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Low Etch Pit Density AlN on Sapphire

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UWBG ULTRAWIDE BANDGAP POWER ELECTRONICS

U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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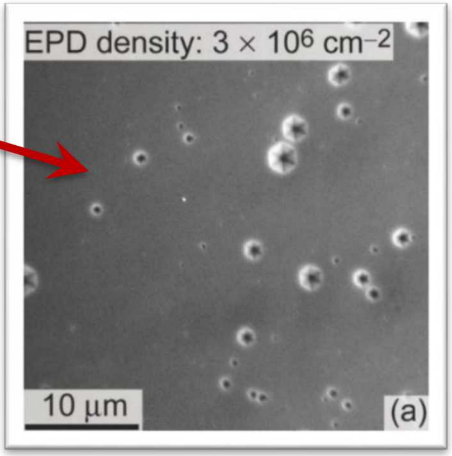
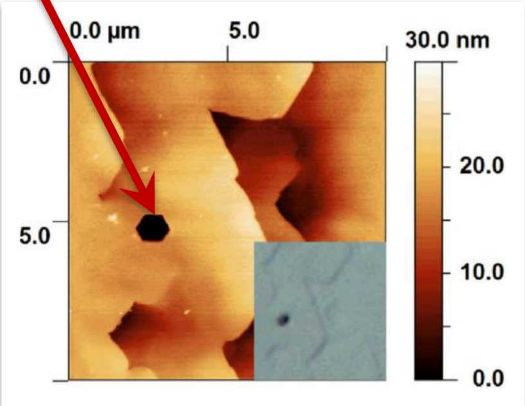
Issues with AlN growth on sapphire: Etch Pit Density (EPD) and Reproducibility

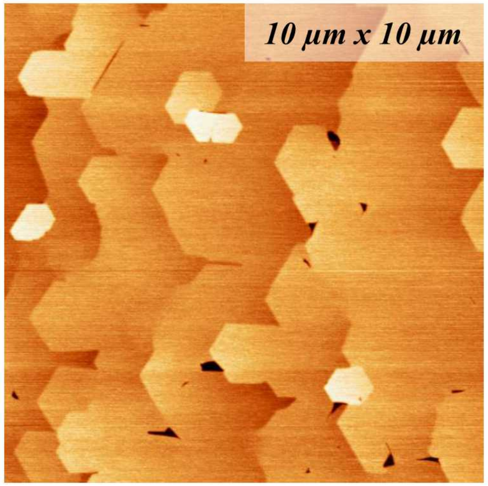
- Hot KOH etching can reveal open core screw dislocations. These are denoted as etch pits.
- Open core screw dislocations cause electrical leakage in $Al_{0.7}Ga_{0.3}N$ UV-LEDs.
- Reproducible AlN growth on sapphire is difficult due to unknown factors – coatings, adducts?



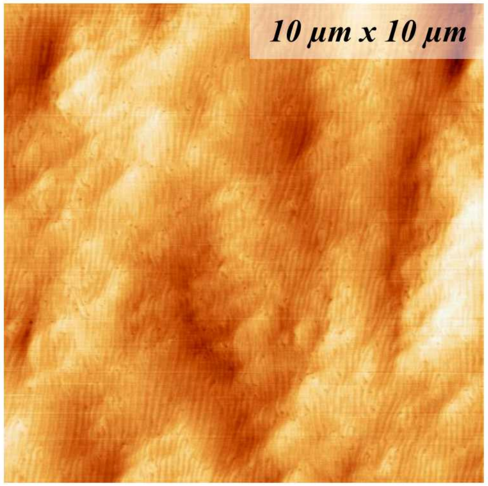
Xi, APL ***89****, 103106 (2006).*



Moseley, JAP ***117****, 095301 (2015).* Conductive AFM confirmed current leakage pathways. Etching in hot H_3PO_4 revealed open core screw dislocation at this location.



Macro steps, σ_{RMS} = 3.7 nm



Single layer steps, σ_{RMS} = 0.39 nm

AlN in Taiyo Nippon Sanso HT SR4000 System

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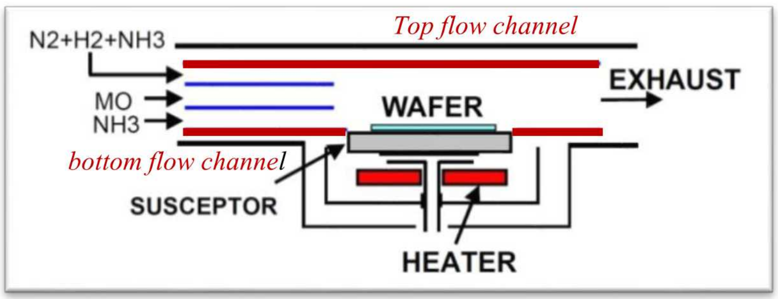
<u>Advertised advantages of TNSC system</u>

