F69 Laue X-Ray Diffraction

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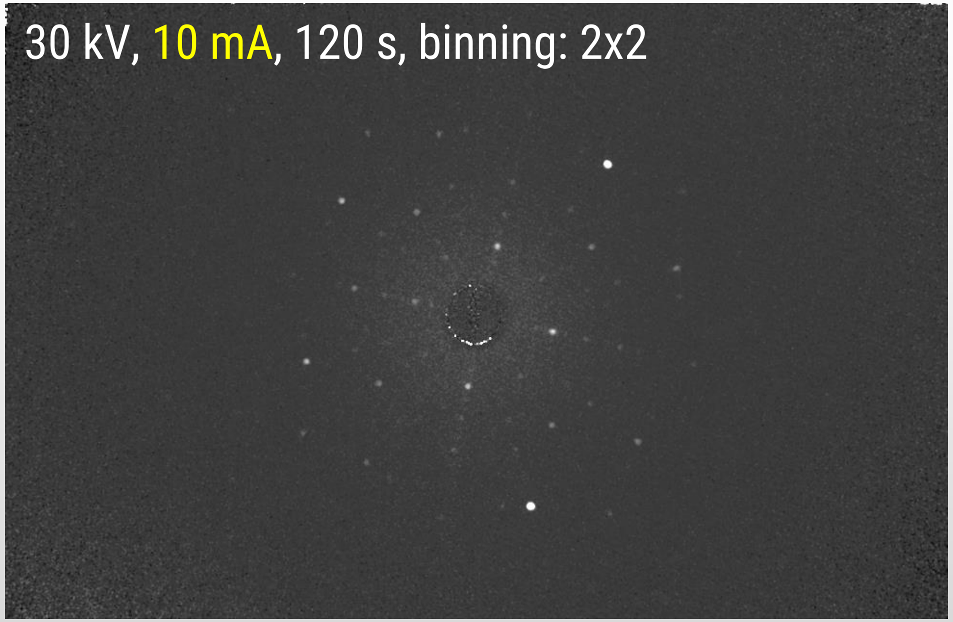
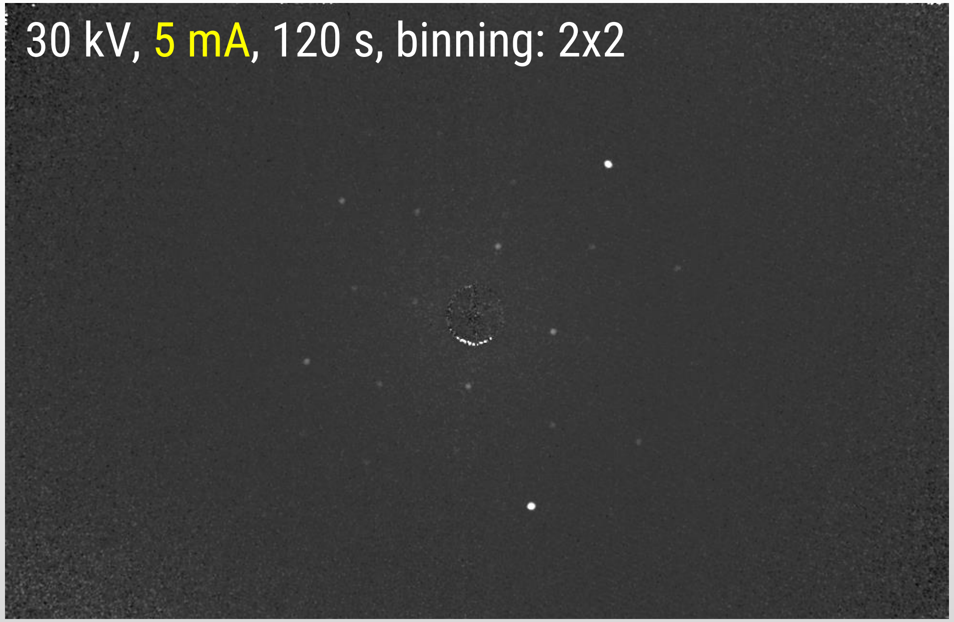
9.1 Silicon Wafer

Varying X-ray tube current (heating current)

- **Observation:**
  - Higher intensity
    ➔ More reflexes visible
- **Explanation:**
  - A higher current = more electrons
  - More electrons = more X-ray photons
    ➔ Higher intensity



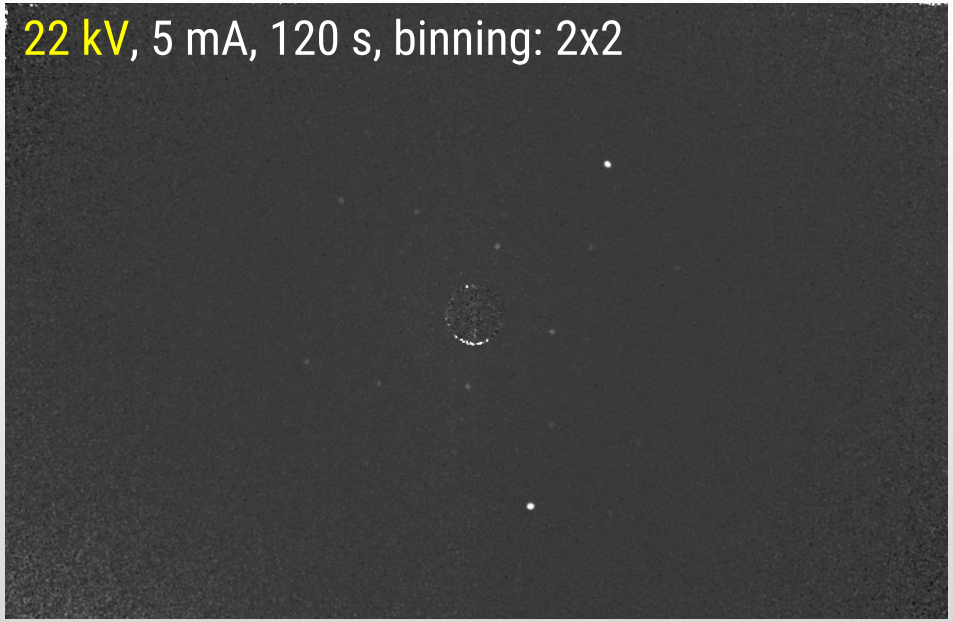


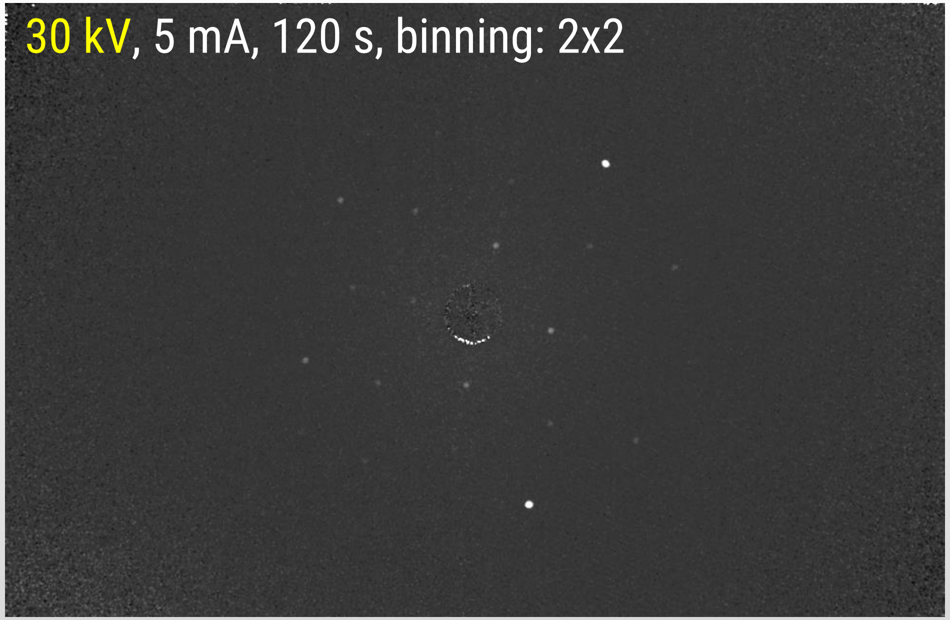
9.1 Silicon Wafer

Varying X-ray tube voltage (acceleration voltage)

- **Observation:**
  - The intensity of the diffraction reflexes is a little bit higher

- **Explanation:**
  - The higher voltage leads to higher energies of the e^-
    ➔ higher intensity of γ



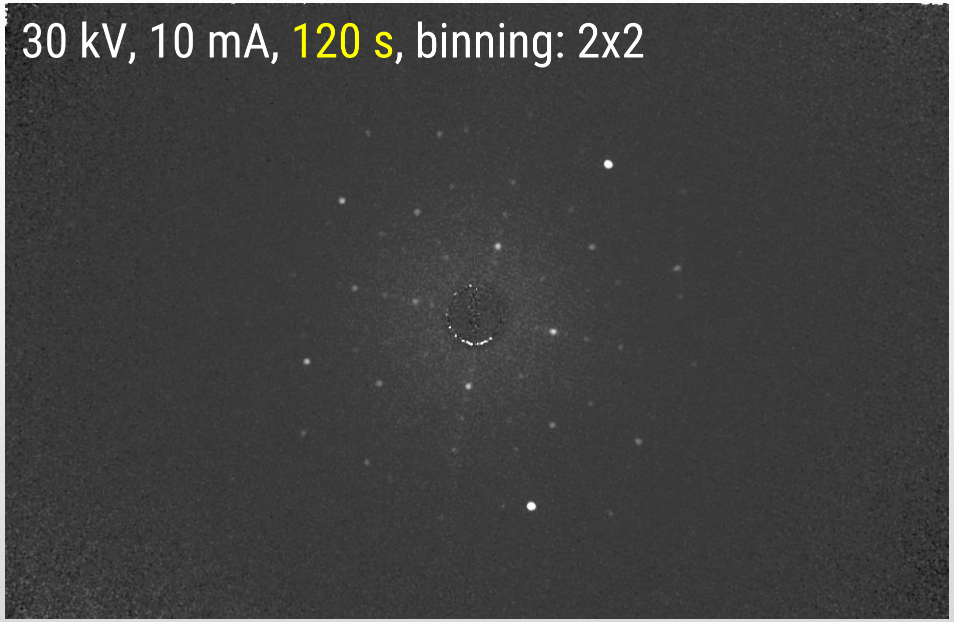


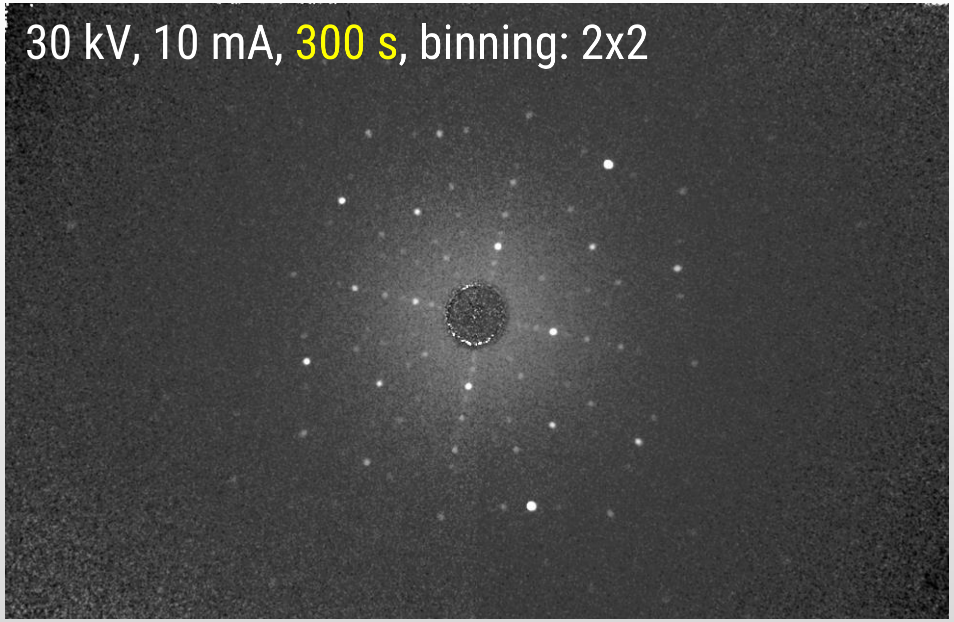
9.1 Silicon Wafer

Varying image-acquisition integration time

- **Observation:**
  - The image gets brighter
  - The intensity of the reflexes gets larger but at the same time the background gets more noisy

- **Explanation:**
  - The larger the integration time, the brighter *all* pixels



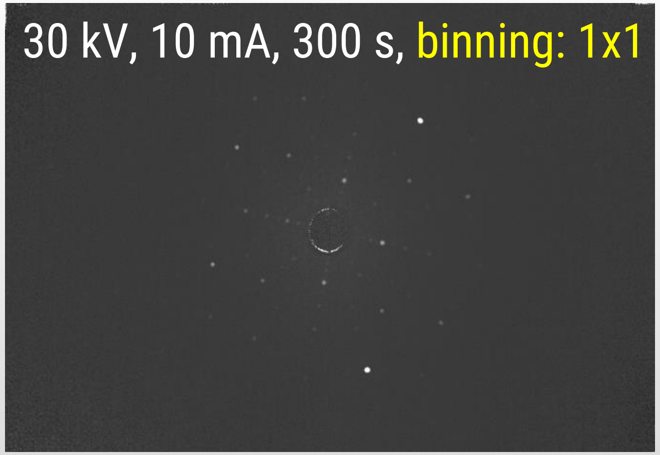


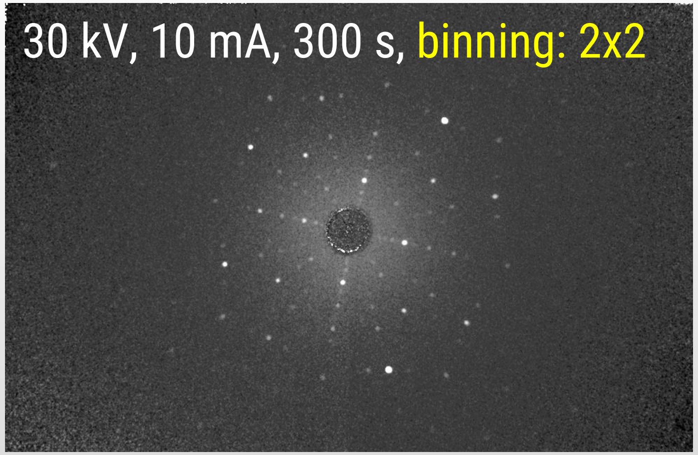
9.1 Silicon Wafer

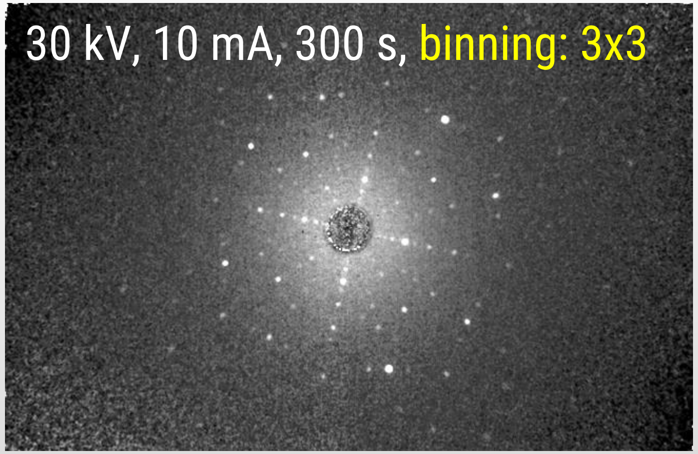
Varying software binning

- **Observation:**
  - The image and the dots get brighter
  - At the same time the background gets more and more noisy

- **Explanation:**
  - The binning defines how many pixels get evaluated together, i.e. with a binning of 1x1 each pixel gets evaluated individually (integrated individually)



