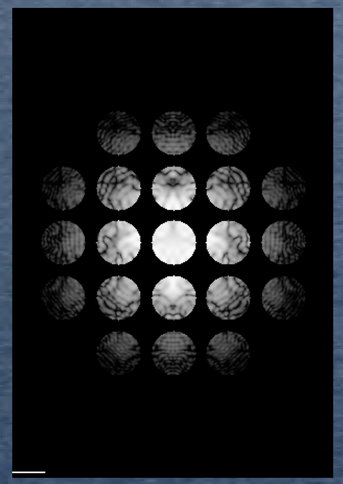
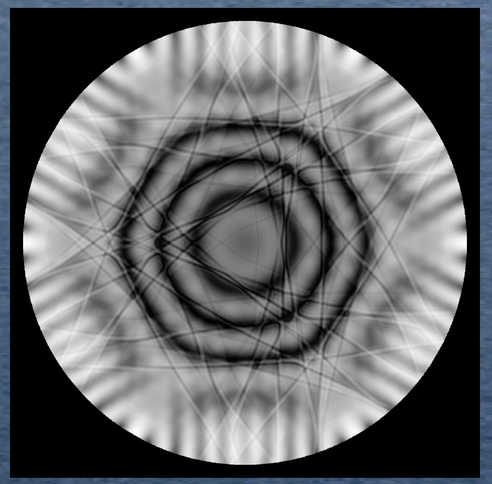
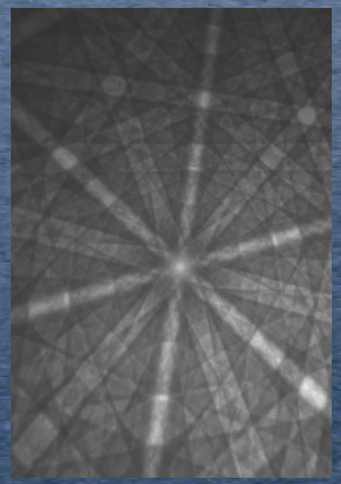
Introduction to Crystallography and Electron Diffraction

Marc De Graef
Carnegie Mellon University







Invited Speaker	Topic
Marc De Graef Carnegie Mellon University	Introduction to crystallography and electron diffraction
John Mansfield University of Michigan	Case studies utilizing crystallography and electron diffraction
Joe Michael Sandia National Laboratories	Introduction to EBSD for orientation mapping
Ute Kolb University of Mainz	Precession diffraction for TEM-based orientation mapping
Ben Britton Imperial College	Strain mapping by HR-EBSD
Jiong Zhang Intel Inc.	TEM-based strain mapping
Michael Mills Ohio State University	Imaging defects by diffraction contrast in the TEM
Yoosuf Picard Carnegie Mellon University	Non-destructive defect imaging in the SEM

Overview

- Introductory remarks
- Basic crystallographic concepts
- Diffraction basics
- Dynamical electron scattering
- Basics of cross-correlations

A dual view of the world

S

measuring stick

-> distance between trees: d = 1 [S]

S'

-> distance between trees: d = 1/2 [S']

numerical value of d decreases when length of measuring stick increases

-> d is contravariant quantity

real or direct space

S

measuring stick

-> # trees per unit S: r = 2 [S^{-1}]

S'

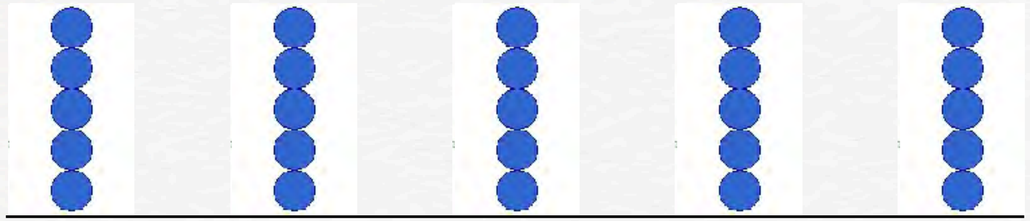
-> # trees per unit S': r = 4 [S'^{-1}]

numerical value of r increases when length of measuring stick increases

-> r is covariant quantity

reciprocal space

A dual view of the world

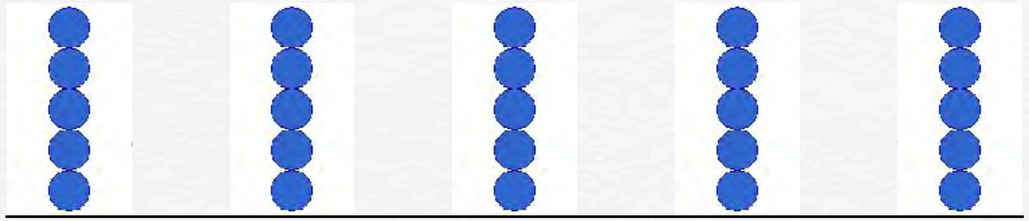


distance between lattice planes:
d [nm]

we use distances to describe the coordinates of atoms, usually scaled in appropriate units

-> we need a tool to compute distances, angles, etc...

real or direct space



lattice plane “lineal density”:
g [nm^{-1}]

[nm^{-1}] is also the unit of a spatial gradient (d/dx), and a gradient evokes the concept of “normal”...

-> we need a tool to compute normals and lineal densities

reciprocal space

Real or Direct Space

the contravariant view

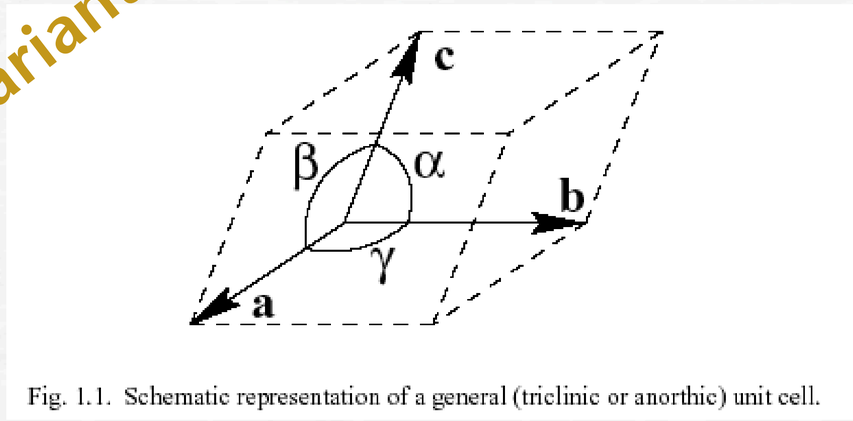


Fig. 1.1. Schematic representation of a general (triclinic or anorthic) unit cell.

- 3-D lattices are represented by a parallelepiped or unit cell;
- the edge lengths and angles between the edges make up the lattice parameters;

$$\{a, b, c, \alpha, \beta, \gamma\}$$

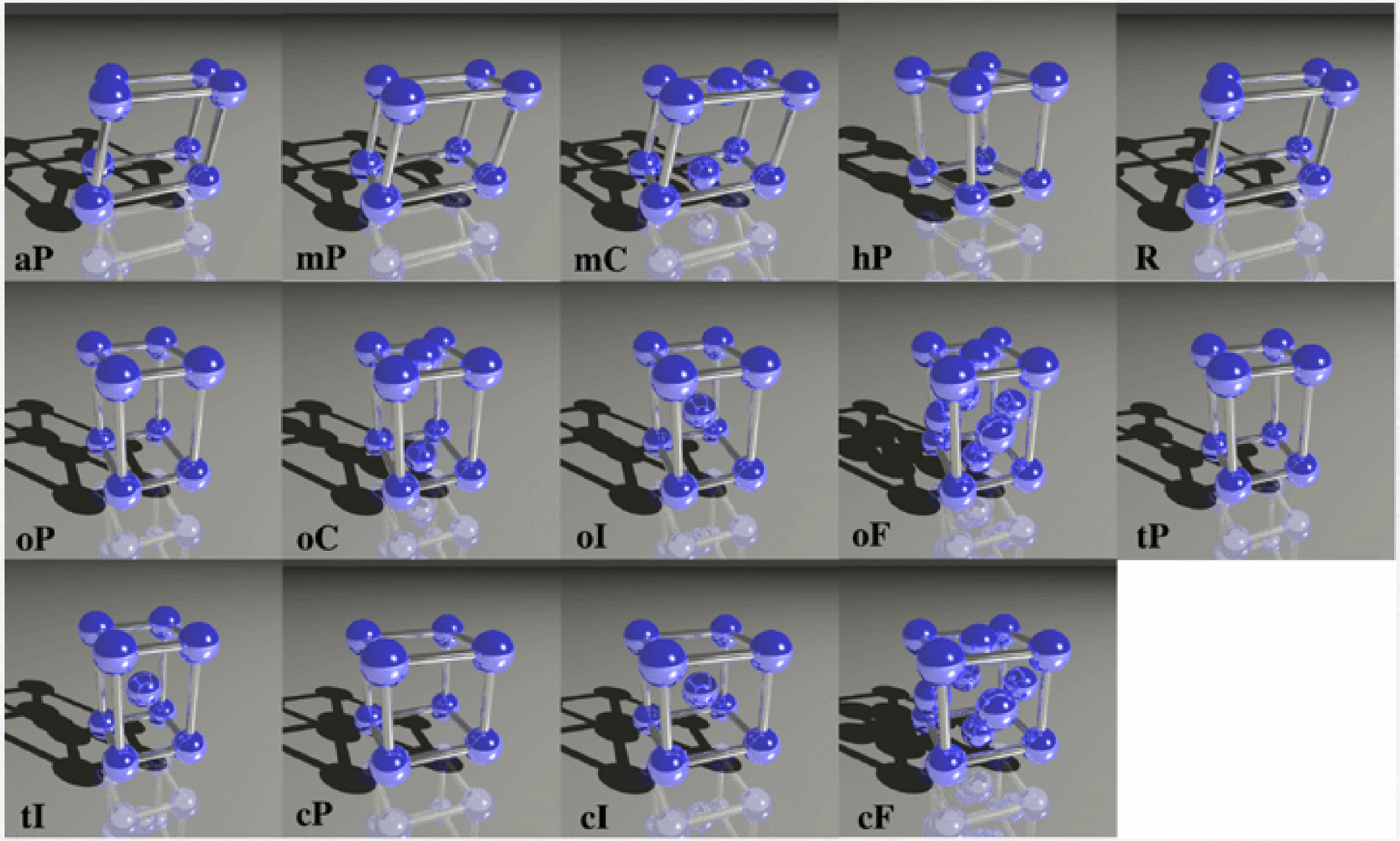
- There are seven different unit cell types, known as the crystal systems:

Parallelepiped :

"a 6-faced polyhedron all of whose faces are parallelograms lying in pairs of parallel planes"

$\{a, b, c, \alpha, \beta, \gamma\}$	$a \neq b \neq c; \alpha \neq \beta \neq \gamma$	triclinic or anorthic (a);
$\{a, b, c, \frac{\pi}{2}, \beta, \frac{\pi}{2}\}$	$a \neq b \neq c; \beta \neq \frac{\pi}{2}$	monoclinic (m);
$\{a, a, c, \frac{\pi}{2}, \frac{\pi}{2}, \frac{2\pi}{3}\}$	$a = b \neq c$	hexagonal (h);
$\{a, a, a, \alpha, \alpha, \alpha\}$	$a = b = c; \alpha \neq \frac{\pi}{2}$	rhombohedral (R);
$\{a, b, c, \frac{\pi}{2}, \frac{\pi}{2}, \frac{\pi}{2}\}$	$a \neq b \neq c$	orthorhombic (o);
$\{a, a, c, \frac{\pi}{2}, \frac{\pi}{2}, \frac{\pi}{2}\}$	$a = b \neq c$	tetragonal (t);
$\{a, a, a, \frac{\pi}{2}, \frac{\pi}{2}, \frac{\pi}{2}\}$	$a = b = c$	cubic (c).

