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In-situ Reflectance and Correlation to AlN quality on sapphire

UWBG
ULTRAWIDE BANDGAP
POWER ELECTRONICS

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U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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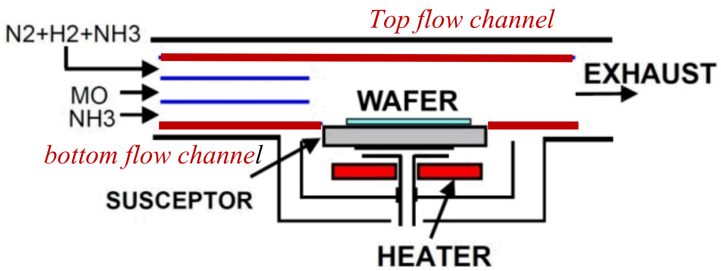
Recap: AlN in Taiyo Nippon Sanso SR4000

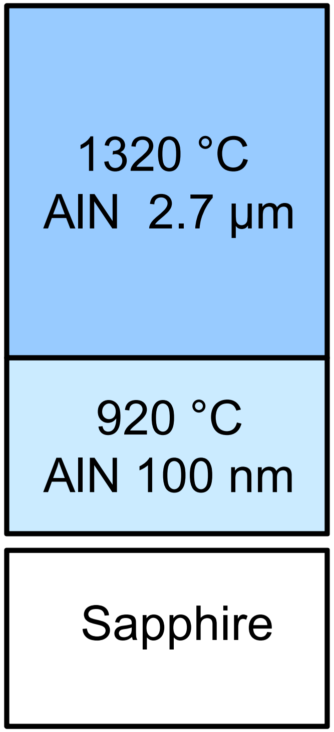
<u>*Advertised advantages of TNSC system*</u>

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.

<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). Decrease 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.

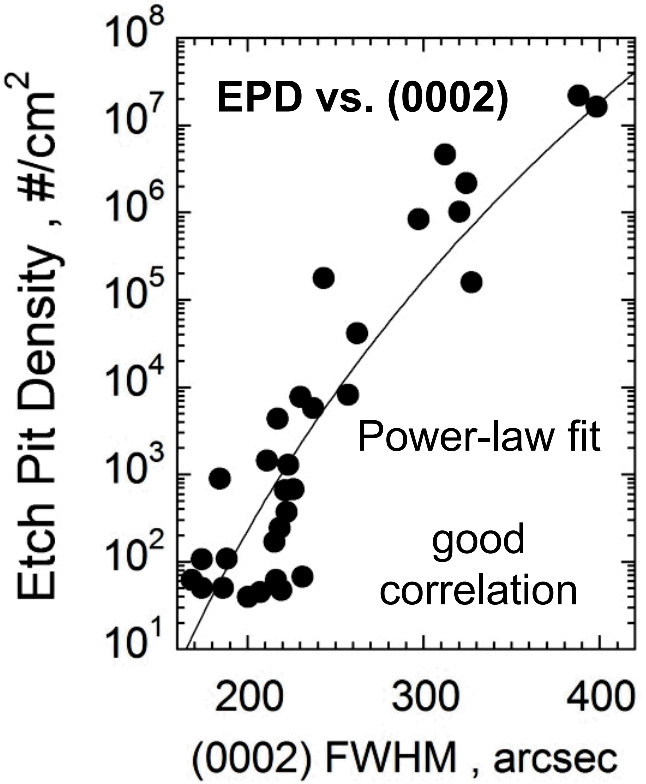
2). Increase pressure to 40 kPa.

1). Heat sapphire to 920 ºC.

Key finding: Exposing the quartz-ware to room air overnight allows reproducible HT AlN on sapphire. Three sets of quartz-ware were used in rotation to demonstrates this.

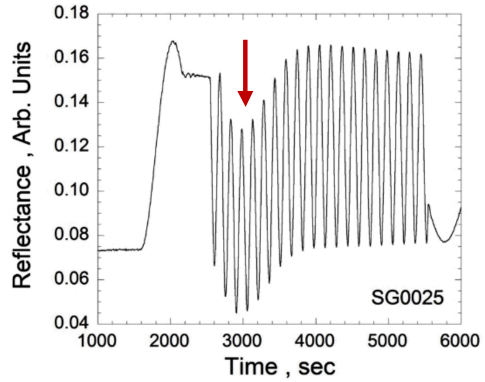
Correlations between XRD, reflectance, and EPD