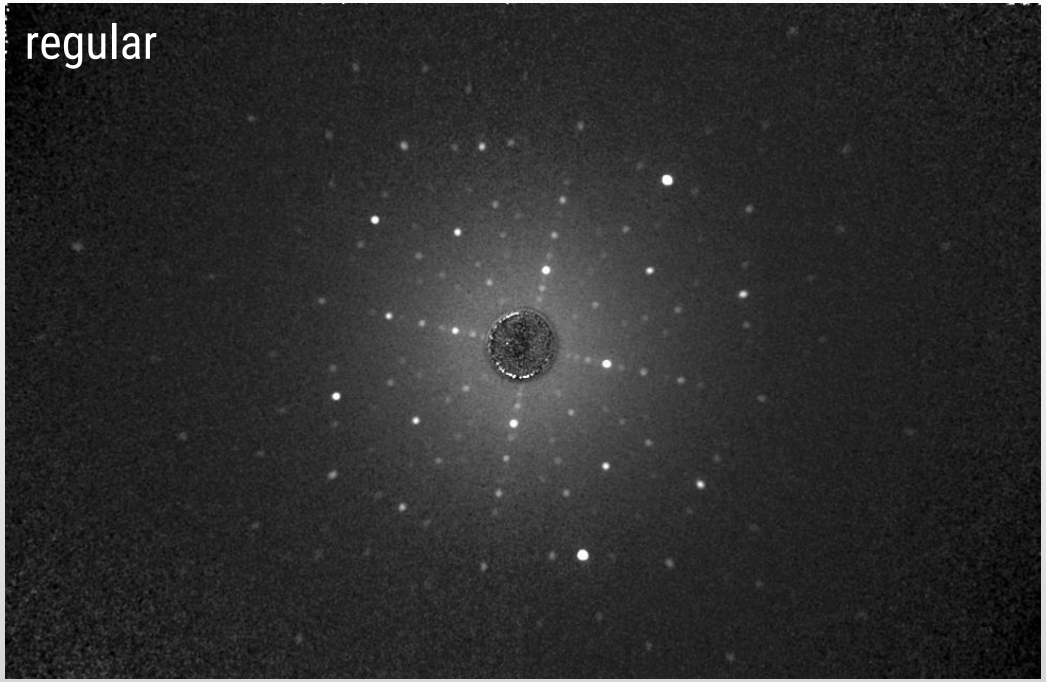


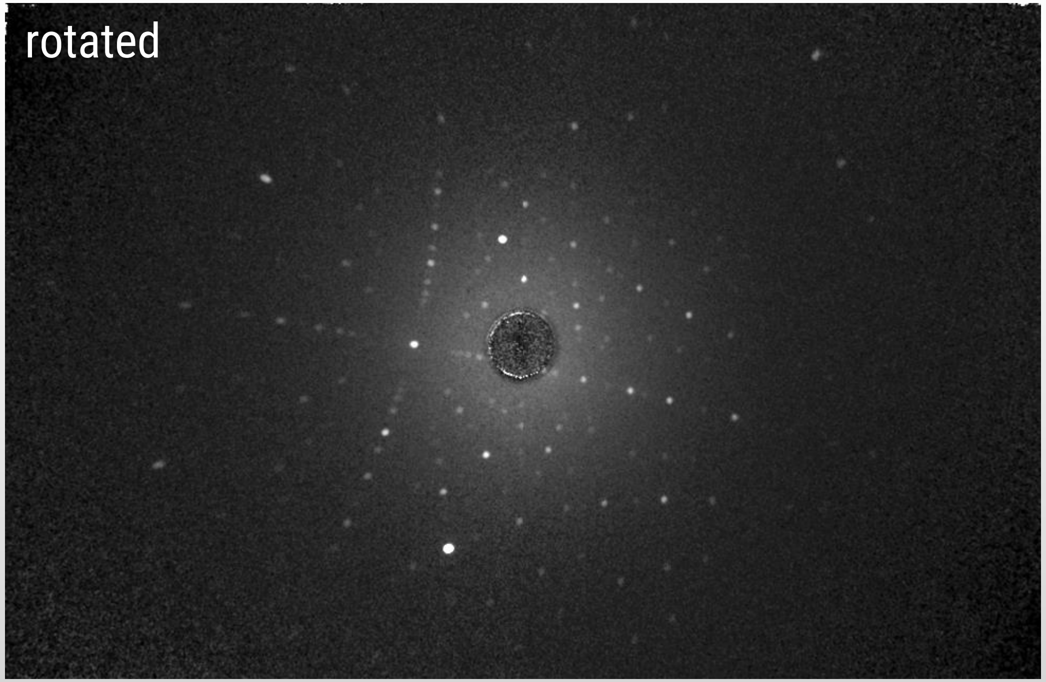


9.1 Silicon Wafer

Results of adjusting the parameters

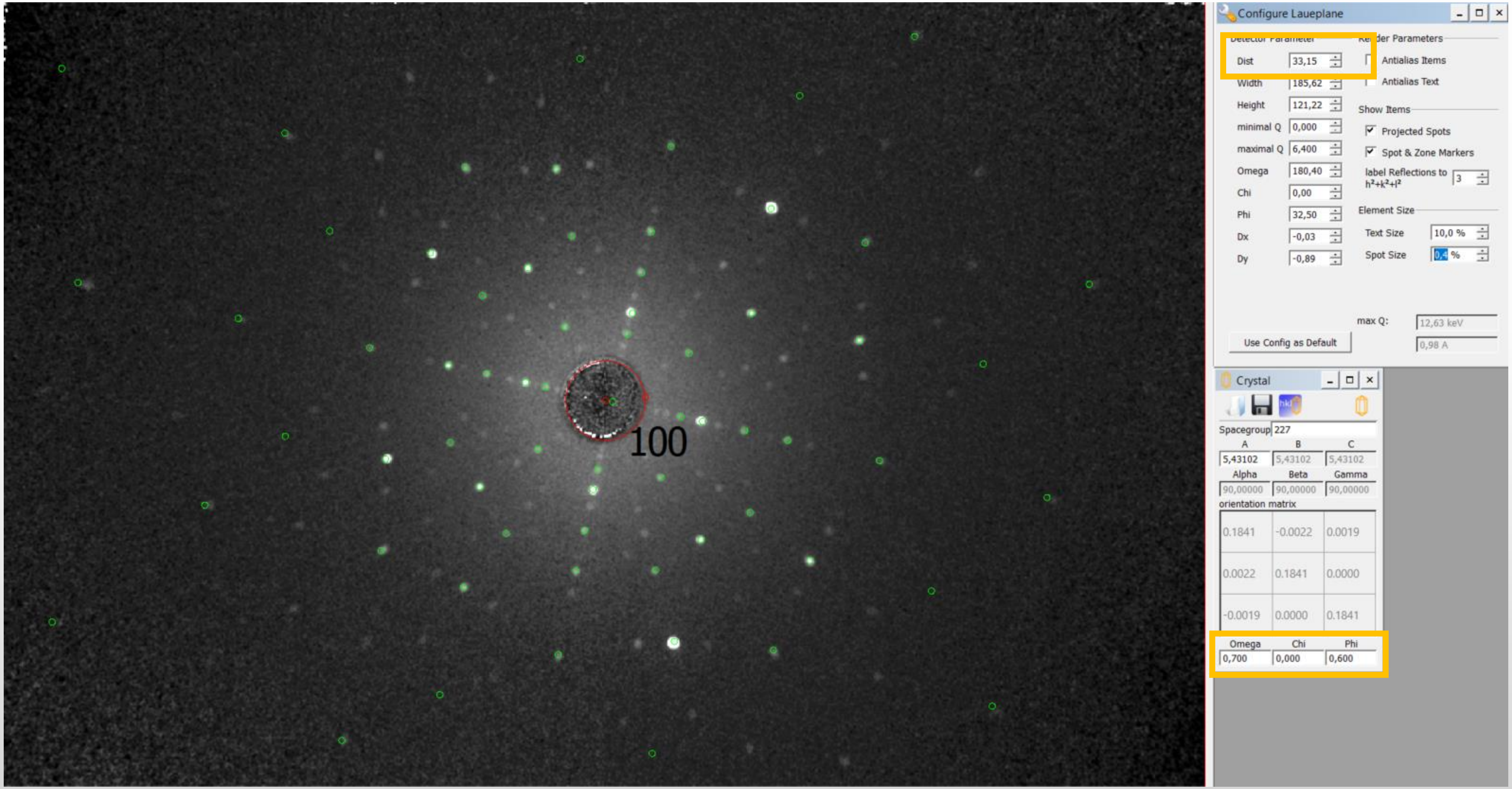
- The best recordings of the diffraction patterns that we received are done with the following parameters:
  - $U_a = 30kV$
  - $I_h = 15mA$
  - $\Delta t = 500s$
  - Binning: $2x2$