14 Bravais lattices



Lattice geometry

- Each lattice node can be reached by means of a translation from the origin.
- Such a translation vector is represented by:

$$\mathbf{t} = u\mathbf{a} + v\mathbf{b} + w\mathbf{c}$$

- Translation (or lattice) vectors are integer linear combinations of the basis vectors of the Bravais lattice.
- Short hand notation: [uvw] (known as direction indices)
- Often, we will use the Einstein summation convention to represent vectors in a compact way. A summation is implied over each subscript that occurs twice on the same side of the equation.

$$\mathbf{t} = \sum_{i=1}^{3} u_i \mathbf{a}_i = u_i \mathbf{a}_i$$

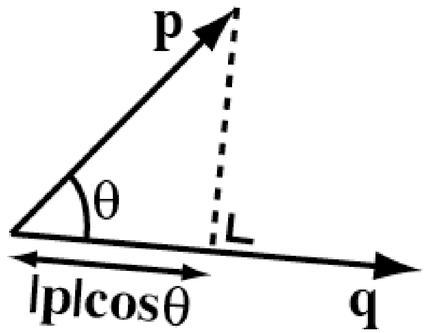
- Position vectors are written like this: the components are known as fractional coordinates.

$$\mathbf{r} = x\mathbf{a} + y\mathbf{b} + z\mathbf{c} = \sum_{i=1}^{3} r_i \mathbf{a}_i = r_i \mathbf{a}_i$$

Lattice geometry

- dot product definition:

$$\mathbf{p}\cdot\mathbf{q} \equiv |\mathbf{p}||\mathbf{q}|\cos\theta.$$



- the length of a vector is then equal to: $\mathbf{p}\cdot\mathbf{p} = |\mathbf{p}|^2$ from which follows: $|\mathbf{p}| = \sqrt{\mathbf{p}\cdot\mathbf{p}}$
- so, how do we compute a dot product in practice ?

$$|\mathbf{p}| = \sqrt{p_i\mathbf{a}_i \cdot p_j\mathbf{a}_j} = \sqrt{p_i(\mathbf{a}_i\cdot\mathbf{a}_j)p_j} \qquad \left(= \sqrt{\sum_{i=1}^{3}\sum_{j=1}^{3} p_i(\mathbf{a}_i\cdot\mathbf{a}_j)p_j}\right)$$

- The dot products between the basis vectors form a 3 by 3 matrix, known as the metric tensor:

$$g_{ij} = \begin{bmatrix} \mathbf{a}\cdot\mathbf{a} & \mathbf{a}\cdot\mathbf{b} & \mathbf{a}\cdot\mathbf{c} \\ \mathbf{b}\cdot\mathbf{a} & \mathbf{b}\cdot\mathbf{b} & \mathbf{b}\cdot\mathbf{c} \\ \mathbf{c}\cdot\mathbf{a} & \mathbf{c}\cdot\mathbf{b} & \mathbf{c}\cdot\mathbf{c} \end{bmatrix} = \begin{bmatrix} a^2 & ab\cos\gamma & ac\cos\beta \\ ab\cos\gamma & b^2 & bc\cos\alpha \\ ac\cos\beta & bc\cos\alpha & c^2 \end{bmatrix}$$

Lattice geometry

- using our short-hand notation, the length of a vector is then equal to: $|\mathbf{p}| = \sqrt{p_i g_{ij} p_j}$
- or, explicitly:

$$|\mathbf{p}| = \sqrt{[p_1\, p_2\, p_3] \begin{bmatrix} a^2 & ab\cos\gamma & ac\cos\beta \\ ab\cos\gamma & b^2 & bc\cos\alpha \\ ac\cos\beta & bc\cos\alpha & c^2 \end{bmatrix} \begin{bmatrix} p_1 \\ p_2 \\ p_3 \end{bmatrix}}$$

Valid for ALL crystal systems!

- similarly, the dot product between two vectors is: $\mathbf{p} \cdot \mathbf{q} = p_i \mathbf{a}_i \cdot q_j \mathbf{a}_j = p_i g_{ij} q_j$
- or, explicitly:

$$\mathbf{p} \cdot \mathbf{q} = [p_1\, p_2\, p_3] \begin{bmatrix} a^2 & ab\cos\gamma & ac\cos\beta \\ ab\cos\gamma & b^2 & bc\cos\alpha \\ ac\cos\beta & bc\cos\alpha & c^2 \end{bmatrix} \begin{bmatrix} q_1 \\ q_2 \\ q_3 \end{bmatrix}$$

- for the angle between two vectors we have:

$$\theta = \cos^{-1}\left(\frac{\mathbf{p}\cdot\mathbf{q}}{|\mathbf{p}||\mathbf{q}|}\right) = \cos^{-1}\left(\frac{p_i g_{ij} q_j}{\sqrt{p_i g_{ij} p_j}\sqrt{q_i g_{ij} q_j}}\right)$$

Examples

- for a tetragonal crystal with a= 1/2 nm and c=1 nm, compute the distance between the points (1/2,0,1/2) and (1/2,1/2,0).

first we need the metric tensor:

$$g_{ij} = \begin{bmatrix} \frac{1}{4} & 0 & 0 \\ 0 & \frac{1}{4} & 0 \\ 0 & 0 & 1 \end{bmatrix}$$

$$\begin{bmatrix} a^2 & ab\cos\gamma & ac\cos\beta \\ ab\cos\gamma & b^2 & bc\cos\alpha \\ ac\cos\beta & bc\cos\alpha & c^2 \end{bmatrix}$$

Answer : *The distance between two points is equal to the length of the vector connecting them, in this case* $\left(\frac{1}{2}-\frac{1}{2}, 0-\frac{1}{2}, \frac{1}{2}-0\right) = \left(0, -\frac{1}{2}, \frac{1}{2}\right)$. *Using the tetragonal metric tensor derived previously, we find for the length of this vector:*

$$|\mathbf{p}| = \sqrt{\begin{bmatrix} 0 & \frac{-1}{2} & \frac{1}{2} \end{bmatrix} \begin{bmatrix} \frac{1}{4} & 0 & 0 \\ 0 & \frac{1}{4} & 0 \\ 0 & 0 & 1 \end{bmatrix} \begin{bmatrix} 0 \\ \frac{-1}{2} \\ \frac{1}{2} \end{bmatrix}};$$

$$= \sqrt{\begin{bmatrix} 0 & \frac{-1}{2} & \frac{1}{2} \end{bmatrix} \begin{bmatrix} 0 \\ \frac{-1}{8} \\ \frac{1}{2} \end{bmatrix}} = \frac{\sqrt{5}}{4}\, nm.$$

Examples

- For the same crystal, compute the angle between the directions [120] and [311]

Answer : *The dot product is found from the expression for the metric tensor, as follows:*

$$\mathbf{t}_{[120]} \cdot \mathbf{t}_{[311]} = [1\,2\,0] \begin{bmatrix} \frac{1}{4} & 0 & 0 \\ 0 & \frac{1}{4} & 0 \\ 0 & 0 & 1 \end{bmatrix} \begin{bmatrix} 3 \\ 1 \\ 1 \end{bmatrix} = [1\,2\,0] \begin{bmatrix} \frac{3}{4} \\ \frac{1}{4} \\ 1 \end{bmatrix} = \frac{5}{4}\, nm^2.$$

The angle is found by dividing the dot product by the lengths of the vectors, $|[120]|^2 = \frac{5}{4}\, nm^2$ *and* $|[311]|^2 = \frac{14}{4}\, nm^2$*, from which we find*

$$\cos\theta = \frac{\frac{5}{4}}{\sqrt{\frac{14}{4}}\sqrt{\frac{5}{4}}} = \frac{5}{\sqrt{70}} \rightarrow \theta = 53.30°.$$

alternative:

Answer : *Consider the following formal relation:*

$$\begin{pmatrix} \mathbf{p} \\ \mathbf{q} \end{pmatrix} \cdot \begin{pmatrix} \mathbf{p} & \mathbf{q} \end{pmatrix} = \begin{pmatrix} \mathbf{p}\cdot\mathbf{p} & \mathbf{p}\cdot\mathbf{q} \\ \mathbf{q}\cdot\mathbf{p} & \mathbf{q}\cdot\mathbf{q} \end{pmatrix}.$$

The resulting 2×2 *matrix contains all three dot products needed for the computation of the angle* θ*, and only one set of matrix multiplications is needed. We can apply this short cut to the previous example:*

$$\begin{pmatrix} 1 & 2 & 0 \\ 3 & 1 & 1 \end{pmatrix} \begin{pmatrix} \frac{1}{4} & 0 & 0 \\ 0 & \frac{1}{4} & 0 \\ 0 & 0 & 1 \end{pmatrix} \begin{pmatrix} 1 & 3 \\ 2 & 1 \\ 0 & 1 \end{pmatrix} = \begin{pmatrix} \frac{5}{4} & \frac{5}{4} \\ \frac{5}{4} & \frac{14}{4} \end{pmatrix},$$

Reciprocal Space

the covariant view

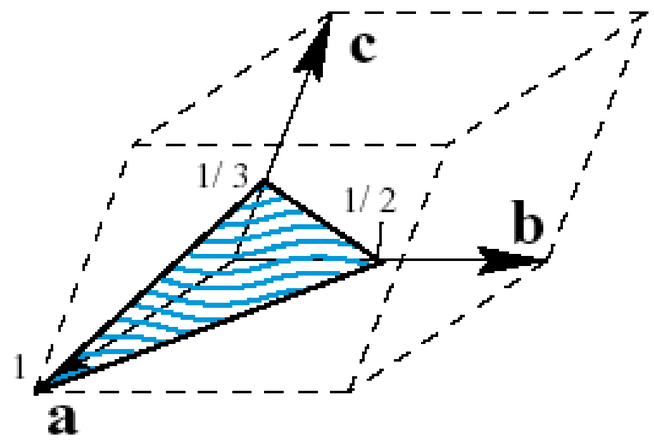


Fig. 1.3. Illustration of the determination of the Miller indices of a plane.

- Miller indices are determined as follows:
  - determine the intercepts of the plane with the basis vectors; (if plane is parallel to one or more basis directions, take intersection to be at infinity)
  - invert the intercepts;
  - reduce to common integers and write between parentheses.
- so, the plane on the left is the (123) plane.
- general notation: (hkl)

Reciprocal Space

- In direct space, we represent directions as vectors. It would be nice, if we could also represent planes by vectors (i.e., the normal to the plane). It would be even better if the components of these normal vectors would be equal to the Miller indices!
- We know that an integer linear combination of basis vectors results in a vector that is not usually normal to a plane with the same integers as Miller indices.
- So, we define a second coordinate system, in such a way that the Miller indices of a plane are the components of the normal to that plane. This new set of basis vectors is known as the reciprocal basis.
- The reciprocal basis vectors are defined by: $\mathbf{a}_i \cdot \mathbf{a}_j^* \equiv \delta_{ij}$
- Let's take a closer look at those new basis vectors.

Reciprocal Space

- The reciprocal basis vectors are defined as follows:

$$\begin{aligned}\mathbf{a}^* &= \frac{\mathbf{b}\times\mathbf{c}}{\mathbf{a}\cdot(\mathbf{b}\times\mathbf{c})};\\ \mathbf{b}^* &= \frac{\mathbf{c}\times\mathbf{a}}{\mathbf{a}\cdot(\mathbf{b}\times\mathbf{c})};\\ \mathbf{c}^* &= \frac{\mathbf{a}\times\mathbf{b}}{\mathbf{a}\cdot(\mathbf{b}\times\mathbf{c})}.\end{aligned}$$

- It is easy to see that a* is orthogonal to b and c, and that a* . a = 1.

- The reciprocal lattice is then the set of vectors of the type:

$$\mathbf{g} = h\mathbf{a}^* + k\mathbf{b}^* + l\mathbf{c}^* = \sum_{i=1}^{3} g_i\mathbf{a}_i^* = g_i\mathbf{a}_i^*$$

Reciprocal Space

- One can show that the reciprocal lattice vectors have the following properties:
  - the reciprocal lattice vector $\mathbf{g}$, with components (h,k,l), is orthogonal to the plane with Miller indices (hkl);
  - the reciprocal lattice vector $\mathbf{g}$, with components (h,k,l), has as its length the inverse of the distance between the planes (hkl).

$$|\mathbf{g}_{hkl}| = \frac{1}{d_{hkl}}$$

lineal density

- We can use the metric tensor formalism to compute the interplanar spacing:

$$\frac{1}{d_{hkl}} = |\mathbf{g}| = \sqrt{\mathbf{g} \cdot \mathbf{g}} = \sqrt{(g_i \mathbf{a}_i^*) \cdot (g_j \mathbf{a}_j^*)} = \sqrt{g_i (\mathbf{a}_i^* \cdot \mathbf{a}_j^*) g_j}$$

Define the reciprocal metric tensor as

$$g_{ij}^* \equiv \mathbf{a}_i^* \cdot \mathbf{a}_j^*$$

Reciprocal Space

- and we find:

$$\frac{1}{d_{hkl}} = |\mathbf{g}| = \sqrt{\mathbf{g} \cdot \mathbf{g}} = \sqrt{g_i g_{ij}^* g_j}$$

- and for the angle between two reciprocal lattice vectors (i.e., plane normals) we have:

$$\theta = \cos^{-1}\left(\frac{f_i g_{ij}^* g_j}{\sqrt{f_i g_{ij}^* f_j}\sqrt{g_i g_{ij}^* g_j}}\right)$$

- One can show that the direct and reciprocal metric tensors are each others inverse, so once the direct metric tensor is known from the lattice parameters, a simple matrix inversion results in the reciprocal metric tensor g*

Example

- Compute the angle between the (120) and (311) plane normals for the tetragonal crystal from slide 10.

$$
\begin{aligned}
\cos\theta &= \frac{[1\,2\,0]\begin{bmatrix}4&0&0\\0&4&0\\0&0&1\end{bmatrix}\begin{bmatrix}3\\1\\1\end{bmatrix}}{\sqrt{[1\,2\,0]\begin{bmatrix}4&0&0\\0&4&0\\0&0&1\end{bmatrix}\begin{bmatrix}1\\2\\0\end{bmatrix}}\sqrt{[3\,1\,1]\begin{bmatrix}4&0&0\\0&4&0\\0&0&1\end{bmatrix}\begin{bmatrix}3\\1\\1\end{bmatrix}}}, \\
&= \frac{20}{\sqrt{20\times 41}} = 0.69843, \\
\rightarrow \theta &= 45.7^\circ.
\end{aligned}
$$

Diffraction Experiments

- The "unknowns" in crystallography are the lattice parameters, the atom coordinates, the crystal symmetry, and the lattice orientation w.r.t. an external reference frame.
- Diffraction techniques allow us to determine all these quantities.
- in Materials Science, we use photons, neutrons and electrons for diffraction experiments
  - important distinction 1: neutrons and photons (x-rays) have similar wave lengths (0.1 nm), electron wave lengths are 10-100 times shorter (1-10 pm);
  - important distinction 2: electrons interact very strongly with matter, much stronger than neutrons and photons.
- Diffraction modalities are conveniently described in reciprocal space.

