EBSD Measurement of Strains in GaAs due to Oxidation of Buried AlGaAs Layers

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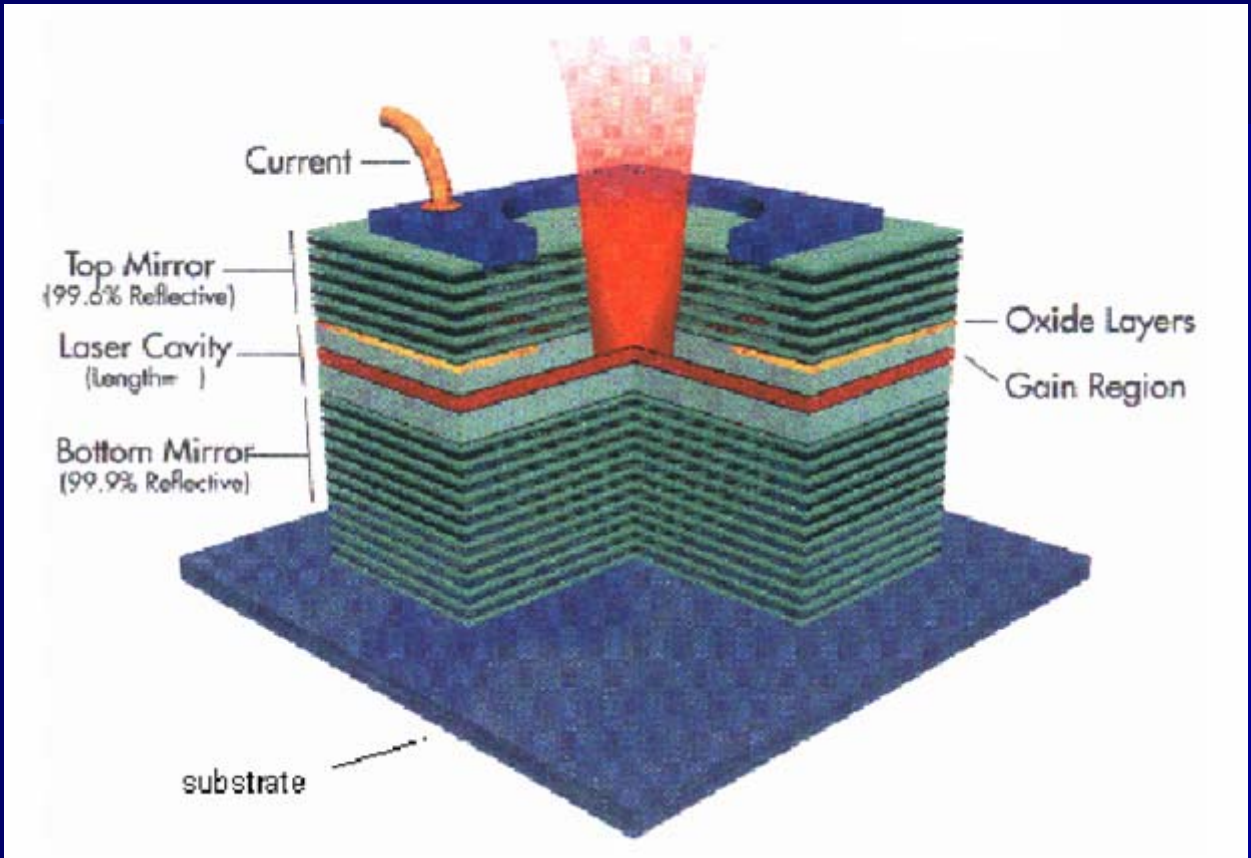
Outline

- Buried oxide layers in AlGaAs/GaAs heterostructures
- The reliability concern – very large elastic strains!
- The need for strain mapping
- Electron backscatter diffraction for strain mapping
- Results – elastic strain fields
- Summary

