Reciprocal Space and Precession Images

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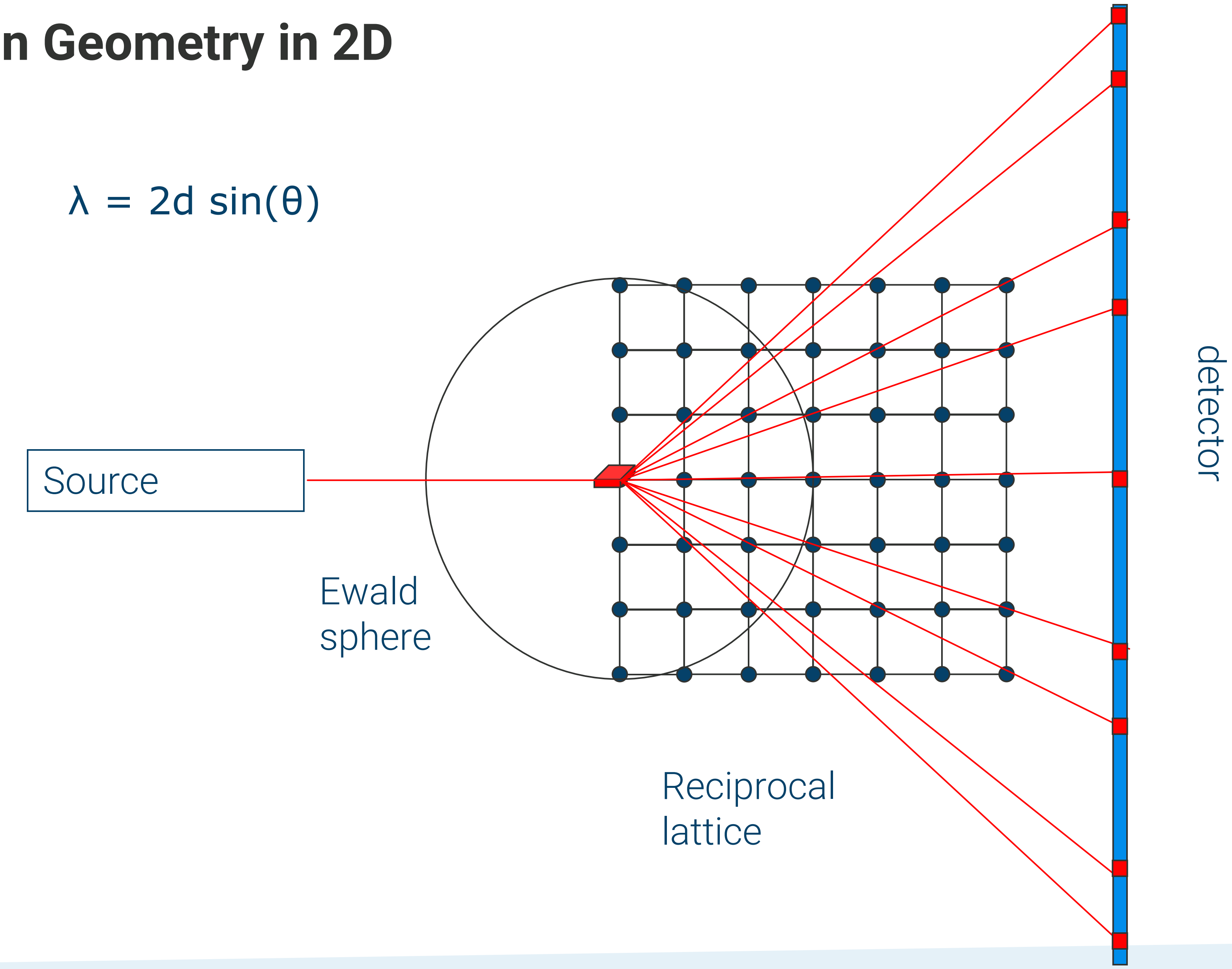
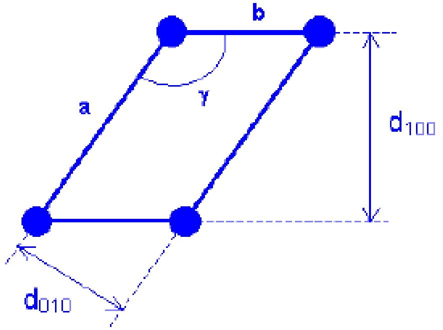
Diffraction Geometry in 2D

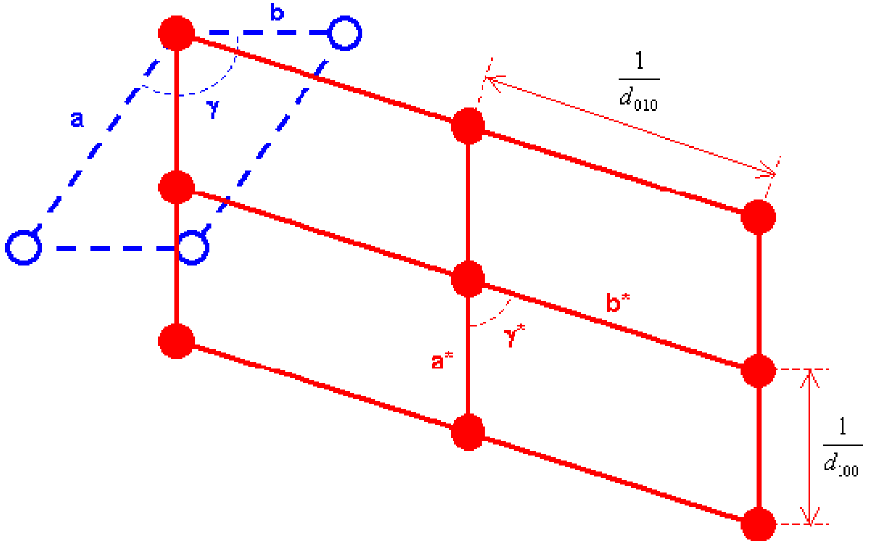
$$\lambda = 2d\sin(\theta)$$



Reciprocal Lattice

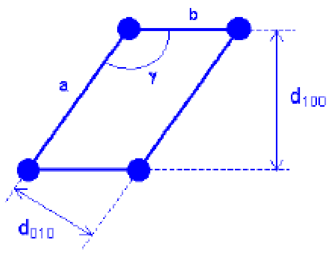
- Due to the reciprocal nature of the spaced $\mathbf{d_{hkl}}$ and the angle θ in Bragg's Law, the observed diffraction pattern can be related to the crystal lattice by a mathematical construction called the Reciprocal Lattice. That is, the beams are reflected according to a pattern that forms a lattice that we can use to obtain information from the crystal.
- The reciprocal lattice is built as follows:
- Choose a point to be the origin of the crystalline lattice.
- Let the normal vector be a set of lattice planes. Translated to the origin and with a modulus equal to 1/d for each plane family. e.g.: the plane vector (hkl) will have a modulus of $1/d_{hkl}$.
- Repeat for all network plans

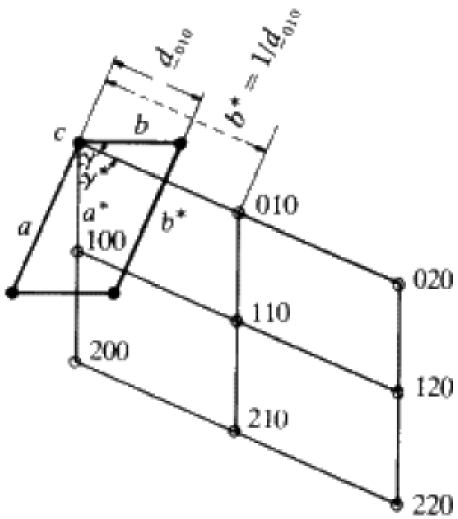


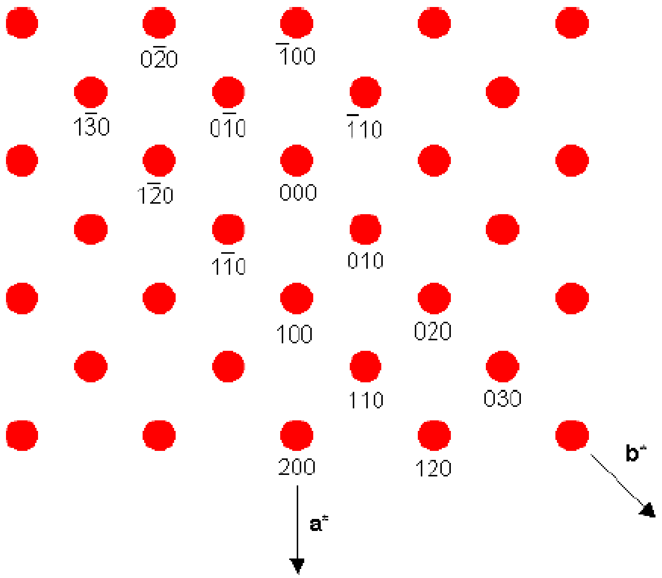


Reciprocal Lattice

- This procedure builds the reciprocal lattice (RL) in which each point corresponds to the reflection generated by a particular family of planes. This network can be indexed by giving the appropriate value (*hkl*) to each point.



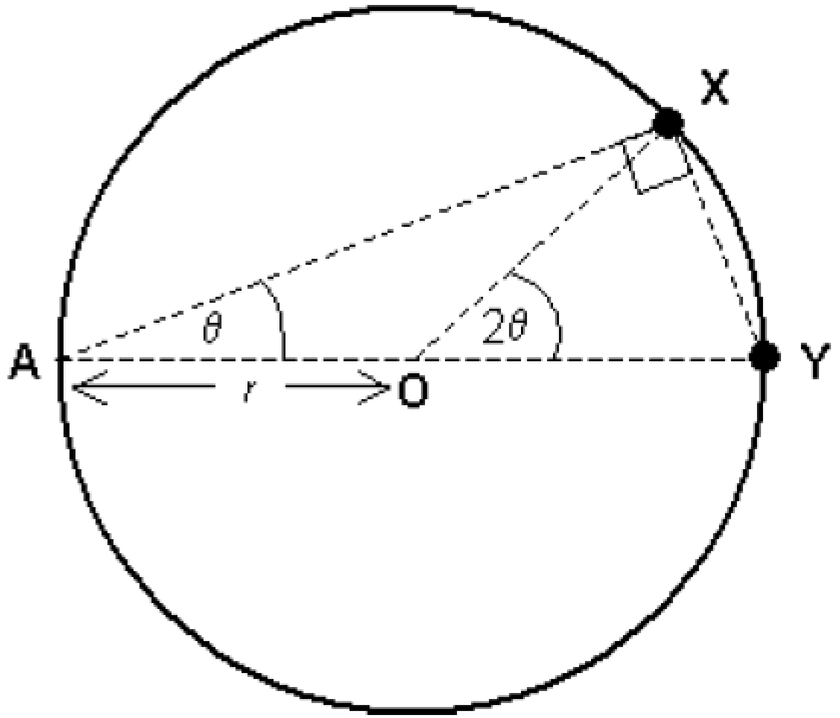




- Consequences of the reciprocal relationship:
  - Large *d* spaced in the Direct Lattice (DL) corresponds to smaller spaces in RL.
  - Obtuse angles in the DL correspond to obtuse angles in the RL.

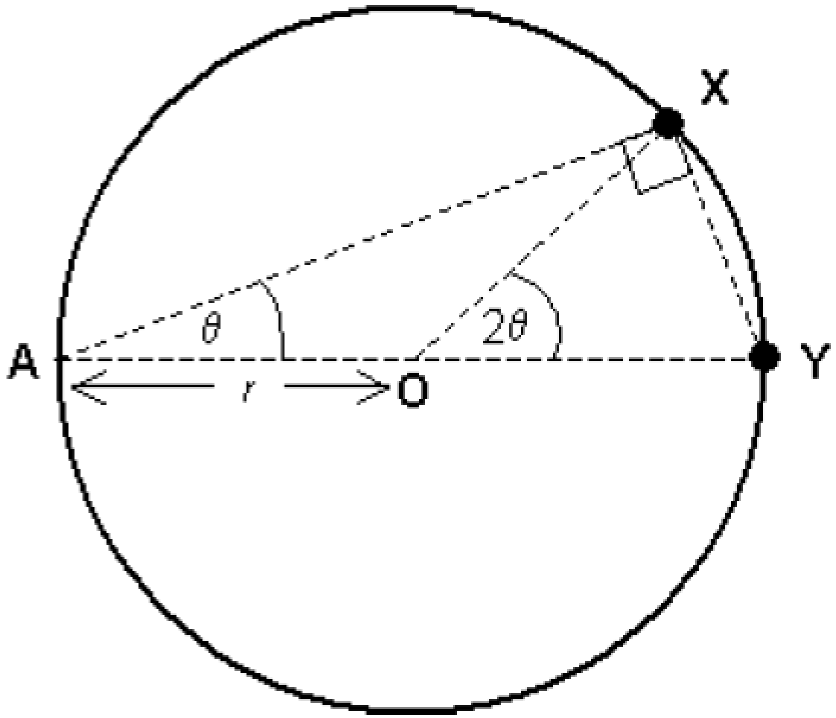
Ewald Sphere

- A very useful mathematical structure in crystallography is the Ewald sphere which tells us the angle at which each family of planes will diffract.
- Consider a circle of radius r, with X and Y points on the circumference.
- If the XAY angle is defined as θ, then the XOY angle will be 2θ by geometry and sin(θ) = XY/2r
- If this geometry is constructed in reciprocal space, then this has some important implications



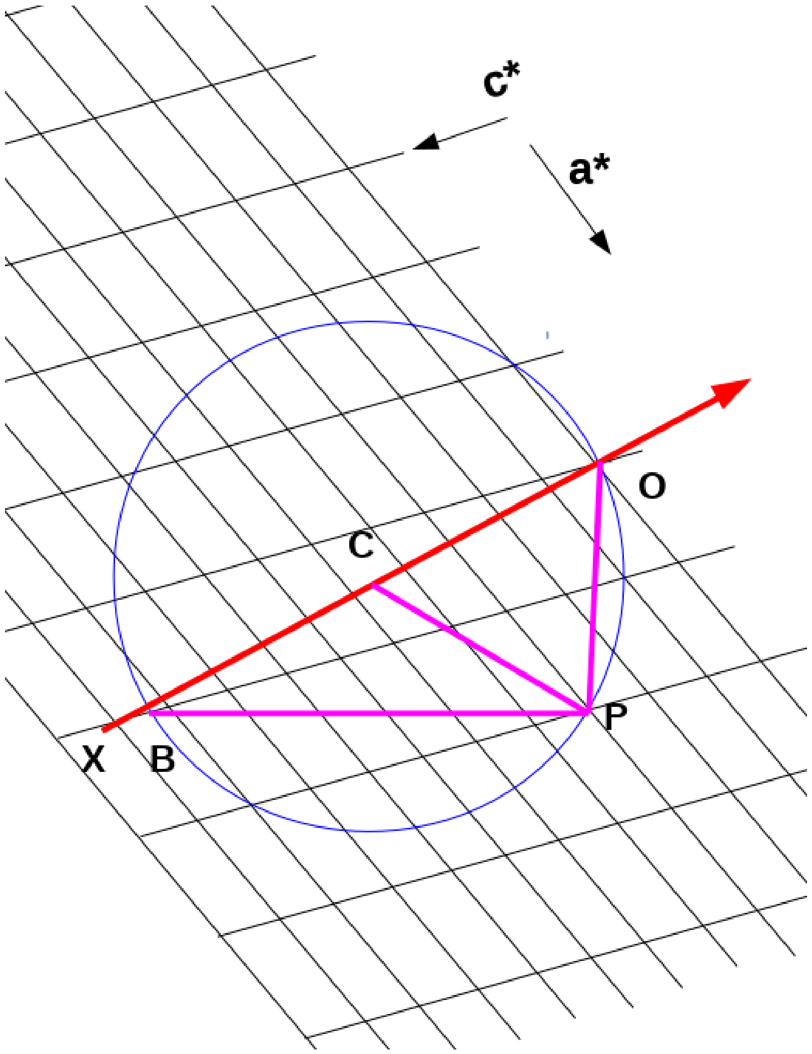
Ewald Sphere

- The radius is fixed as being $1/\lambda$
  - λ = wavelength of the X-ray beam.
- If Y is the point 000 of the Reciprocal Lattice (center of the crystal), and X is a general point *hkl* (point of the Reciprocal Lattice touching the sphere), then the distance XY is $1/d_{hkl}$
- Therefore: $\sin(\theta) = (1/d_{hkl}) / (2/\lambda)$ or, rearranged: $\lambda = 2\, d_{hkl}\sin(\theta)$
- Then Bragg's Law will be satisfied!



