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Growth Evolution of AlN on Sapphire at High Temperature

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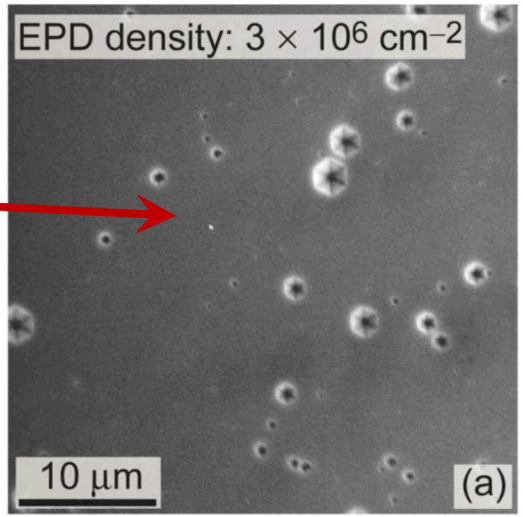
Issues with AlN: Etch Pits and Reproducibility

Etch Pits cause electrical leakage in AlGaN Devices

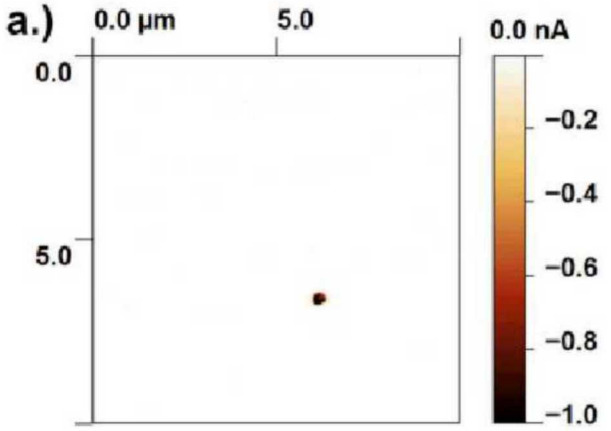
*Xi, et al. Appl. Phys. Lett **89**, 103106 (2006).*

The AlN etched in 50% KOH solution heated to 70 °C.

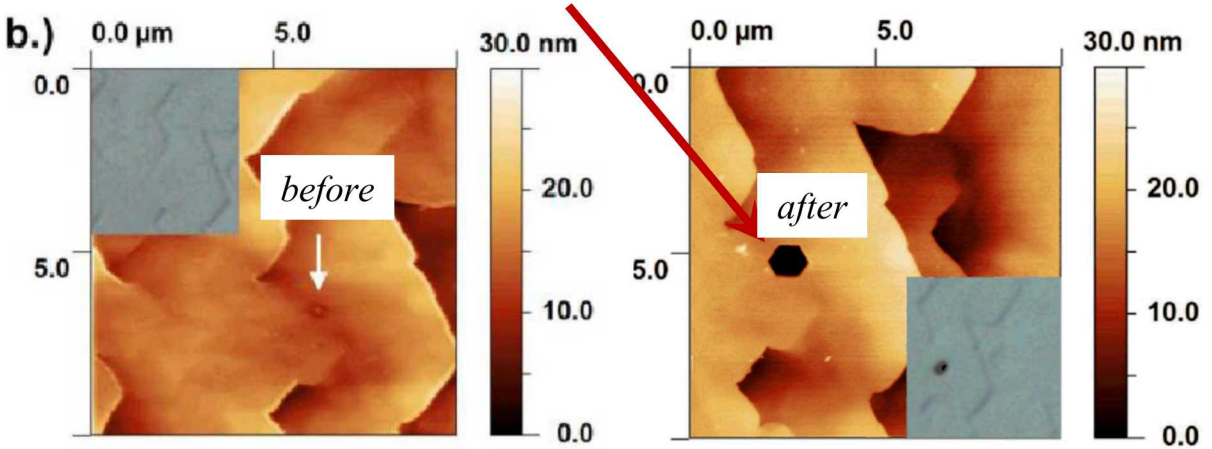


*Moseley, et al., J. Appl. Phys. **117**, 095301 (2015).*
Moseley, et al., Phys. Status Solidi A 212, 709 (2015).

Open-core dislocations that contributed to <u>electrical leakage</u> identified in $Al_{0.7}Ga_{0.3}N$ UV-LEDs. Used hot Phosphoric acid to decorate the open-core dislocations.



Conductive AFM measurement

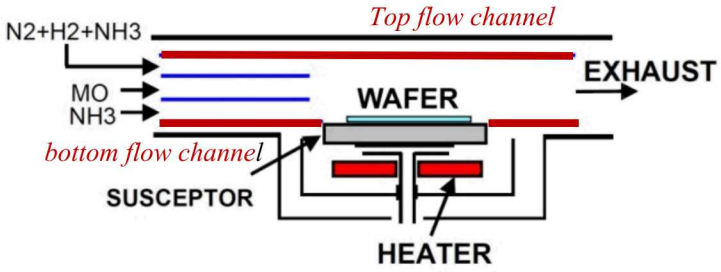


AFM Images before and after etching in hot phosphoric acid. Conductive EP is revealed.

HT SR4000 System

Advertised advantages of TNSC system

1. *3 layer horizontal laminar flow.*
2. *Growth at atmospheric pressure.*
3. *Prevent gas phase mixing reactions.*



4. Top and bottom quartz-ware flow channels, susceptor, and susceptor cover can be easily exchanged between growths.

GaN → AlN → GaN

Growth monitoring LayTec epiTT and EpiCurve

Reproducibility? – Repeat same growth recipe over and over

1320 °C AlN 2.7 µm	7). Grow AlN,1 SLM NH_3 for 50 min ~ GR > 3 µm/hr. 6). Decrease pressure to 13 kPa. 5). Heat to 1320 ºC.
920 °C AlN 100 nm	4). Grow AlN NL, 0.3 SLM NH_3. 3). Dose 1 SLM NH_3 for 7 min.
Sapphire	2). Increase pressure to 40 kPa. 1). Heat sapphire to 920 ºC.

