MSE 582 - Introduction to transmission electron microscopy
&
MSE 640 - Transmission electron microscopy & crystalline imperfections

Instructor: Eric Stach

Goals

- **Goals:**
  - MSE 582: Perform simple imaging and diffraction experiments with the microscope
  - MSE 640:
    - In depth understanding of fundamentals of scattering, diffraction and imaging
    - Practical application of these concepts towards materials characterization
- **Taught in four units:**
  - Operation of the TEM (MSE 582 content)
  - Diffraction
  - Imaging
  - Spectroscopy

About me …

Education

- B.S.E. - Duke University
- M.S.M.S.E. - University of Washington
- Ph.D. - University of Virginia

Professional Experience

- Graduate research - IBM Watson Research Labs
- Staff Scientist, Principal Investigator and Program Leader, National Center for Electron Microscopy & Materials Sciences Division, Lawrence Berkeley National Laboratory
- Associate Prof @ Purdue since January 2005

Research expertise

- Crystalline defects, crystal growth, electronic thin films, electron microscopy, mechanical behavior, nanostructured materials

Course mechanics

MSE 582: Pass/Fail

- Can you take a picture with the microscope?

MSE 640:

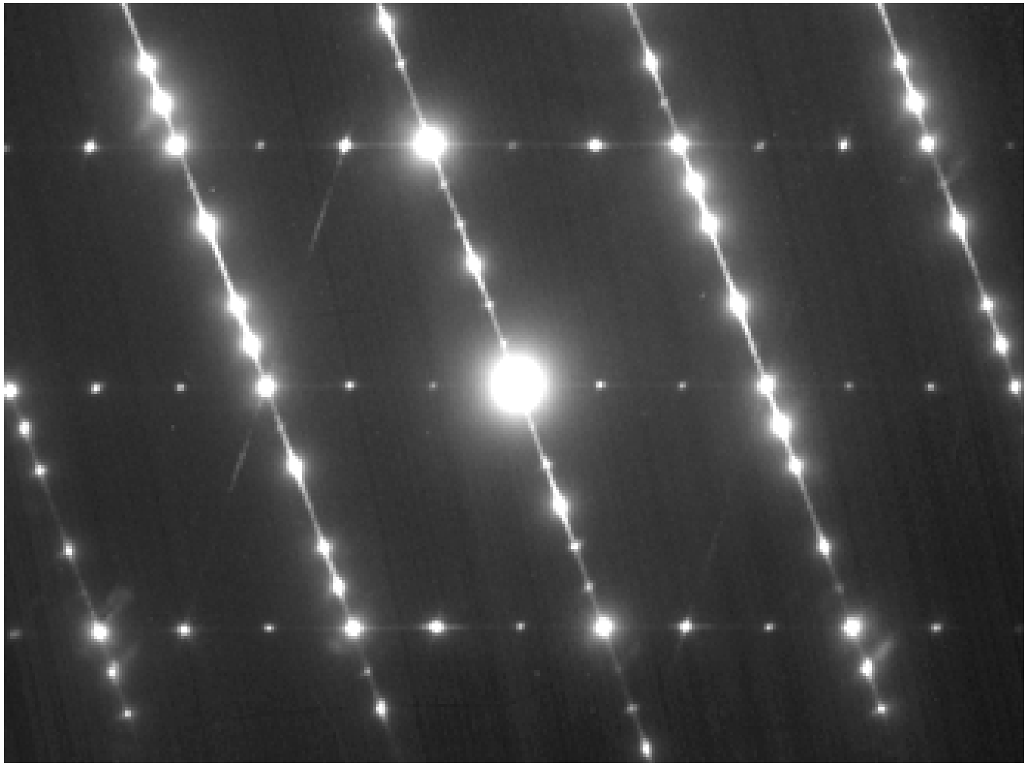
- Homework (20%)
- Laboratories (30%)
  - We'll engage in a systematic study of the microstructure of an advanced aluminum alloy
- Exams (50%)
  - Midterm (after diffraction unit - 20%) and a Final (30%)

What can you do with a TEM?

What can you do with a TEM?

Selected Area Diffraction (SAD)

- Crystallographic structure from particular areas of a sample.
- Used to distinguish and identify crystalline (and amorphous) phases in a material.



Selected area diffraction pattern

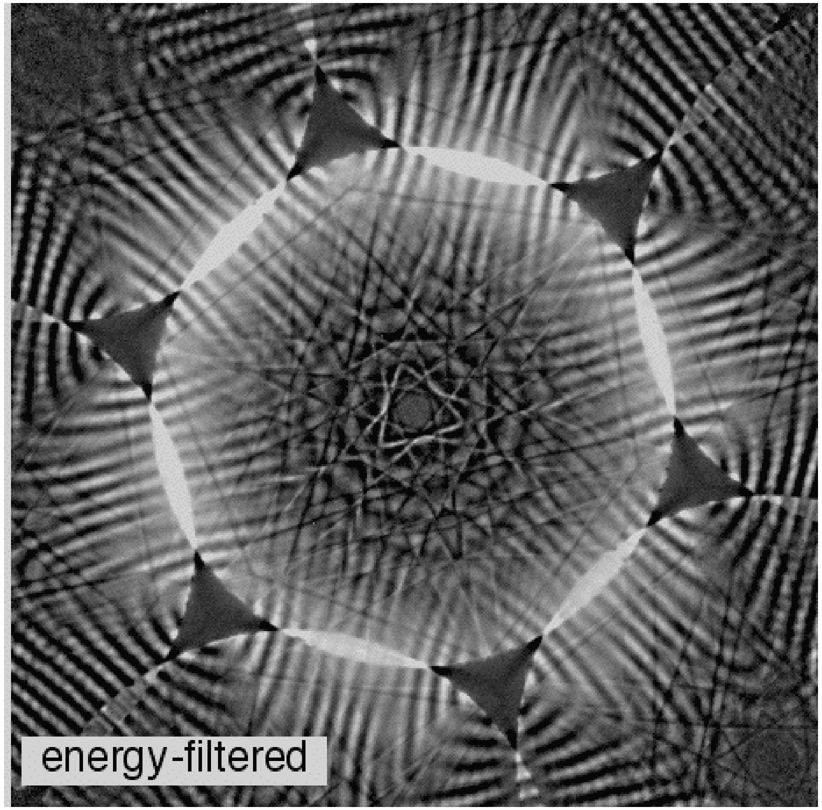
$10\bar{1}0$ Zone axis pattern of a hexagonal GaN / cubic GaN heterostructure

Pattern reveals extensive twinning in the cubic layer

What can you do with a TEM?