ASME Summer Meeting – Applied Mechanics and Materials, Scottsdale, AZ, June 2003

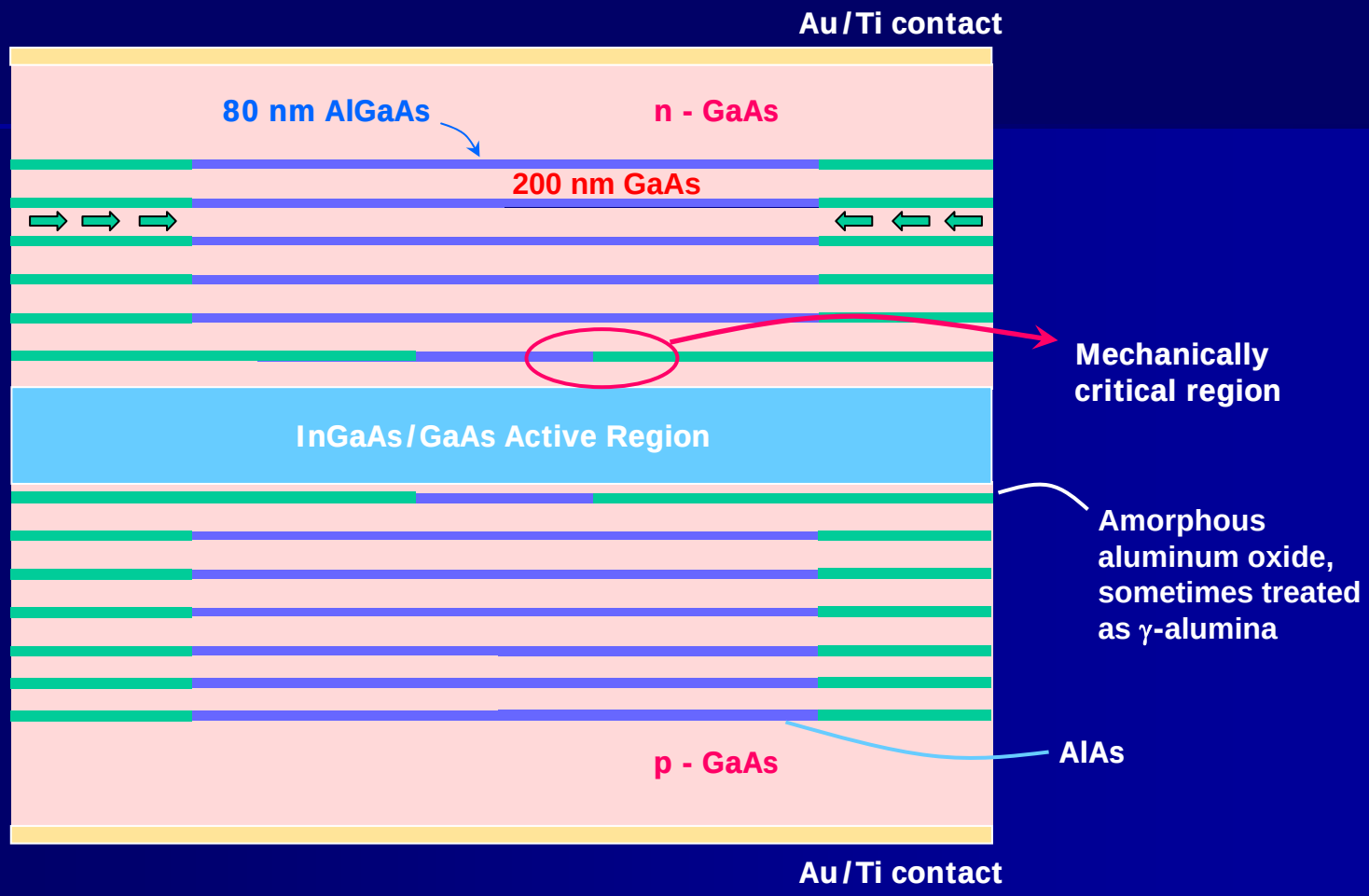
Lateral Oxide Confined VCSELs



From: III-Vs Review 14, 45 (2001).

Lateral Oxide-Confined VCSEL

460°C wet thermal oxidation proceeds from edge of wafer



Oxide Interfaces: Sites of Reliability Threats

$$2\ AlAs + 6\ H_2O_{(g)} = Al_2O_3 + As_2O_{3(l)} + 6\ H_2$$
$$As_2O_{3(l)} + 3\ H_2 = 2\ As\ (\uparrow) + 3\ H_2O_{(g)}$$

Accompanying the AlAs to alumina transformation is a volume contraction of the resulting material.

Example, for γ-Al_2O_3:

AlAs:

zincblende, a_o = 0.5636 nm,

4 Al atoms/cell

Volume/Al atom = 0.0448 nm^3

→ Al atom spacing = 0.355 nm

γ-Al_2O_3:

deficient cubic spinel, a_o = 0.790 nm,

21 Al atoms/cell

Volume/Al atom = 0.0235 nm^3

→ Al atom spacing = 0.286 nm

Expect alumina layers to contract by ~ 20% (!)

DELAMINATION is observed

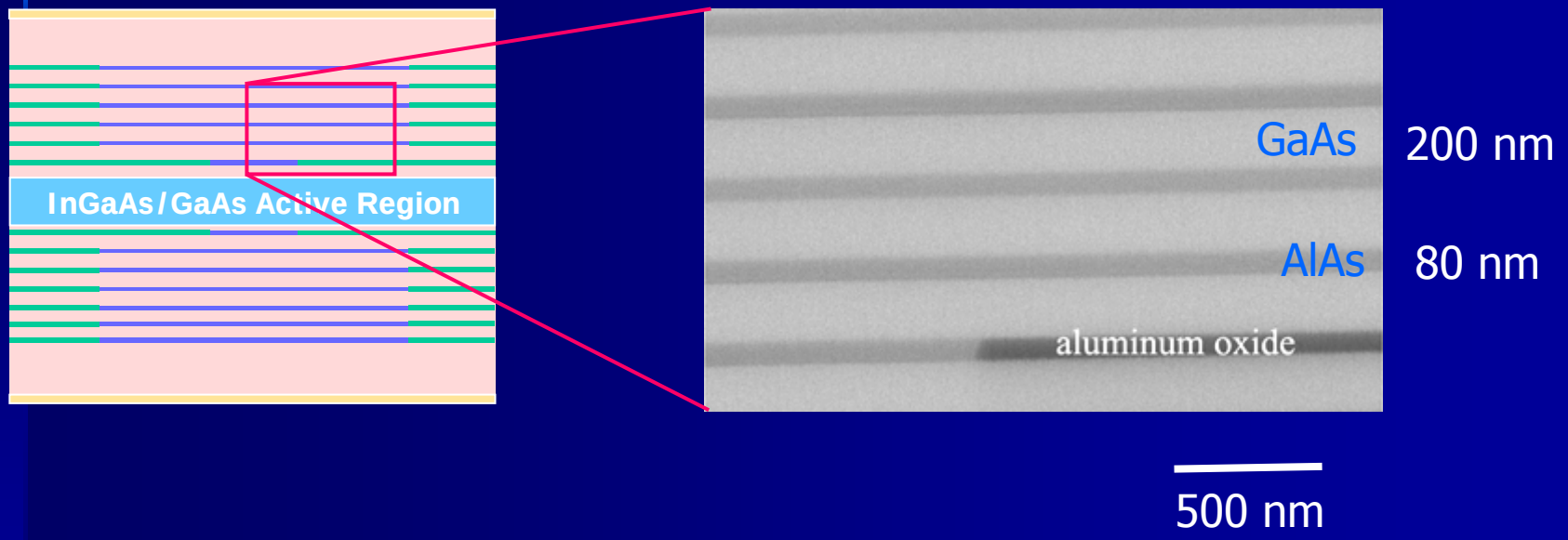
Oxide Interfaces: Sites of Reliability Threats

- Note that the actual aluminum oxide formed in this structure is an amorphous $(Al_xGa_{1-x})_2O_3$, which is nearly all Al-based.
- Properties of this oxide are unknown, so we consider γ-Al_2O_3 as an approximation.
  - Amorphous oxide likely less dense;
  - TEM observations show ~ 7% linear contraction, in the constrained condition.

  - NOTE: high energy (> 100 keV) electron beam has been shown to transform oxide from amorphous to nanocrystalline.

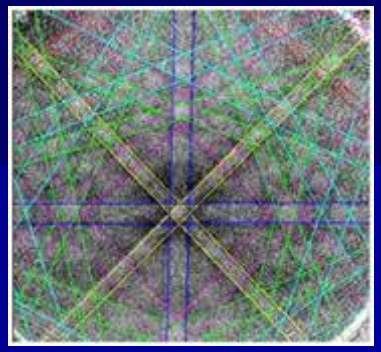
Strain Determination

- Effort underway to reduce effect of strains, through compositional control during MBE growth
- Accompanied by metrology to measure strains



To MAP elastic strain, need a high spatial resolution method for measuring strain.