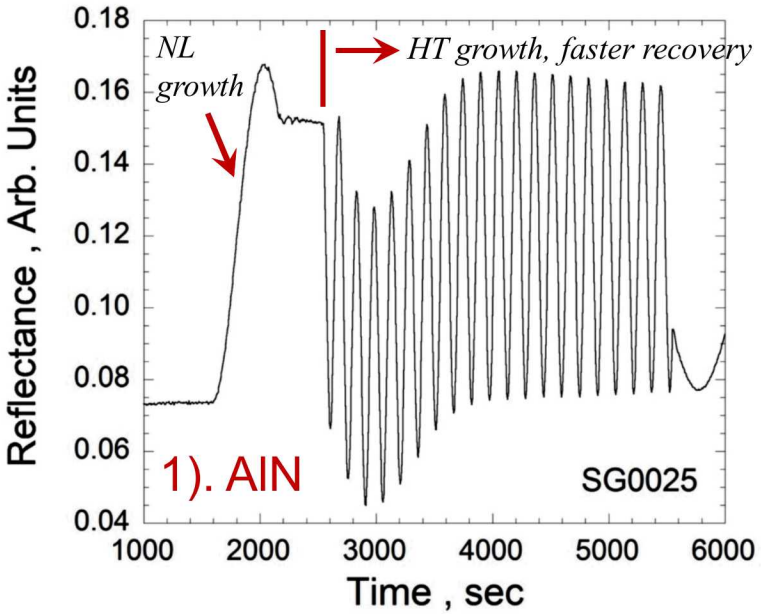
Recipe developed by A. Mishima and K. Ikenaga during tool install.

40 kPa ~ 300 torr, 13 kPa ~ 100 torr

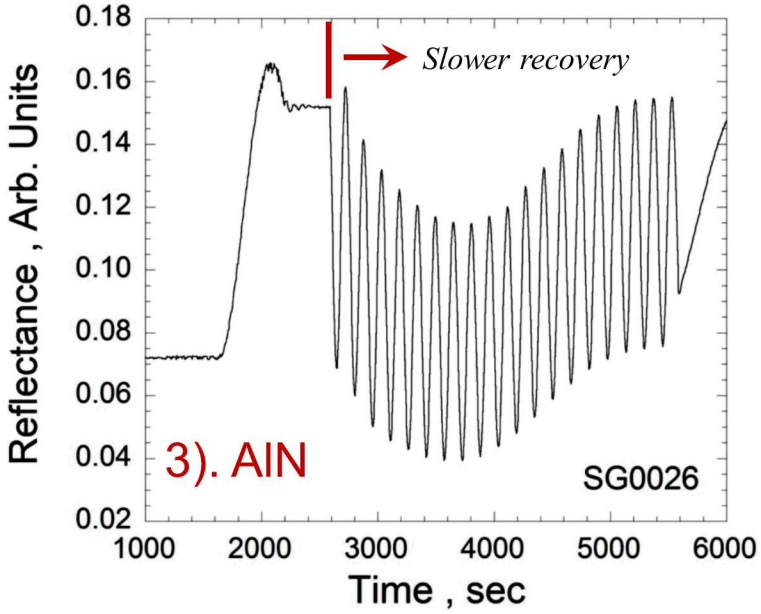
Two consecutive AlN growths

Process:
1). AlN growth recipe (last slide)
2). Do something (ex. H_2 bake)
3). AlN growth recipe.
…repeat 2) and 3).

Film Quantification:
1). Morphology: AFM and Nomarski microscope
2). XRD: (0002) and (10-12) FWHM
3). Etch Pit Density: 70 ºC KOH solution
4). Reflectance: LayTec epiTT (405, 633, 951 nm)



2). Do Nothing



Smoother film, Low Etch Pit Density
(0002) = 219 arcsec (10-12) = 528 arcsec

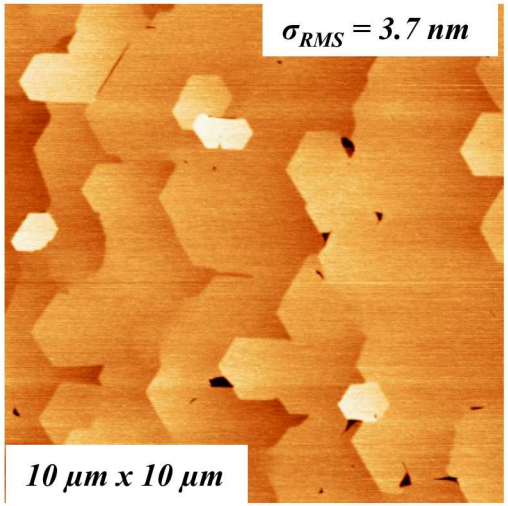
Rougher film, High Etch Pit Density
(0002) = 398 arcsec (10-12) = 558 arcsec

The Somethings That We Tried and What Worked

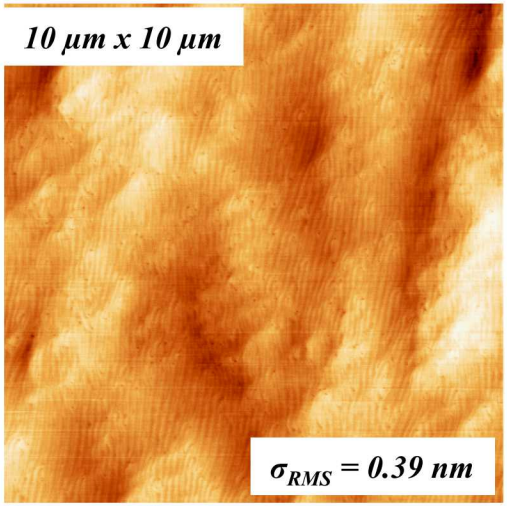
1. Nothing (data from the last slide) – no Bake.
2. Clean quartz-ware.
3. Bake in N_2.
4. Bake in H_2 (1100 ºC and 1300 ºC).
5. AlN growth with scrape afterwards – 3rd best.
6. Scrape the wafer susceptor cover – 2nd best.
7. Swap different sets of quartz-ware – best.