Convergent Beam Electron Diffraction (CBED)

- Point and Space Group determination
- Local strain
- Nanoscale diffraction



Convergent beam electon diffraction pattern

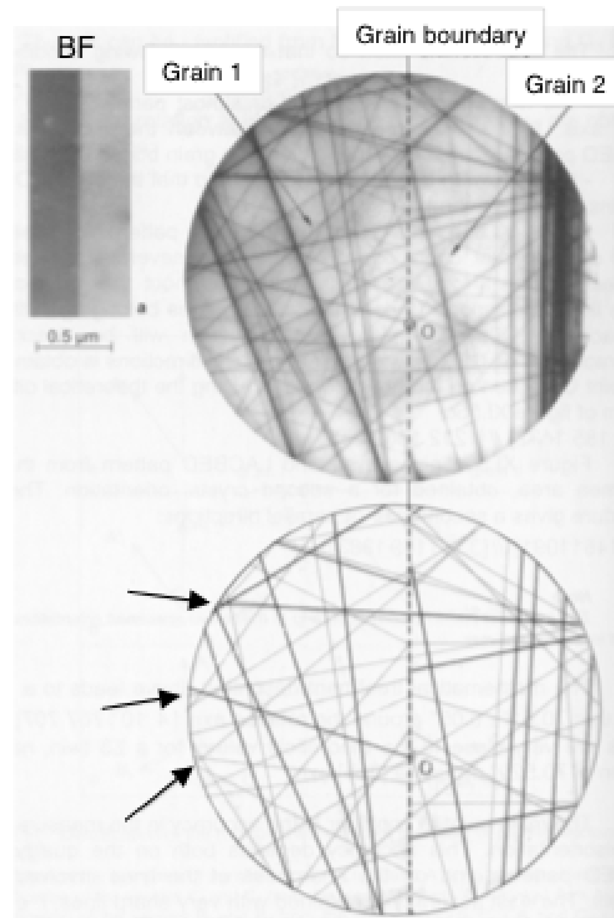
111 zone axis pattern of silicon

Note detailed structure in the central disk

What can you do with a TEM?

Large-angle Convergent Beam Electron Diffraction (LACBED)

- Misorientation across grain boundaries
- Dislocation Burgers vector
- Crystalline symmetry



at 0: $\mathbf{A}_I$ = [1142 185 1432] $\mathbf{A}_{II}$ = [1212 1362 255] at 0:

→ $\mathbf{u}_R$ = (-4·10^{-7} 707 707), θ_R = 70.51° ± 0.05°

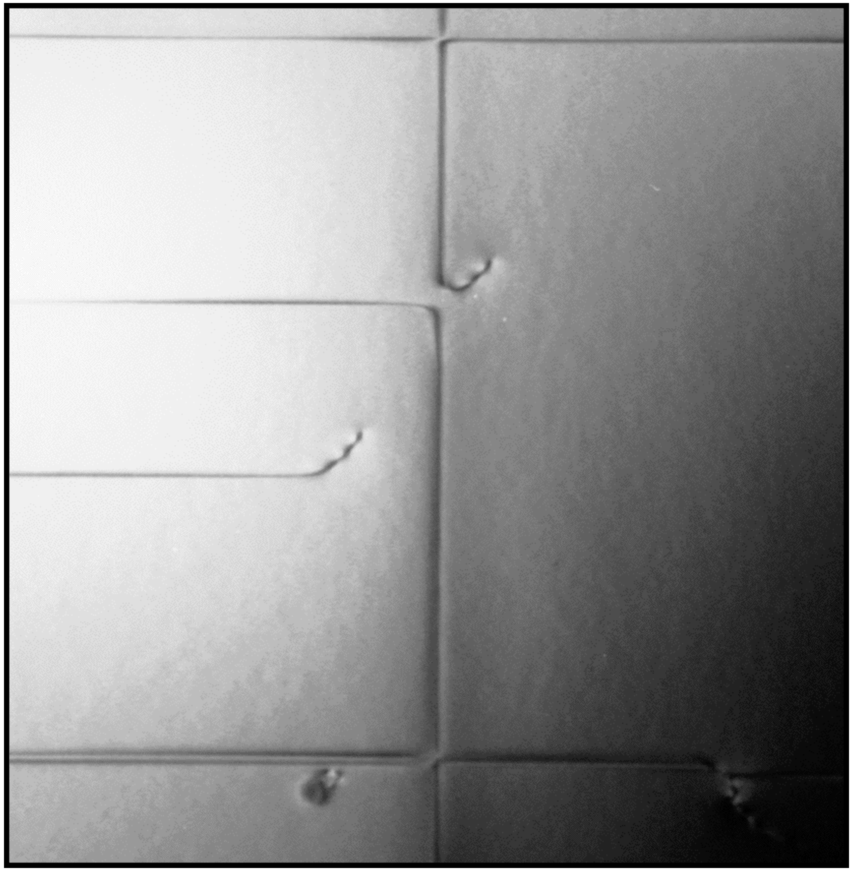
From J.P Mornoroli

What can you do with a TEM?

