HOW DO YOU MAKE CONTINUOUS IMPROVEMENT PROFITABLE?

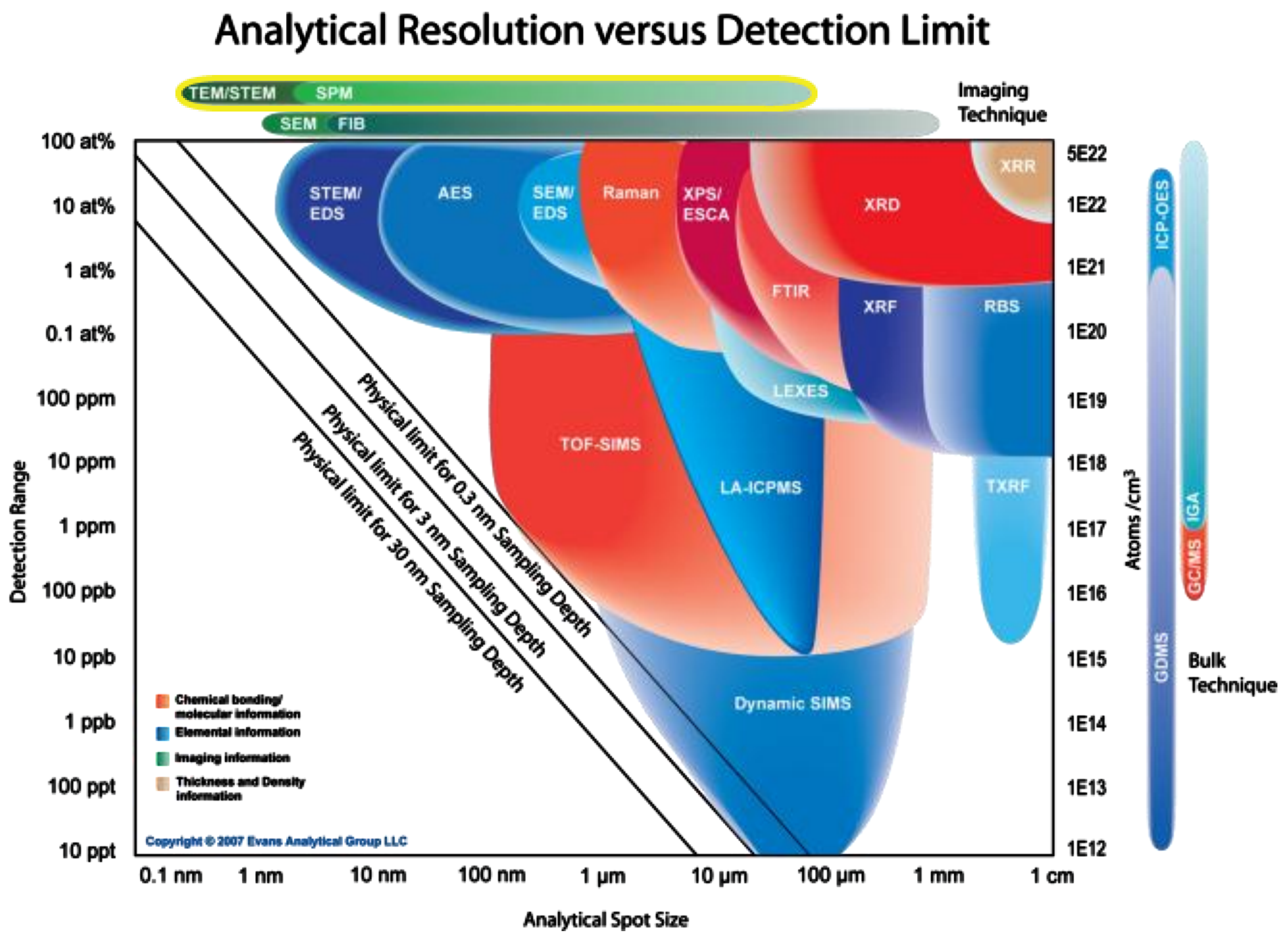
透射电子显微镜(TEM)和扫描透射电子显微镜(STEM)

统称为S/TEM

禹重科技® ÜZONGLAB
成分分析仪器|表面测试仪器|样品前处理仪器

EAG LABORATORIES
WE KNOW HOW™

TEM/STEM



S/TEM能提供最高的图象分辨率和元素识别

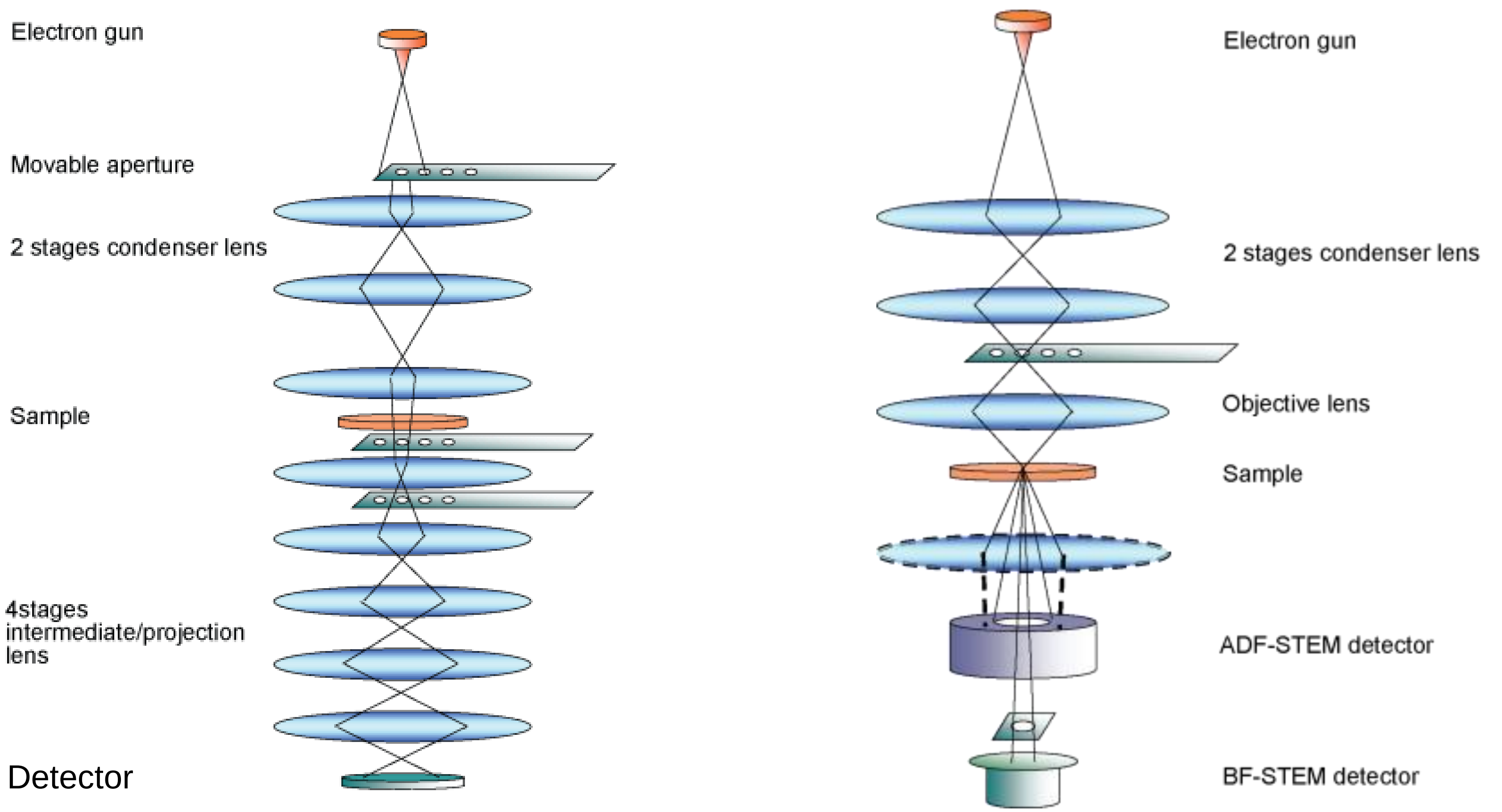
EAG LABORATORIES

(S)TEM 理论背景

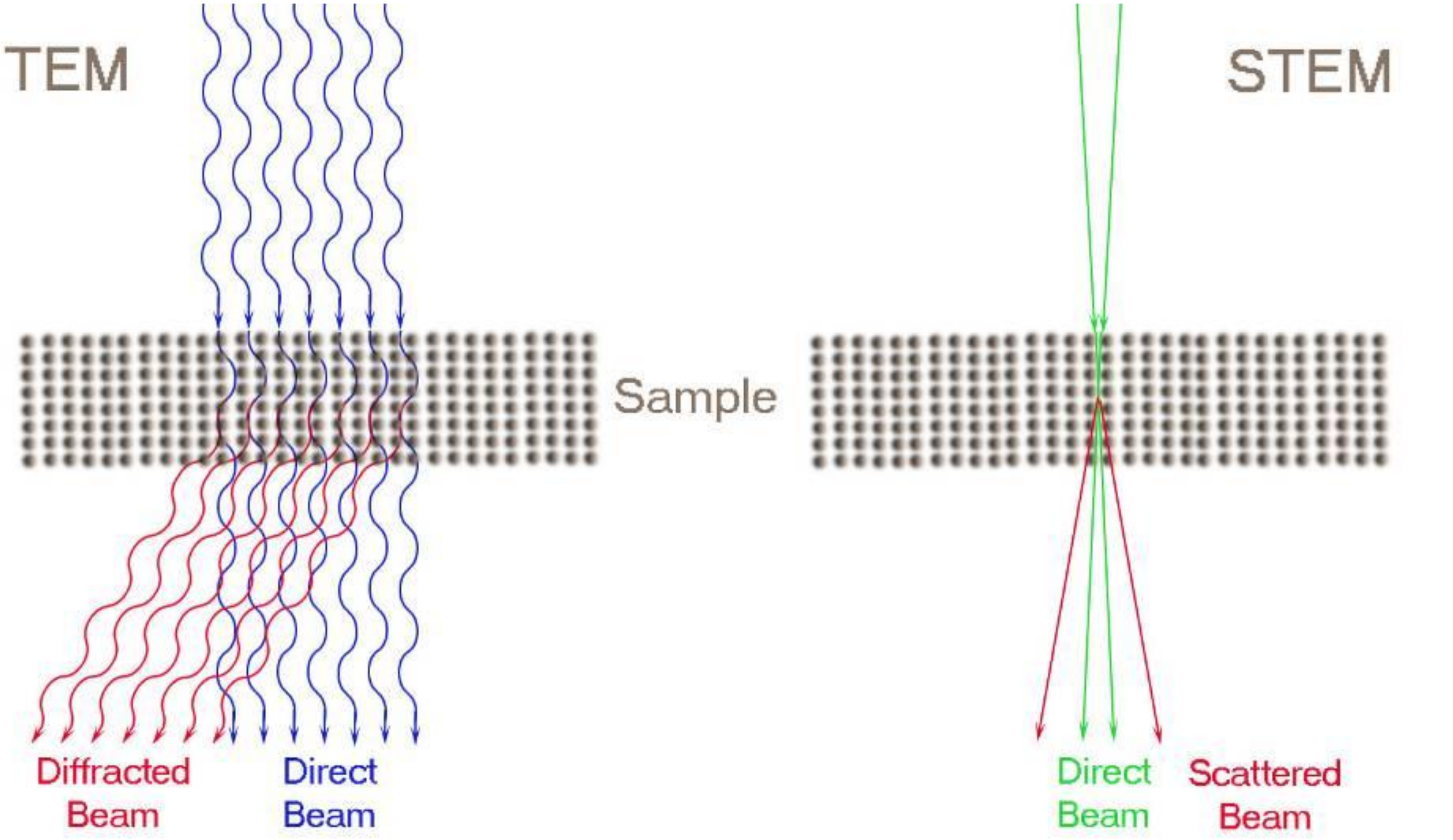
TEM 和 STEM 结构图

TEM

STEM



TEM 和 STEM 比较图



理论背景

- TEM或STEM成像薄样品都用30-300 KeV的电子束.
- 电子束穿透样品以透射模式成像，样品厚度一般是50～100nm。
- S/TEM’s 可以和EDS or EELS (electron energy loss spectroscopy)耦合， 探测器提供元素或化学态信息.

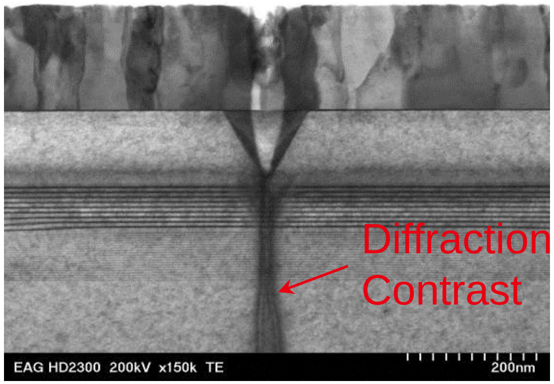
禹重科技® UZONGLAB

理论背景

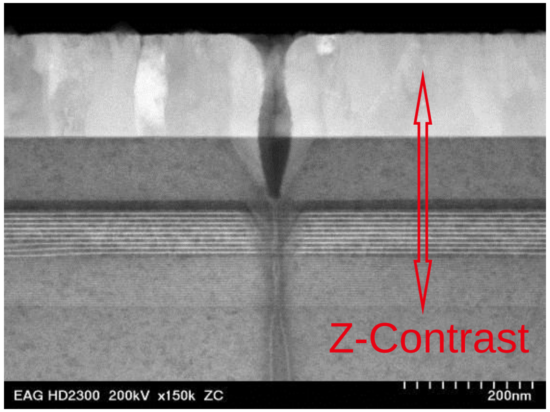
- TEM成像利用不同的衬度机制可以在不同的模式下拍照：
  - 明场像(非散射电子）
  - 暗场像 (晶体形貌衬度）
  - 高分辨率 (衍射电子和非衍射电子的干扰).
  - 电子衍射.
  - 分析模式: EDS or EELS.
- STEM数据也可以采用不同的模式成像 :
  - 明场像(非散射电子)
  - 暗场像(散射电子: High Angle Annular Dark Field; HAADF, Z-contrast, ZC)
  - 二次电子
  - 分析模式: EDS or EELS

(S)TEM DATA TYPES

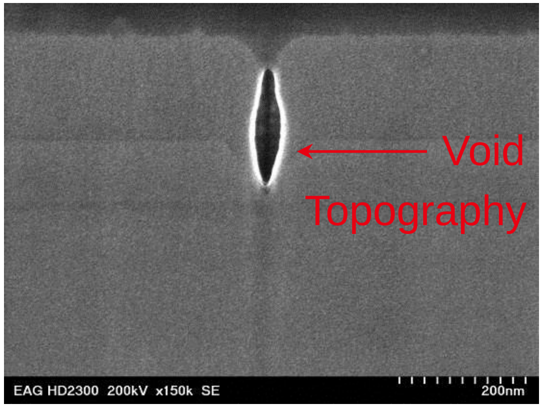
STEM BF Image



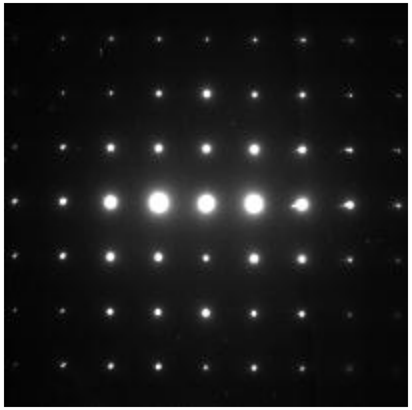
STEM Dark Field/Z-contrast



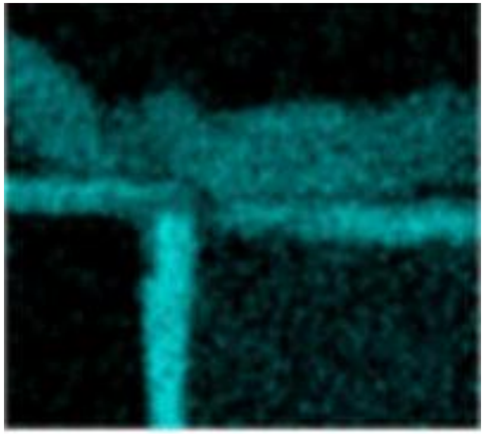
STEM SE Image



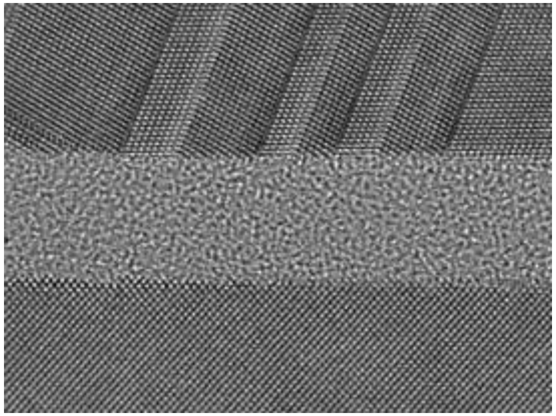
Electron Diffraction



Elemental Maps



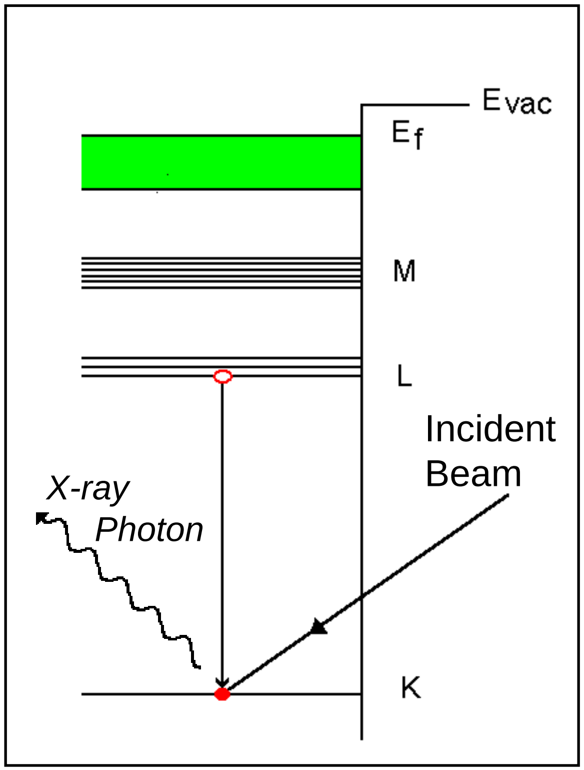
HR-TEM



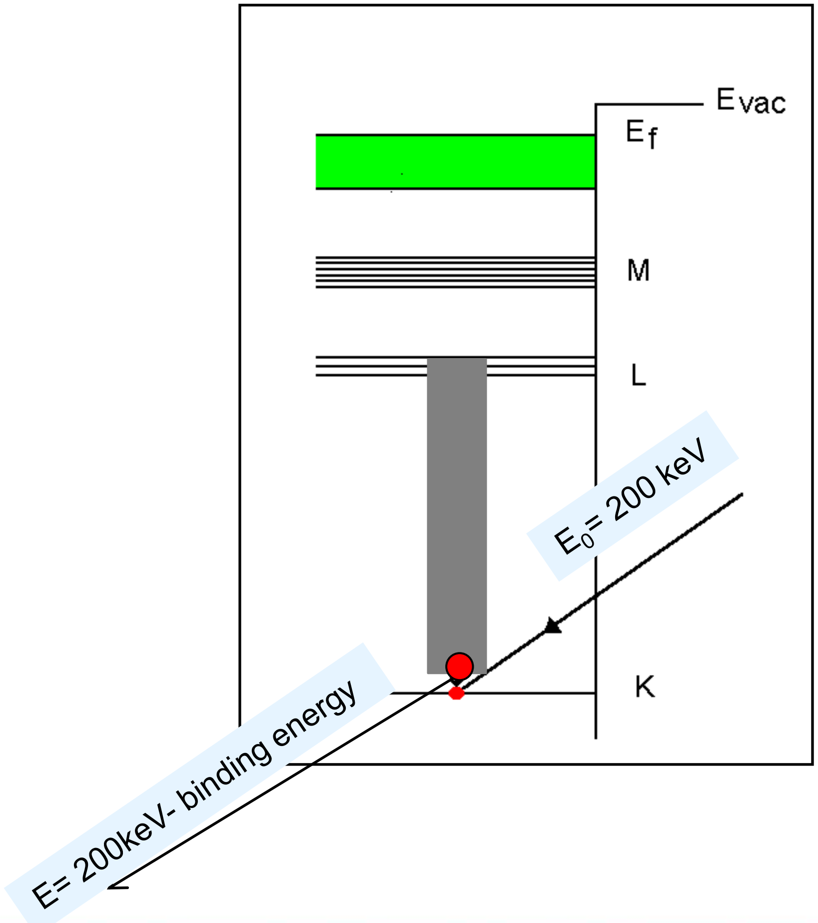
Some images and figures courtesy of Hitachi

理论背景- ELEMENTAL ID

X-ray emission -EDS

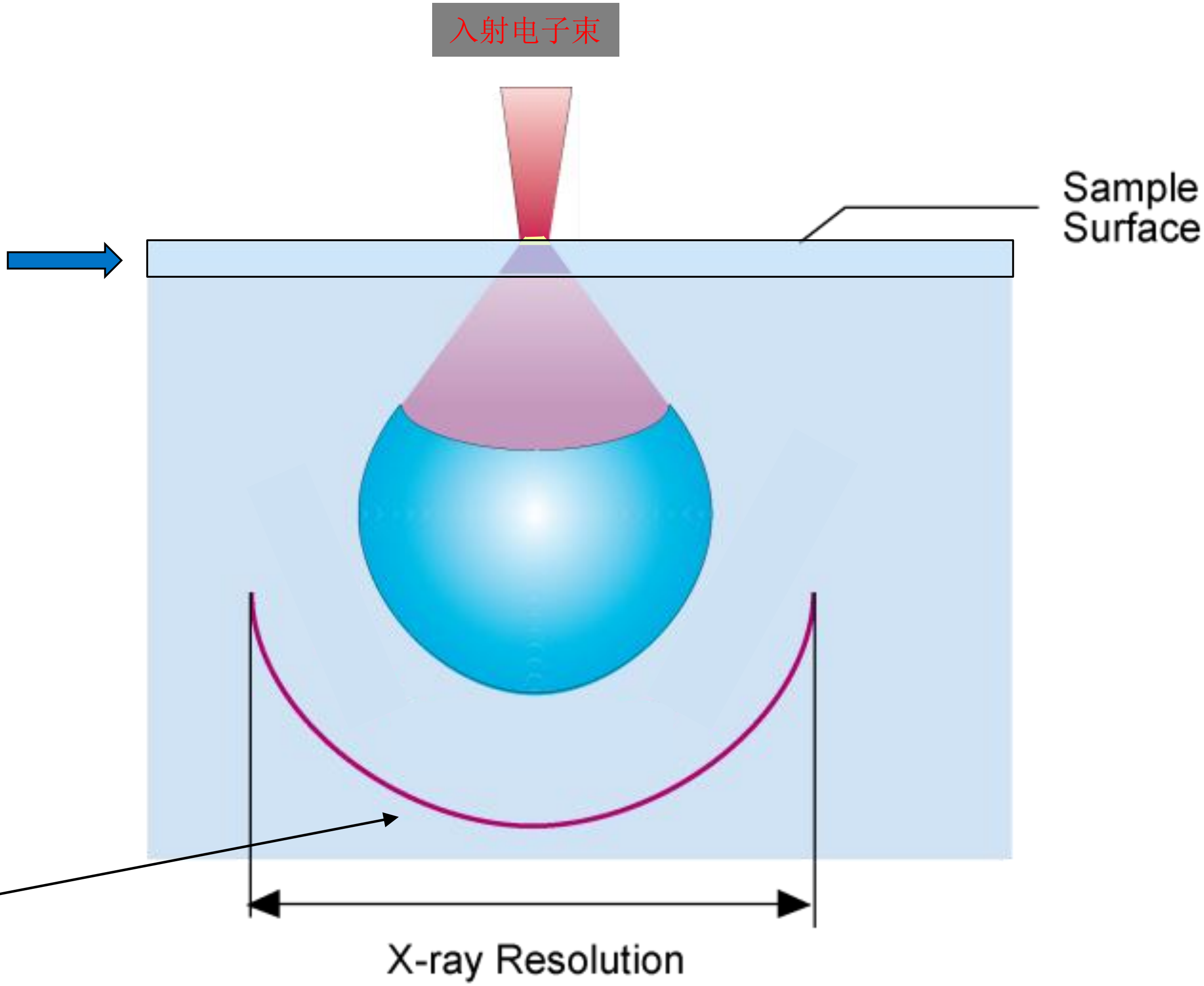


Electron Energy Loss Spectroscopy-EELS



理论背景 - EDS

TEM样品厚度使得EDS分辨率可到 ~3 nm



Source of secondary fluorescence

0.5-5 um fpr bulk sample

ELECTRON ENERGY LOSS SPECTROSCOPY

在电子能量损失谱中，样品在TEM电子束照射下，电子经历非弹性散射，会损失能量。

非弹性散射包括声子激发，带内或带间散射，等离子体激发，内壳层电子电离.