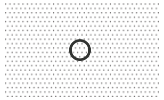
Ewald Construction

- Graphical depiction of Bragg's Law
- Circle has radius of 1/λ, center at C such that origin of reciprocal lattice, O, lies on circumference
- XO is the X-ray beam, P is the reciprocal lattice point (in this case the 202 reflection)
- OP is the reciprocal lattice vector (**d***) and is normal to the (202) set of planes [aka the Scattering Vector]
- Angle OBP is θ, the Bragg angle
- Angle OCP is 2θ
- CP is the direction of the diffracted beam
- BP is parallel to the set of (202) planes
- Any time a reciprocal lattice point falls on the circumference, Bragg's Law is fulfilled

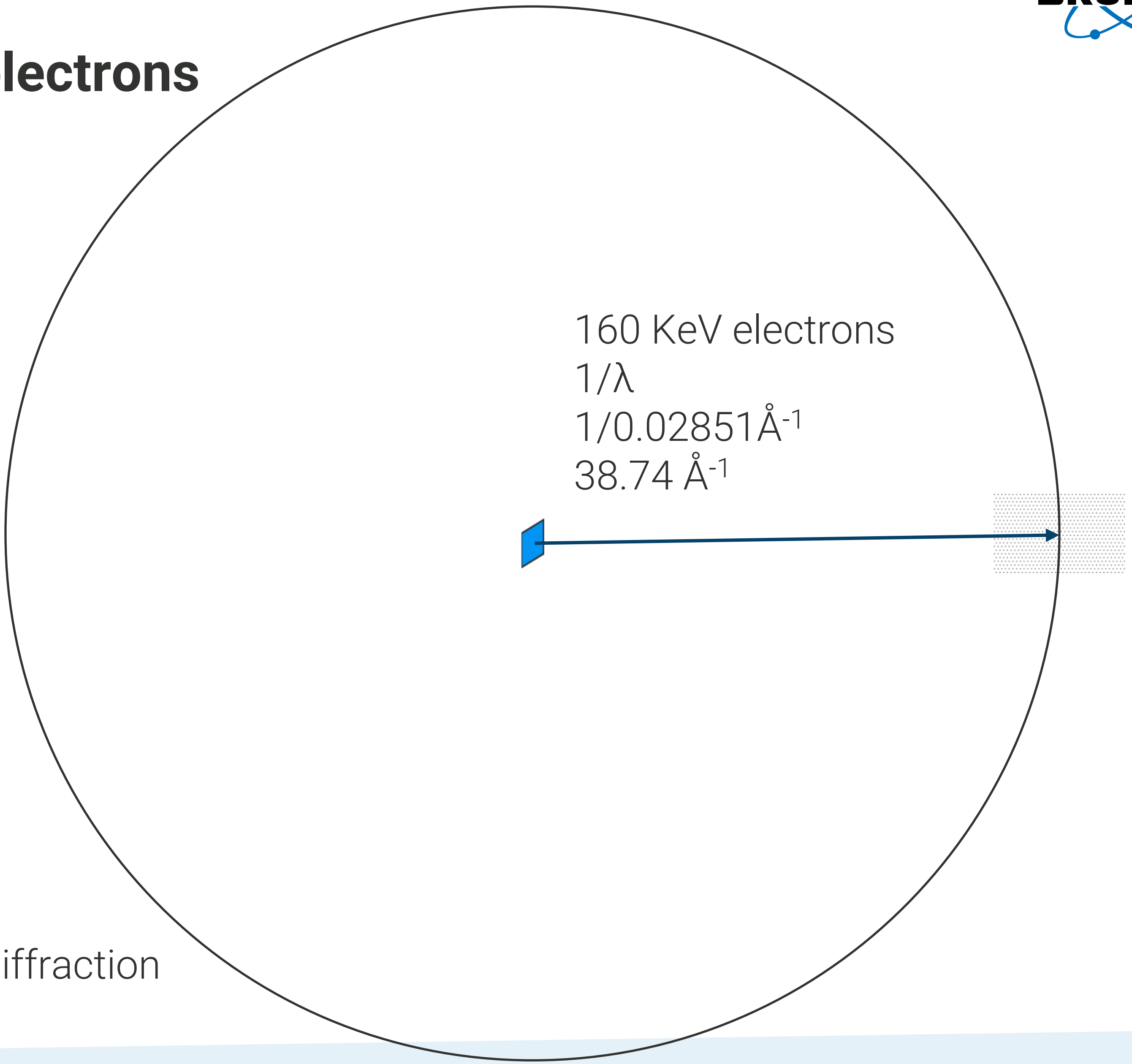


Diffraction geometry: X-ray vs electrons

Mo radiation
$1/\lambda$
1/0.71073 Å^{-1}
1.41 Å^{-1}



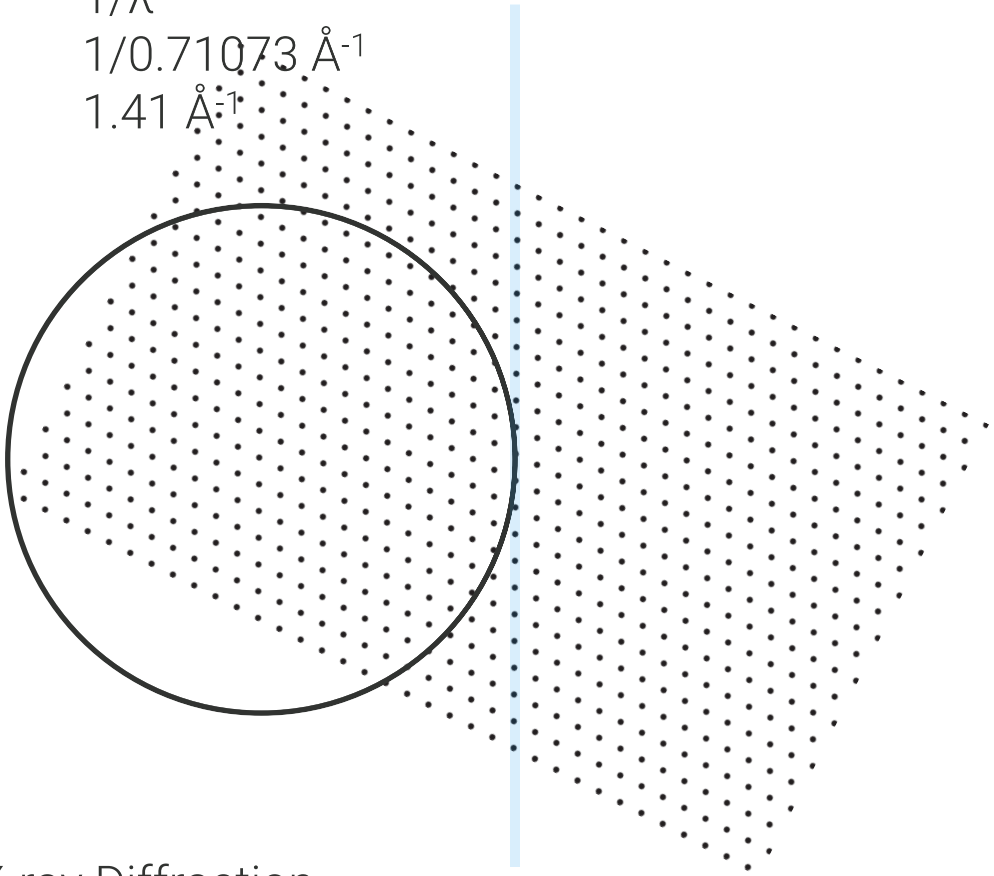
X-ray Diffraction



Electron Diffraction

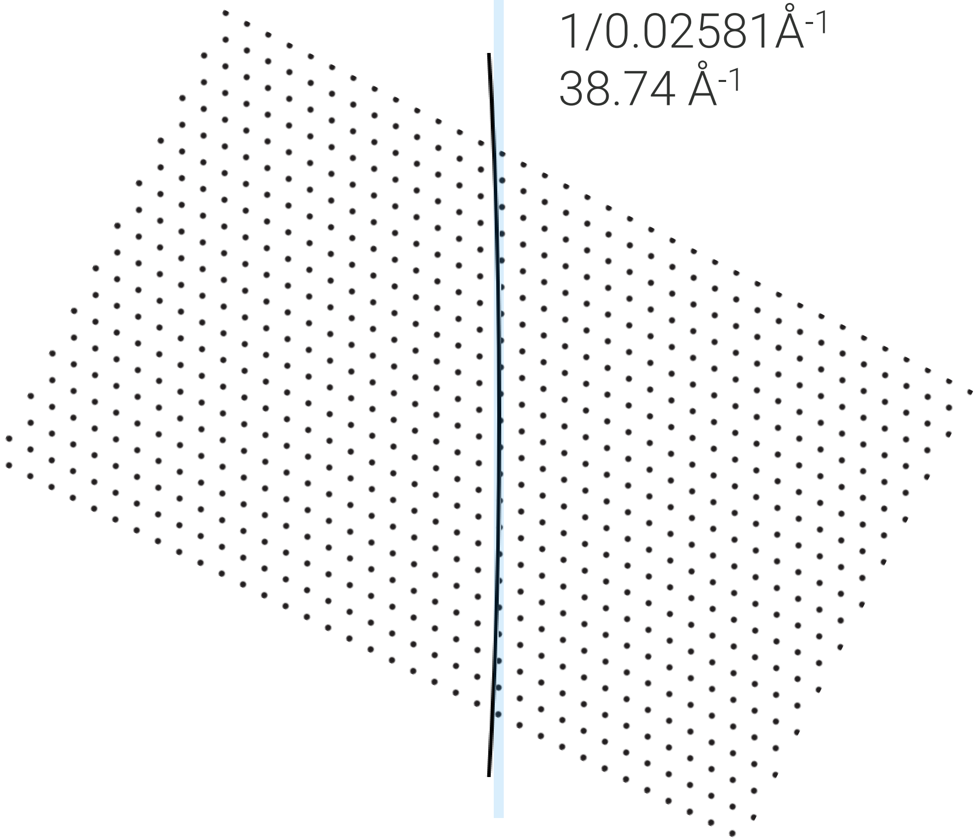
Diffraction geometry: X-ray vs electrons