EBSD for Elastic Strain Measurement



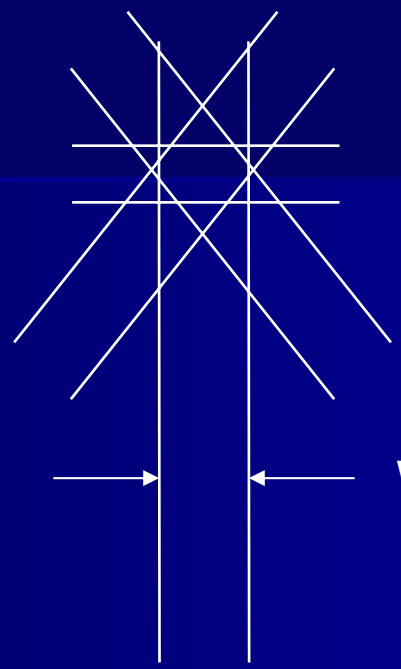
Spatial resolution sufficient (more on this later)

Two effects of elastic strain field on EBSD patterns:

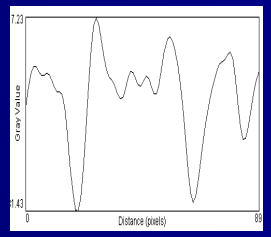
- Elastic strain
  - Kikuchi band width ∝ 1/(strain)
  - In principle, can extract individual strain components
- Elastic strain with gradients
  - Kikuchi band (edge) broadening ∝ strain gradient
  - Pattern sharpness/ "image quality" parameter describes intensity distributions and overall broadening of all bands in pattern

Elastic Strain and EBSD Band Width

- Band width can be measured by intensity profiling, and an interplanar spacing directly determined.
- Need a reference crystal for absolute determinations.
- Relative changes measurable to ~ 0.1 to 0.2% strain.



$W = 2 \cdot \theta_{hkl}$



$\theta_{hkl} = \arcsin(n\lambda/2d_{hkl})$

$$\mathbf{d_{hkl} = \lambda / 2 \cdot \sin\theta_{hkl}}$$

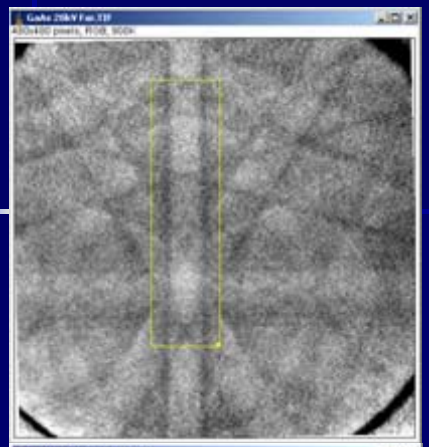
Get λ from reference crystal.

Uncertainties:

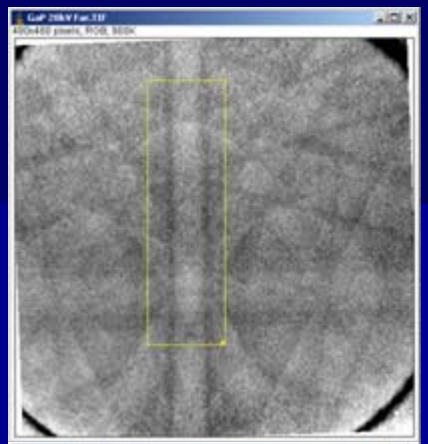
- Detector resolution
- Bands are really hyperbolas, i.e. curvature present
- Beam coherency (field emission best)

$\varepsilon \rightarrow \theta_{hkl}$(unstrained) becomes θ_{hkl} (strained), i.e. a discrete change in θ_{hkl}

EBSD Band Width Measurements



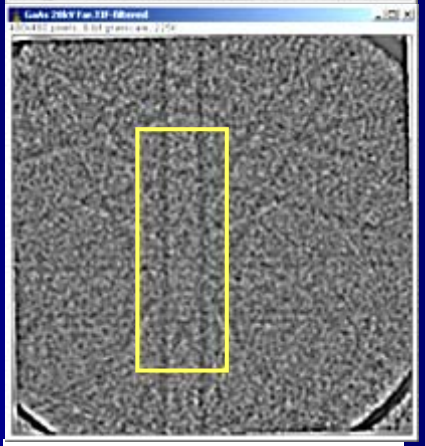
Patterns, as collected, background-subtracted



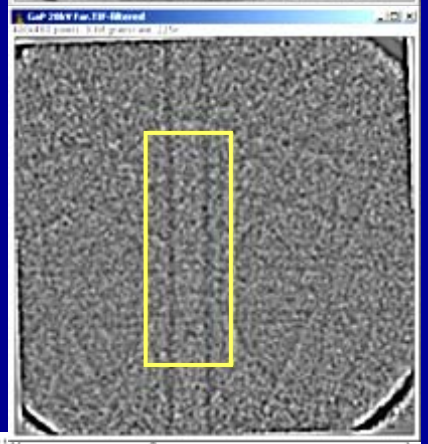
GaAs
a_o=0.5653 nm

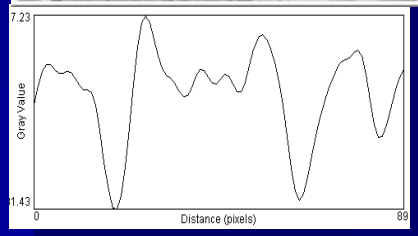
GaP
a_o=0.5451 nm

Δ = 3.6%

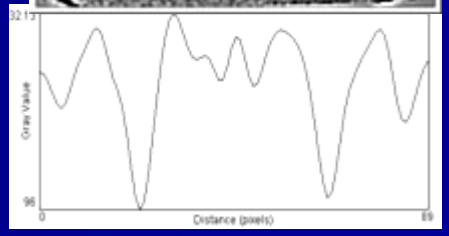


Patterns, FFT filtered, i.e. pseudo high-frequency filtered

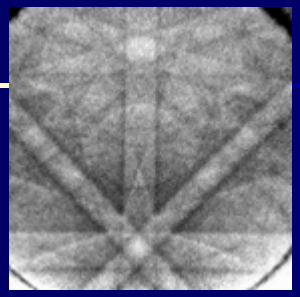


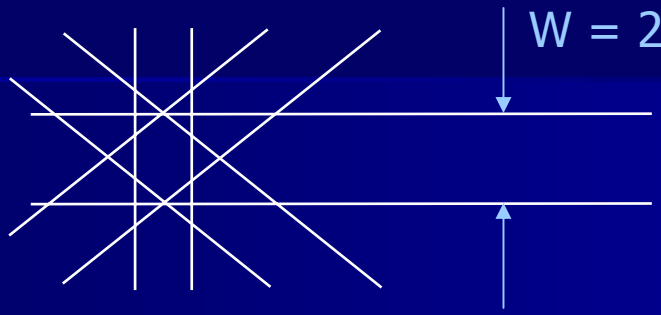


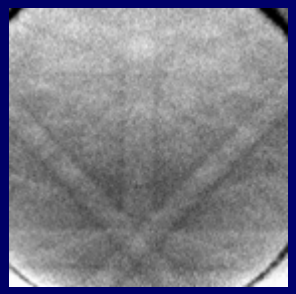
Intensity profiles
Δ = 3.4%

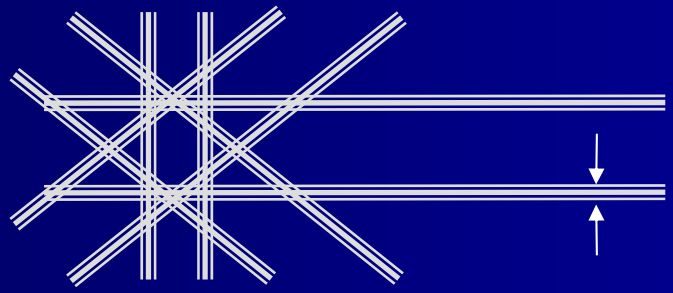


Elastic Strain Gradients and EBSD Kikuchi Band Edges









$\Delta W = 2 \cdot \Delta\theta_{hkl}$

Strain gradient: variation in d(hkl), leading to $\Delta\theta_{hkl}$.