内壳层电子电离对探测材料的元素成份非常有用。

- 比EDS收集更多信号
- 1 nm probe size
- 高能量分辨率(<1 eV)
- 对原子序数低的元素灵敏度高

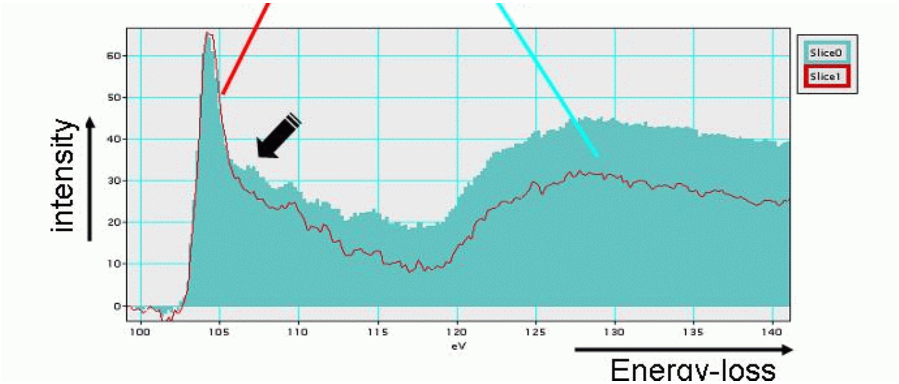


Image source:
http://pruffle.mit.edu/~ccarter/NANOAM/images/van_benthem_EELS.gif

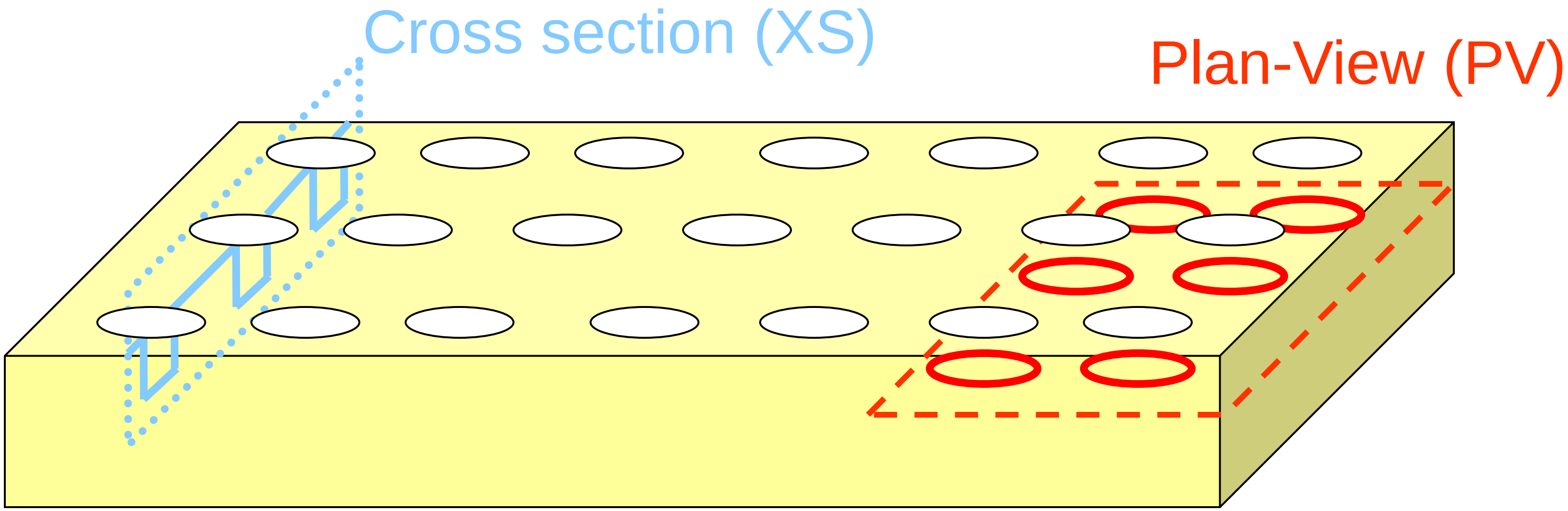
怎么制备100NM厚的样品?

- 要很小心…
  – 对于100nm厚的样品，每边的损伤层不能大于20nm，有些样品要小于10nm。
  – 样品不同，制备技术也不同:
    - Hard or soft samples
    - Hard and soft layered/composite samples
    - Heat sensitive samples
    - Small features
    - How much time and $$ are available?
      – Duel beam FIB’s cost >$1M
    - Biologicals
    - Solvent sensitive samples

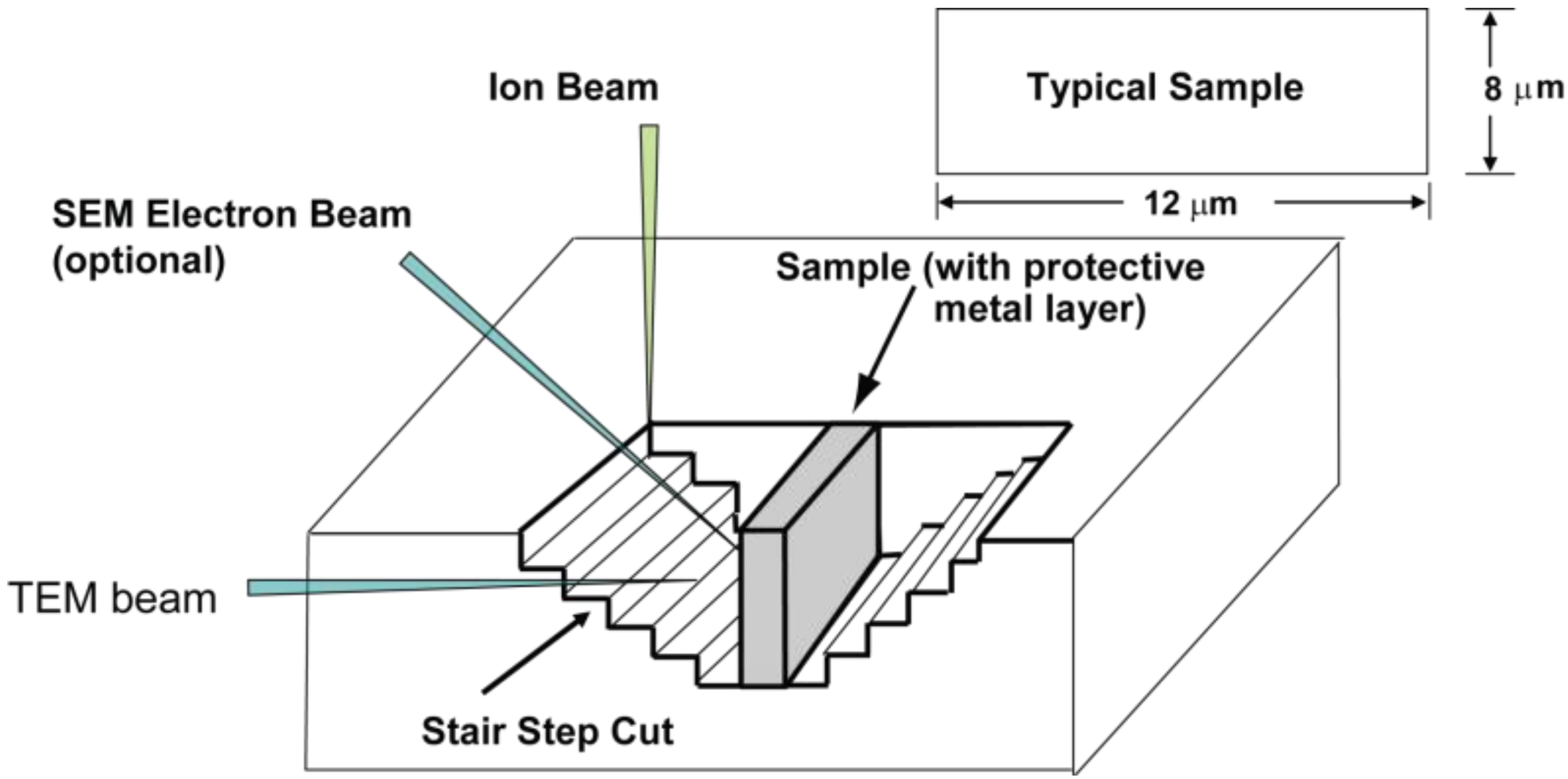
TEM 样品制备方法

- 会聚离子束
- Wedge Polishing+ Ion Milling
  - 离子束最终减薄
  - 对于半导体材料好用，可测量区域要大.
  - 样品制备质量要求高
- 直接成像
  - 仅用于很薄的样品，比如纳米颗粒
- 机械钉薄
- 电解抛光和化学抛光
  - 需要特殊的侵蚀液配方
- 超薄切片
  - 不能用于脆性材料-经常用于生物材料和聚合物材料.
- Crushing
- 脆性或陶瓷材料

样品制备: 样品取向

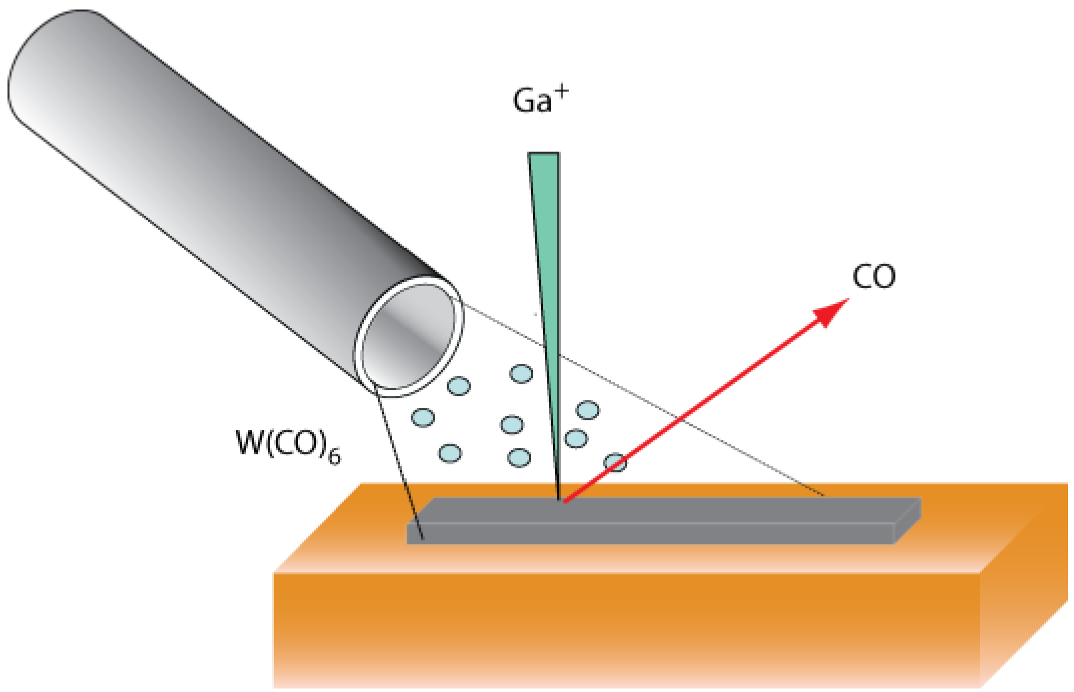


S/TEM 样品制备



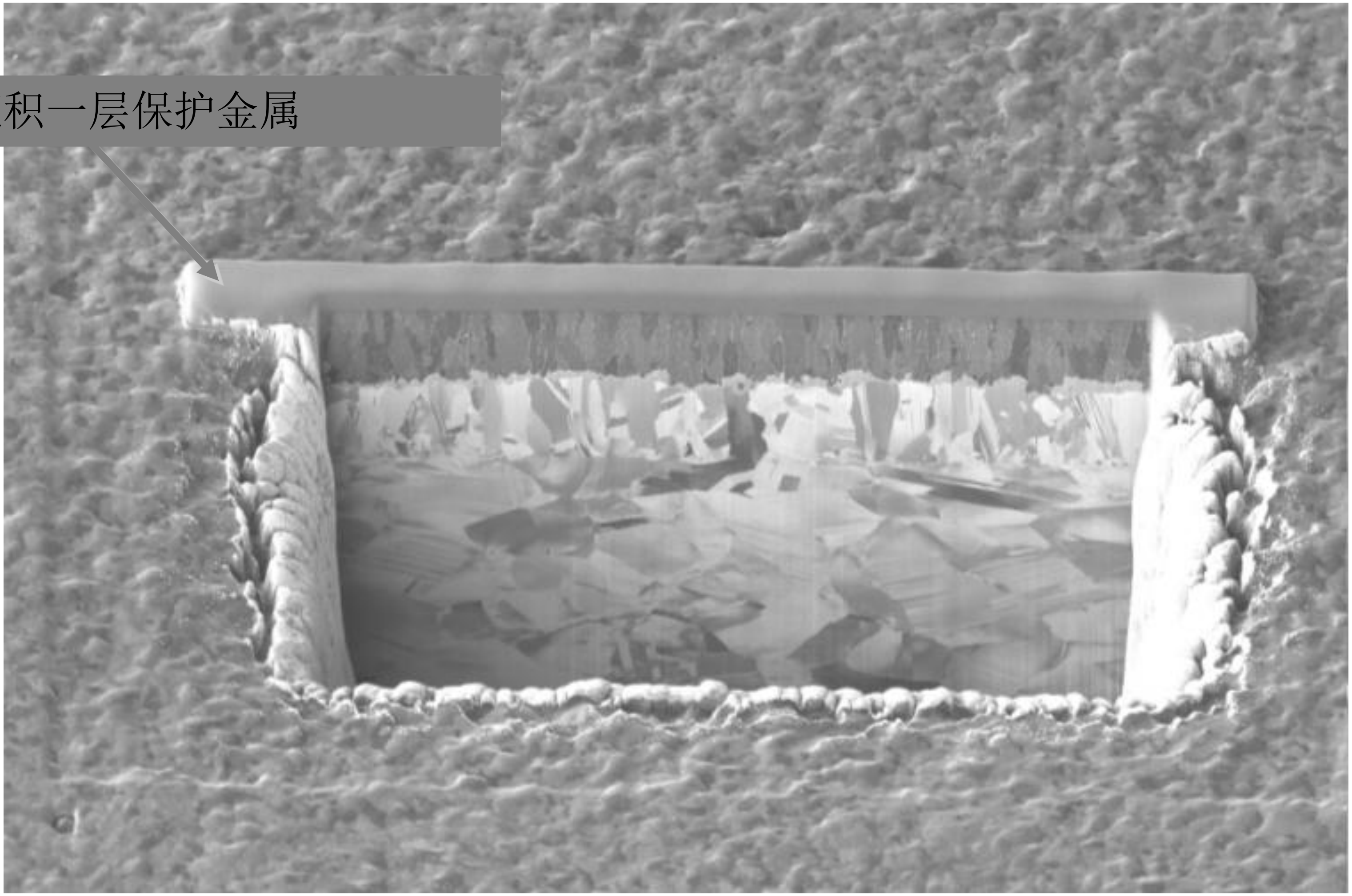
称作“lift-out”；最终的样品要从挖的沟里拿出来，沾到TEM 铜网上。

沉积

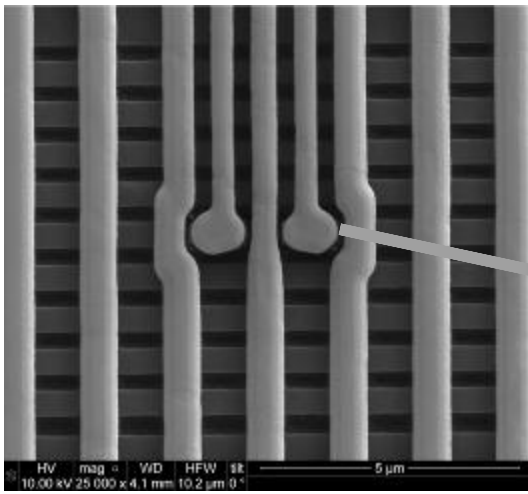


用于刻蚀或沉积的气相可能引入其它材料。

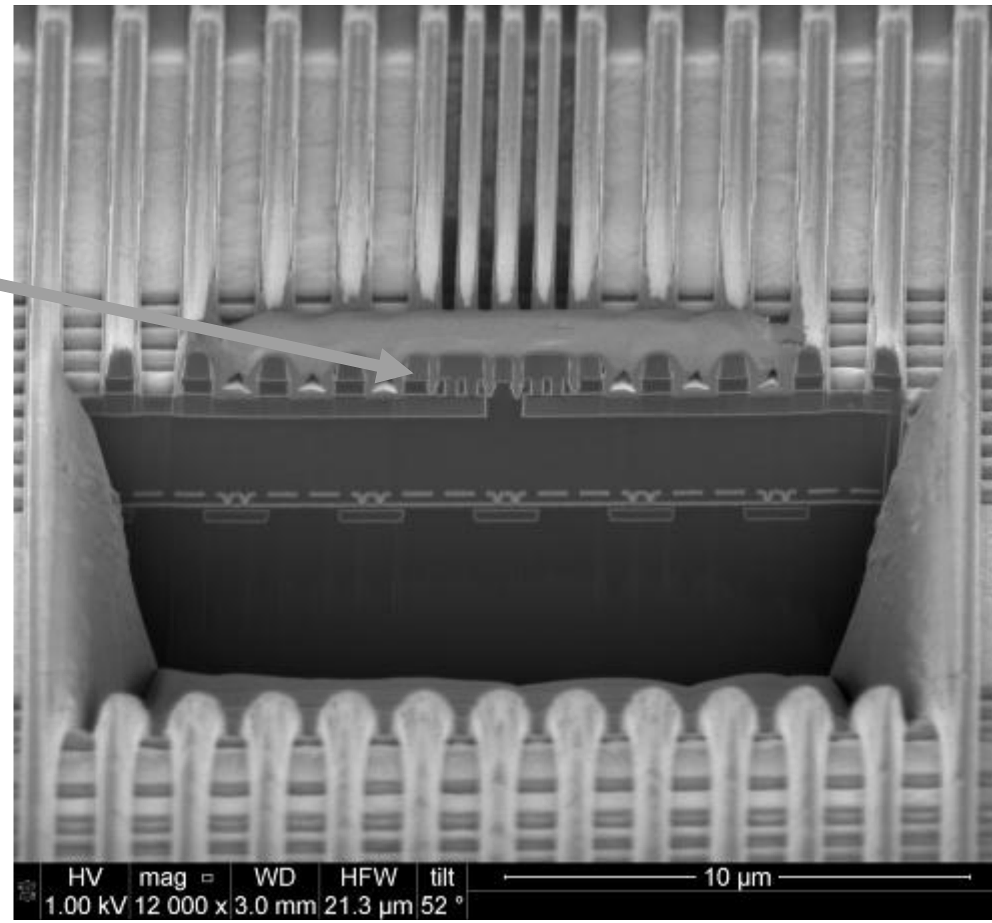
TYPICAL RESULTS



集成电路的DUAL BEAM FIB CROSS SECTION

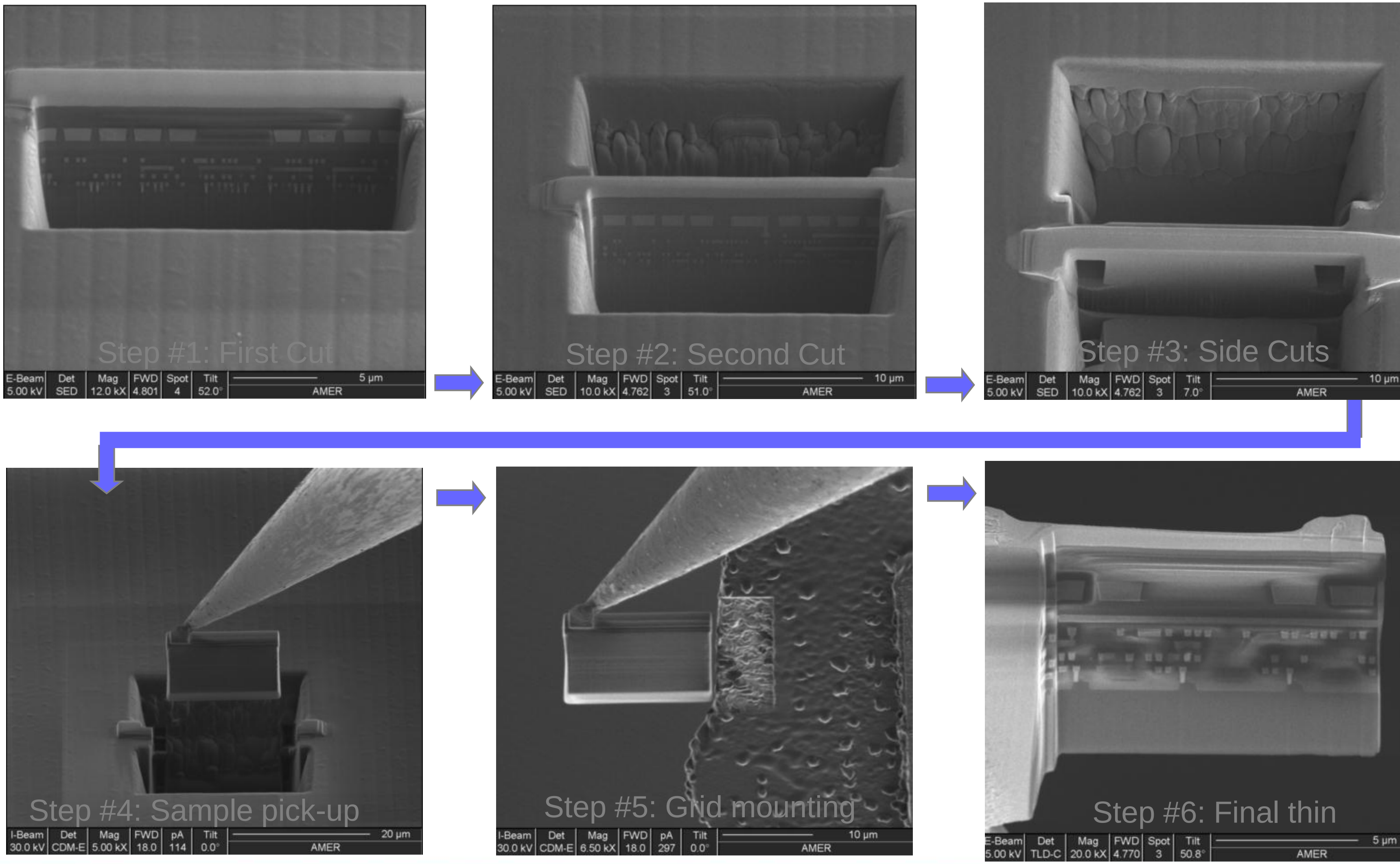


很小的图形有die map(GDS II files)的帮助可以做横界面制样，线或连接可以用高精度的横切。



EAG LABORATORIES

TEM in Situ lift out

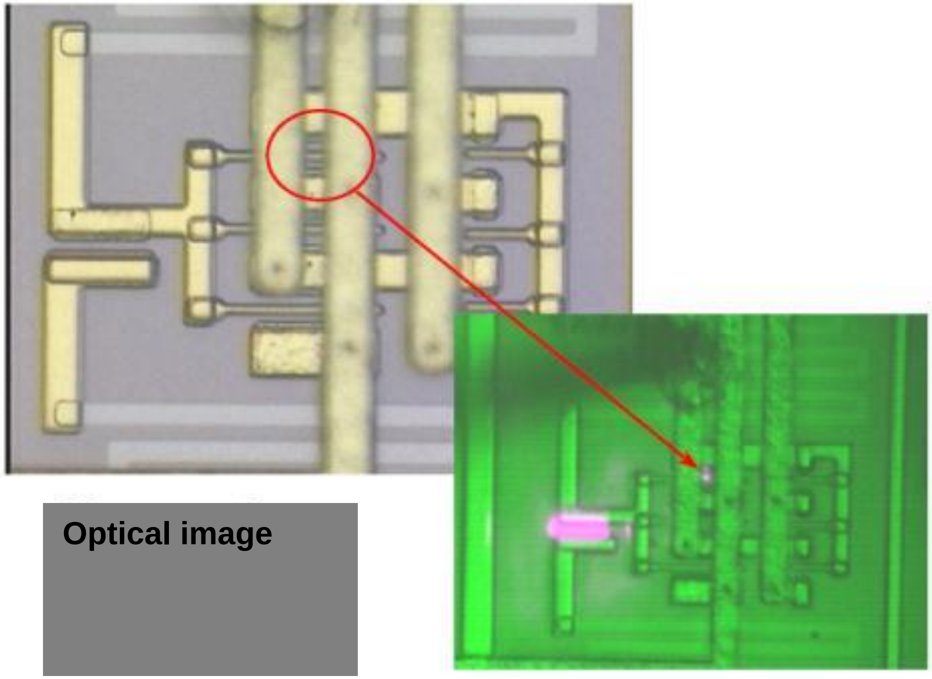


WHAT (S)TEM CAN DO…

- HR Imaging (Bright Field/Z-contrast/SE)
- Composition Identity (EDS, EELS…)
- Crystal Defects/Dislocations (Diffraction Contrast)
- Crystal Orientation/Structure (Phase Contrast, Atomic Resolution Imaging, SAD, NBD, CBED,FFT)
- Electronic Structure (EBIC)
- And much more (3D imaging, Strain Field, EMF by Electron Holography, …)

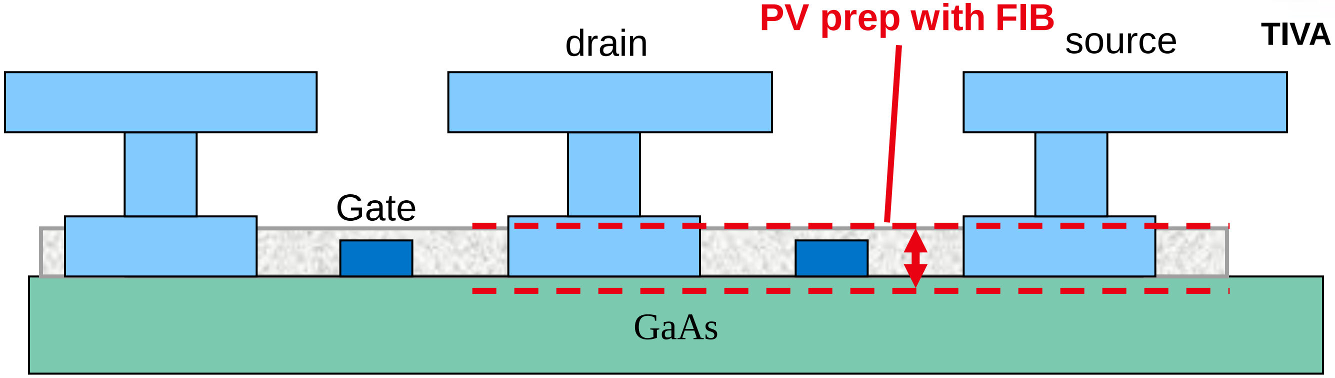
FAULT LOCALIZATION VIA PV AND XS FROM A PV

- 电性能测试证明在source和gate之间有短路
- FIB lift out 首先用plan view样品制备，包含 gate and source 欧姆接触区域