Achieved reproducible growth by swapping three sets of quartz-ware

Limited room in glove-box, so we stored quartz-ware in room air



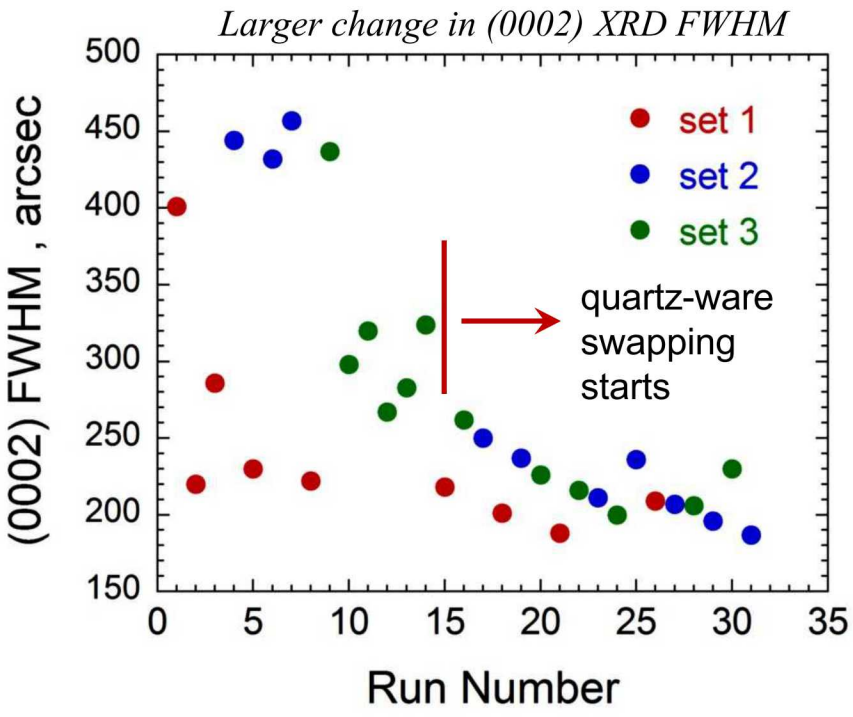
Slower recovery

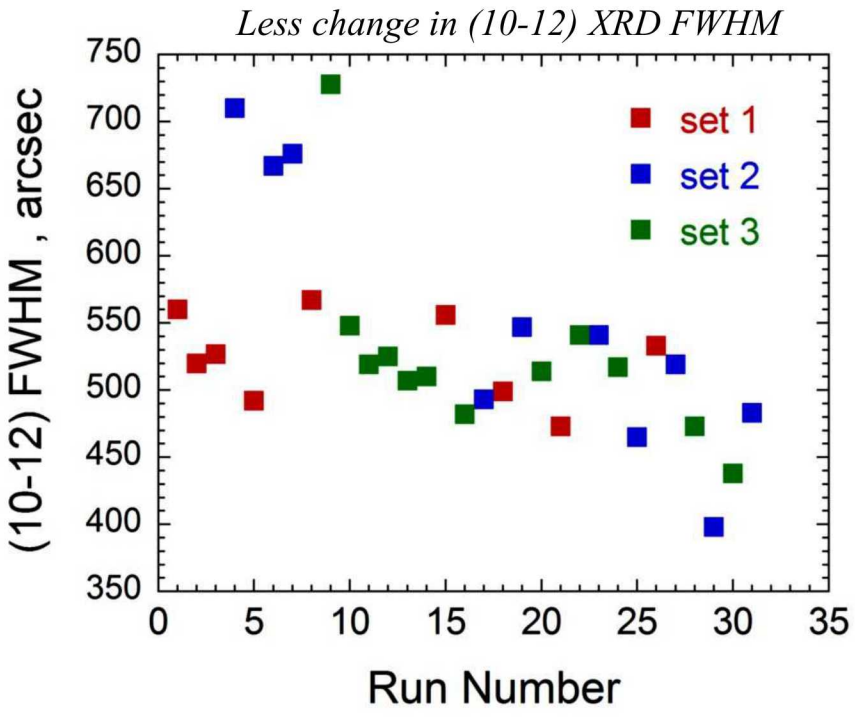


Faster recovery

uartz-ware Sets

- First 14 growth runs – less reproducibility – less influence on growth.
- At run 15 started to rotate three different sets of quartz-ware.





Achieved (0002) < 200 arcsec and (10-12) < 500 arcsec

Dislocation Density of 3×10^9 cm^{-2} based on Lee *et al*. APL 86, 241904 (2005).

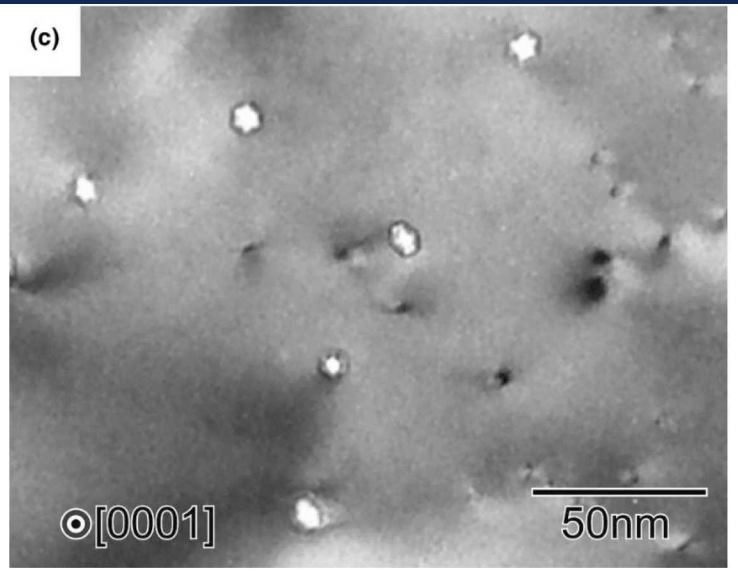
Etch Pits – Nanopipes – Open Core Screw Dislocations

Qian, et al. Appl. Phys. Lett. 67, 2284 (1995).

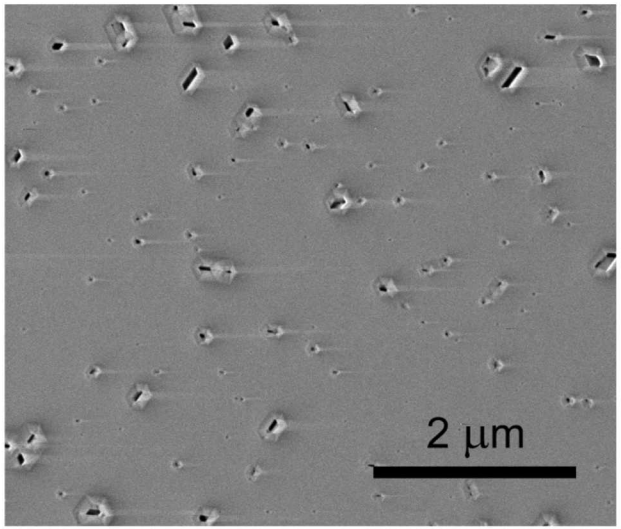
Observed nanopipes in GaN films on sapphire.
Identified as open core screw dislocations.

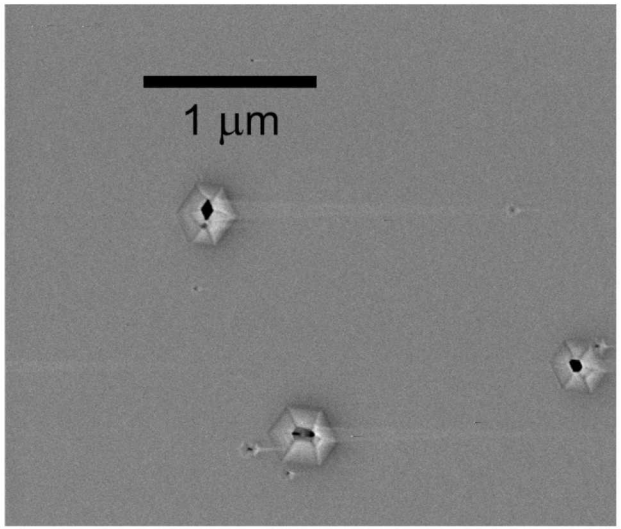
Tokumoto, et al. J. Mater. Res. 23, 2188 (2008).