Diffraction Contrast Imaging

Strain fields

- Dislocations
- Stacking faults
- Grain boundaries
- Precipitates
- Second phases



Typical bright field image

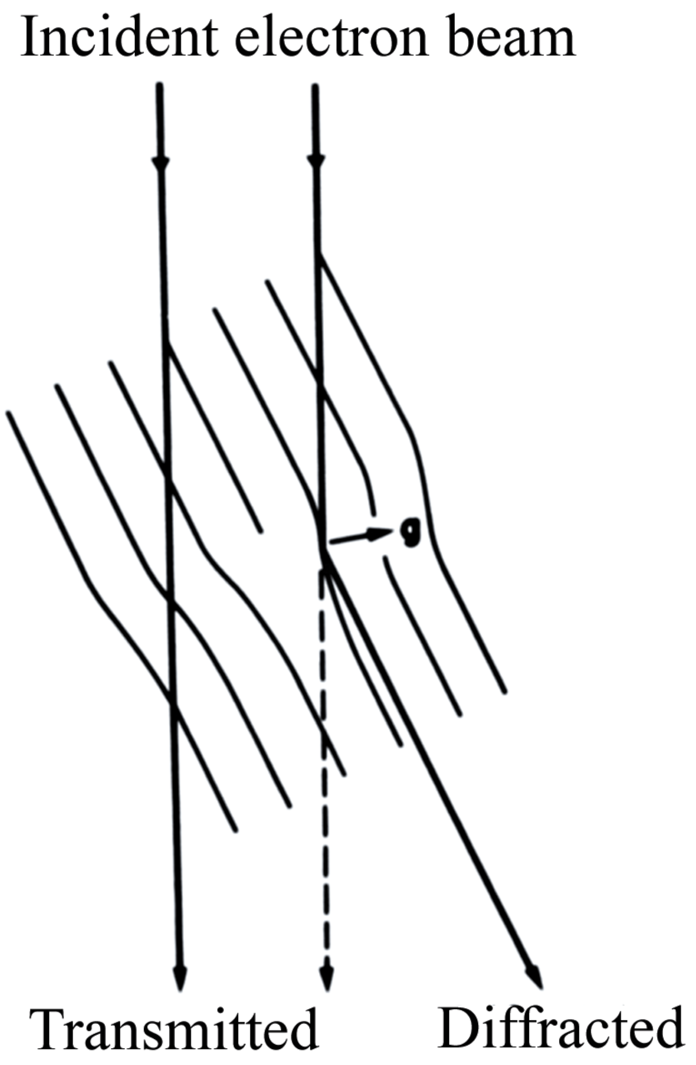
Dislocation configurations at the interface between a SiGe heteroepitaxial layer and a Si (100) substrate viewed in plan view (along [100])

What can you do with a TEM?

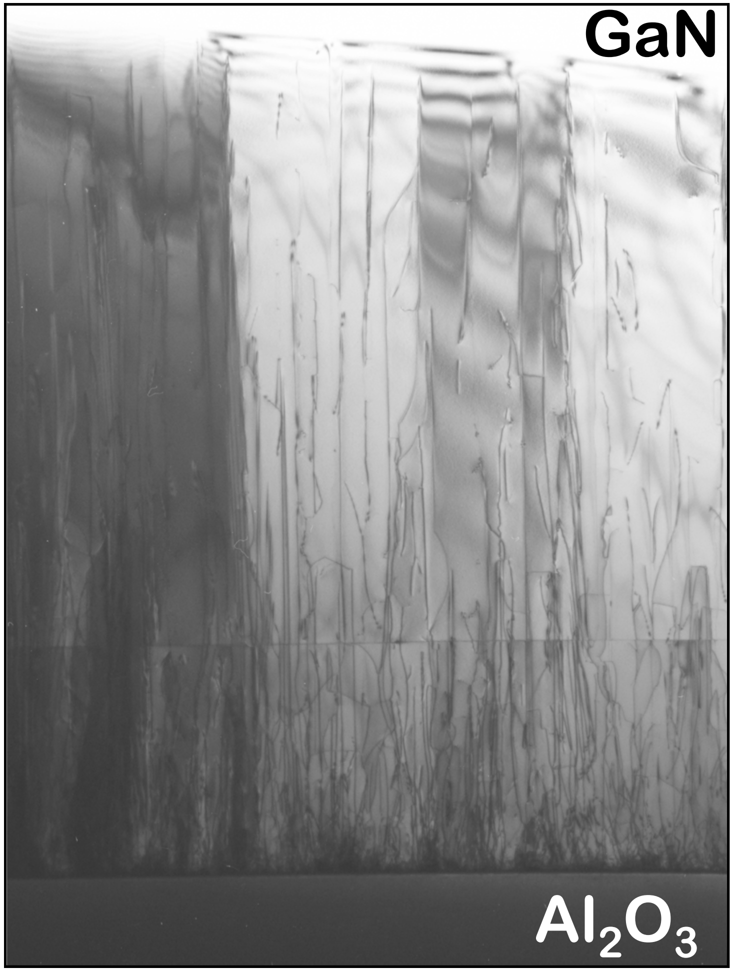
Diffraction Contrast Imaging

One beam selected for imaging

- Transmitted - “bright field"
- Diffracted - “dark field”



What can you do with a TEM?



Bright field image

Dark field image

What can you do with a TEM?

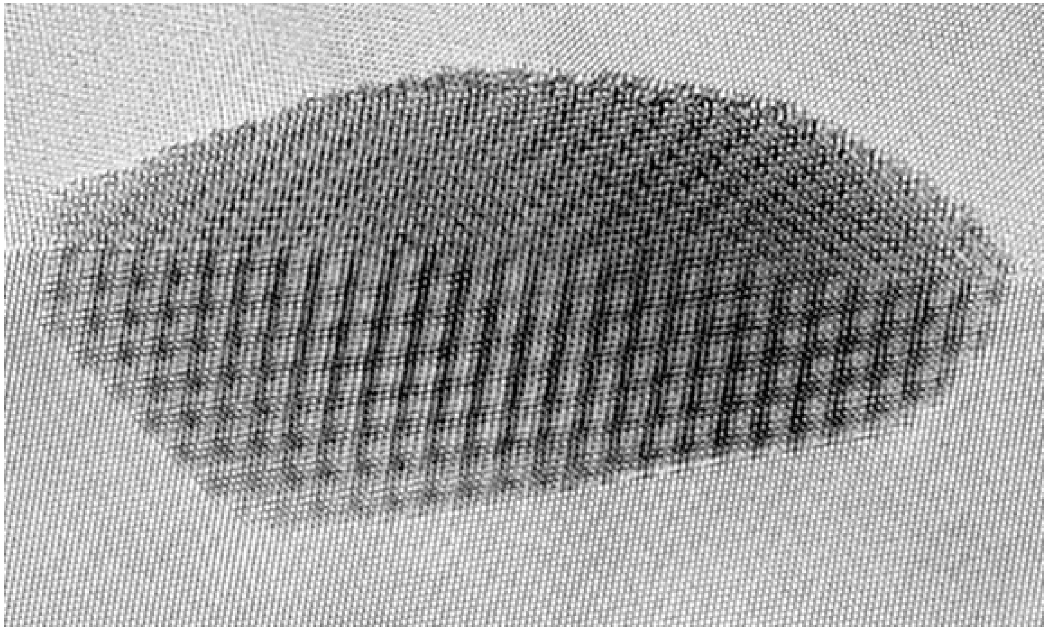
'Weak beam' dark field image

What can you do with a TEM?