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

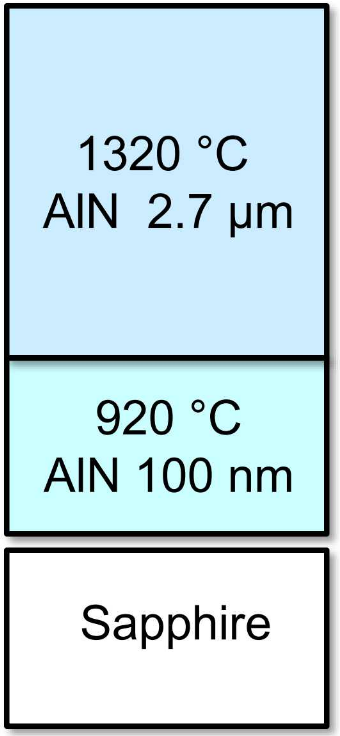


4. Top and bottom quartz-ware flow channels, susceptor, and susceptor cover can be easily exchanged between growths.

GaN → AlN → GaN

Growth monitoring LayTec epiTT and EpiCurve

<u>Reproducibility? – Repeat same growth recipe over and over</u>



7). Grow AlN,1 SLM NH_3 for 50 min ~ **GR > 3 µm/hr**.

6). Lower pressure to 13 kPa.

5). Heat to **1320 ºC**.

4). Grow AlN NL, 0.3 SLM NH_3.

3). **Dose 1 SLM NH_3 for 7 min**.

2). Raise pressure to 40 kPa.

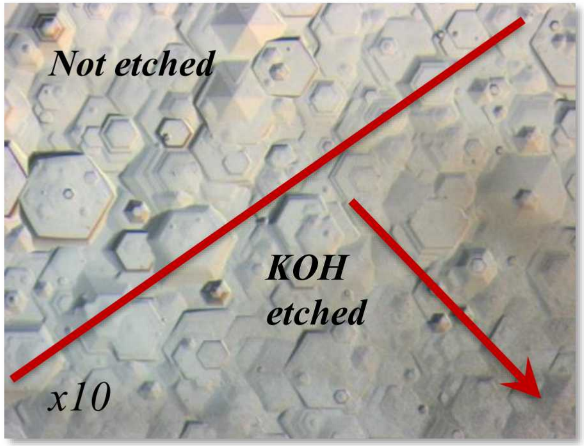
1). Heat sapphire to 920 ºC.

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

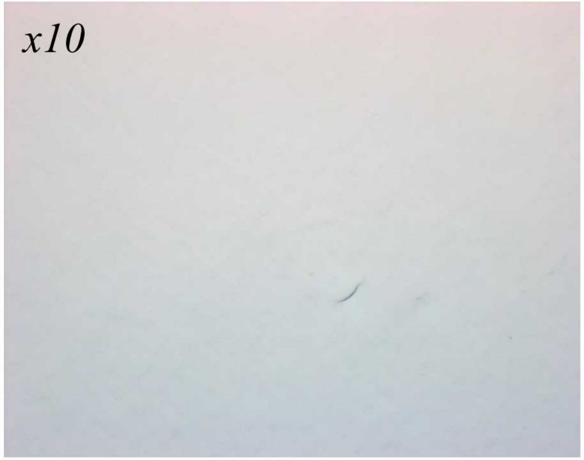
40 kPa ~ 300 torr, 13 kPa ~ 100 torr

Polarity of the AlN nucleation and HT AlN layers

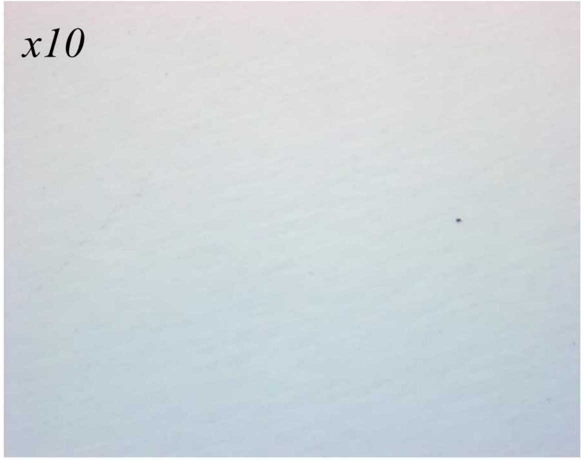
- Nitridation (7 min.) of sapphire at 920 °C produces N-polar AlN NL.
  - Get N-polar GaN when we grow on the AlN NLs.