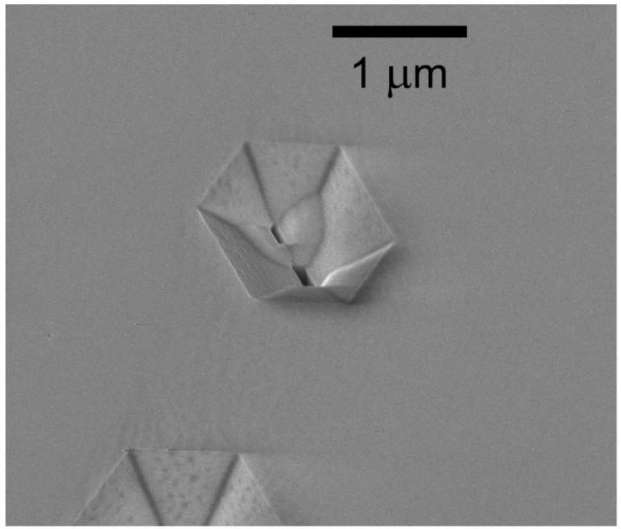
Identified similar structures for AlN on sapphire in TEM plan-view.



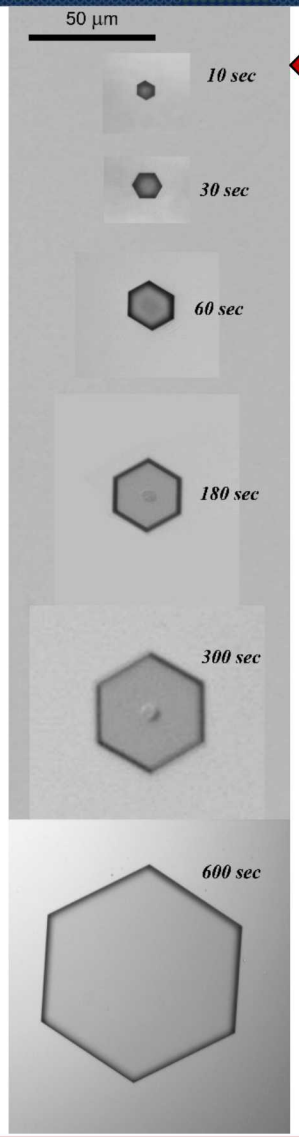
SEM images of nanopipes in our AlN on sapphire



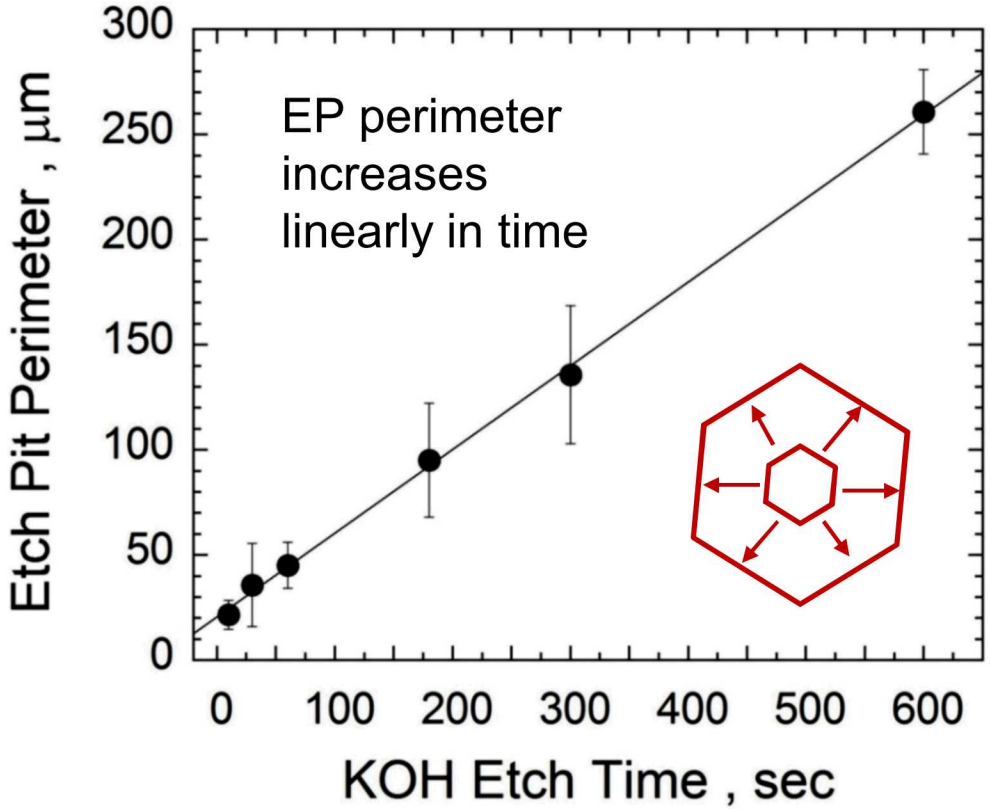




How the Etch Pit Density Was Quantified



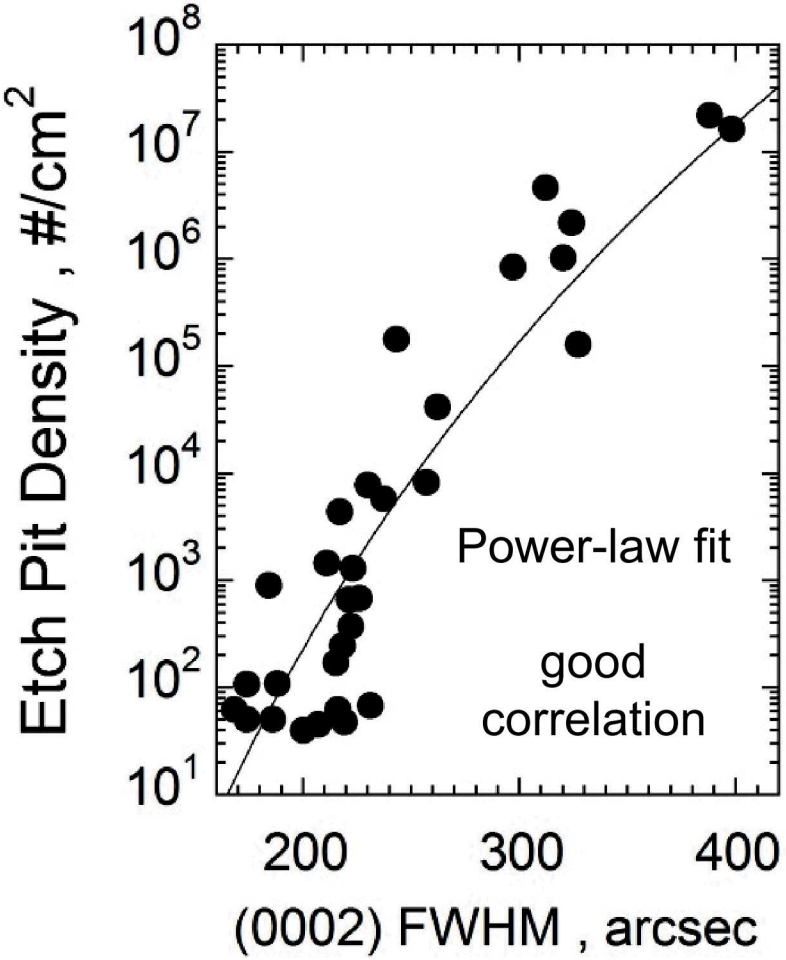
Etch pit size depended on KOH etching time and KOH concentration