High-resolution imaging

Atomic column images at resolutions from 0.7Å and above

- Interference of transmitted and diffracted electron waves



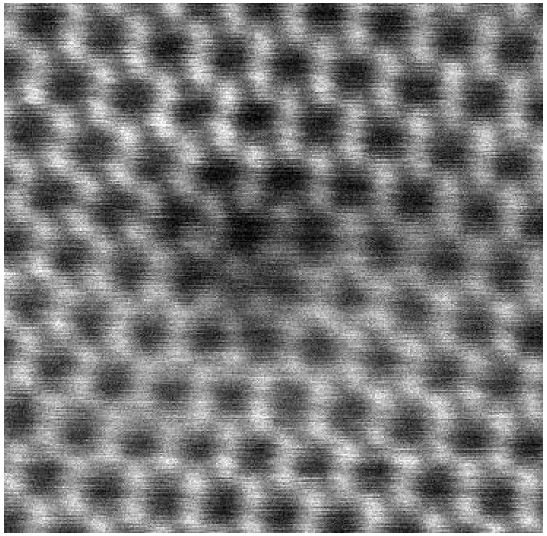
High resolution micrograph of a precipitate at a high angle grain boundary in aluminum

Courtesy of U. Dahmen

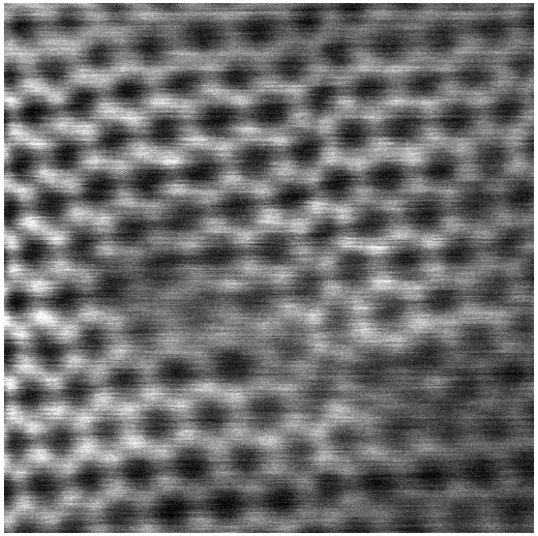
What can you do with a TEM?

High angle annular dark field (HAADF) imaging

- Accomplished in a dedicated Scanning TEM (STEM)
- Collects incoherent scatter, yields atomic resolution



Mixed dislocation in GaN



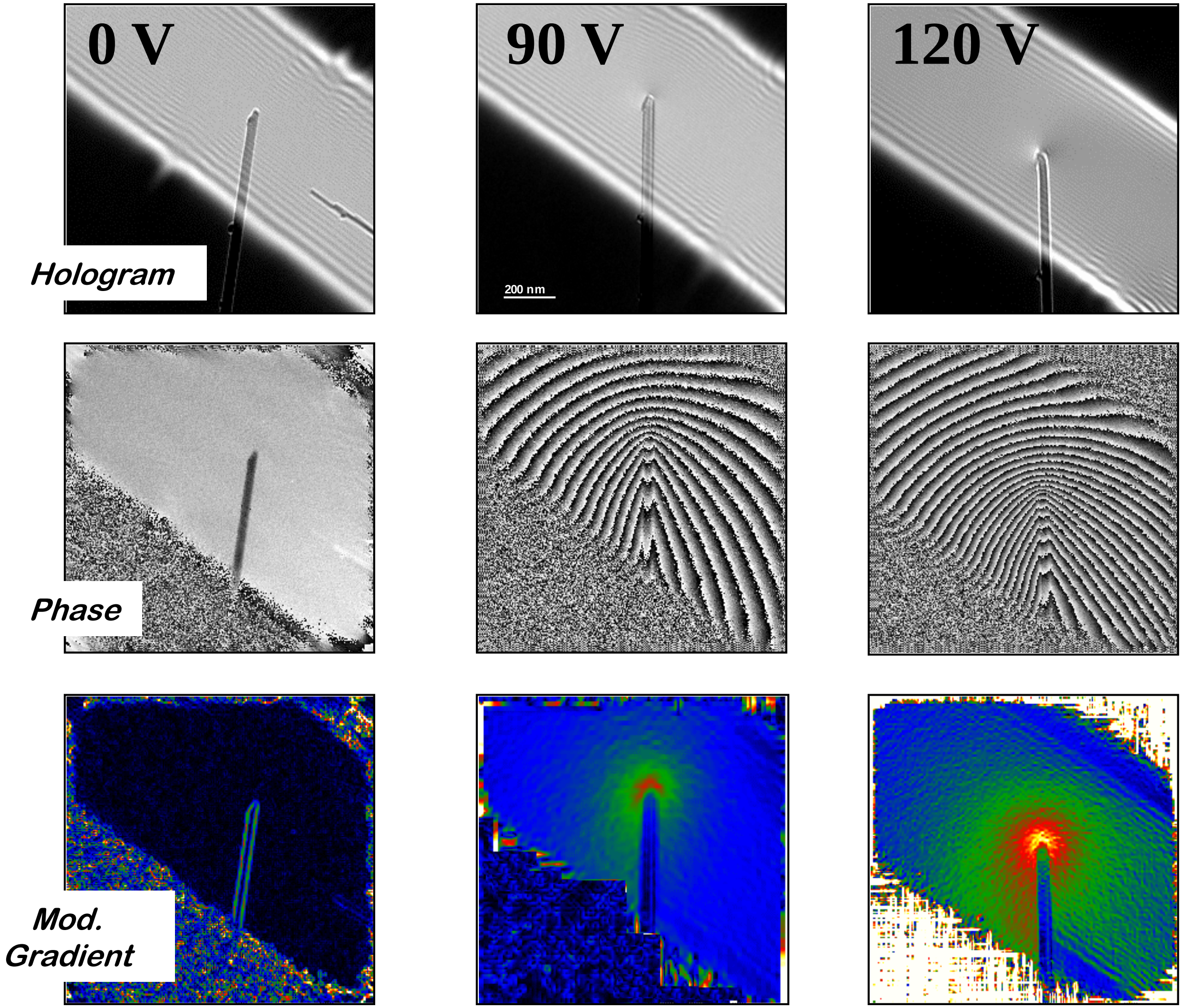
Dissociated mixed dislocation in GaN

Courtesy of I. Arslan

What can you do with a TEM?