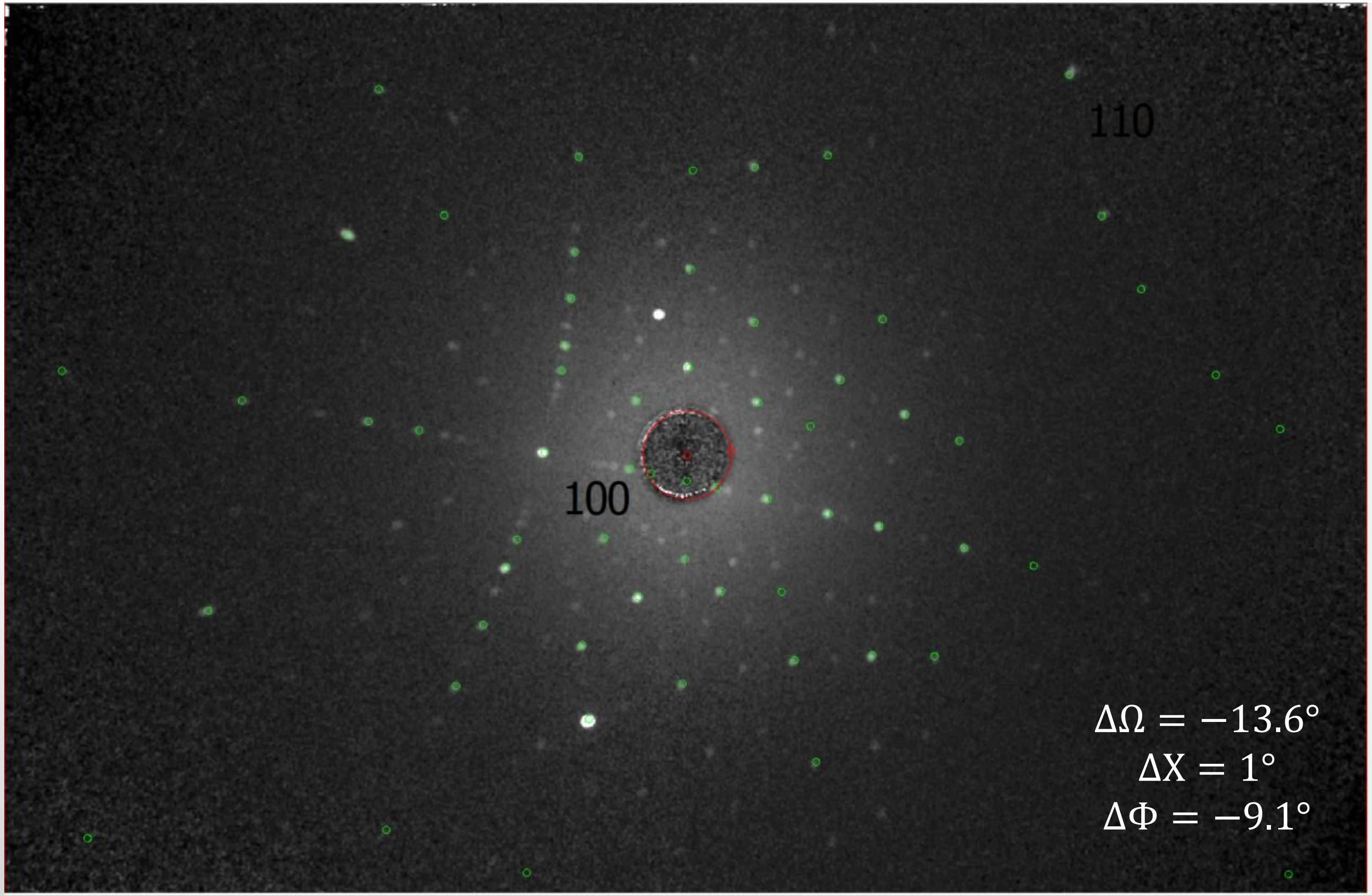
9.1 Silicon Wafer

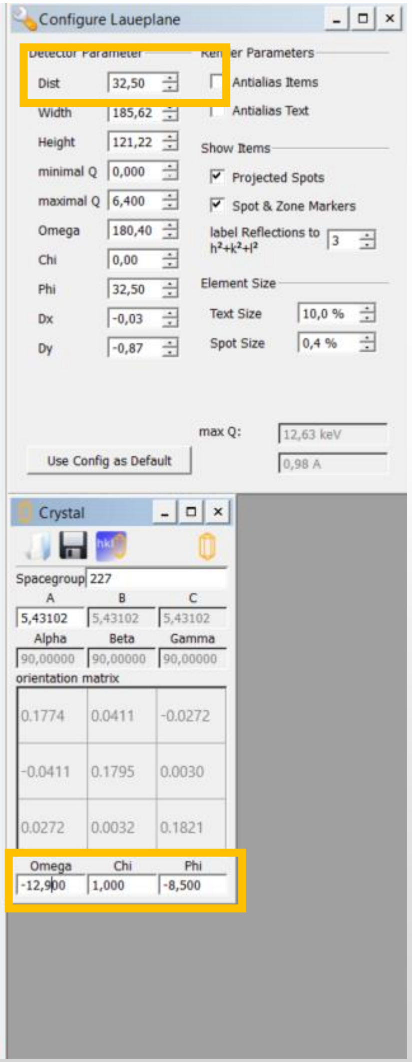
30kV_15mA_500s.PNG



9.1 Silicon Wafer

30kV_15mA_500s_rotiert.PNG

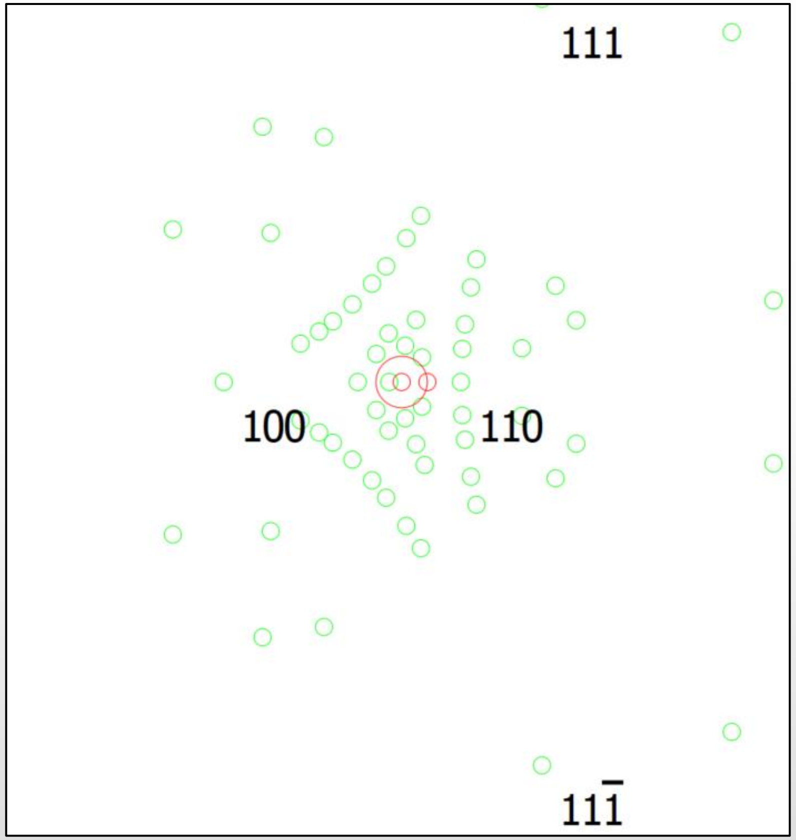




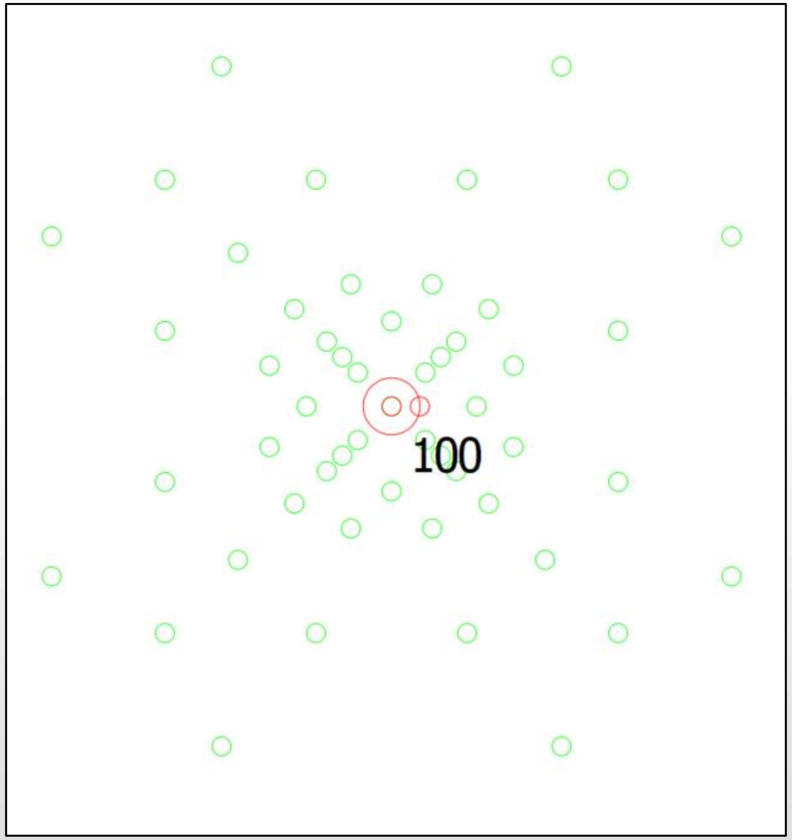
9.1 Silicon Wafer

Effect of the rotation around Ω

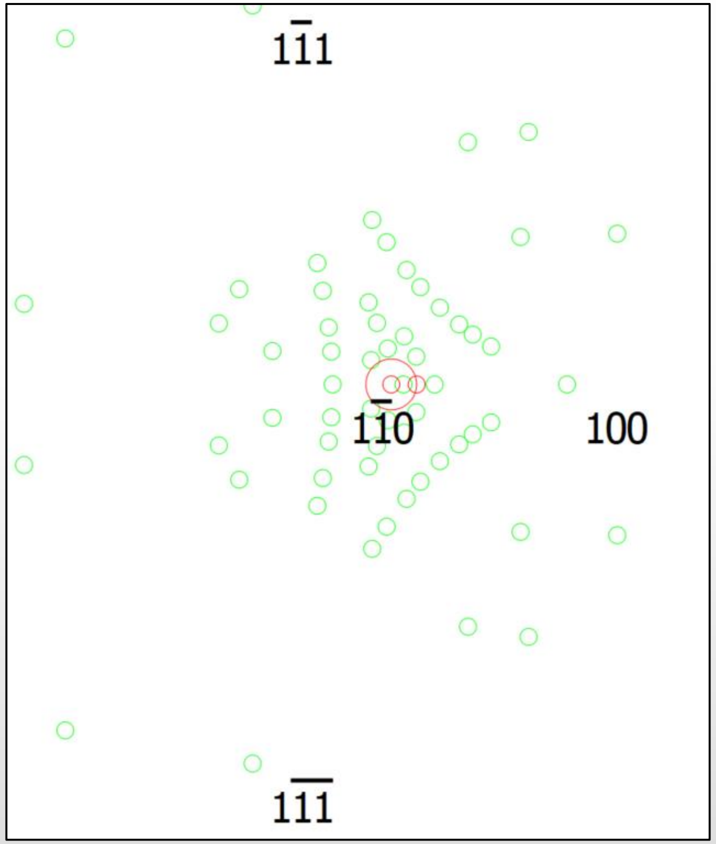
Ω = -30°



Ω = 0°



Ω = +30°

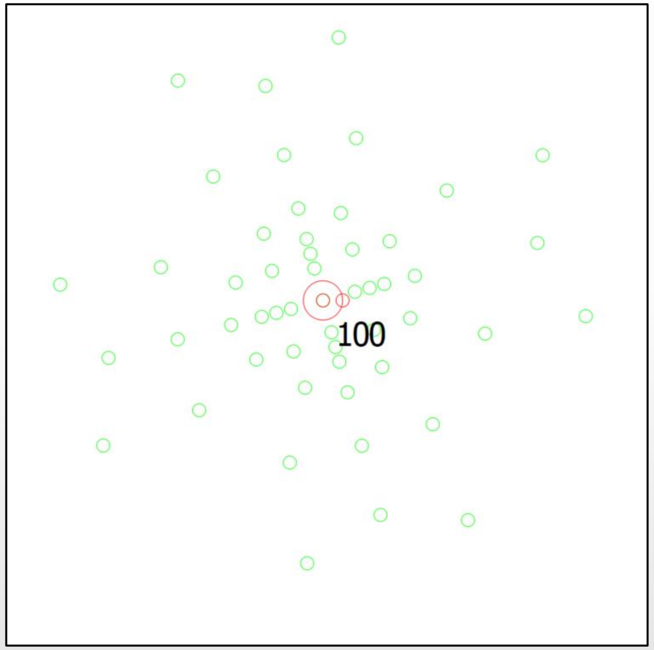


➔ Rotation around z-axis

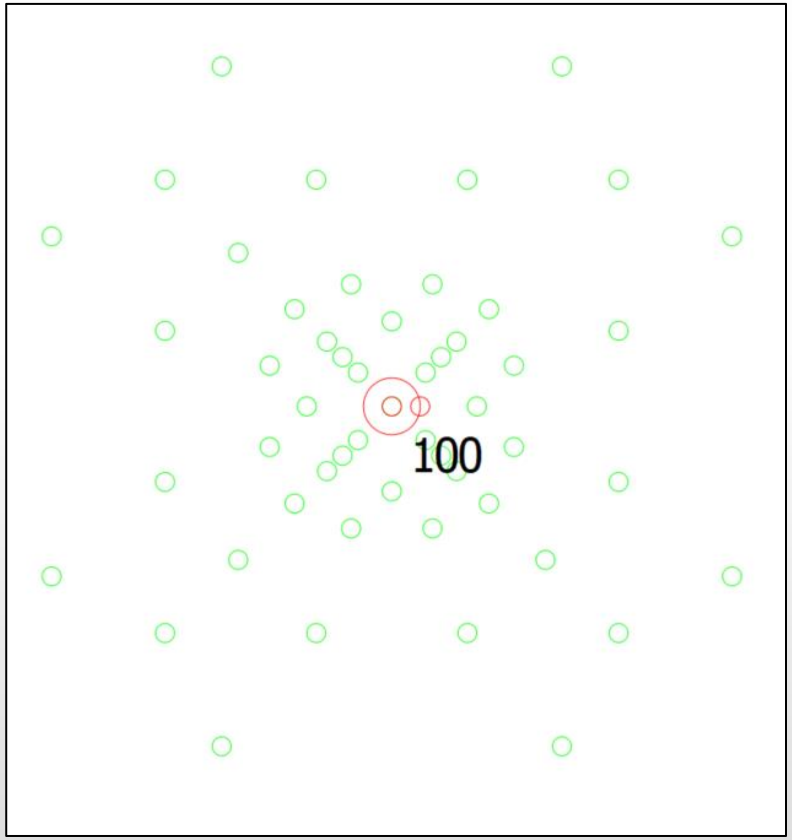
9.1 Silicon Wafer

Effect of the rotation around X

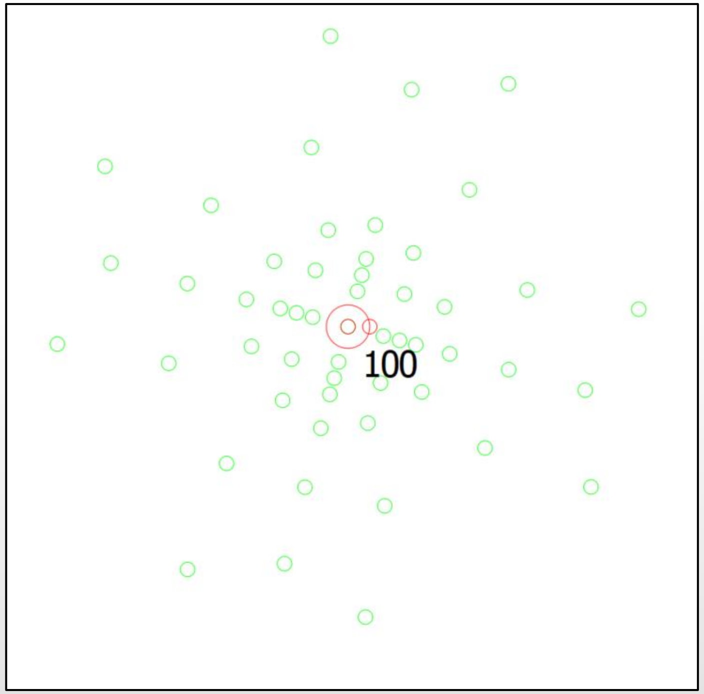
X = -30°



X = 0°



X = +30°

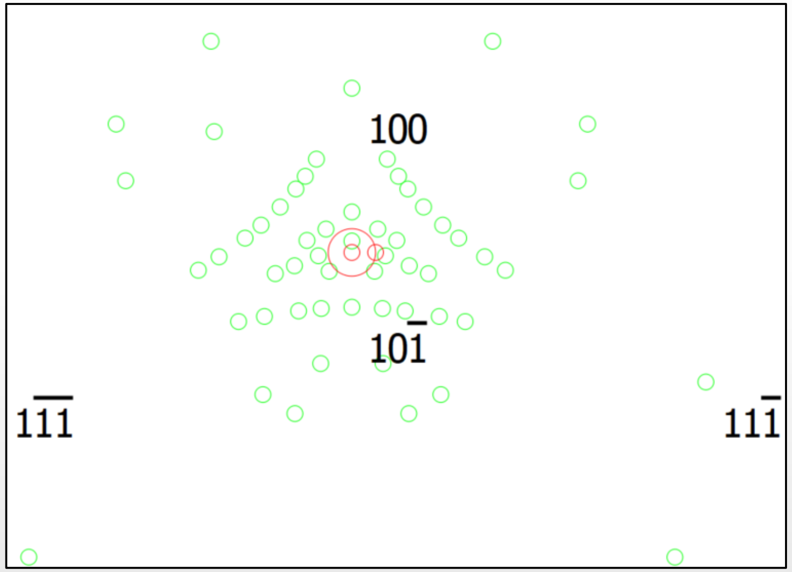


➔ Rotation around x-axis (axis of view)

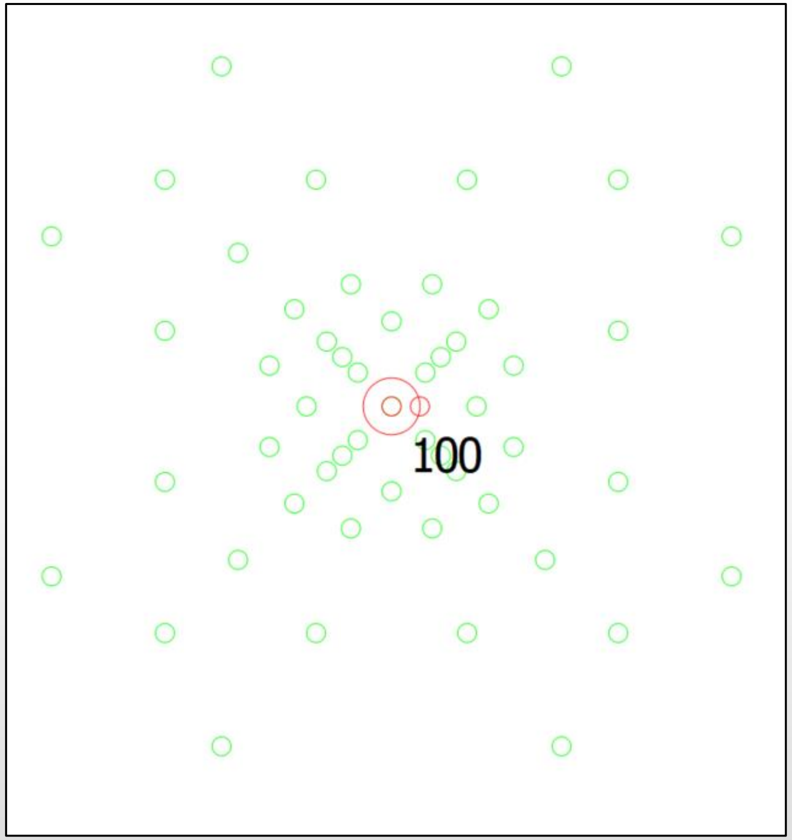
9.1 Silicon Wafer

Effect of the rotation around Φ

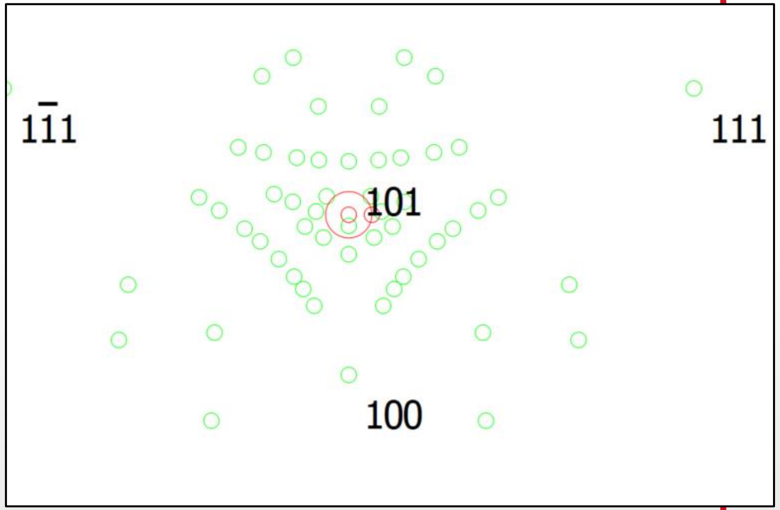
Φ = -30°



Φ = 0°



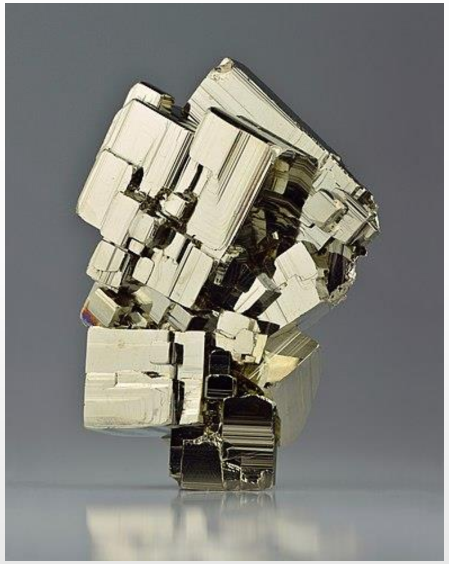
Φ = +30°



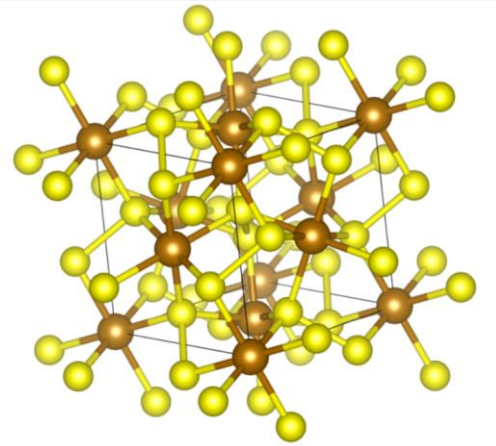
➔ Rotation around y-axis

9.2 Crystal of your choice

Pyrite FeS_2

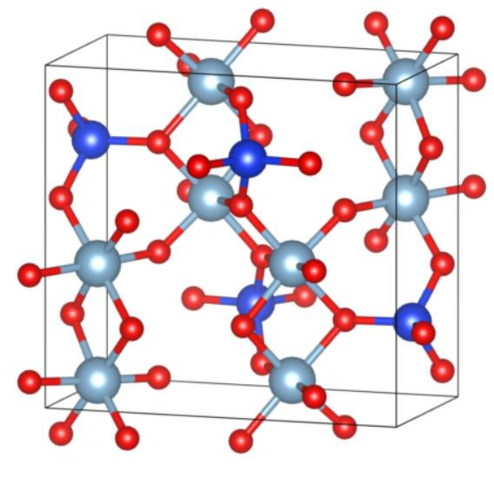


https://en.wikipedia.org/wiki/Pyrite



Topaz $Al_2SiO_4(F,OH)_2$

https://en.wikipedia.org/wiki/Topaz



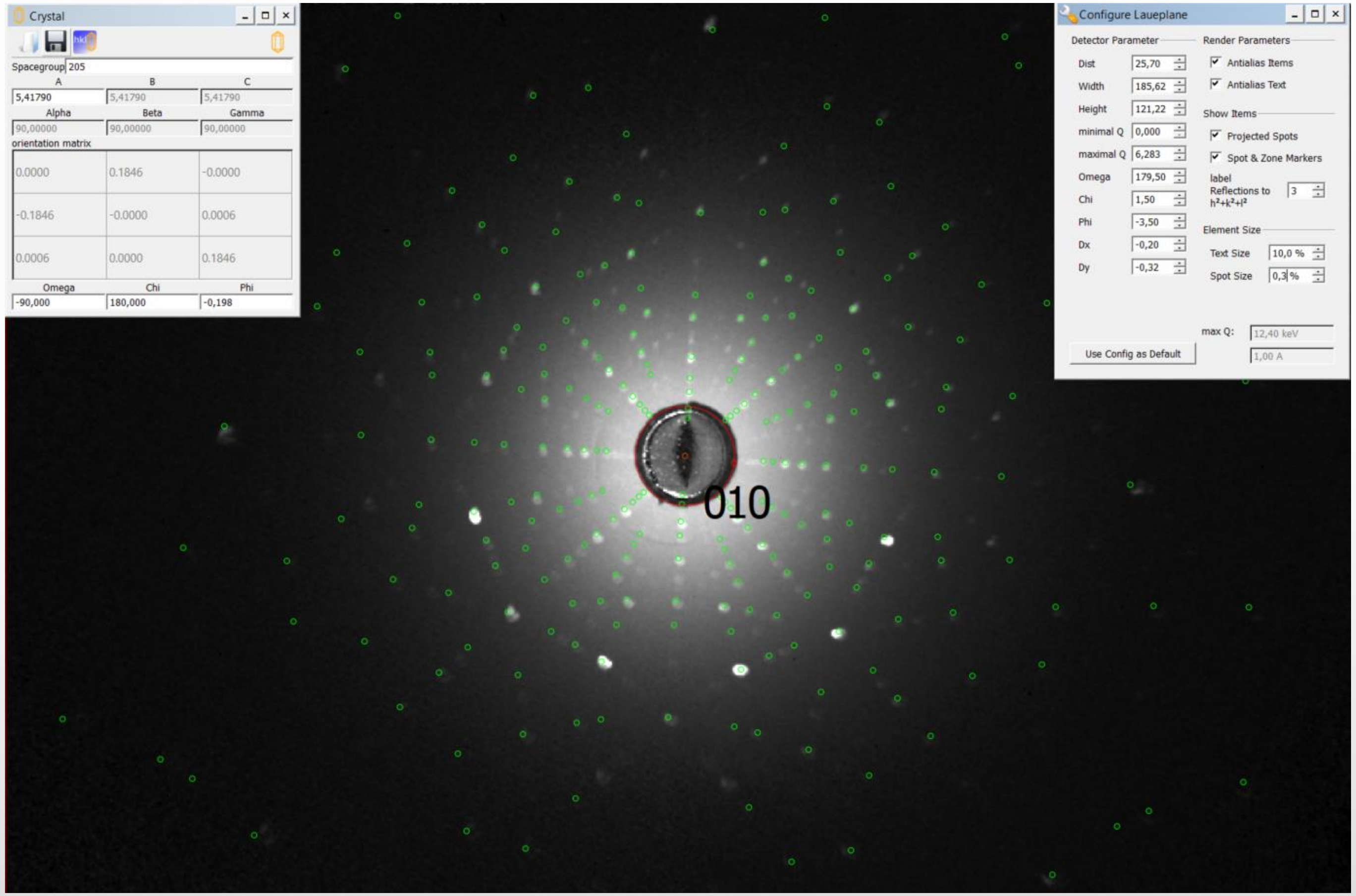
Crystal 1

Pyrite

9.2 Crystal of your choice

Pyrite 1

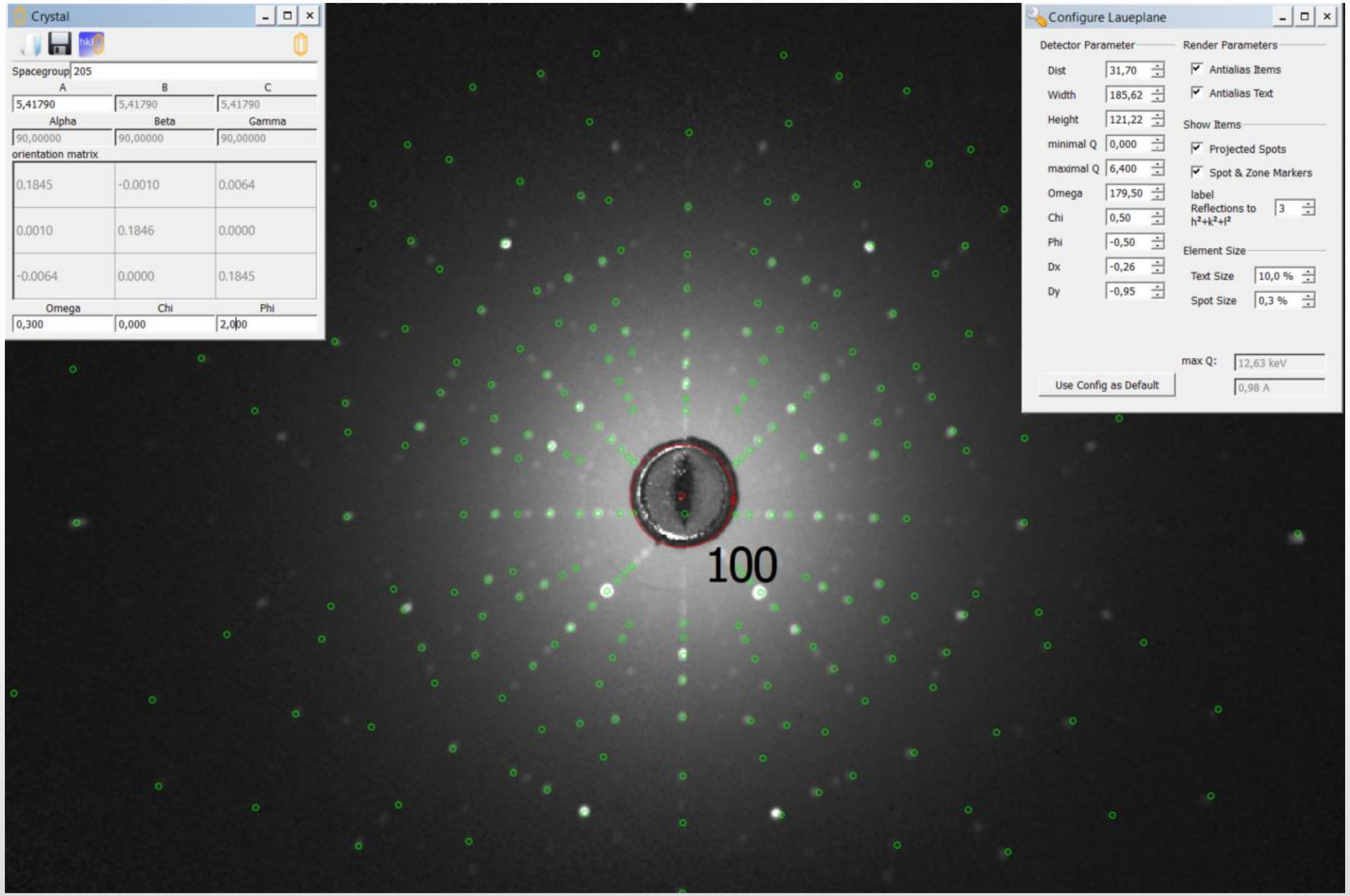
- Determined direction: 010
- Rotational symmetries: 4
- Mirror symmetries: 2
- Defects: not visible (a defect would disturb the symmetry)