The de Broglie relation

- Louis de Broglie (1924) postulated a relation between a particle's momentum and the wavelength of the quantum mechanical wave associated with that particle: $\lambda = \frac{h}{p}$
- This is known as the particle-wave duality.
- We introduce the wave vector $\mathbf{k}$ as a vector directed along the travel direction of the particle, and with length equal to the inverse of the wavelength. The de Broglie relation is then rewritten as: $\mathbf{p} = h\mathbf{k}$
- Since the wave vector has dimensions of reciprocal length, it belongs to reciprocal space. Also, since $\mathbf{p}$ and $\mathbf{k}$ are proportional, this means that, apart from a scaling factor, reciprocal space and momentum space are identical.

Basic Diffraction Law

Bragg's Law

$$2d_{hkl}\sin\theta = \lambda$$

$$\mathbf{k}' = \mathbf{k} + \mathbf{g}$$

Necessary condition for diffraction to occur.

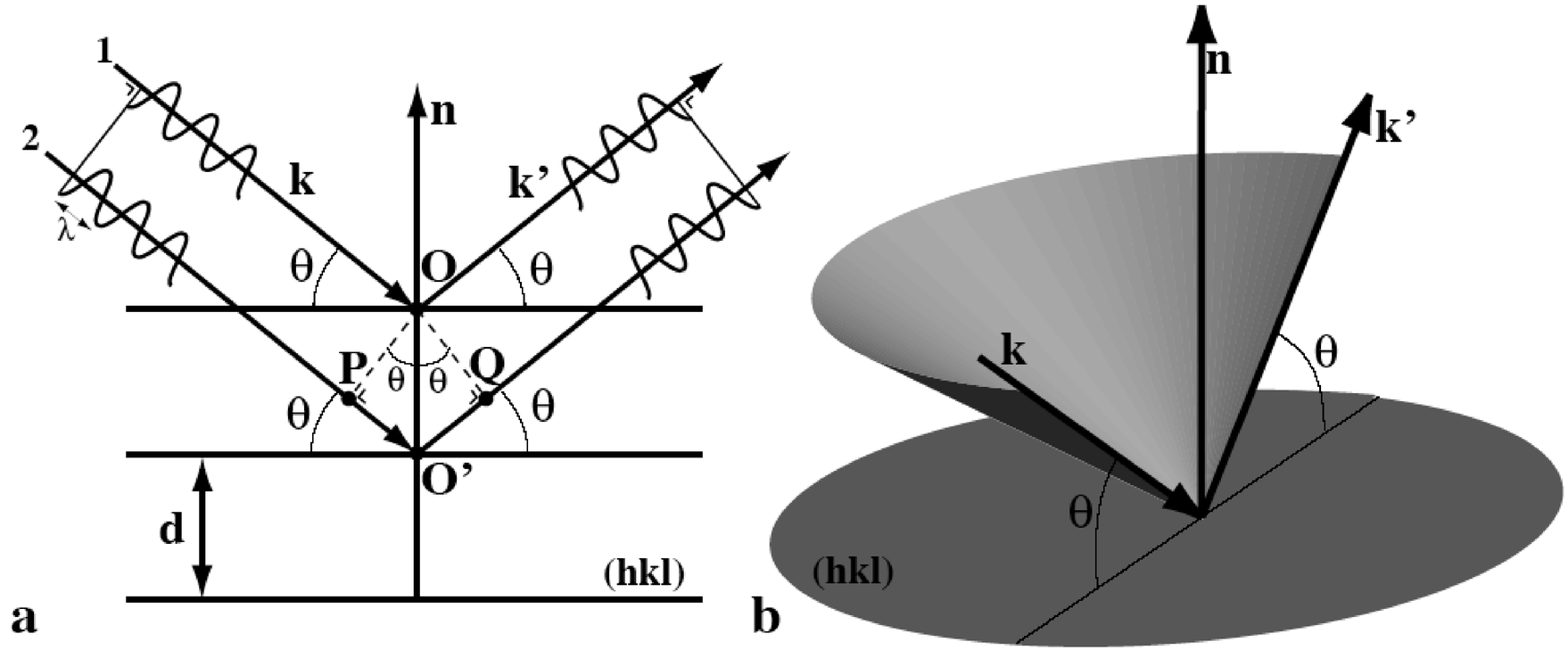


Fig. 2.2. a) Geometrical construction leading to the direct space Bragg equation; b) the incident and diffracted directions and the plane normal must lie in a planar section through a conical surface with top in the plane.

Electron Wave Length

- Classical expression for the electron wavelength follows from equating the kinetic energy to the potential energy due to a potential drop E:

$$\lambda_{nr} = \frac{h}{\sqrt{2m_0 eE}} = \frac{1,226.39}{\sqrt{E}}$$

- For low accelerating voltages, the non-relativistic expression is quite accurate:

E (Volt)	λ_{nr} (pm)	λ (pm)
100	122.64	122.63
500	54.84	54.83
1,000	38.78	38.76
5,000	17.34	17.30
10,000	12.26	12.20
20,000	8.67	8.59

relativistic wavelength

$$\lambda = \frac{h}{\sqrt{2m_0 eE(1 + \frac{e}{2m_0 c^2}E)}}$$

Table 2.2. *Relativistic acceleration potential $\hat{\Psi}$, electron wavelength λ, wavenumber $K_0 = \frac{1}{\lambda}$, mass ratio $\gamma = m/m_0$, relative velocity $\beta = \frac{v}{c}$, and interaction constant σ for various acceleration voltages E.*

E (kV)	$\hat{\Psi}$ (V)	λ (pm)	K_0 (nm^{-1})	m/m_0	$\beta = v/c$	σ (V^{-1}nm^{-1})
100	109,784	3.701	270.165	1.196	0.548	0.009244
120	134,090	3.349	298.577	1.235	0.587	0.008638
200	239,139	2.508	398.734	1.391	0.695	0.007288
300	388,062	1.969	507.937	1.587	0.777	0.006526
400	556,556	1.644	608.293	1.783	0.828	0.006121
800	1,426,224	1.027	973.761	2.566	0.921	0.005503
1,000	1,978,475	0.872	1,146.895	2.957	0.941	0.005385
1,250	2,778,867	0.736	1,359.228	3.446	0.957	0.005296
2,000	5,913,900	0.504	1,982.876	4.914	0.979	0.005176
3,000	11,806,277	0.357	2,801.657	6.871	0.989	0.005122

Ewald Sphere Construction

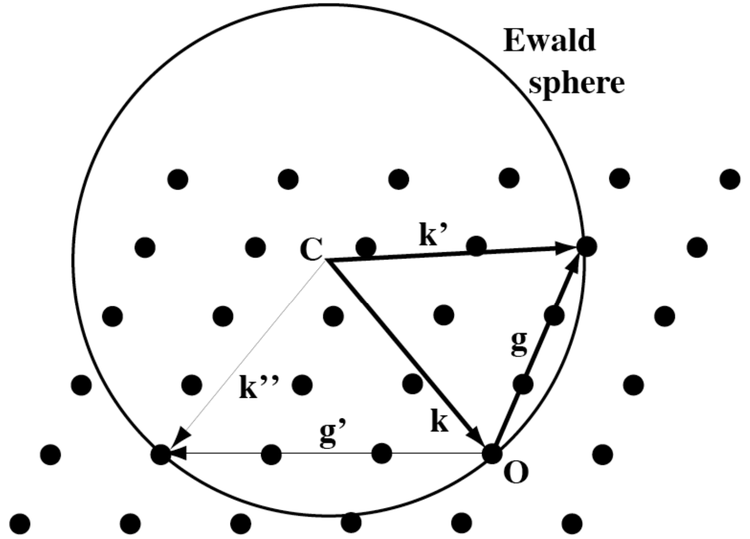


Fig. 2.4. Ewald sphere construction.

X-rays & neutrons

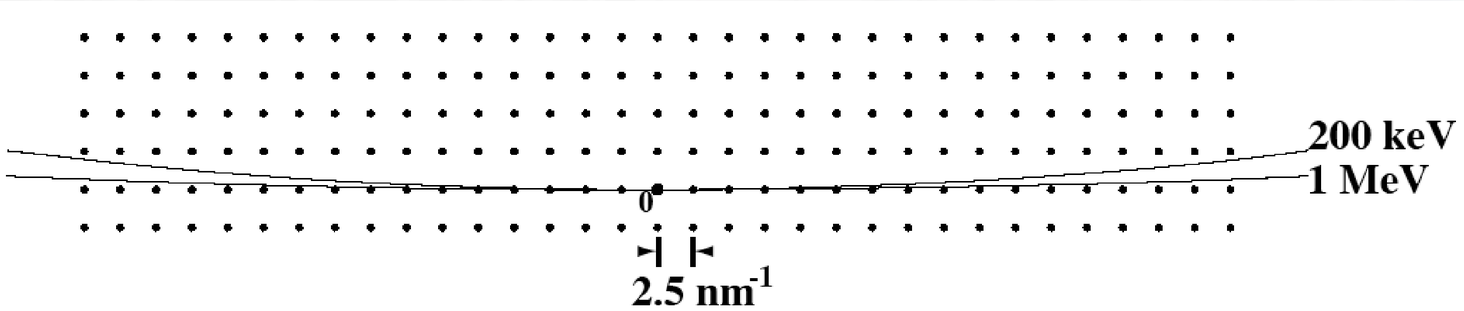


Fig. 2.7. Ewald sphere drawn to scale for the reciprocal lattice of a square crystal with lattice parameter 0.4 nm, and a 200 keV and 1 MeV incident electron beam.

electrons

Table 2.3. *Diffraction angles 2θ for the (200), (400), and (600) lattice planes in Aluminum for $E = 200$ kV and $E = 1$ MV in mrad (degrees). The last column shows the corresponding angles for x-ray diffraction using Cu-K_α radiation with $\lambda = 0.1542838$ nm; the (600) planes do not give rise to a diffracted beam for this wavelength.*

Plane	$2\theta_{200\text{ kV}}$	$2\theta_{1\text{ MeV}}$	*Cu-K_α* x-rays
(200)	12.38(0.71)	4.31(0.25)	781.31 (44.76)
(400)	24.77(1.42)	8.61(0.49)	1,731.52 (99.21)
(600)	37.16(2.13)	12.92(0.74)	—