Etch pits are open core screw dislocations – decorated by KOH etching

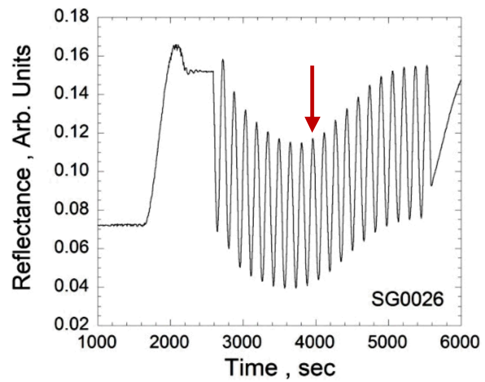


Lower (0002) FWHM – lower EPD

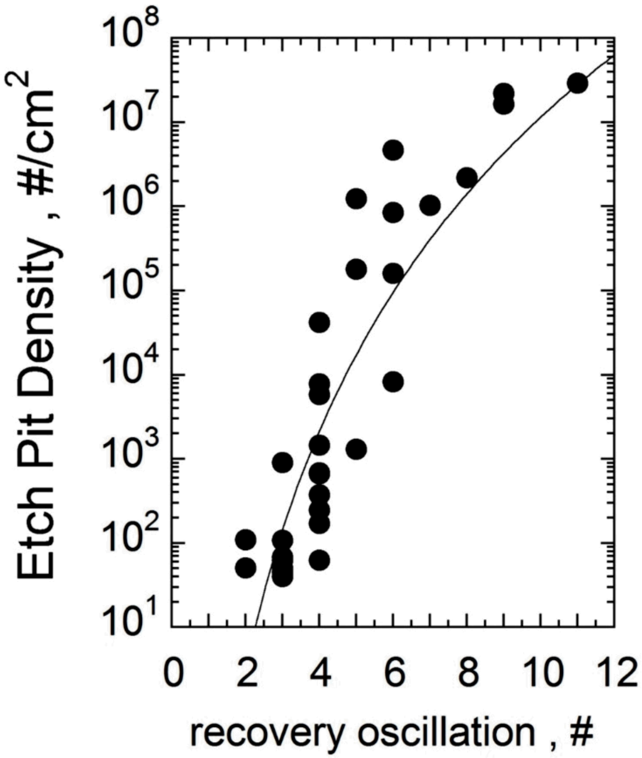
Recovery begins after 4 oscillations



Recovery begins after 9 oscillations



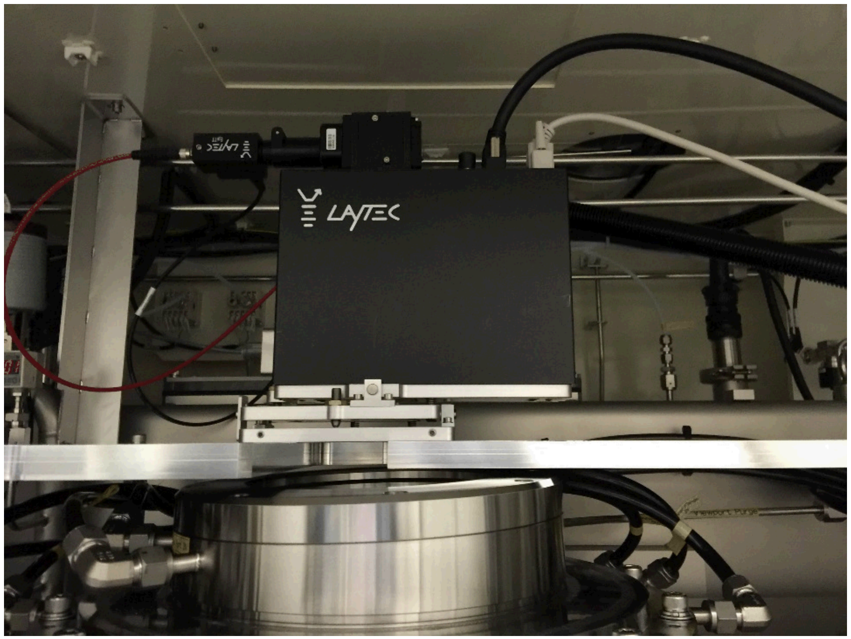
waveform predictive of AlN film quality



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

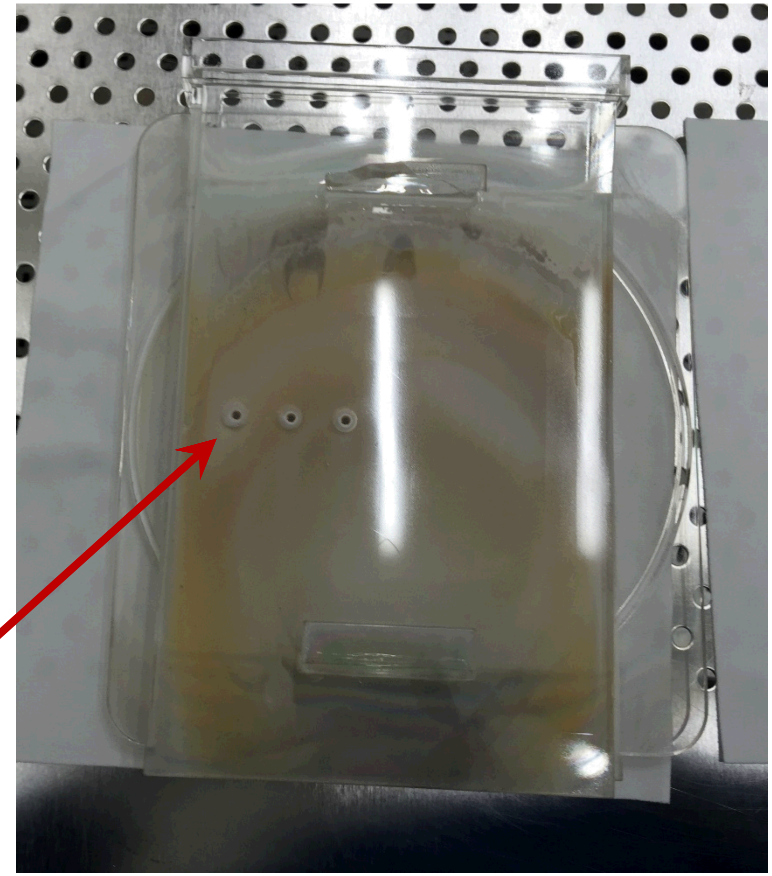
What causes the differences in the reflectance waveforms?

LayTec epiTT and EpiCurve on the TNSC SR4000



Mounted on top access window

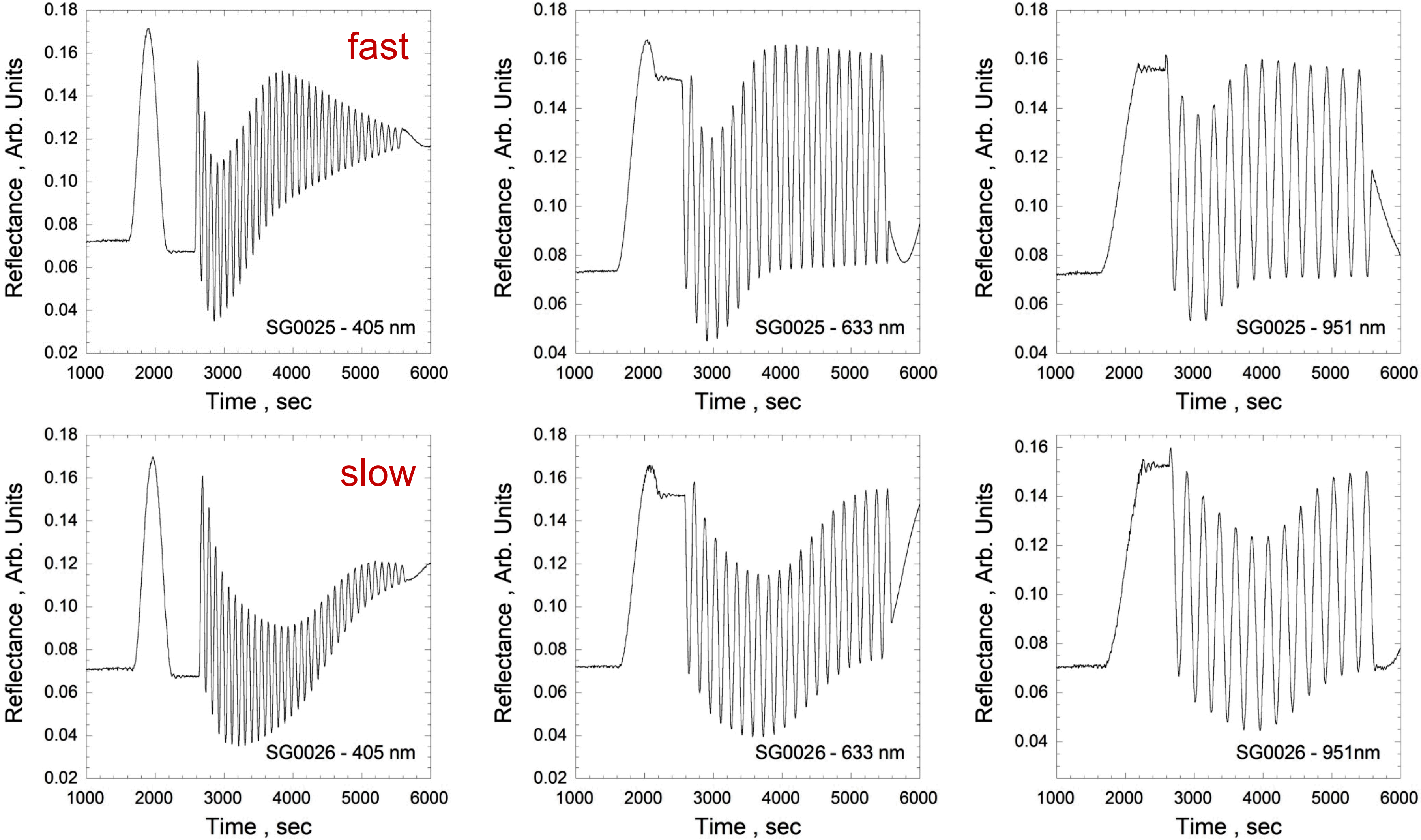
Optical access through holes in the top quartz channel. Coatings do not change the optical access.