9.2 Crystal of your choice

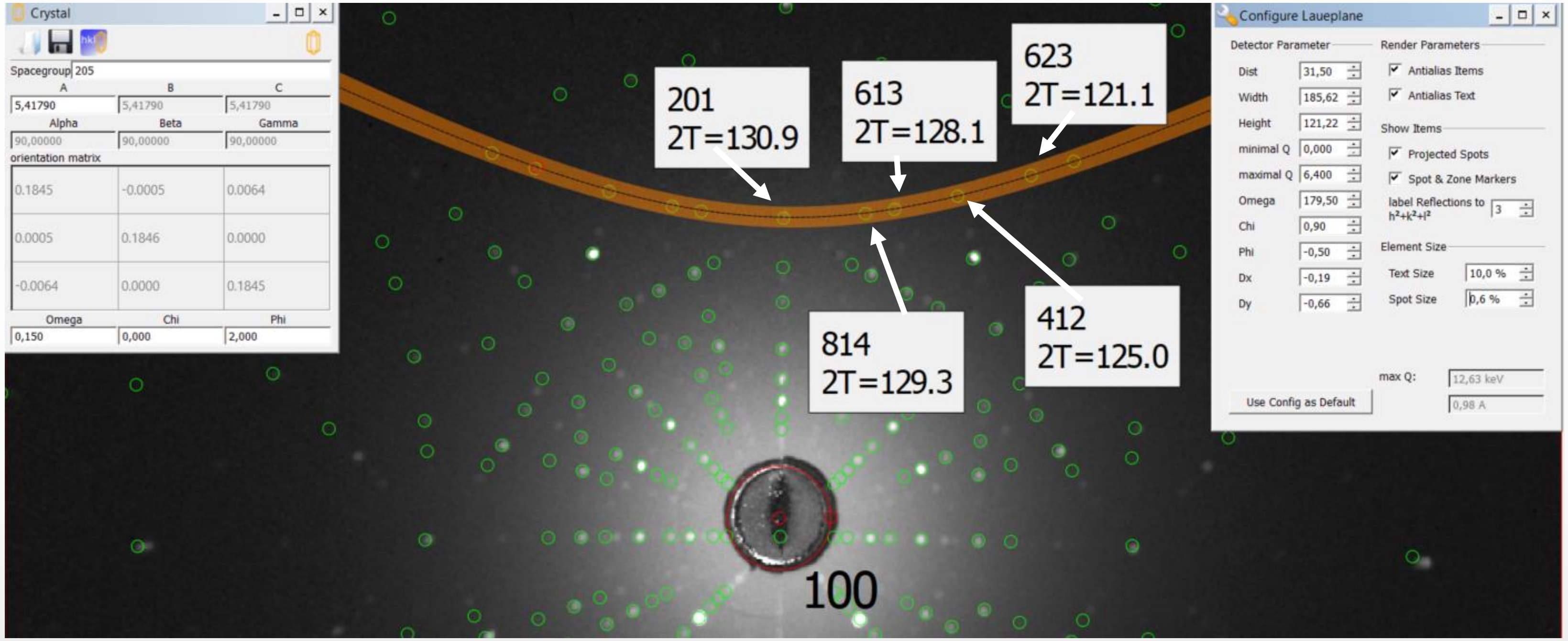
Pyrite 2

- Determined direction: 100
- Rotational symmetries: 4 (but skewed)
- Mirror symmetries: 2 (but skewed)



9.2 Crystal of your choice

Pyrite 2 – zone axis for a particular set of five planes



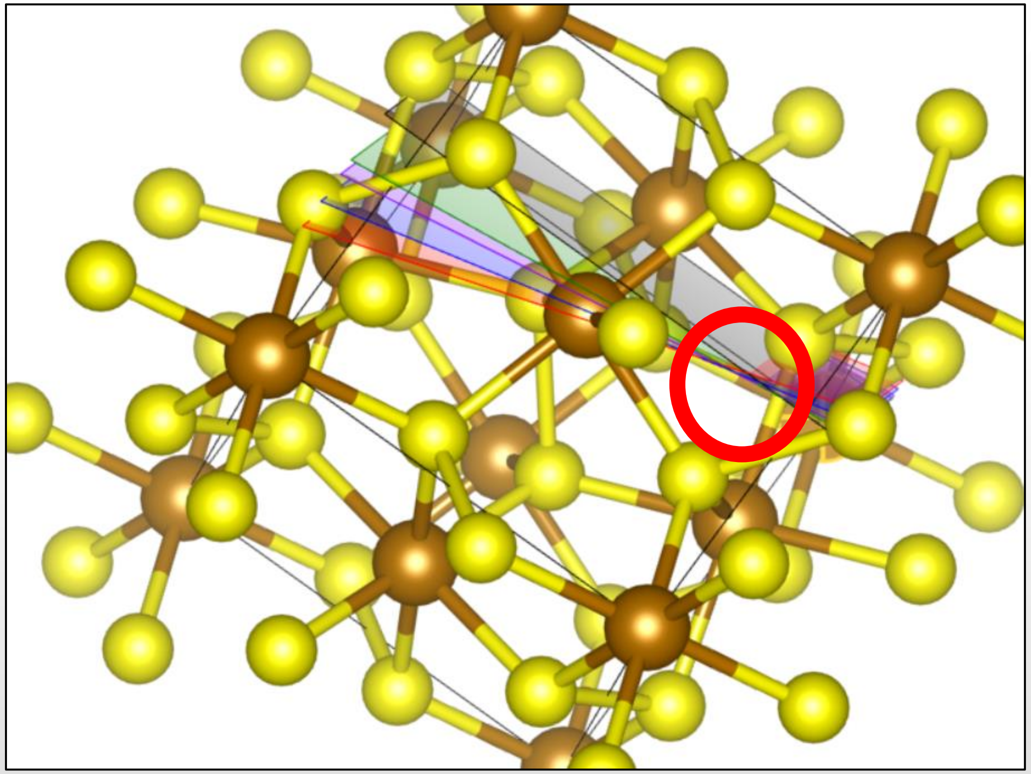
- Five planes $(201), (814), (613), (412), (623)$
- The zone axis z can be calculated via

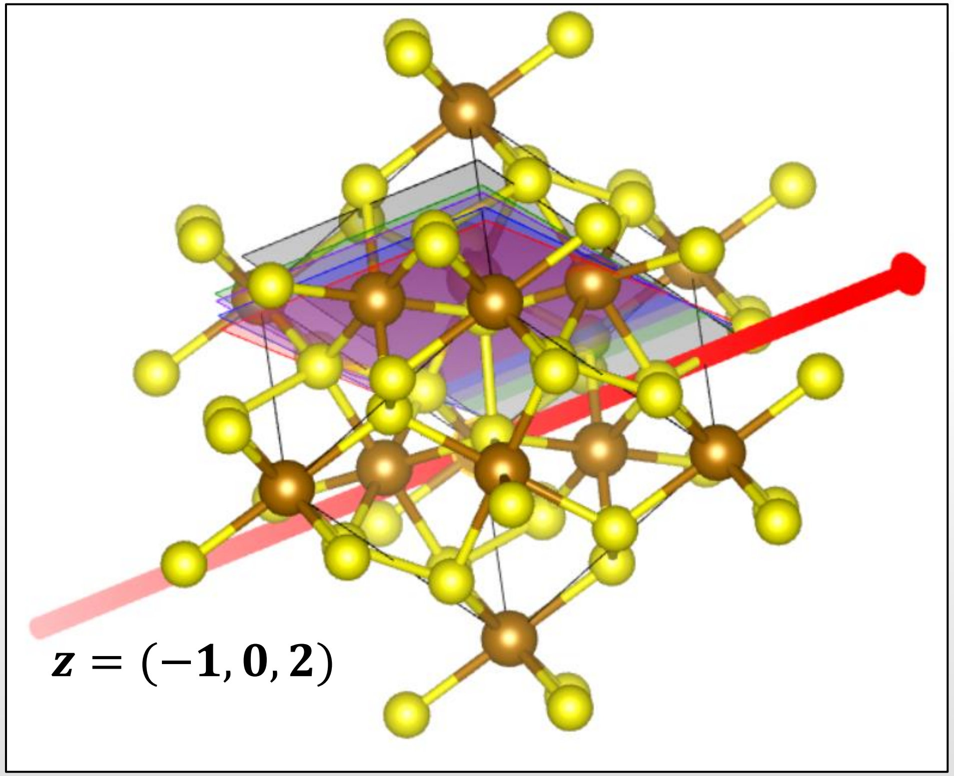
$$p_i \cdot z = 0 \;\; for\ all\ i = 1, \dots, 5$$

- The result is: $\boldsymbol{z} = (\boldsymbol{-1}, \boldsymbol{0}, \boldsymbol{2})$

9.2 Crystal of your choice

Pyrite 2 – zone axis for a particular set of five planes – visualization with VESTA

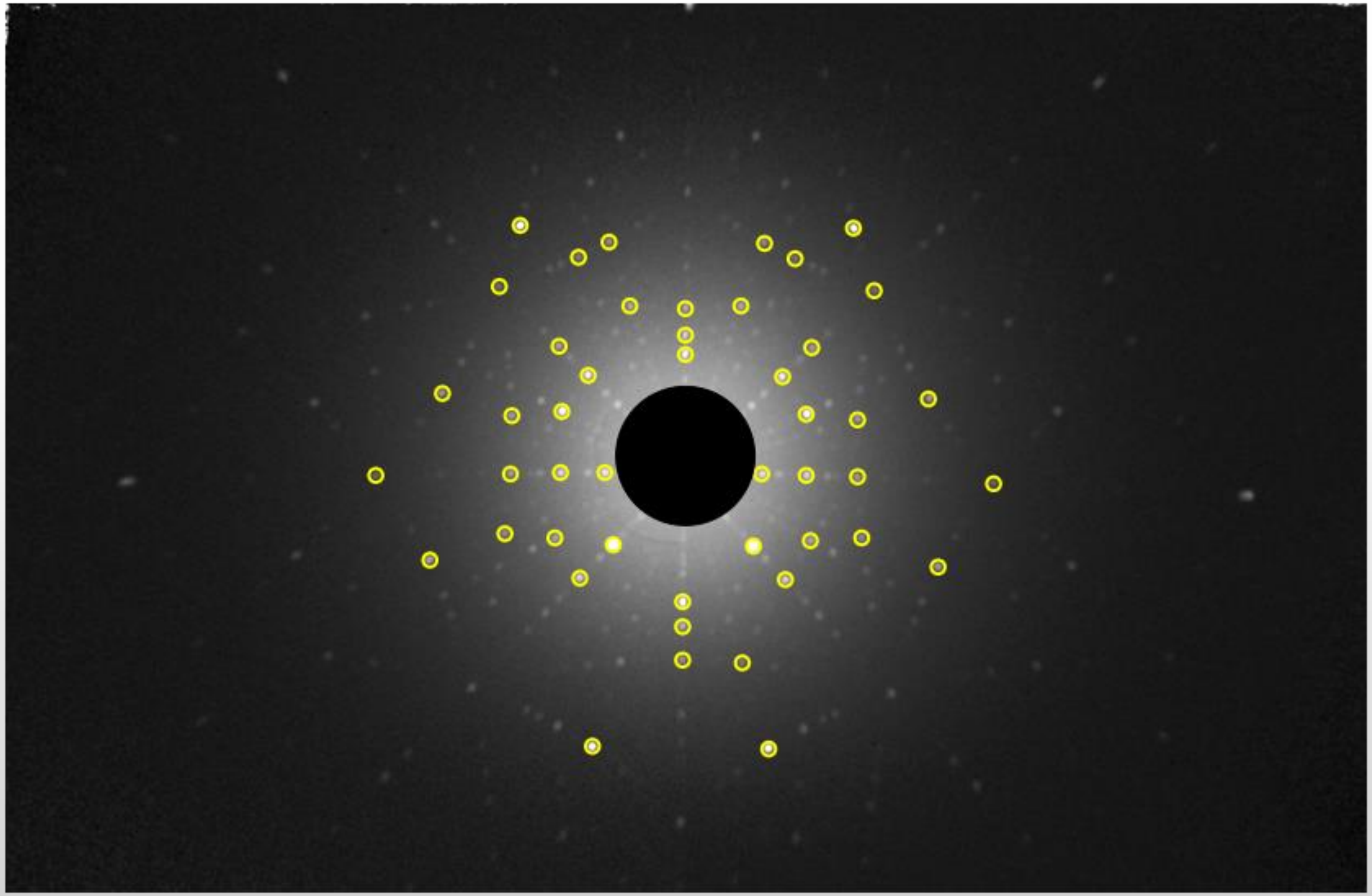




9.2 Crystal of your choice

Pyrite 2 – zone axis for a particular set of five planes – fit spots

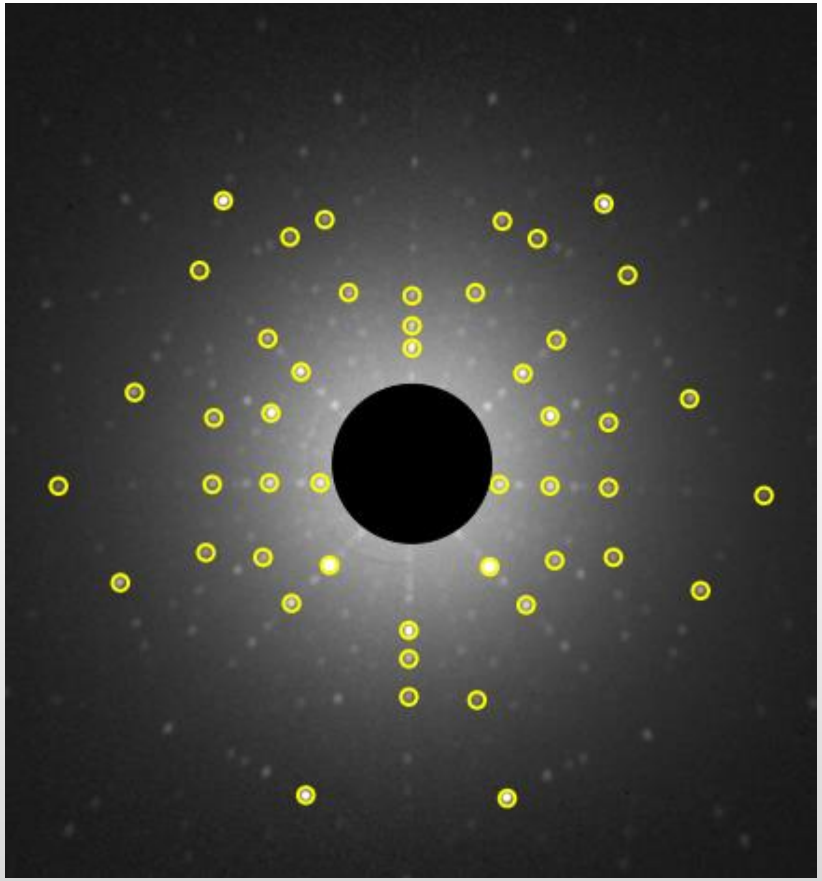
- We use a small python script to fit the spots (“less spots but no errors”)