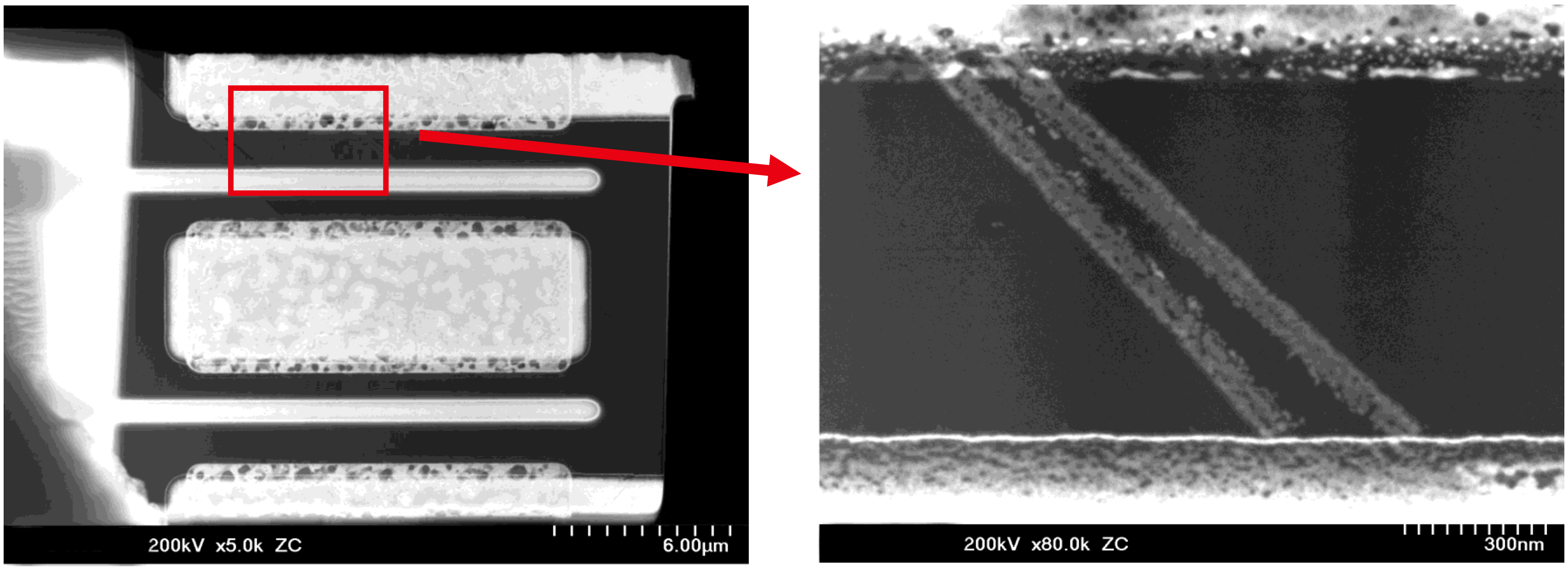
Optical image

TIVA 像显示了缺陷位置



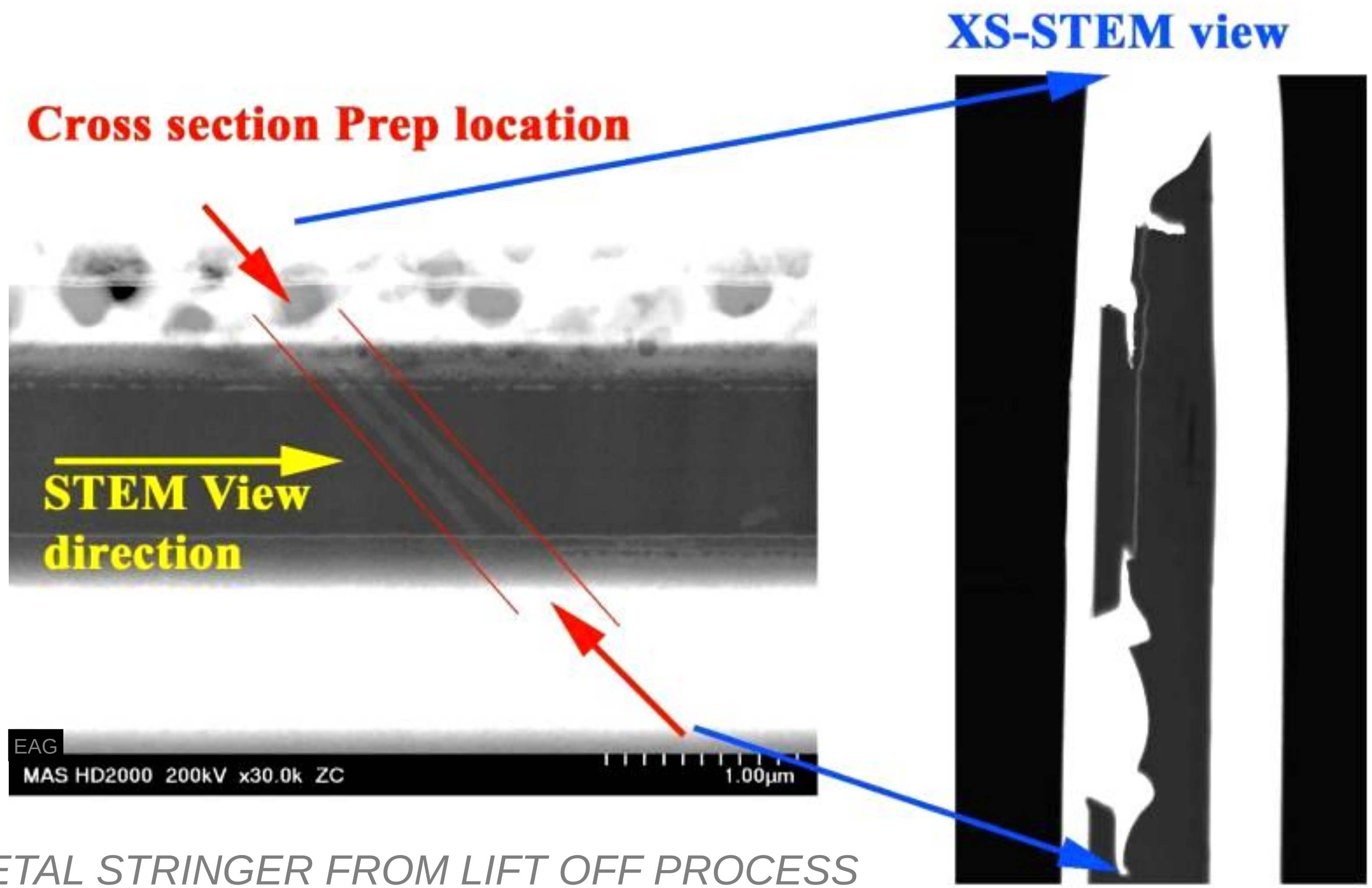
STEM的集成电路分析

15 BY 15 MICRON PLAN VIEW



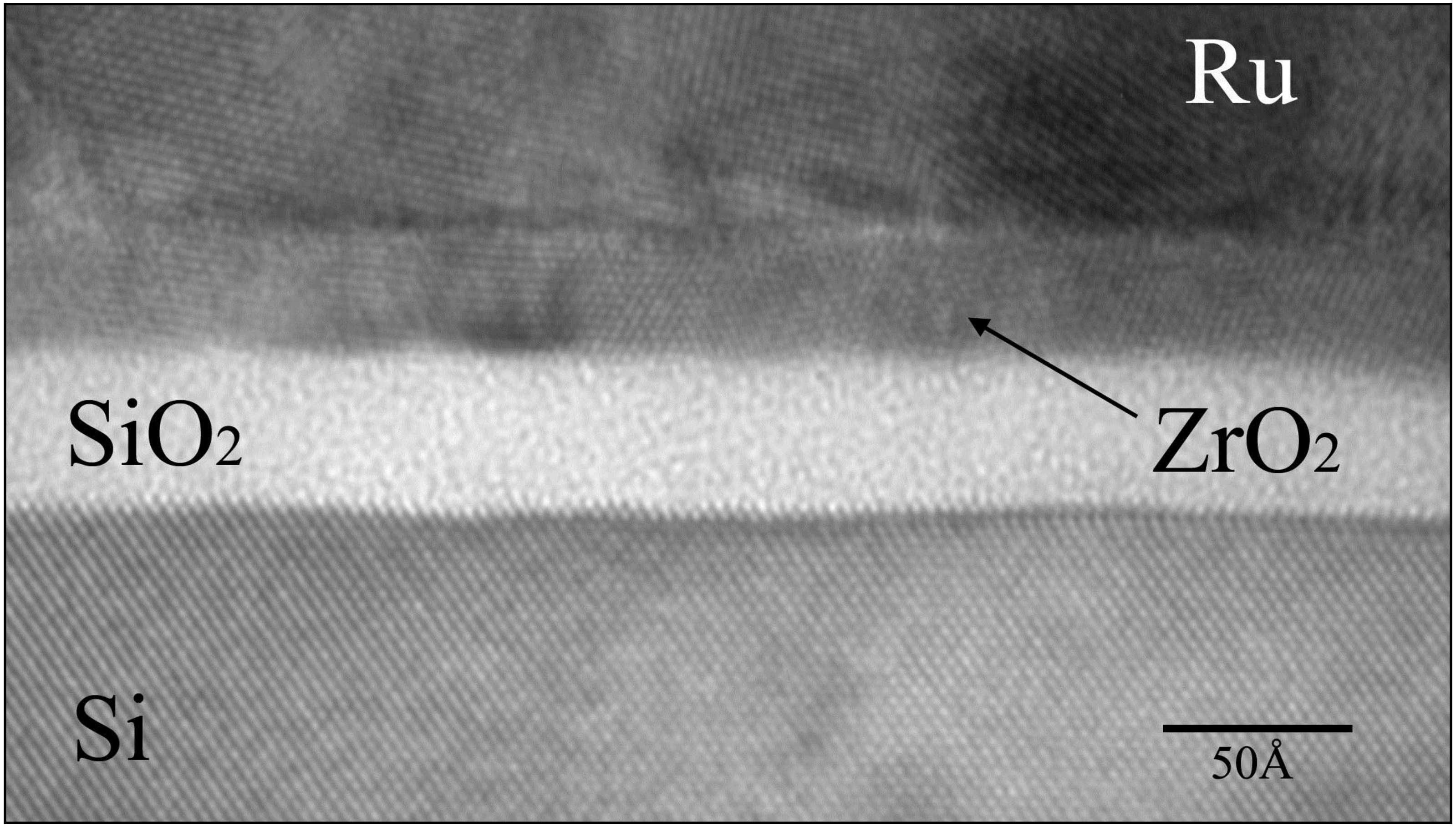
样品通过减薄表面制备到适合TEM的厚度.

Fault Localization via PV and XS from a PV



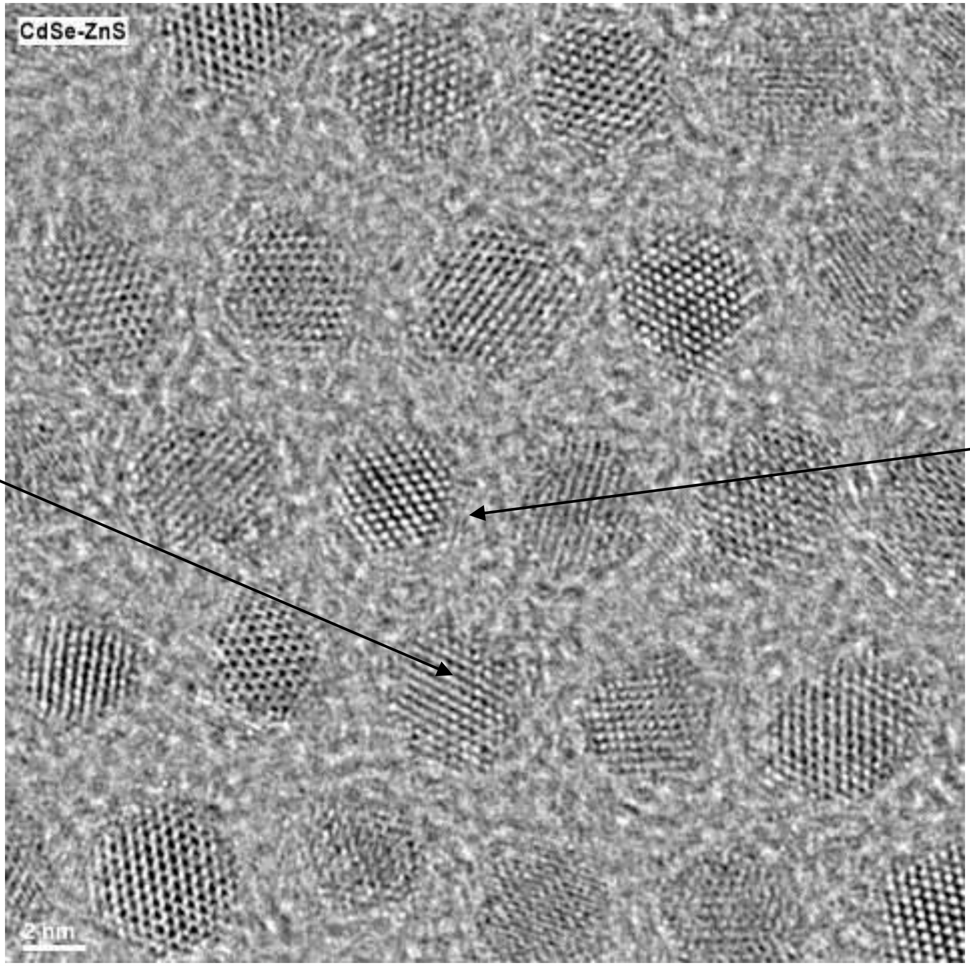
METAL STRINGER FROM LIFT OFF PROCESS IDENTIFIED!

HRTEM PREPARATION



可以避免FIB 损伤

HR-TEM of CdSe/ZnS 壳-核纳米颗粒



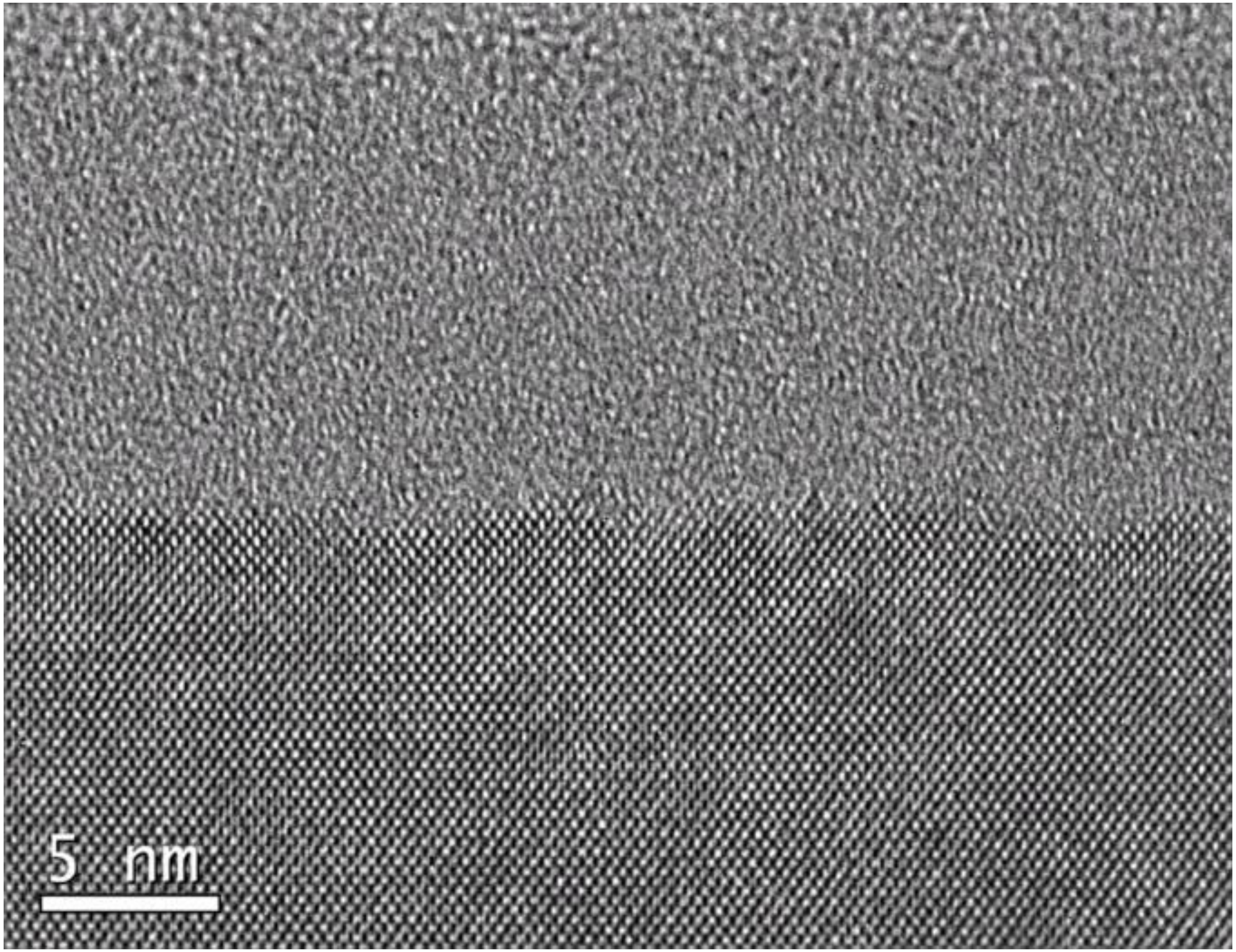
每个点是 核颗粒的晶格点.