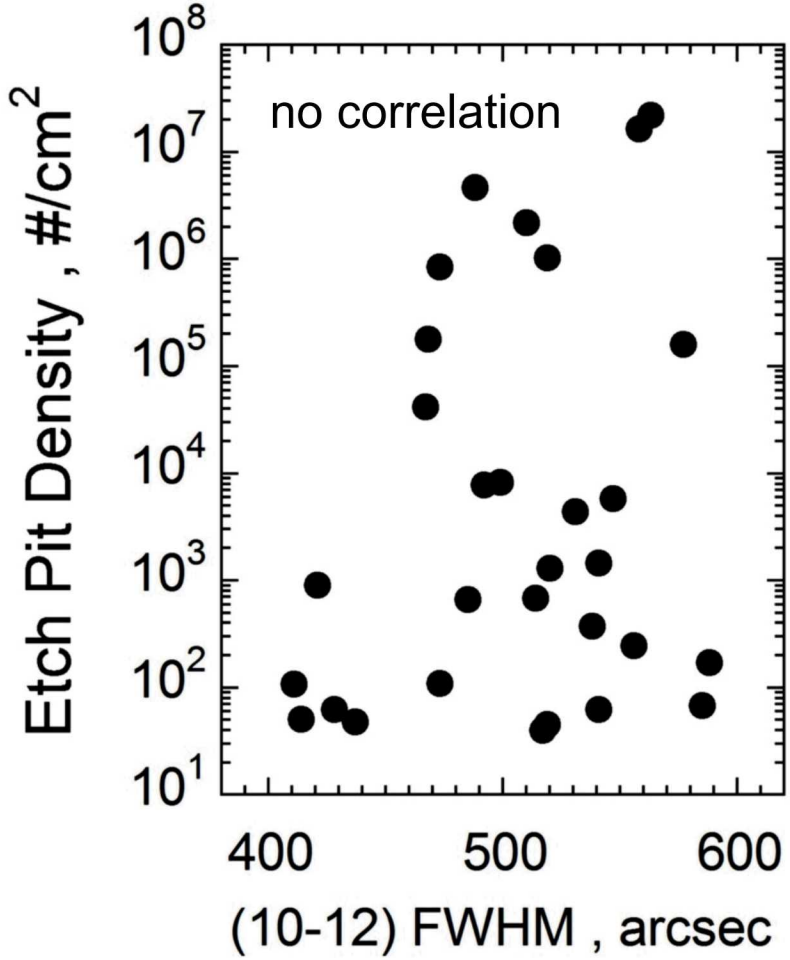


Useful for distinguishing open from closed core screw dislocations.

For AlN films suspected of having fewer EPDs, use longer etch times.