EM modalities

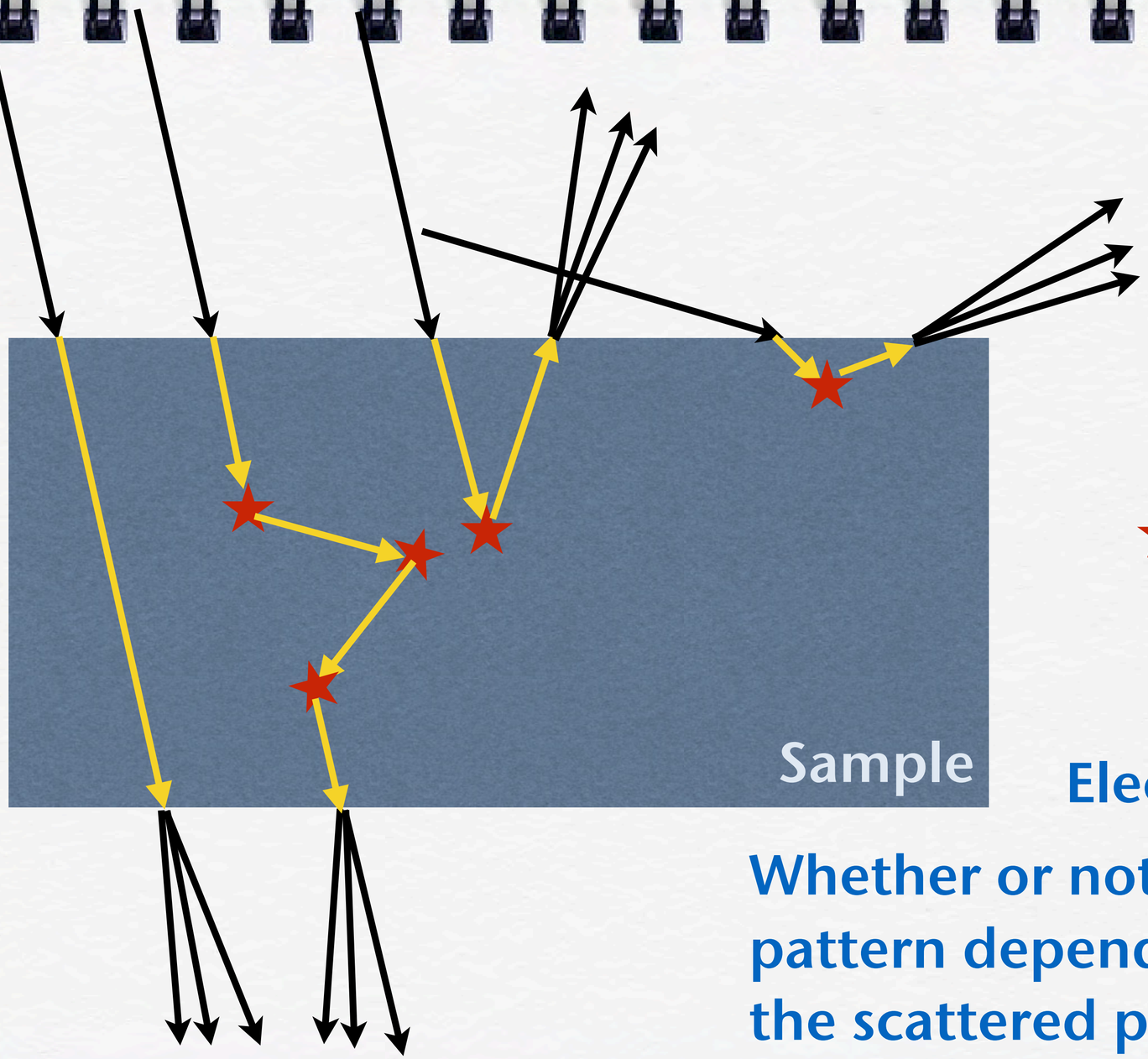
- Diffraction
  - SEM: EBSD, ECP — Michael, Britton
  - TEM: ZADP, CBED, PED, … — Mansfield, Zhang; Kolb
- Imaging
  - SEM: SE, BSE, ISE, … — Picard
  - TEM: DCI, HREM, … — Mills
- Spectroscopy (not considered here)

All of these can be described by a single theory, with adaptations for the various geometries…

EM modalities

- Elastic: Bragg (dynamical), Rutherford (Z)
  - (differential) scattering cross sections are known (f, Z^2)
- Inelastic: collective excitations (phonons, plasmons, etc), core and outer-shell excitations
  - These can be modeled, but often they are replaced by an effective (phenomenological) absorption potential
  - Often, the models involve Monte Carlo simulations to account for the stochastic nature of these processes

Simplistic view



Channeling (dynamical)

★ Inelastic events (stochastic)

Electrons ALWAYS channel !

Whether or not we observe a diffraction pattern depends on how we interrogate the scattered plume of electrons...

QM governing equation

kinetic total potential

$$\Delta\Psi + 4\pi^2 k_0^2 \Psi = -4\pi^2 \left[U + \mathrm{i}U'\right] \Psi,$$

wave function

$$U(\mathbf{r}) + \mathrm{i}U'(\mathbf{r}) \equiv \frac{2me}{h^2} \left(V'(\mathbf{r}) + \mathrm{i}W(\mathbf{r})\right)$$

electrostatic lattice potential absorption potential

periodic functions !

$$\begin{aligned} U(\mathbf{r}) &= \frac{2me}{h^2} \sum_{\mathbf{g}\neq 0} V_{\mathbf{g}} e^{2\pi \mathrm{i}\mathbf{g}\cdot\mathbf{r}}; \\ U'(\mathbf{r}) &= \frac{2me}{h^2} \sum_{\mathbf{g}} W_{\mathbf{g}} e^{2\pi \mathrm{i}\mathbf{g}\cdot\mathbf{r}}. \end{aligned}$$

$$k_0 \equiv \frac{\sqrt{2me\hat{\Psi}_c}}{h}.$$

$$\hat{\Psi}_c = \hat{\Psi} + \gamma V_0$$

Relativistic wave number

Simplifying Assumptions

- High Energy Approximation
  - turns equation into first-order dif. eq.
- Perfect flat crystal
  - eliminates the x,y derivatives, resulting in z-derivative only
- Column approximation
  - electron does not leave a vertical column through the side-faces

Solution Methods

- Superposition of plane waves with wave vectors according to Bragg equation

$$\Psi(\mathbf{r}) = \sum_{\mathbf{g}} \psi_{\mathbf{g}} e^{2\pi \mathrm{i}(\mathbf{k}_0+\mathbf{g})\cdot\mathbf{r}}$$

Coupled Diff. Eqs.

- Superposition of plane waves with periodicity of lattice (Bloch waves)

$$\Psi(\mathbf{r}) = C(\mathbf{r}) e^{2\pi \mathrm{i}\mathbf{k}\cdot\mathbf{r}} = \sum_{\mathbf{g}} C_{\mathbf{g}} e^{2\pi \mathrm{i}(\mathbf{k}+\mathbf{g})\cdot\mathbf{r}}$$

Eigenvalue problem

Coupled Diff. Eqs.

- Darwin-Howie-Whelan equations

Beam *g* amplitude

$$\frac{\mathrm{d}\psi_{\mathbf{g}}}{\mathrm{d}z} - 2\pi \mathrm{i} s_{\mathbf{g}} \psi_{\mathbf{g}} = \mathrm{i}\pi \sum_{\mathbf{g}'} \frac{e^{\mathrm{i}\theta_{\mathbf{g}-\mathbf{g}'}}}{q_{\mathbf{g}-\mathbf{g}'}} \psi_{\mathbf{g}'}.$$

Excitation error

Interaction parameter

$$\frac{1}{q_{\mathbf{g}}} \equiv \frac{1}{\xi_{\mathbf{g}}} + \mathrm{i}\frac{e^{\mathrm{i}\beta_{\mathbf{g}}}}{\xi'_{\mathbf{g}}}$$

$$\text{(extinction distance)}^{-1} \rightarrow \quad \frac{1}{\xi_{\mathbf{g}}} \equiv \frac{|U_{\mathbf{g}}|}{|\mathbf{k}_0 + \mathbf{g}| \cos\alpha};$$

$$\text{(absorption length)}^{-1} \rightarrow \quad \frac{1}{\xi'_{\mathbf{g}}} \equiv \frac{|U'_{\mathbf{g}}|}{|\mathbf{k}_0 + \mathbf{g}| \cos\alpha}.$$

Coupled Diff. Eqs.

- Darwin-Howie-Whelan equations

$$\frac{\mathrm{d}\psi_{\mathbf{g}}}{\mathrm{d}z} - 2\pi \mathrm{i} s_{\mathbf{g}} \psi_{\mathbf{g}} = \mathrm{i}\pi \sum_{\mathbf{g}'} \frac{e^{\mathrm{i}\theta_{\mathbf{g}-\mathbf{g}'}}}{q_{\mathbf{g}-\mathbf{g}'}} \psi_{\mathbf{g}'}.$$

typically rewritten as a matrix equation

$$\frac{\mathrm{d}\mathbf{S}}{\mathrm{d}z} = \mathrm{i}\mathcal{A}\mathbf{S}$$

$$\mathcal{A}_{nn} = 2\pi s_n;$$

$$\mathcal{A}_{nn'} = \frac{\pi}{q_{n-n'}} \qquad n \neq n'.$$

Structure Matrix

which has an exponential solution:

$$\mathbf{S}(z_0) = e^{\mathrm{i}\mathcal{A}z_0}\mathbf{S}(0) \equiv \mathcal{S}\mathbf{S}(0)$$

Scattering Matrix

Bloch waves

- Bloch wave eigenvalue equation

$$2k_0 s_{\mathbf{g}} C_{\mathbf{g}}^{(j)} + \sum_{\mathbf{h}\neq\mathbf{g}} U_{\mathbf{g}-\mathbf{h}} C_{\mathbf{h}}^{(j)} = 2k_n \gamma^{(j)} C_{\mathbf{g}}^{(j)}$$

$$\begin{pmatrix} \mathrm{i}U'_0 & U_{-\mathbf{g}} + \mathrm{i}U'_{-\mathbf{g}} & \dots & U_{-\mathbf{h}} + \mathrm{i}U'_{-\mathbf{h}} \\ U_{\mathbf{g}} + \mathrm{i}U'_{\mathbf{g}} & 2k_0 s_{\mathbf{g}} + \mathrm{i}U'_0 & \dots & U_{\mathbf{g}-\mathbf{h}} + \mathrm{i}U'_{\mathbf{g}-\mathbf{h}} \\ \vdots & \vdots & \ddots & \vdots \\ U_{\mathbf{h}} + \mathrm{i}U'_{\mathbf{h}} & U_{\mathbf{h}-\mathbf{g}} + \mathrm{i}U'_{\mathbf{h}-\mathbf{g}} & \dots & 2k_0 s_{\mathbf{h}} + \mathrm{i}U'_0 \end{pmatrix} \begin{pmatrix} C_{\mathbf{0}}^{(j)} \\ C_{\mathbf{g}}^{(j)} \\ \vdots \\ C_{\mathbf{h}}^{(j)} \end{pmatrix} = 2k_n \Gamma^{(j)} \begin{pmatrix} C_{\mathbf{0}}^{(j)} \\ C_{\mathbf{g}}^{(j)} \\ \vdots \\ C_{\mathbf{h}}^{(j)} \end{pmatrix}$$

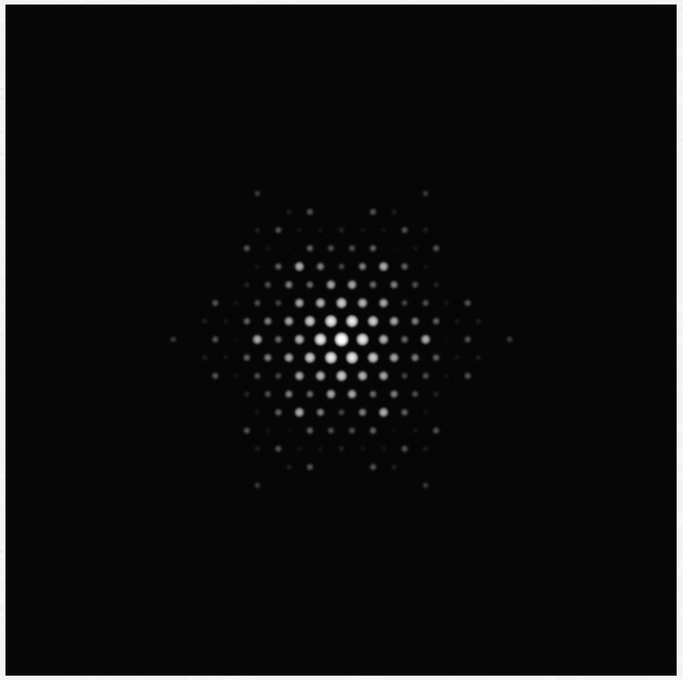
$$\Psi(\mathbf{r}) = \sum_j \alpha^{(j)} \sum_{\mathbf{g}} C_{\mathbf{g}}^{(j)} e^{2\pi\mathrm{i}(\mathbf{k}^{(j)}+\mathbf{g})\cdot\mathbf{r}} = \sum_j \alpha^{(j)} C^{(j)}(\mathbf{r}) e^{2\pi\mathrm{i}\mathbf{k}^{(j)}\cdot\mathbf{r}}$$