晶格条纹来自很薄的壳材料

FE-TEM 高分辨率成像

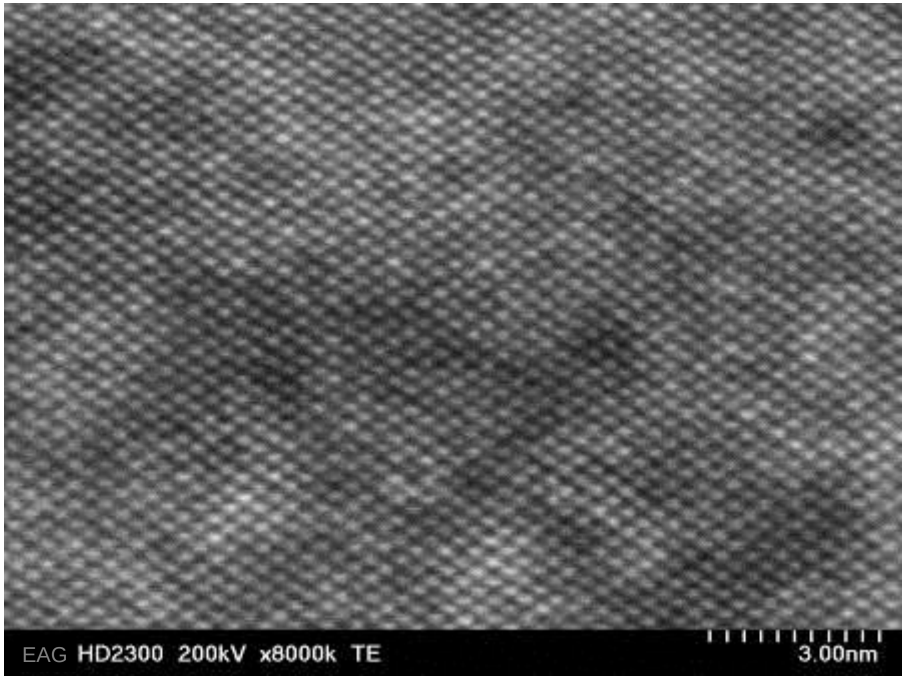
非晶层

Si 基体

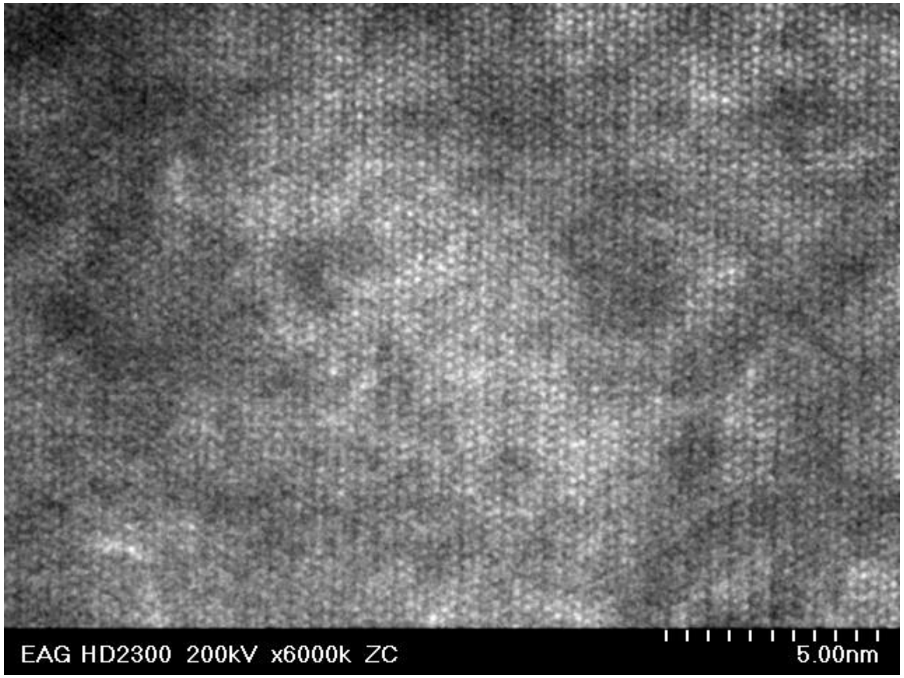


STEM 高分辨率成像

GaAs



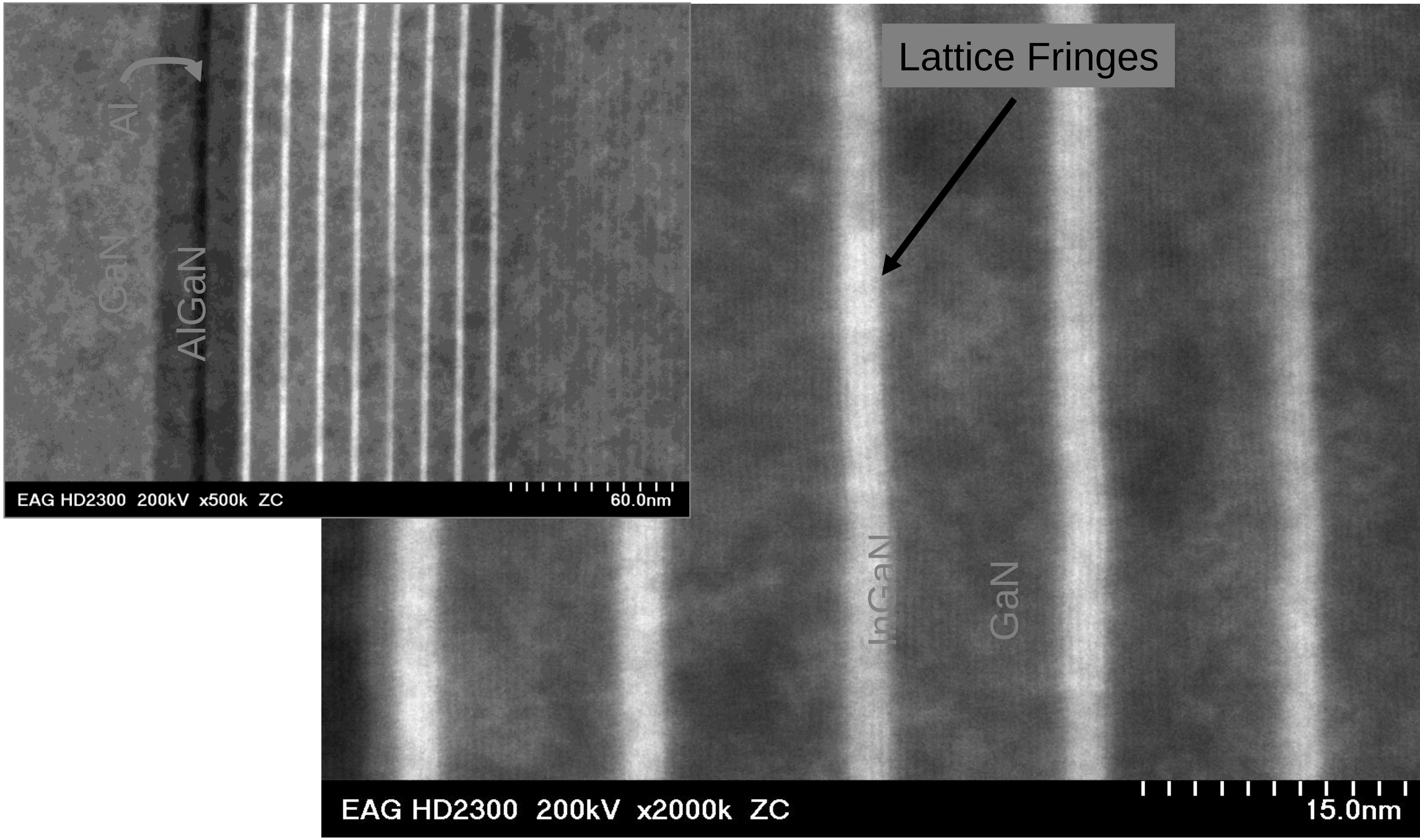
GaN



~0.2 nm Resolution

原子排列直接成像

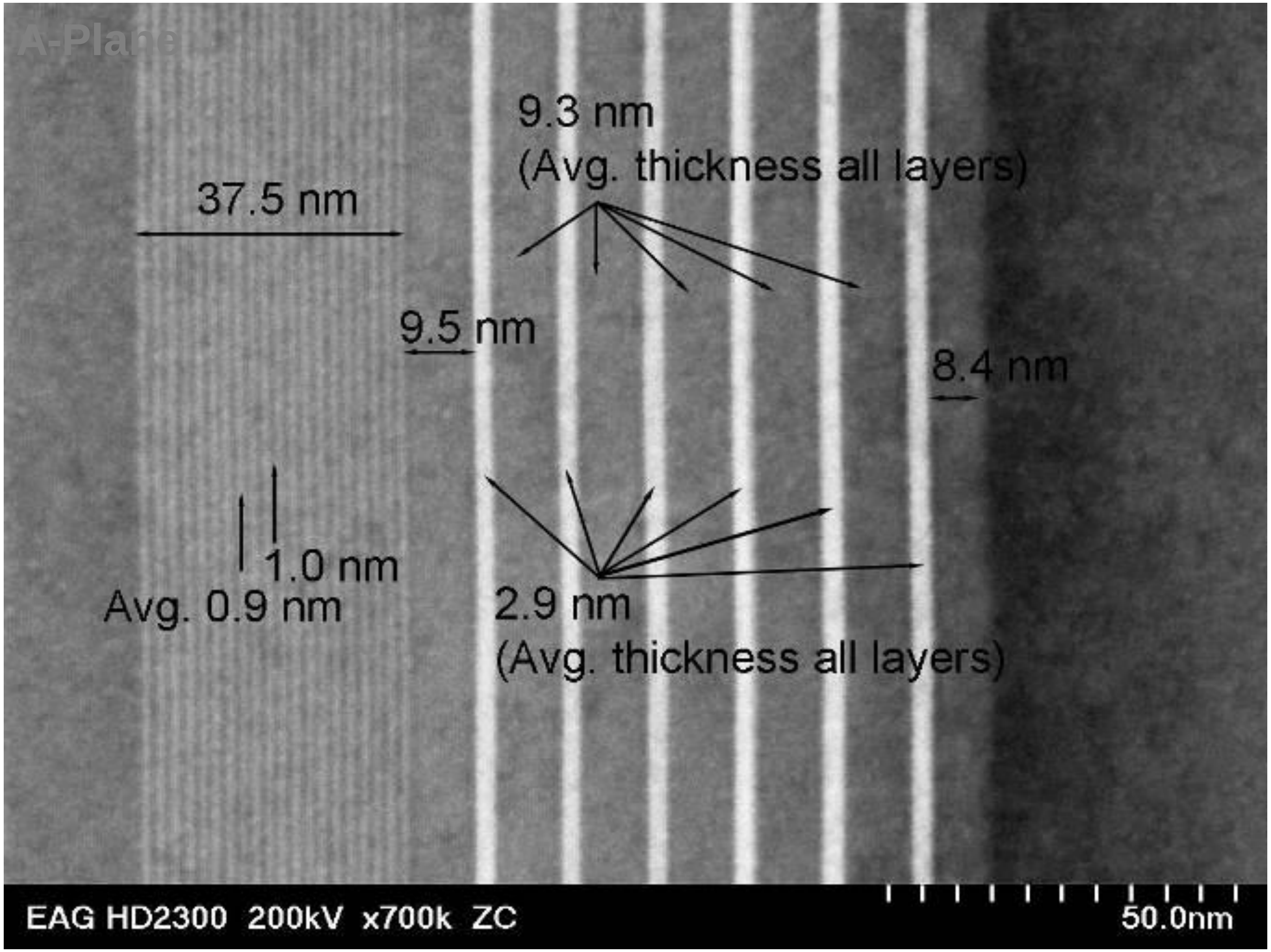
量子阱结构的Z- CONTRAST IMAGE



Z-衬度像, 显示了GaN和AlGaN阻挡层和InGaN的量子阱层.， STEM具有独特的Z-衬度成像能力 。

GAN BASED LED结构分析

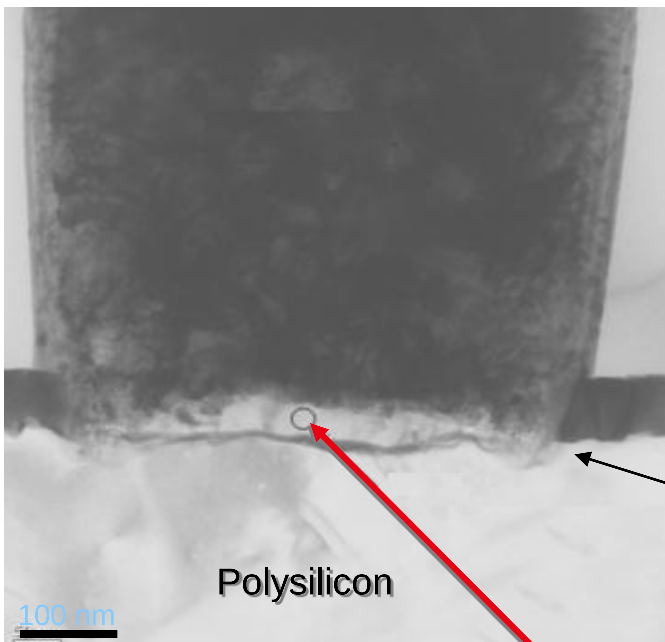
测量每层的厚度

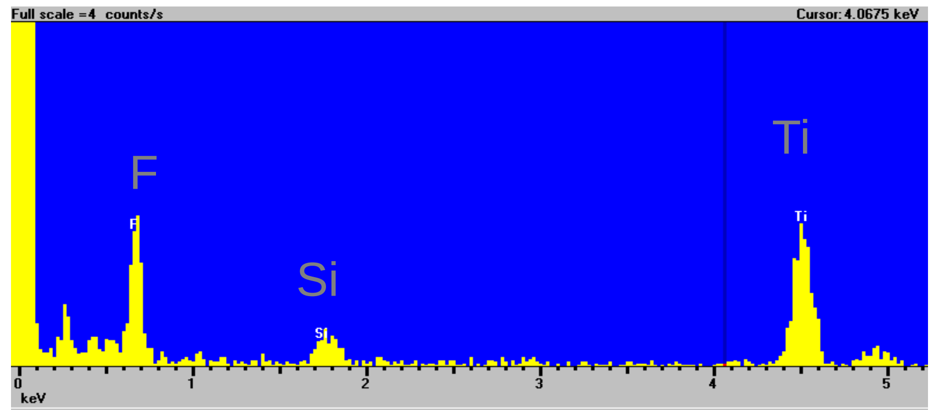


实例应用

W
(Poly contact)

通过界面的EDS 显示有F沾污

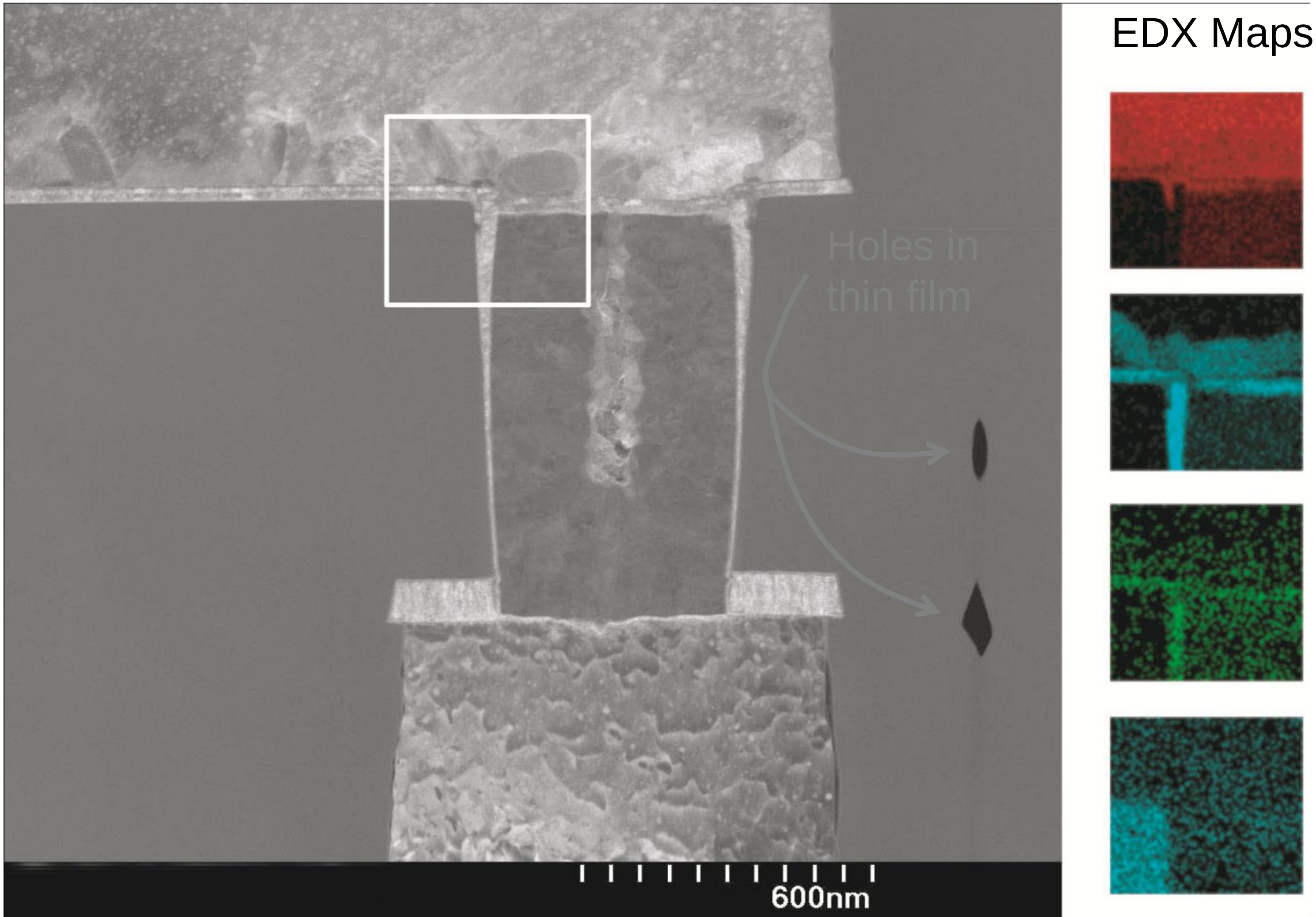




TiSi layer

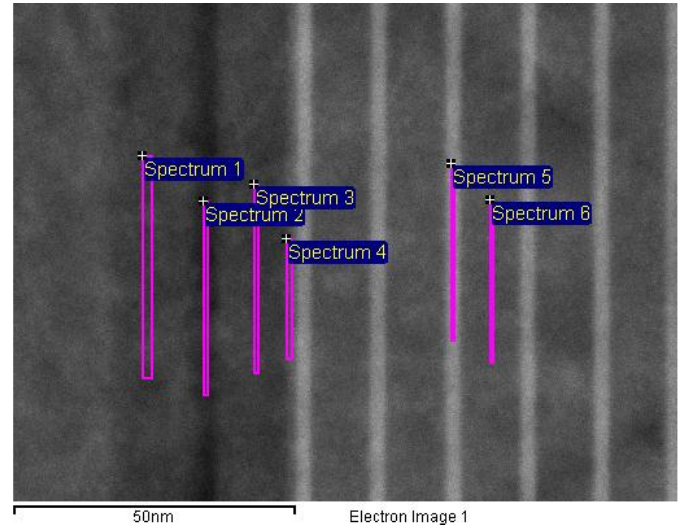
EDS Site

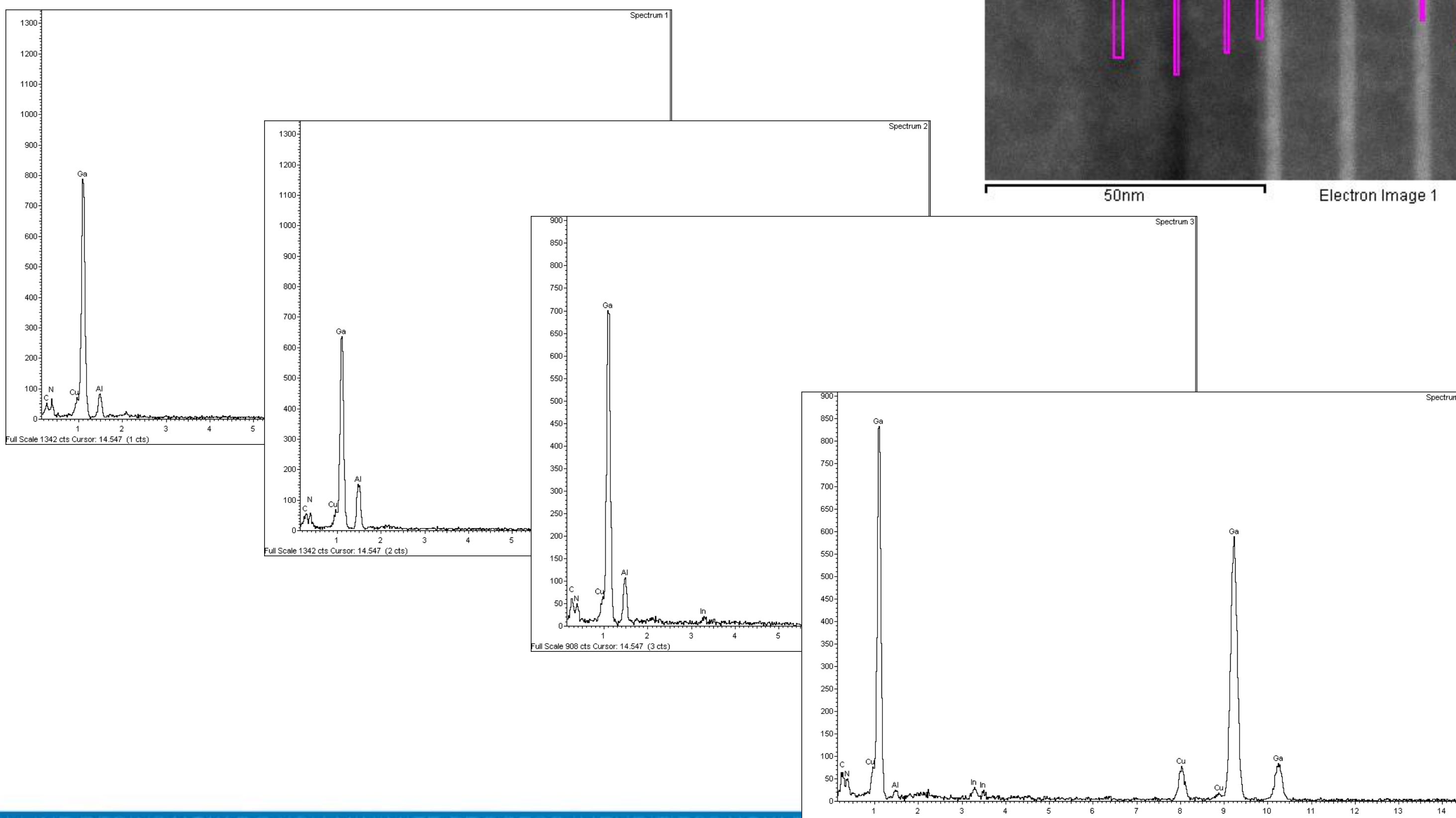
ELEMENTAL MAPS BY STEM



GAN BASED LED结构分析

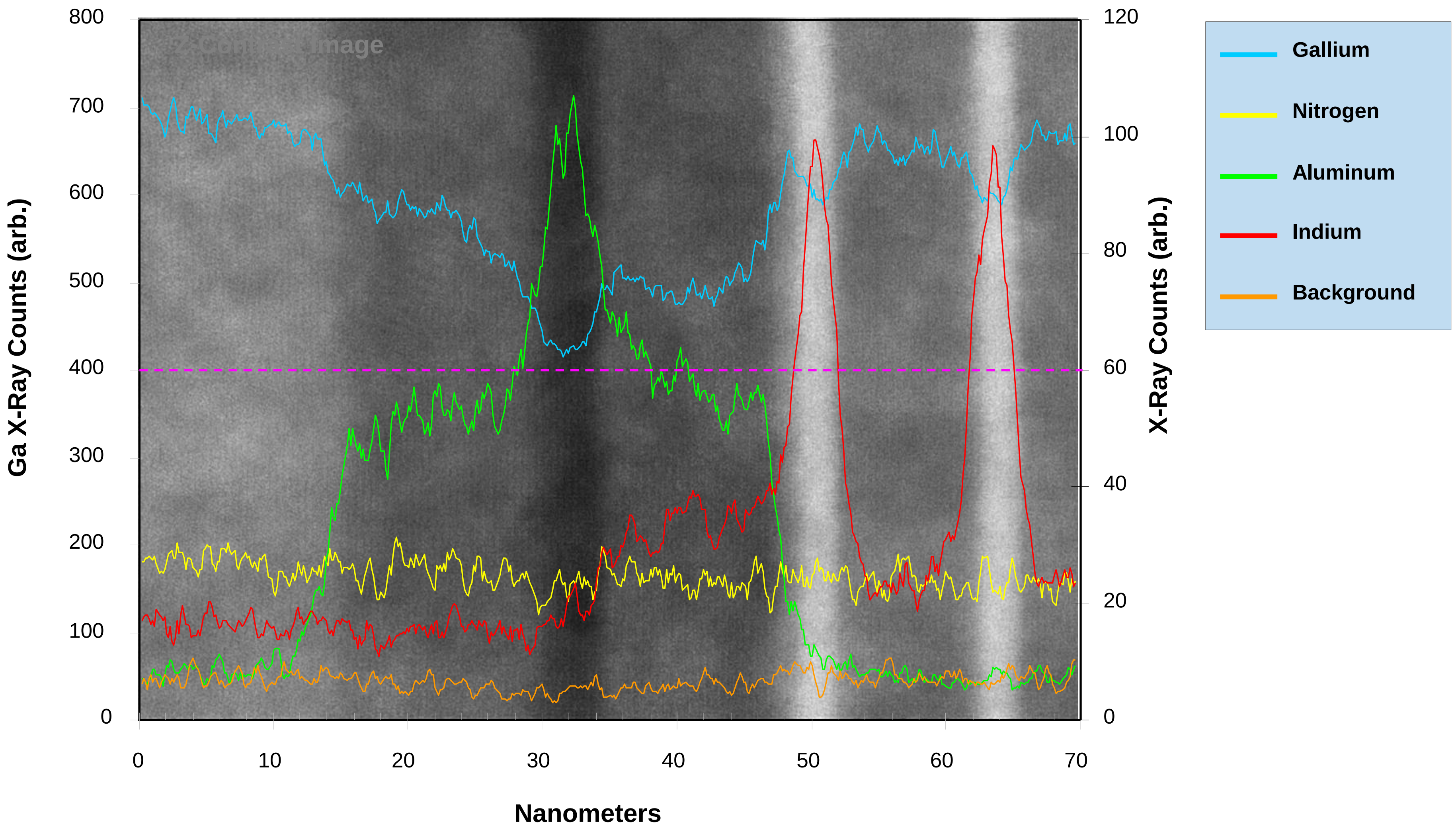
识别每层的曾分



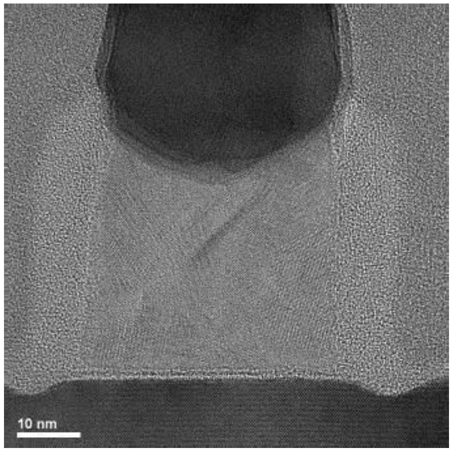


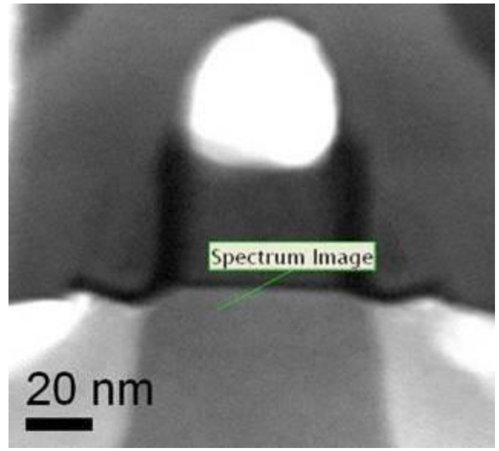
GaN Based LED结构分析

元素线扫描



反相工程：gate是氧化物还是氧氮化物？EELS





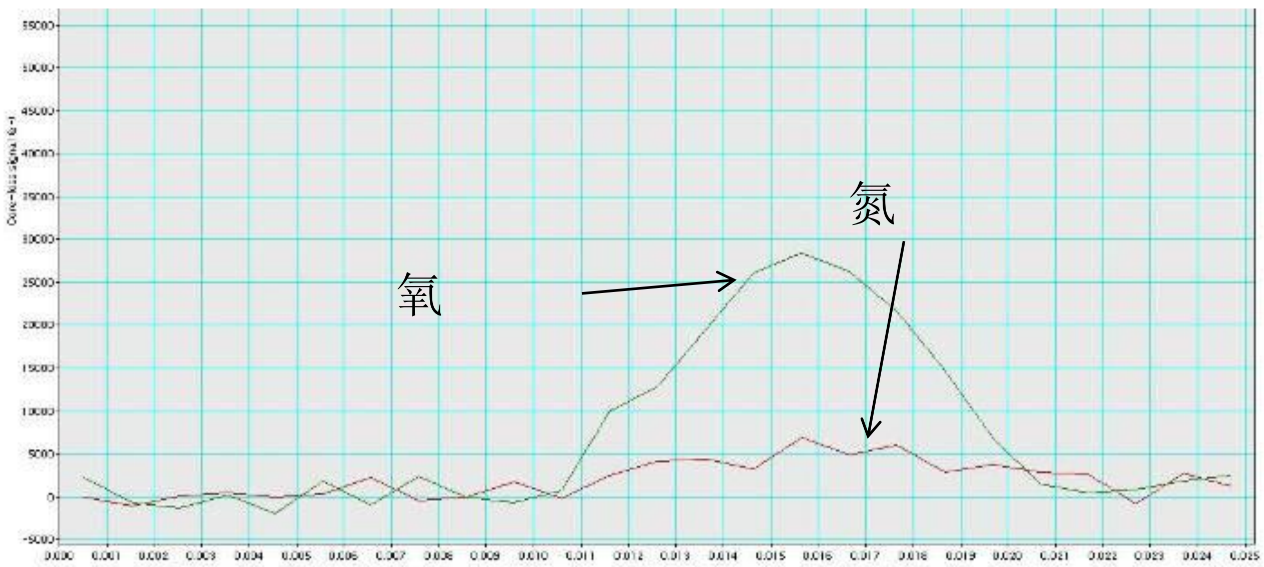


Fig. A: HRTEM image of a MOSFET gate. Gate 介电层在~2nm厚.

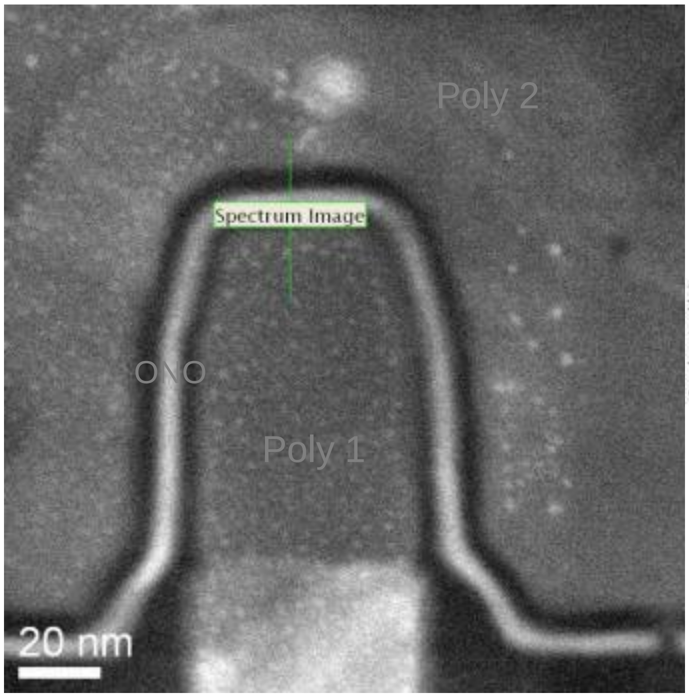
Fig. B: 同一区域的暗场 STEM image 显示EELS线扫描从Si基体开始。

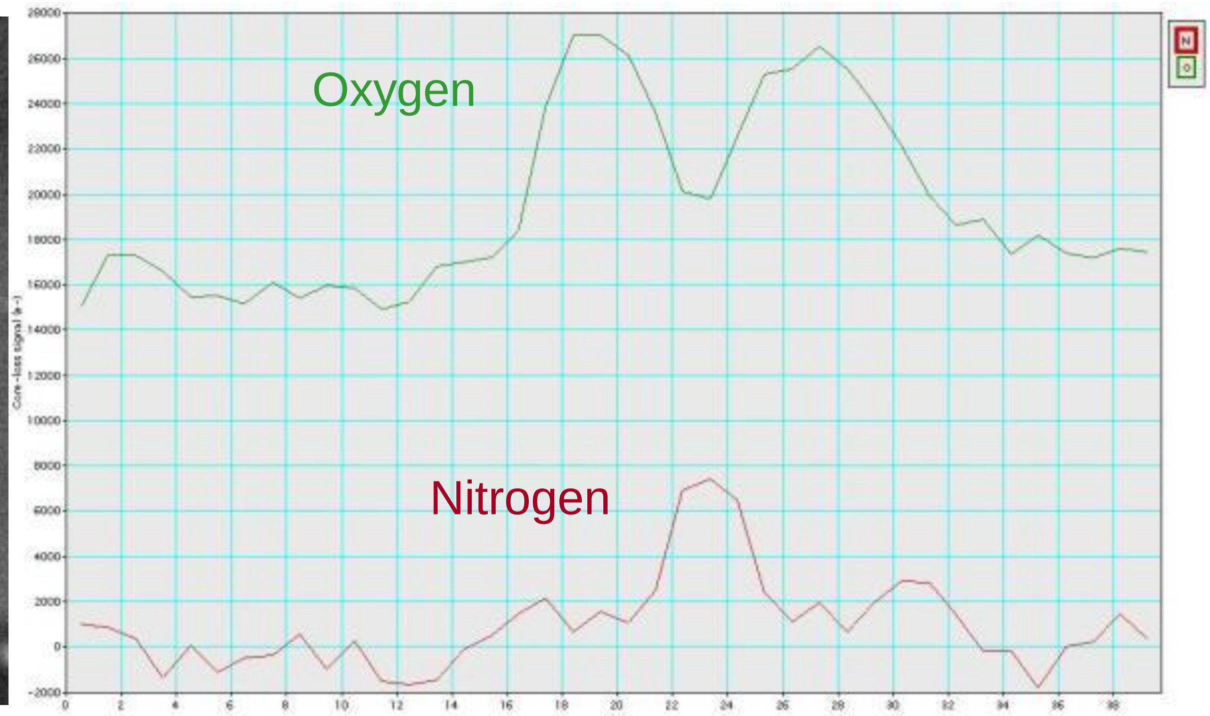
Fig C:另外在gate的介电层测到了氮和氧，证实是氧氮化物.