Electron holography

Map the mean inner potential of a material

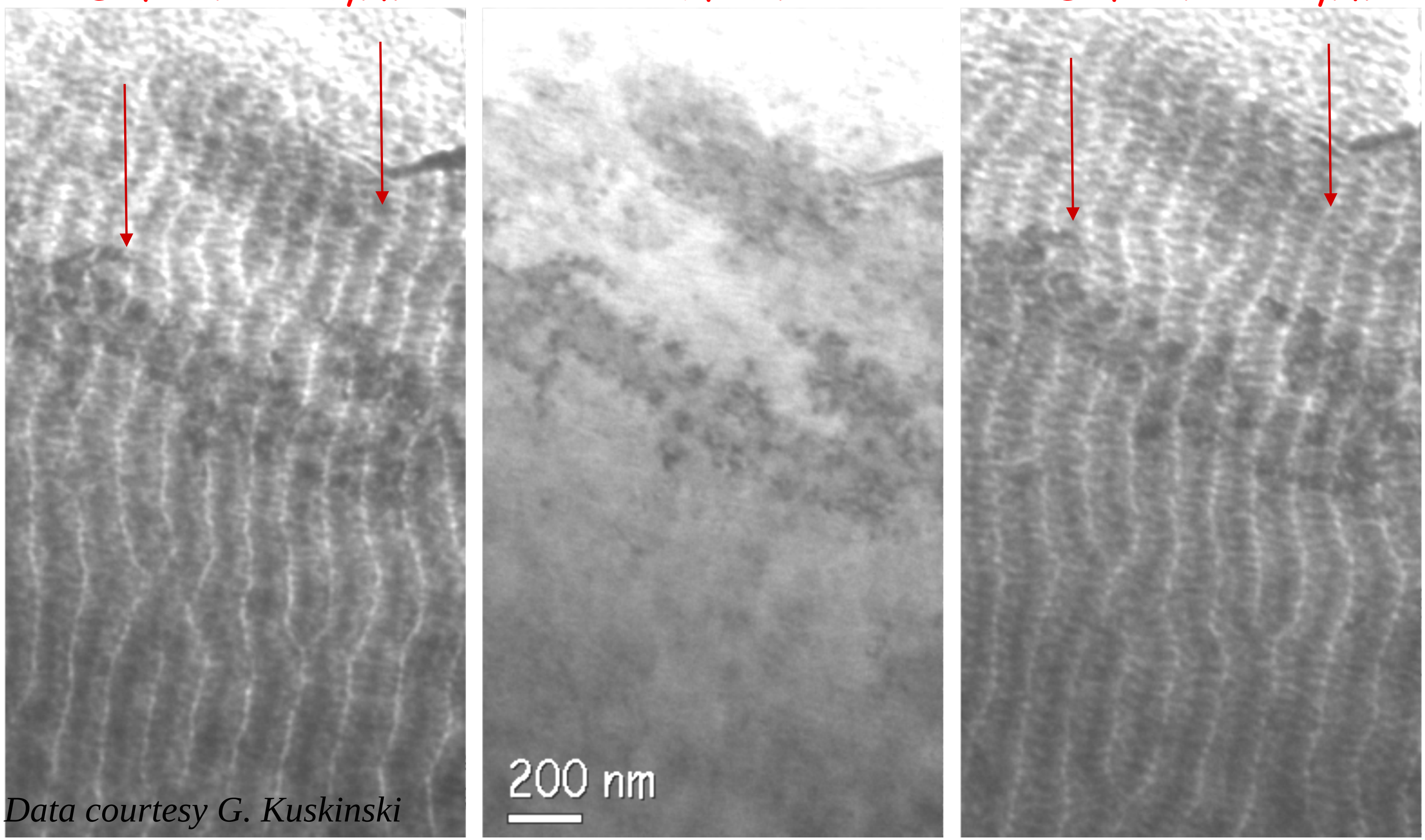


Courtesy of J. Cumings

What can you do with a TEM?