Top and bottom quartz-ware channel

Special Thanks to Jaime Beltrán for setting up the Laytec epiTT and EpiCurve

Optical Reflectance at Three Wavelengths



Simulation of optical reflectance

From Breiland and Killeen, JAP 78, 6726 (1995).

From Koleske et al., J. Cryst. Growth 78, 6726 (1995).

Reflectance is calculated using the virtual interface model of Breiland.

The observable real reflectance, $R=|r|^2$, is given by

$$R(t)=\frac{R_\infty-2\sqrt{R_\infty R_i}e^{-\gamma t}\cos(\delta t-\sigma-\varphi)+R_i e^{-2\gamma t}}{1-2\sqrt{R_\infty R_i}e^{-\gamma t}\cos(\delta t-\sigma+\varphi)+R_\infty R_i e^{-2\gamma t}}. \tag{6}$$

The following definitions are used in Eq. (6):

$$R_\infty=|r_\infty|^2=\frac{(1-n)^2+k^2}{(1+n)^2+k^2},$$

$$\varphi=\tan^{-1}\left(\frac{2k}{n^2+k^2-1}\right),$$

$$R_i=|r_i|^2=|\sqrt{R_i}e^{i\sigma}|^2=\frac{(n-n_s)^2+(k-k_s)^2}{(n+n_s)^2+(k+k_s)^2},$$

$$\sigma=\tan^{-1}\left(\frac{2(nk_s-n_s k)}{n^2-n_s^2+k^2-k_s^2}\right), \tag{7}$$

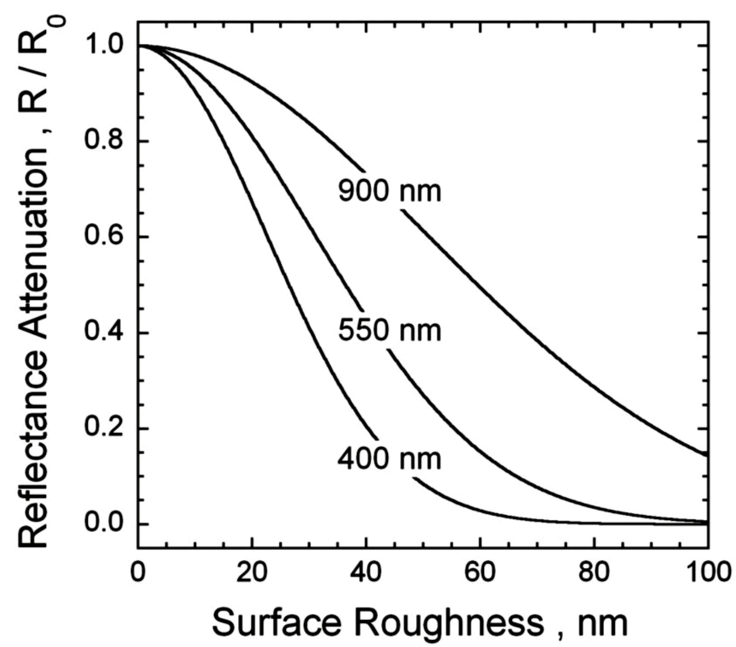
$$\gamma=4\pi kG/\lambda,$$

$$\delta=4\pi nG/\lambda.$$

G = growth rate
λ = wavelength
s = substrate
N = n – ik

Film roughness is calculated using the formula. *ρ(t) is the roughness.*

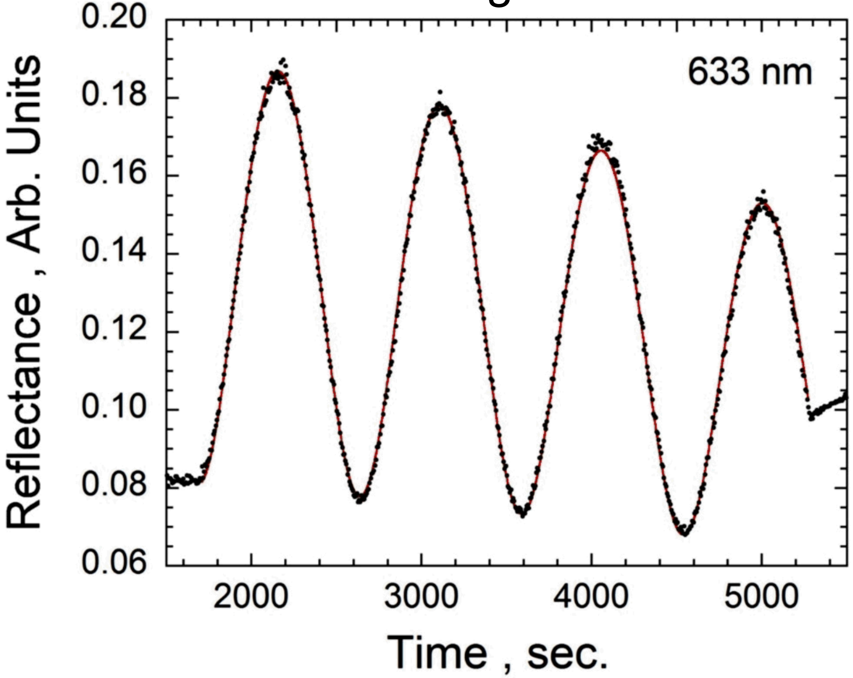
$$R'(t) = R(t)\exp(-[4\pi\rho(t)/\lambda]^2)$$

