## GaN Crystal Growth & TEM Sample Preparation

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Special Thanks to: Ben Haskell & Patrick Waltereit

#### Project Focus

- First 3 weeks:
  - TEM Manual Sample Preparation
- Last 3 weeks:
  - Automated Sample Preparation (FIB) &
  - GaN Crystal Growth

#### Gallium Nitride

Blue Light Emitting Diodes (LEDs)
& Lasers
– Wide Band Gap (3.4 ev)



- Transistors
  - Electrical Properties
  - Stability



#### 3 Methods to Grow GaN

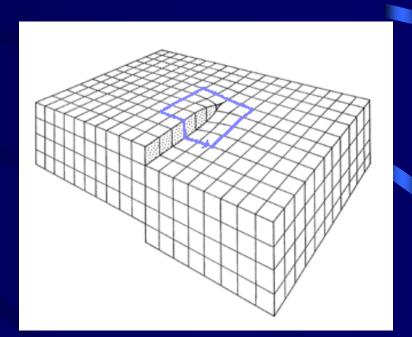
- HVPE
  - Hydride Vapor Phase Epitaxy
- MOCVD
  - Metal Organic Chemical Vapor Deposition
- MBE
  - Molecular Beam Epitaxy

## Comparison of GaN Crystal Growth Methods

|                 | HVPE    | MOCVD   | MBE    |
|-----------------|---------|---------|--------|
| Temp            | highest | medium  | lowest |
|                 | 1050 °C | 1000 °C | 700 °C |
| Growth<br>Rate  | highest | medium  | lowest |
| Film<br>Quality | lowest  | highest |        |

### Crystal Defect Structures

- Caused by slight incompatibility between GaN
   & Substrate
  - Lattice Structure
  - Thermal Properties
  - Other
- Examples:
  - Point Defect 0-D
  - Dislocation 1-D
  - Stacking Fault 2-D



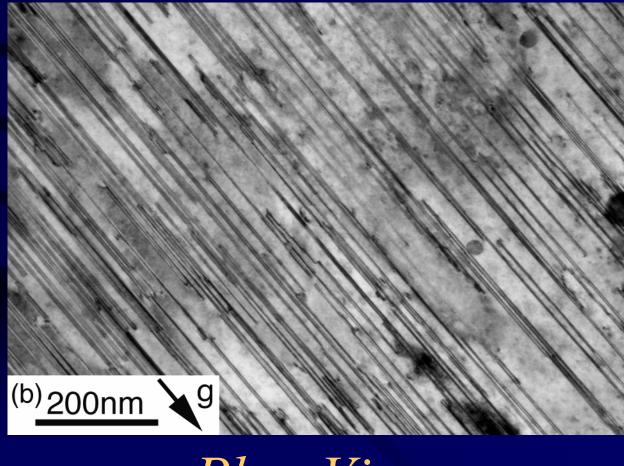
http://www.tf.uni-kiel.de/matwis/amat/def\_en/kap\_5/backbone/r5\_2\_2.html

## Structural Characterization Tools

- X-Ray Diffraction
- Atomic Force Microscope (AFM)
- Optical Microscope
- Scanning Electron Microscope (SEM)
- Transmission
   Electron
   Microscope
   (TEM)

