Transmission Electron Microscopy of Semiconductors and Heterostructures

D. Cherns

University of Bristol, UK
Outline

- Background
- Scattering theory
- Applications
  - Imaging (defects, interfaces, atomic structure …)
  - Diffraction (strain, polarity …)
  - Microanalysis (chemical composition etc)
- Recent developments
Transmission electron microscope (TEM)

- Heated W, LaB6 or field emission source
- Electromagnetic lenses, giving direct imaging or diffraction using a parallel probe, or microanalysis using a focused probe
- Thin samples (10-500 nm)
Electrons as particles (200kV)

- Electrons travel at 0.7c (relativistic)
- Up to $10^{10}$ e/sec. Focused probe (field emission gun) can generate up to 1nA into 1nm probe, or greater than $10^8$ e/atom/sec
- An electron can transfer up to 44eV to a carbon atom in a head-on collision. This can generate point defects (bulk) and sputtering (surface)
- Less energetic collisions generate phonons, excitation of inner and outer shell electrons, plasmons and photons. This inelastic scattering gives microanalysis and imaging using a variety of signals
- Radiation damage can be a problem, with some organic materials damaging at down to 1 e/atom. Conversely there is potential for lithography and hole drilling
Inelastic scattering: some useful signals

- Secondary electrons
- Electron energy loss spectroscopy (EELS)
- High angle annular dark field detection (HAADF)
- X-rays (EDX)
- Cathodoluminescence
Electrons as waves: diffraction

\[ \lambda = h/p \text{ (de Broglie)} = 0.0025\text{nm (200kV)} \]

c.f. \( \lambda = 0.1 \text{ nm (X-rays), 500nm (light)} \)

\[ \lambda = 2d\sin\theta \]

Bragg’s Law
Spatial resolution

\[ d = \frac{\lambda}{\alpha} \]

Abbe criterion (\( \alpha = \text{convergence angle} \))

Light microscope: \( \alpha \sim 1 \text{ rad}, \ d \sim \lambda \)

TEM: \( \alpha \sim 10^{-2} \text{ rad}, \ d \sim 100\lambda \ (0.2\text{nm})! \)

i.e. resolution is comparable to atom spacings, and \( \alpha \) is comparable to the Bragg angle
The presence of aberrations requires that imaged beams must be as close as possible to the optic axis. Selection is by means of an objective aperture.

**Imaging modes**

- **Bright field**
- **Dark field**
- **Lattice imaging**
Scattering theory

Amplitude scattered into $g$ (thin crystal limit):

$$\frac{d\phi_g}{\phi_0} = \frac{i\pi \Delta t}{\xi_g}$$

$\Delta t = \text{specimen thickness}$

$\Phi = \text{amplitude}$

$\xi_g = \text{extinction distance}$

For electrons, $\xi_g \sim 10 - 100 \text{ nm}$

For X-rays, $\xi_g \sim 2-3$ orders of magnitude greater
TEM: why so many reflections?

\[ K = g \]

\[ K = g + s \]
Two-beam imaging

In general, electron diffraction is a many beam problem.

Fortunately, it is possible to orient a single crystal sample until only one diffracted beam is strong. Understanding diffraction is then a relatively simple two-beam problem:
Two-beam imaging: significance of “deviation parameter” $s$

Large $s$ is simple (kinematical):

$$\phi_g \phi_0 = \left( \frac{i\pi}{\xi g} \right) \int_0^\infty \exp(-2\pi isz)dz$$

$$\frac{\phi_g^2}{\phi_0^2} = \frac{\pi^2}{\xi g^2} \frac{\sin^2(\pi s)}{(\pi s)^2}$$

$$\Delta t = \frac{1}{s}$$

$s = 0$

$s = 0.2\text{nm}^{-1}$

$s = 1.3\text{nm}^{-1}$ (surface steps)
Two-beam imaging: significance of “deviation parameter” $s$

$s = 0$: behaviour is dynamical:

\[
\frac{\phi_g^2}{\phi_0^2} = \frac{\pi^2}{\xi_g^2} \frac{\sin^2 \pi \sqrt{s^2 + \xi_g^{-2}}}{\pi^2 (s^2 + \xi_g^{-2})}
\]

\[
\Delta t = \frac{1}{\sqrt{s^2 + \xi_g^{-2}}}
\]

$s = 0, \Delta t = 1/s = \xi_g$

Bloch waves

Channelling
Two-beam imaging: defects

A good qualitative understanding of contrast can be obtained using the kinematical formula

\[
\phi_g / \phi_0 = \frac{i \pi}{\xi_g} \int_t^0 \exp(-2\pi i (s \zeta + g \cdot R)) d\zeta
\]

E.g. for dislocations \( g \cdot R \) defines bending of diffracting planes
Two-beam imaging: defects

Dislocations are seen when the diffracting planes are distorted, i.e. when the dot product $g \cdot b$ is non-zero.

Analysis of misfit dislocations in $\text{NiSi}_2/(001)\text{Si}$ interface
Core structure of dislocations: weak beam technique

Image is seen where planes are bent towards $s = 0$, i.e. progressively closer to the core as $s$ increases.

$K = g + s$

Dislocations in semiconductors are often dissociated.
Stacking faults are visible when the diffracting planes are fractionally displaced, i.e. contrast depends on $g \cdot R$. Two-beam imaging: defects
Lattice imaging: many (strong) beams

Scattered amplitudes from Si viewed along [110] as a function of film thickness. Phases vary also!

Can we believe what we see?
Lattice imaging

Hence two problems:

• Seeing is not believing

• Limited resolution described by the contrast transfer function

• However, with computation many problems can be solved

“B” NiSi2/(111)Si along [110]

Si nanocrystal (Takeguchi JEM 48, 1087)
Lattice imaging

Current advances:

• Resolution improvements from 0.2nm to better than 0.1nm through aberration correction

• Smaller focused probes

• Improved resolution of structure (e.g. closely spaced atoms in semiconductors), lattice imaging by scanning TEM (STEM) using chemically sensitive signals
HAADF: Sb dopants in Si (courtesy of D. Muller)

NiSi_2/(001)Si 1984 - 2004

Direct image

HAADF image, courtesy of A. Bleloch showing higher resolution and chemical sensitivity
Convergent beam electron diffraction

Selected area diffraction

CBED

LACBED
Electron rocking curves

CBED Si 220

LACBED InP/InGaAs MQW 200 disc (Vincent et al Inst Phys Conf Ser 90, 233 (1987))

Ewald sphere

$k = 1/\lambda$

20
High order (weak) reflections: grain boundaries in Si

High order reflections: Rotation of wings in GaN ELOG structures
Z Liliental-Weber and D Cherns JAP 89 7833 (2001)
Asymmetry in the CBED patterns is a dynamical effect depending on double diffraction between 0002 and 000-2 reflections. It represents breaking of Friedel’s Law...
Phase shift depends on the “inner potential”, which can include contributions from internal (and external) magnetic or electric fields.
Examples of holography

Phase map around a charged latex sphere (K Yamamoto et al, JEM 49 (2000) 31)

Electric fields in GaN/InGaN LEDs


D. Cherns and C. Jiao
PRL 87, 205504 (2001)
A problem requiring a combination of techniques!

Ref. A Briggs (www.nanotech.org)
Some references and acknowledgements

- P.B. Hirsch et al “Electron Microscopy of Thin Crystals”
- M.H. Loretto “Electron Beam Analysis of Materials”
- J-P. Morniroli “Large Angle Convergent Beam Electron Diffraction”