<u>2 effects:</u>

- line edges become diffuse
- maximum band intensity decreases

Note that we are considering strictly the material volume sampled by the electron beam.

Image Quality Parameter

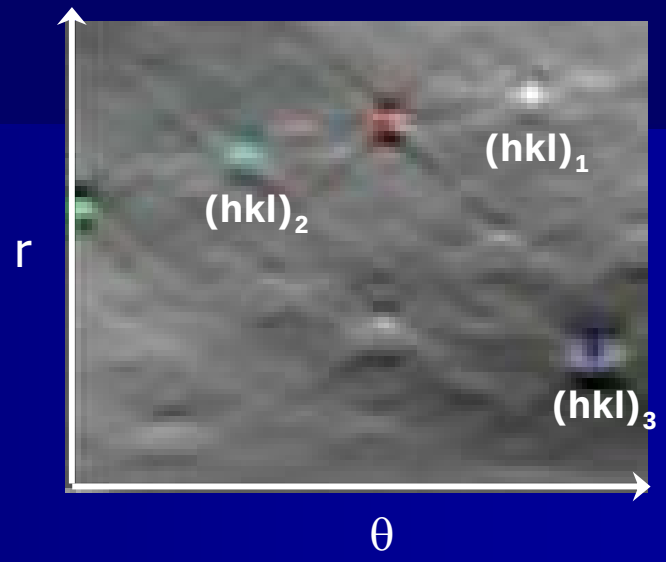
Image quality parameter:

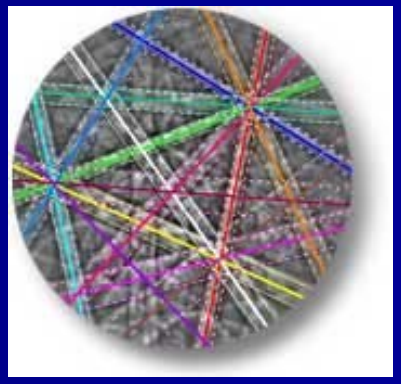
IQ ≡ Σ (max. Hough peak intensities) for many (hkl)

Σ (pixel intensities in one (hkl) Hough peak) ∝ Σ (pixel intensities in one Kikuchi band)

(Note: this is different from max. Hough intensity)

Image quality is a measure of Kikuchi band intensities





Images from http://www.edax.com/technology/EBSD/OIM/intro3.html
Disclaimer: use of any product or company names does not constitute an endorsement of the product or company by NIST or the U.S. government.

Strain Gradients and IQ Parameter

- For a given number of electrons contributing to a Kikuchi band, the maximum intensity of the band decreases if the band edges become broadened due to a strain gradient.
  - Stronger gradients should decrease average intensity more than weaker gradients, *even for the same overall strain.*
  - Anticipate different effects for gradients mostly in tension versus those mostly in compression.
  - Measurable?
- Therefore, the IQ parameter should be directly correlated to the severity of the elastic strain gradient within the material volume sampled by the electron beam.

EBSD Image Quality Mapping of Strain Fields

- IQ map shows contrast based on IQ parameter.
  - Darker pixels: more diffuse patterns
- Consistently observe distortion region about the oxide growth front.
  - Elongated in direction of oxide layer
  - Extent: ~ 1-2 μm in either direction of front.