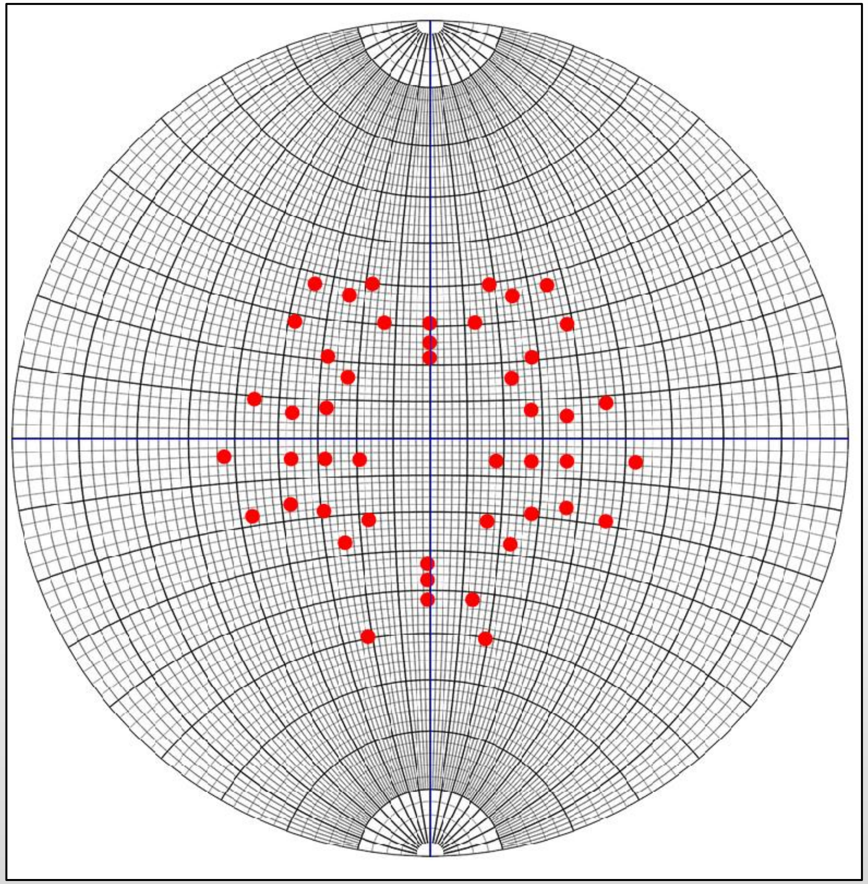


9.2 Crystal of your choice

Pyrite 2 – zone axis for a particular set of five planes – stereographic projection

- We use a small python script to fit the spots (“less spots but no errors”)

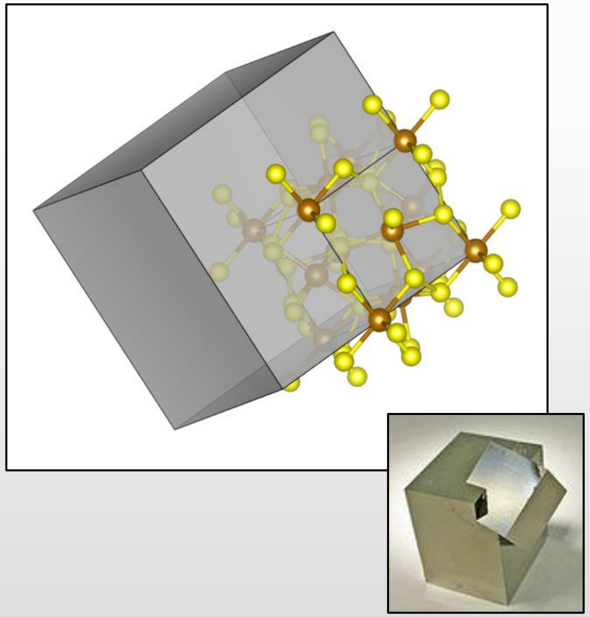


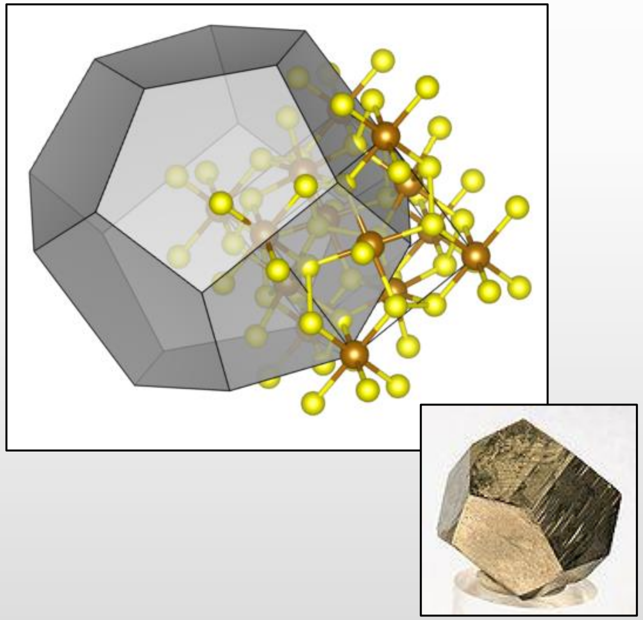


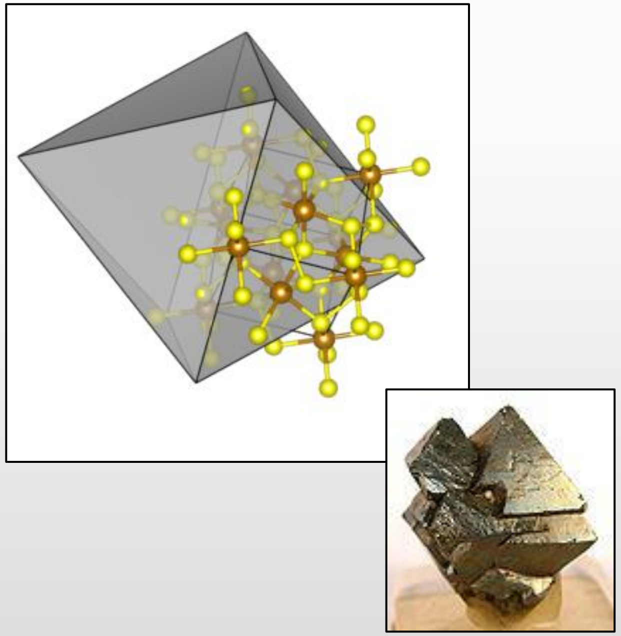
9.2 Crystal of your choice

Pyrite – crystal shape

- Crystal-system: cubic
- Energetically favourable: Growing into directions of small (hkl) → (100), (010), (001)
- Most common: {100}, {210}, {111}



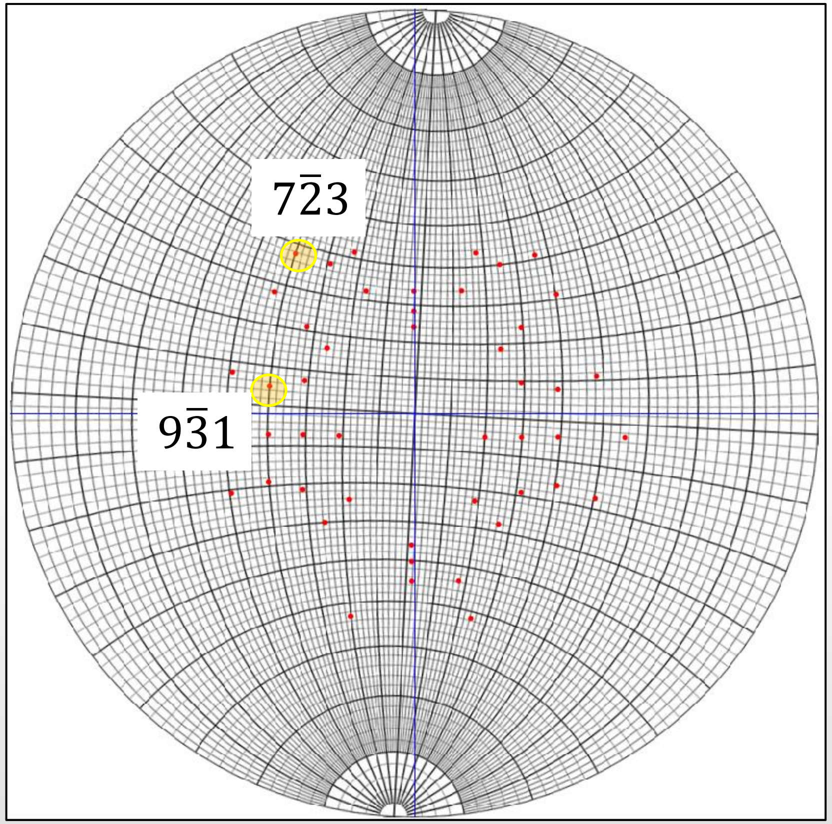




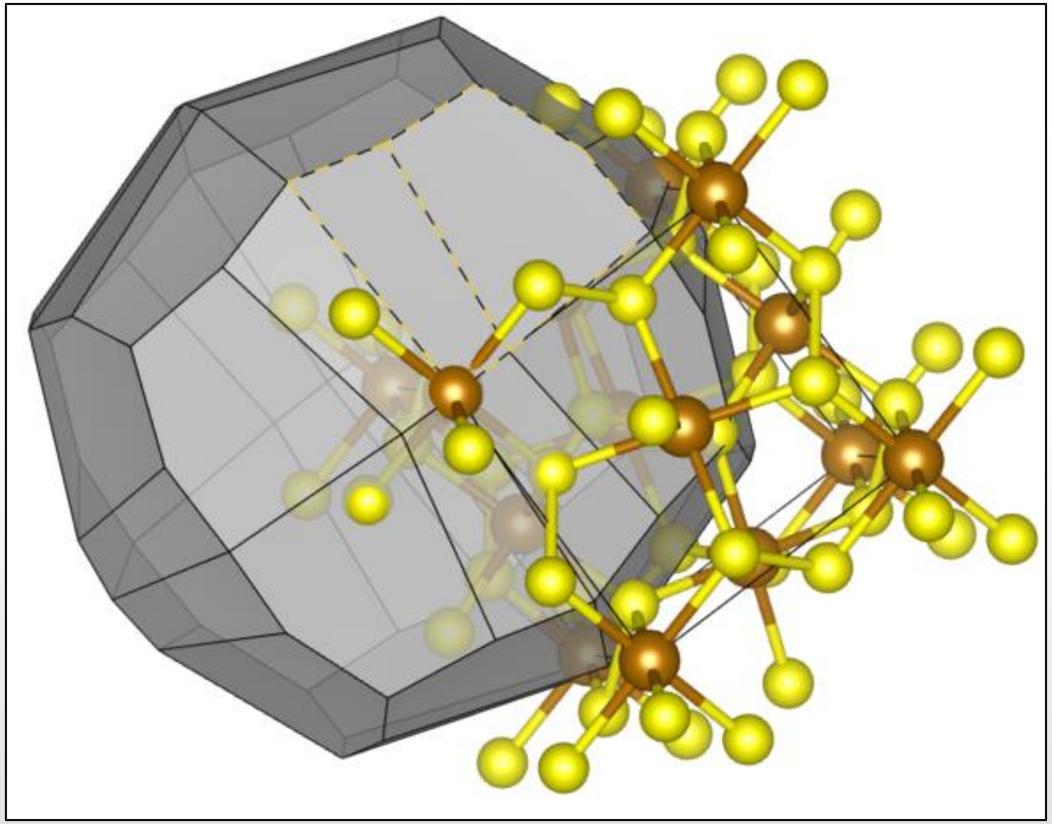
https://de.wikipedia.org/wiki/Pyrit

9.2 Crystal of your choice

Pyrite 2 – angle between planes



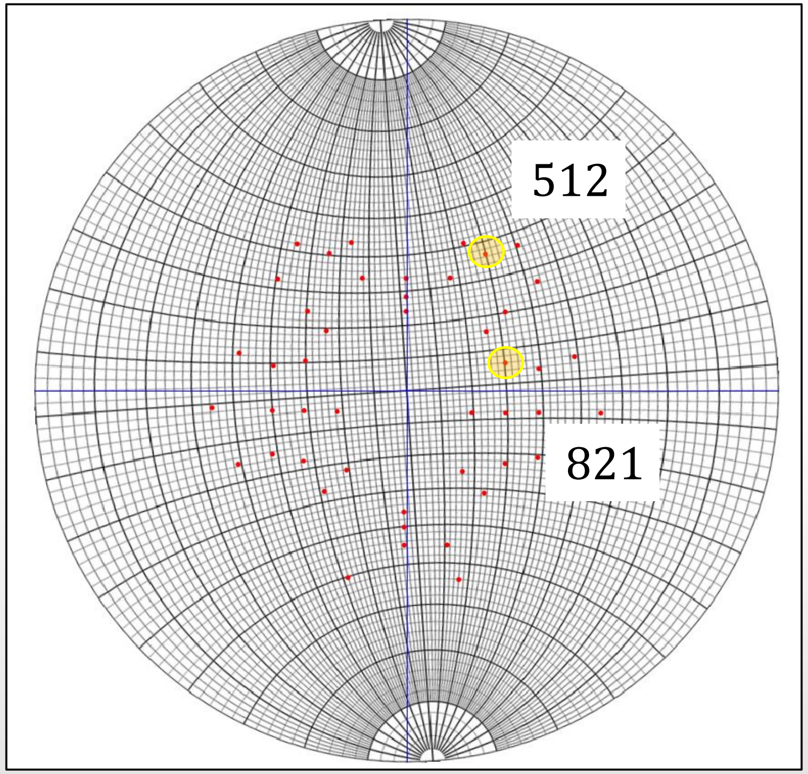
$\rightarrow (16.5 \pm 1.0)°$



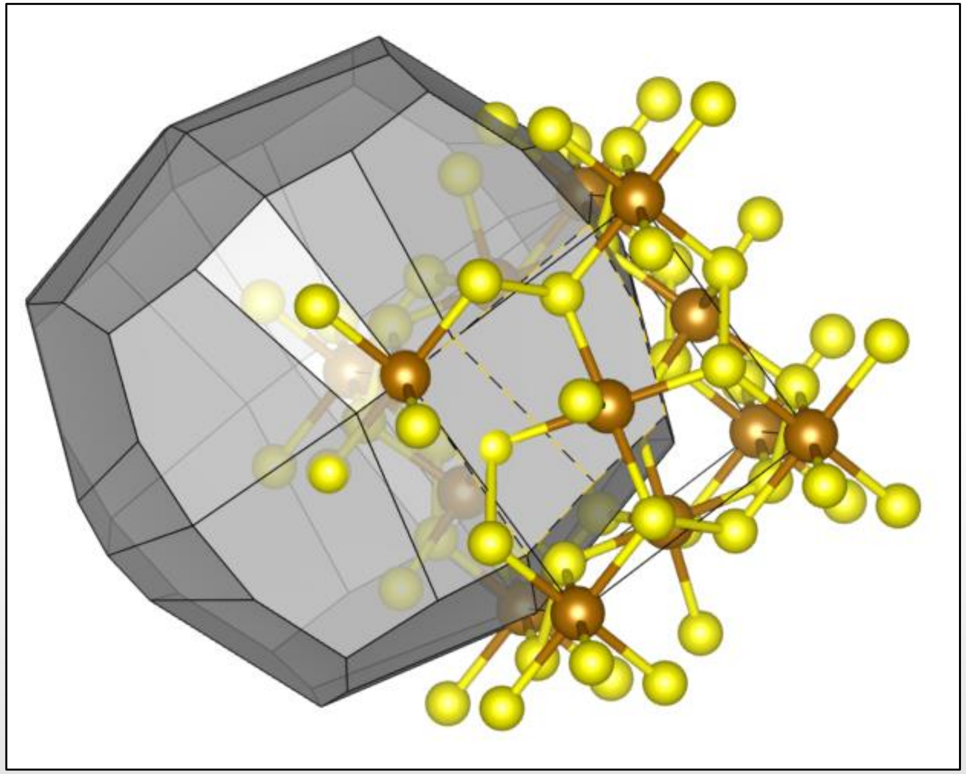
Angle between (9 -3 1) and (7 -2 3) = 16.5538°