Correlation between (0002) XRD FWHM and EPD

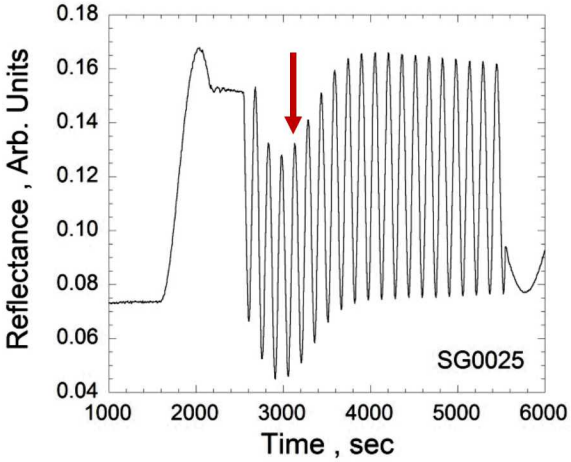




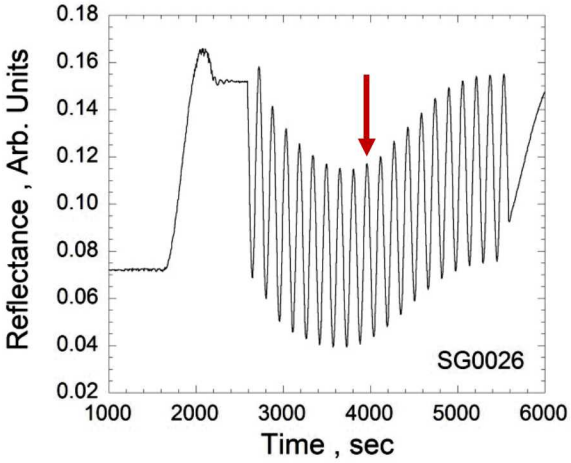
Achieved AlN with EPD < 100 cm^{-2}

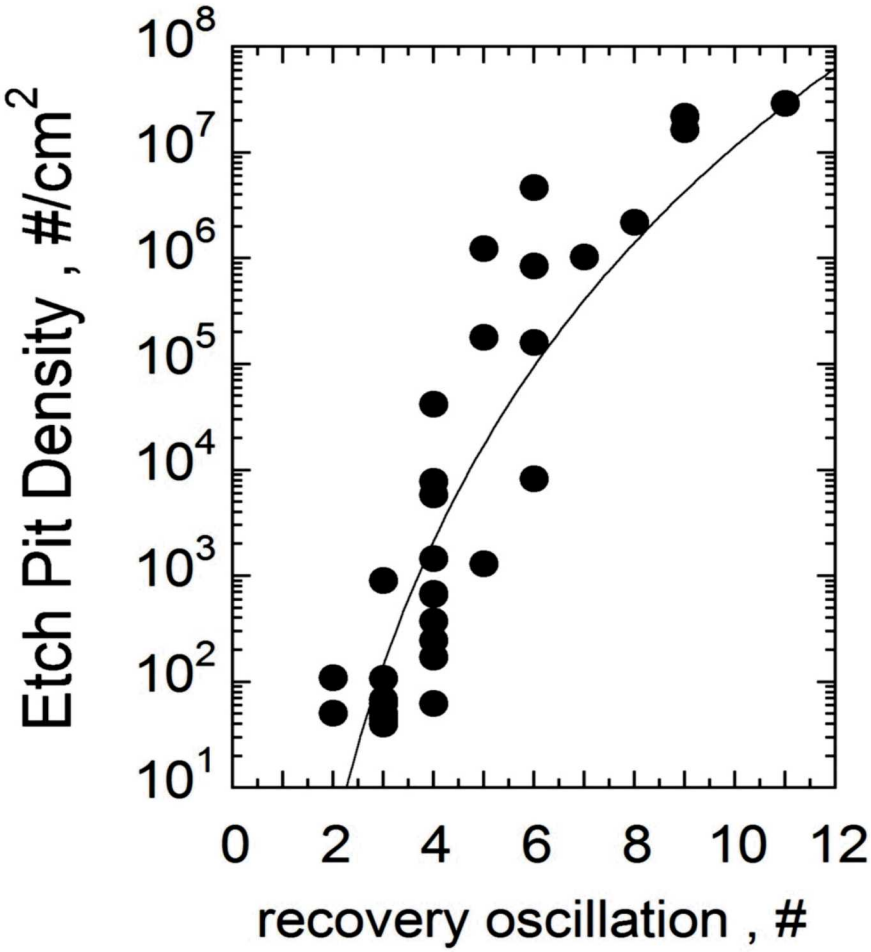
Correlation between reflectance recovery and EPD

Recovery begins after 4 oscillations



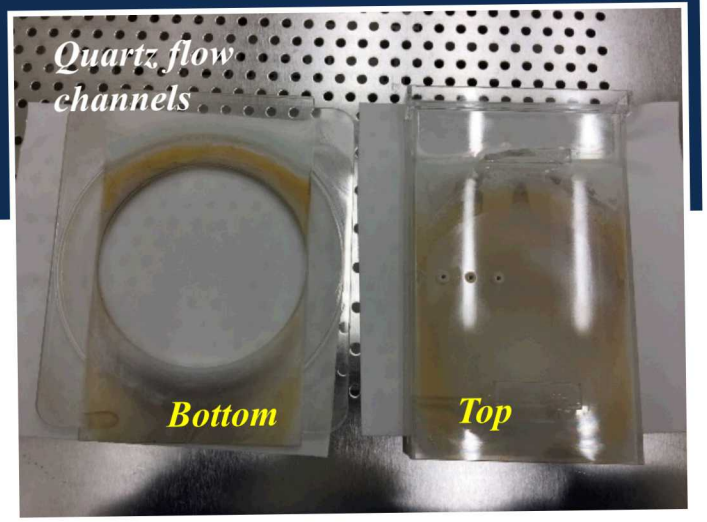
Recovery begins after 9 oscillations



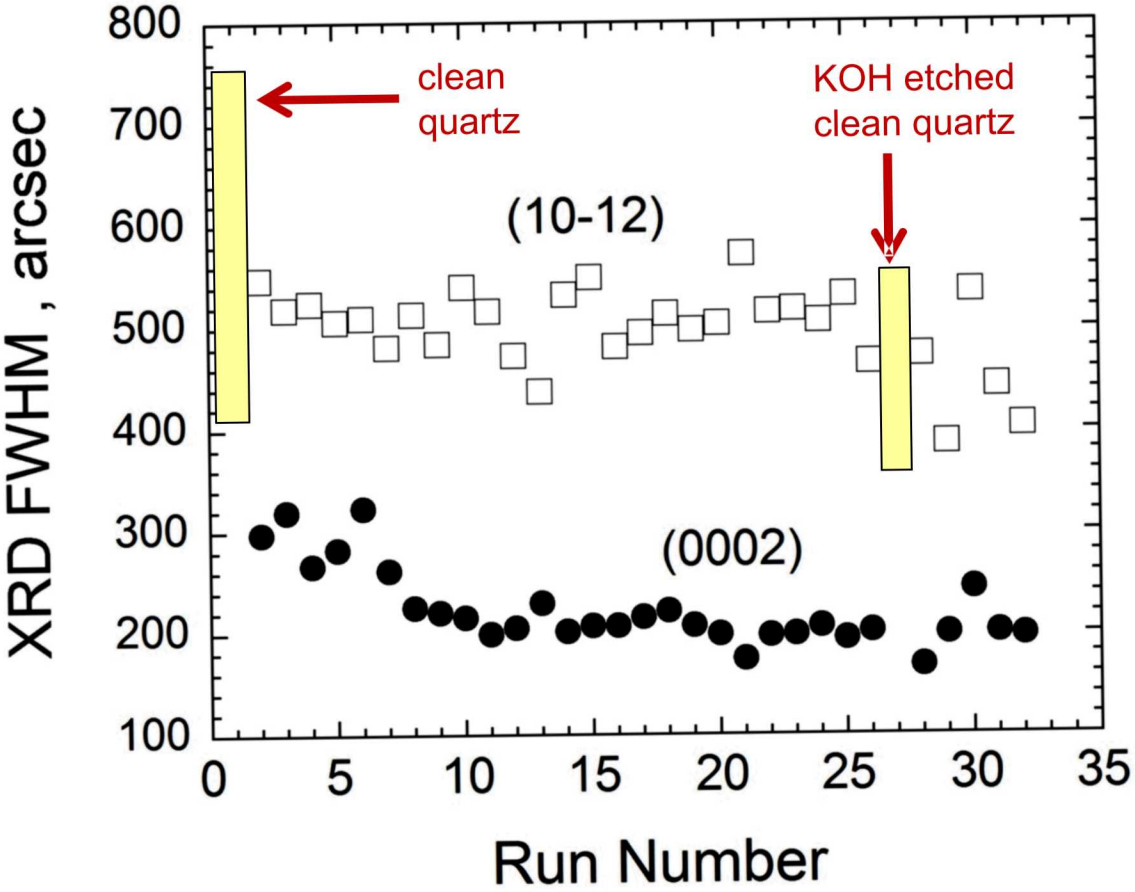


Use the reflectance waveform as a predictor of EPD → faster recovery → lower EPD

Set 3 Quartz-ware – Clean to Coated



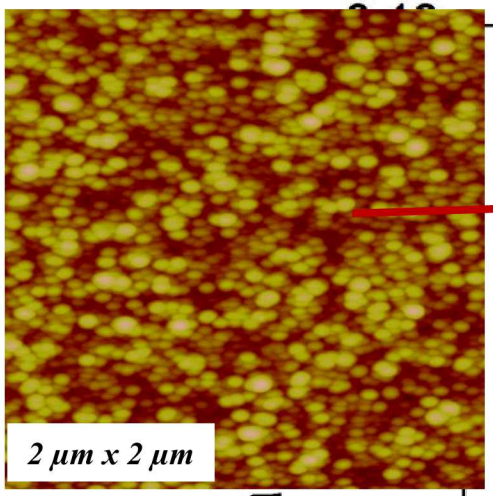
Caveat: Takes a couple of growth runs to sufficiently coat the quartz-ware.