Mo radiation
$1/\lambda$
$1/0.71073$ Å^{-1}
1.41 Å^{-1}



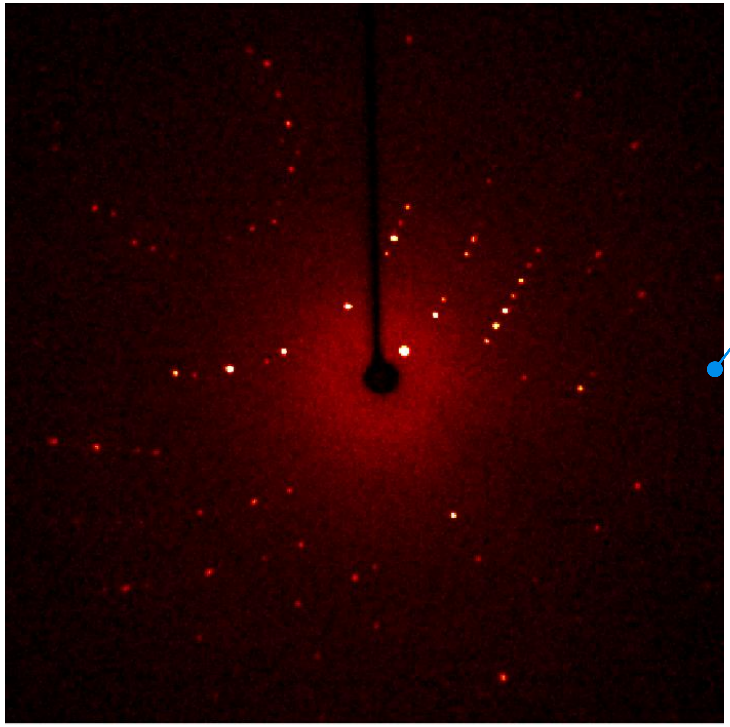
X-ray Diffraction

200 KeV electrons
$1/\lambda$
$1/0.02581$Å^{-1}
38.74 Å^{-1}



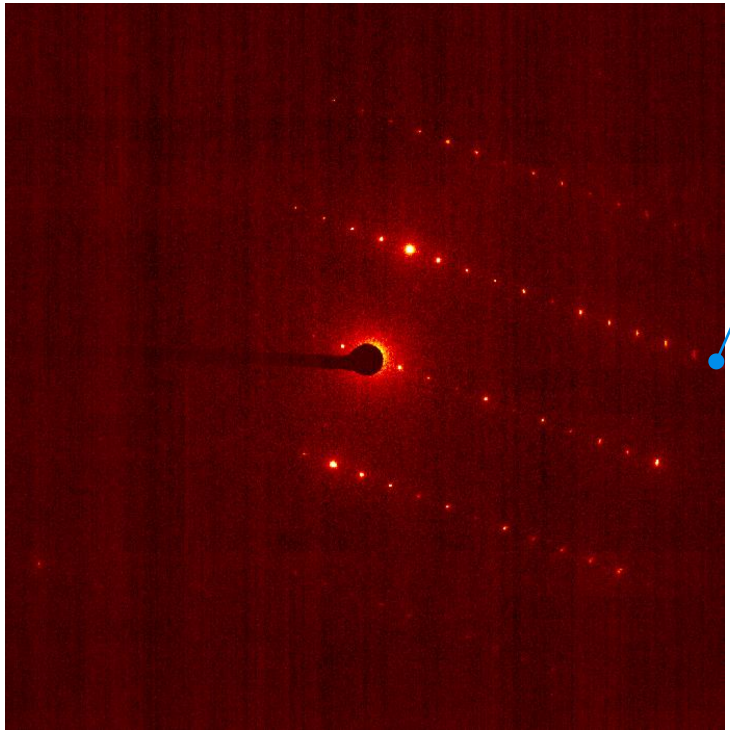
Electron Diffraction Diffraction

X-ray diffraction vs electron diffraction



2Theta=45 deg

Mo radiation
0.71073 Å

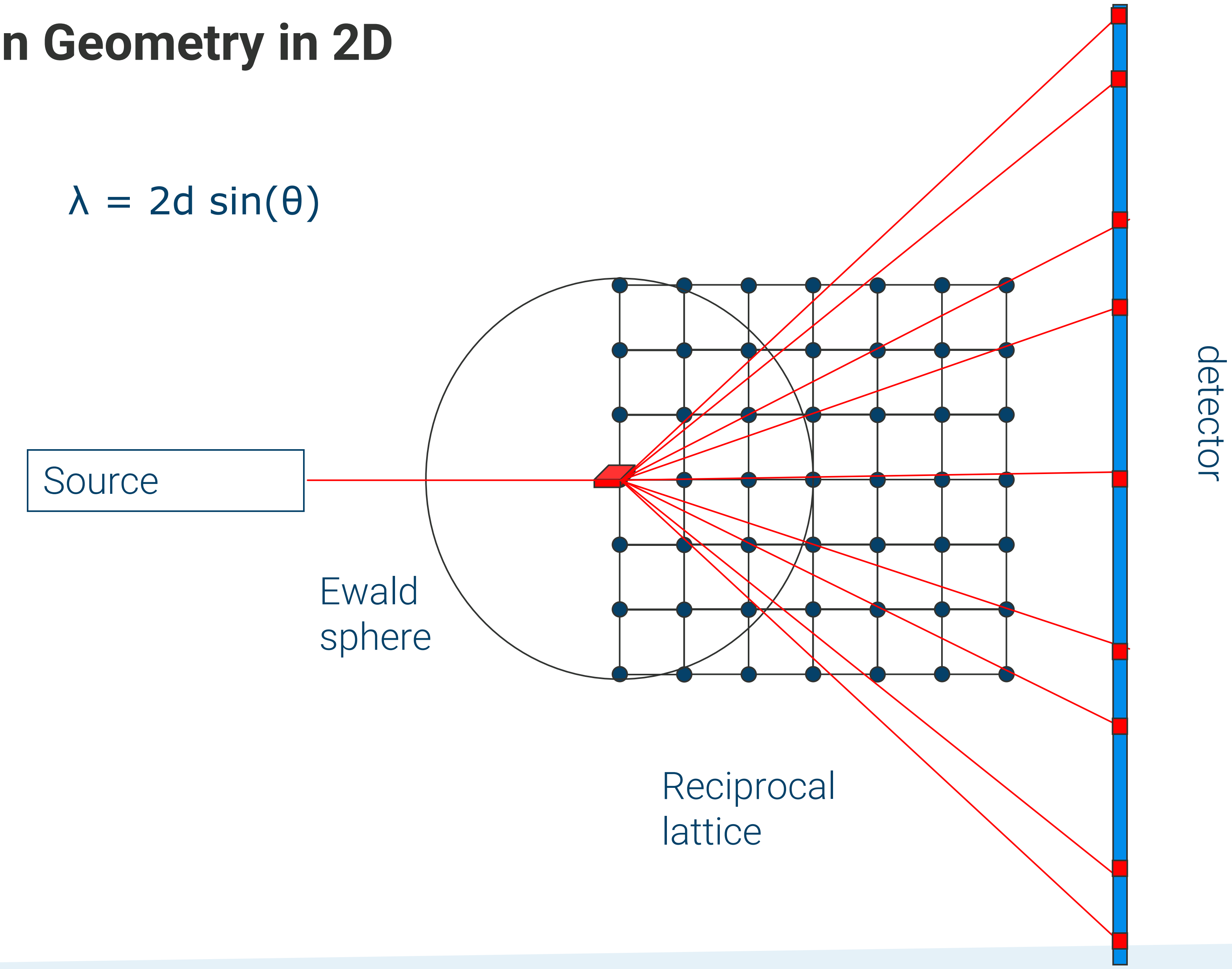


2Theta=2 deg

200 KeV electrons
0.02851Å

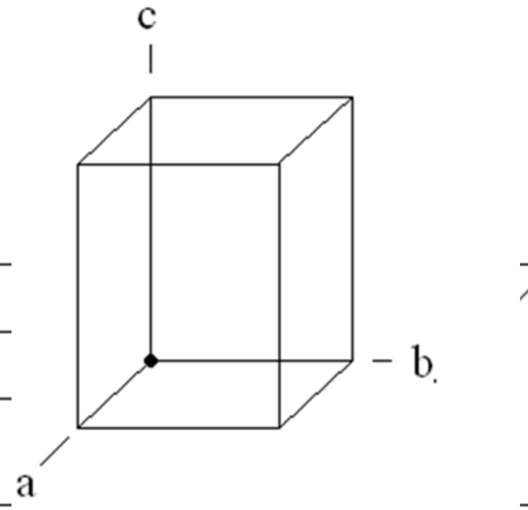
Diffraction Geometry in 2D

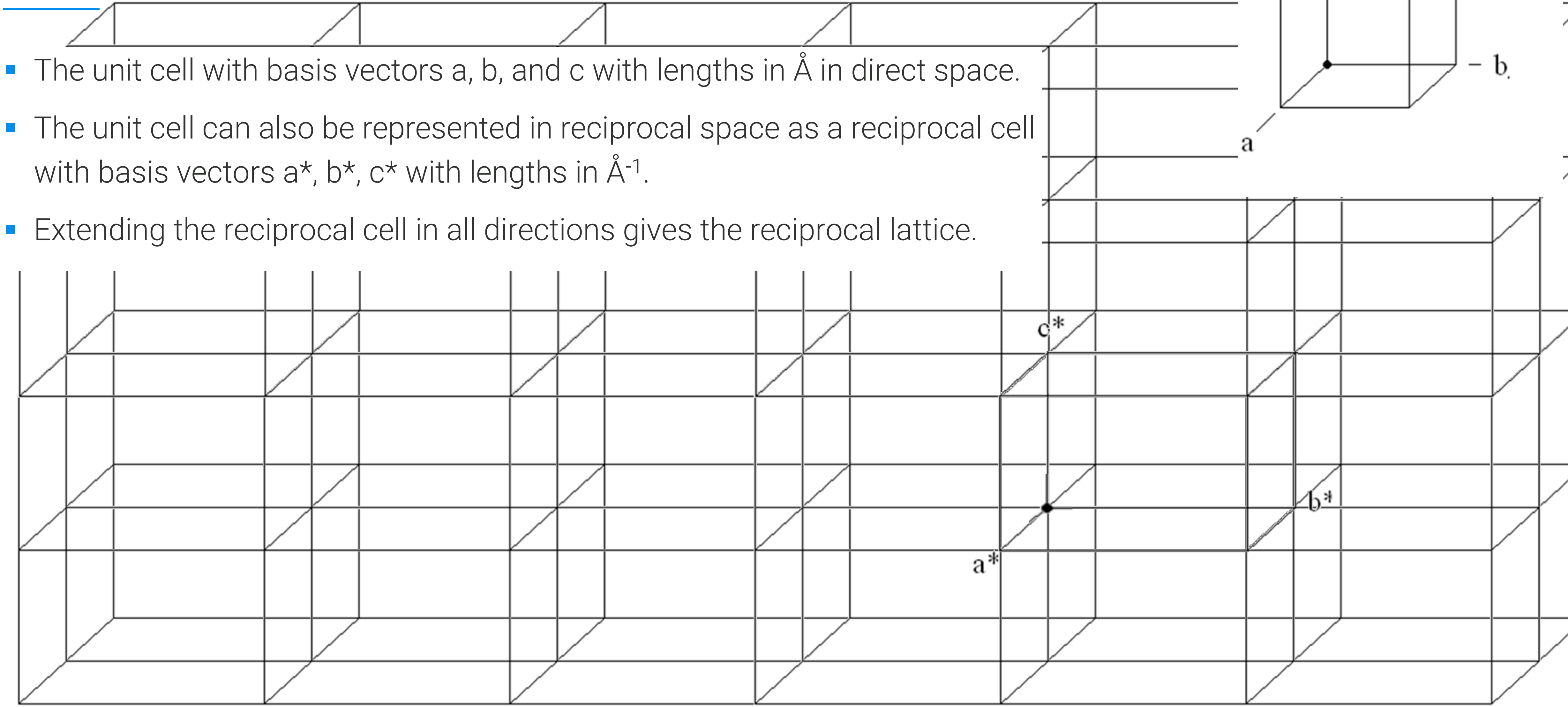
$$\lambda = 2d \sin(\theta)$$



The Reciprocal Lattice

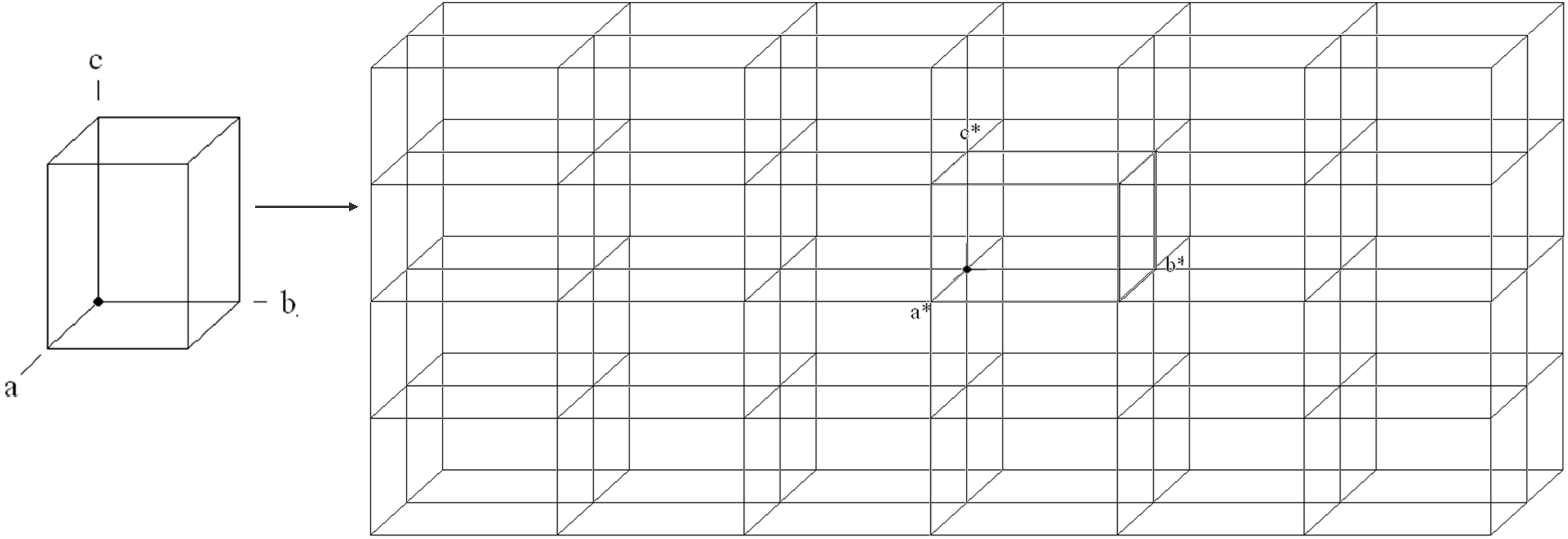
- The unit cell with basis vectors a, b, and c with lengths in Å in direct space.
- The unit cell can also be represented in reciprocal space as a reciprocal cell with basis vectors a*, b*, c* with lengths in $Å^{-1}$.
- Extending the reciprocal cell in all directions gives the reciprocal lattice.





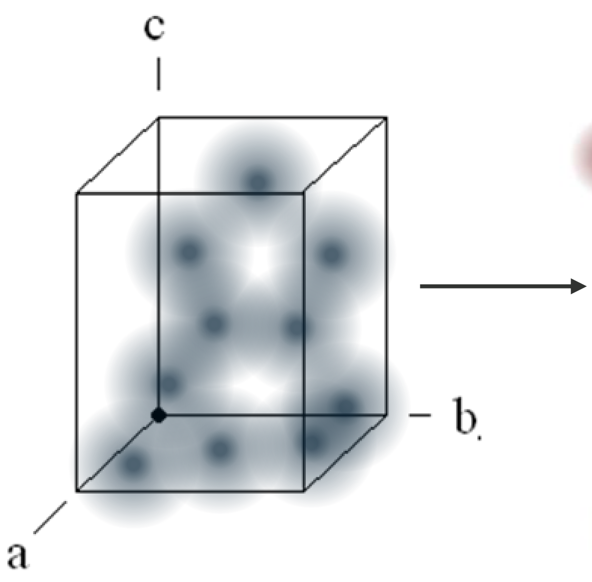
The reciprocal lattice is the diffraction pattern

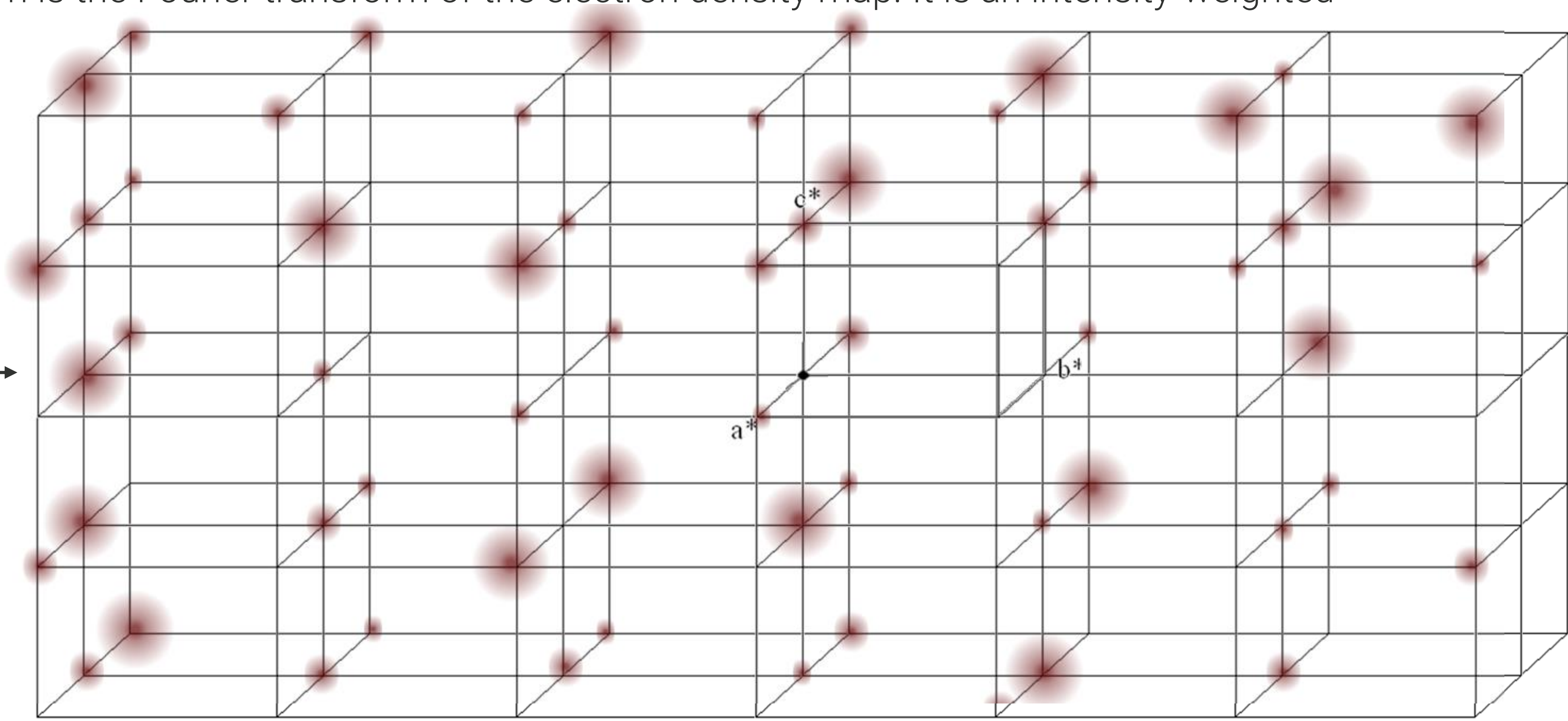
- Each reciprocal lattice point hkl corresponds to the X-rays reflected from the lattice planes hkl
- The diffraction pattern only tells you about the size/shape of the unit cell, not its contents