9.2 Crystal of your choice

Pyrite 2 – angle between planes



→$(15.0 \pm 1.0)°$



Angle between (5 1 2) and (8 2 1) = 14.7398°

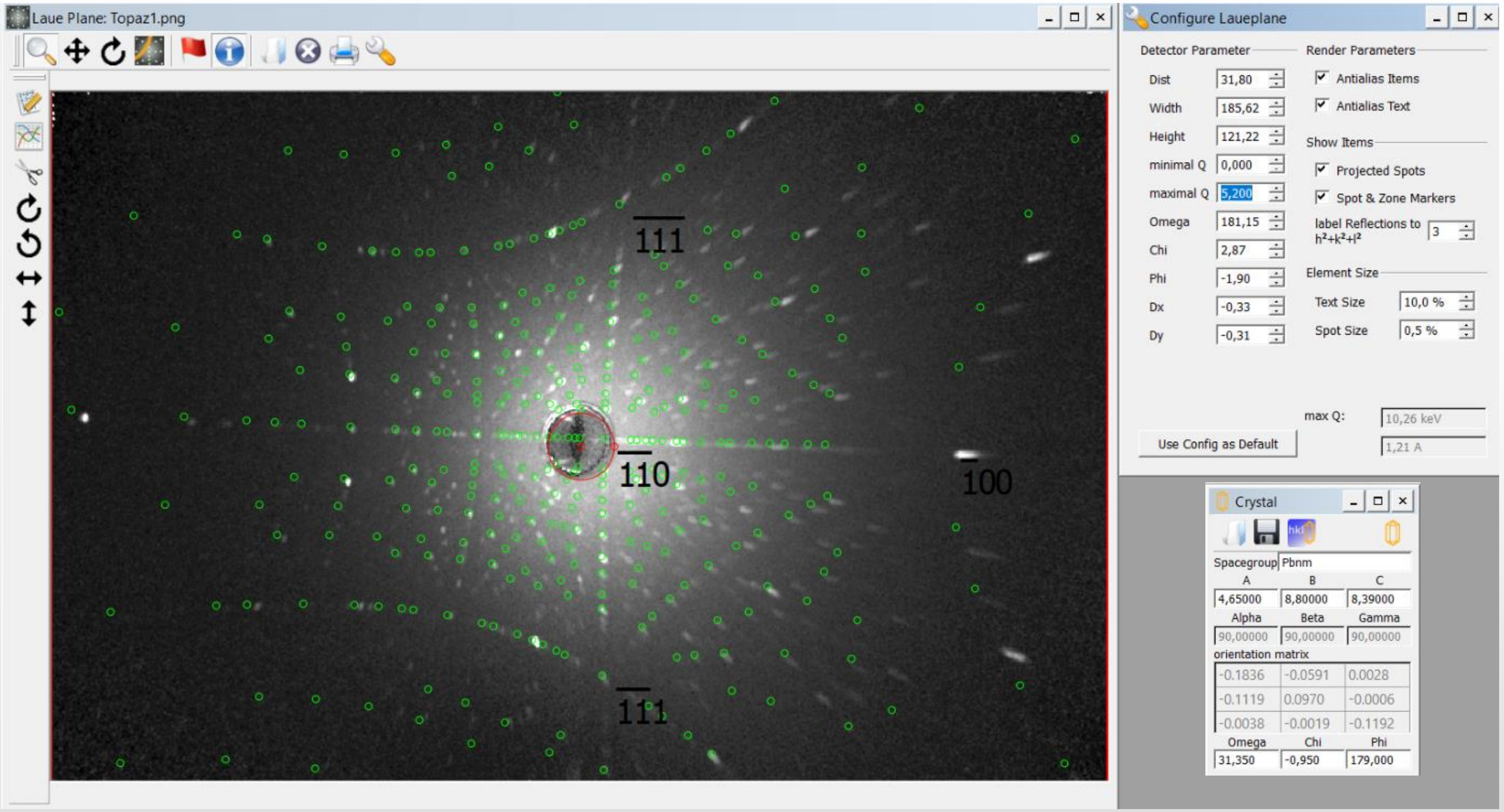
Crystal 2

Topaz

9.2 Crystal of your choice

Topaz 1

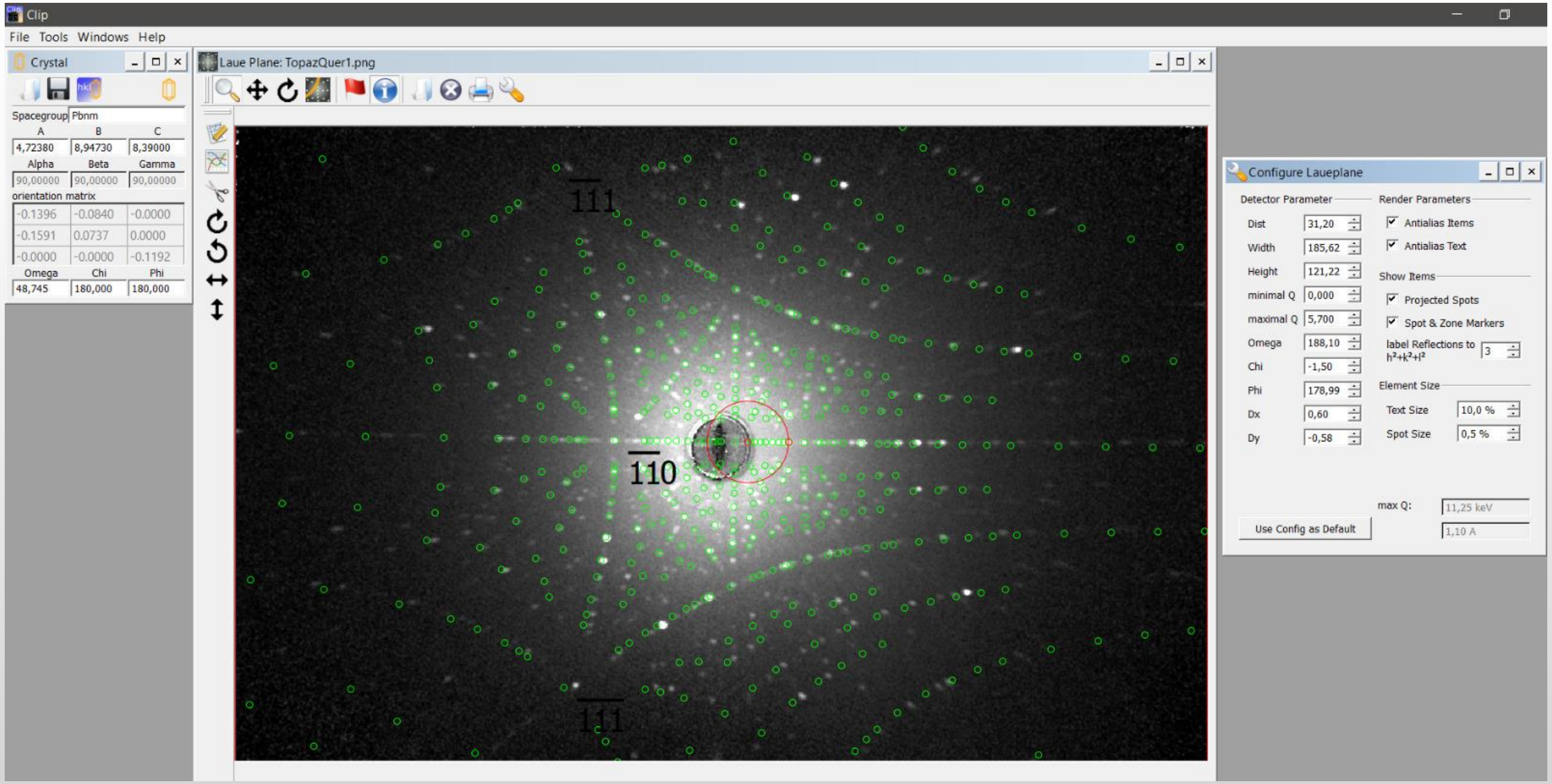
- Determined direction: $\bar{1}\bar{1}0$
- Rotational symmetries: <…>
- Mirror symmetries: <…>
- Defects: <…>



9.2 Crystal of your choice

Topaz cross

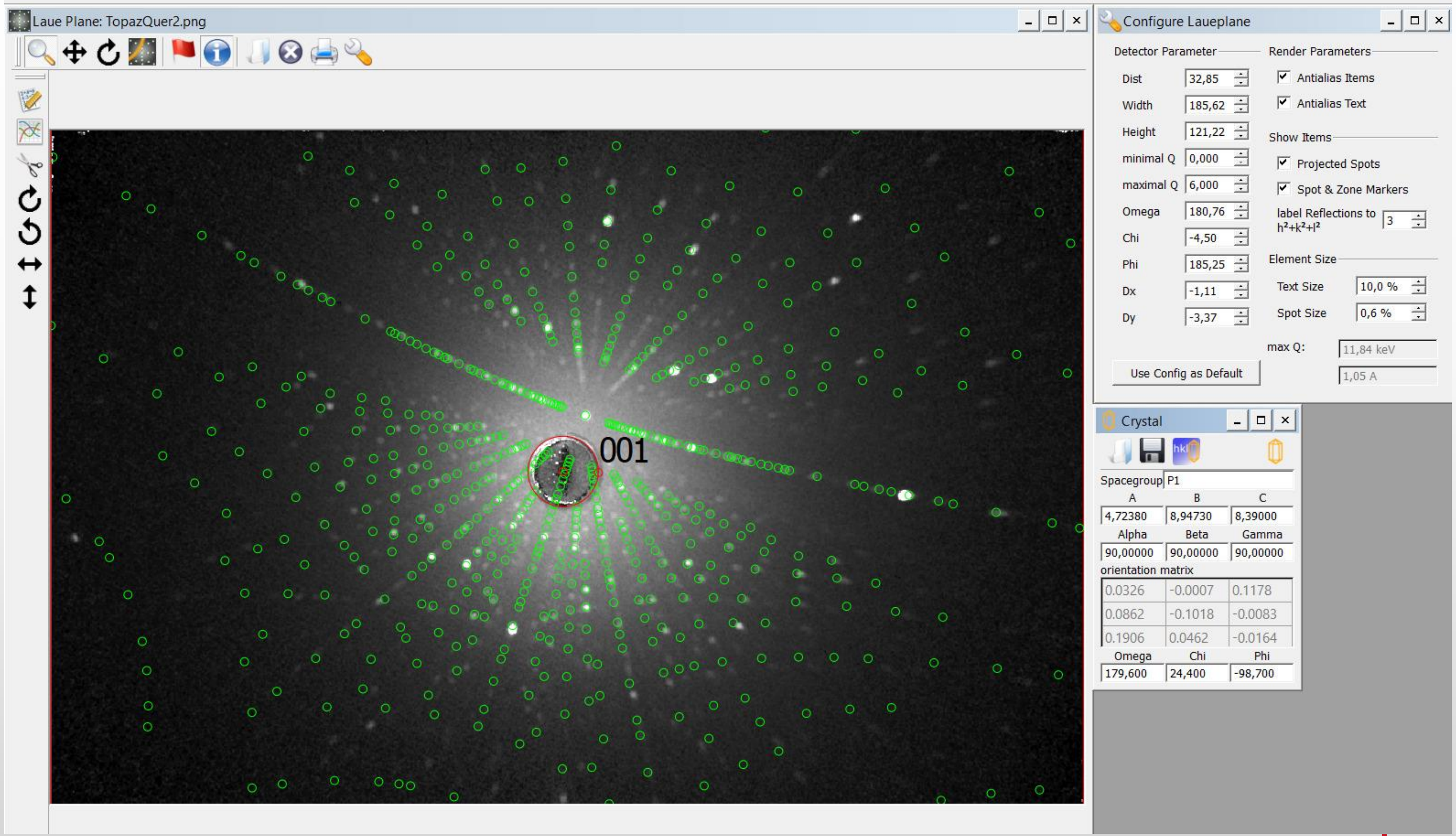
- Determined direction: $\bar{1}\bar{1}0$
- Rotational symmetries: <…>
- Mirror symmetries: <…>



9.2 Crystal of your choice

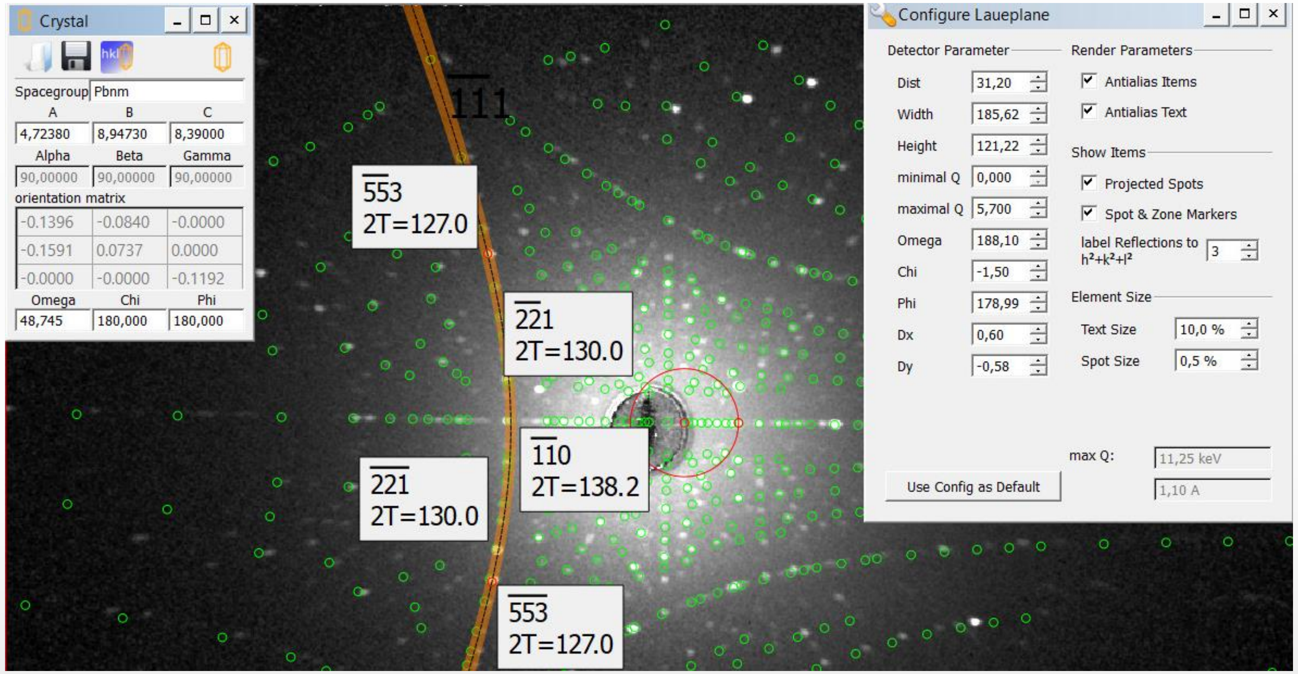
Topaz cross 2

- Determined direction: 100
- Rotational symmetries: <…>
- Mirror symmetries: <…>



9.2 Crystal of your choice

Topaz cross – zone axis for a particular set of five planes



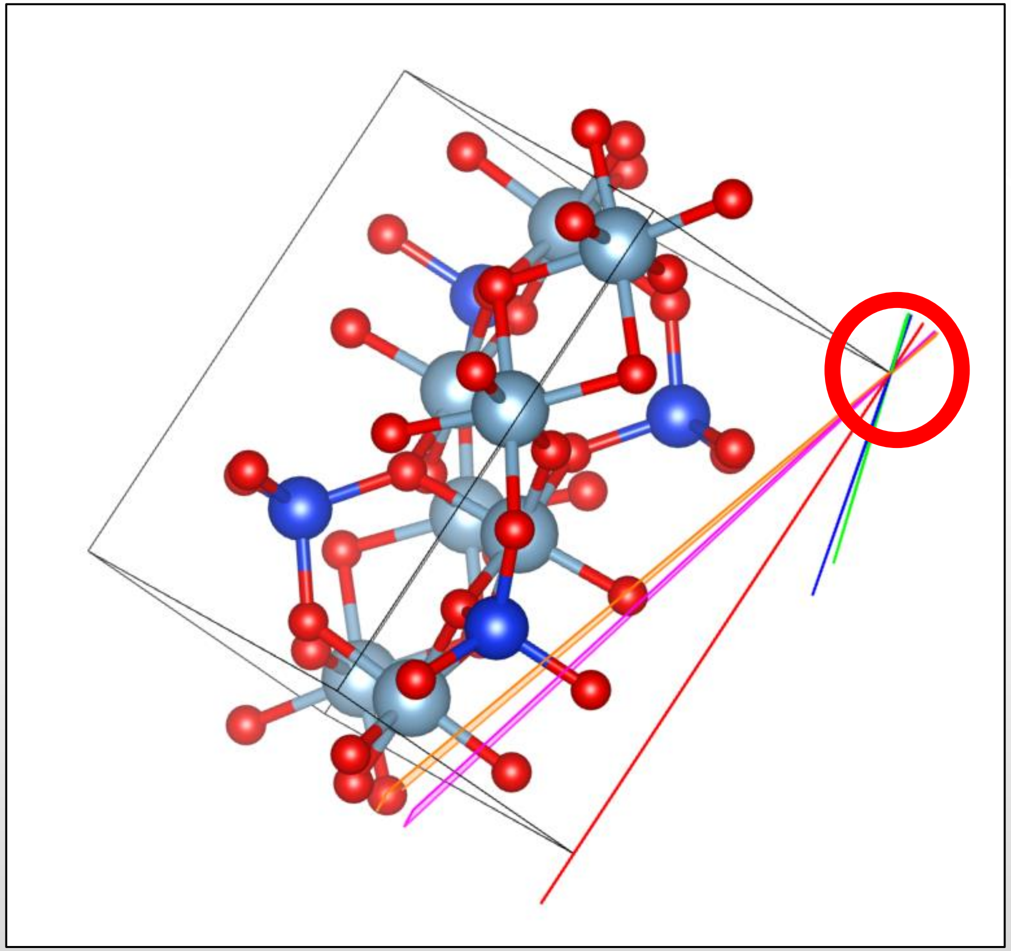
- Five planes $(\bar{5}\bar{5}3), (\bar{2}\bar{2}1), (\bar{1}\bar{1}0\ ), (\bar{2}\bar{2}\bar{1}\ ), (\bar{5}\bar{5}\bar{3})$
- The zone axis z can be calculated via

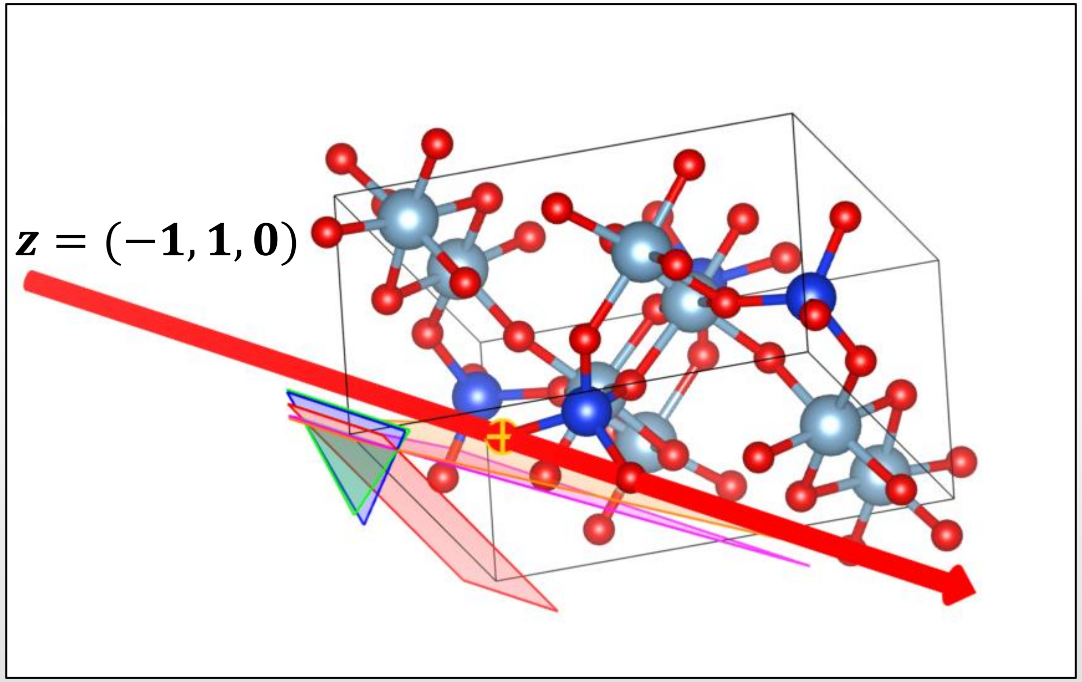
$$p_i \cdot z = 0 \;\; for\; all\; i = 1, \dots, 5$$