"Lorentz" microscopy for imaging of magnetic structures

Defocus = -15μm | In focus | Defocus = +15μm

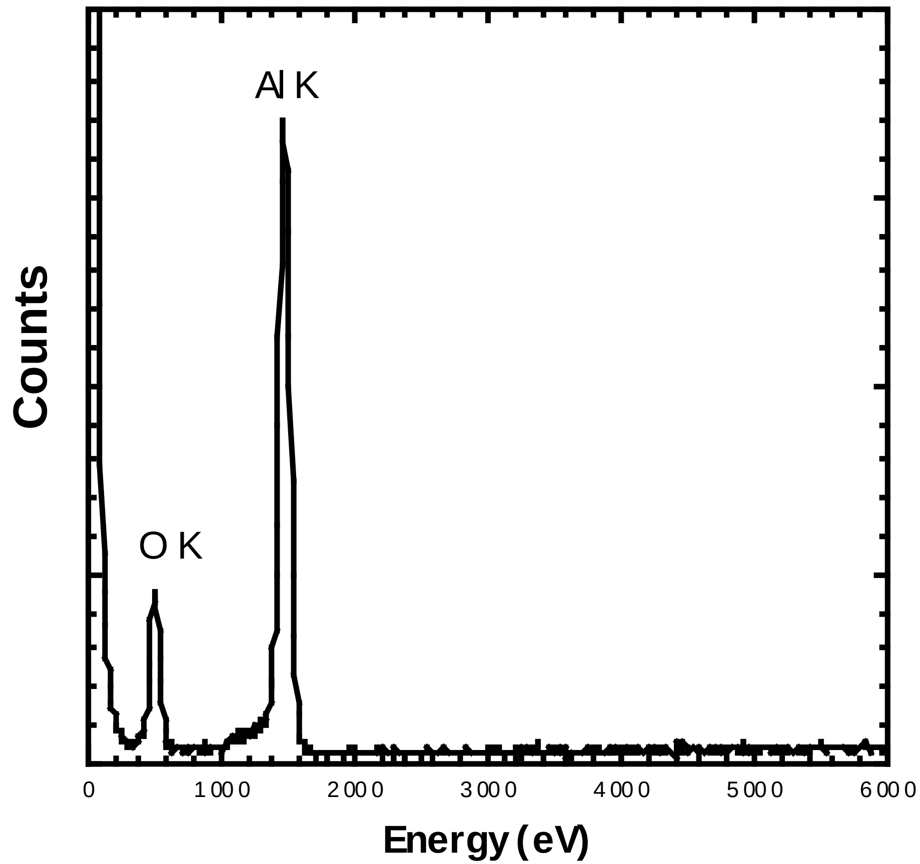


Data courtesy G. Kuskinski

What can you do with a TEM?

Energy Dispersive X-ray Spectroscopy (EDS)

- Detection of characteristic x-rays excited by incident electrons.
- Spatial resolution on the order of probe size (can be as low as 2-3 Å)



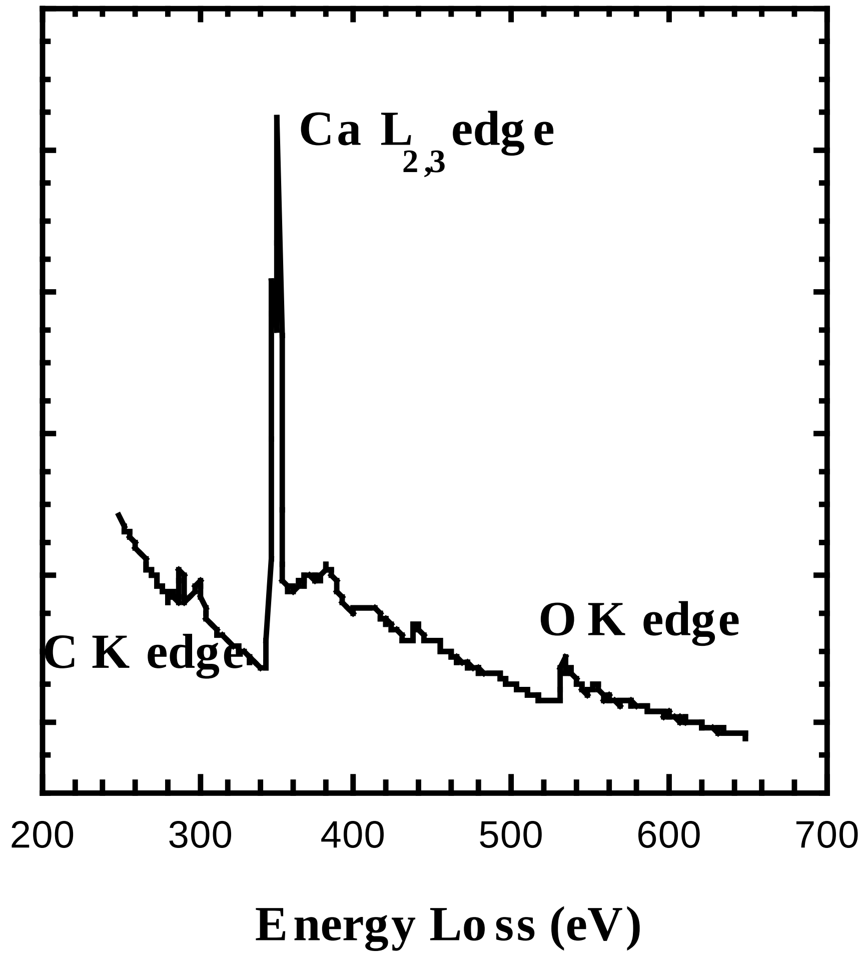
Simple EDS spectrum from Al_2O_3

From Williams & Carter

What can you do with a TEM?

Electron energy loss spectroscopy

- Measures the amount of energy lost by the incident electrons.
- Similar spatial resolution, energy resolution of ≈ 1 eV.
- Probes density of state (DOS) locally.