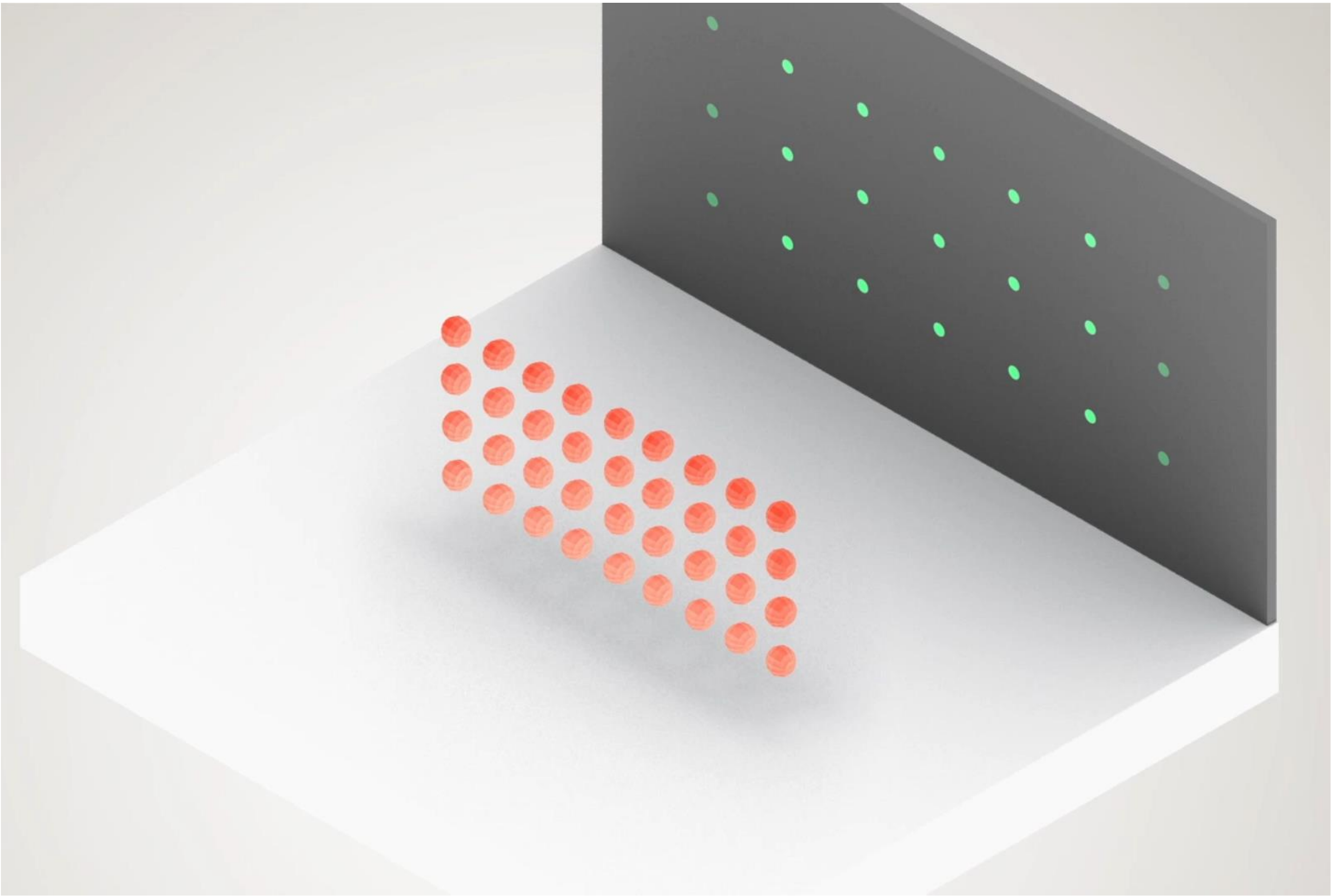
The reciprocal lattice is the diffraction pattern

- How do we know the structure of the molecule? The **intensities** of the diffracted spots.
- The diffraction pattern is the Fourier transform of the electron density map. It is an intensity-weighted reciprocal lattice.





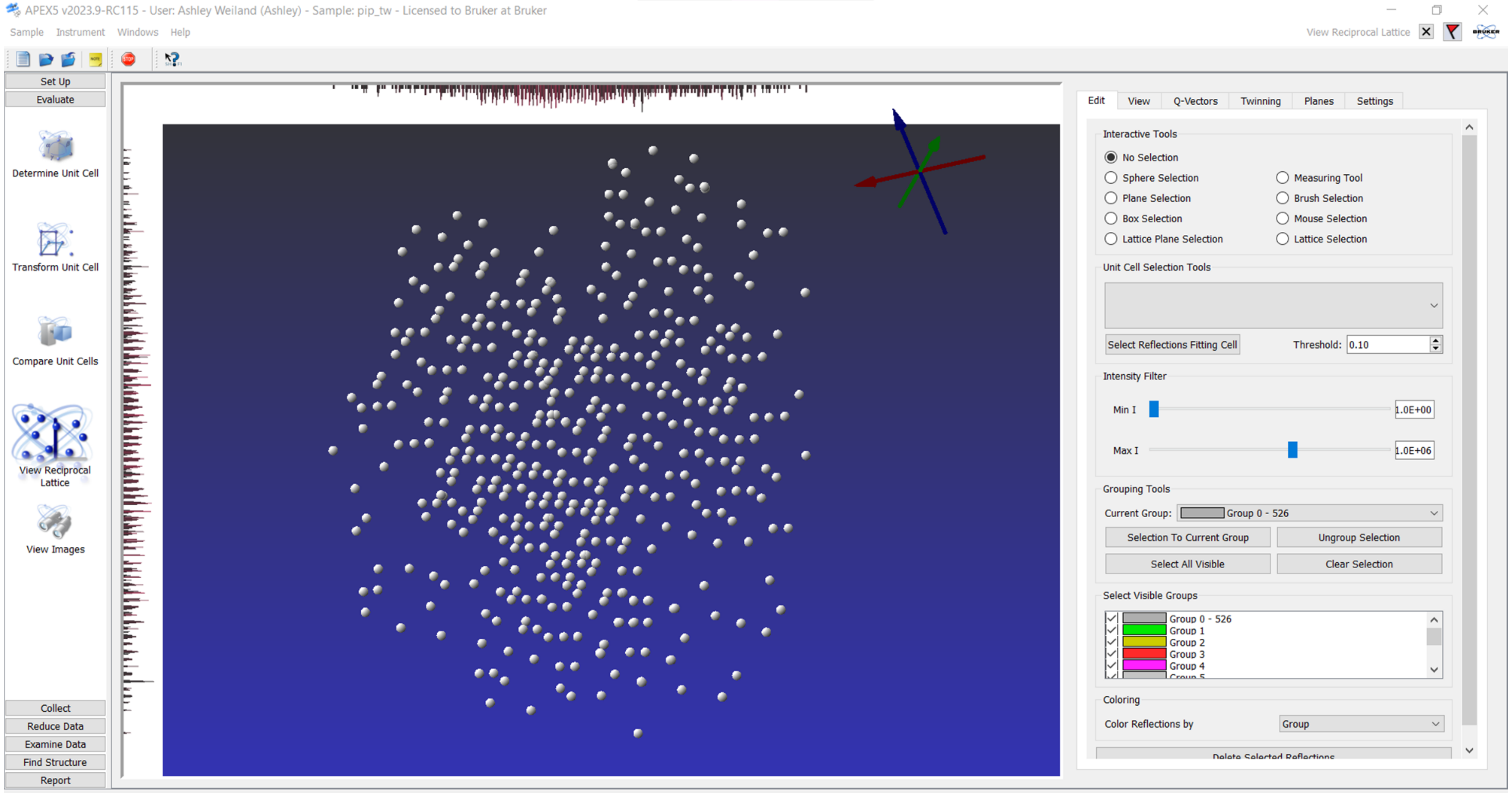
Reciprocal Space Visual Representation



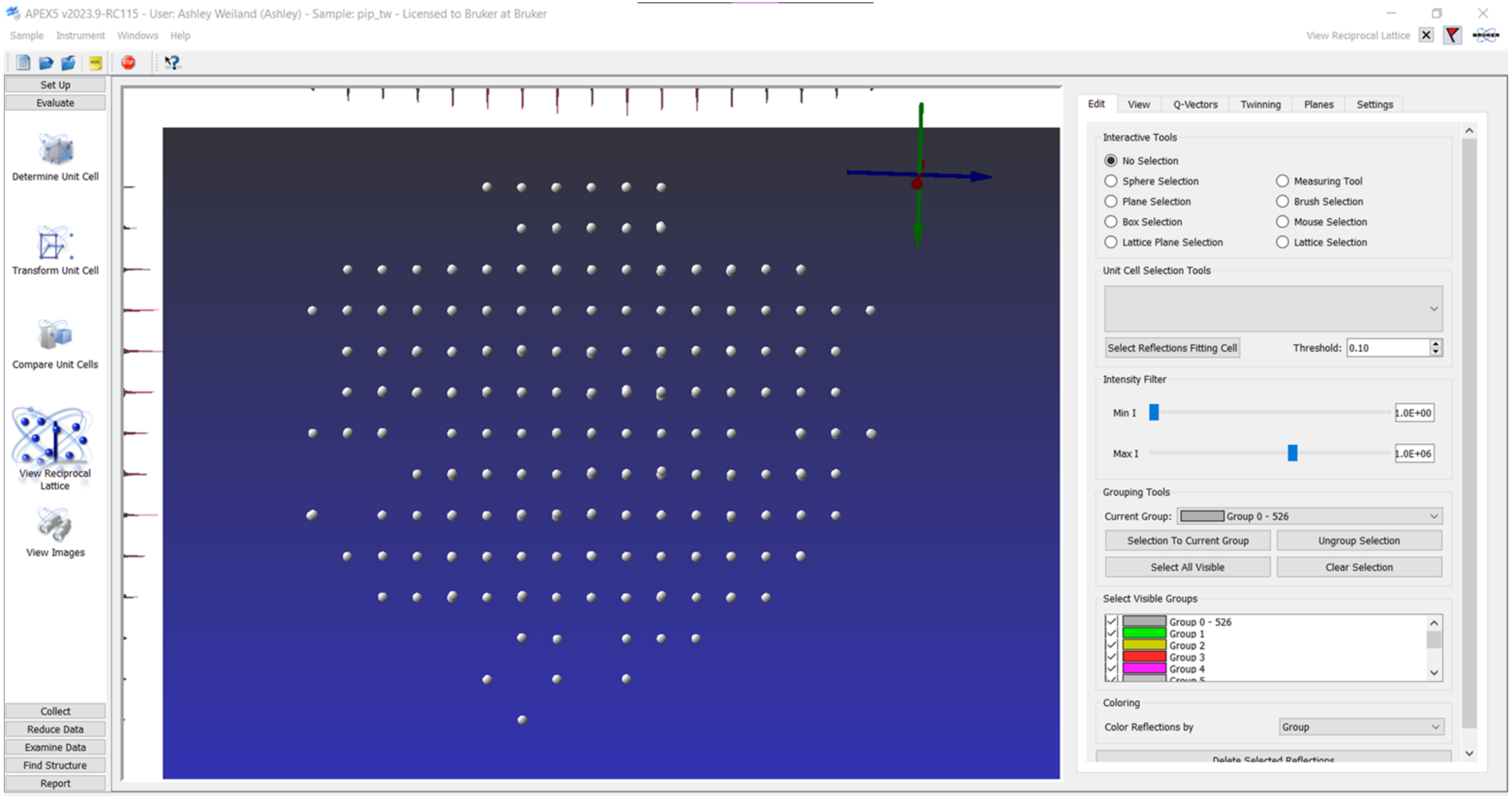
https://www.youtube.com/watch?v=DFFU39A3fPY