EDS 是测不到N的.

EAG LABORATORIES

Electron Energy Loss spectroscopy



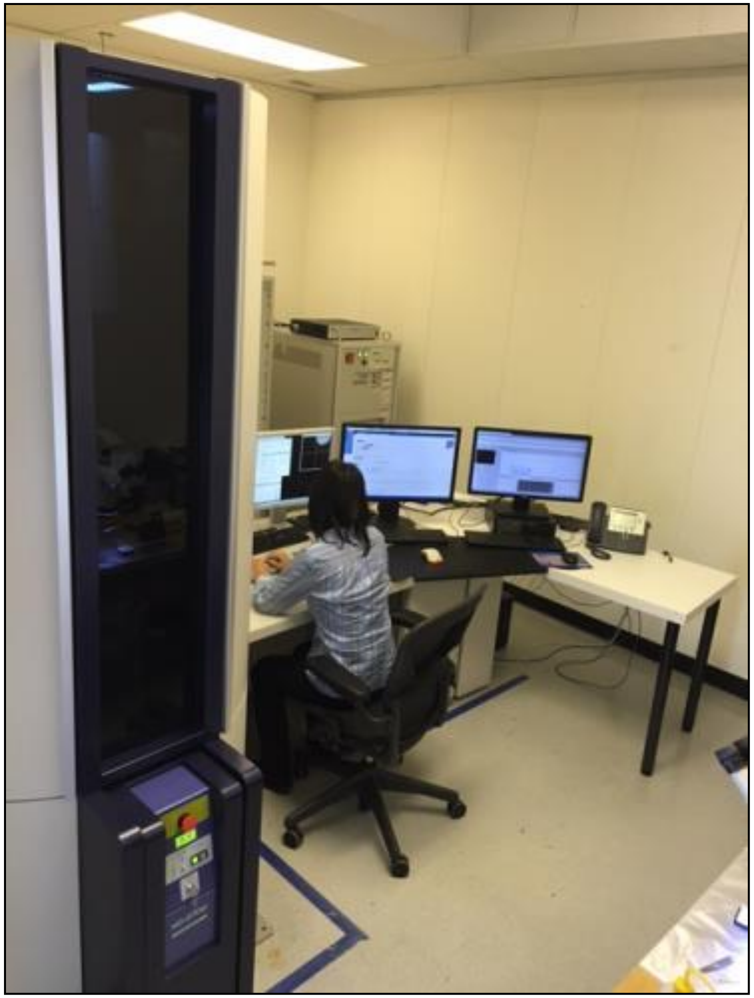


STEM Image of Flash memory cross sectioned parallel to the word line. The poly1 floating gate, oxide/nitride/oxide(ONO) dielectric stack, and the poly2 gate electrode were imaged. The ONO stack was investigated using EELS. The EELS 数据显示ONO stack 层大概在5nm : 4nm : 5nm.

High Resolution Imaging

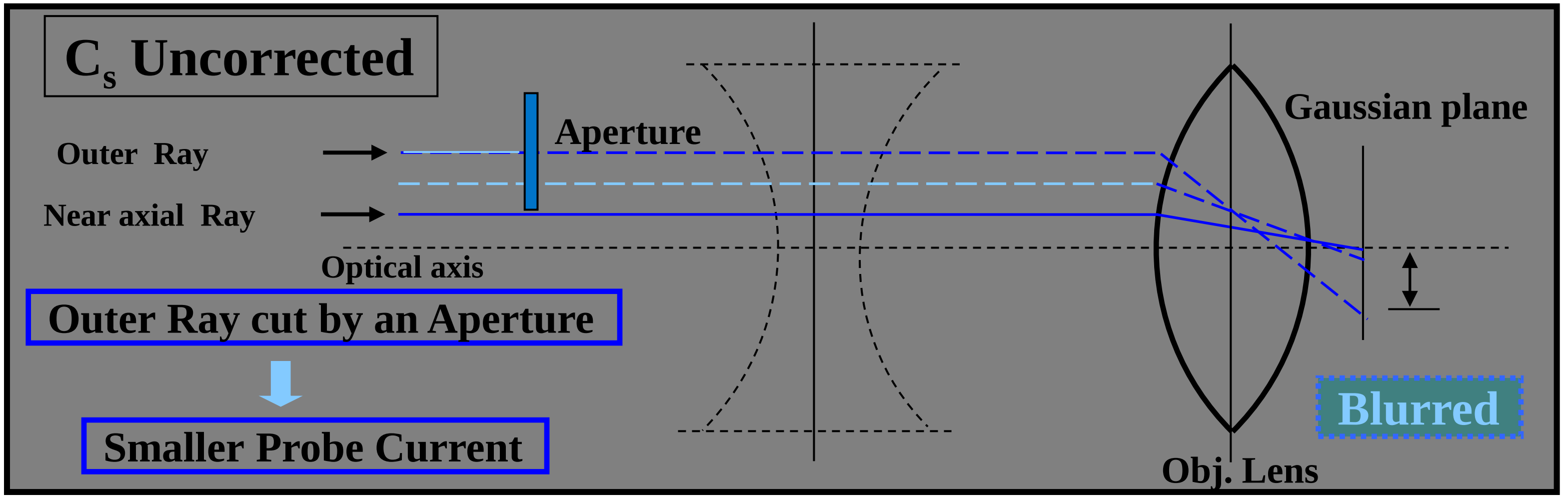
with Aberration Corrected STEM
(AC-STEM)

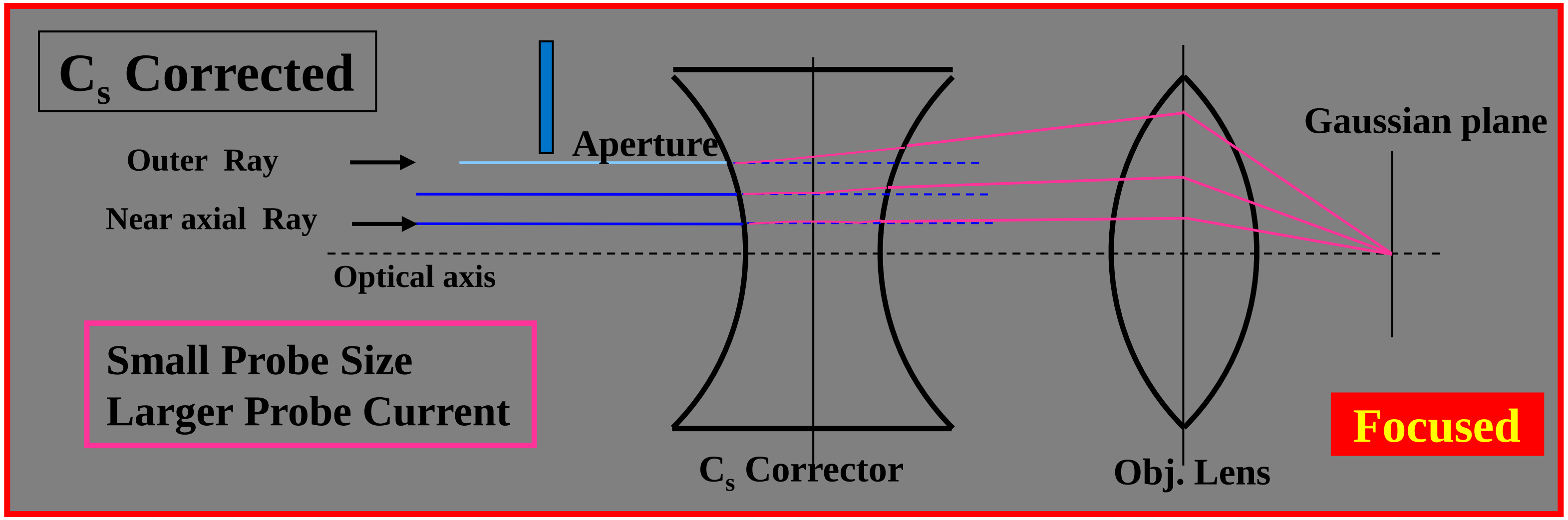
EAG-NC AC-STEM



- Hitachi HD2700 STEM
- Hitachi C_S-Corrector
- Bruker QUANTAX EDS

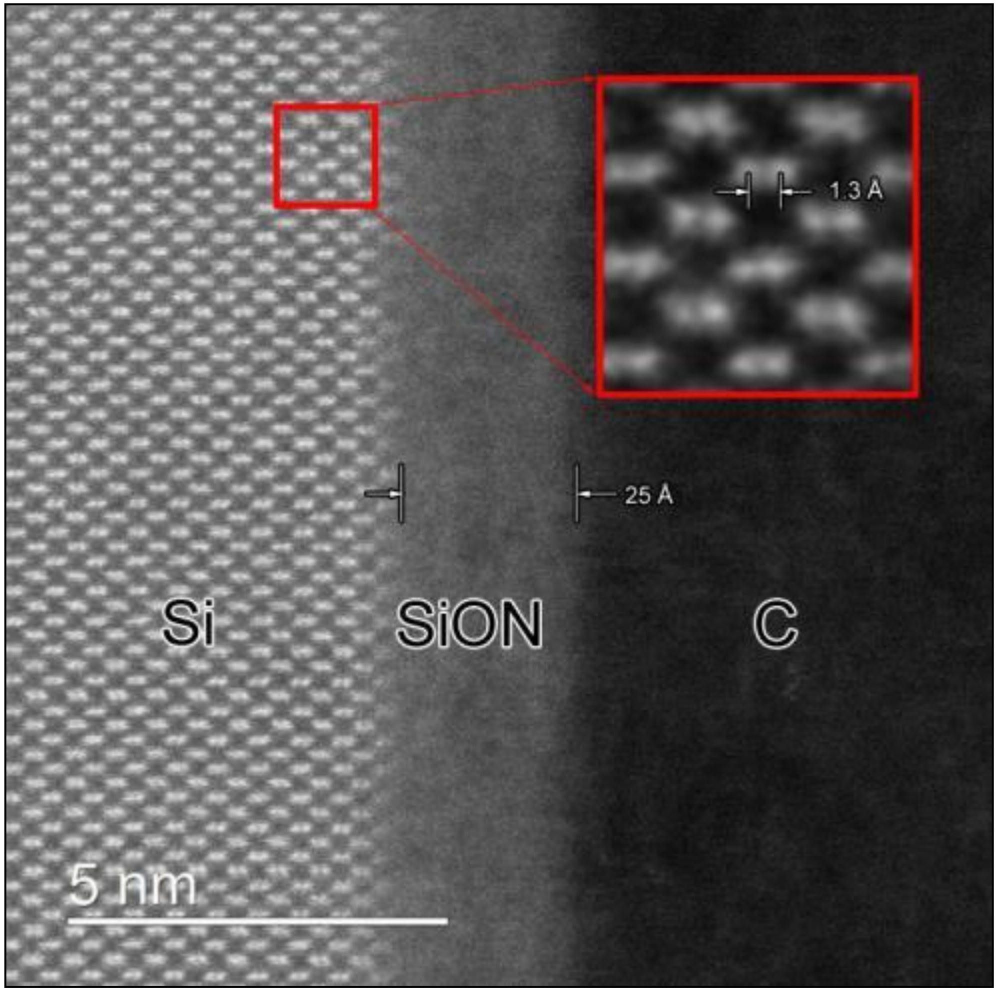
AC-STEM: CORRECTING FOR SPHERICAL ABERRATION





AC-STEM ADVANTAGE: FINER PROBE=HIGHER RESOLUTION

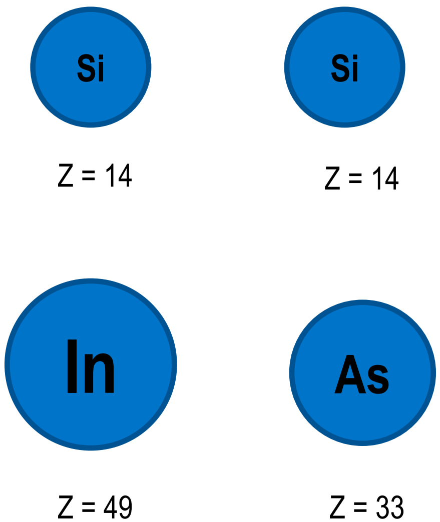
SiON on Si



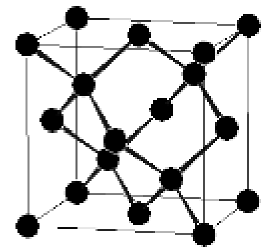
- FIB prepared Samples
- 0.13 nm resolution
- Great for examining thin layers
- ZC images are directly interpretable

Atomic Resolution: Structure with Composition

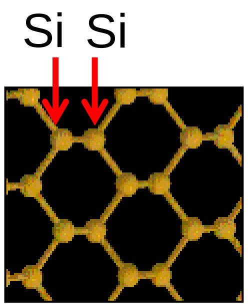
AC-STEM Z-Contrast:
Relative Intensity varies
with atomic number (Z)



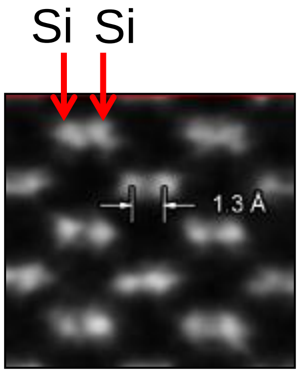
Si – Diamond Cubic



Unit cell

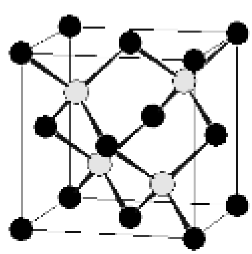


110 projection

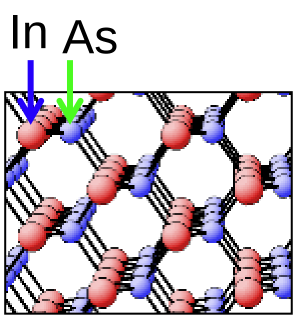


STEM ZC

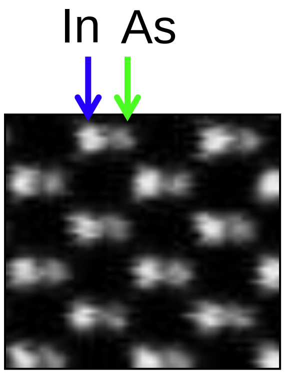
InAs - Zinc Blende



Unit cell



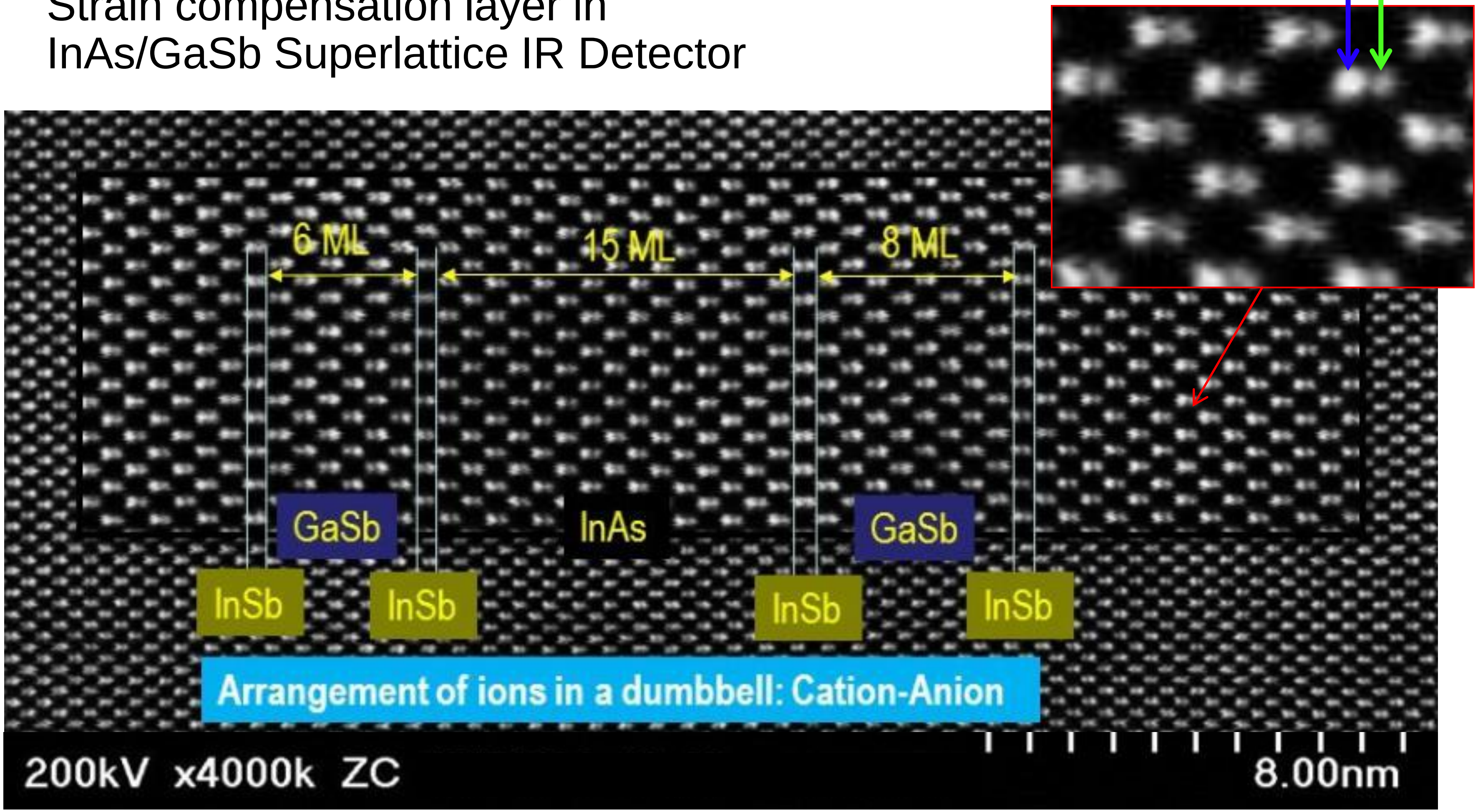
110 projection



STEM ZC

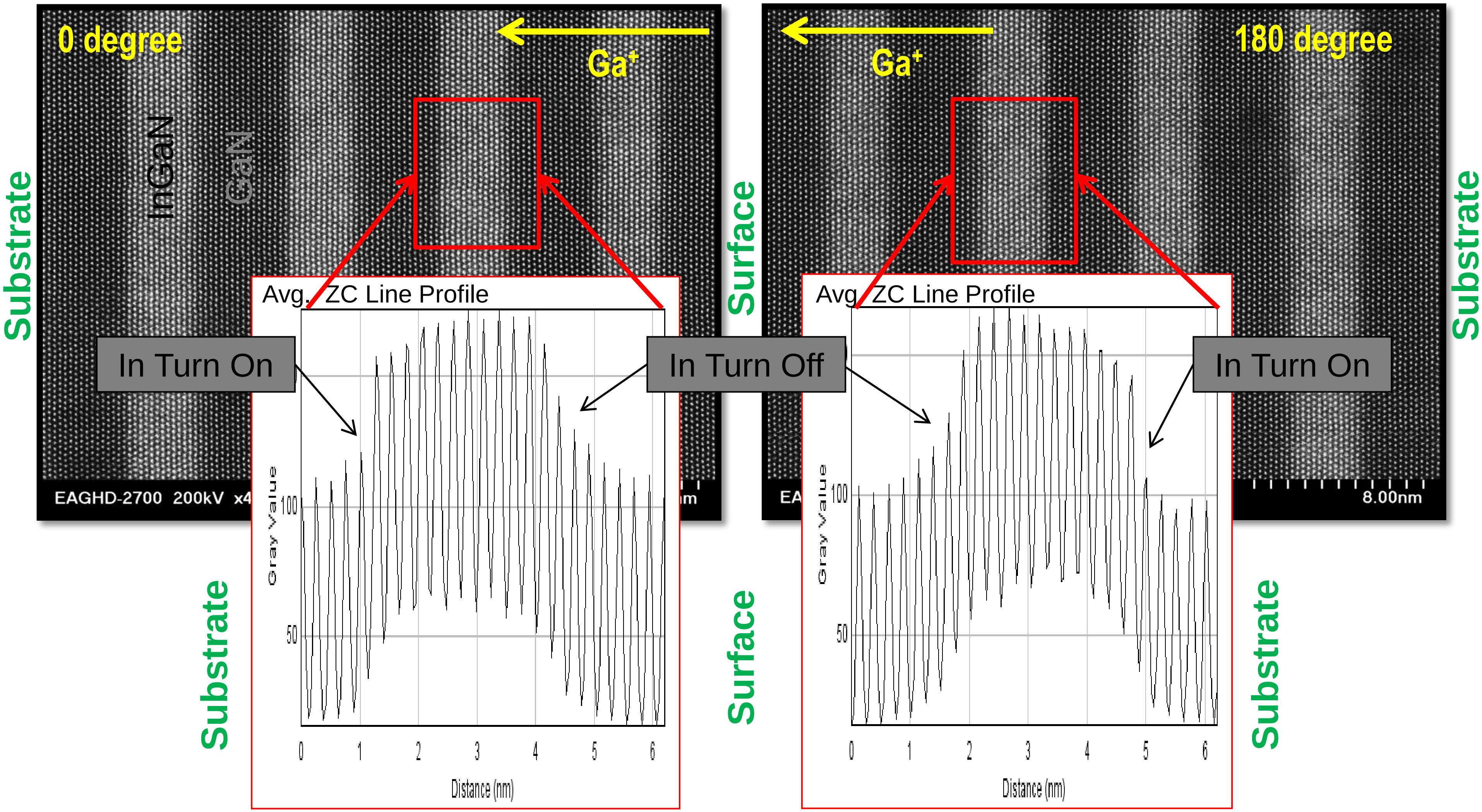
ATOMIC RESOLUTION: ENGINEERING STRUCTURE

Strain compensation layer in
InAs/GaSb Superlattice IR Detector



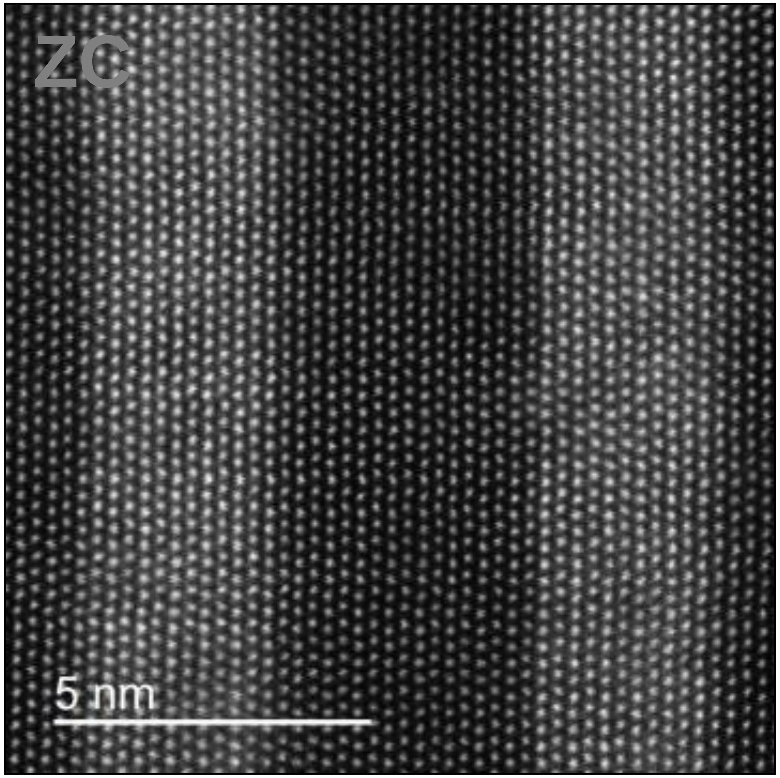
- AC-STEM ZC images combined with computational modelling and electrical performance data confirms there is a single monolayer of InSb at the interfaces of the GaSb and InAs

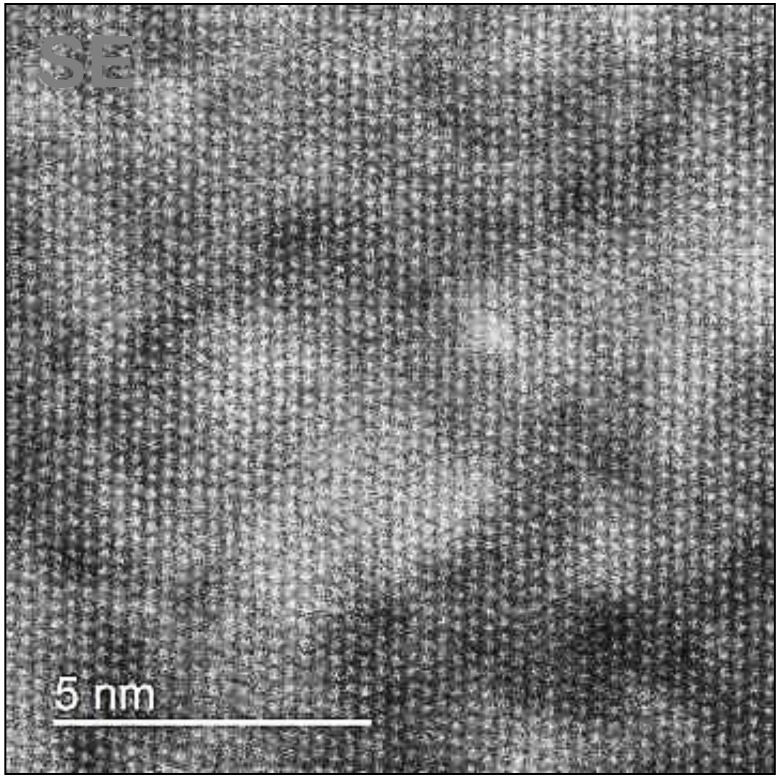
ATOMIC RESOLUTION: MONITORING INDIUM COMPOSITION



In composition ~5-10 at%. ZC intensity variance not from FIB sample prep!