- High temperature AlN on the N-polar NLs is Al-polar.
  - Get Ga-polar GaN when we grow on the HT AlN. No pits after KOH etch.



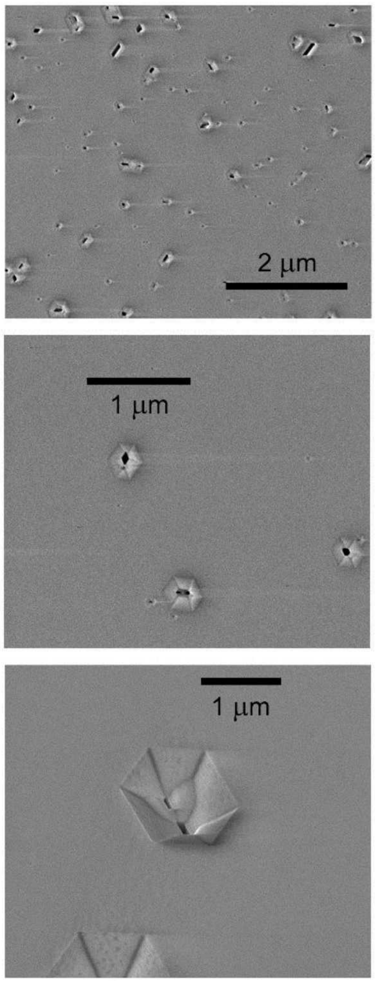
GaN growth on top of HT AlN.



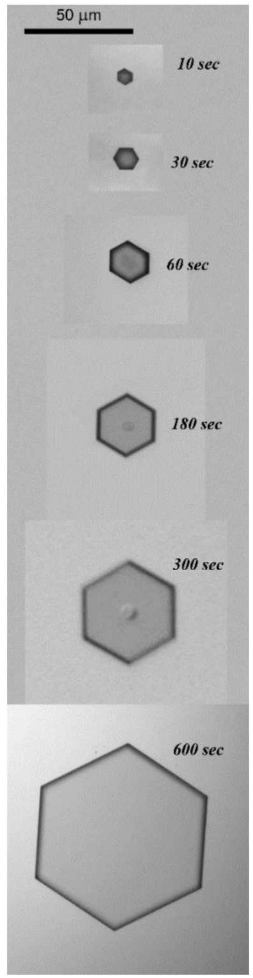
GaN after 10 min. KOH etching at 70 °C

Quantifying the Etch Pit Density (EPD)

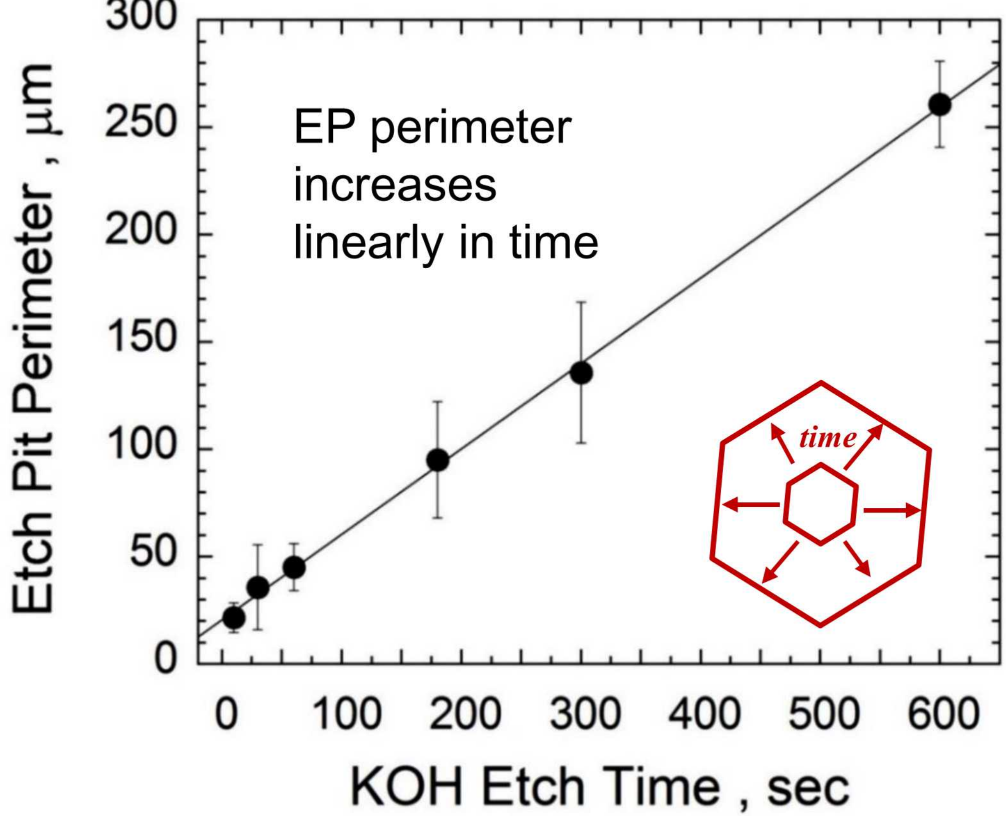
Not all features are open core screw dislocations