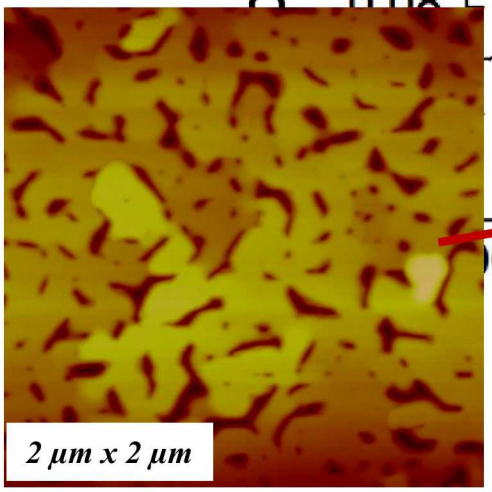


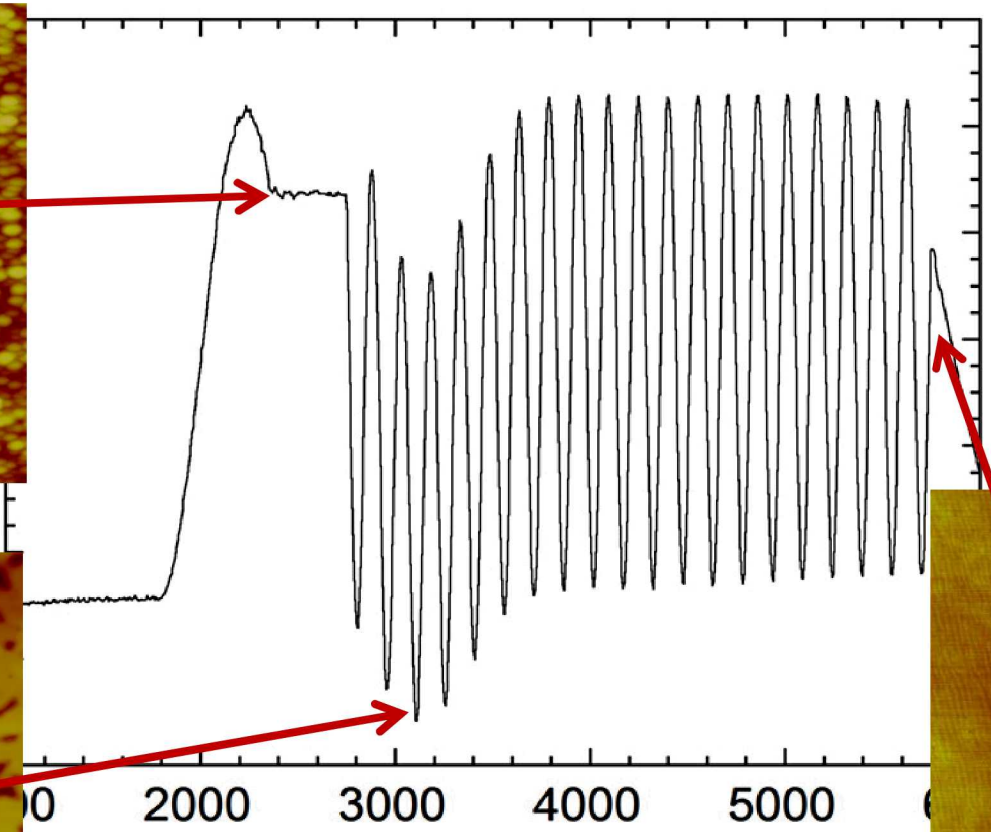
- Consistent XRD and EPD results over time.
- The first run after KOH etching (run 28) showed higher (0002) XRD and higher EPD.
- The second run showed AlN with similar quality to before KOH etching.

progress

100 nm AlN NL growth, σ_{RMS} = 6.8 nm





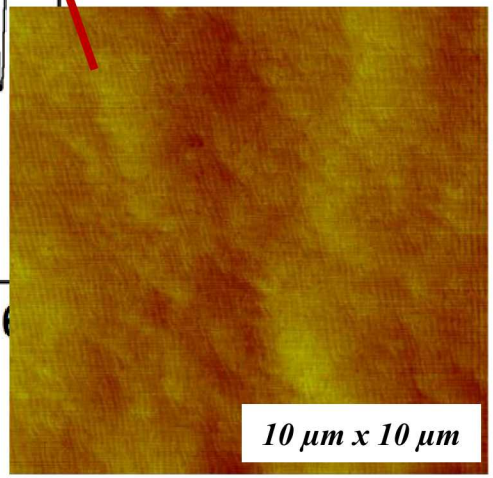


350 nm HT AlN growth

σ_{RMS} = 6 nm

HT AlN growth 50 min.

σ_{RMS} = 0.30 nm

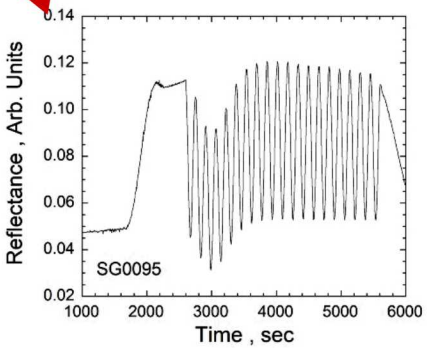


More on this at LayTec's 21st In-situ Seminar

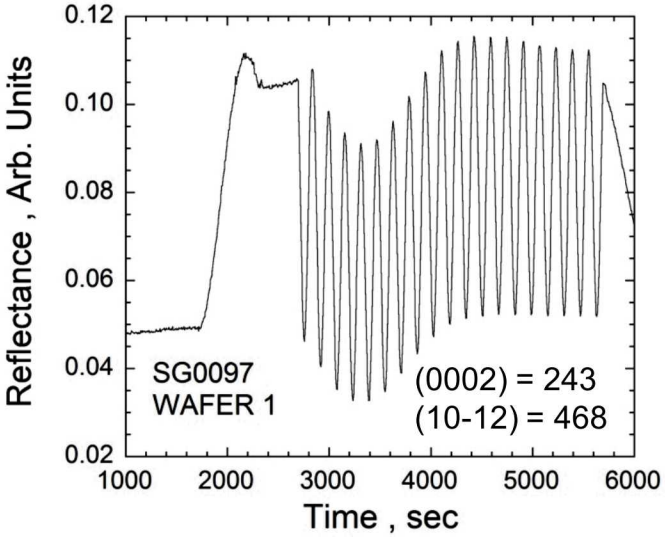
Before NL Growth

H_2 1100 ºC bake in between each run
Keep set 2 quartz-ware in whole time.