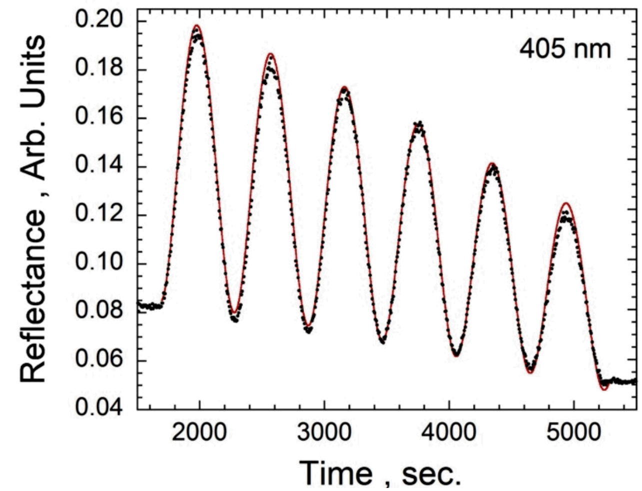


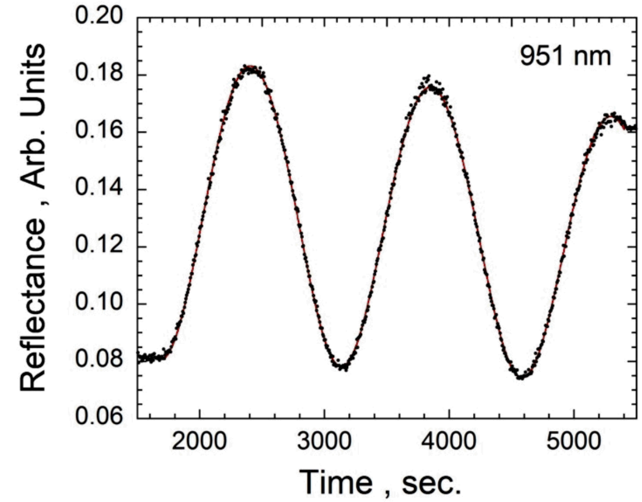
How does the roughness vary vs. time

Thick AlN NL roughens during growth

60 min AlN NL growth at 920 ºC



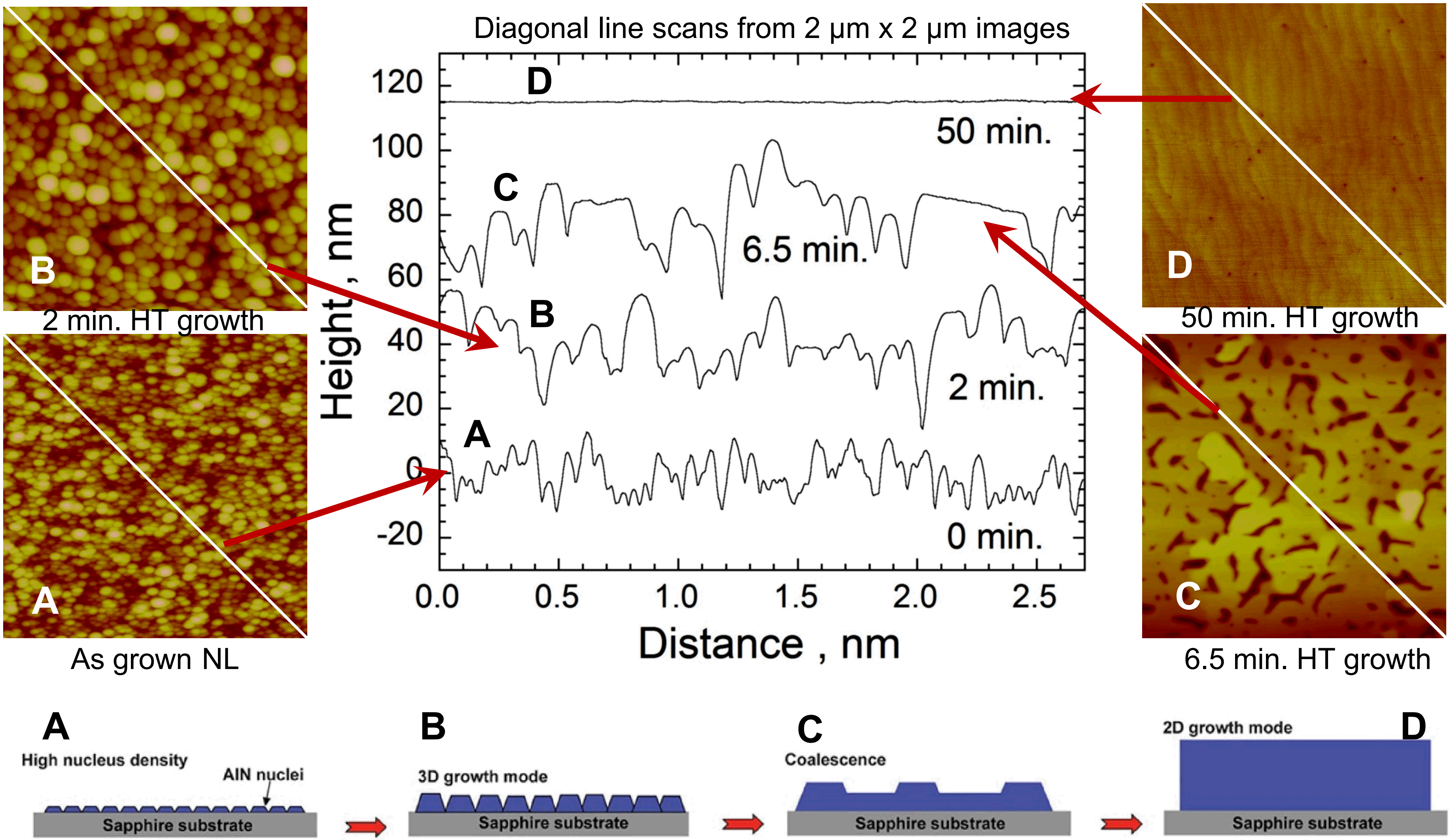




σ_{RMS}	60 min.	9.5 min.
405 nm	17.3 nm	2.75 nm
633 nm	17.9 nm	2.84 nm
951 nm	18.3 nm	2.90 nm