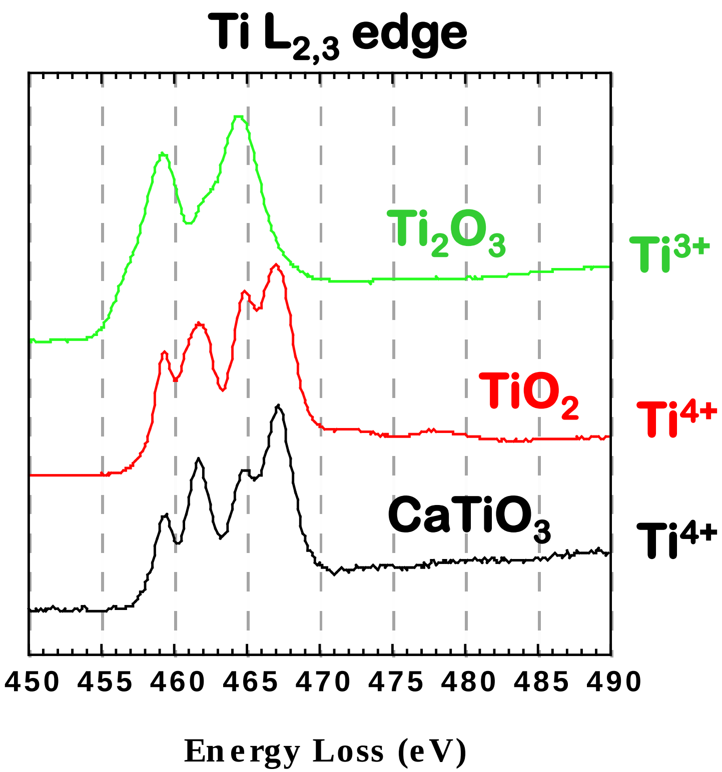


EELS spectrum from $CaCO_3$

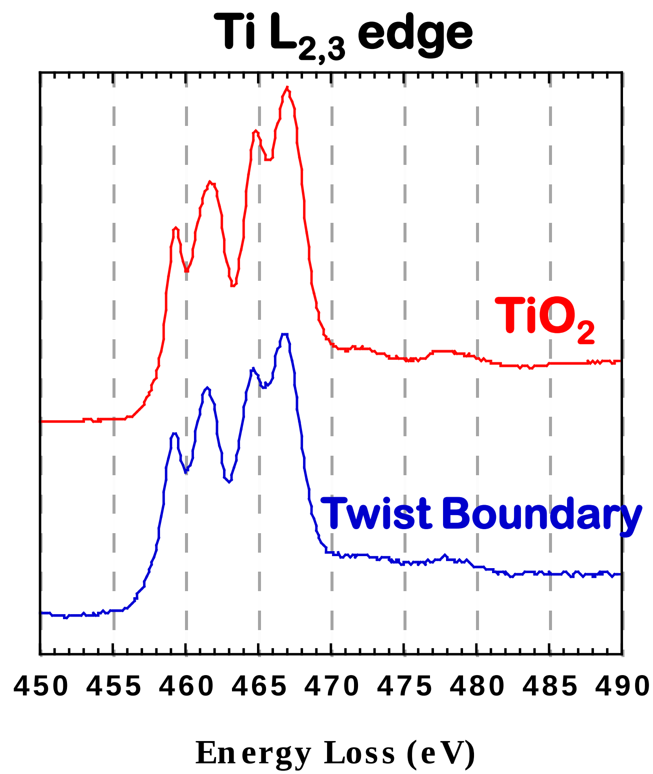
From Williams & Carter

What can you do with a TEM?

Ti valence determination using EELS



Ti $L_{2,3}$ edge from trivalent Ti_2O_3 differs markedly from tetravalent compounds TiO_2 and $CaTiO_3$



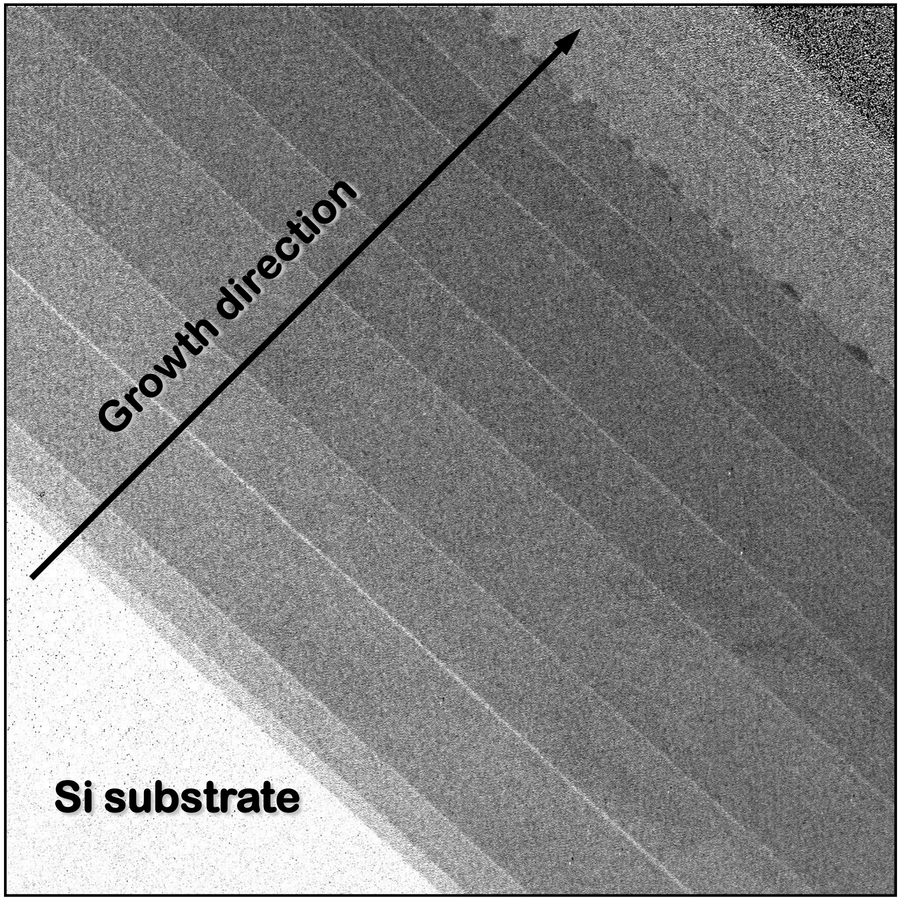
Ti $L_{2,3}$ edge from twist boundary closely matches edge structure of TiO_2 standard (Ti^{4+}).

Data courtesy Seth Taylor

What can you do with a TEM?

Energy Filtered Imaging

- Zero loss imaging removes inelastically scattered electrons from image
- Selective imaging of electrons that have lost a particular energy
- Most commonly used to create a map of local (≈ 1 nm) chemistry



Energy filtered image of a SiGe graded buffer structure using the Si K edge.

The brighter the pixel, the larger the concentration of Si.

What can you do with a TEM?

Dynamical behavior

- Possible to apply many type of stimuli to samples during simultaneous imaging.
- Probe interrelationships between structure / properties and processing
- Stimuli include:
  - Temperatures to 1300°C
  - Temperatures to LN_2
  - Chemical flux
  - Nanomanipulation
  - Nanoindentation
  - Electrical bias in combination with heating
  - Uniaxial strain