Etch pit size vs. KOH etching time



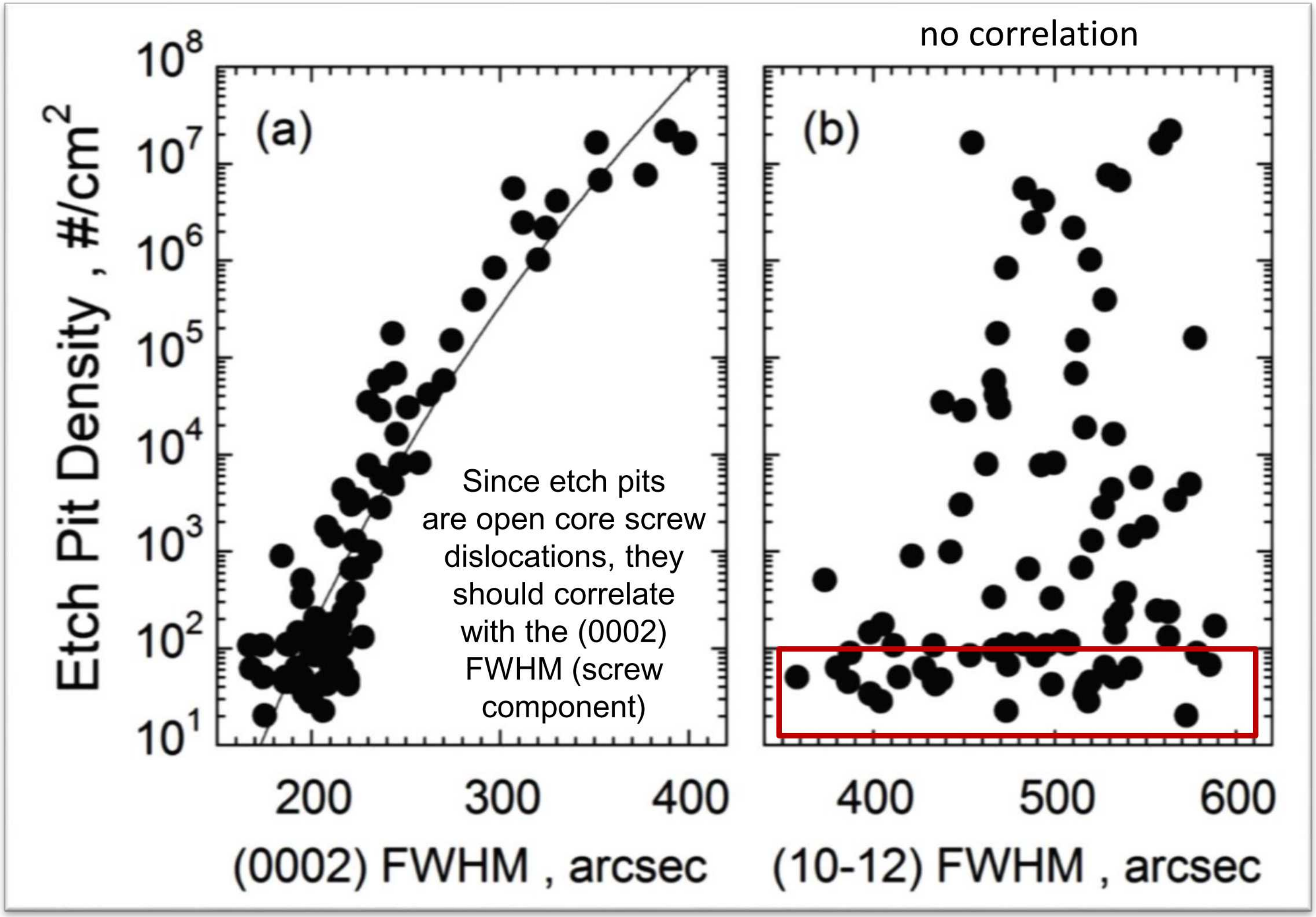
Etch pit size depends on etching time



Largest etch features are what we are measuring.