ATOMIC RESOLUTION: SURFACE IMAGING





- AC-STEM of A-plane GaN – ZC/SE
- Images are of the same region on the same ~ 50nm thick FIB prepared sample

Composition Identity

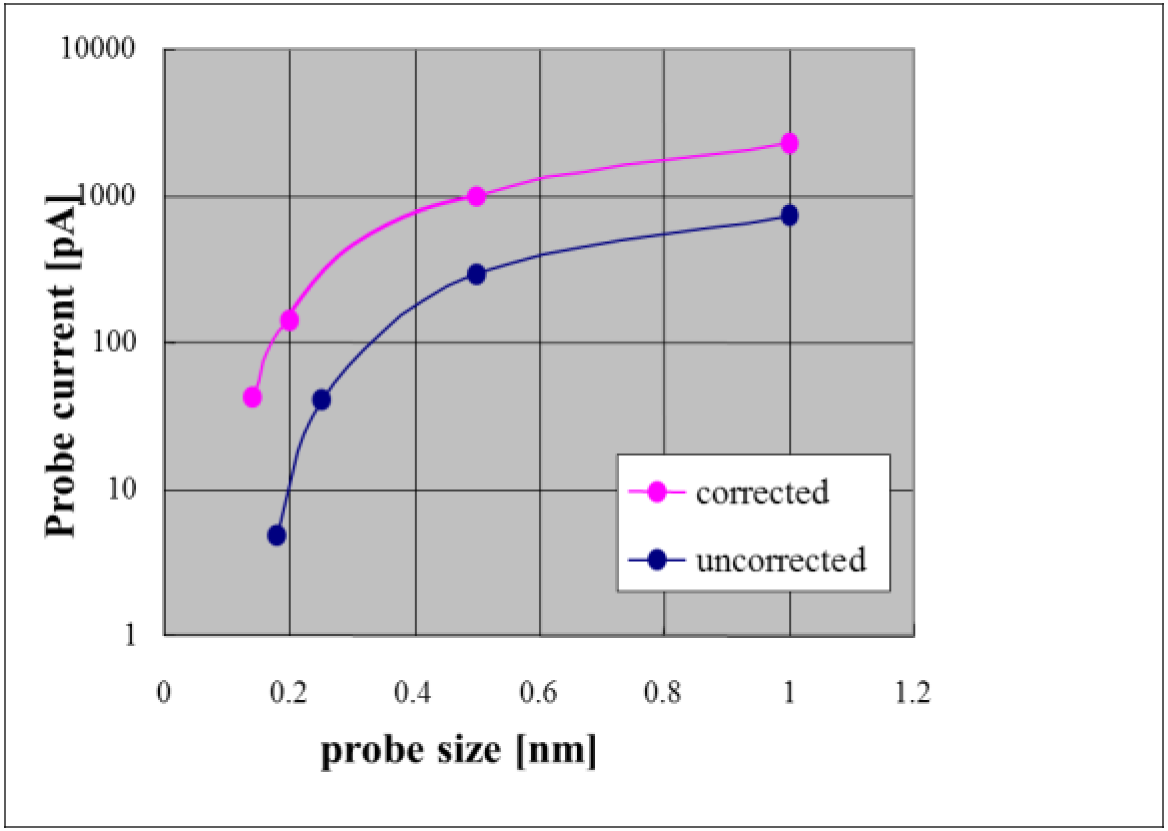
with EDS on AC-STEM

AC-STEM ANALYTICAL ADVANTAGE

More beam current in a smaller e-probe!

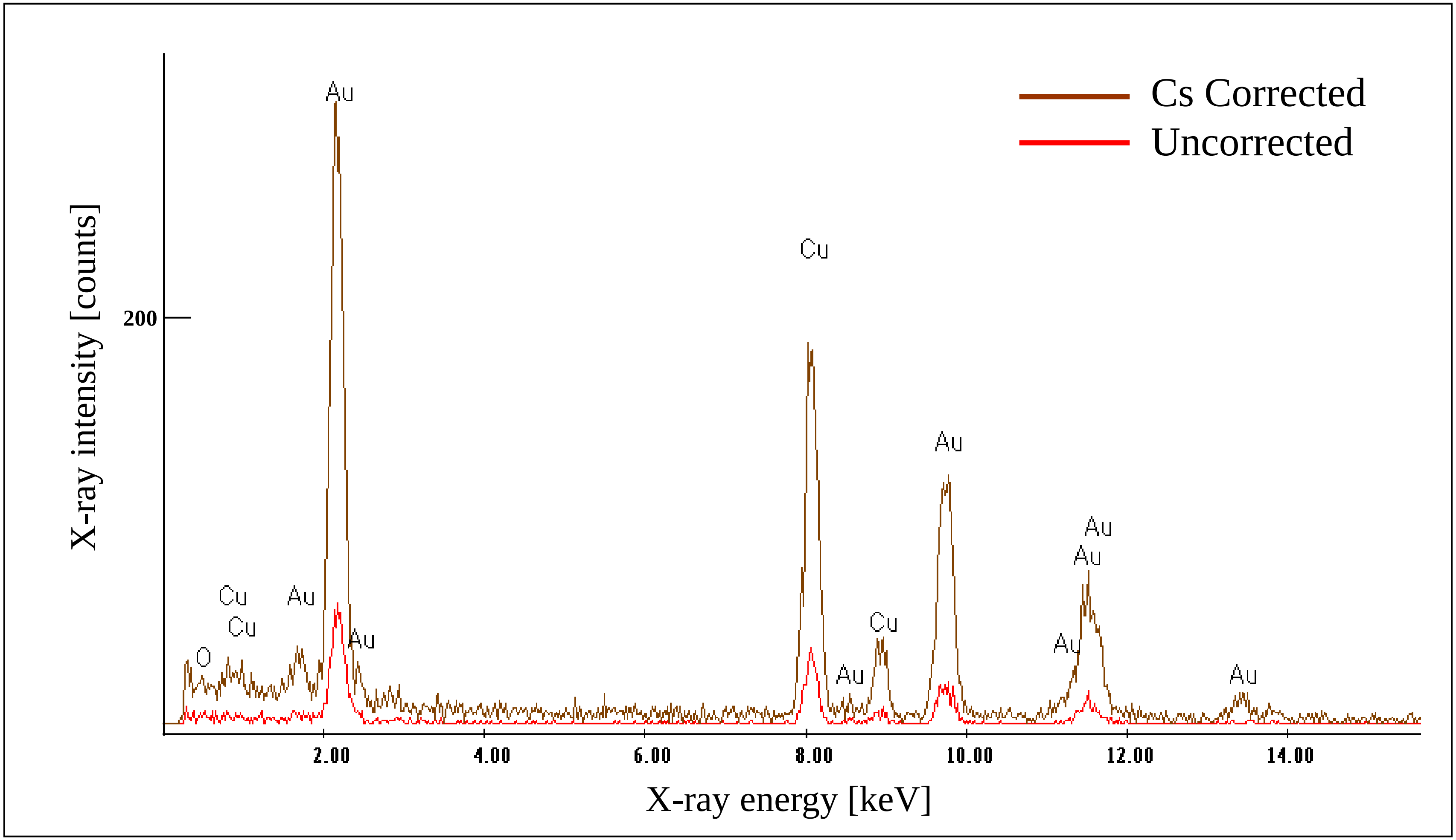
10x
probe current

2x
resolution



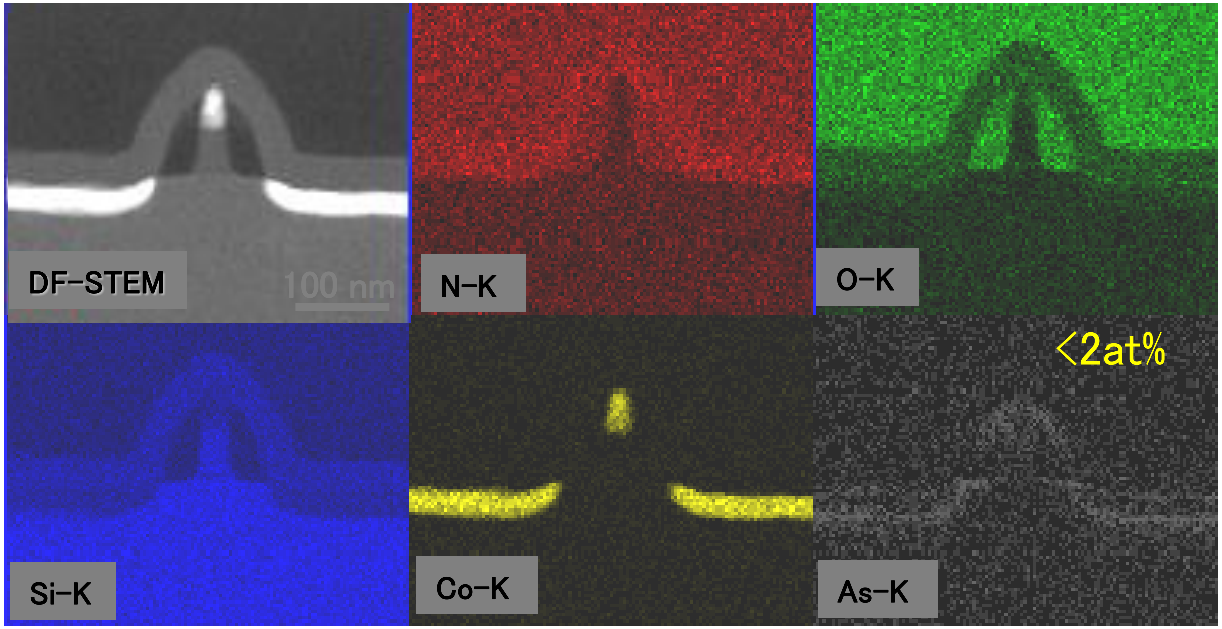
The spatial resolution for Imaging and EDS is improved by a Cs Corrector system.

HIGHER BEAM CURRENT = BETTER ANALYTICAL PERFORMANCE



EDS spectra of Au particles. Si(Li) detector, Acquisition time = 20 sec.
0.2 nm-probe (Red line = Uncorrected state, Brown line = Cs Corrected).

EDS MAPPING – LOW CONCENTRATIONS



Si Device EDS Mapping

>0.3 sr solid angle*

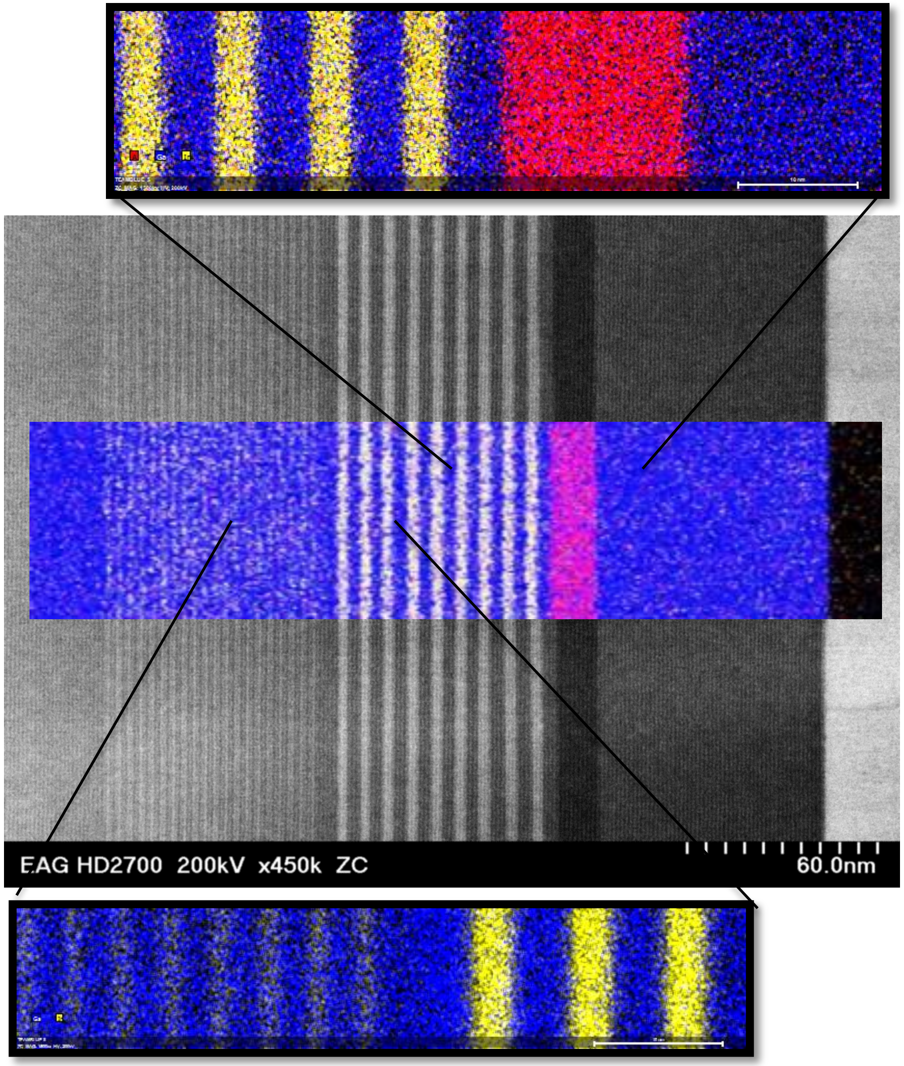
128x100 Pixels

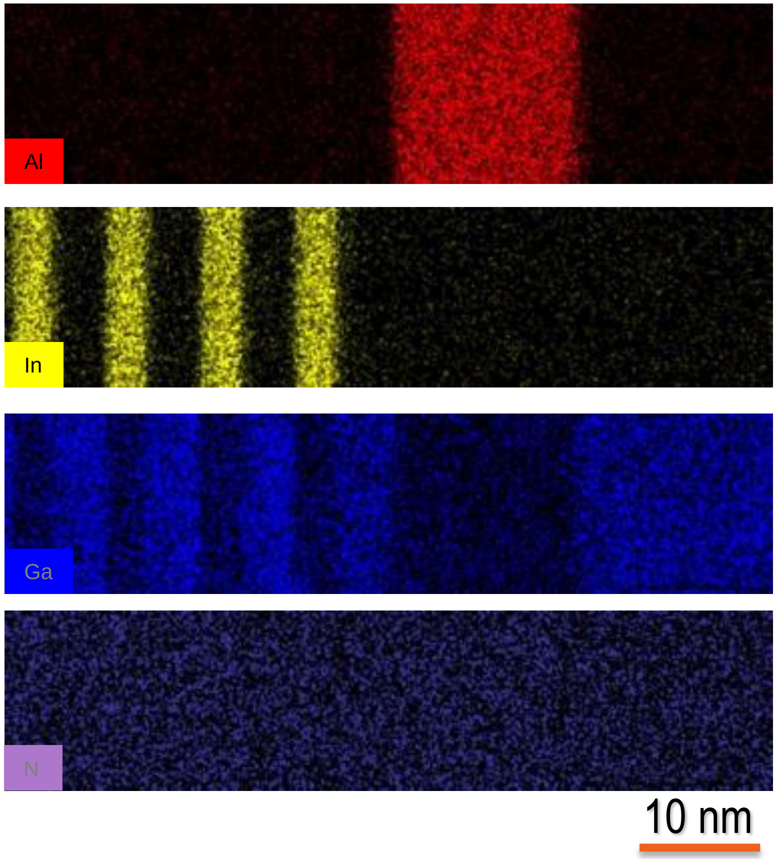
Dwell time: 0.5 ms/pixel

Total acquisition time: 20min

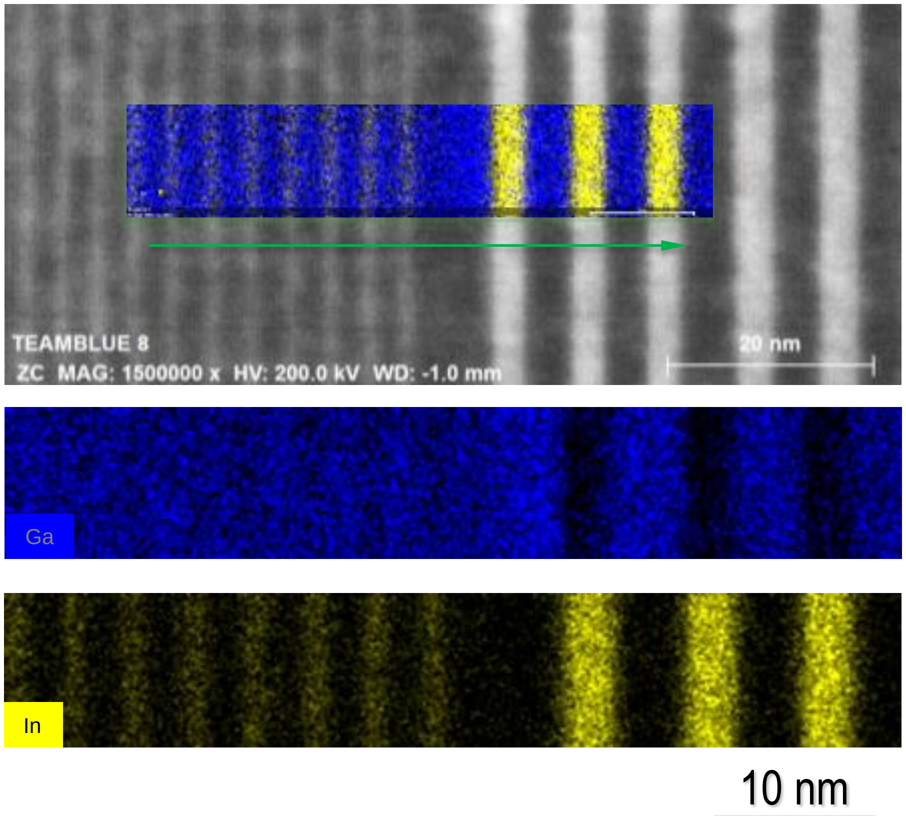
The aberration corrected STEM allows high-speed elemental mapping by using large probe current. The above shows HAADF-STEM and As-elemental mapping of a Si device. As-K can already been seen at only 1 minute acquisition time, and the distribution of As is clearly visualized after 20 minutes acquisition. This type of low concentration mapping cannot be realized by using the conventional STEM.

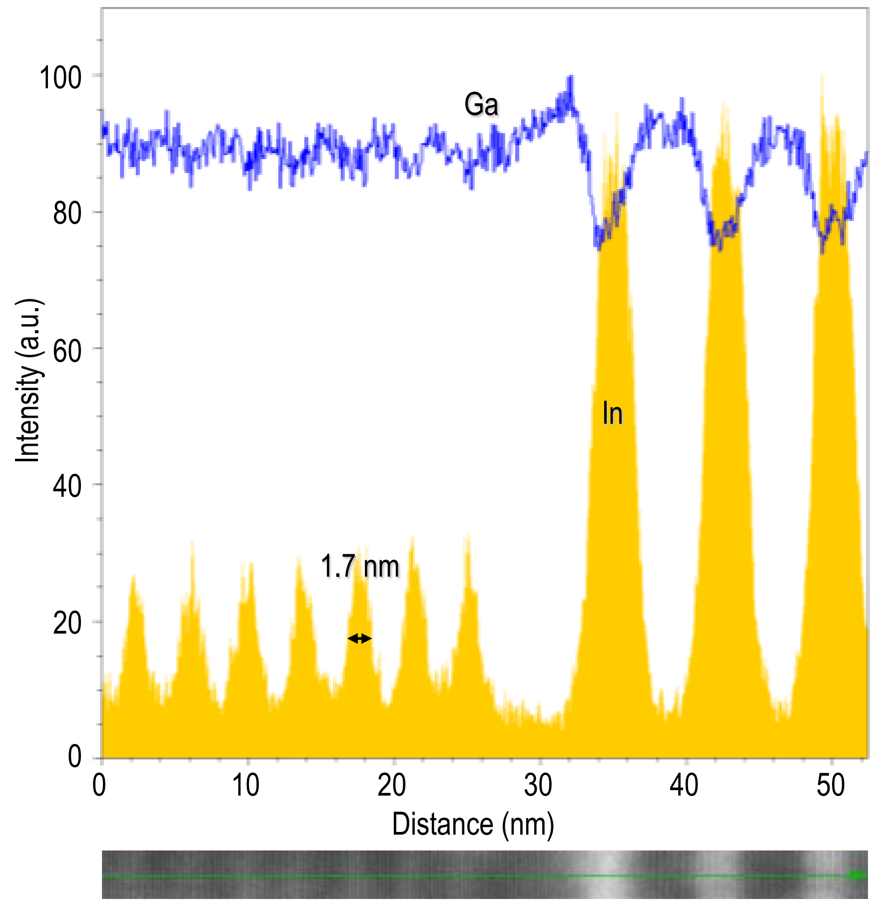
EDS for Chemical Analysis at Nanoscale





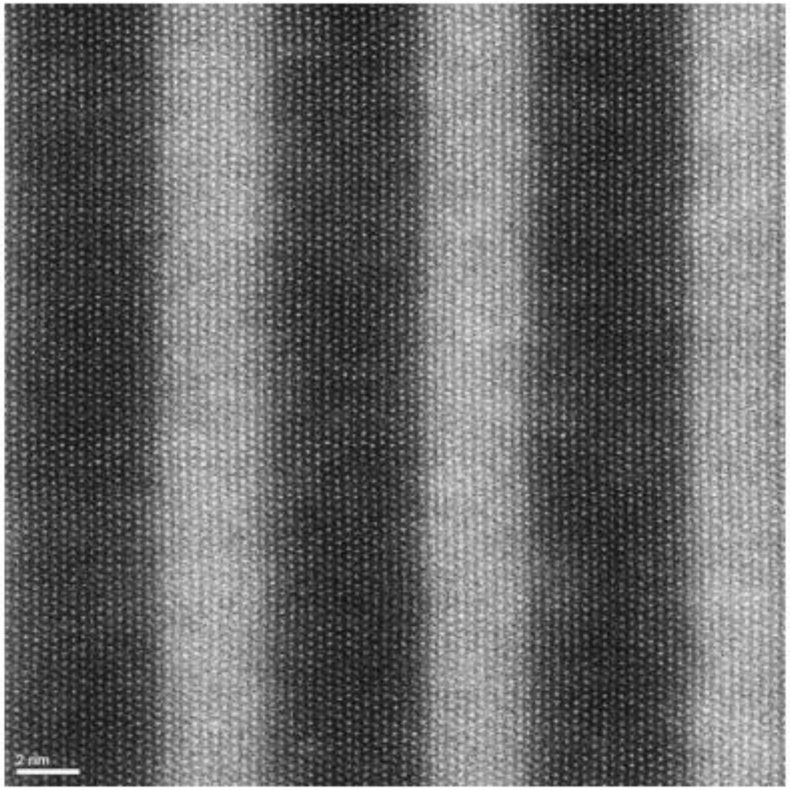
EDS for Chemical Analysis at Nanoscale





EDS QUANTIFICATION - ACCURACY

Application Note: Quantification of Ultrathin Layers by STEM-EDS
http://www.eag.com/documents/quantification-of-ultrathin-layers-by-STEM-EDS-AN472.pdf



RESULTS & DISCUSSION

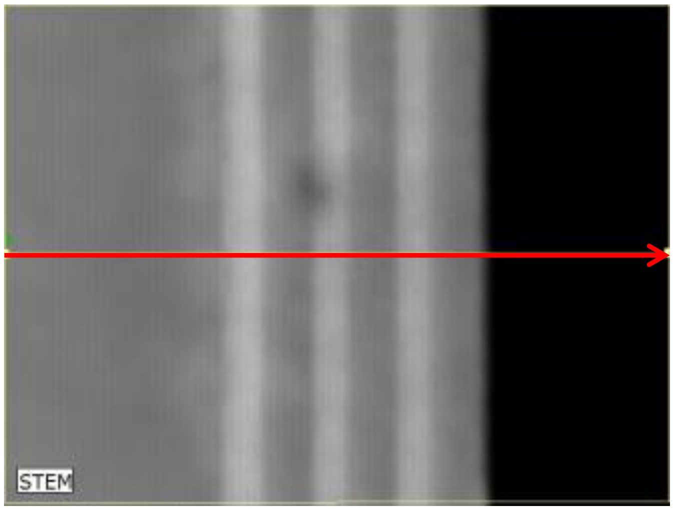
As can be seen in Figure 1, the AC-STEM image provides highly detailed images of the sample. This image clarity is not possible with traditional STEM or TEM images. With image resolution better than 1.5Å, AC-STEM can provide unparalleled layer thickness accuracy and detail.

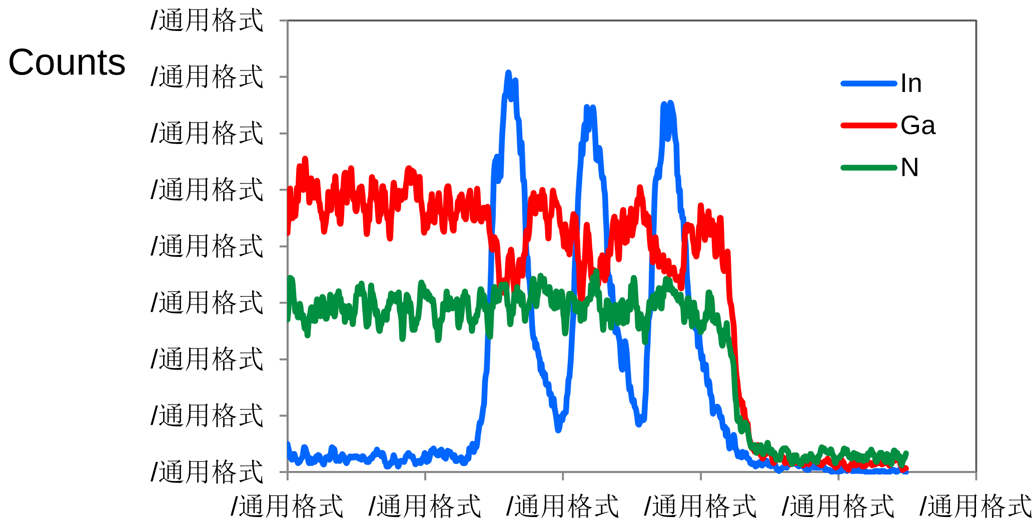
In addition to excellent image quality; the AC-STEM coupled to an advanced x-ray detector allows for reliable quantification of materials. As an example of the accuracy that can be obtained by STEM-EDS, Table 1 shows the results comparing the measured values by STEM-EDS and by Rutherford Backscattering Spectrometry (RBS). RBS is used to create reference standards and is considered to be one of the most accurate analytical techniques for thick films. The agreement between RBS and STEM-EDS is within the expected RBS measurement uncertainty of 5% (relative).