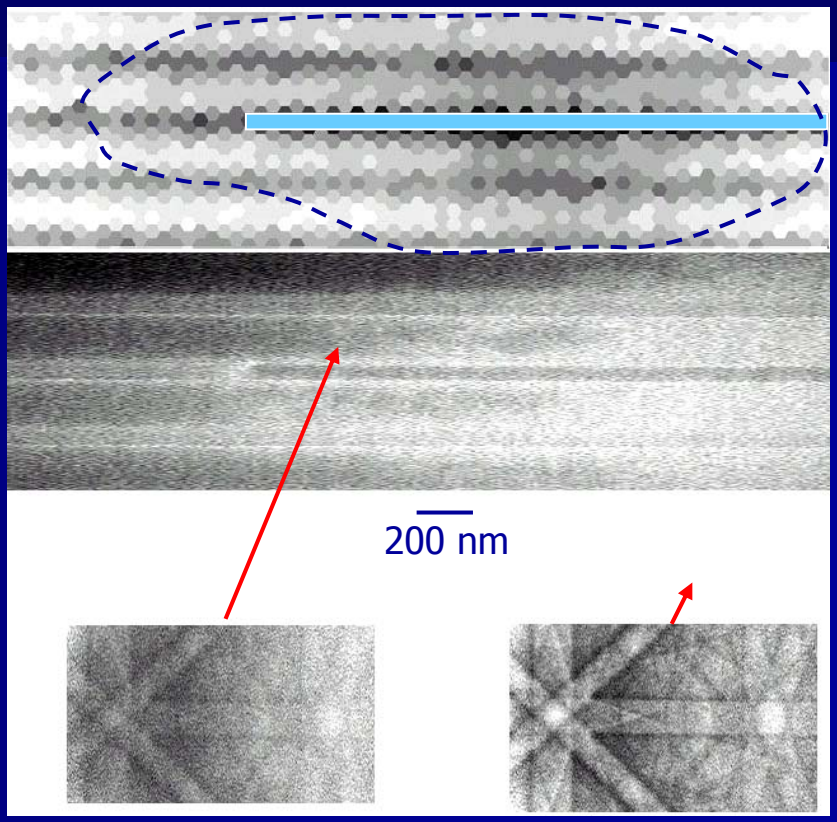
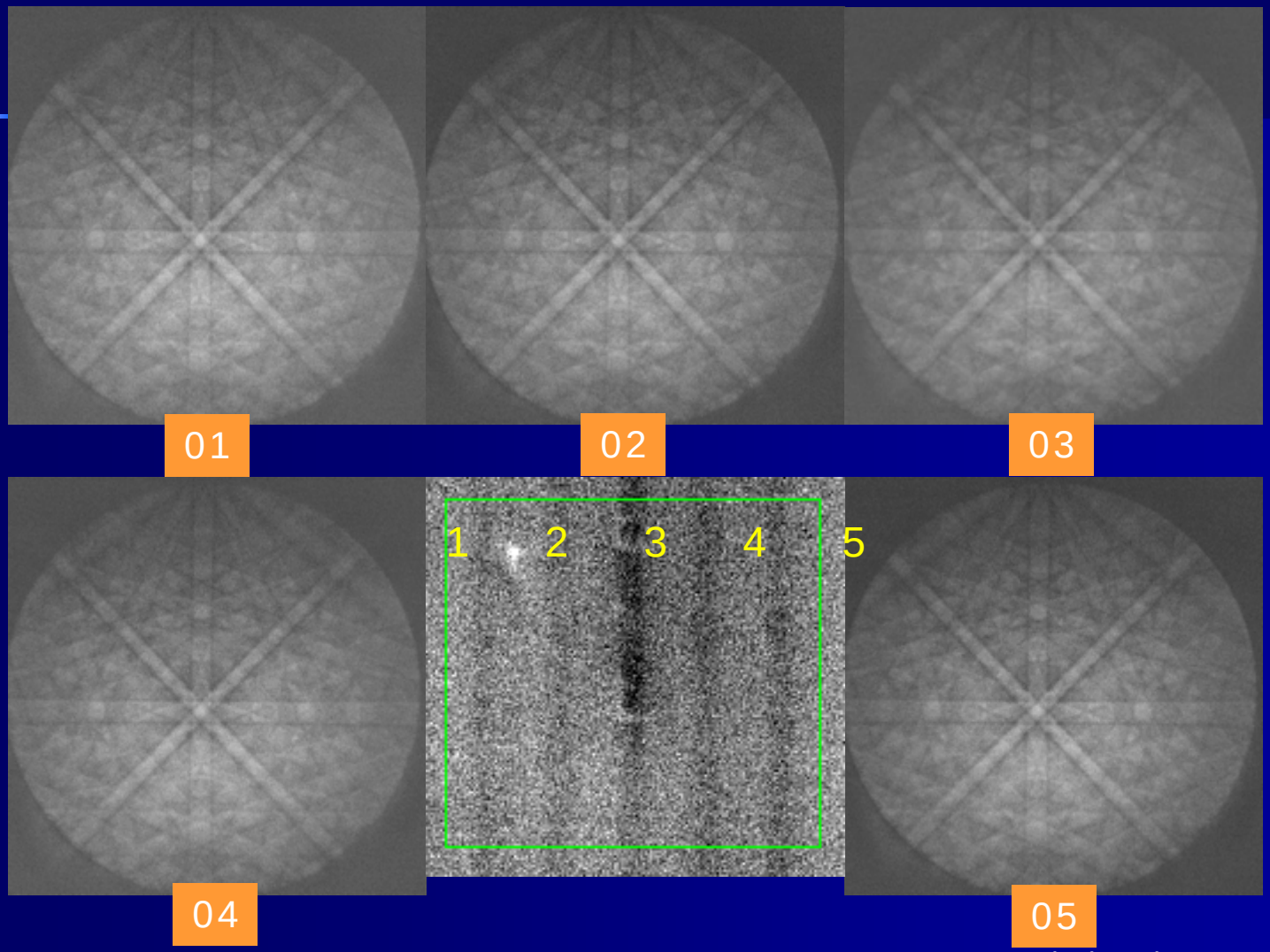
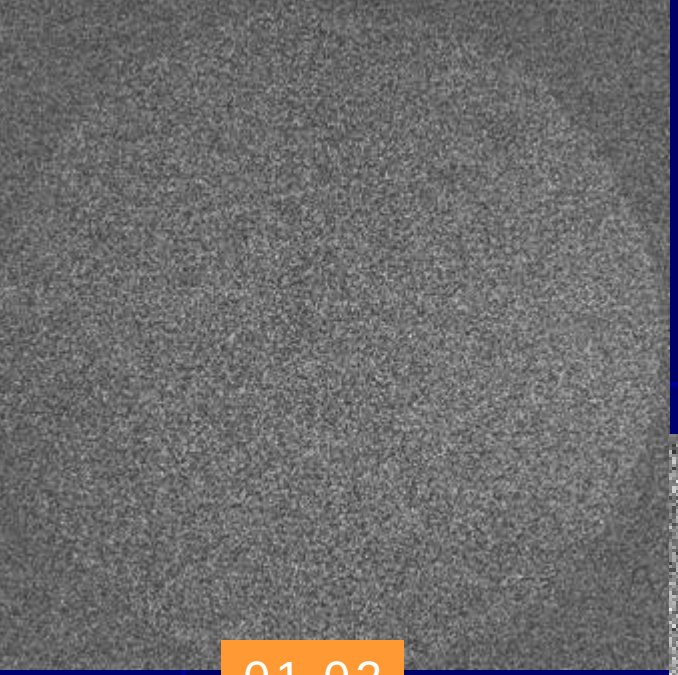