Use longer etch times for films with lower EPDs.

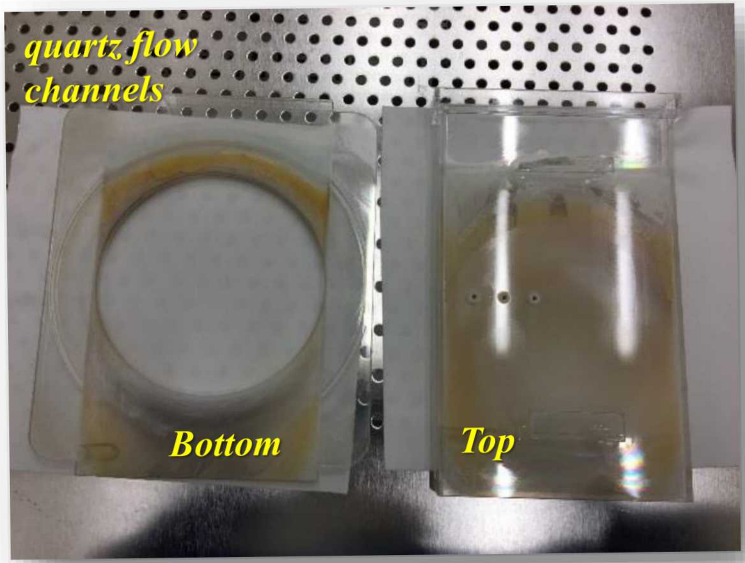
Correlation between (0002) XRD FWHM and EPD



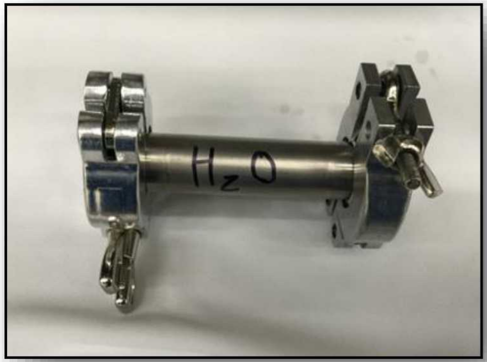
27 samples of AlN on sapphire with EPD < 100 cm^{-2}

How AlN films with low EPD were achieved.

- Initially, noticed better AlN films were achieved when quartz-ware was taken out and placed in room air. Over night - best!
- Achieved lower EPD when 3 sets of quartz-ware were swapped out, exposed to room air, then put back in for growth.
- Baking quartz in H_2 and/or N_2 had little effect, although standard practice was to bake quartz in H_2 to 1300 ^oC before AlN growth.
- Quartz had yellow/brownish coating. This coating is needed for best results.
- Suspected that O_2 or H_2O reacted with the AlN coated quartz which passivates or conditions the surface.

