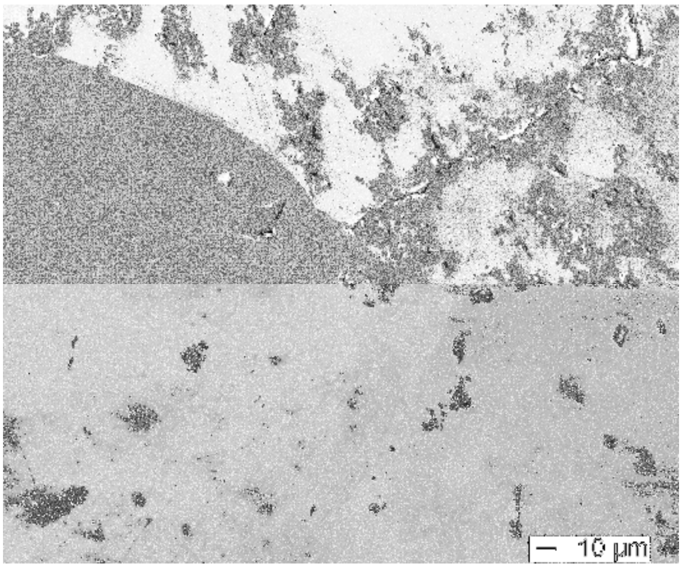

Fig. 1: Wafer surface in scanning electron microscopy. Two neighboring but differently oriented grains demonstrate different density of structural defects (voids and cracks).

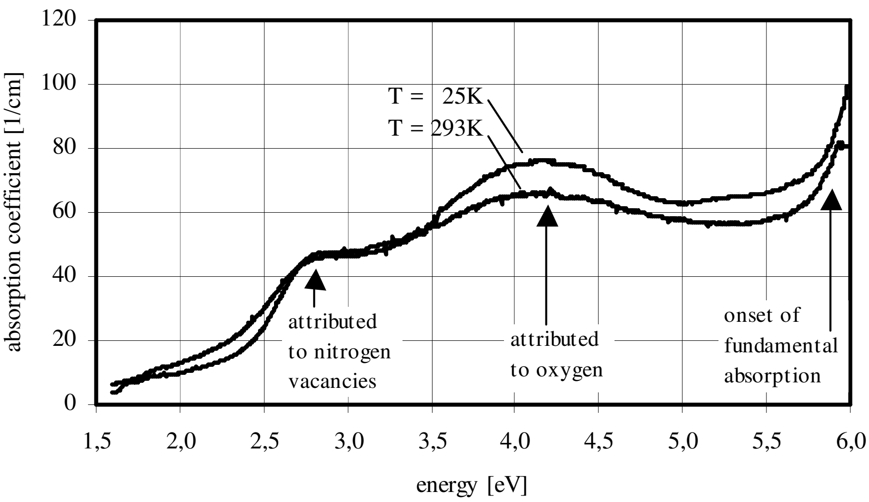

Fig. 2. Optical absorption spectra (visible/UV range) of an AlN wafer at 25 K and 293 K.