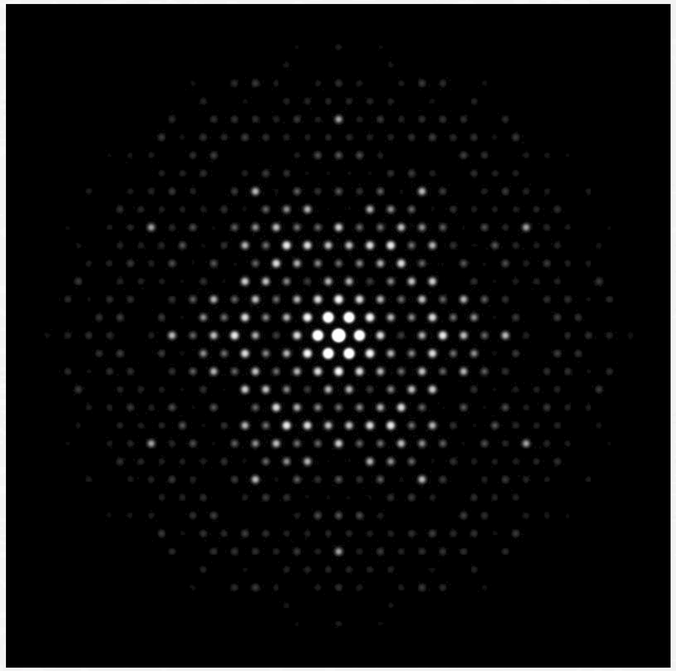
Perfect Crystal Examples

Ute Kolb

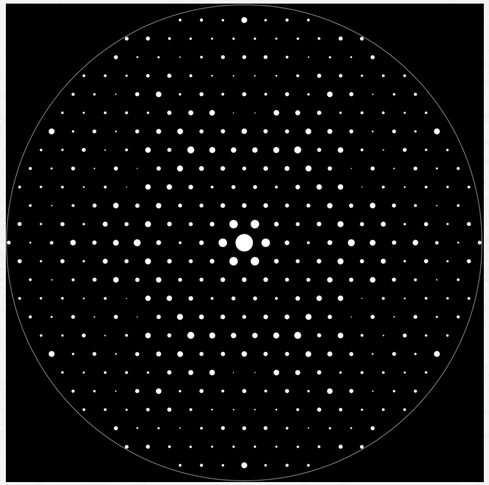
- ZADP vs. beam tilt for Beryl, dynamical PED simulation (200 kV)

Beryl





kinematical

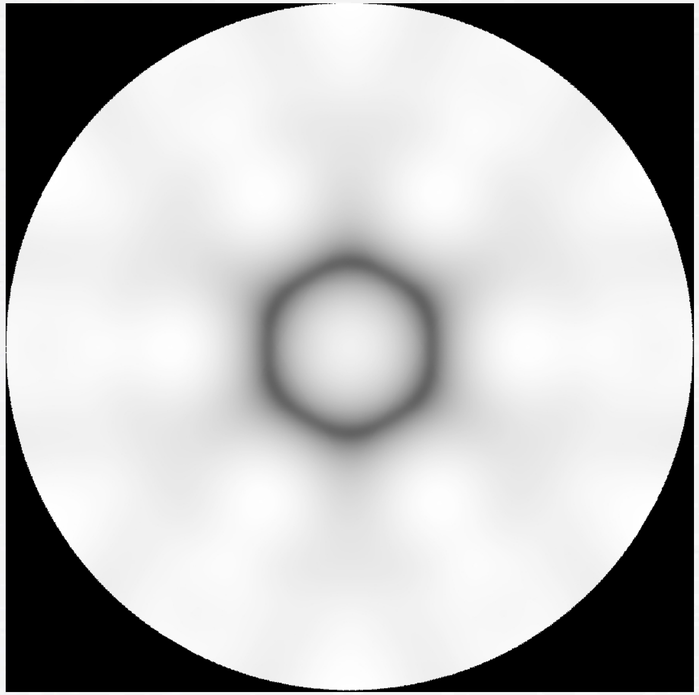


parallel incident beam, focused in back focal plane

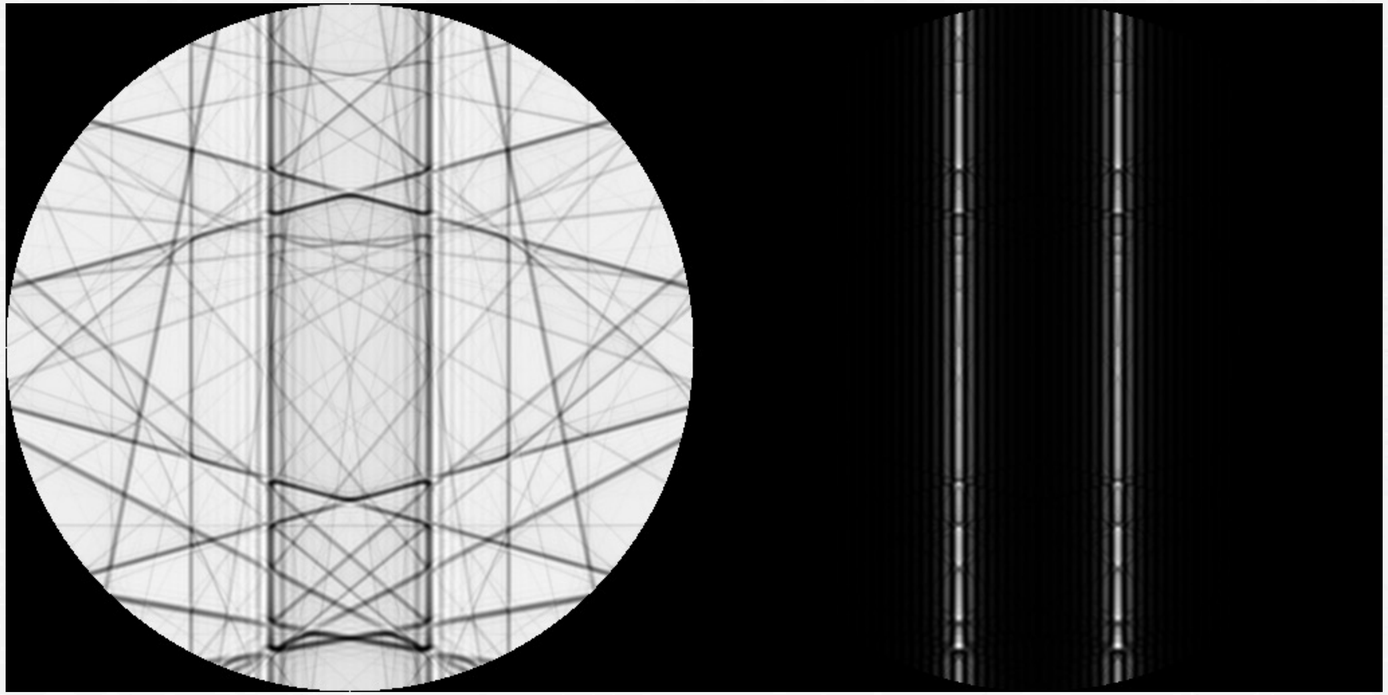
Perfect Crystal Examples

John Mansfield
Jiong Zhang

- CBED simulation: separate ZADP simulation for each incident beam direction followed by combining all (shifted) zaps into a CBED pattern



Si [111], 200 kV



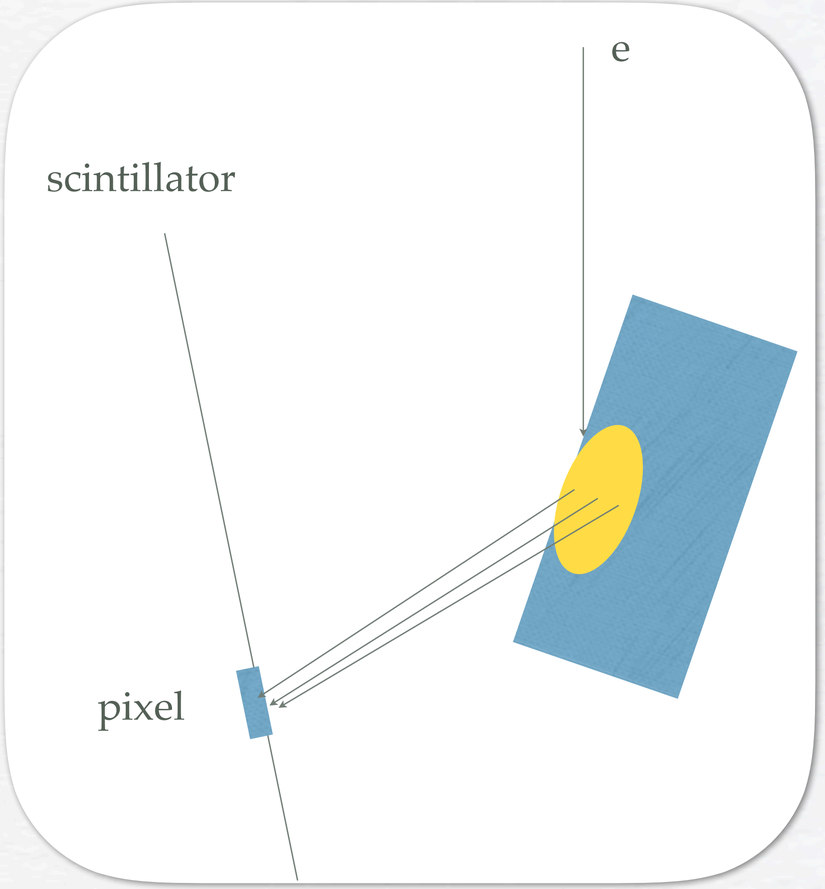
Si [230], 200 kV; 004 DF

converged incident beam, focused on sample

SEM diffraction modalities

- Let's consider BSEs only, created by Rutherford scattering events
- scattering cross section proportional to Z^2
- Since scattering events are stochastic, we need to integrate the probabilities over depth inside the sample.

$$\mathcal{P}(\mathbf{k}) = \sum_i \frac{Z_i^2 D_i}{z_0} \int_0^{z_0} \mathrm{d}z \, |\Psi_{\mathbf{k}}(\mathbf{r}_i)|^2$$



SEM diffraction modalities

$$\mathcal{P}(\mathbf{k}_0) = \sum_{\mathbf{g}} \sum_{\mathbf{h}} S_{\mathbf{gh}} L_{\mathbf{gh}},$$

$$S_{\mathbf{gh}} \equiv \sum_{n} \sum_{i \in \mathcal{S}_n} Z_n^2 \, e^{-M^{(n)}_{\mathbf{h}-\mathbf{g}}} \, e^{2\pi \mathrm{i}(\mathbf{h}-\mathbf{g})\cdot\mathbf{r}_i};$$

$$L_{\mathbf{gh}} \equiv \sum_{j} \sum_{k} C^{(j)*}_{\mathbf{g}} \alpha^{(j)*} \mathcal{I}_{jk} \alpha^{(k)} C^{(k)}_{\mathbf{h}}.$$

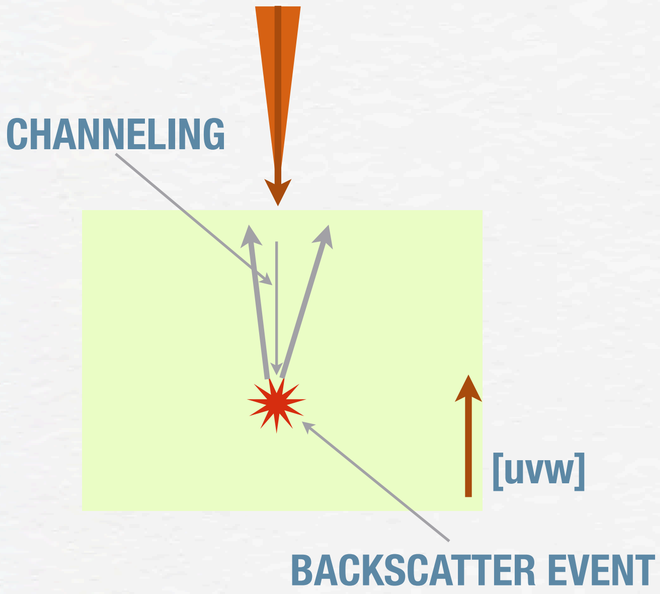
The same formalism works for EBSD, ECP, and also the TEM-based ALCHEMI technique...

$$\mathcal{I}_{jk} = \frac{1}{z_0(E)} \int_0^{z_0(E)} \lambda(E,z) e^{-2\pi(\alpha_{jk}+\mathrm{i}\beta_{jk})} \, \mathrm{d}z$$