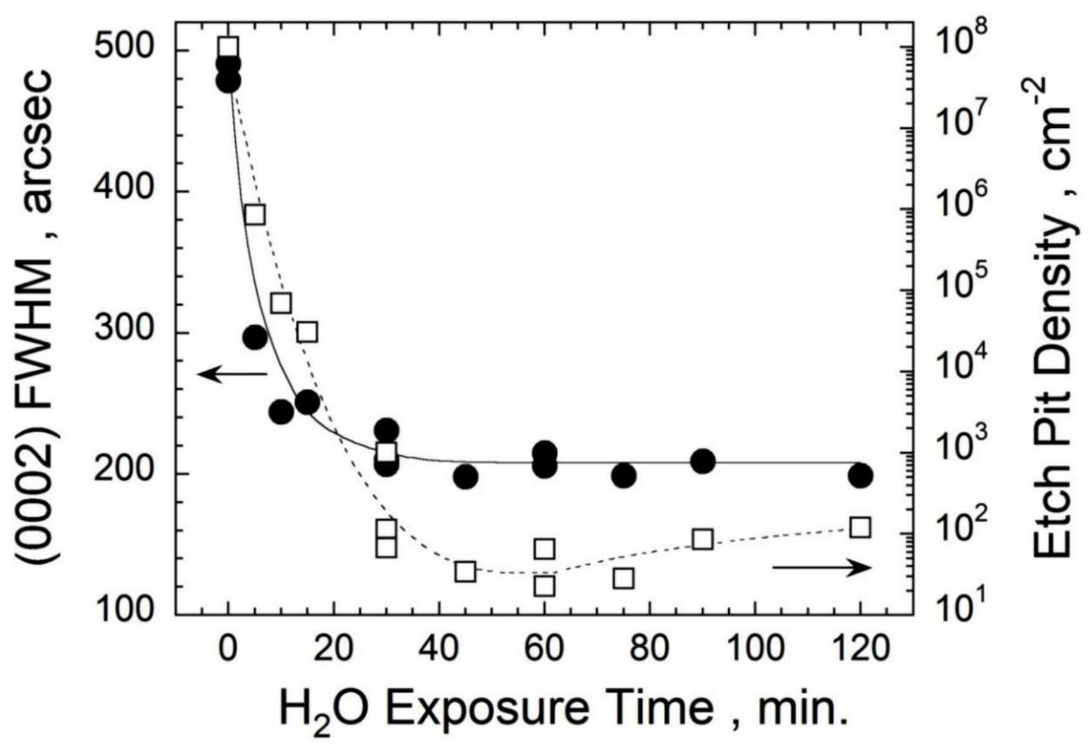


Expose quartz-ware to H_2O in N_2 purged glovebox



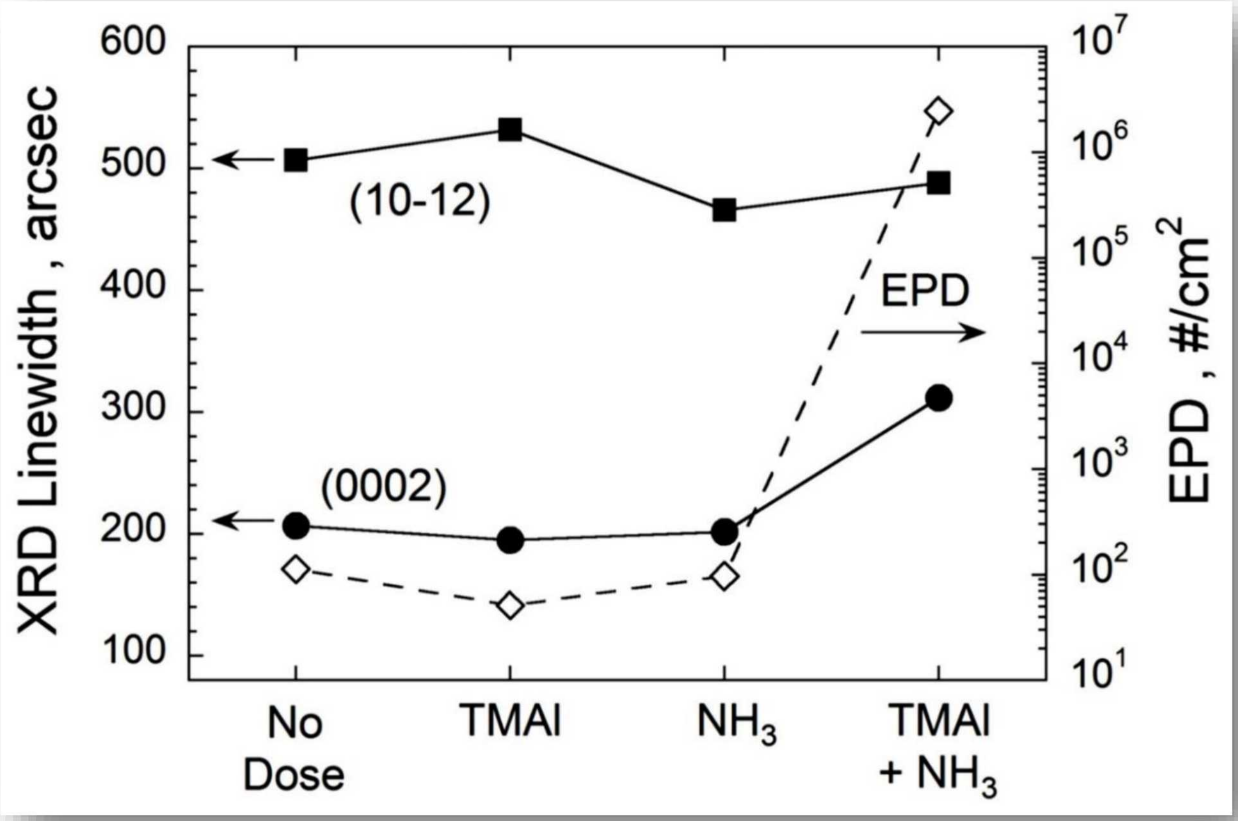
- Loaded DI H_2O into KF fitting. Keep in glovebox.
- Take quartz out of reactor, close reactor, expose quartz to H_2O in glovebox, seal H_2O in KF, wait 30 min., return quartz to reactor, 1300 ^{o}C bake in H_2.