The Reciprocal Lattice Viewer

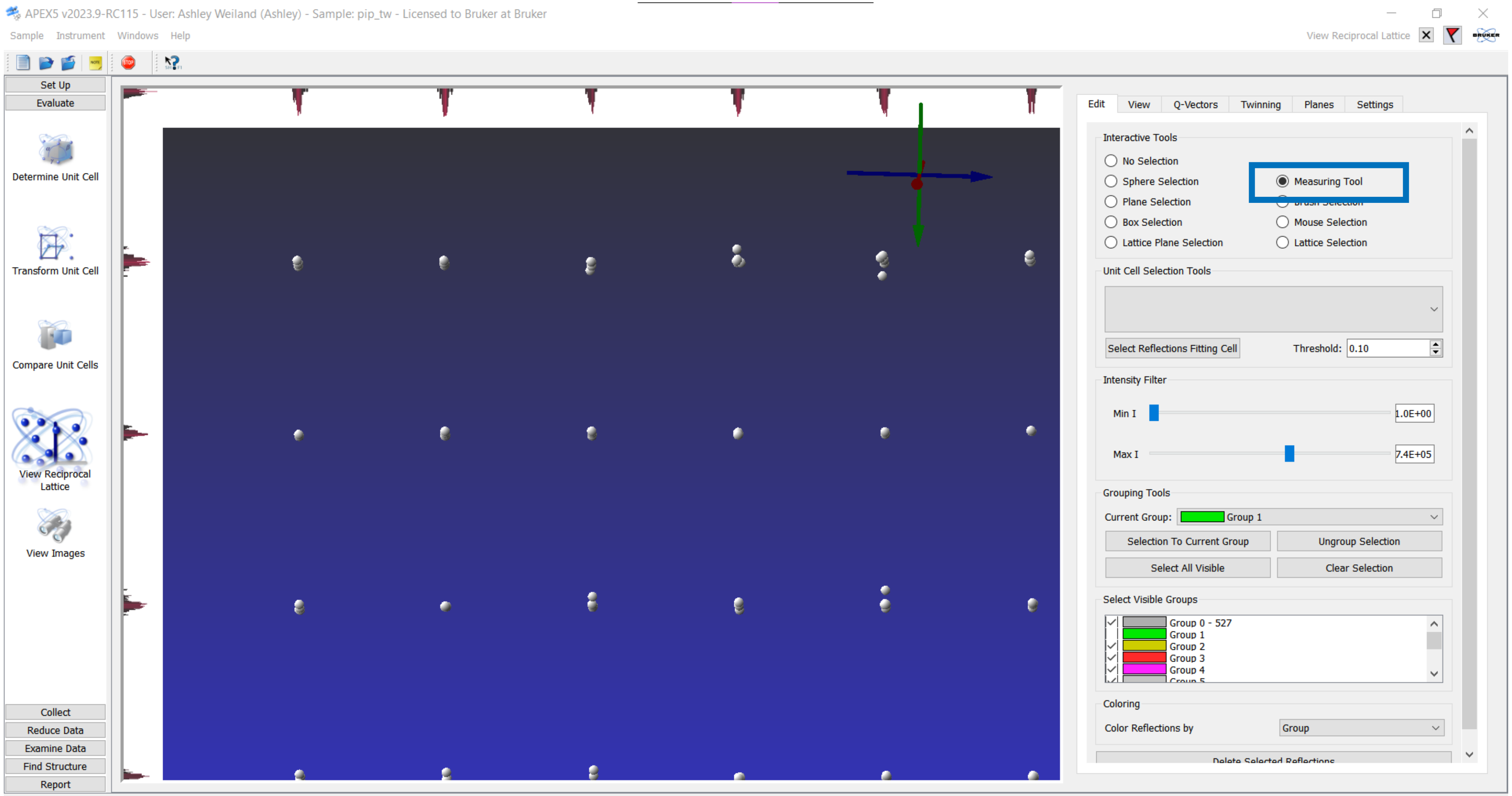
View Reciprocal Lattice



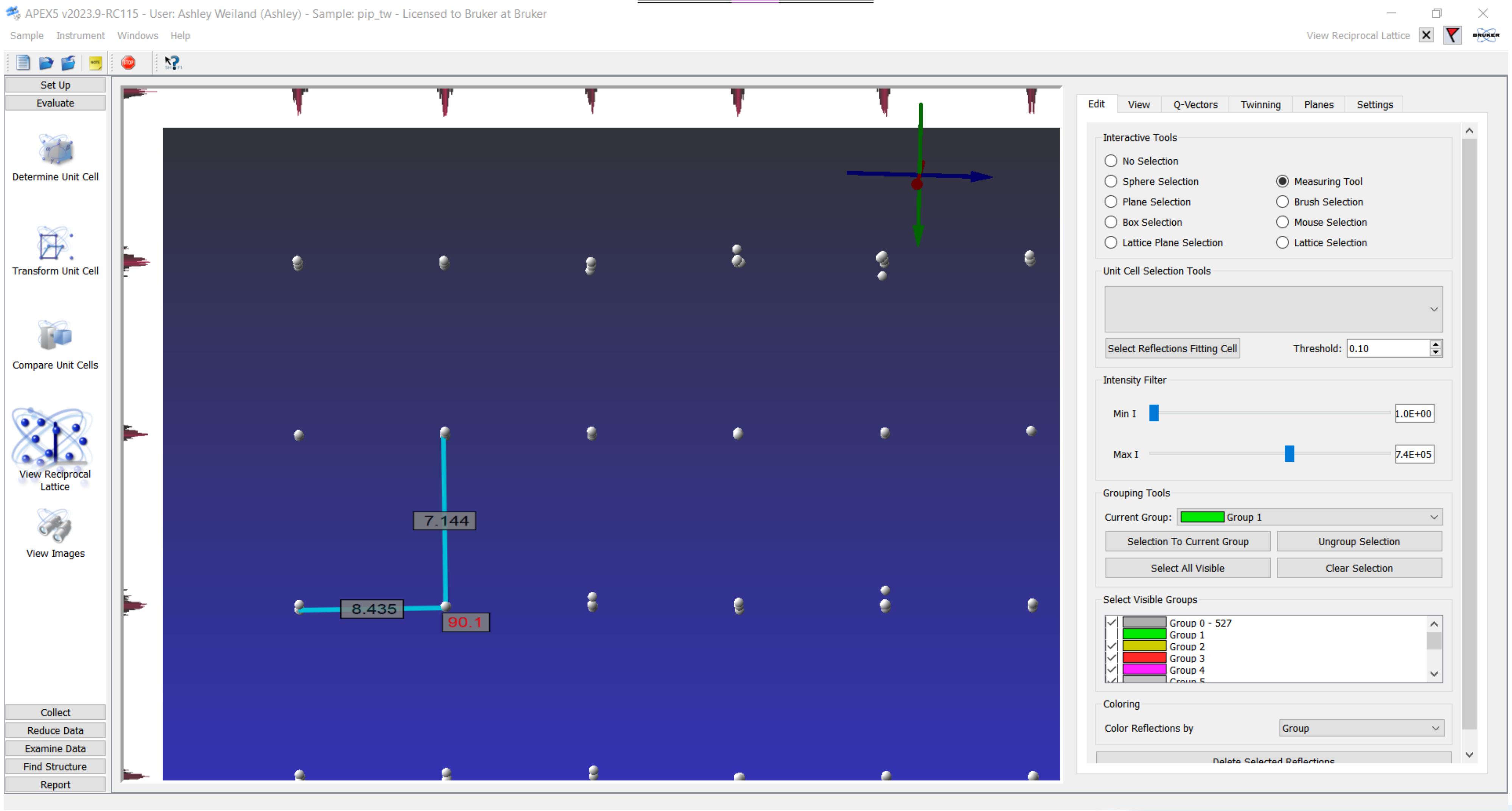
View Reciprocal Lattice



Measuring Tool

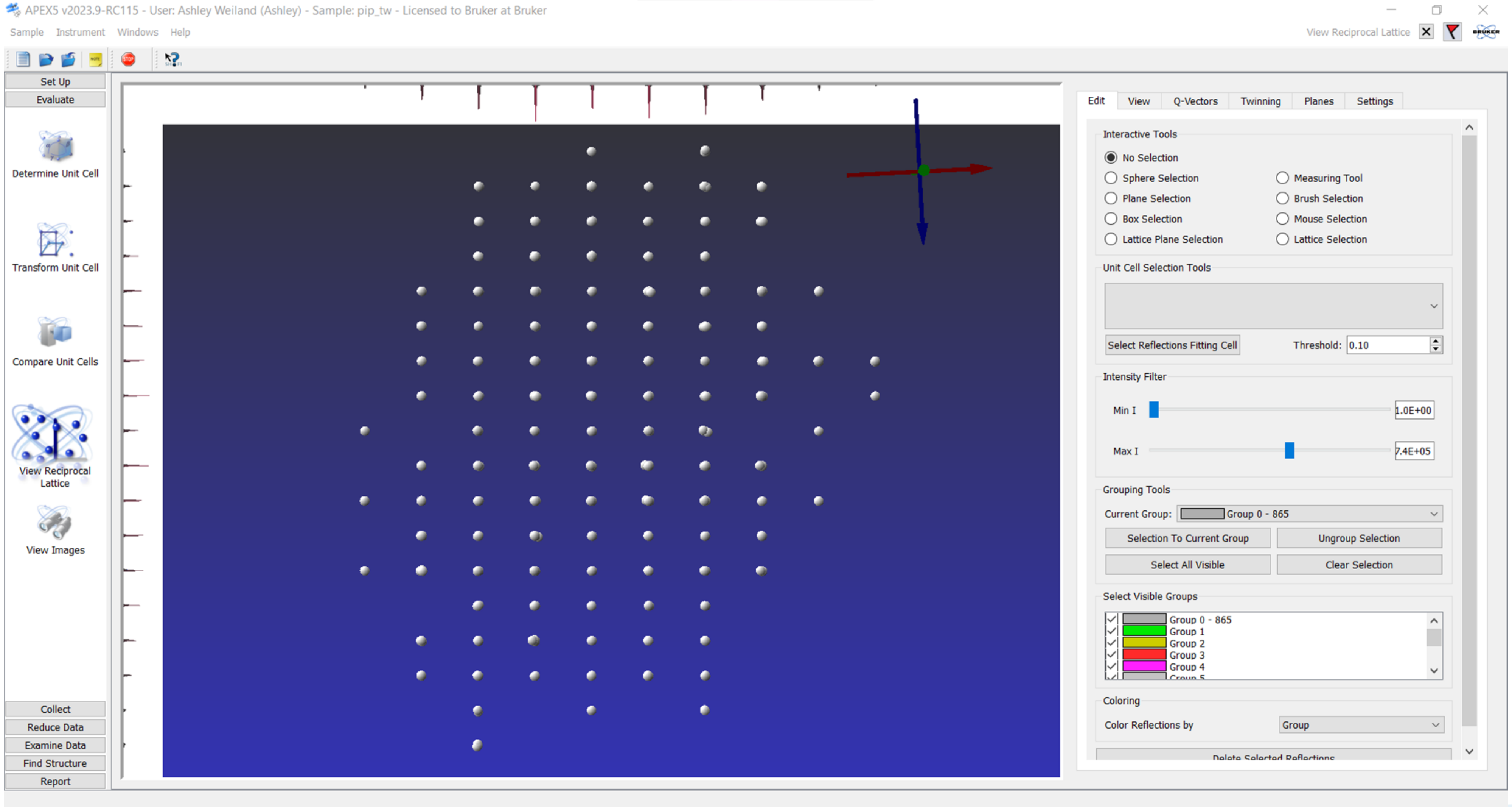


Measuring Tool

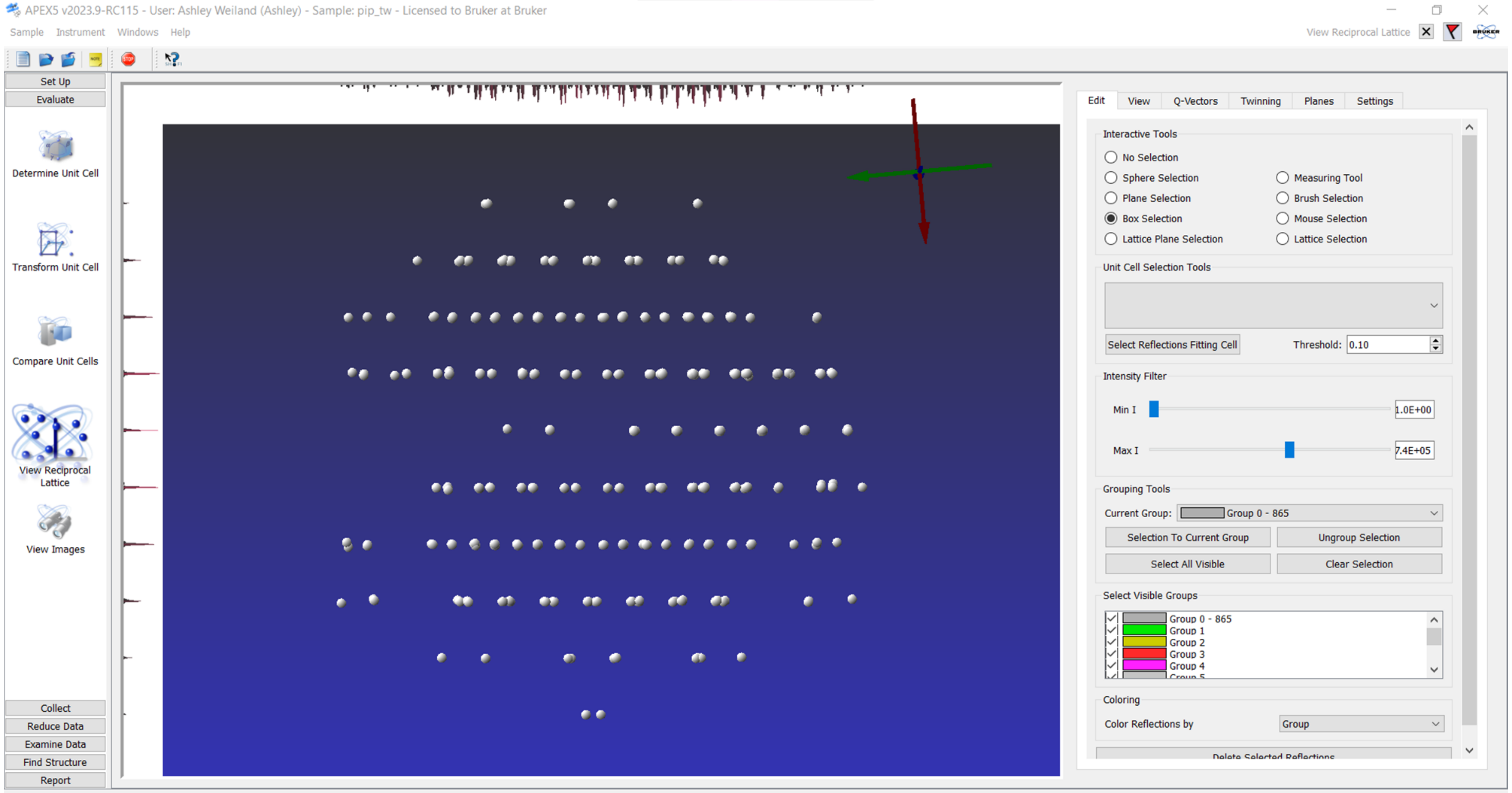


Twinning

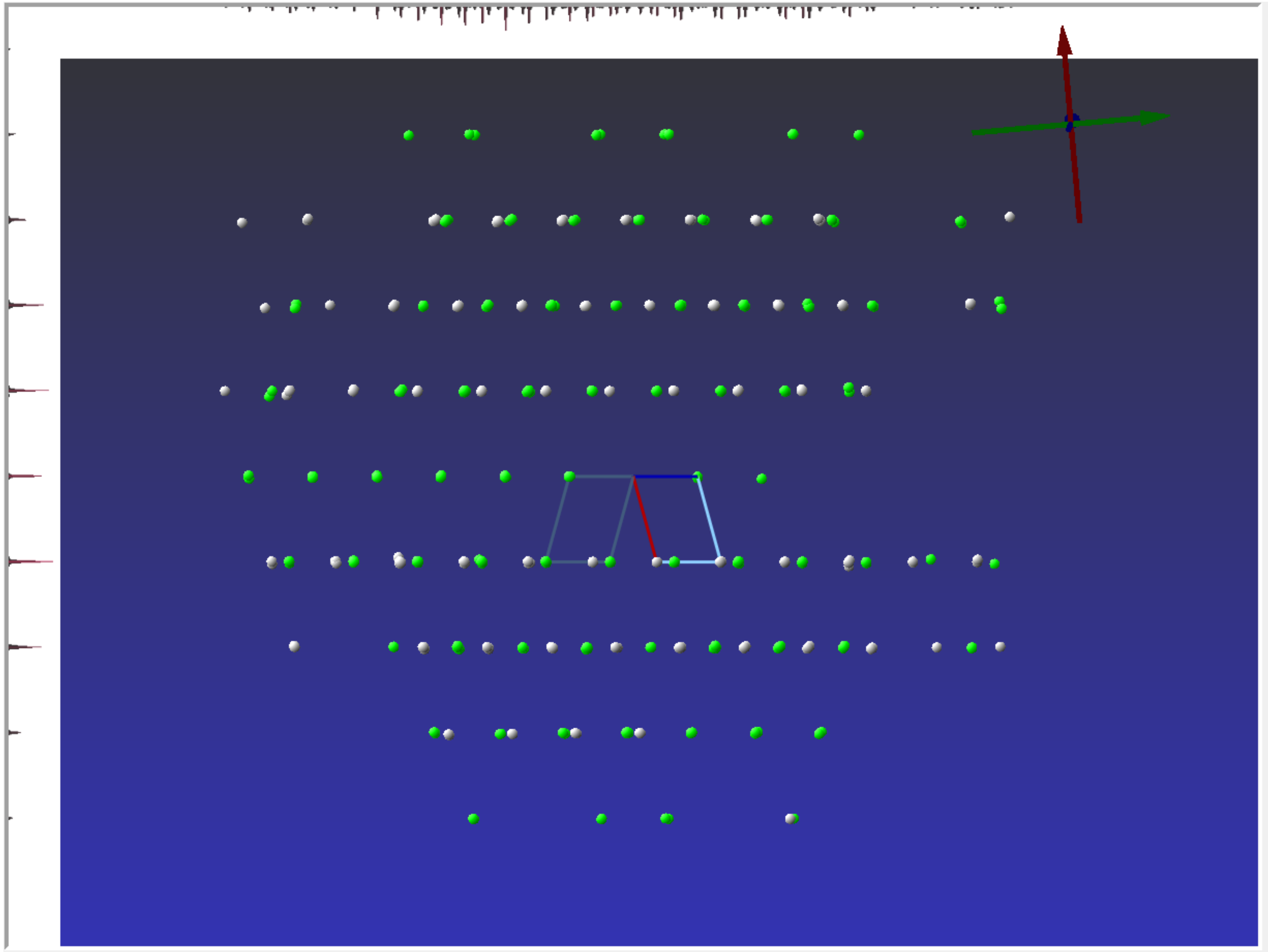
Seemingly Normal Lattice Lines



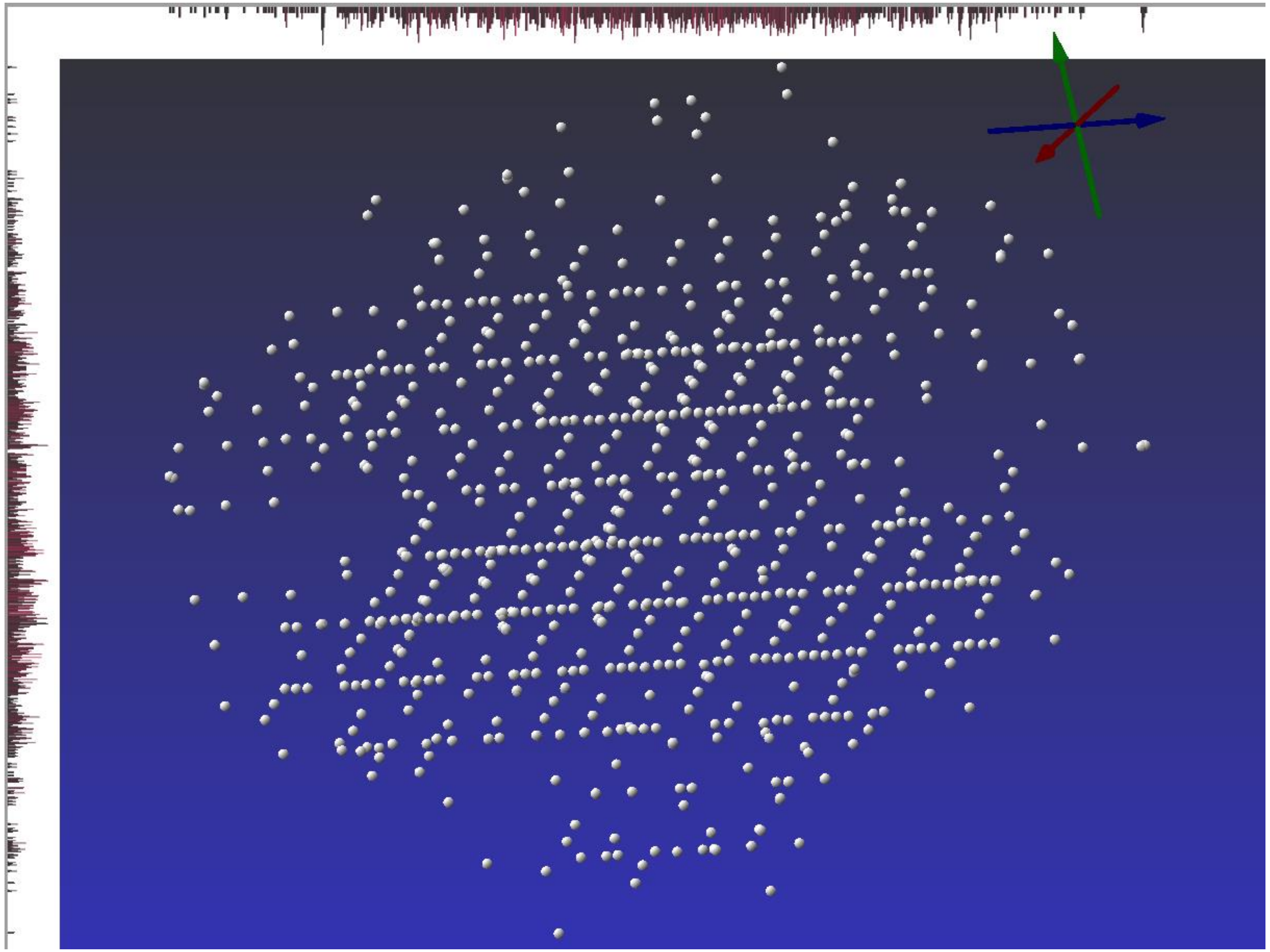
Indications of Twinning



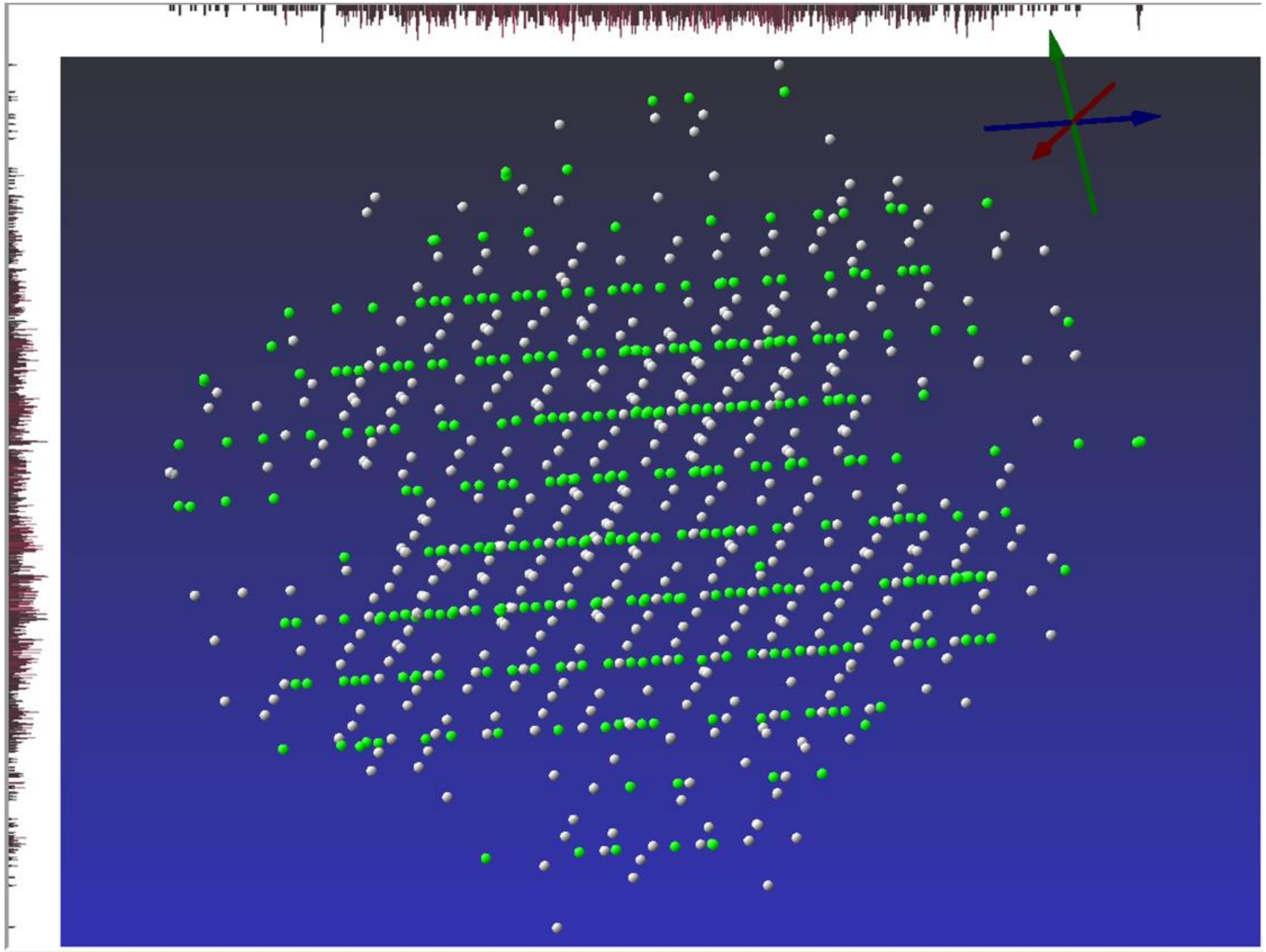
Twinning



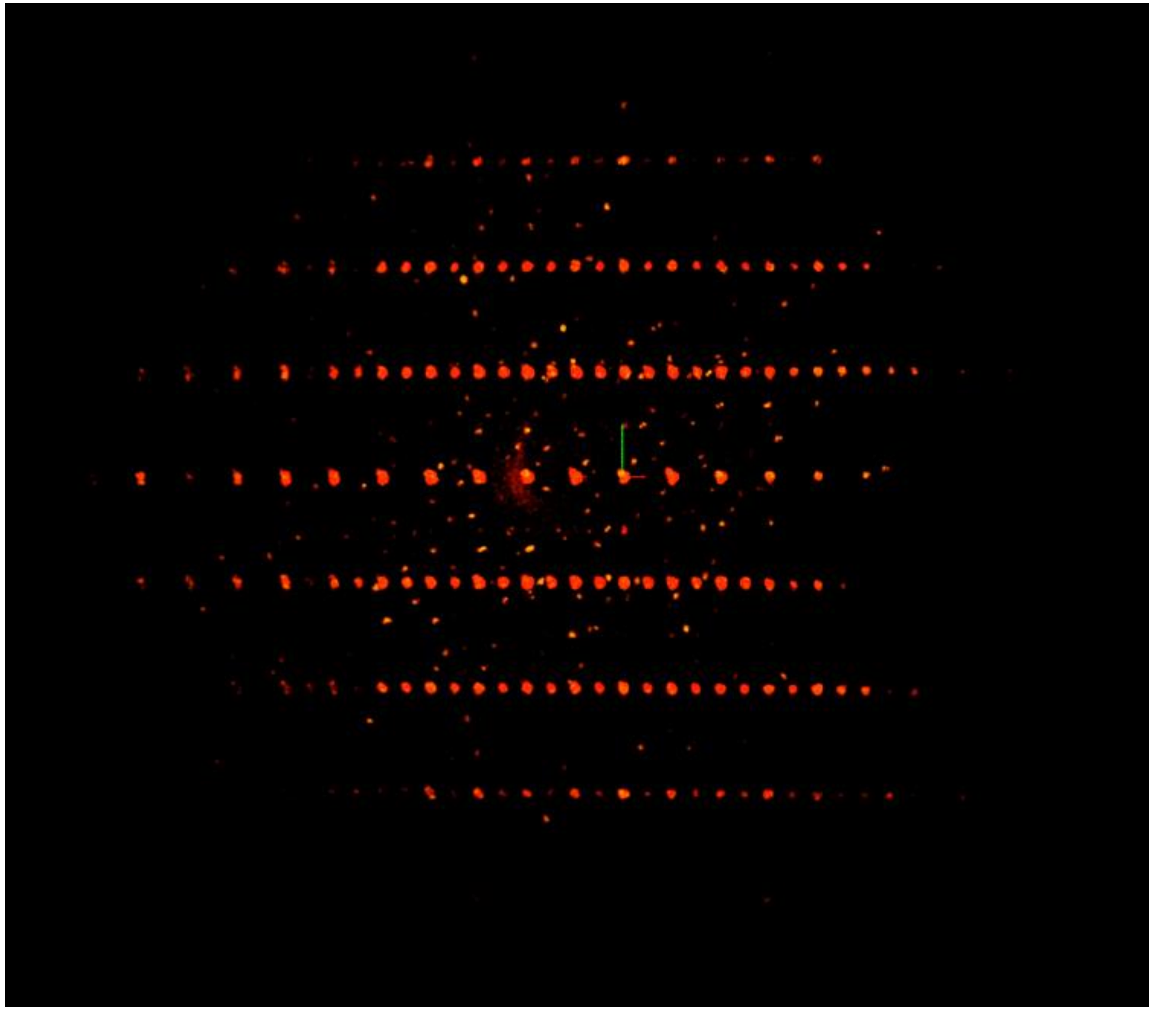
Twinning