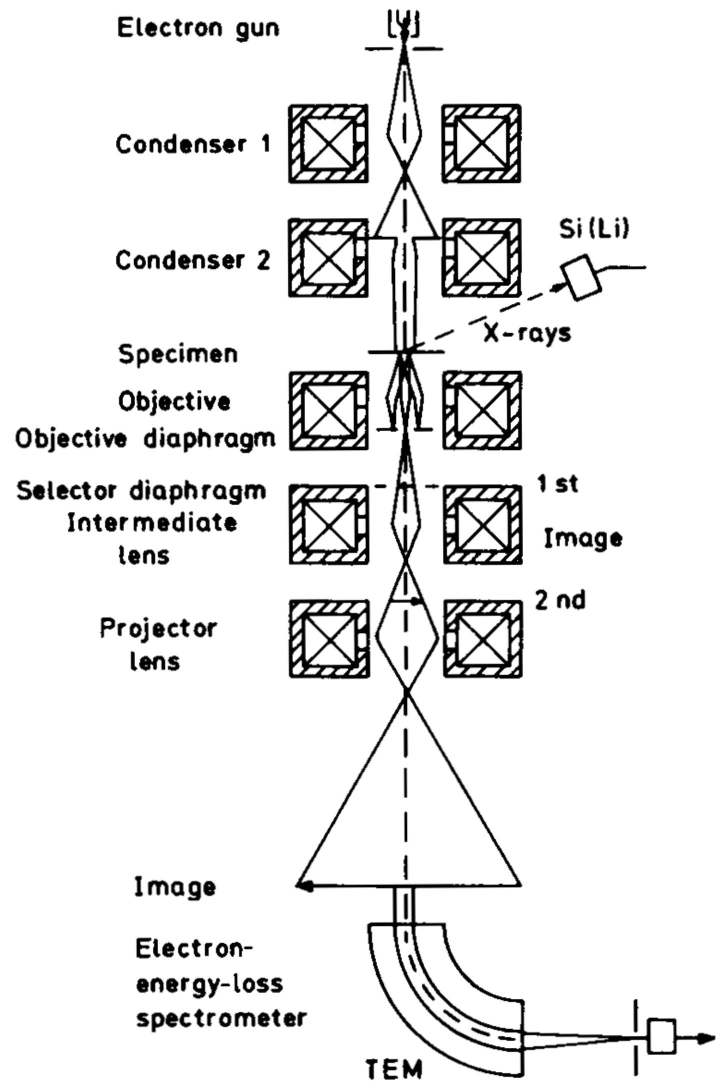
Interaction between threading and misfit dislocations in a SiGe heterostructure

A way to organize our thoughts

In MSE 582 we will learn how to "use" the microscope

We will discuss:

- Electrons & scattering, briefly
- Electron sources
- Lenses & aberrations
- How the instrument goes together
- Alignment
- The sample

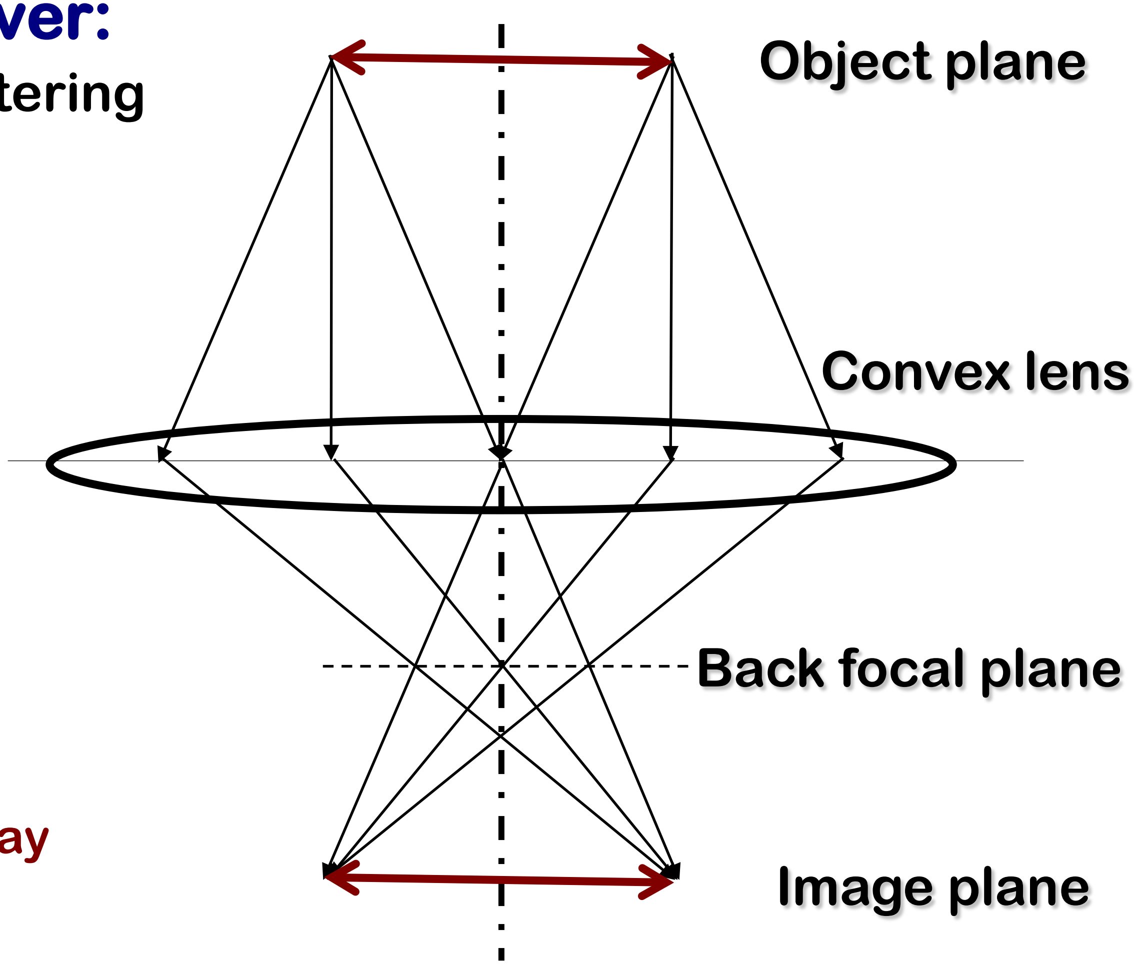


From Reimer

A way to organize our thoughts

In MSE 640, we will cover:

- Elastic & inelastic scattering
- Diffraction
  - Single scattering (kinematical)
  - Multiple scattering (dynamical)
- Image formation
  - Diffraction contrast
  - Phase contrast
  - Incoherent imaging
- Spectroscopy
  - Energy Dispersive X-ray Spectroscopy
  - Electron Energy Loss Spectroscopy



Basic properties of electrons

a reminder …