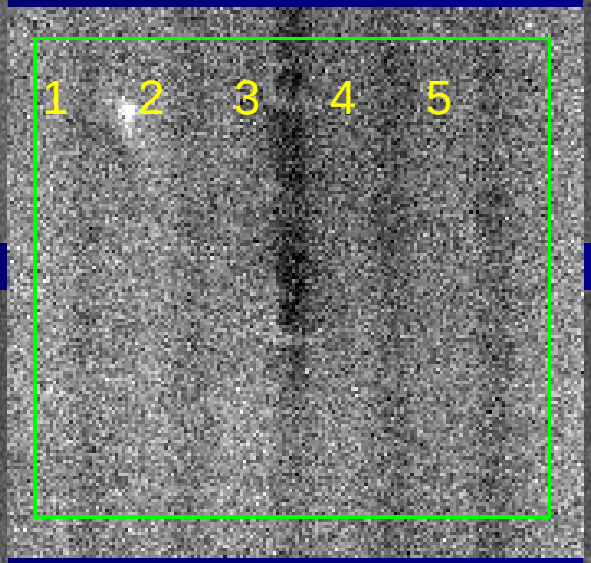
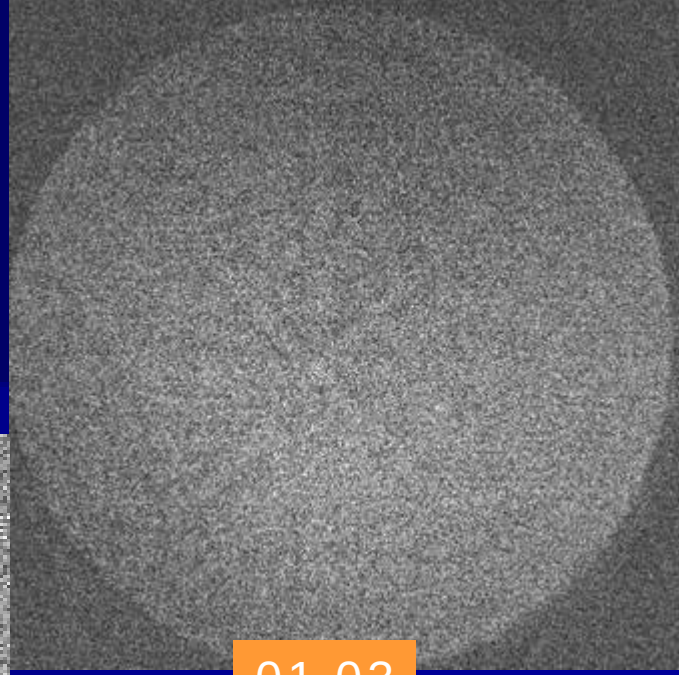