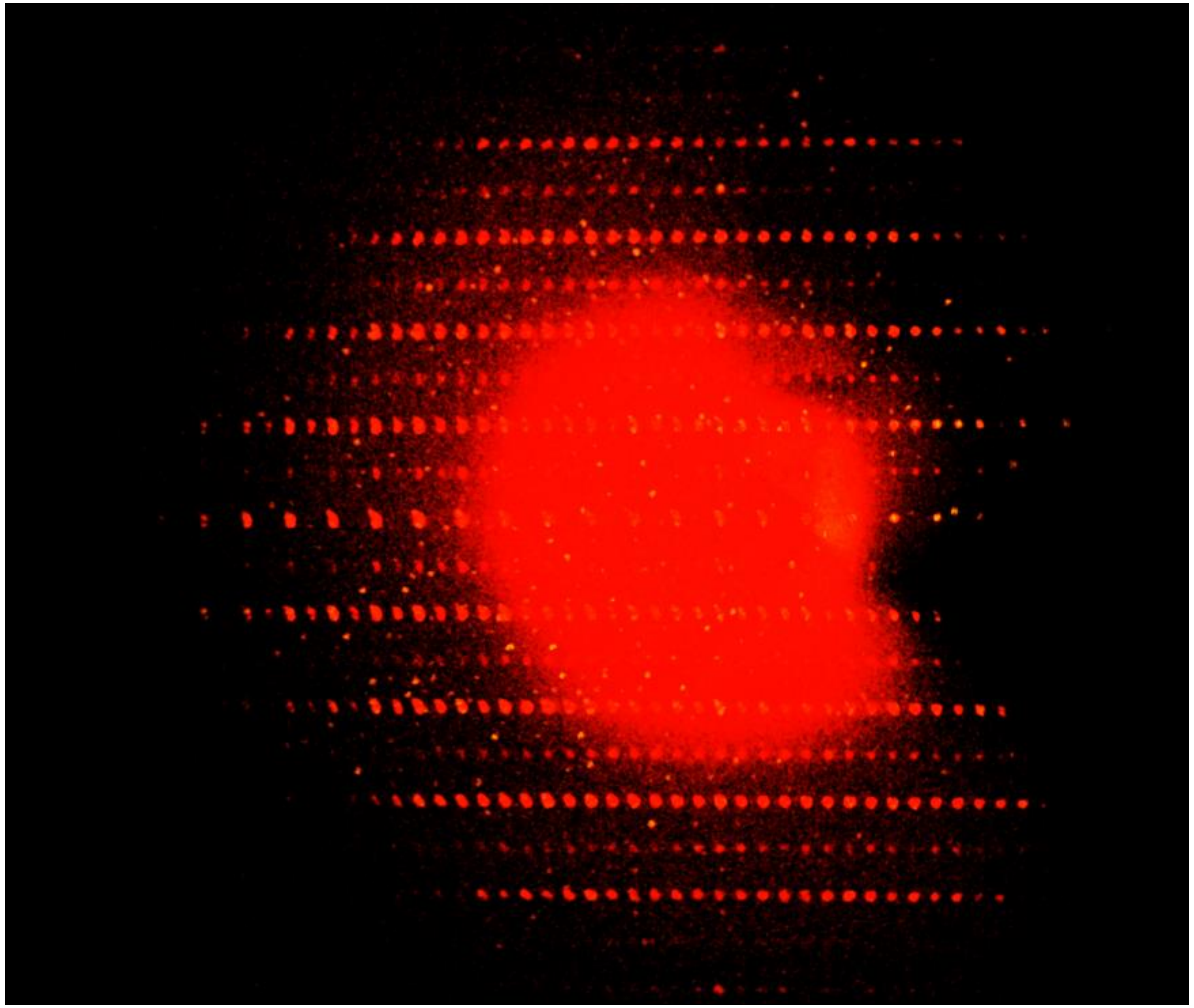


Twinning

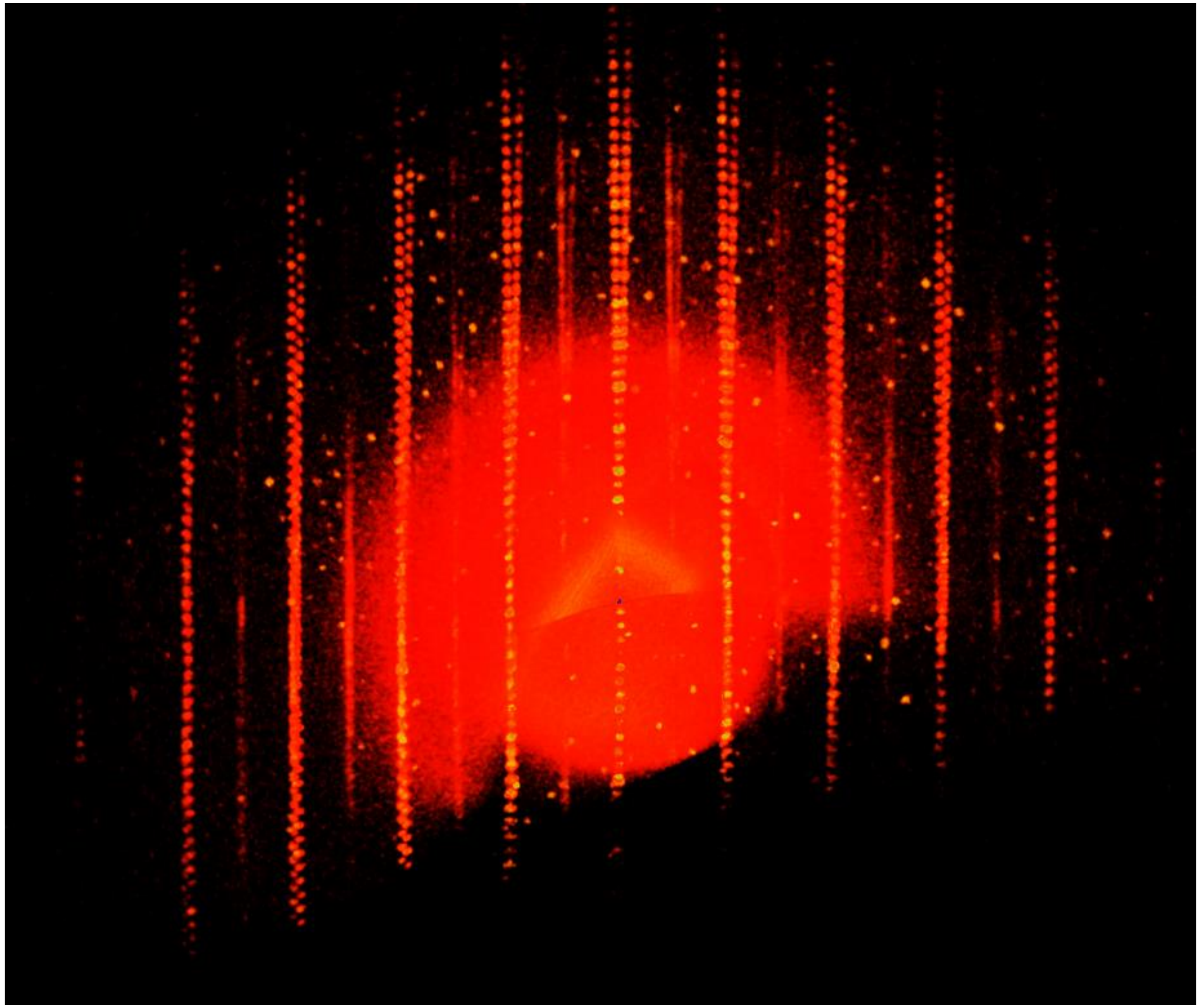


MAX3D





MAX3D

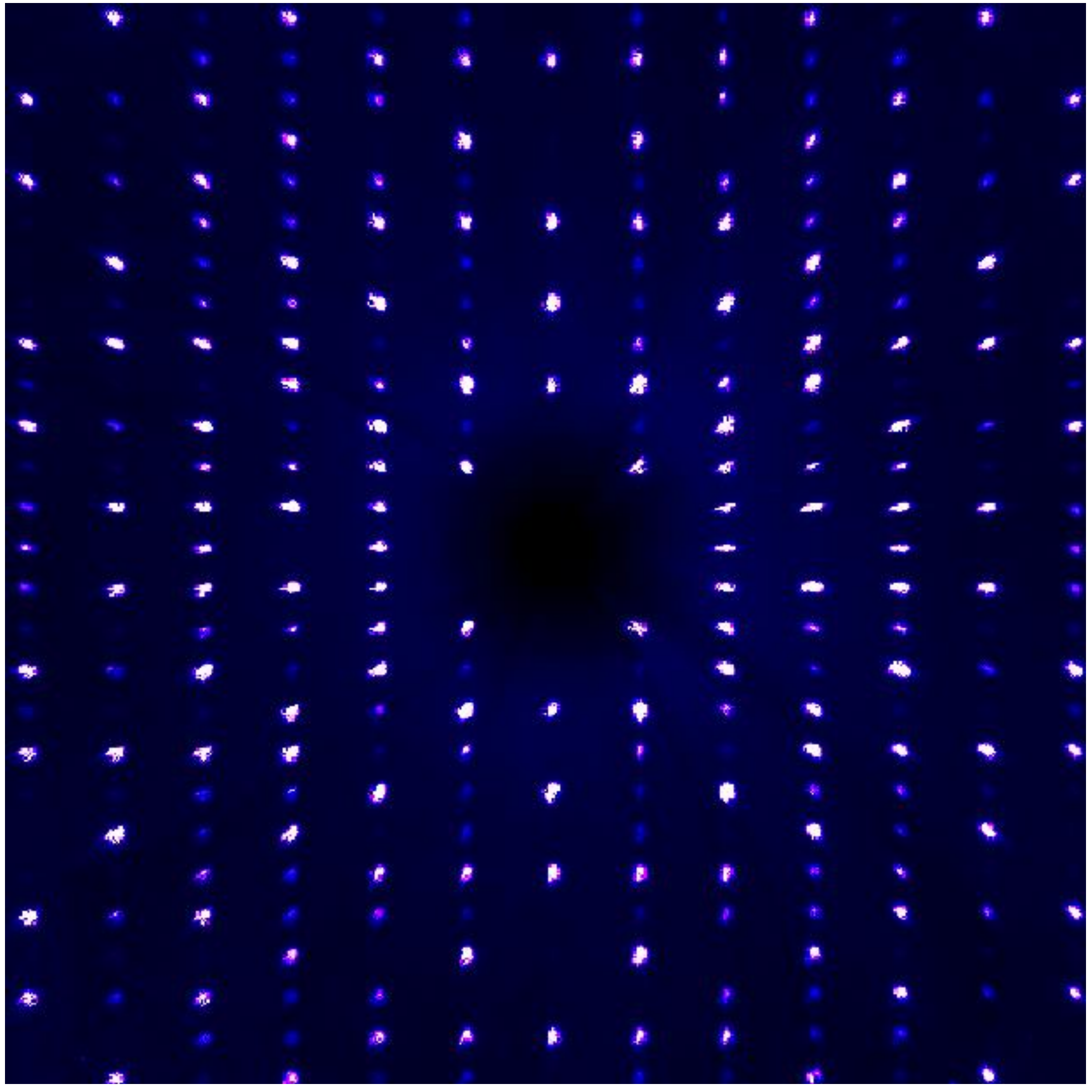


Reciprocal Lattice Points

- Are designated by their Miller index, hkl
- Assigning hkl values to the reciprocal lattice points is called **indexing the crystal** or **indexing the diffraction pattern**
- Reciprocal lattice points represent the diffraction from a **set of planes** designated by the hkl value and have a corresponding d* value
- Normal to the set of planes and therefore represent a direction in reciprocal space