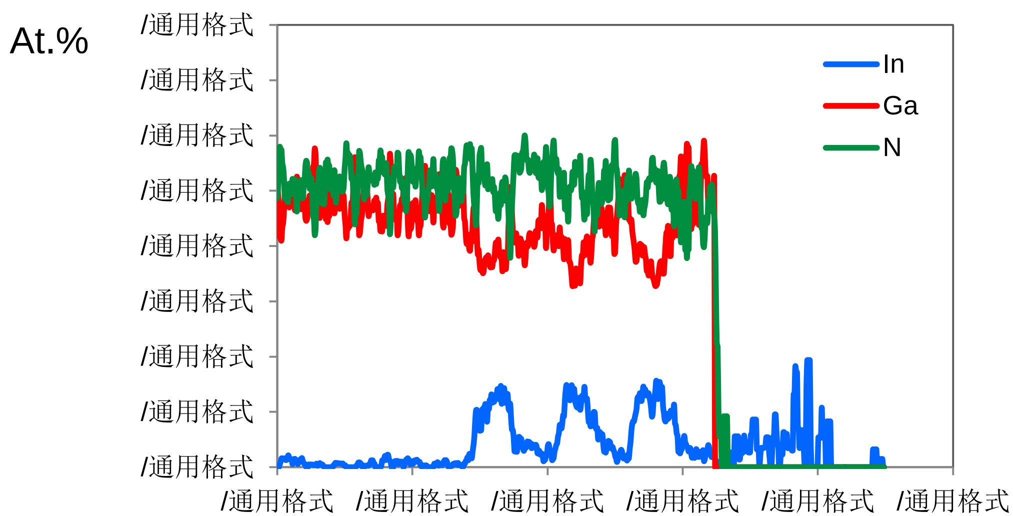
	RBS	AC-STEM-EDS	Relative difference
In concentration	5.3%	5.5%	3.8%

Table 1 In concentration on a InGaN thick film standard sample.

EDS QUANTITATIVE LINESCAN



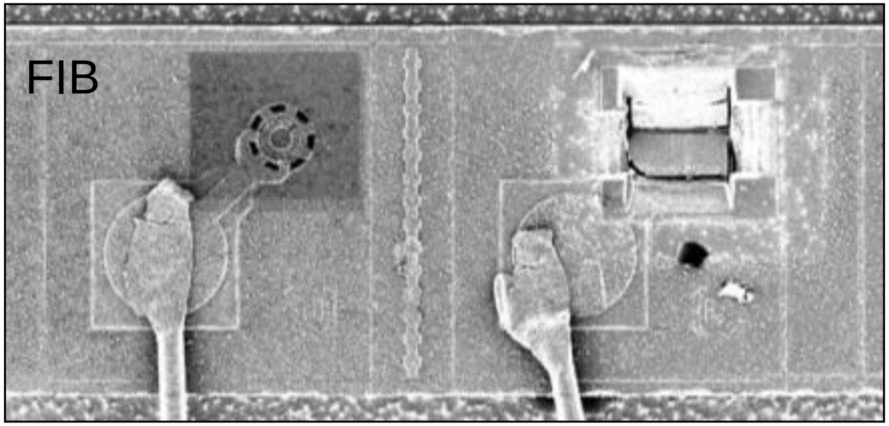


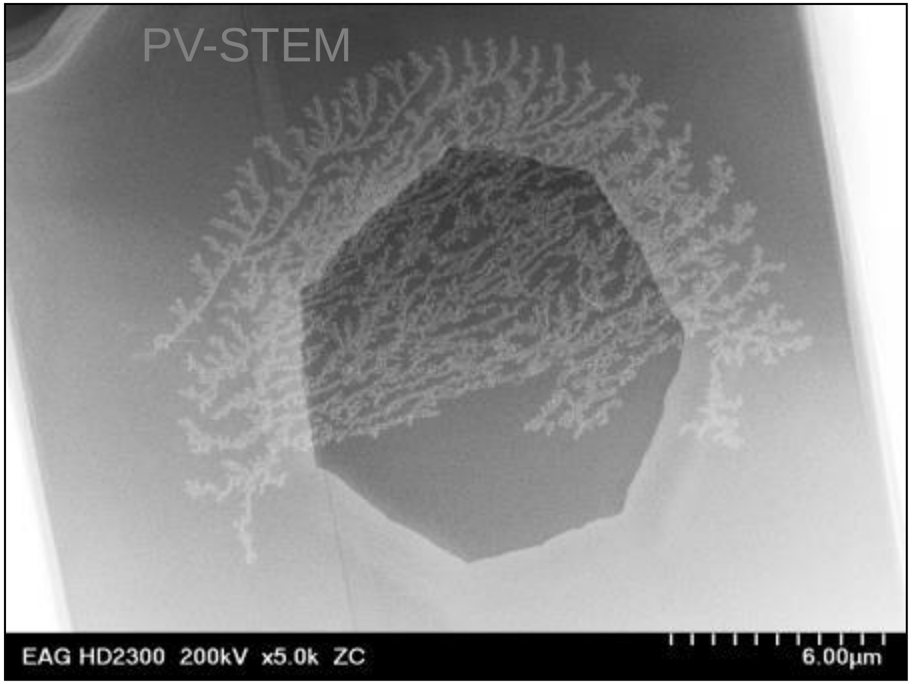


Crystal Defects/Dislocations

with Diffraction Contrast

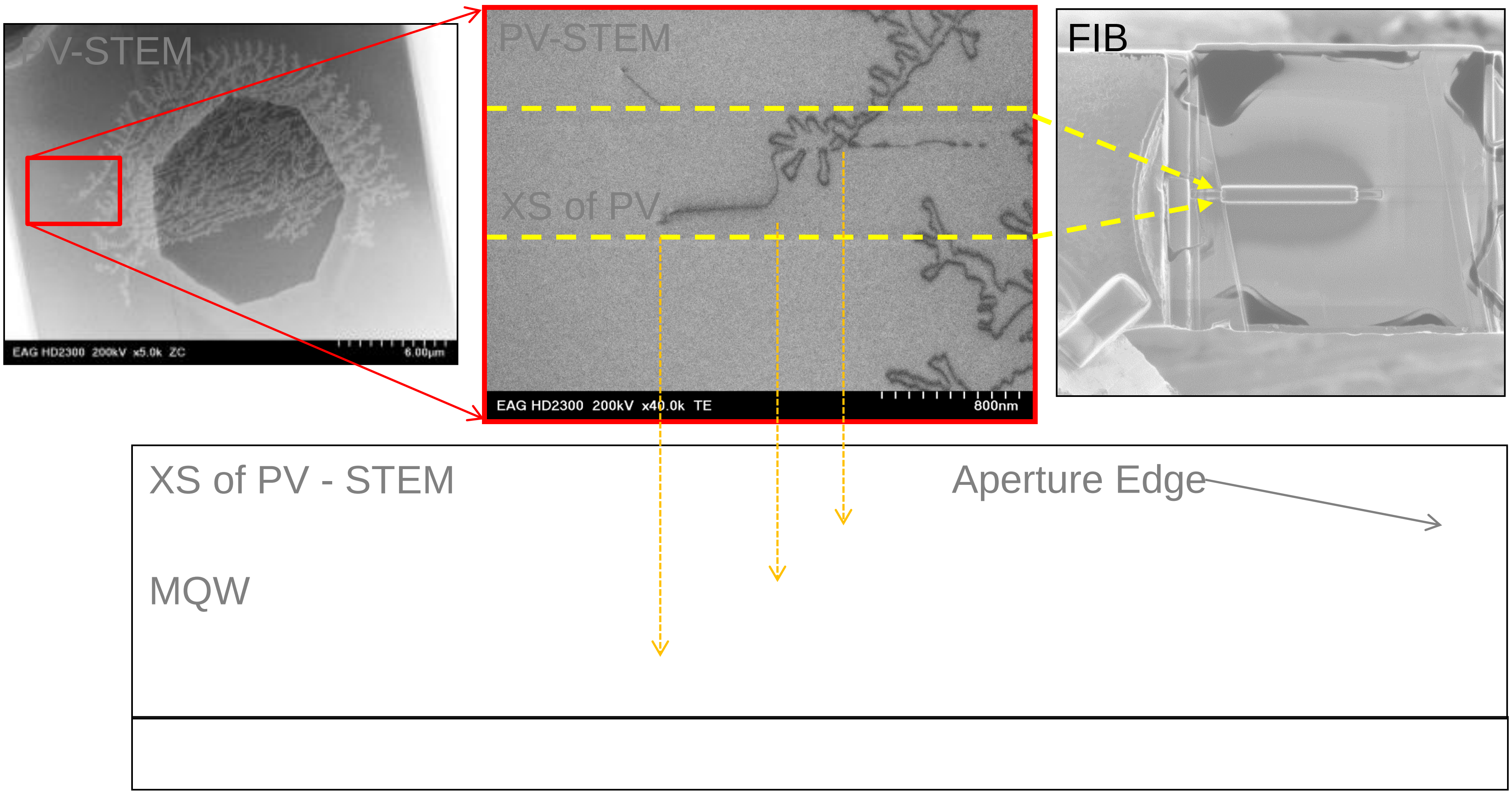
VCSEL Plan View (PV) STEM of Field Failures





- All PV STEM prep is performed using in-situ FIB liftout
- 30um x 30um x 1.5um thick
- Imaged with 200kV STEM
- Thickness: 1-2 periods p-DBR above oxide and 1-2 periods n-DBR below MQW

Targeted VCSEL XS of PV



- XS of PV reveals out-of-plane (OOP) dislocation behavior

EAG LABORATORIES

Catching the Failure Early

Corrosion-Based Failure of Oxide-Aperture VCSELs, Robert W. Herrick, et. al., IEEE JOURNAL OF QUANTUM ELECTRONICS, VOL. 49, NO. 12, DECEMBER 2013

EL

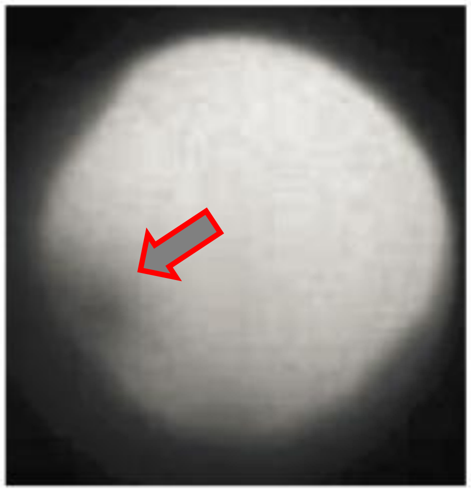
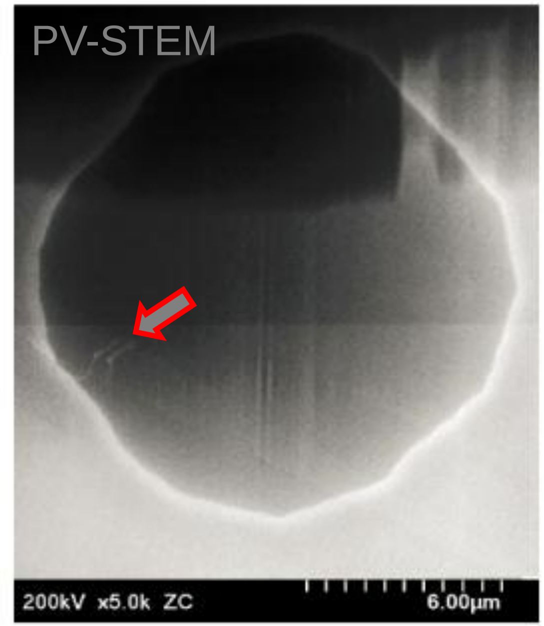
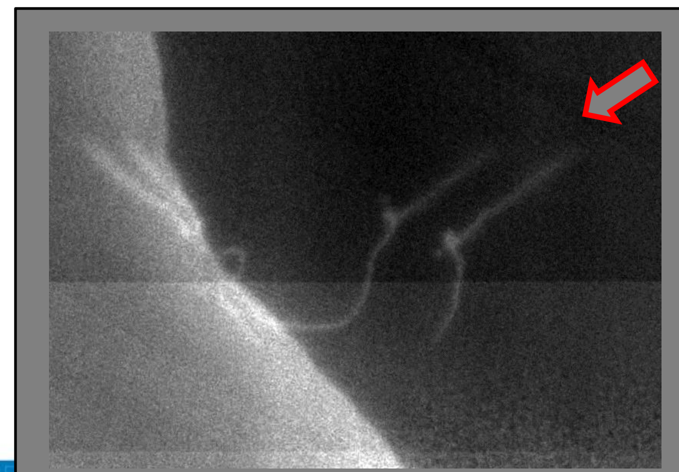


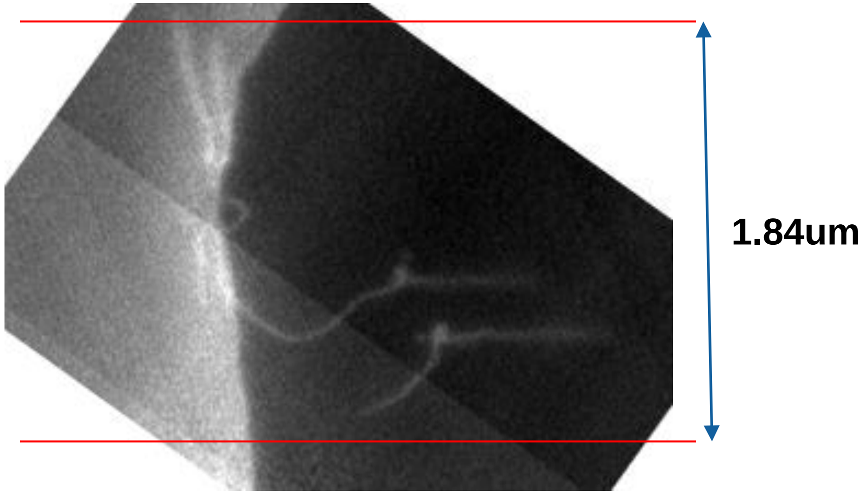
Fig. 7. EL image of partially-degraded VCSEL, imaged at ~1 mA (below threshold). Note dark area at lower left.

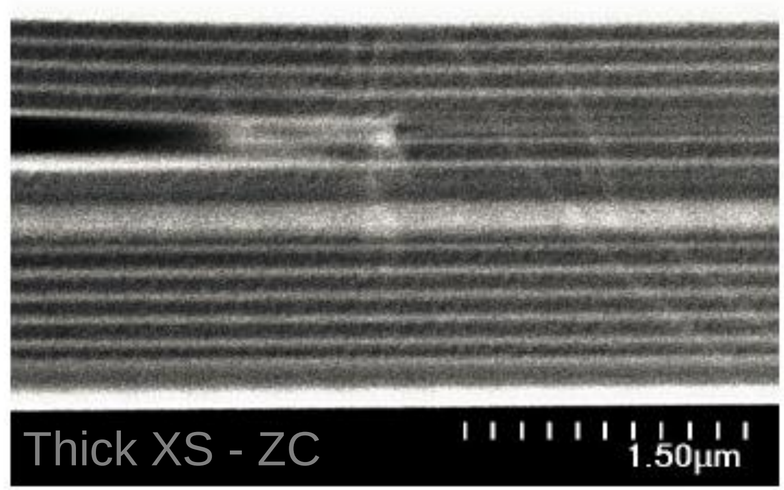


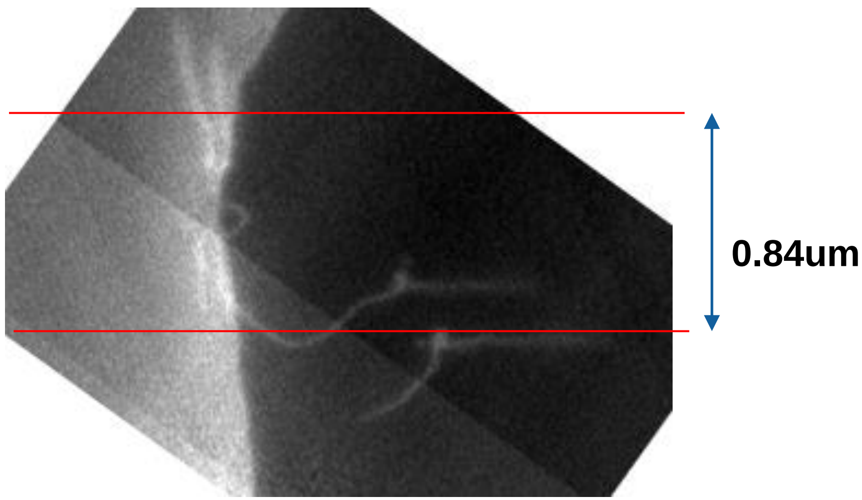


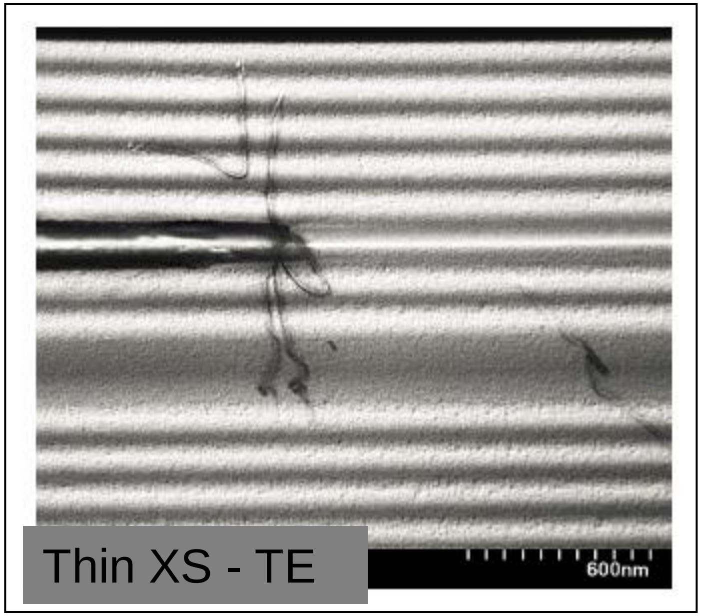
- 26% decrease in output after
  - Aged at 70C 85%RH for 49 days @ 1mA
  - Additional 9 days @ 6mA
- Checked output every 24hrs

Iterative XS of PV









Dislocations grow from the oxide aperture and down into the MQW

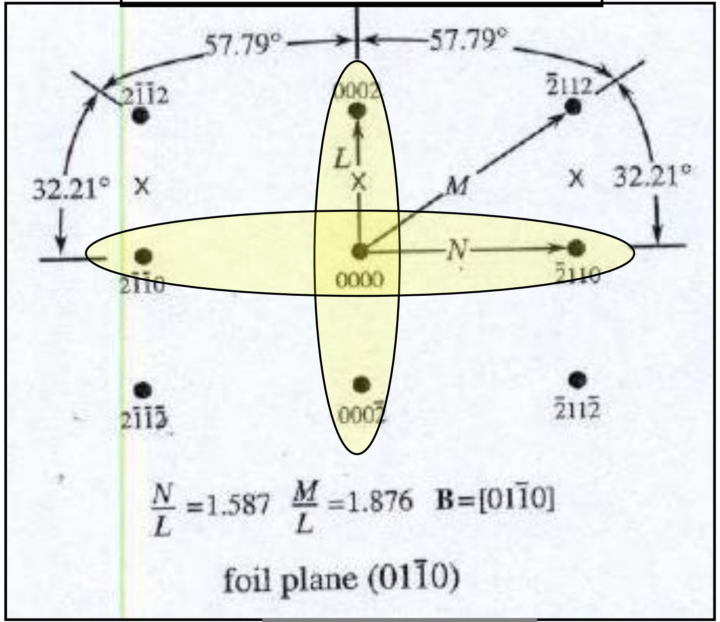
VTD Typing – m-plane GaN

- Reflection vector g = 11-20 & 0002

- Screw dislocations
  - Visible only for g = 0002 (C)
  - Invisible for g = 11-20: |g·b| = 0

- Edge dislocations
  - Visible only for g = 11-20 (A)
  - Invisible for g = 0002: |g·b| = 0

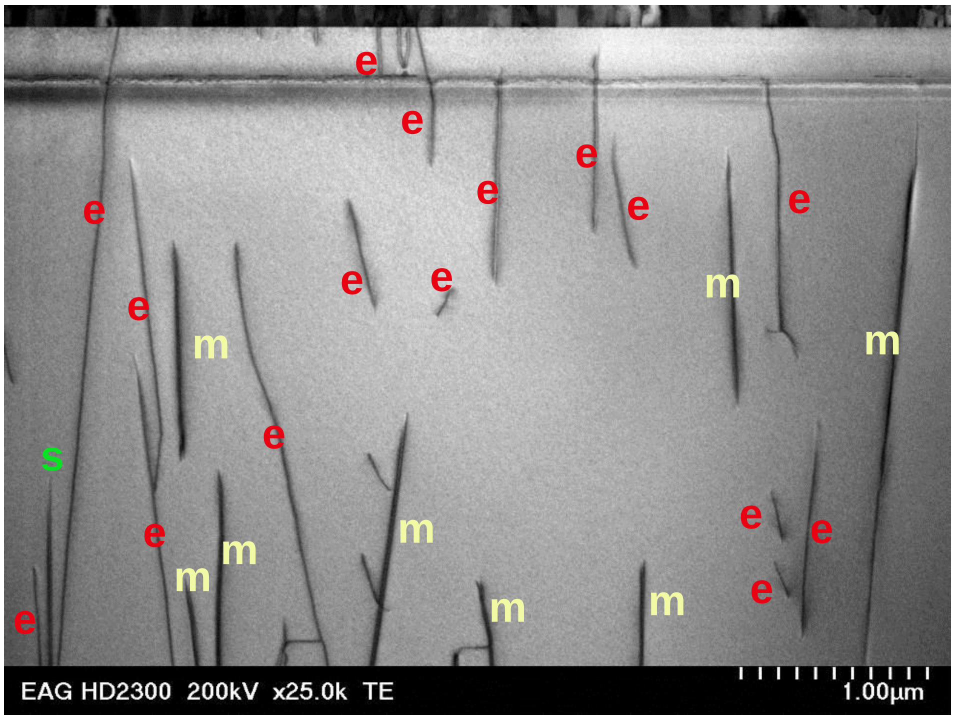
- Mixed dislocations
  - Visible for both reflections

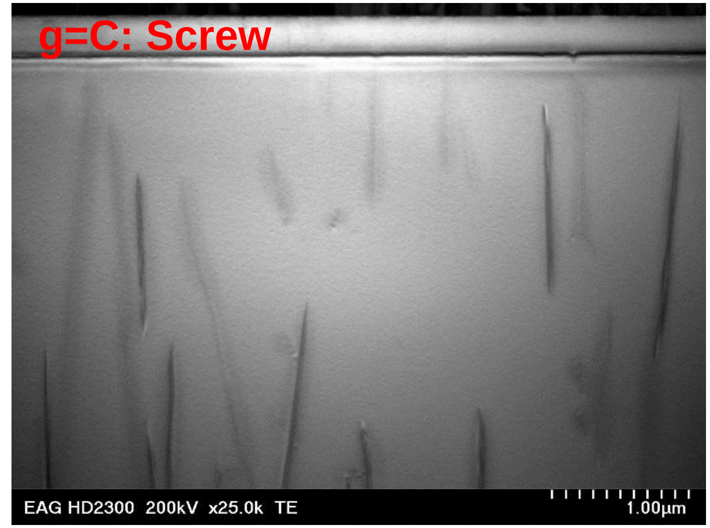


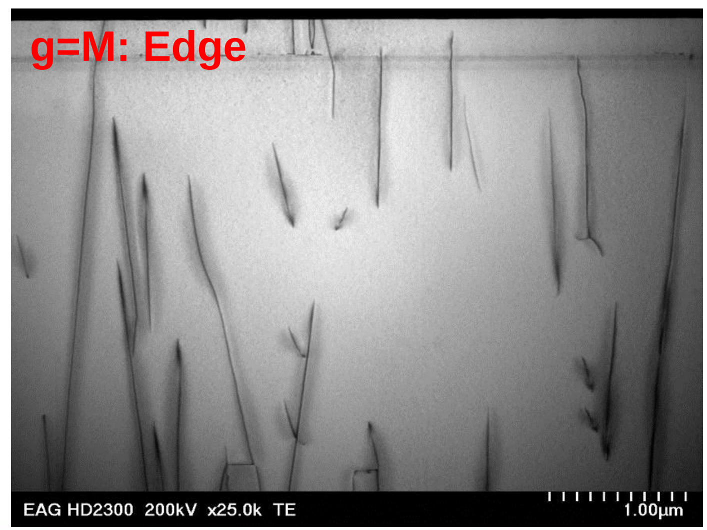
Williams and Carter

VTD Typing – m-plane GaN

On Zone: All dislocations







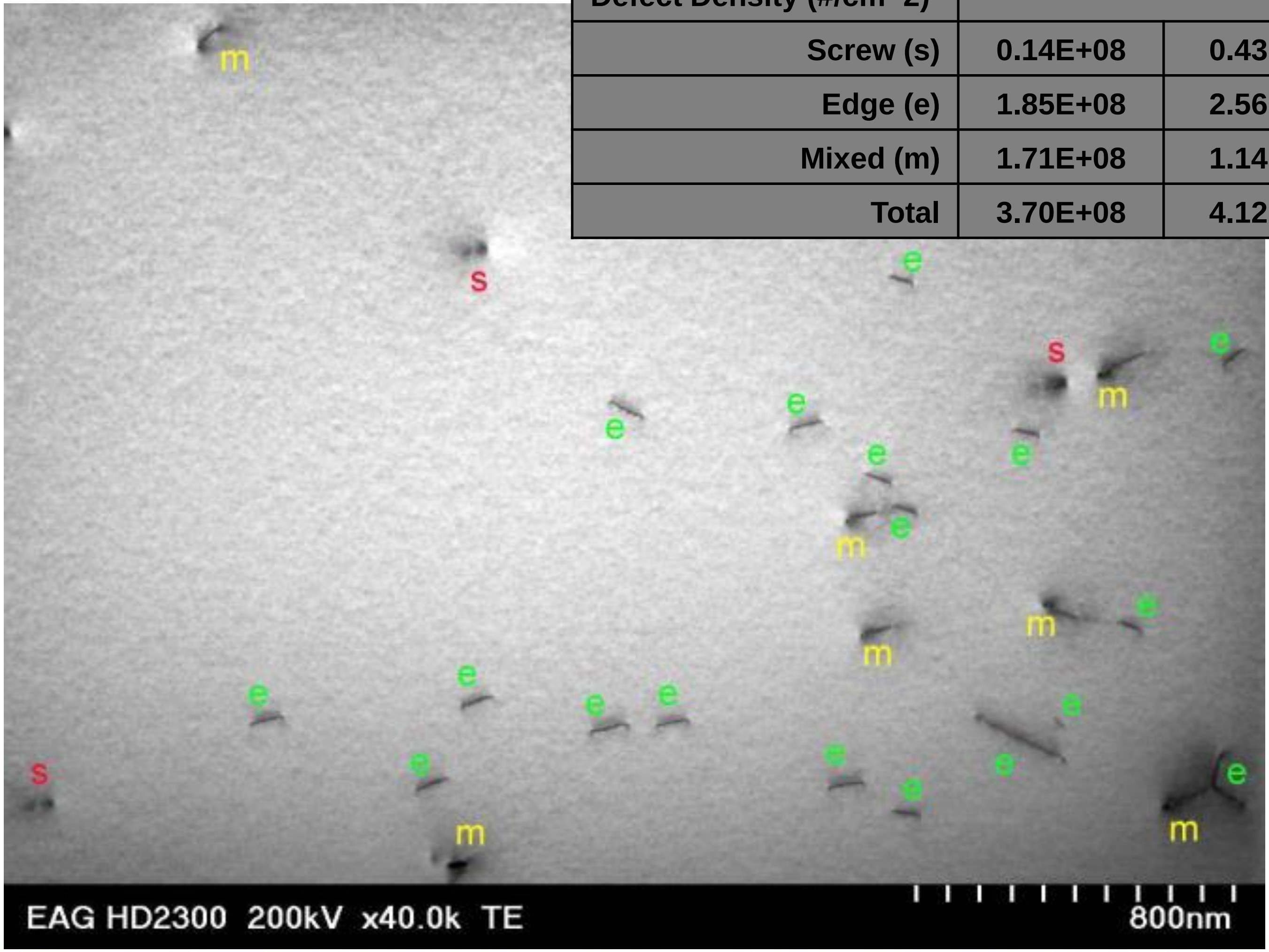
m: mixed dislocation
e: edge dislocation
s: screw dislocation