- The result is: $\boldsymbol{z} = (\mathbf{-1, 1, 0})$

9.2 Crystal of your choice

Topaz cross – zone axis for a particular set of five planes – visualization with VESTA

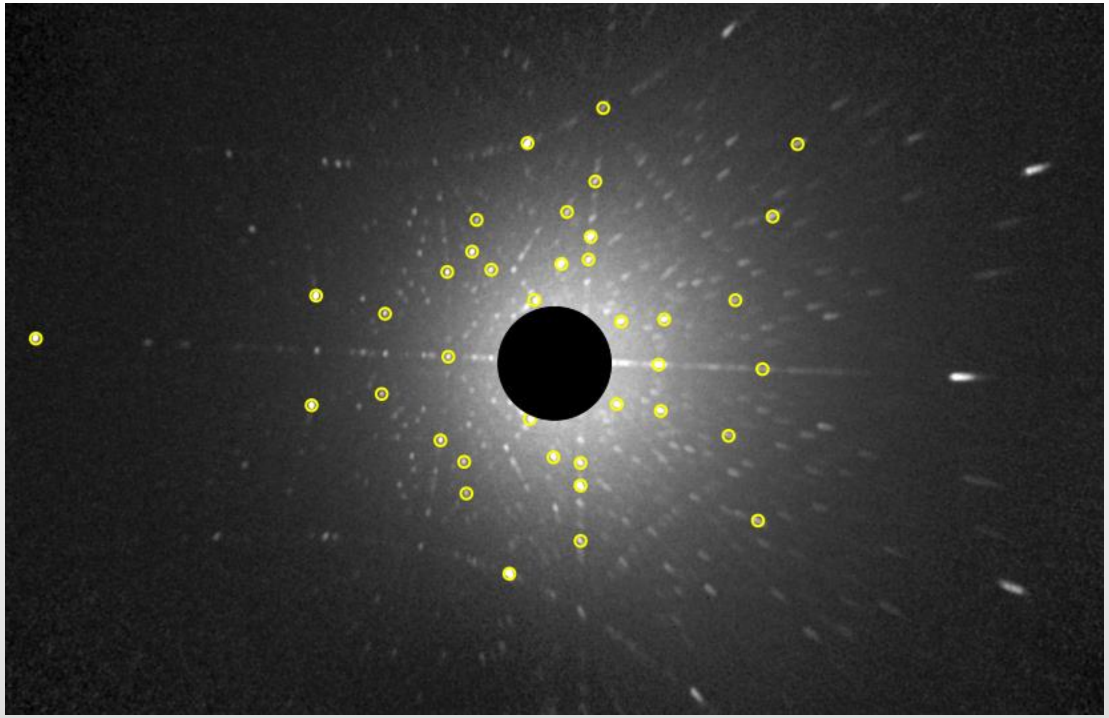


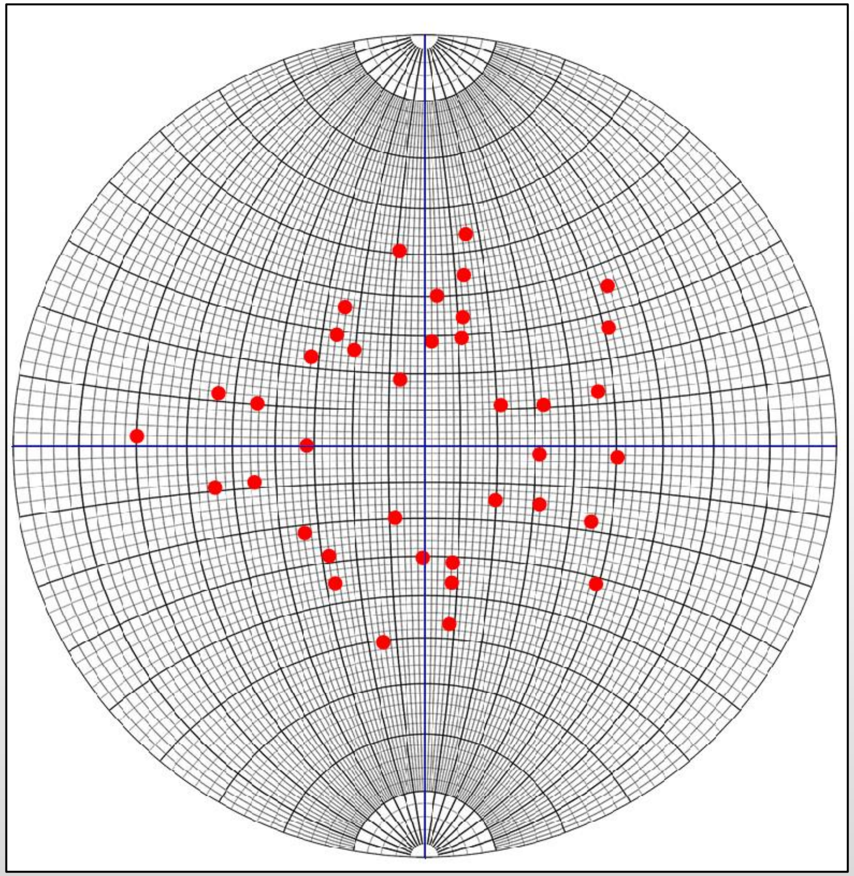


9.2 Crystal of your choice

Topaz – zone axis for a particular set of five planes – fit spots

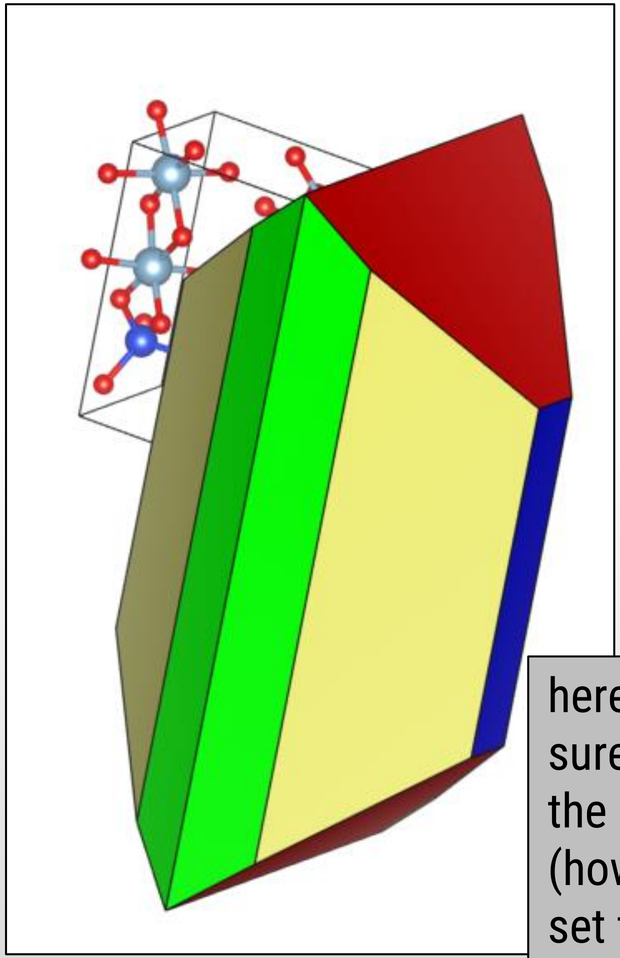
- We use a small python script to fit the spots ("less spots but no errors")

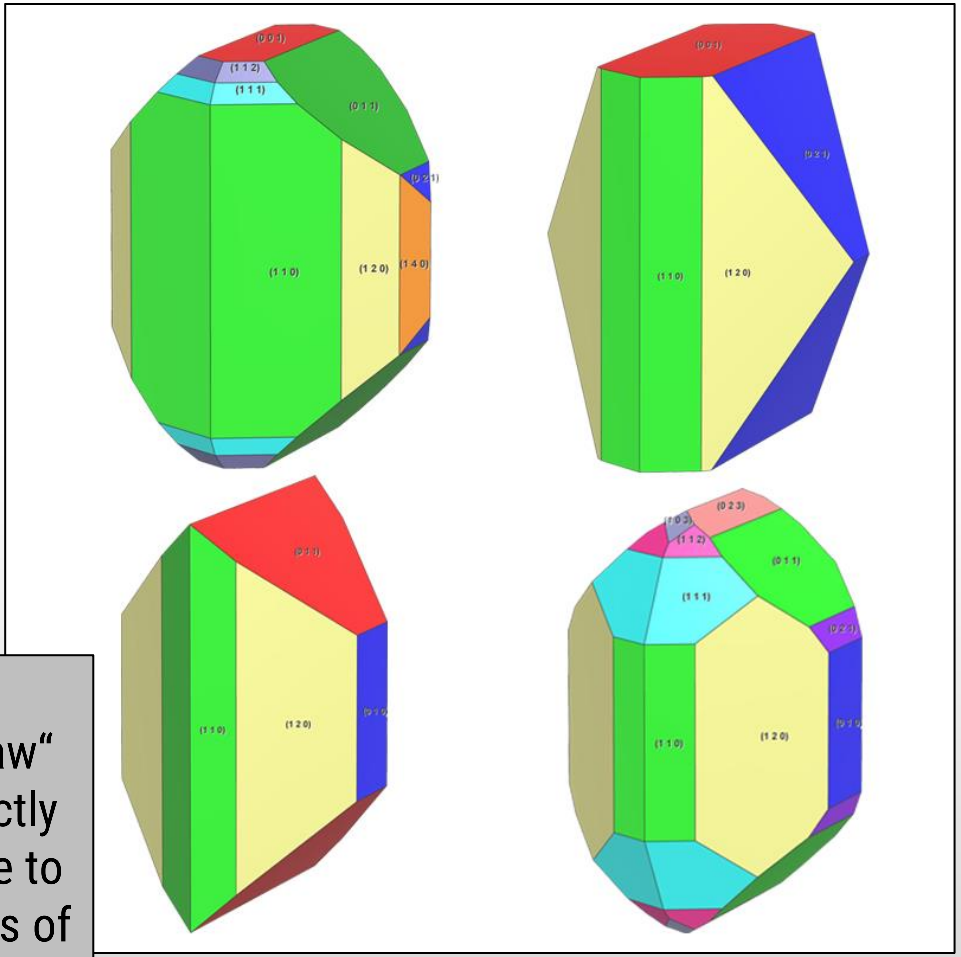




9.2 Crystal of your choice

Topaz – crystal shape



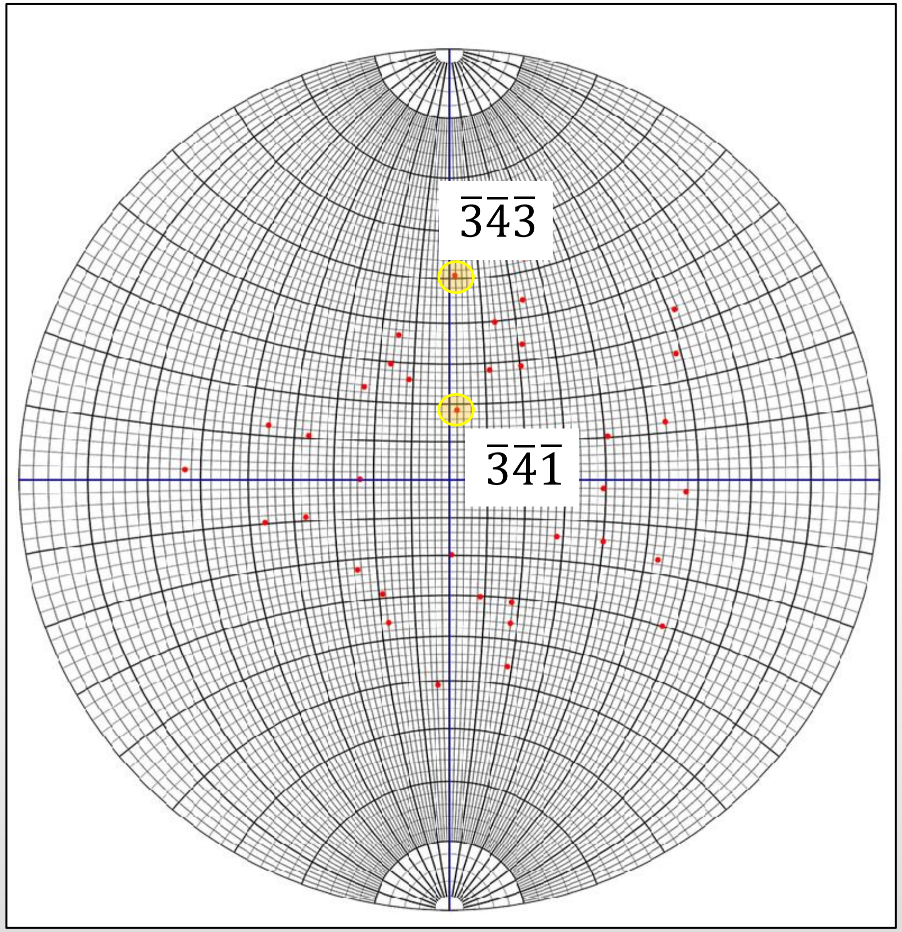


here we are not sure how to „draw“ the shape correctly (how do we have to set the distances of the planes?)

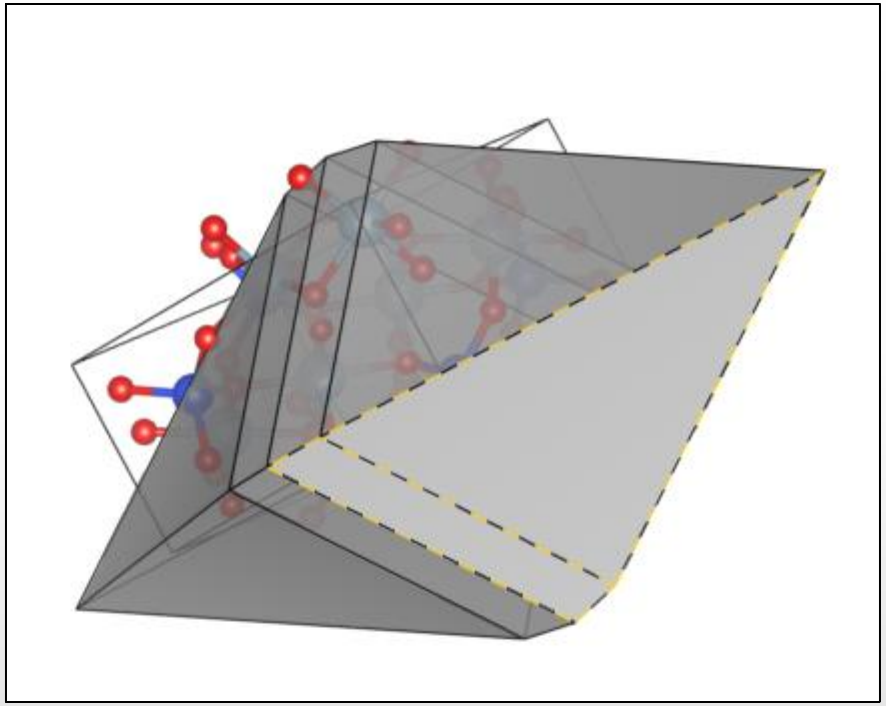
http://www.meelis-bluetopaz.com/about-topaz.html

9.2 Crystal of your choice

Topaz – angle between planes



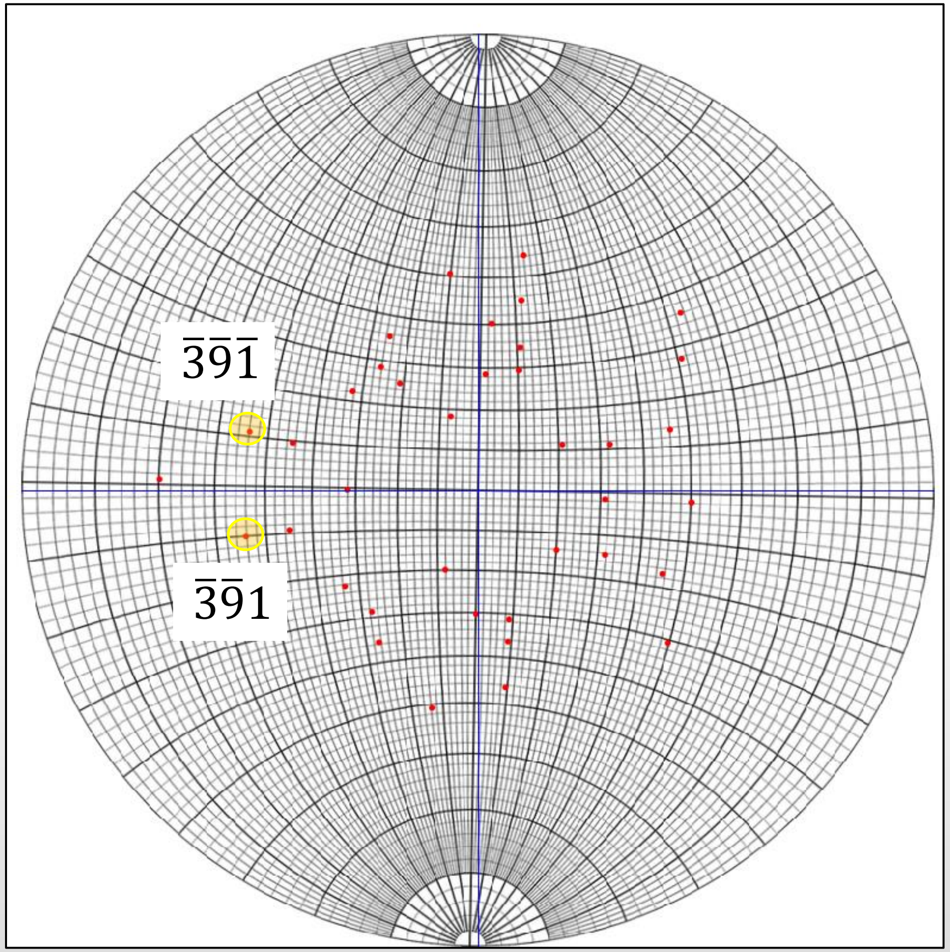
$\rightarrow (16.0 \pm 1.0)°$



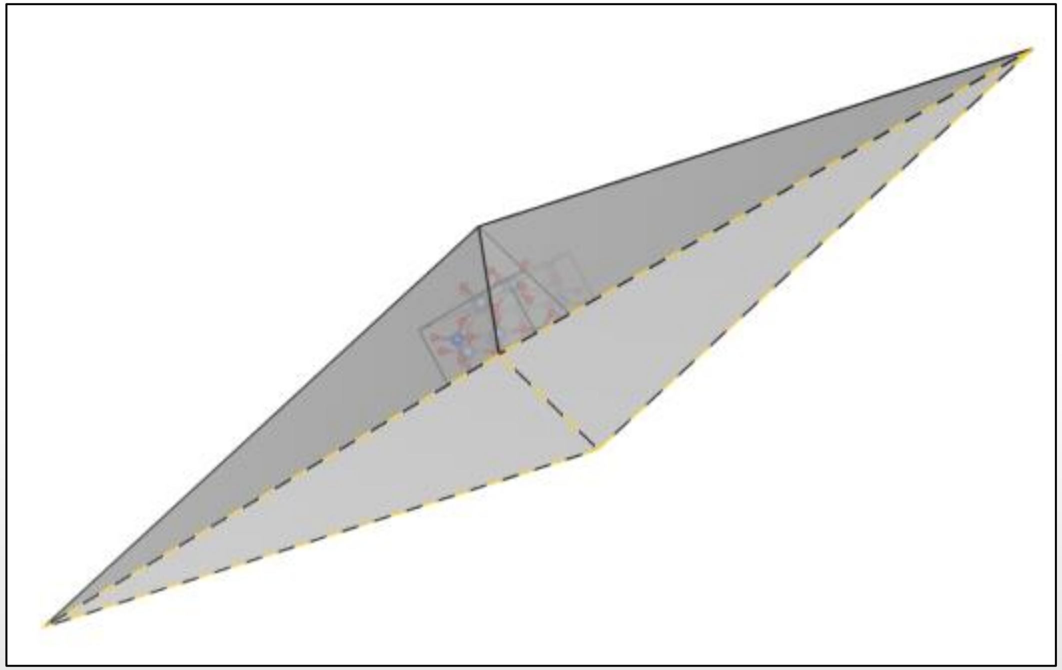
Angle between (-3 -4 -1) and (-3 -4 -3) = 15.9963°

