Structural Properties of Aluminum Nitride Bulk Single Crystals Grown by PVT

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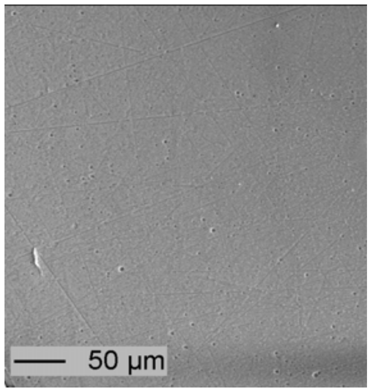
Aluminum nitride (AlN) is a promising substrate material for epitaxy of Al-rich III-nitrides, to be employed e.g. in deep-UV optoelectronic and high-power microwave devices. Bulk AlN single crystals are preferentially grown by physical vapor transport (PVT), but structural quality, especially dislocation content, of such material remains an important issue for substrate use. In this study, crystals were grown on single-crystalline AlN seeds by directional sublimation, yielding AlN single crystals up to 1.2 inch in diameter and 12 mm in height [1]. For comparison, crystals spontaneously nucleated on crucible walls during growth under nearly isothermal conditions were also investigated; they are virtually unstressed, but size and habit is insufficient for industrial application [2]. All crystals were cut to approx. 1 mm in thickness and subsequently polished on the Al-polar face. We investigated distribution and density of dislocations as well as domain structure (mosaicity) of these wafers by wet chemical etching [3], optical and electron microscopy, and X-ray transmission rocking curve experiments.

Crystals grown on AlN seeds show average dislocation densities of $1x10^4 \ldots 10^7$ cm^{-2}. Thus, structural quality even of large-area bulk AlN wafers is highly competitive to epitaxial material on foreign substrates in respect to substrate use. As shown in Fig. 1a, etch pits are inhomogeneously distributed on the wafer surface, some are aligned in clusters, and some areas with etch pit densities higher than 10^5 cm^{-2} clearly show cellular dislocation patterns. The cellular structure was found to be partially inherited from the AlN seed. This is confirmed by rocking curve experiments where seeded crystals as well as the AlN seeds used for seeding show FWHM values of 0.1…1.5° depending on rocking orientation. Additionally, smaller etch pits show up regularly in such samples which are only rarely seen on spontaneously nucleated crystals [2]. We conclude that dislocations of different types are present with different densities in the crystals. X-ray topography is underway. In the presentation, we will discuss etch pit distribution in further detail and present the evolution of dislocation density with growth time for seeded AlN crystals. The comparison with spontaneously nucleated crystals demonstrates that structural properties of AlN bulk single crystals depend strongly on the growth process. Such crystals have average dislocation densities of $1x10^3 \ldots 5x10^5$ cm^{-2}; an exemplary etch pit distribution is shown in Fig. 1b. Although the dislocation distribution is also inhomogeneous, no cellular patterns were observed. Rocking curve FWHM values of approx. 30 arcsec prove that mosaicity is absent in those crystals.

As a conclusion, large seeded AlN crystals have been grown and investigated. Crystalline quality of them is still sufficiently lower compared to spontaneously nucleated crystals. As it stands, mosaicity is a challenging problem in directional sublimation; both growth conditions and seed quality have to be improved to ensure AlN bulk growth with low dislocation density.



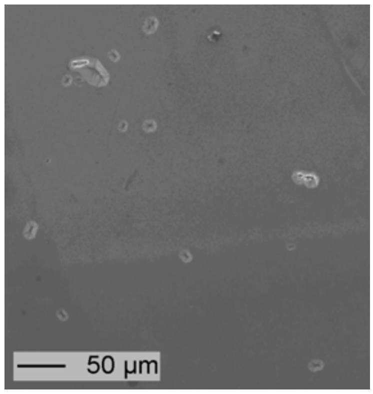


Fig. 1: Etch pit distributions of bulk AlN wafers. a) cut from a crystal grown on AlN seed; etch pit density is 3.3×10^5 cm^{-2}; b) cut from spontaneously nucleated AlN, etch pit density is 2.4×10^4 cm^{-2}. The latter sample was etched for a longer time intentionally to increase the etch pit diameter.

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[2] B. Epelbaum, M. Bickermann, A. Winnacker: J. Crystal Growth 275 (2005) e479.

[3] M. Bickermann, S. Schmidt, B. M. Epelbaum, P. Heimann, S. Nagata, A. Winnacker: J. Crystal Growth 300 (2007) 299.