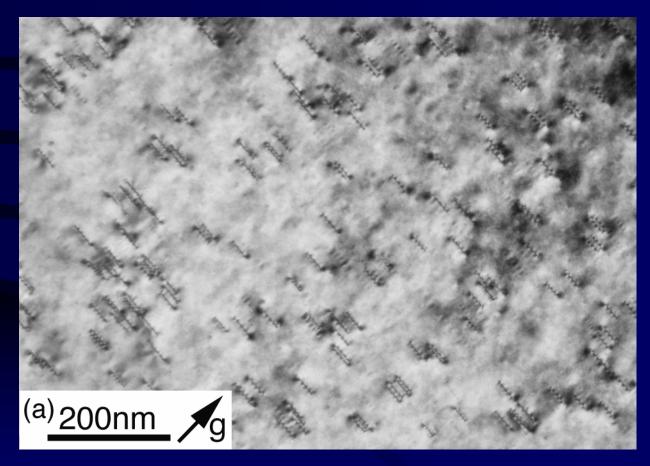


## TEM Image Stacking Fault



Plan View

## TEM image Dislocation



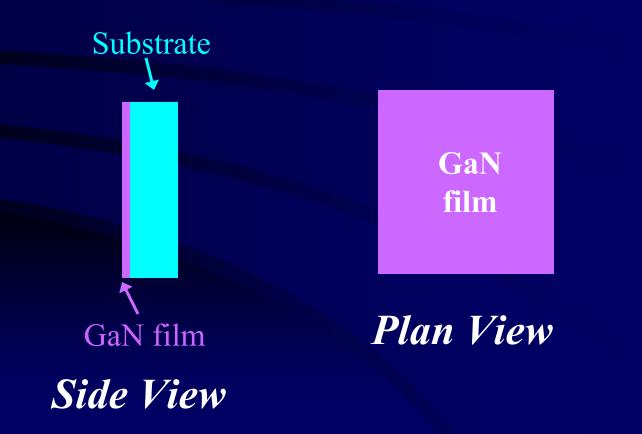
Plan View

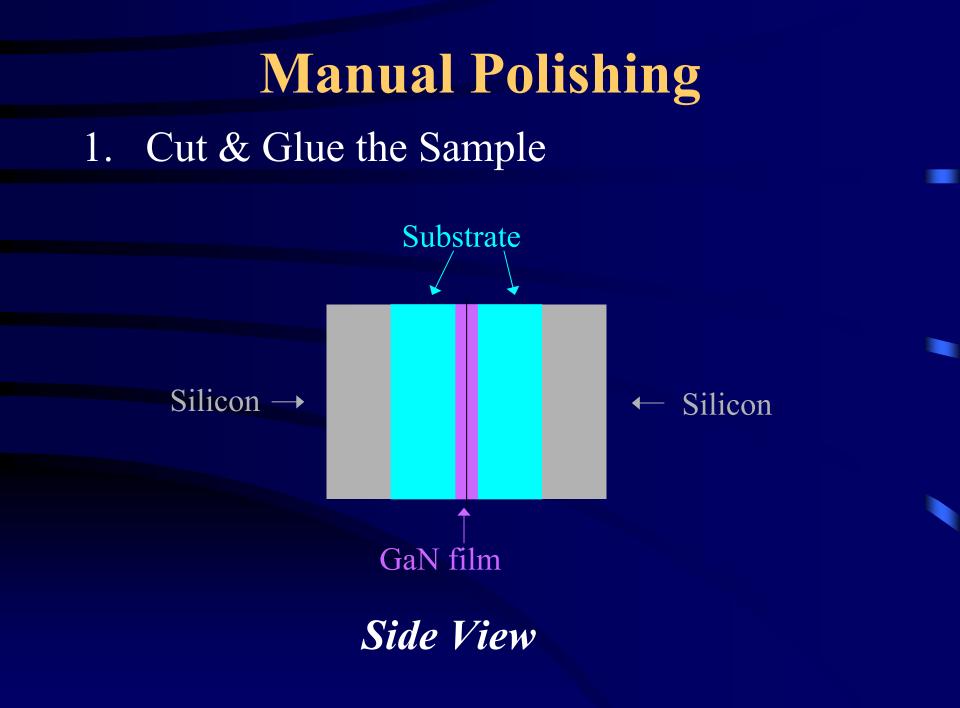
#### **TEM Sample Preparation**

- Manual Polishing
  - -1 day 2 weeks
- Automated Polishing
  - -2-4 hours
  - Focused Ion Beam (FIB)

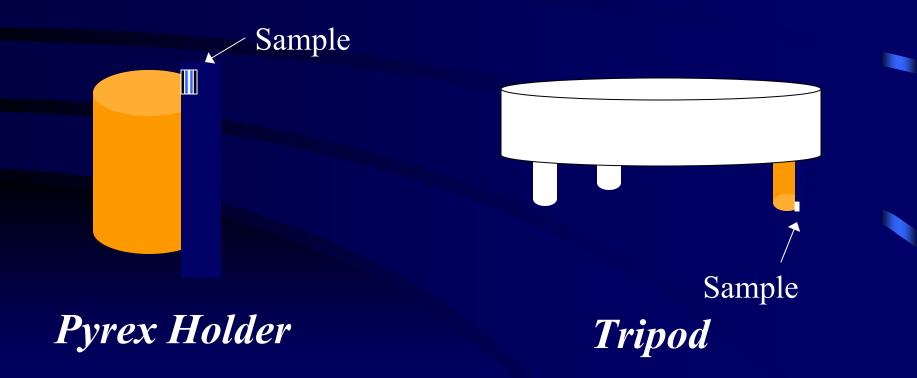
#### **TEM Sample**

- Thin film (GaN)
- Substrate (Sapphire or Silicon Carbide)