Assume: AlN NL roughness increases linearly with time, no k, no T dependence

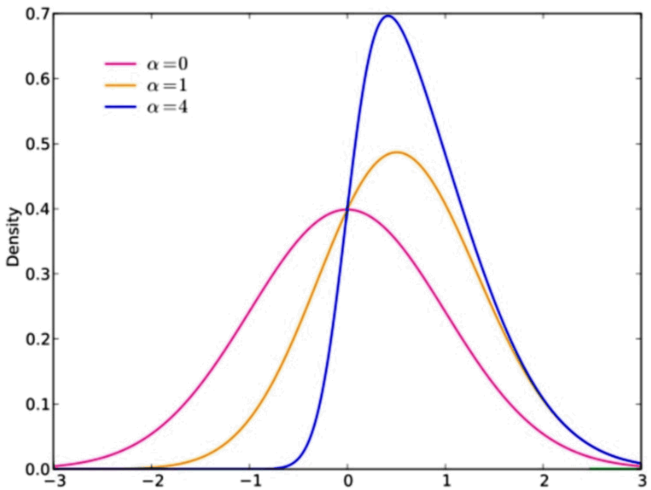
Roughness increases height and width during growth



Images from Sun et al., Cryst. Eng. Comm. 15, 6066 (2013).

Use a Skewed Normal Distribution for Roughness

α = 0 ≡ normal Gaussian



Let $\phi(x)$ denote the standard normal probability density function

$$\phi(x) = \frac{1}{\sqrt{2\pi}} e^{-\frac{x^2}{2}}$$

Gaussian

with the cumulative distribution function given by

$$\Phi(x) = \int_{-\infty}^{x} \phi(t)\, dt = \frac{1}{2}\left[1 + \operatorname{erf}\left(\frac{x}{\sqrt{2}}\right)\right],$$

Area under the curve is conserved

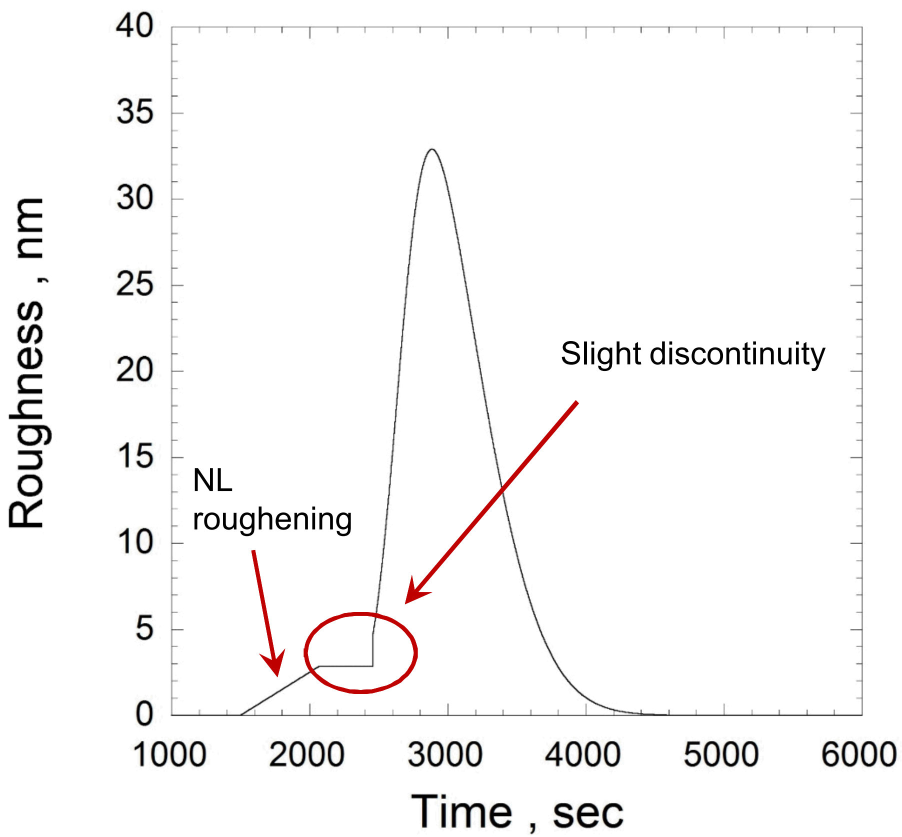
where **erf** is the error function. Then the probability density function (pdf) of the skew-normal distribution with parameter α is given by

$$f(x) = 2\phi(x)\Phi(\alpha x).$$

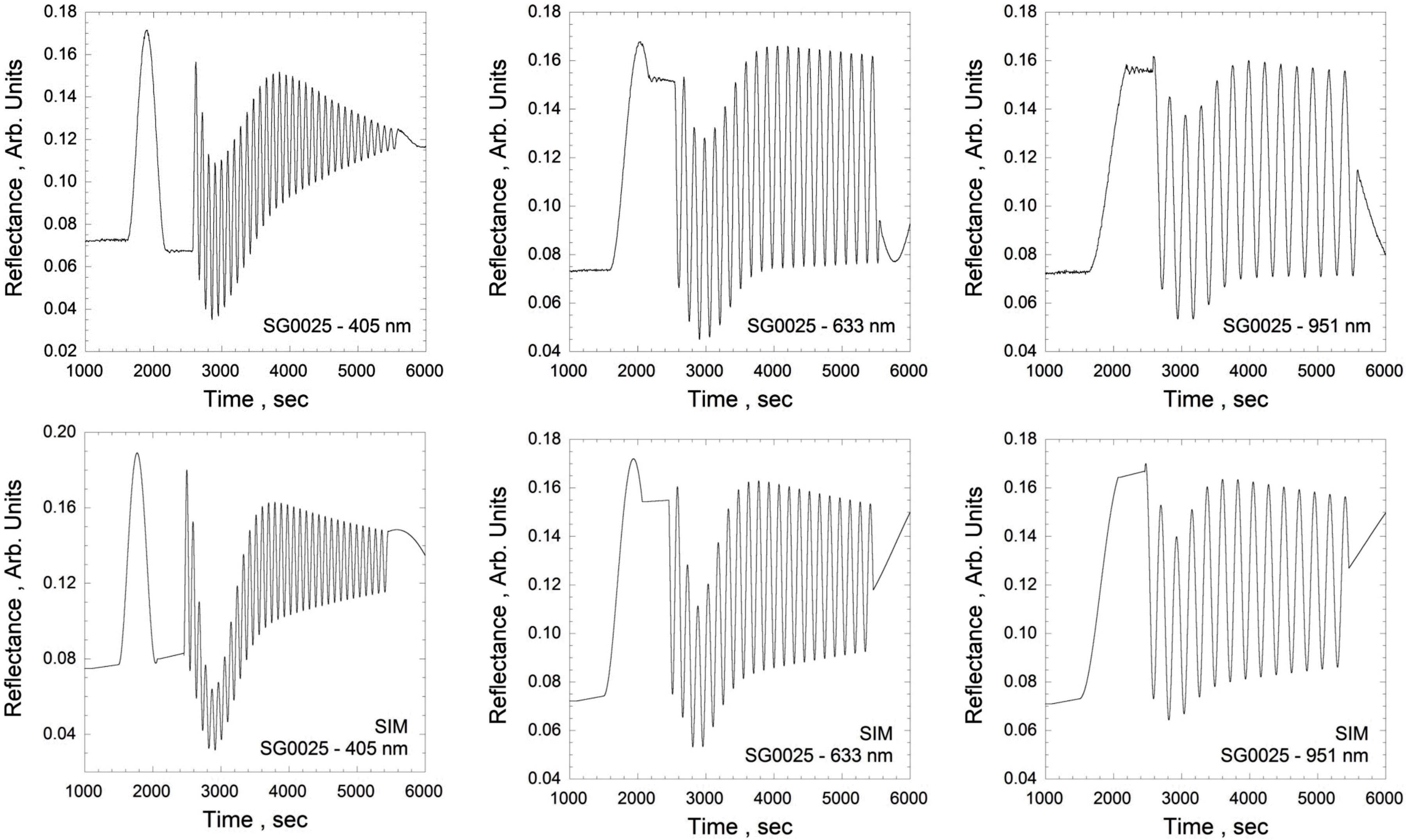
Skew-normal distribution with location, ξ, and scale, ω.

