Comparative Etching Study of Dislocations in Bulk GaN grown by HVPE, Na-flux and Ammonothermal method

Bhavpreeta P. Charan¹*, Ricksen Tandryo¹, Masayuki Imanishi¹, Kosuke Murakami¹, Shigeyoshi Usami¹, Mihoko Maruyama¹, Masashi Yoshimura², Yusuke Mori¹

*bhavpreeta@cryst.eei.eng.osaka-u.ac.jp

1 Graduate School of Engineering, Osaka University, Osaka, Japan

2 Institute of Laser Engineering, Osaka University, Osaka, Japan

Na-flux, hydride vapor phase epitaxy (HVPE), and ammonothermal methods are the three most popular growth processes used to crystalize bulk gallium nitride (GaN). Each growth method exhibits a different amount and type of threading dislocations (TDs) in the grown crystal. Therefore, comparison of the dislocation characteristics in crystals grown with each method is an engaging topic of study.

Defect-selective chemical etching can be employed for the quick characterization of TDs in a crystal. However, the etching rate of TDs can differ depending on the growth method of the crystal, as each growth method incorporates different kinds and concentration of impurities into the crystal ^[1]. Consequently, a direct comparison of TD character using chemical etching is challenging. Considering that crystals grown using same growth method will have similar impurity concentration, we performed a comparative etching study of TDs observed in Na-flux GaN crystal grown on GaN seed substrate grown using HVPE, Na-flux and Ammonothermal method. Here, we assumed that TDs originated in seed substrate propagated directly into the grown substrate.

In order to perform a fair evaluation, smooth interface Na-flux GaN layers were grown on three different seed substrates grown under same experimental conditions (Temperature: 870°C, Pressure: 3MPa, Duration: 24 h). Next, the samples were then chemically etched for the analysis of TD character in grown crystals by immersing them in a molten KOH/NaOH eutectic mixture at 450°C for 15 minutes. Sizes of 300 etch-pits were measured from each sample using an optical microscope via a 100x objective lens.

TD density of Na-flux crystal grown on HVPE, Na-flux-grown and Ammonothermal-grown seed substrate was of the order of 10^6 cm⁻², 10^5 cm⁻² and 10^5 cm⁻² respectively. The etch-pit size measurement results revealed the differences in etch-pit size range of the samples. GaN crystal grown on HVPE seed had the largest etch-pit size range of $[0.4 \, \mu m \sim 5.6 \, \mu m]$, followed by GaN crystal grown on Ammonothermal GaN with range of $[0.4 \, \mu m \sim 3.5 \, \mu m]$, and GaN crystal grown on Na-flux GaN had the smallest etch-pit size range of $[0.4 \, \mu m \sim 2.5 \, \mu m]$. Since, all the samples were grown and etched under the same experimental conditions, it can be derived that the TDs propagated from seed substrate to grown crystal are contributing to these variations in etch-pit sizes. Such variations in the etch-pit size range implies the presence of different types of TDs in each sample. We plan to classify the TDs based on etch-pit sizes and further investigate the burger vectors of TDs in each sample using transmission electron microscopy.

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References

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