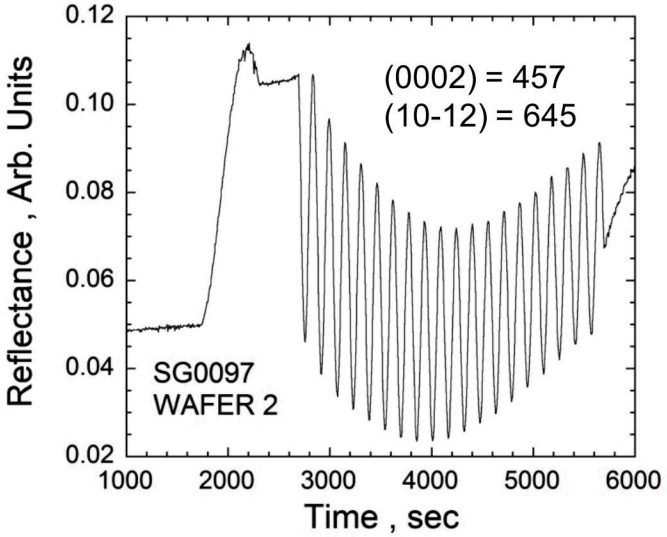
- Put in quartz-ware after sitting out overnight
- Run SG0094: Grow only 30 sec. of the AlN NL (WAFER 1)
- Run SG0095: Full AlN growth run, 50 min. HT AlN.
- Run SG0096: Grow only 30 sec. of the AlN NL (WAFER 2)
- Run SG0097: On WAFER 1 and 2 complete AlN NL (9 min.) and HT AlN.



(0002) = 219
(10-12) = 498



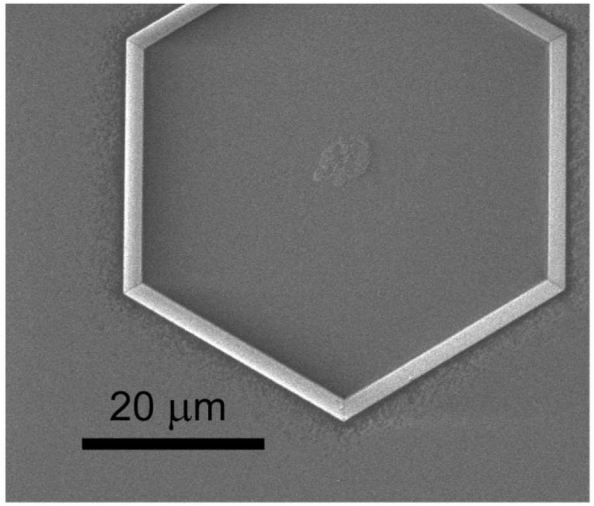
EPD = 2.1×10^5

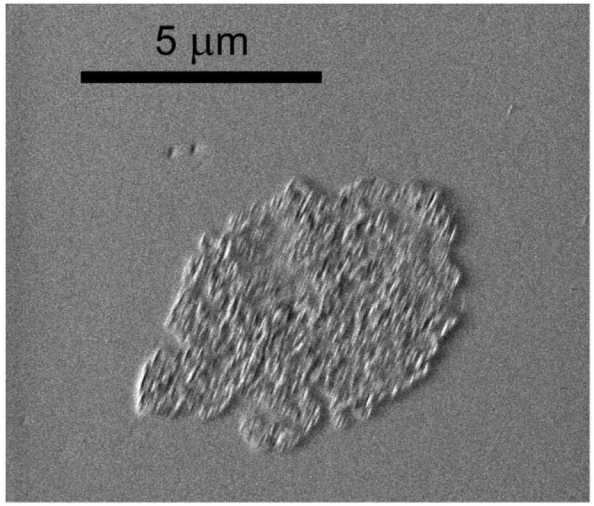


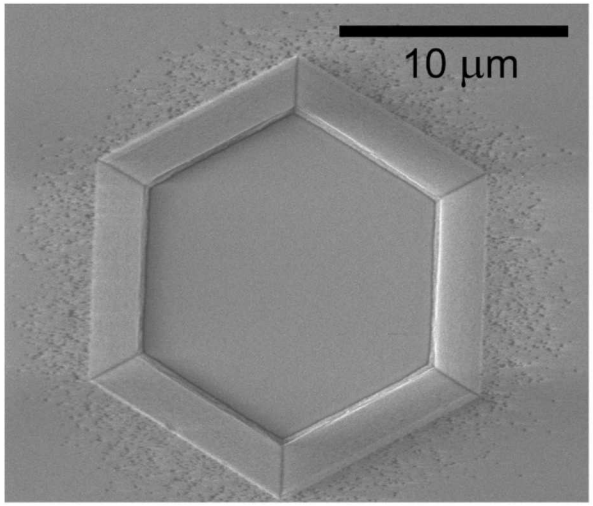
EPD = 1.1×10^7

What causes AlN with high EPDs?

- Leaving quartz-ware out of the glove box leads to reproducible AlN growth on sapphire – demonstrated using three sets of quartz-ware.
  - Use the same recipe - only change reactor conditioning between growth runs.
  - Need to test individual gaseous components of air.
- AlN fate is set early on – possibly during heat up, NH_3 exposure?
- Observe residue in the center of larger etch pits (not smaller pits).
  - Possible particulate deposited before heating to AlN NL growth.







Summary

- Achieved reproducible AlN growth on sapphire in TNSC system.
  - Involves conditioning quartz-ware in room air – overnight best.
  - Best AlN has (0002) = 189 arcsec, (10-12) = 386 arcsec, and EPD = 45/cm^2.
  - Estimated total dislocation density of 1.5x10^9 cm^{-2} from XRD.
- Etch Pit Density correlates with (0002) FWHM.
  - Etch pits are open core screw dislocations that are formed from surface contamination early on during the AlN growth.
- The reflectance waveform can be used to predict AlN film quality.
  - Shorter recovery time → lower (0002) FWHM and lower EPDs.
- In abstract, the degree of sapphire nitridation, AlN NL growth temperature and thickness, and HT growth rate have less influence on the AlN quality that the quartz-ware conditioning.