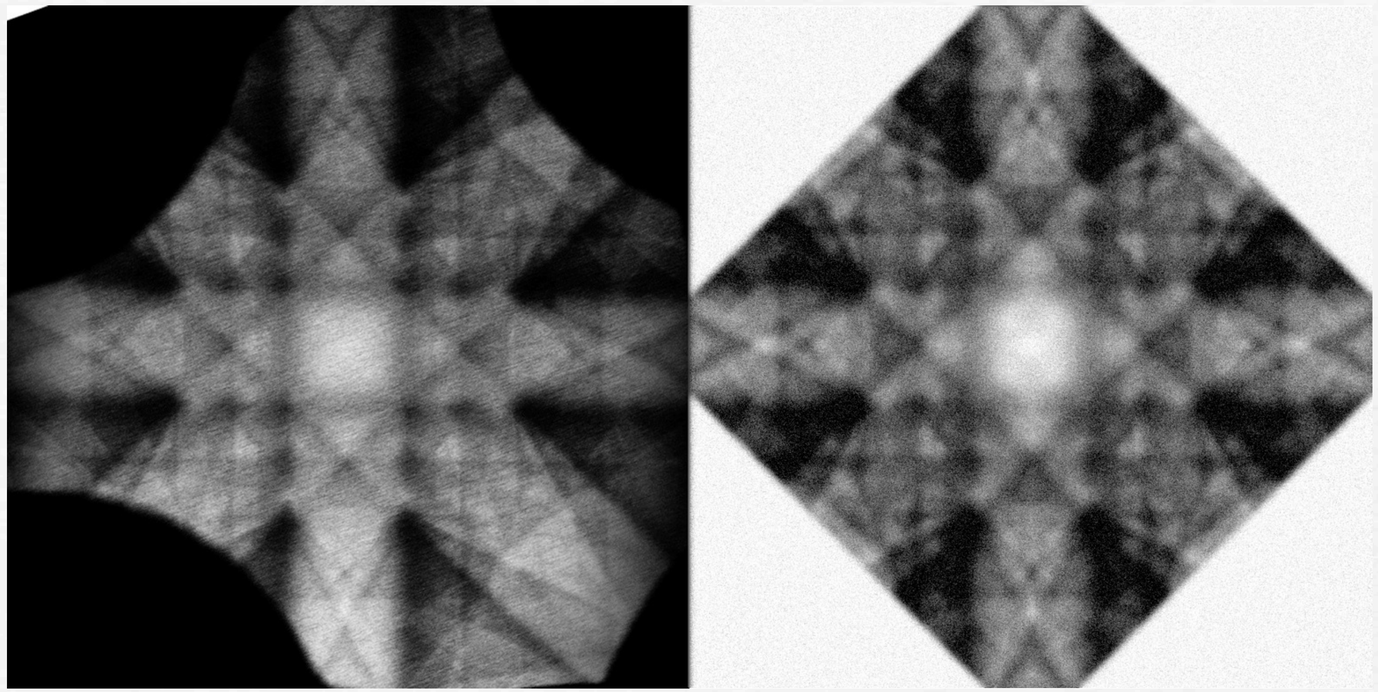
from Monte Carlo simulations

SEM diffraction modalities

Yoosuf Picard



ECP



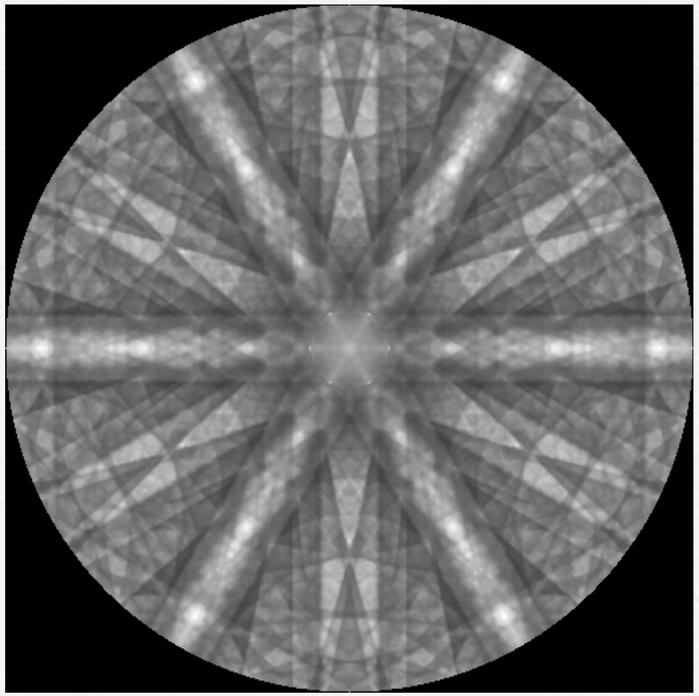
GaSb [001], 20kV

simulated pattern blurred
to resemble experimental pattern

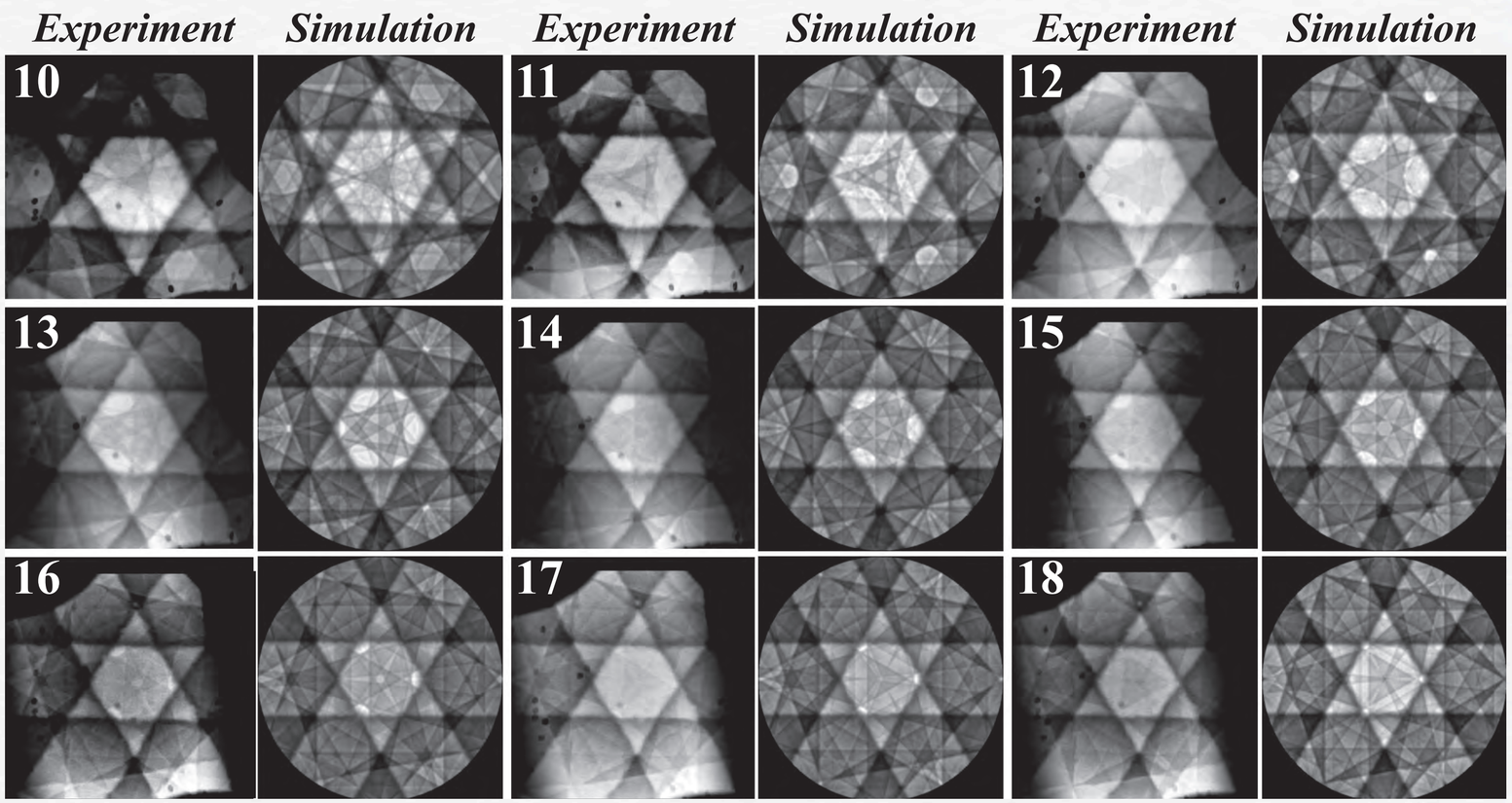
focused incident beam, rocked inside cone with apex on sample

Perfect Crystal Examples

- Electron Channeling Patterns (ECP), SEM, annular BSE detector



$BaMnO_3$, [00.1]



Al, [111]

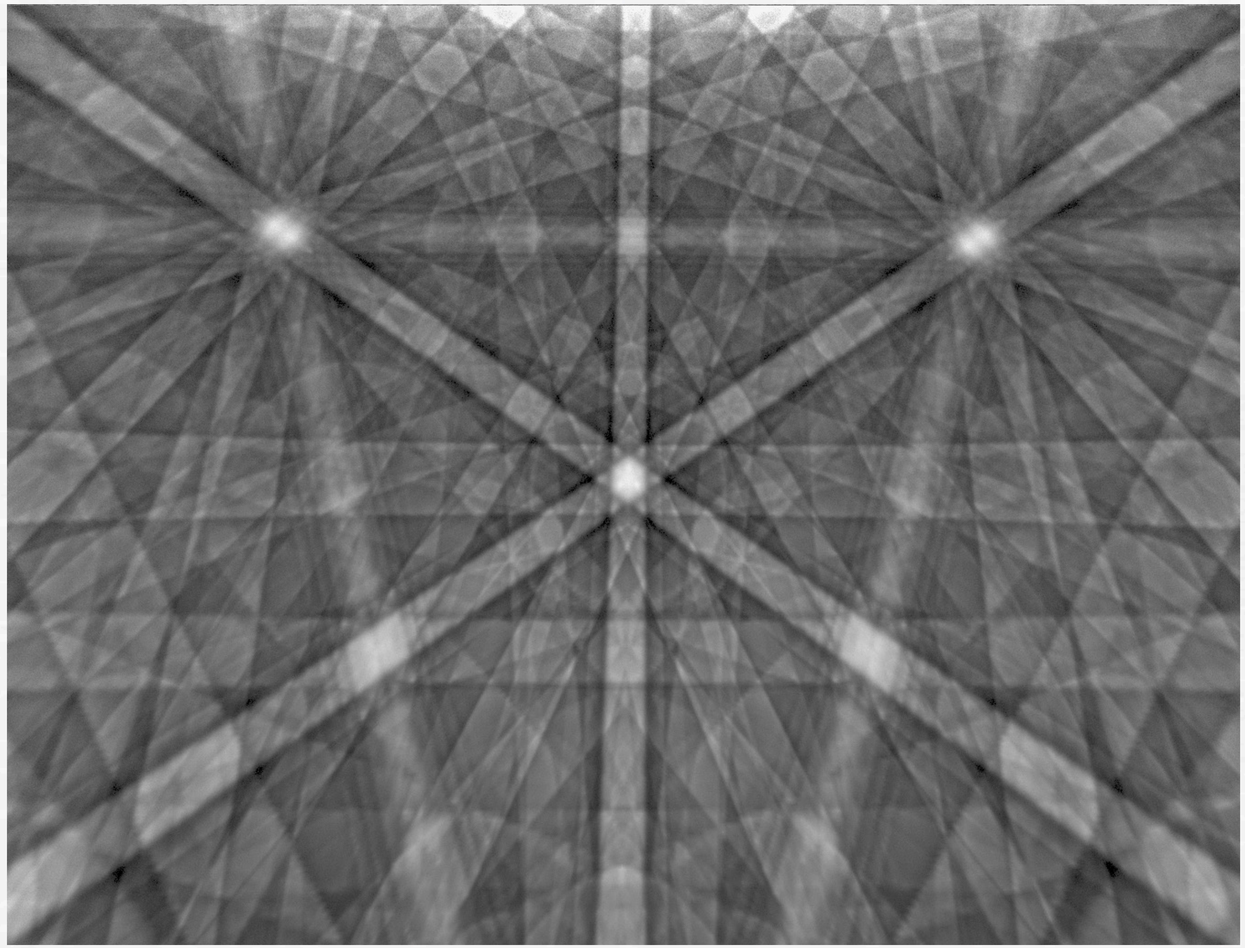
focused incident beam, rocked inside cone with apex on sample

Perfect Crystal Examples

Joe Michael

EBSD

Si [111]



focused beam, scanned across region of interest

Defects in crystals

- A defect causes a local perturbation in the lattice potential, which manifests itself as a phase shift of the Fourier coefficients of the potential…

$$V(\mathbf{r}) = \sum_{\mathbf{g}} V_{\mathbf{g}}\, e^{\mathrm{i}\mathbf{g}\cdot\mathbf{r}}$$