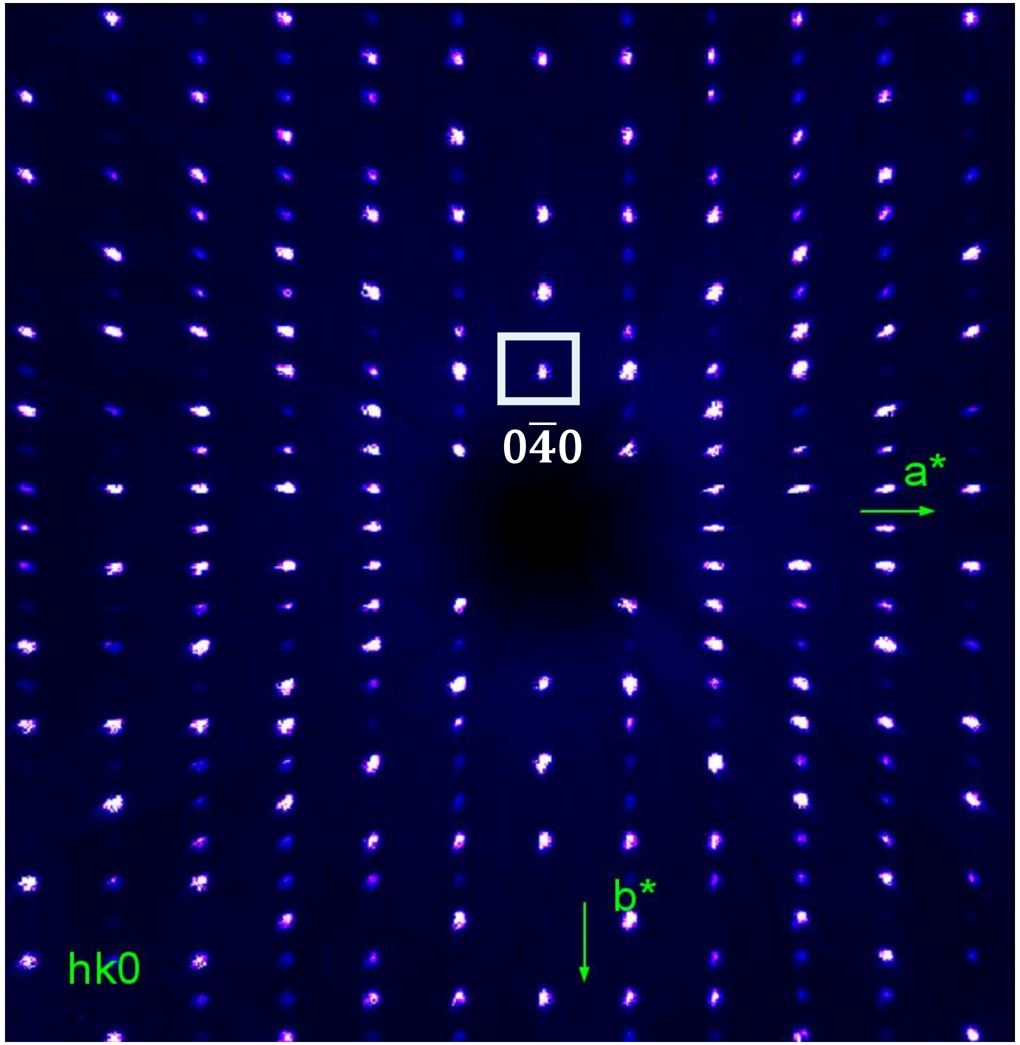
Indexing a Diffraction Pattern

- Synthesized reciprocal lattice layer (hk0) from an actual crystal
- Vertical axis has closer packed reciprocal lattice points
- Vertical axis has larger direct space unit cell parameter



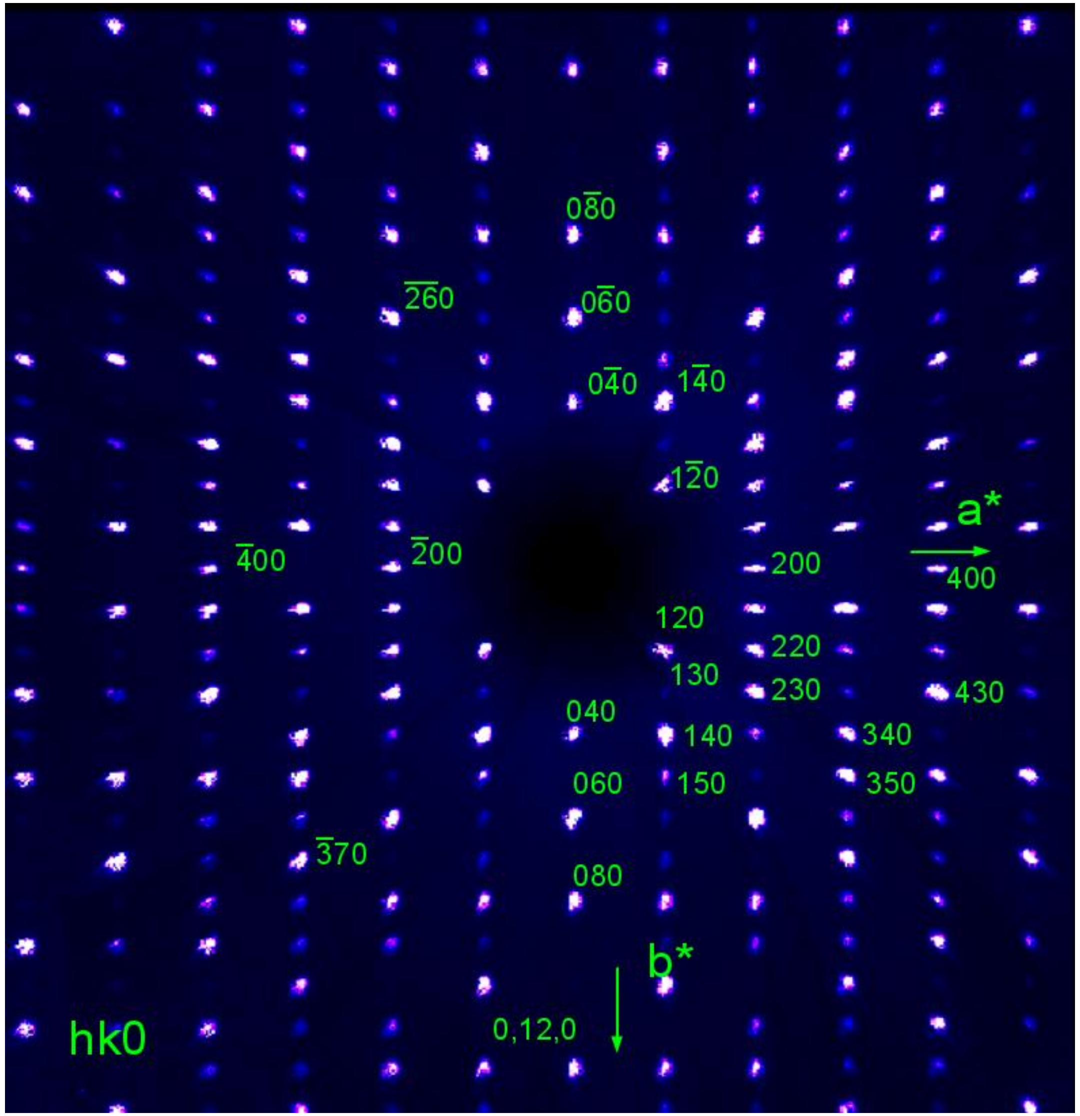
Indexing a Diffraction Pattern

- First assign the lattice directions
- Notice there are systematic absences along the h00 and 0k0 reciprocal axes
- Indicative of two screw axes (translational symmetry elements)