Manual Polishing
 Attach sample to Pyrex holder
 Insert Pyrex holder into Tripod



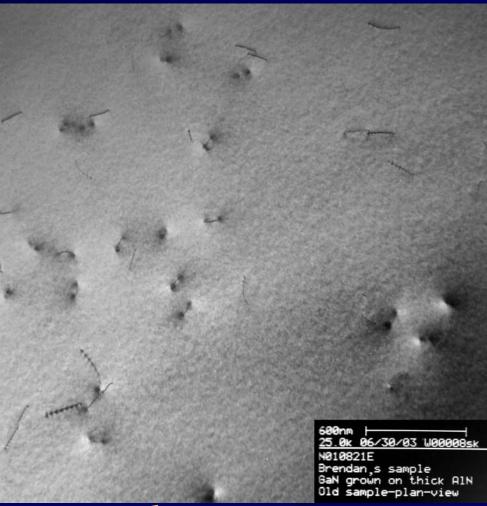
## **Manual Polishing**

- Polish 1<sup>st</sup> side of Sample on Diamond Lapping Film until smooth
- 5. Glue Sample onto Copper Grid
- Polish 2<sup>nd</sup> side of Sample until 10 μm thick
- 7. Ion Milling until 100 nm thick



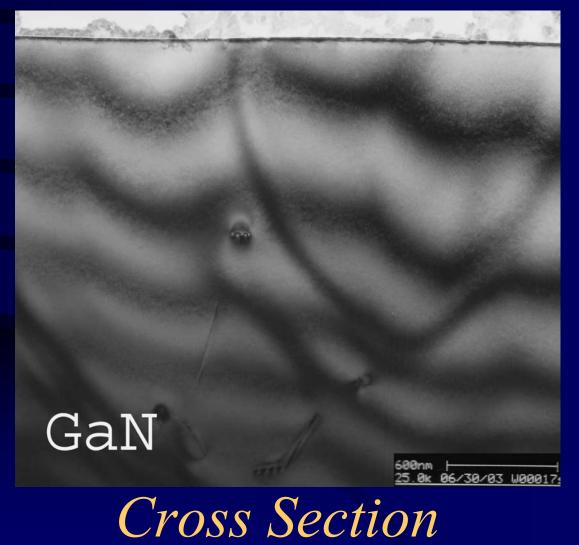


## TEM image Dislocation



Plan View

# TEM image Defects?



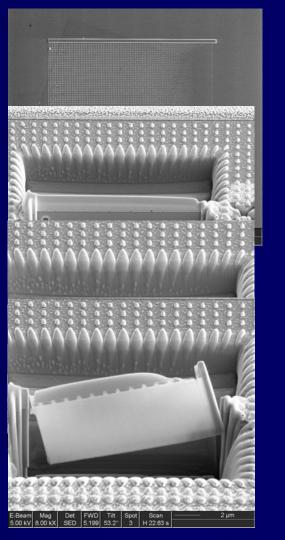
## FIB Focused Ion Beam

- No Sample
   Preparation
- 2 Beams
  - Electron
  - Ion

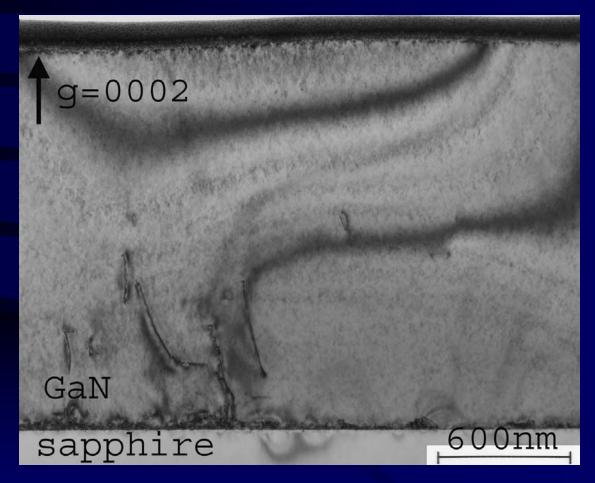


#### **FIB** Procedures

Locate Site of Interest
 Mill a trench on either side
 Thin the wall
 Cut it out



## TEM image Dislocation



#### Cross Section

#### Reflections...

- Research Lessons:
  - TEM Sample Preparation
    - Time-Consuming
    - Delicate
    - Easy to Make Mistakes
  - TEM images give most detailed info
  - Other forms of Microscopy used as a gage
- Classroom Applications:
  - Interviewing experts key
  - Learning about other components made it more interesting/meaningful