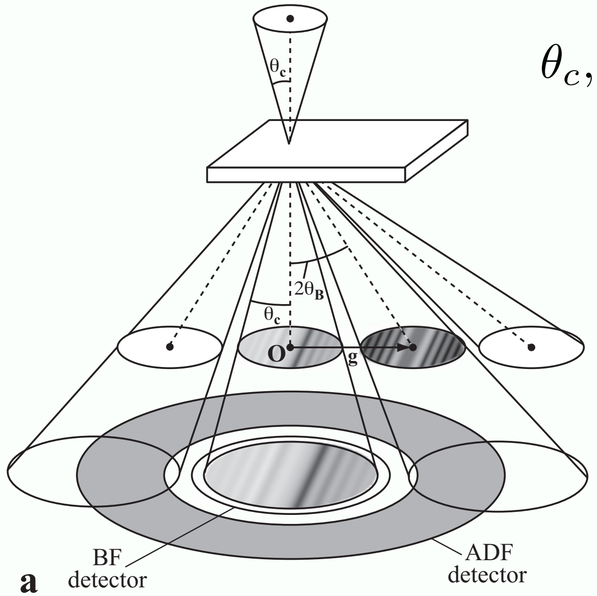
$$V_{\mathbf{g}} \rightarrow V_{\mathbf{g}}\, e^{-\mathrm{i}\alpha_{\mathbf{g}}(\mathbf{r})} \quad \text{with} \quad \alpha_{\mathbf{g}}(\mathbf{r}) \equiv 2\pi\mathbf{g}\cdot\mathbf{R}(\mathbf{r})$$

- The solution methods are still valid, but the sample is now “sliced” into thin slices, with different Fourier coefficients for each slice…

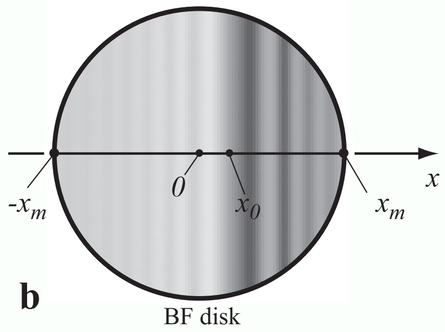
Defects in crystals: examples

STEM-DCI

- converged beam defect imaging, using a circular BF detector and an annular dark field detector, in STEM mode
- produces high quality defect contrast images near zone axis orientations without the strong dynamical background contrast that typically makes zone axis orientations unfavorable for defect imaging

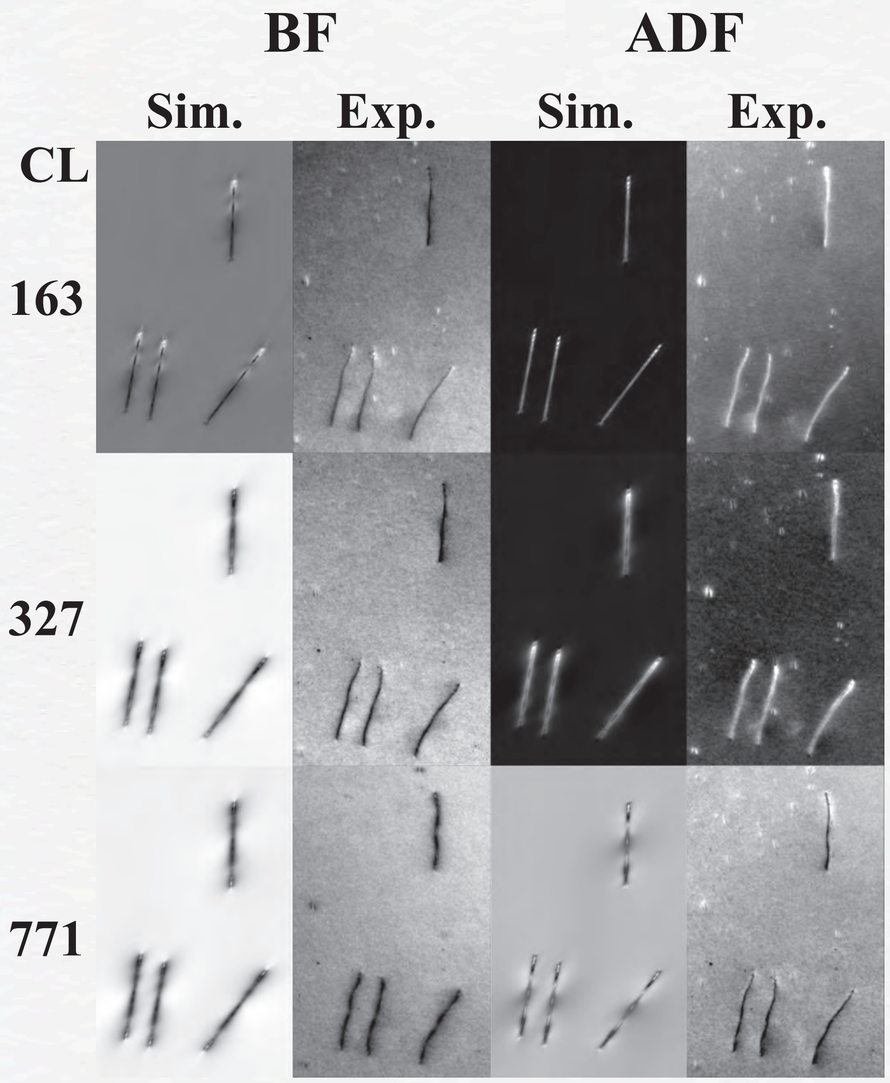


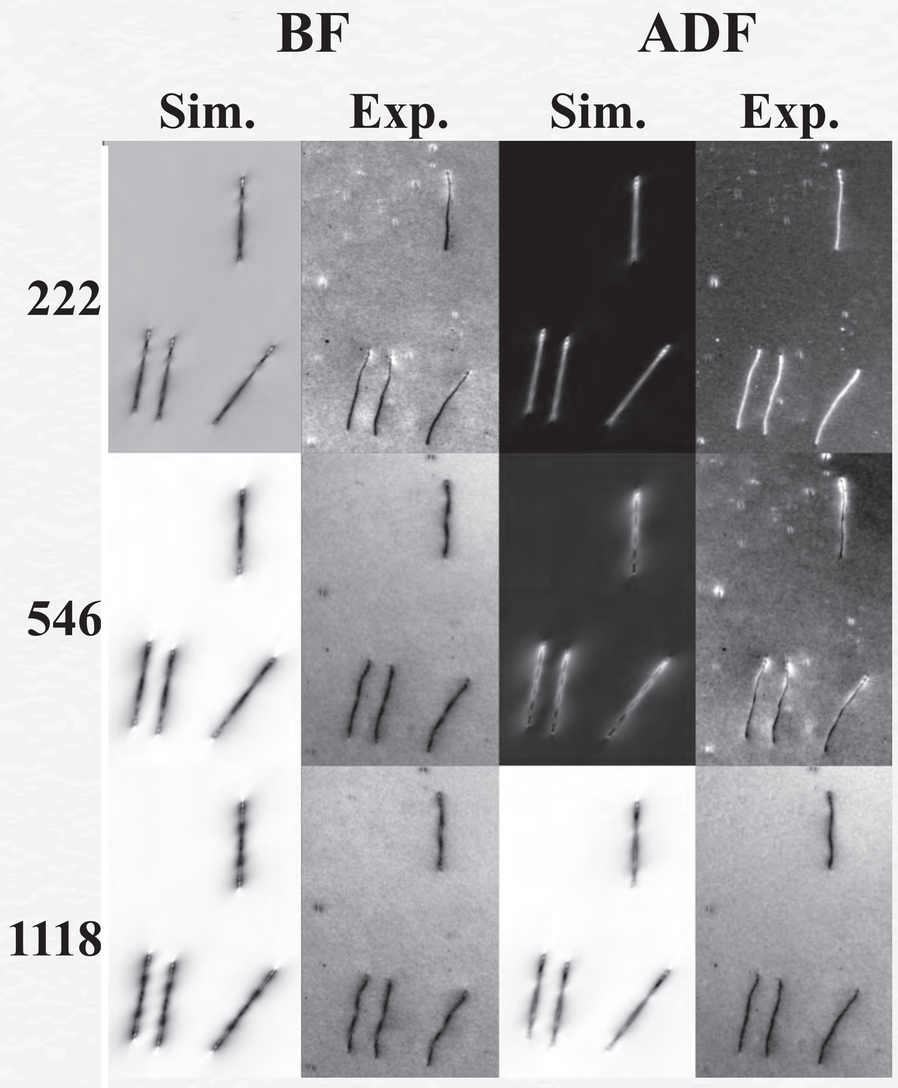
$\theta_c, \lambda L, \rho_{\mathrm{BF}}, \rho_{i,\mathrm{ADF}}, \rho_{o,\mathrm{ADF}}$



focused beam, annular detector(s)

Michael Mills





Zone Axis
defect imaging

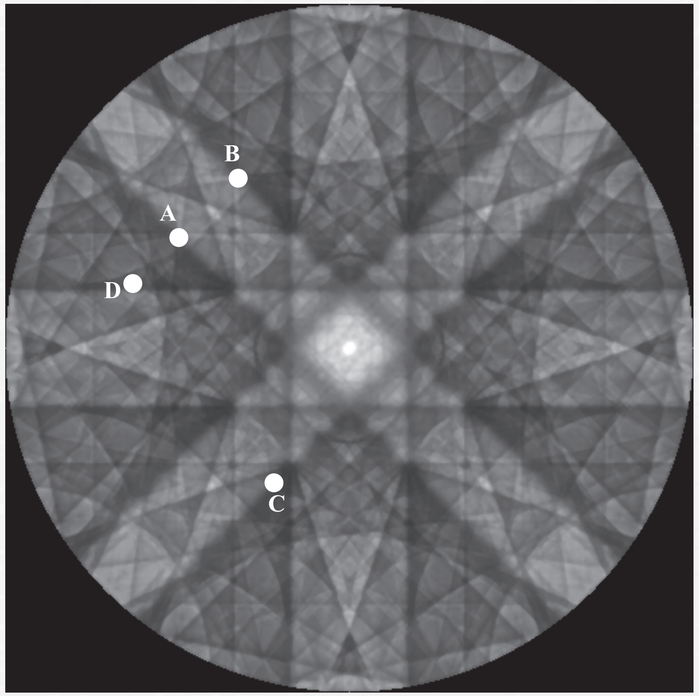
hcp Ti+6wt%Al
200 kV

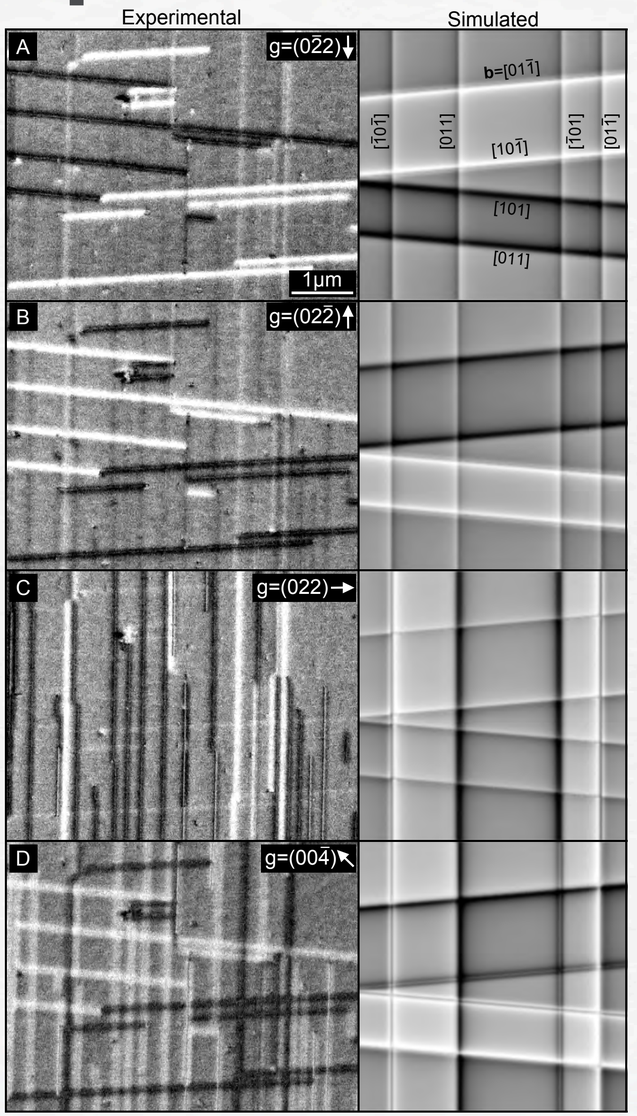
$[1\bar{2}.0]$

Defects in crystals: examples

SEM-ECCI

- Using an ECP, the sample is tilted to a two-beam orientation (on the edge of a Kikuchi band)
- zooming in then produces defect contrast images for near-surface or surface penetrating defects
- Contrast rules (visibility criteria) are similar to those used for TEM and STEM-DCI
- Can be used for large area defect studies