$$f(x) = \frac{2}{\omega}\phi\left(\frac{x-\xi}{\omega}\right)\Phi\left(\alpha\left(\frac{x-\xi}{\omega}\right)\right).$$

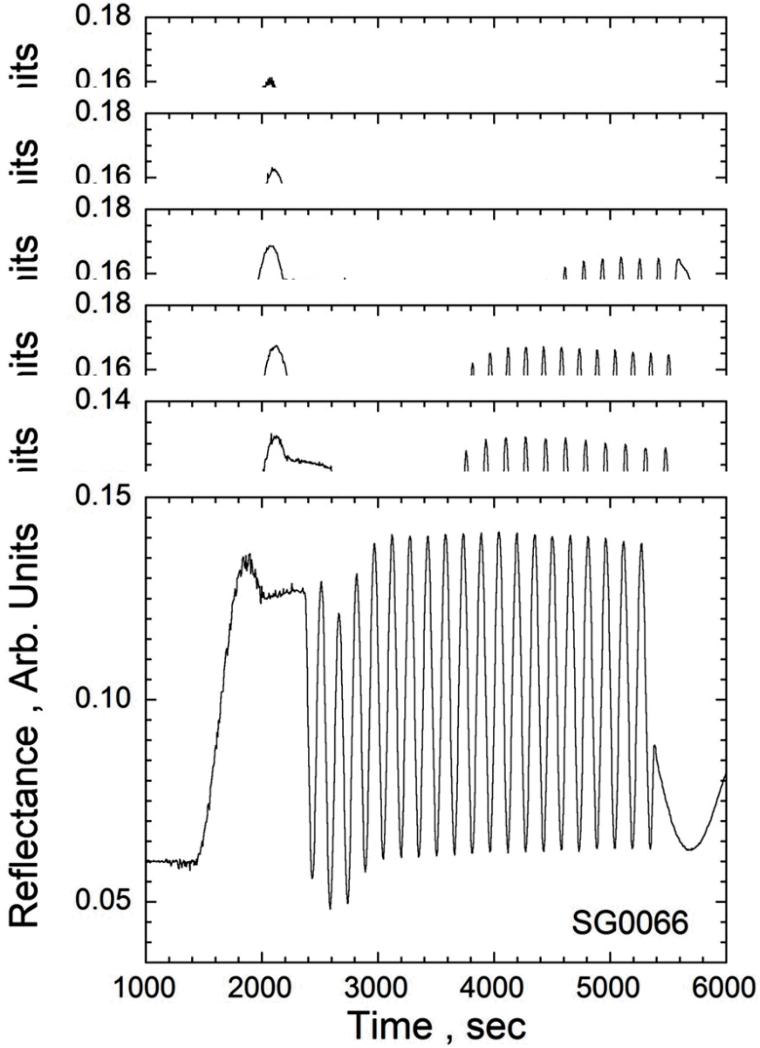
Roughness profile used for the SG0025 reflectance simulations on the next slide



Optical Reflectance at Three Wavelengths



Reflectance Waveforms Predicts Film Quality



run	recovery	XRD (0002)	EPD #/cm^2
SG0034	9	388	2.2×10^7
SG0048	8	324	2.2×10^5
SG0045	7	320	1.0×10^5
SG0039	4	230	7.8×10^3
SG0053	4	237	5.8×10^3
SG0066	3	200	4.0×10^1

Summary

- Good "ease of use" with the LayTec system.
  - Look at it often during growth to determine growth outlook.
- Reflectance waveforms indicative of AlN material quality
  - Delay time correlates to XRD (0002) FWHM and EPD, the shorter- the better.
- Issues with full modeling of the reflectance waveform.
  - High temperature optical constants?
  - Further studies of AlN morphology evolution are required.
    - Stopped growth runs with AFM measurements.
  - What is functional form of the roughness.
    - One or two dimensional, use power spectral density, etc.?
  - How might wafer curvature might influence waveform.

Thanks and Questions?

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MOVPE Growth at Sandia in the Taiyo Nippon Sanso SR4000 MOCVD System.