Image Processing to Reveal Pattern Differences




01-02


1
2
3
4
5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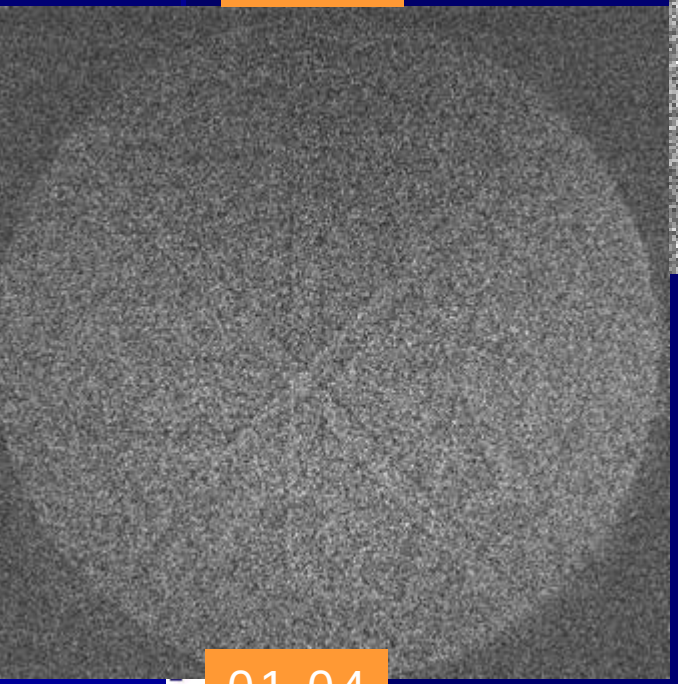
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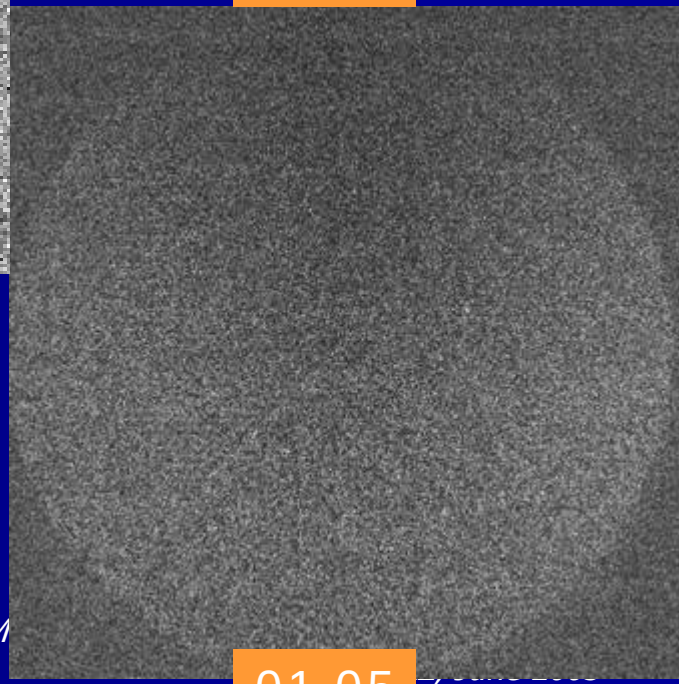
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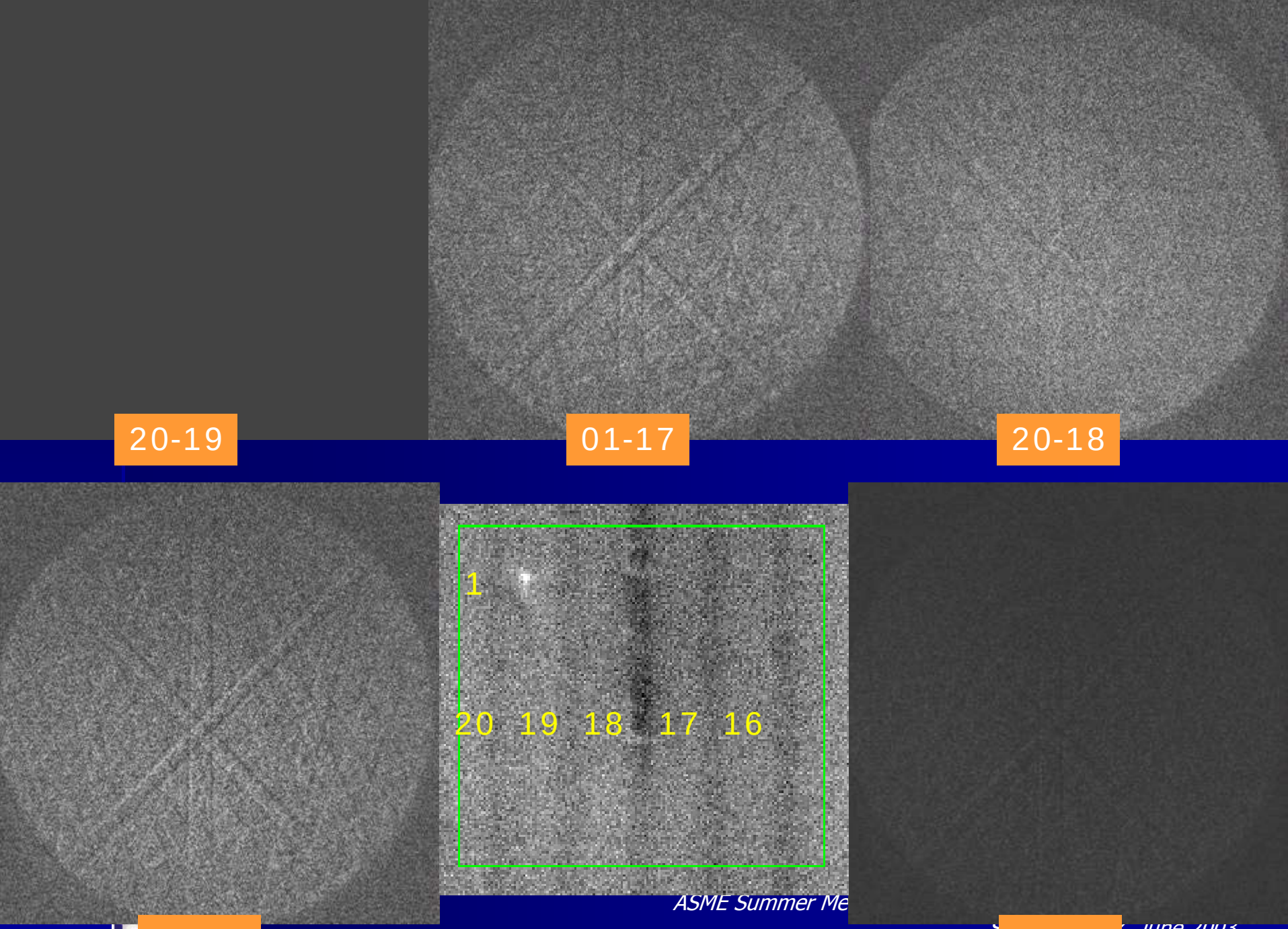
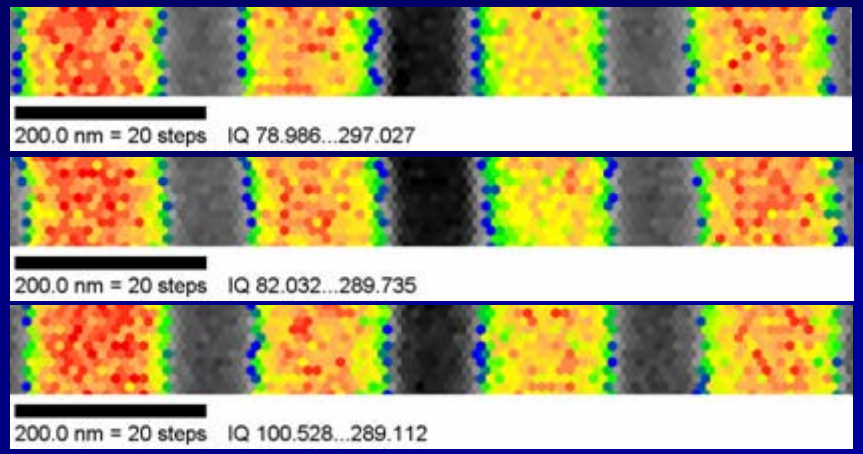
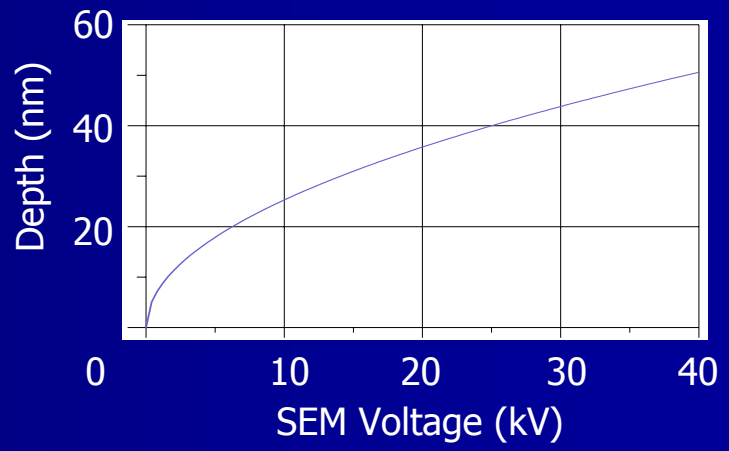