9.2 Crystal of your choice

Topaz – angle between planes



$\rightarrow (10.5 \pm 1.0)^\circ$



Angle between (-3 -9 1) and (-3 -9 -1) = 11.4431°

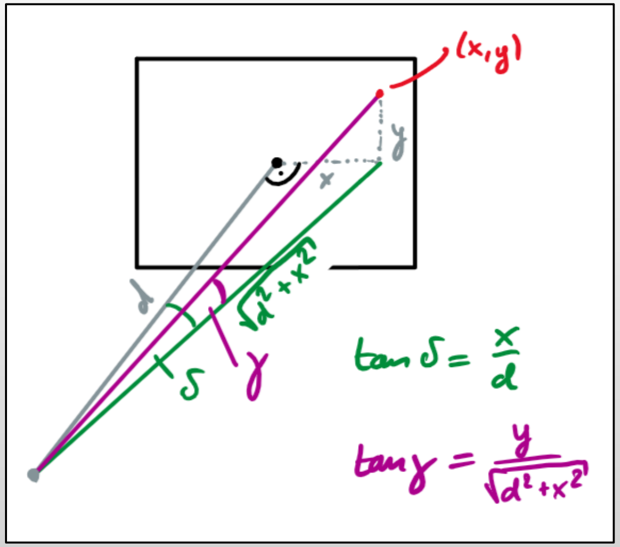
9.3 General tasks

Stereographic projection

- Projection: $(x, y) \rightarrow (\gamma, \delta)$
- Assume the coordinate centre to be $(x_0, y_0) = (0{,}0)$

$$\delta = \begin{cases} 0, & x = 0 \\ \arctan \frac{x}{d}, & x \geq 0 \end{cases}$$

$$\gamma = arctan \frac{y}{\sqrt{d^2 + x^2}}$$



- Mathematica:

```
delta[X1_, Y1_] := If[X1 - X0 == 0, 0, ArcTan[X2[X1, Y1] / d] * 180 / Pi]
```

```
gamma[X1_, Y1_] := ArcTan[Y2[X1, Y1] / Sqrt[d^2 + (X2[X1, Y1])^2]] * 180 / Pi
```

  - X0, Y0: coordinate center
  - X2, Y2: functions that calculate the X and Y value with respect to the coordinate center and the respective quadrants

9.3 General tasks

Maximum hkl

- The energy of the electrons is
$$E_{e^-} = eU_a$$
- Assumption: The emission of the X-ray photons happens without energy loss
➔ maximal energy of the photons
$$\frac{2\pi\hbar c}{\lambda_{min}} = E_X = E_{e^-} = eU_a$$
- With the well-known formula $k = \frac{2\pi}{\lambda}$ this results in
$$k_{max} = \frac{eU_a}{\hbar c}$$
- The formula for the lattice distance is
$$d_{hkl} = \frac{2\pi}{G}$$
with $G = \left|\vec{k} - \overrightarrow{k_0}\right|$ the
- Looking at the Ewald sphere one sees that
$$\left|\vec{k} - \overrightarrow{k_0}\right|_{max} \leq 2k_0 = 2k_{max}$$
- With the previous formulas this results in
$$d_{hkl} = \frac{2\pi}{G} \geq \frac{2\pi}{2k_{max}} = \frac{\pi\hbar c}{eU_a}$$
- For a simple cubic crystal
$$d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$
- This gives us the limiting condition
$$\boxed{\sqrt{\boldsymbol{h^2 + k^2 + l^2}} \leq \frac{\boldsymbol{eU_a a}}{\boldsymbol{\pi\hbar c}}}$$

9.3 General tasks

Maximum hkl - Pyrite

- Pyrite (sc) has the lattice constant $a = 5.4179\text{Å}$
- Assuming that the images are taken at the optimal acceleration voltage $U_a = 30kV$ we get the limit

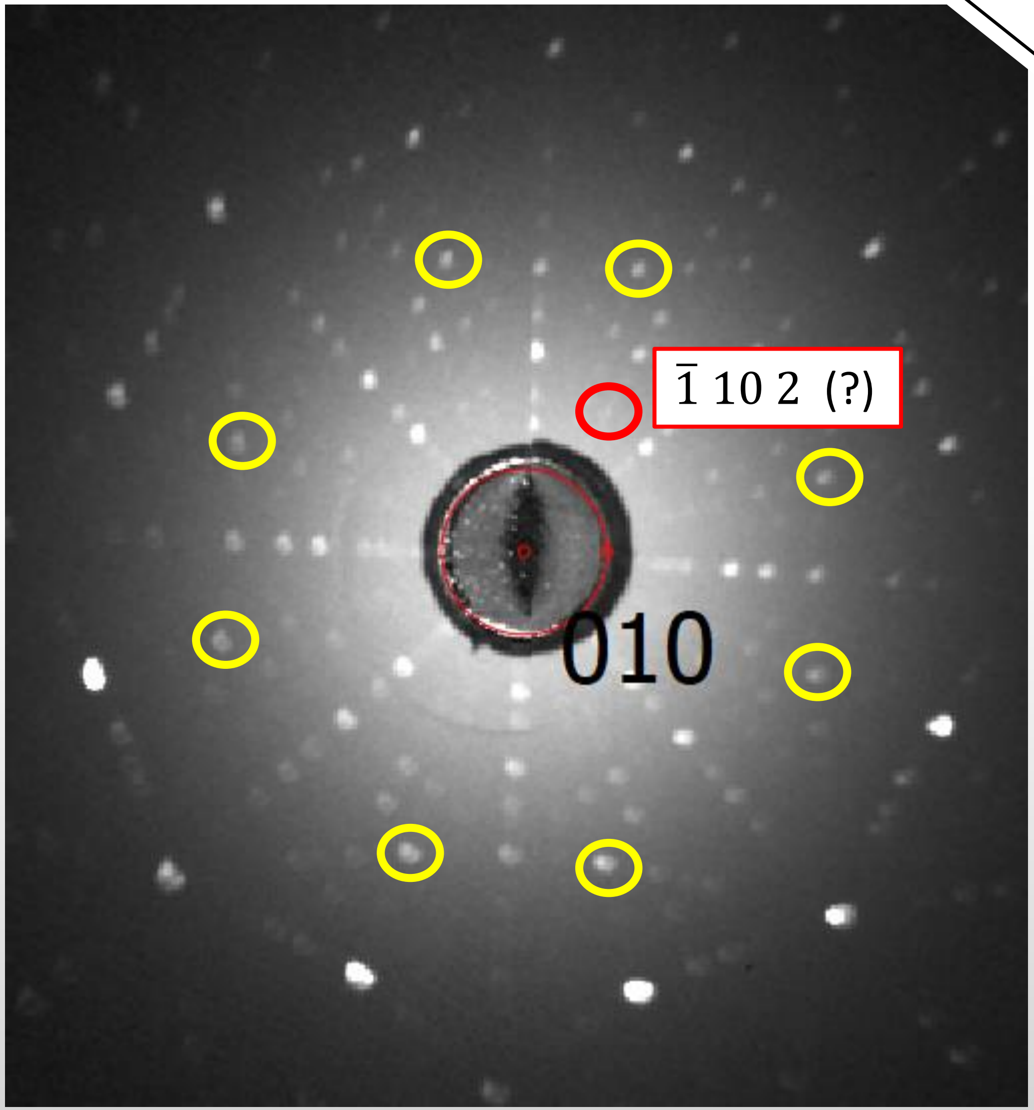
$$\sqrt{h^2 + k^2 + l^2} \leq 26.22$$

- The highest planes we clearly observed on Pyrite are the planes of the combinations 3,9,1

$$\sqrt{3^2 + 9^2 + 1^2} \approx 9.5 < 26.22$$

- Explanation:
  higher planes
  $\leftrightarrow$ smaller plane distances $d_{hkl} \sim \frac{1}{\sqrt{h^2+k^2+l^2}}$
  ➔ one needs smaller λ $\leftrightarrow$ higher energy $E \sim \frac{1}{\lambda}$
  But: These are not the planes that are chosen for the fits (this is not a problem)

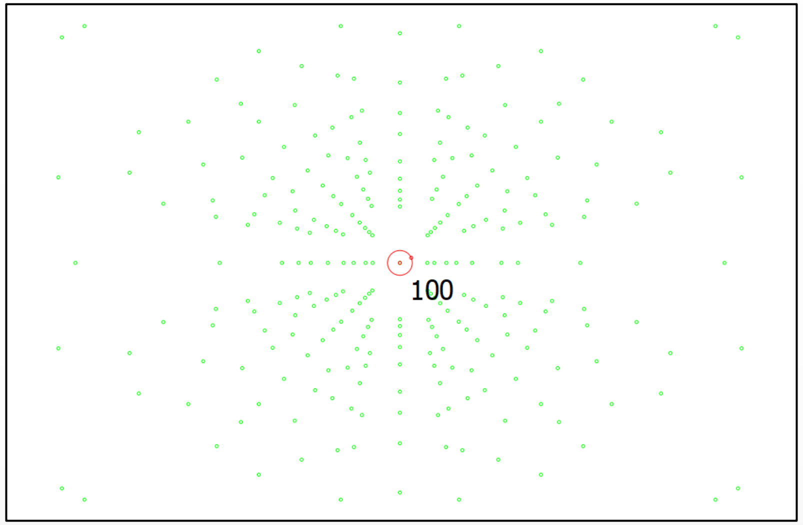
$$\sqrt{h^2 + k^2 + l^2} \leq \frac{eU_a a}{\pi\hbar c}$$

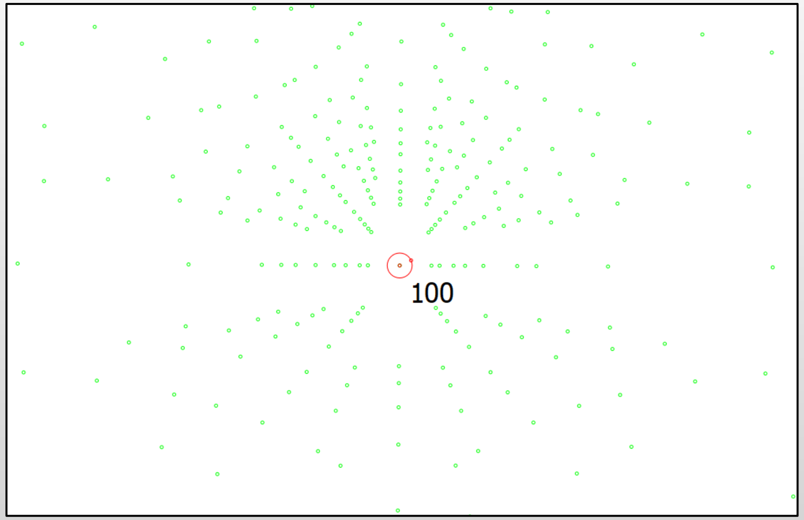


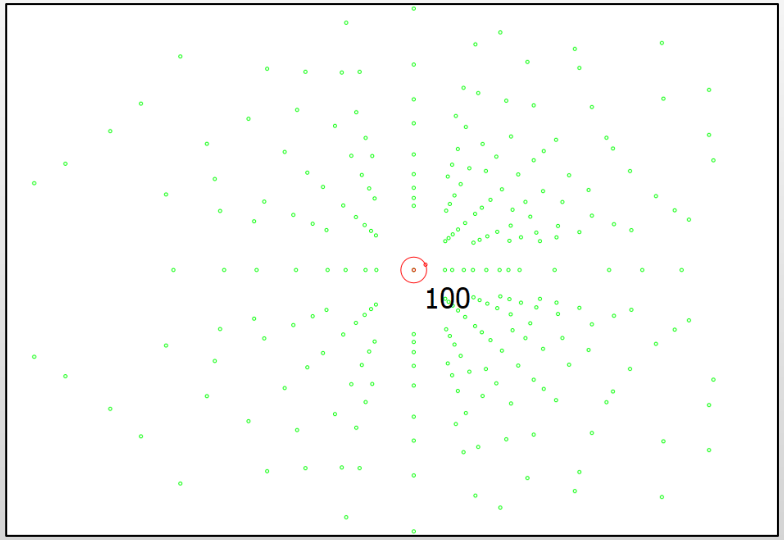
9.3 General tasks

Tilted detector – poorly collimated beam – variable sample-detector distance

- Tilted detector:
  - “Distorted” images
  - Symmetry gets lost







9.3 General tasks

Tilted detector – poorly collimated beam – variable sample-detector distance

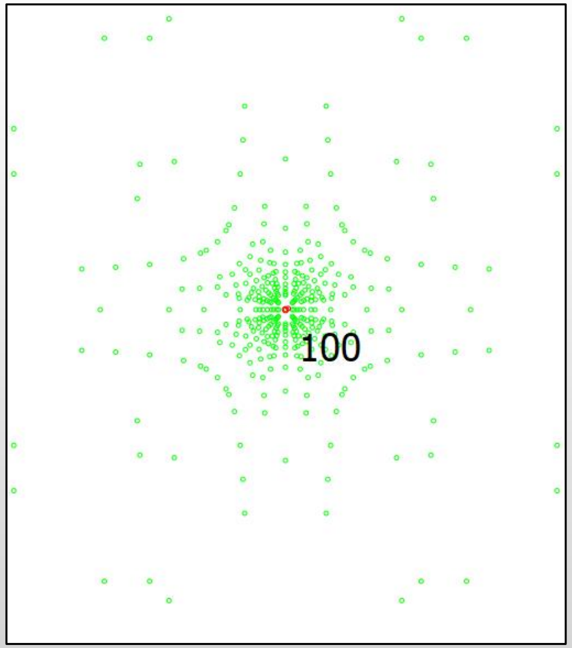
- Poorly collimated beam:

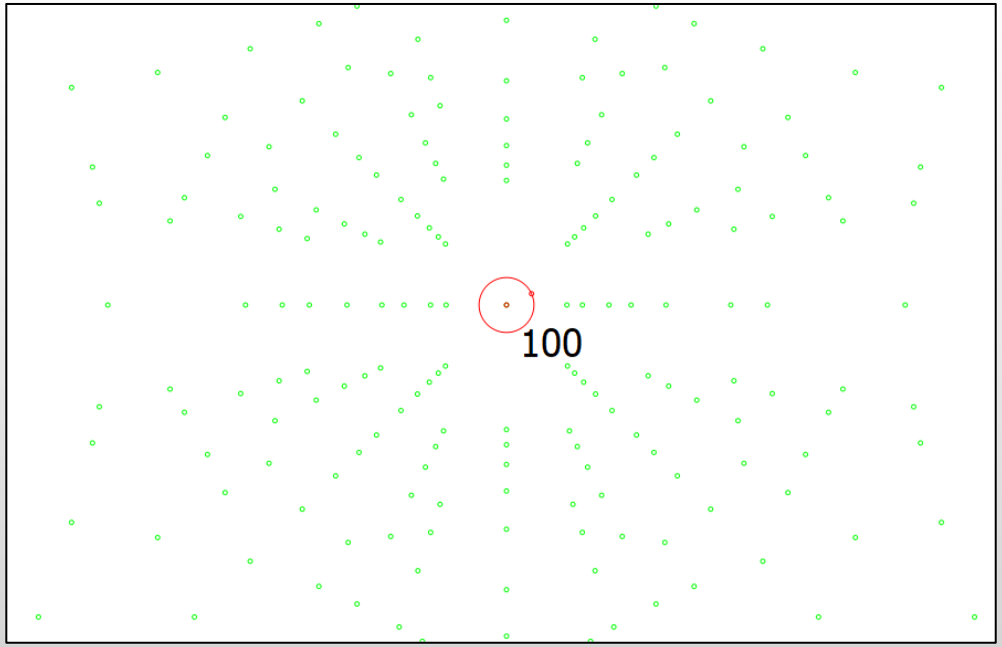
  ➔the angle of incidence is not fixed

  ➔ the spots get washed out

- Variable sample-detector distance:

  ➔ Larger distance ➔ larger spot distance





9.3 General tasks

Experimental errors

- Previously discussed: poorly collimated beam
- Wrong settings in CLIP (can be seen if angle of planes is totally wrong)
- Pixel-accuracy
- Accuracy of the Wulff-net
- Staining on the crystal surface