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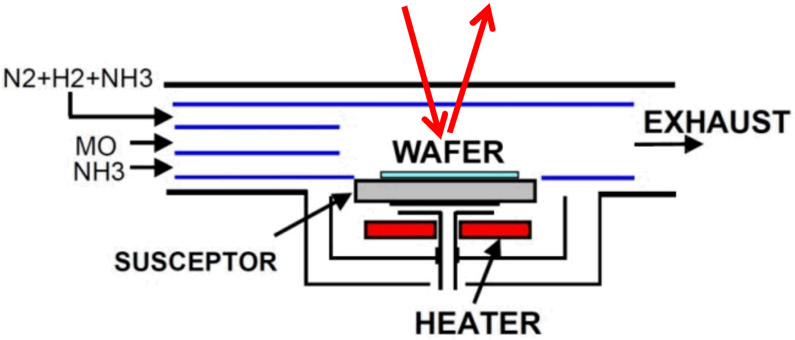
Delivery and Install at Sandia National Laboratories

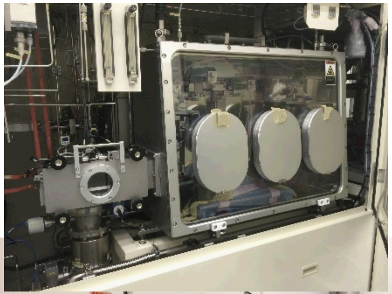
Accepted December 11, 2015

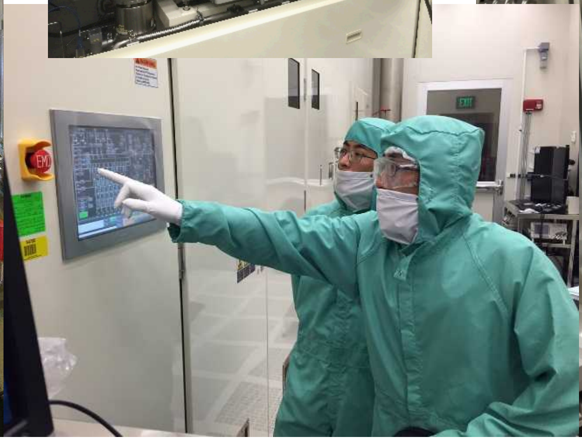
Atmospheric and reduced pressure growth
High Temperature, up to T = 1350 °C

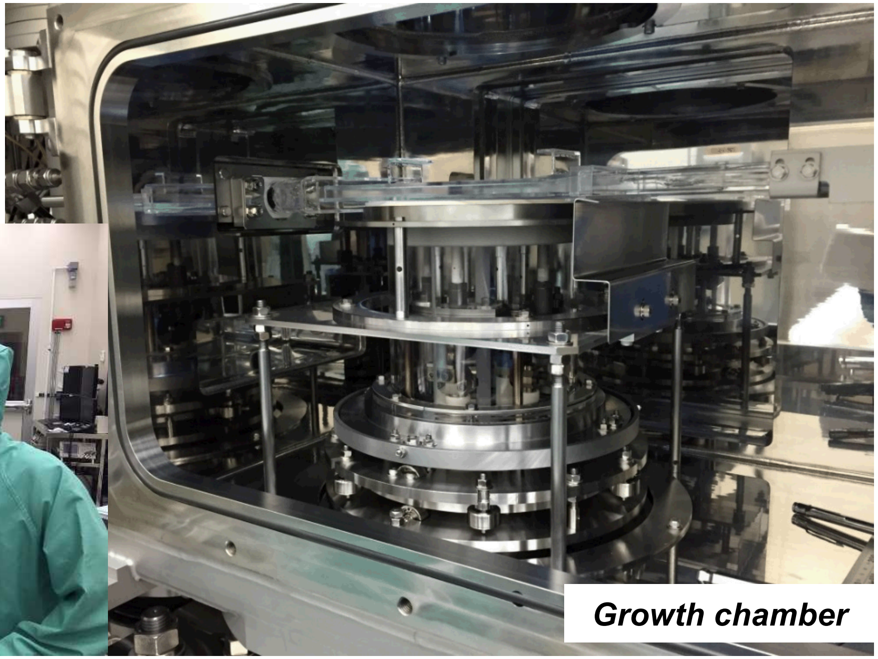
Acceptance Criteria included GaN, InGaN, ar AlGaN thickness and composition uniformity and doping both background and intentional.

Equipped with a Growth monitoring LayTec epiTT and EpiCurve systems









Growth chamber

High Temperature AlN on sapphire

Testing the Reproducibility of AlN growth on sapphire

1320 °C AlN 2.7 μm
920 °C AlN 100 nm
Sapphire

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.

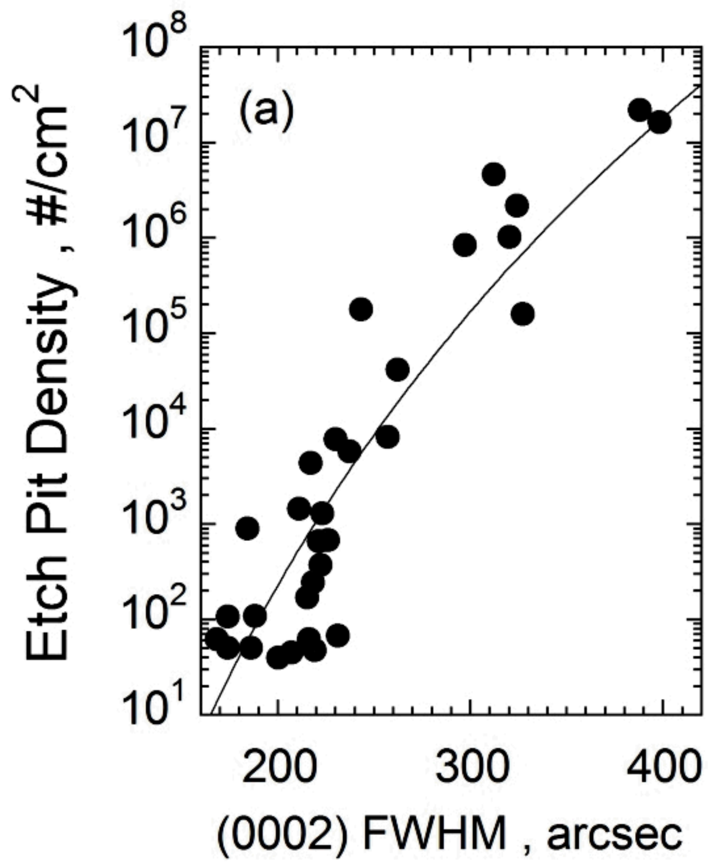
4). Grow AlN NL, 0.3 SLM NH_3.

3). Dose 1 SLM NH_3 for 7 min.

2). Increase pressure to 40 kPa.

1). Heat sapphire to 920 ºC.