20-19
01-17
20-18
1
20 19 18 17 16
20-17
20-16

EBSD Spatial Resolution Considerations

- Higher resolution IQ maps are possible. (example)
- Resolution tied to information volume for backscattered electrons that are dependent upon crystal orientation.
  - This volume has dimensions of the order of 2 absorption lengths. (Spencer *et al.*, Phil. Mag. 26, 193-213 (1972))
    - = f (Z, keV)
  - For GaAs at 15 kV, expect ~30 nm diameter
  - Tied to SEM conditions too.



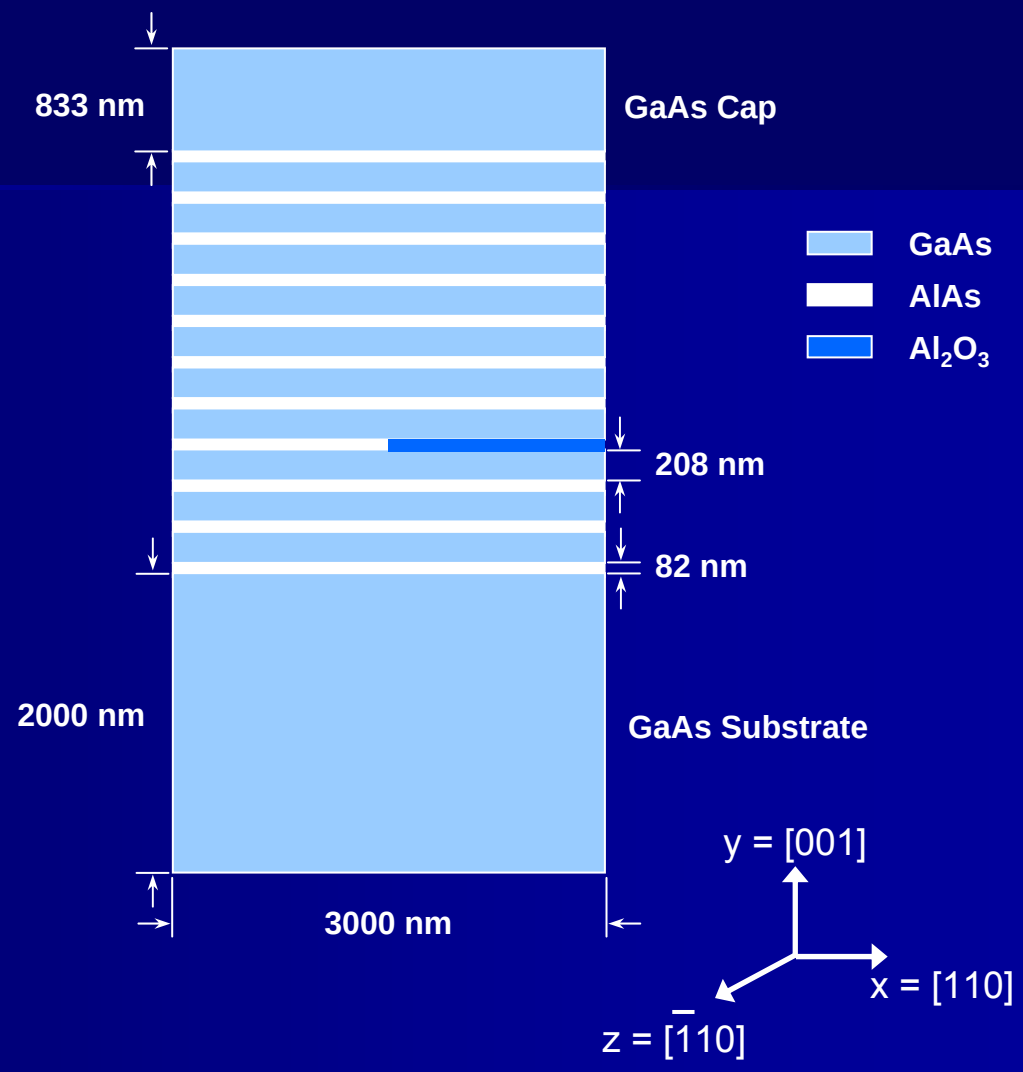


Finite Element Analysis

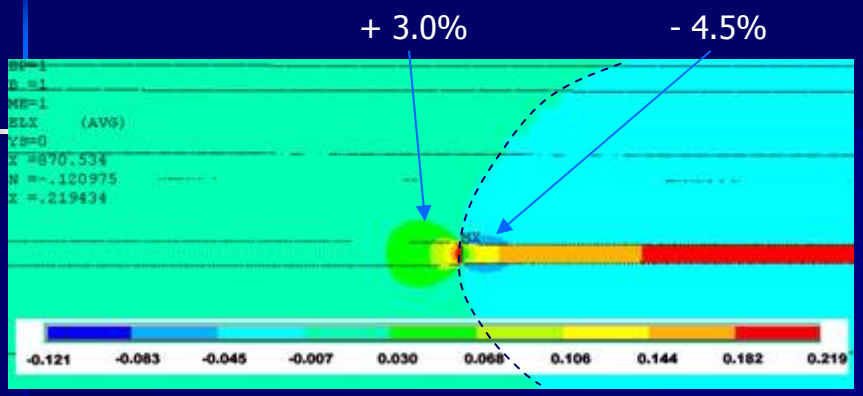
FEA analysis: assess physical validity of measured strain field

- Boundary conditions:
  - Fixed lower left corner, free to expand in +x, +y
- Loading:
  - Heat the structure,
  - Adjust CTEs to give 20% contraction in alumina
  - plane strain

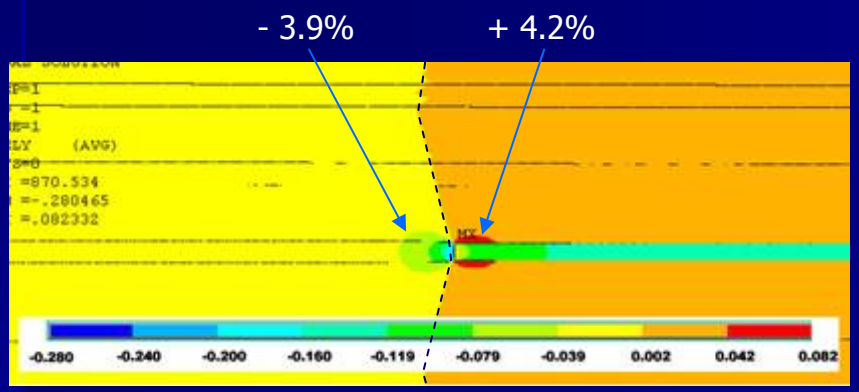
	GaAs	AlAs	γ-Al_2O_3
E[100]	85.7 GPa	83.5	
E[110]	122	120	221 (polyxtal)
E[111]	141	141	
ν	0.32	0.33	0.25



Calculated Elastic Strain Fields



ε_{xx}



ε_{yy}

300 nm

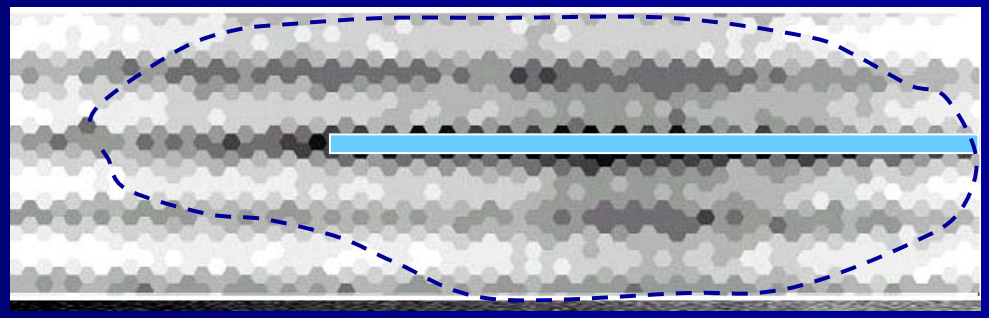
Note that we can measure distortions only in the crystalline regions, i.e. GaAs and AlAs.

In principle, we can extract these strain components, but we have not yet been successful.

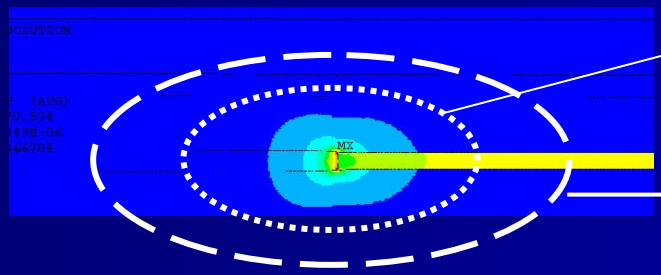
Average (von Mises) Strain Field

$$\varepsilon_{\text{von Mises}} = (1/\sqrt{2})\,[(\varepsilon_x-\varepsilon_y)^2 + (\varepsilon_y-\varepsilon_z)^2 + (\varepsilon_z-\varepsilon_x)^2 + 6(\gamma_{xy}^2+\gamma_{yz}^2+\gamma_{xz}^2)]^{1/2}$$

- Experimental data for the distortion field most resembles von Mises field.
- EBSD pattern sharpness is an averaging method, and so *should* most closely correspond to the von Mises determination of an equivalent strain.



300 nm



Summary and Conclusions

- We have demonstrated the utility of EBSD for characterizing elastic strain fields in a selectively oxidized GaAs/AlAs heterostructure.
- EBSD band widths can be used to directly measure elastic strain, with resolution approaching 0.1% strain.
- EBSD image quality, or pattern sharpness, is sensitive to the magnitude of the strain gradient within the sampled volume of material.
- Image quality maps reveal the elastic distortion field about an oxide front.
- The distortion field shown by EBSD is in reasonably good agreement with finite element simulations , using γ-Al_2O_3.