Defect-Selective Etching of Aluminum Nitride Single Crystals

Matthias Bickermann[a], Boris Epelbaum[a], Sabine Schmidt[a], Paul Heimann[a], Shunro Nagata[b], Albrecht Winnacker[a]

[a] *Department of Materials Science 6, University of Erlangen, Martensstr. 7, 91058 Erlangen, Germany*

[b] *Functional Materials Development Center, Research Laboratories, JFE Mineral Company, Ltd. 1, Niihama-cho, Chuou-ku, Chiba-shi, Chiba 260-0826, Japan*

matthias.bickermann@ww.uni-erlangen.de

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. In this context, preparation of bulk AlN crystals by physical vapor transport (PVT) seems to be most successful. But structural quality and defect formation in such material remain important issues for substrate use. Wet chemical etching is the adequate technology for defect evaluation, as it provides defect decoration and determination of surface polarity [1,2]. However, up to now etching phenomena on different surfaces of AlN single crystals are not understood in detail. In this study, we address open issues and present new results and insights on wet chemical etching of defects in AlN.

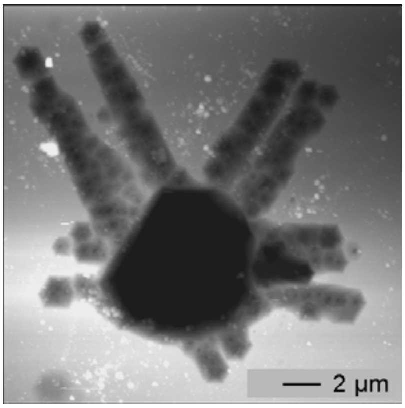

Fig. 1: AFM image of an etched Al-polar surface showing edge dislocation patterns around a bigger etch pit.

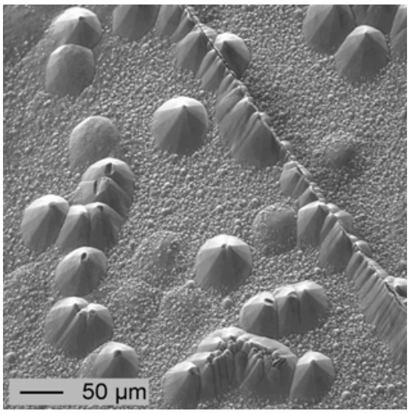

Fig. 2: SEM image of an etched N-polar surface covered by µm-sized pyramids. Additionally, big pyramids form along cracks and on isolated sites.

We used as-grown freestanding AlN single crystals grown in our laboratory [3] as well as polished cuts of such crystals to investigate etching in KOH-NaOH eutectic solutions using various temperatures and etching times. Etched c-plane (both polarities), m-plane and r-plane on-axis surfaces were investigated by SEM and AFM. We find that as-grown surfaces of AlN show characteristic behavior depending on the surface polarity and orientation. The Al-polar surface shows hexagonal pits of different size which can be attributed to screw and edge dislocations. We were able to verify the edge dislocation nature of the smaller etch pits by comparing them with features found on micro-indented, subsequently annealed and etched samples, in analogy to the work of Kamler *et al* on GaN [2]. Furthermore, we found edge dislocation patterns forming around screw dislocation etch pits on repeated etching (see Fig. 1). On the N-polar surface, which is etched much more rapidly compared to the Al-polar surface, oriented pyramids are observed which differ in their dimensions in more than one order of magnitude. In contrast to literature data [1] we clearly attribute the big pyramids to defects, as they are aligned along cracks and polishing scratches (see Fig. 2). But also isolated big pyramids are found; their density roughly correlates with the density of etch pits on the Al-polar face. The pyramids persist on repeated etching up to 100 µm surface ablation, so presumably they indicate the presence of dislocations (TEM studies are underway). Finally, we show that areas in single-crystalline samples having different optical properties (e.g. caused by different concentrations of impurities or intrinsic defects) show significant differences in etch pattern and etching rate; thus, wet etching is appropriate to detect macroscopic inhomogeneities in the samples.

References

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