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



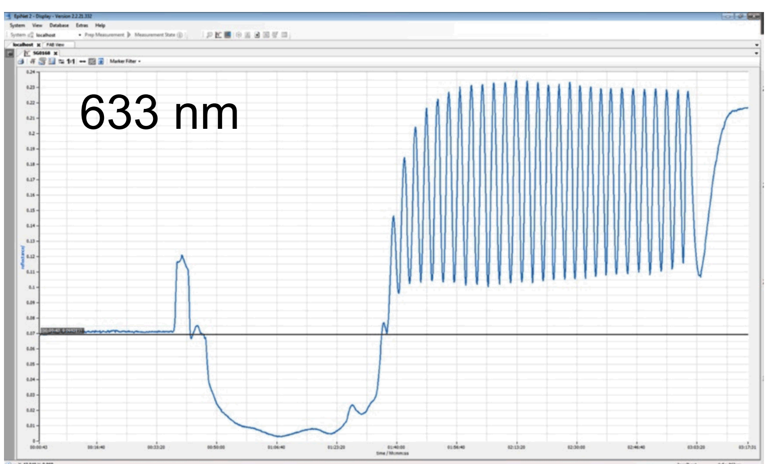
Lower (0002) FWHM – lower EPD

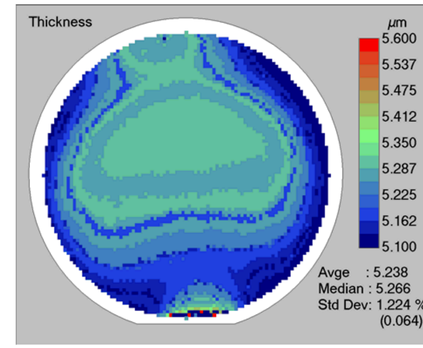
Achieved AlN on sapphire with EPD < 100 cm^{-2}

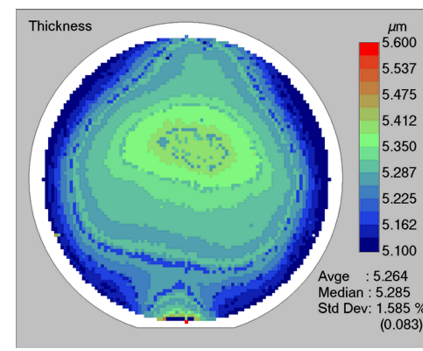
Delayed Recovery GaN on Sapphire

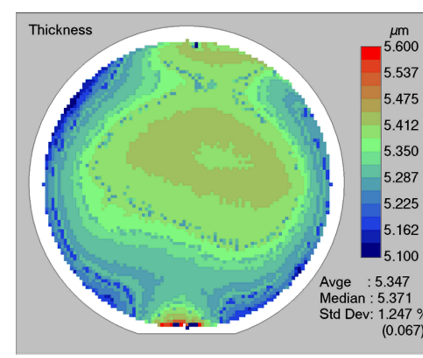
- Qualification GaN growth recipe, modified slightly.
- Thickness uniformity of 1.5 % for ~ 5 µm thick GaN at a growth rate of 2.7 µm/hour.
- XRD linewidths suggest dislocation densities
  - (0004) = 139 arcsec, Screw component ≤ 2×10^{8} cm^{-2}.
  - (10-11) = 247 arcsec, Edge component ≤ 4×10^{8} cm^{-2}.

  following Lee, *et al*. APL 86, 241904 (2005).
- Optical reflectance shows classic delayed recovery.









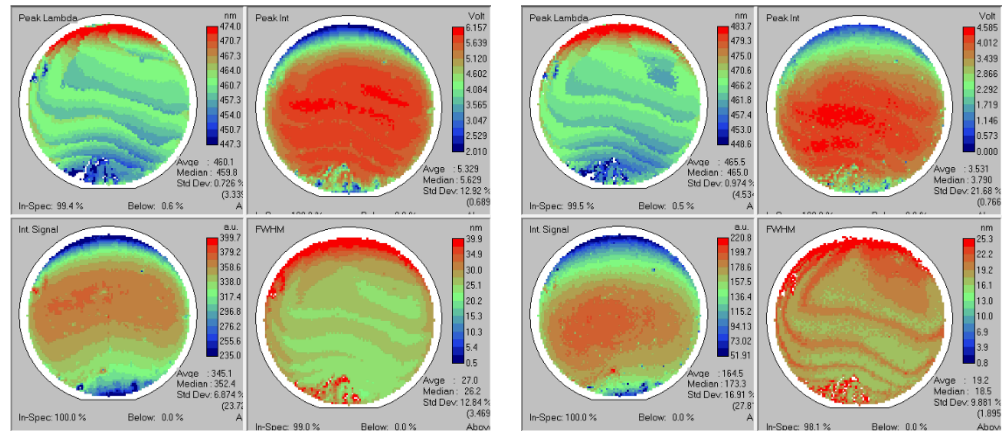
Blue wavelength MQWs on GaN on sapphire

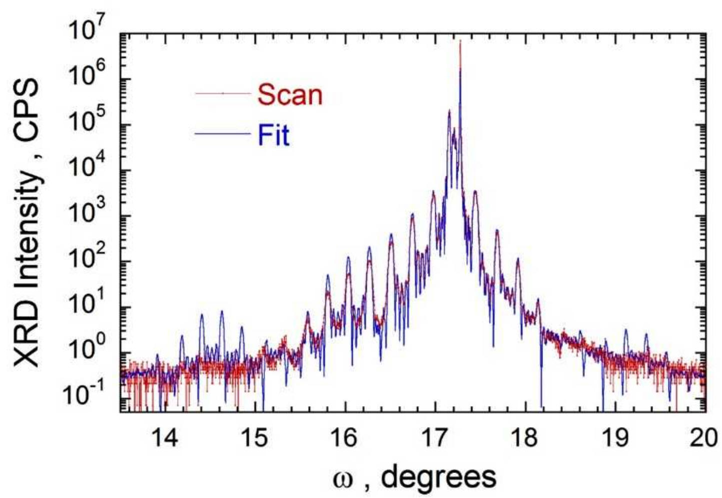
- Changed MQW growth recipe based on qualification runs.
- Used 5 InGaN/GaN MQWs.
- Added InGaN underlayer, 190 nm thick with 4% indium.
- Varied GaN barrier and UL growth temperature.
- To achieve these results ~ 12 growth runs.

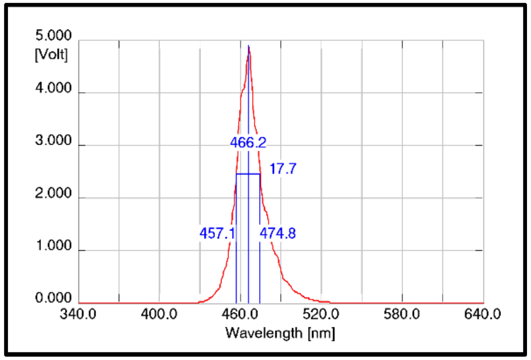
X_UL = 0.0442
h_UL = 190 nm

x_QW = 0.155
h_QW = 2.9 nm
h_B = 16.7 nm

266 nm pulsed laser *325 nm cw HeCd laser*







325 nm: λ = 465.5 ± 1.0 nm
266 nm: λ = 460.1 ± 0.7 nm

Estimate IQE ~ 70 %