EAG LABORATORIES

GaN Dislocation Density and Typing (PV)

Image	m01_Typed	m02_Typed	Average
Defect Density (#/cm^2)			
Screw (s)	0.14E+08	0.43E+08	0.28E+08
Edge (e)	1.85E+08	2.56E+08	2.20E+08
Mixed (m)	1.71E+08	1.14E+08	1.42E+08
Total	3.70E+08	4.12E+08	3.91E+08

Z=[0001], g=(11-20)

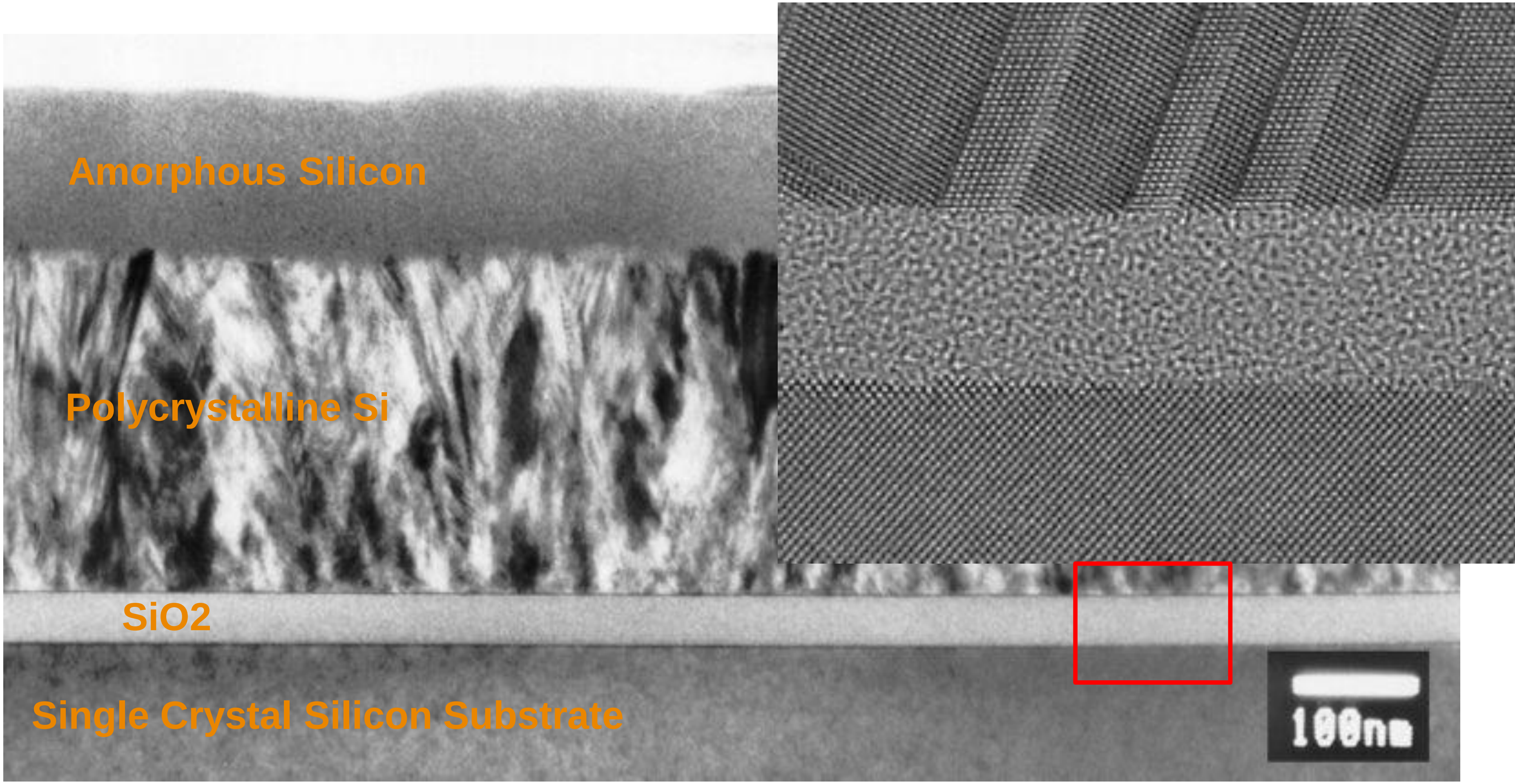


EAG LABORATORIES

Crystal Orientation/Structure

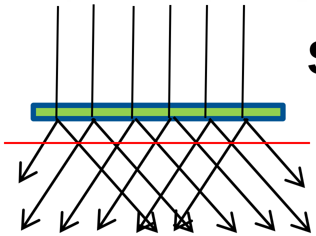
with Phase Contrast, Atomic Resolution Imaging, Electron Diffraction (SAD, NBD, CBED,FFT)

PHASE CONTRAST: ALL PHASES OF SILICON

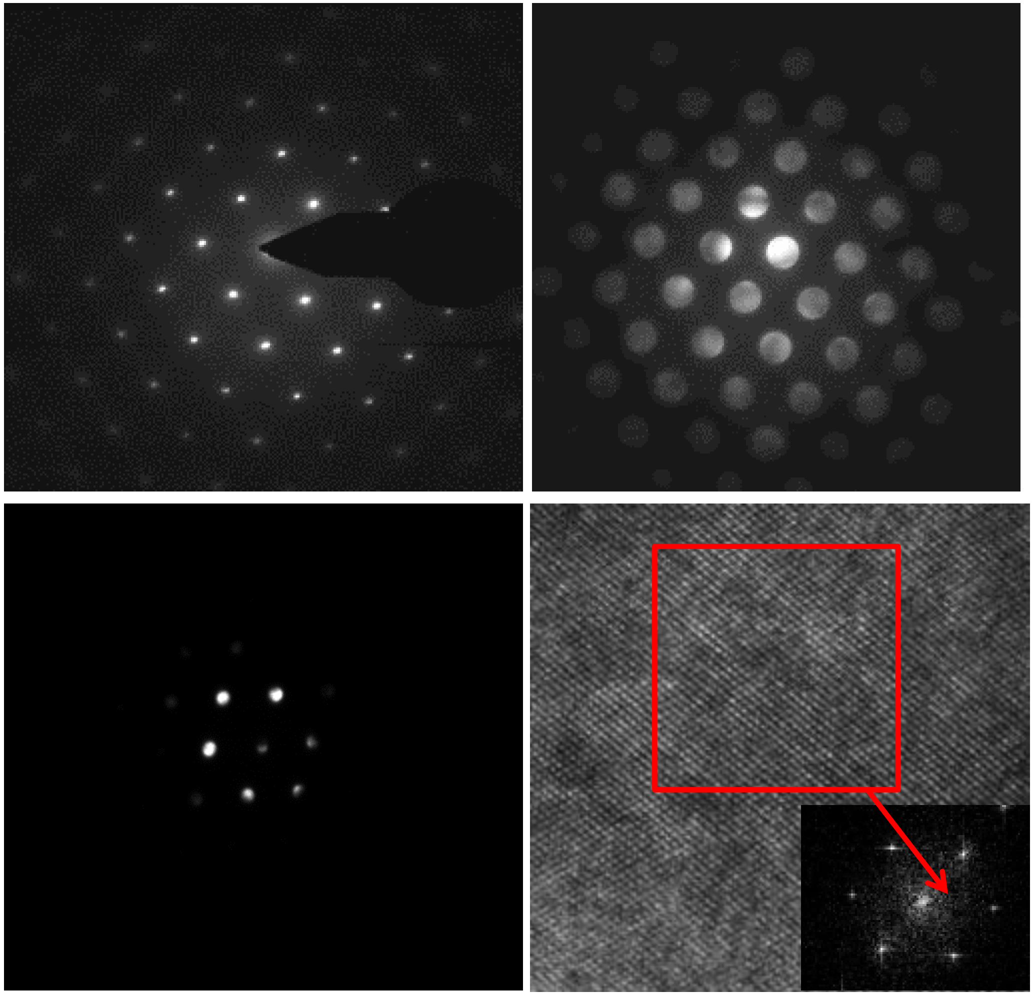


Modes of Electron Diffraction

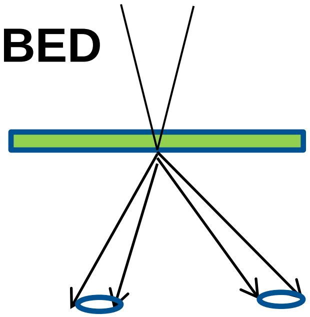
SAD



- Parallel, broad beam
- Spot or ring patterns
- micron-sized areas

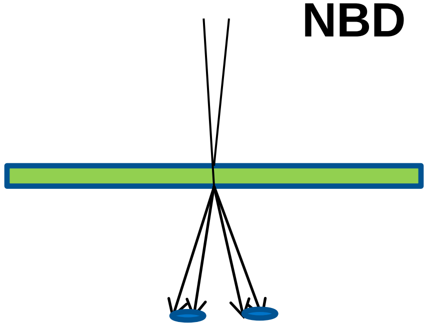


CBED



- Convergent beam
- Disc pattern
- Tens of nanometers

NBD



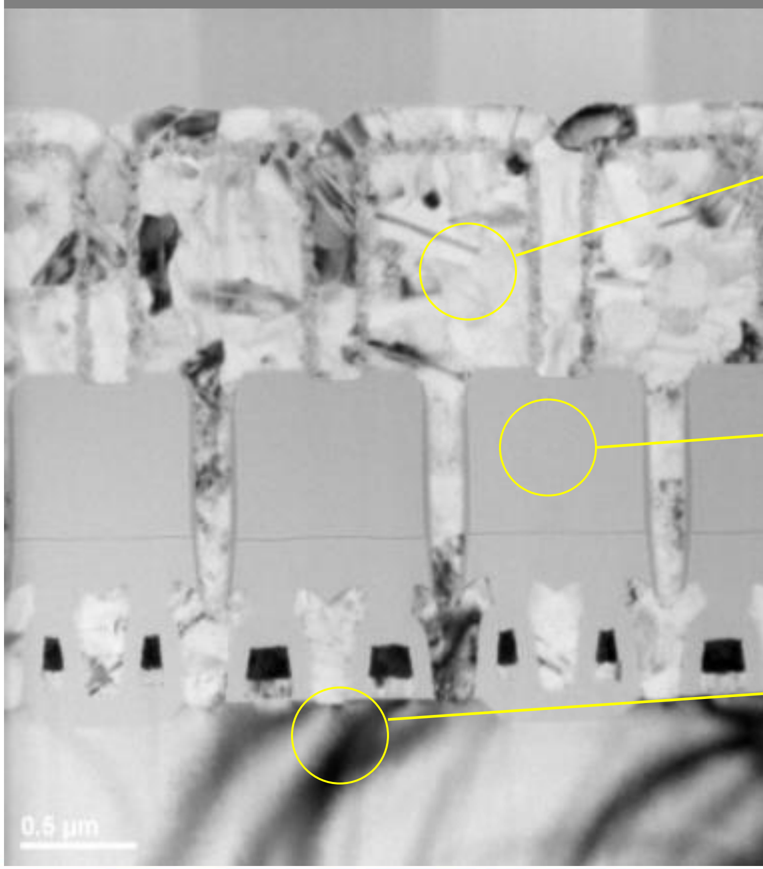
- Parallel Pencil beam
- Small discs
- Nanometer size areas

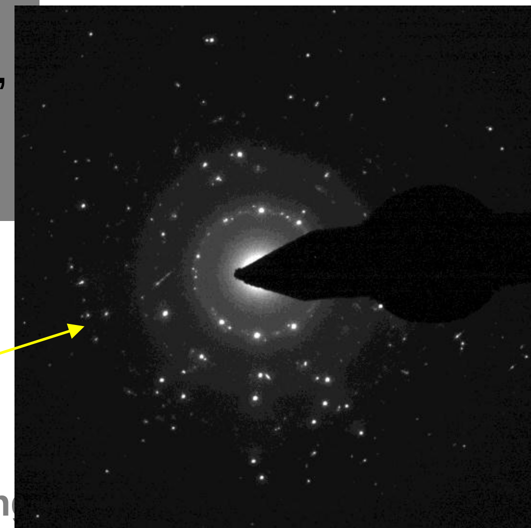
FFT

- Parallel beam
- Spot pattern
- ~2nm area

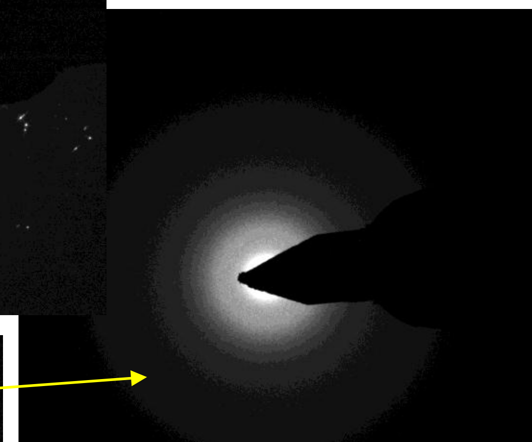
SAD: Selective Area Diffraction

- For large area (>200nm) analysis
- Crystallinity of material (single crystal, poly-crystalline or amorphous)
- Orientation of a single crystal

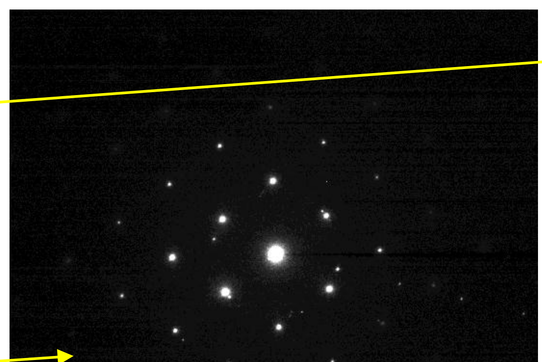




Ring



Diffuse pattern from amorphous SiO2



Single crystal pattern from Si substrate
Identified to be in 110 orientation

NBD – NANO-BEAM DIFFRACTION

Structure and Crystallinity information from very small regions - sub 10nm!

On

Off

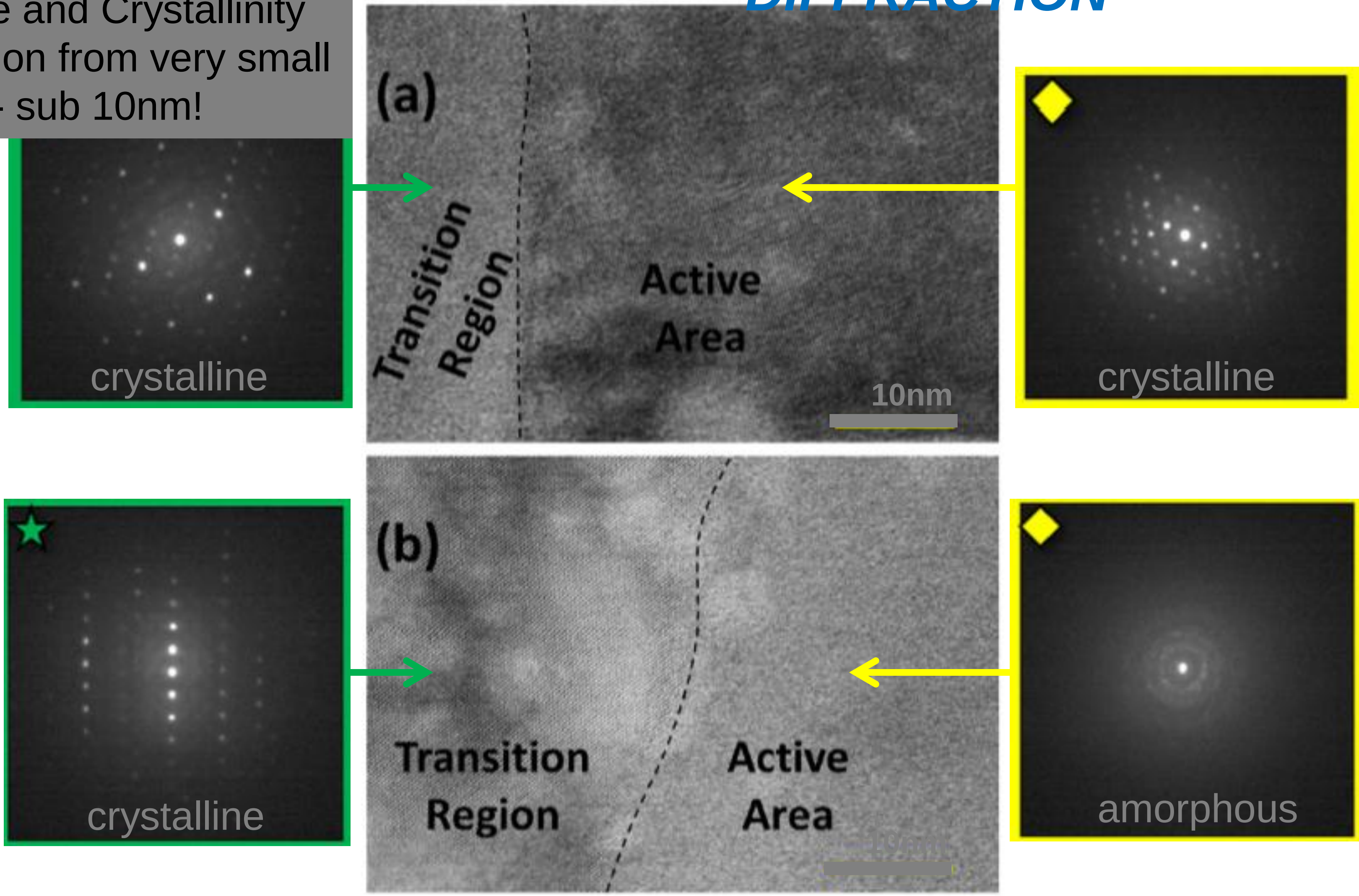
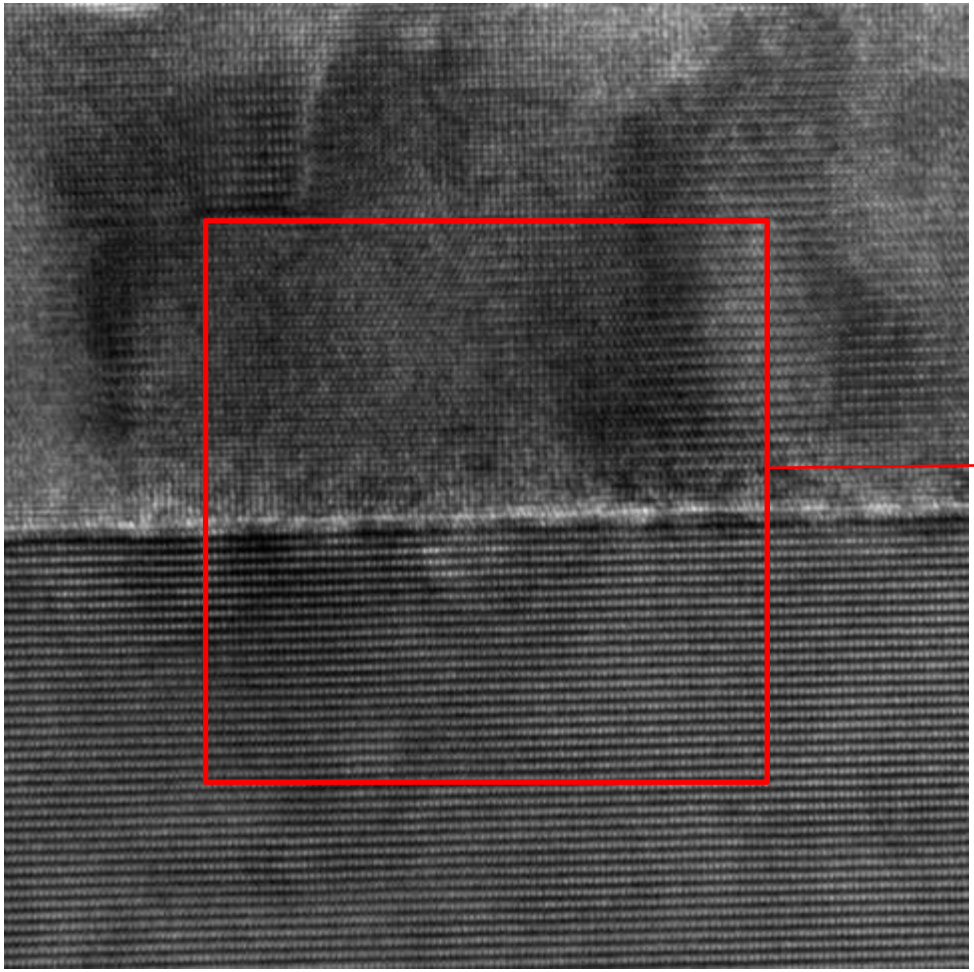
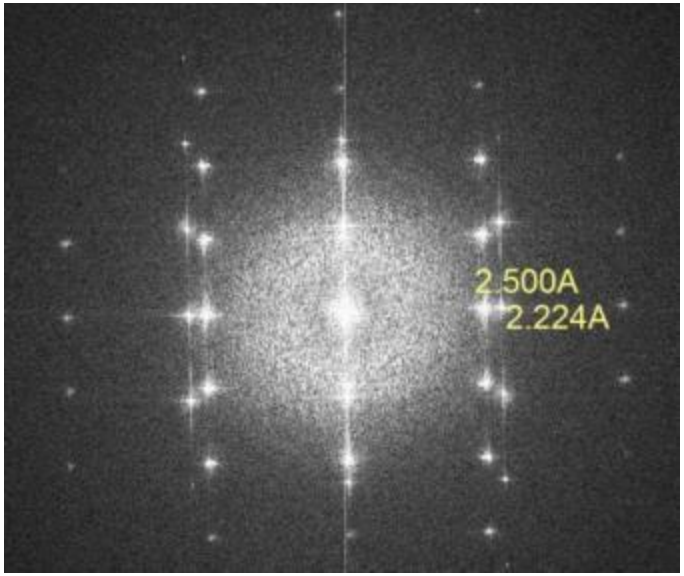


FIG. 7. STEM images of the interface between the active area and transition region for a device in the (a) ON and (b) OFF state. Scale bars 10 nm.

Using FFT's on High Resolution Images





EAG INSTRUMENTATION

样品制备

- Focused Ion Beam
  - 8 FEI dual beam FIBs, 8+ single beam FIBs
  - In-situ Lift Out TEM preparation
- Other TEM Preparation
  - Dimple and ion mill
  - Mechanical and ion mill

Electron Microscopes

- Transmission Electron Microscopy
  - Topcon 002B 200kV TEM
  - Hitachi HF2000 200kV TEM
- Scanning Transmission Electron Microscopy
  - Hitachi HD-2300 Schottky field emission with EDS
  - Hitachi HD-2700, Hitachi Cs corrector, Bruker QUANTX EDS
- Scanning/Transmission Electron Microscopy
  - Three FEI Tecnai TEM/STEM with EDS and EELS

总 结

- 优点
  - –极好的成像和分析分辨率.
  - –很多成像机制.
  - –可以连接很多传感器
- 局限性
  - –样品制备要花很长时间
  - –不是所有的样品适合做TEM或制备TEM样品.

S/TEM能提供最高的图象分辨率和元素识别