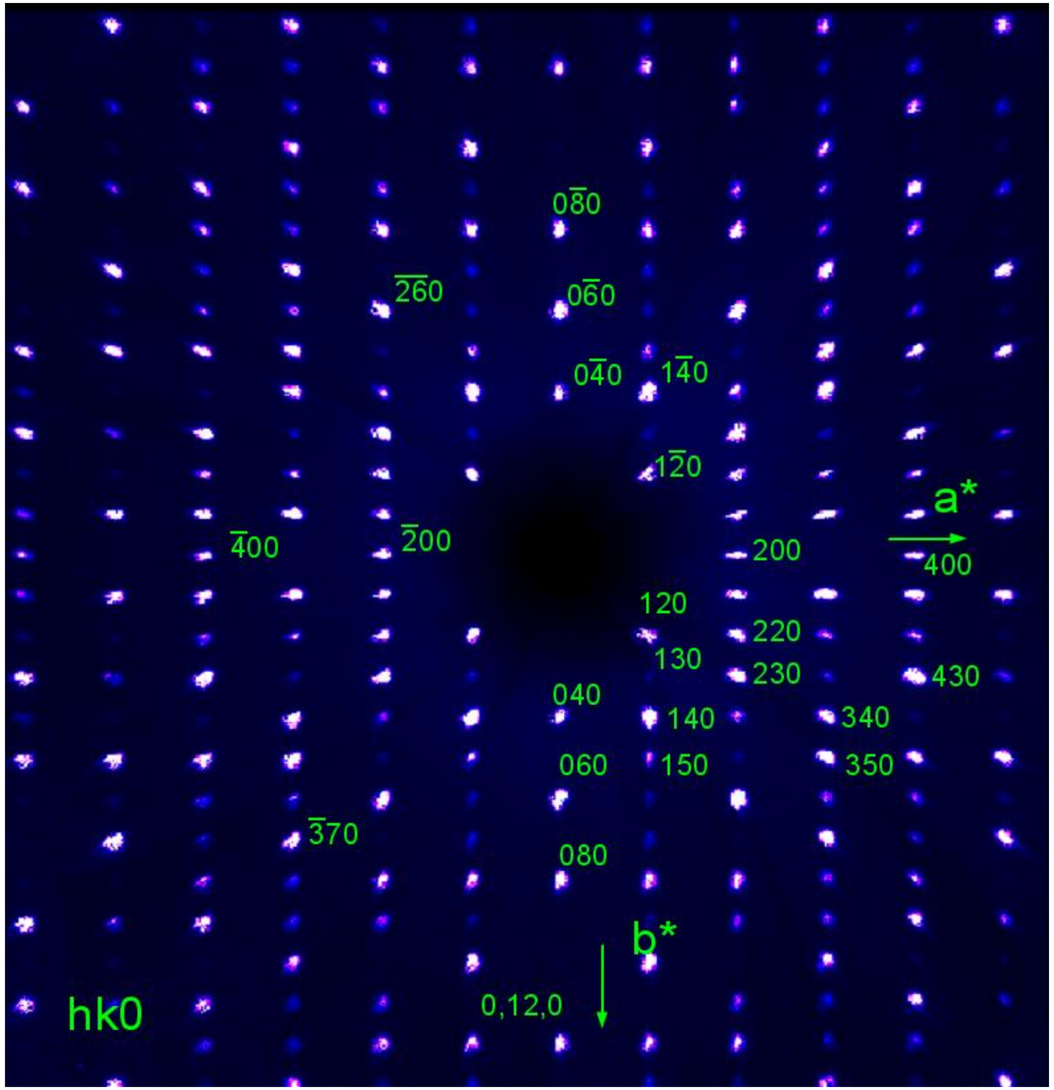
Indexing a Diffraction Pattern

- Assign hkl values to each reciprocal lattice point
- Use Bragg's Law to calculate the interplanar spacing associated with each reciprocal lattice point
- Measure angle between a* and b* to obtain γ*
- Repeat process with other zero layers (0kl and h0l)



How to think about this

- Each reciprocal lattice point represents both a direction and d spacing
- With each reciprocal lattice point measured, we are “sampling” the electron density with certain spatial frequency in a given direction



Practical Considerations for Data Collection

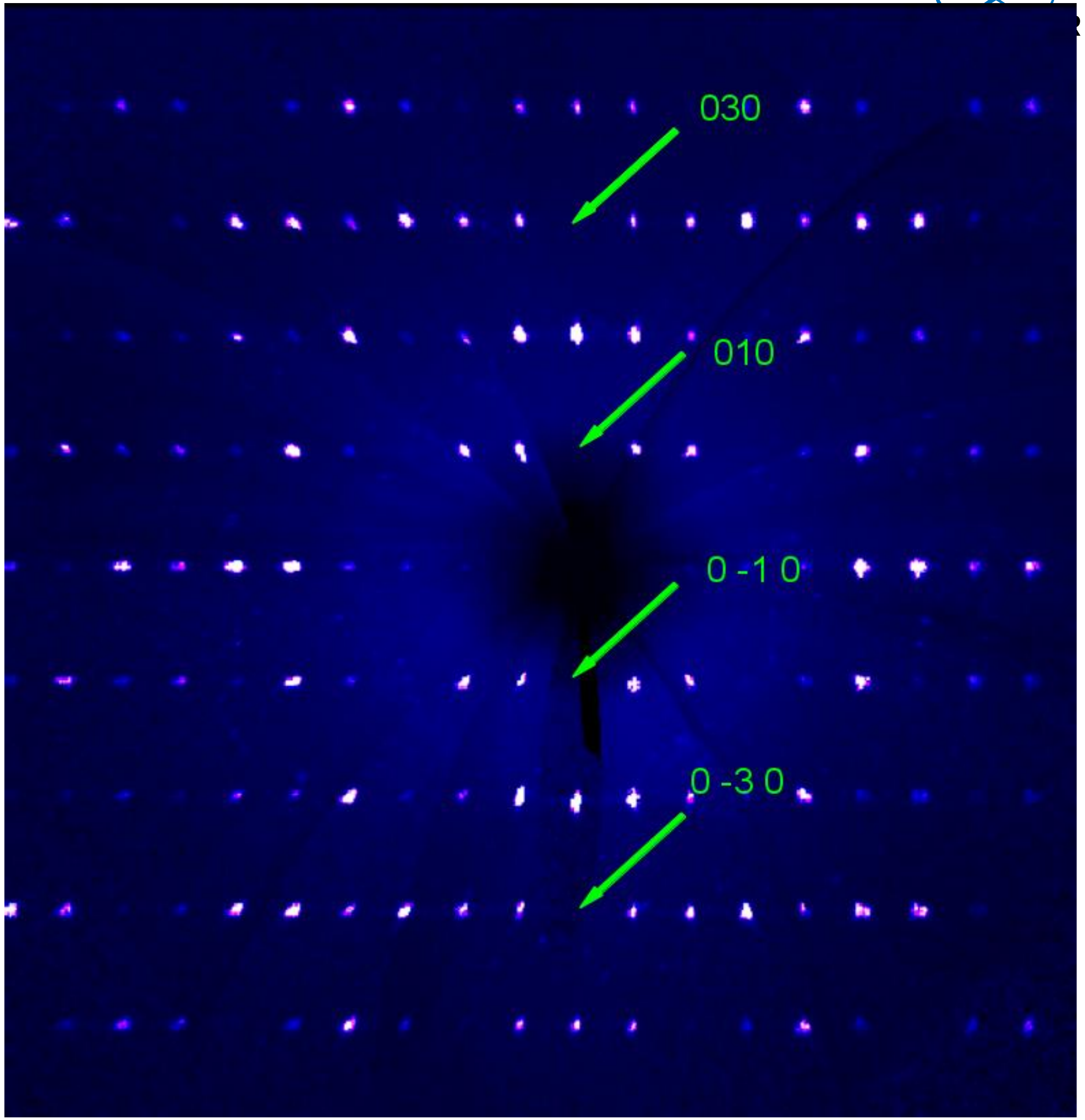
- Long axes give densely packed reciprocal lattice rows
- Integration is better if peaks aren't overlapping
- Choose minimum crystal to detector distance as:
  - DX(mm) = 2 * longest primitive axis (Å) [MoKα]
  - DX(mm) = 1 * longest primitive axis (Å) [CuKα]
- For non-merohedrally twinned samples, move the detector back even farther

Experimental Determination of Space Group

- Space groups are determined primarily through the examination of systematic absences in the diffraction pattern
- Systematic absences arise from the presence of translational symmetry elements
  - Non-primitive lattice centerings
  - Screw axes (rotation with translation)
  - Glide planes (reflection with translation)

Screw Axis Absences

- Screw axes affect the classes of axial reflections: h00, 0k0, and 00l
- The type of screw axis is determined by examining the pattern of the absence
- Example: In this figure there is a 2_1 axis parallel to b*
- 0k0: k = odd



Orientation of Glide Planes

- When a glide plane is present one can determine the orientation and type of glide plane present from the affected class(es) of reflections
- The 0 index of the affected layer indicates the orientation of the glide's reflection
  - 0kl: glide reflects across (100)
  - h0l: glide reflects across (010)
  - hk0: glide reflects across (001)

Identification of Glide Planes

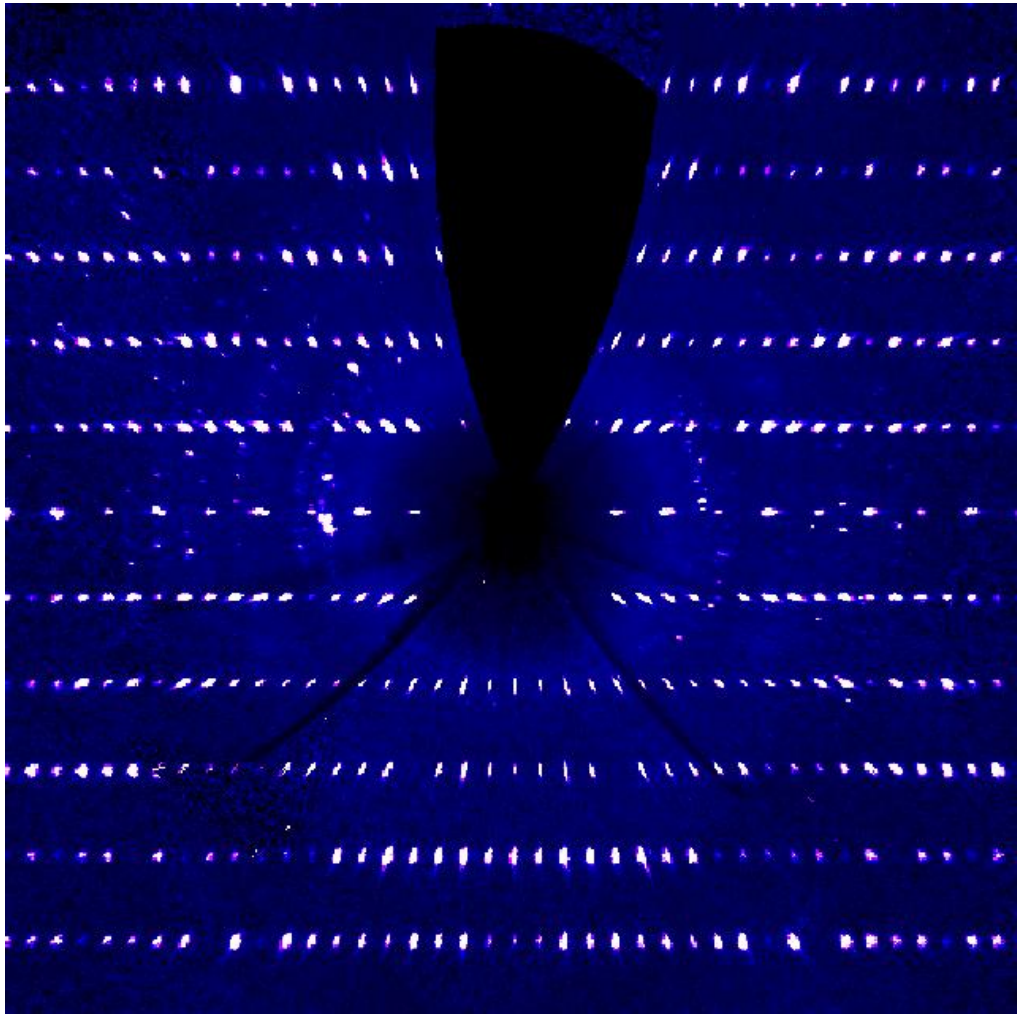
- The translational component identifies the type of glide plane
- The translational component causes absences in along the affected axes
- 0kl:

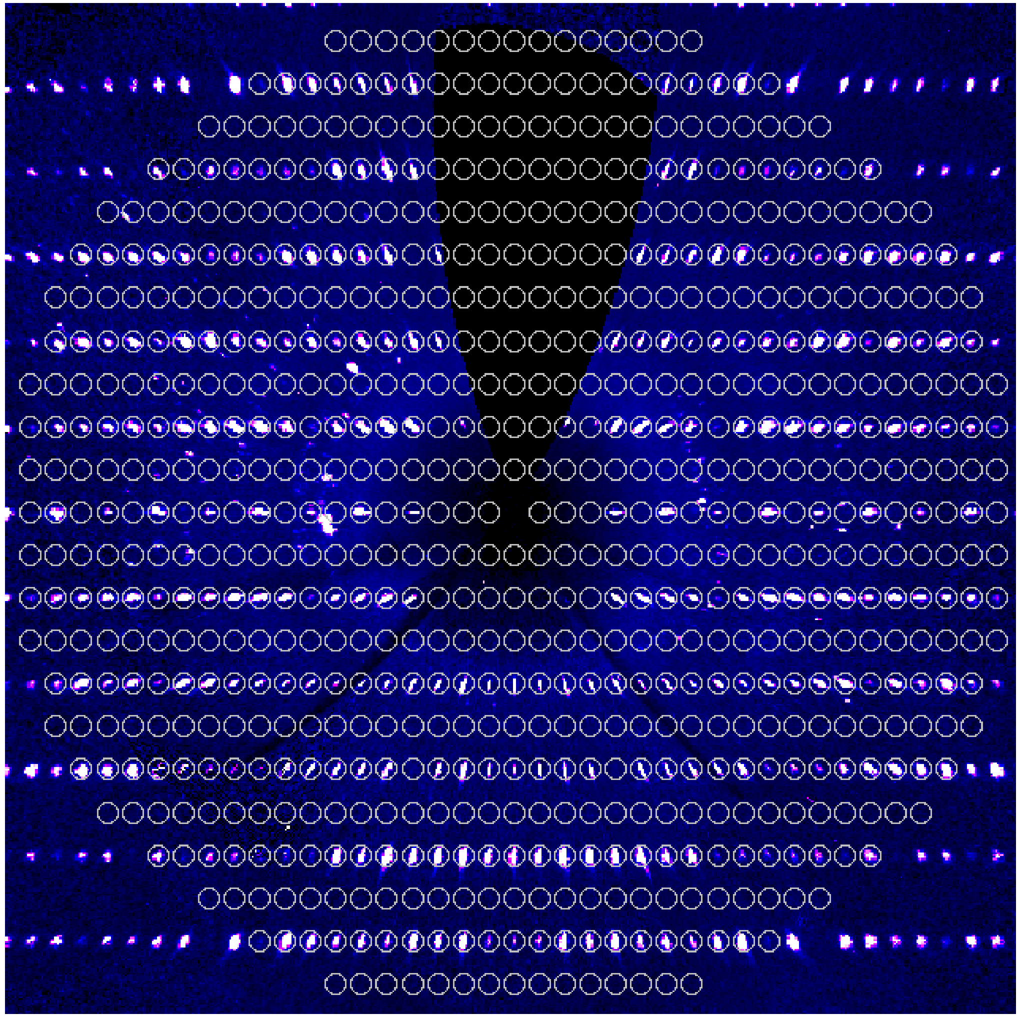
  k = odd → b glide; l = odd → c glide; k+l = odd →n glide
- h0l:

  h = odd → a glide; l = odd → c glide; h+l = odd → n glide
- hk0:

  h = odd → a glide; k = odd → b glide; h+k = odd → n glide

Example of c glide (h0l: l = odd)





Thank you!

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