- (0002) FWHM reaches of 220 arcsec for H_2O exposures of ≥ 30 min.
- EPD < 100 cm^{-2} after 30-90 min. of H_2O exposure.



What is the cause? Chemistry?

- Condition quartz with H_20 treatment. Same starting point growth run.
- During 30 min. H_2 bake, introduce 30 sec. flows of TMAl, NH_3, or both.
- Grow AlN using standard recipe.



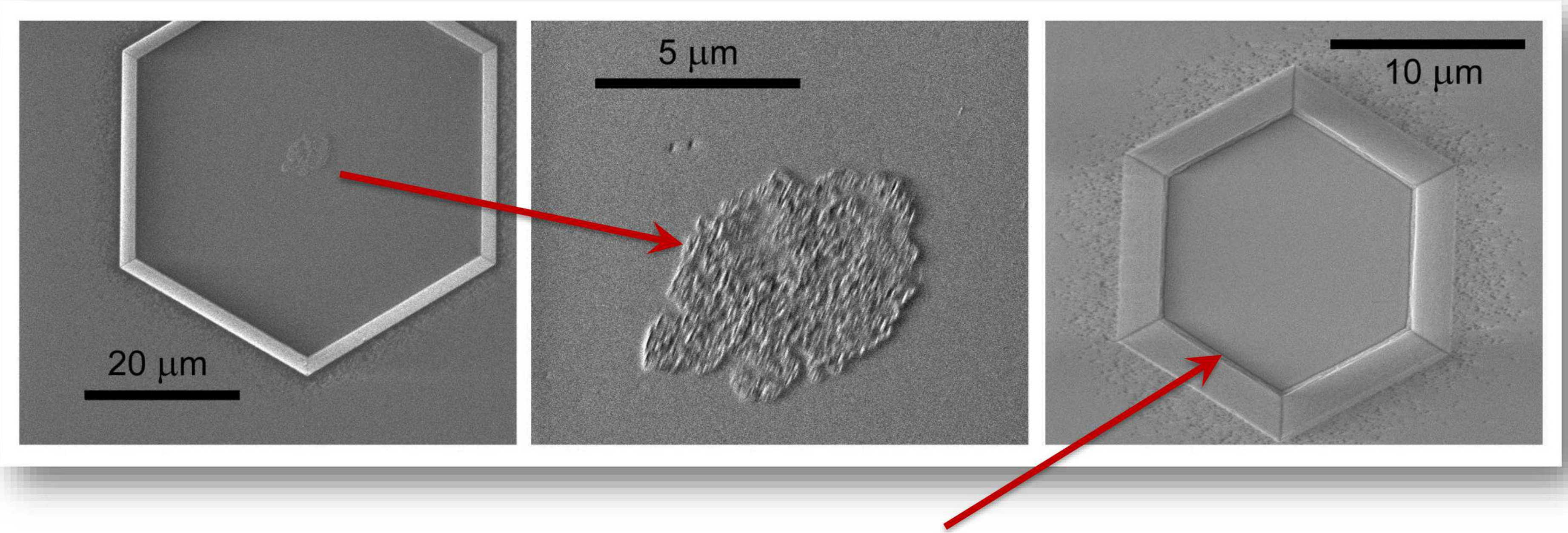
Suggests TMAl + NH_3 adduct?

Other experiments suggest that the AlN film fate is determined early on.

Possible during the heating of the wafer before growth.

What is the Cause? Particulate?

- Observe residue in the center of larger etch pits.
- Possible particulate deposited during initial heat up, before NH_3.
- Is this particulate or some un-etched region of the etch pit?