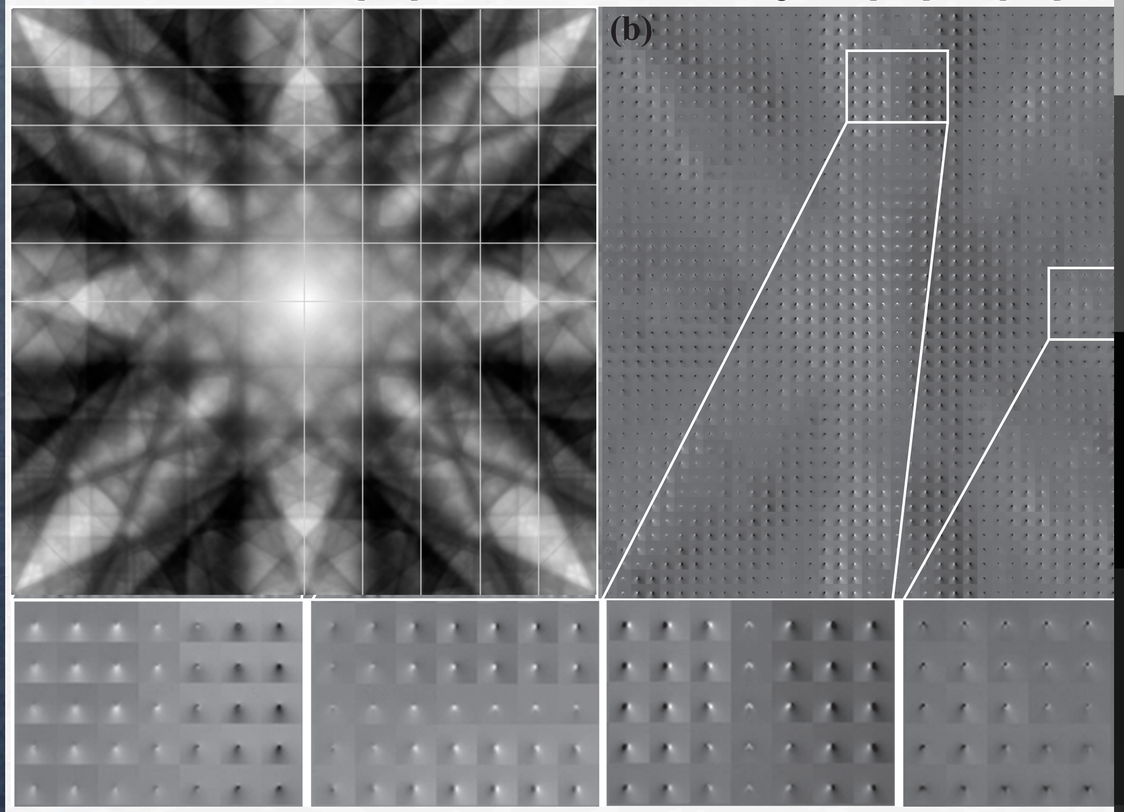


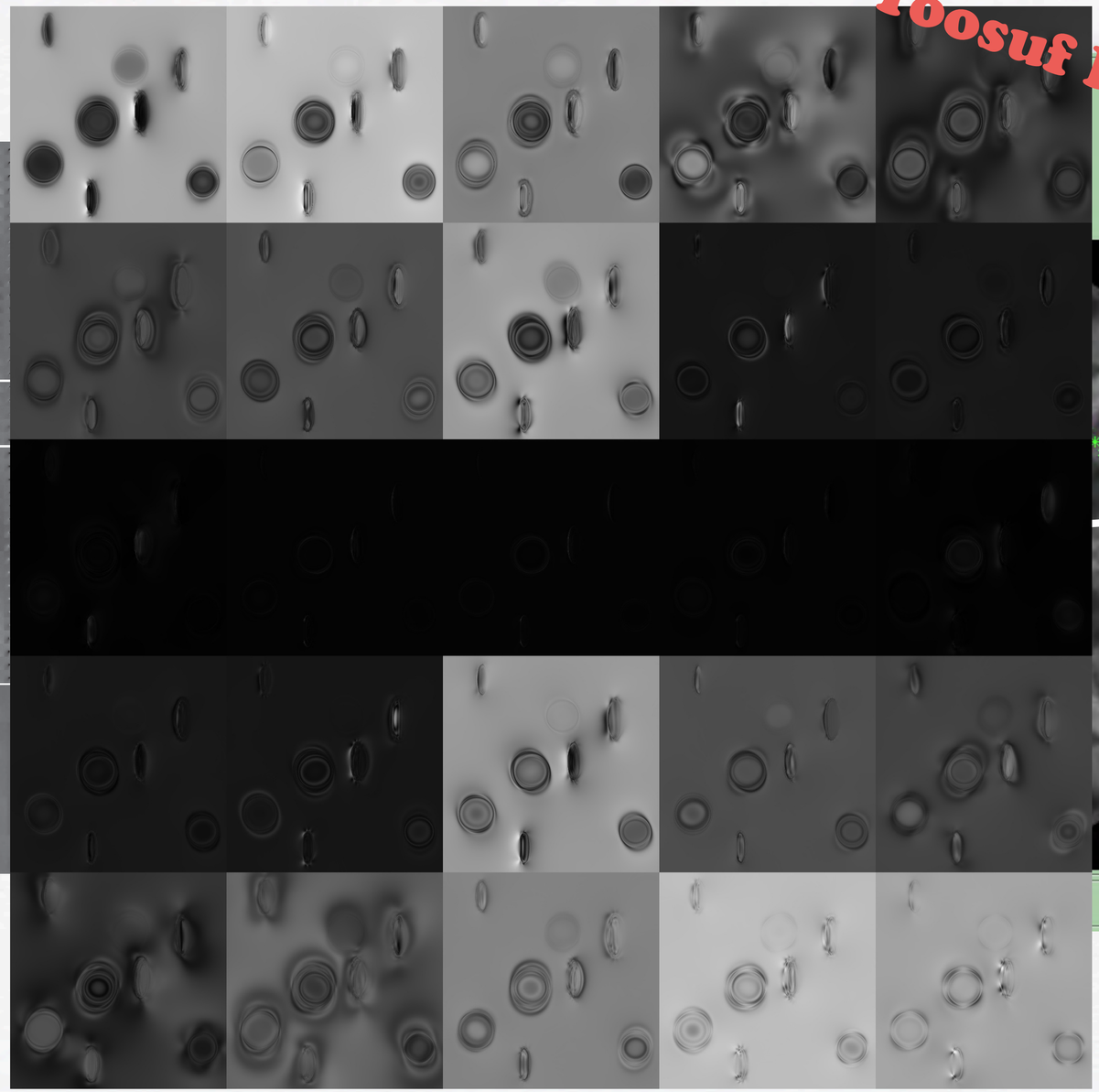
focused beam, annular BSE detector

Yoosuf Picard

Screw **u** = **b** = $[0\bar{1}1]$

Edge **u** = $[0\bar{1}1]$; **b** = [100]





Cross-Correlations

- correlation is a measure for how similar two signals are and is similar to a convolution:

$$(f \otimes g)[\tau] = \int f(x)g(\tau - x)\mathrm{d}x$$

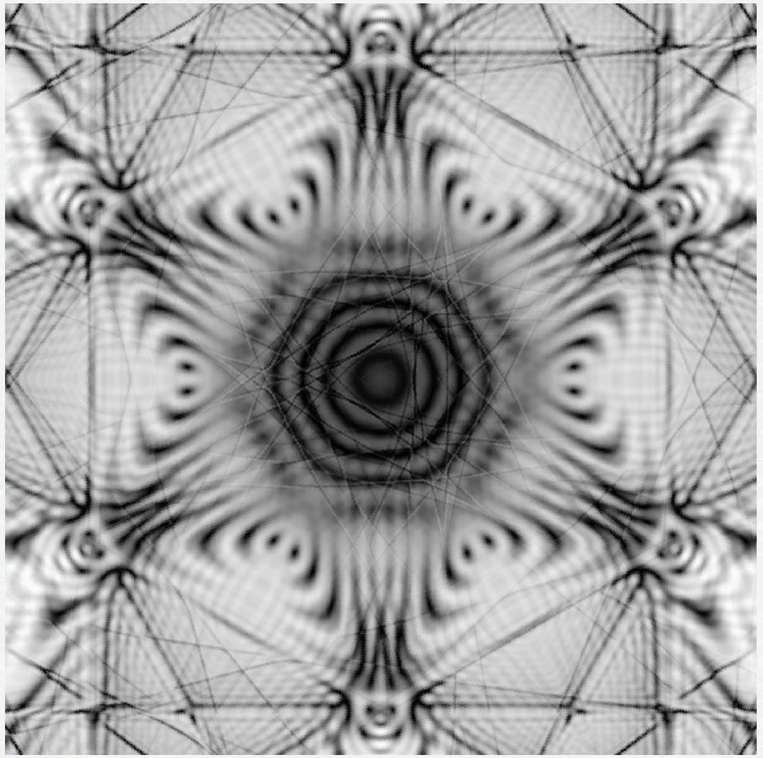
$$C(\tau) = (f \star g)[\tau] = \int f(x)g(\tau + x)\mathrm{d}x$$

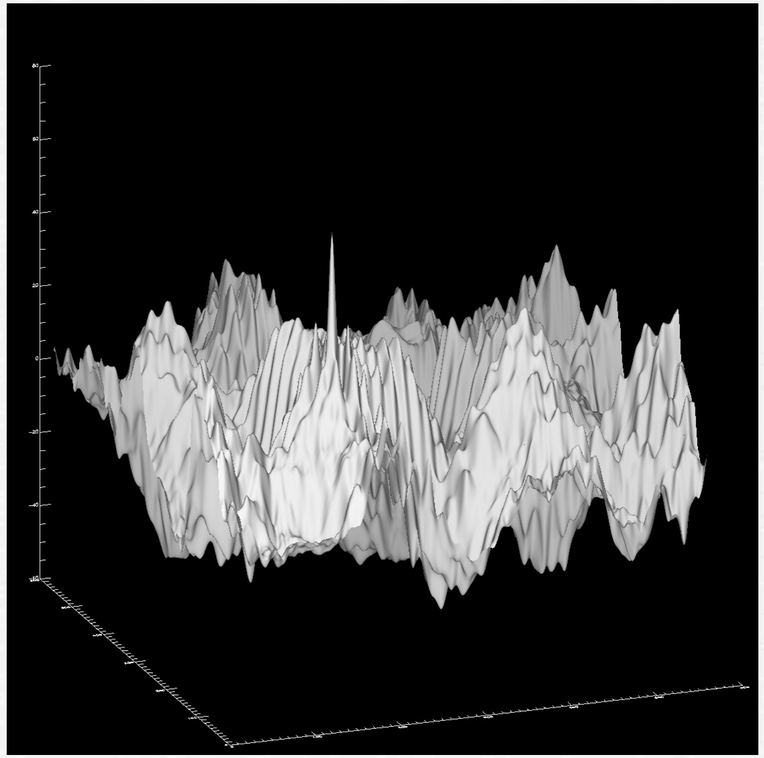
- can be defined in a number of ways, but from a computational point of view the Fourier transform of the signals is used.

$$C = \mathcal{F}^{-1}\left[\mathcal{F}[f]\mathcal{F}^{*}[g]\right]$$

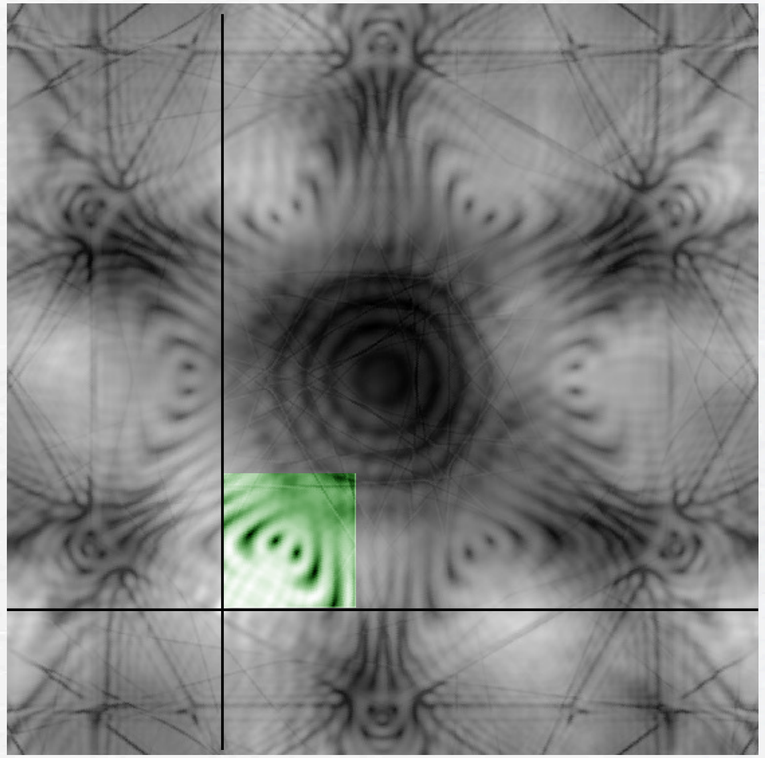
Cross-Correlations

Ben Britton





Cross-correlation



Where is  ?

QUESTIONS ?