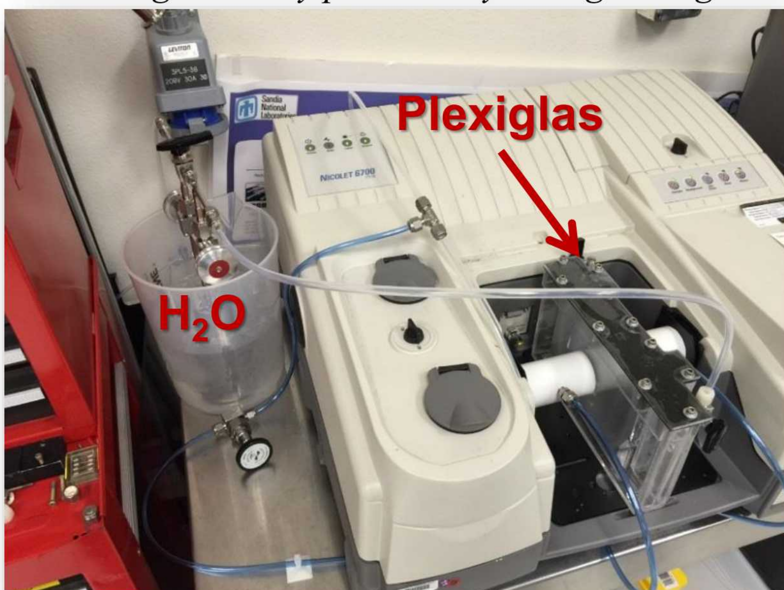


- Do not always observe particulate in etch pits – not quantified.

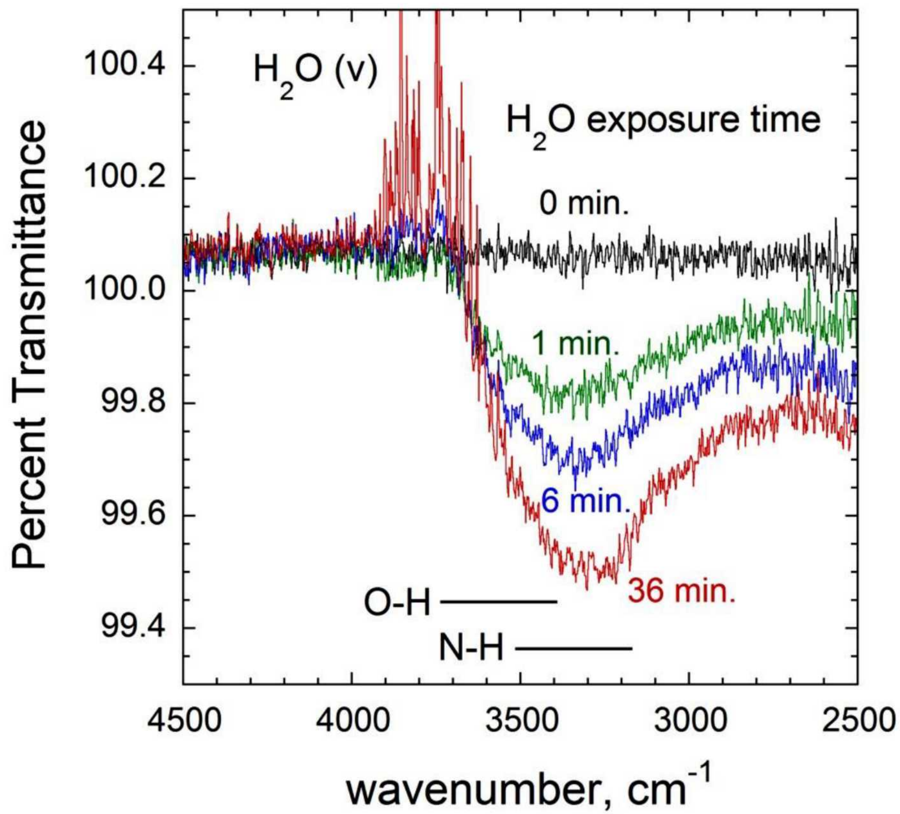
FTIR measurements of AlN coated quartz to H_2O

- On clean quartz grow 10 min. HT AlN.
- Loaded quartz into N_2 purged Plexiglas container with IR windows.
- Flowed H_2O from a bubbler through the Plexiglas container.

FTIR use generously provided by George Wang



Observe an increase in O-H and N-H stretching modes after H_2O exposure.



Summary

- Achieve low EPD AlN on sapphire in TNSC system.
  - Repeat same AlN growth recipe....change quartz conditioning between runs.
  - Best AlN when quartz is exposed to room air (over night) or H_2O (30-90 min.).
  - Achieved 27 growth runs with EPD < 100 cm^{-2}.
- Possible causes of etch pits.
  - Residue from previous growth adsorbs on sapphire (residual MO + NH_3).
    - Reacts during heat up - passivates surface and/or prevent further growth.
    - FTIR measurement suggests H_2O reacts with AlN coated quartz possibly passivating these MO + NH_3 coatings. H_2O exposure reduces EPD.
  - Particulate lands on surface which passivates surface and/or prevents growth.
    - Unetched material located in center of many larger etch pits.

Thank - you