

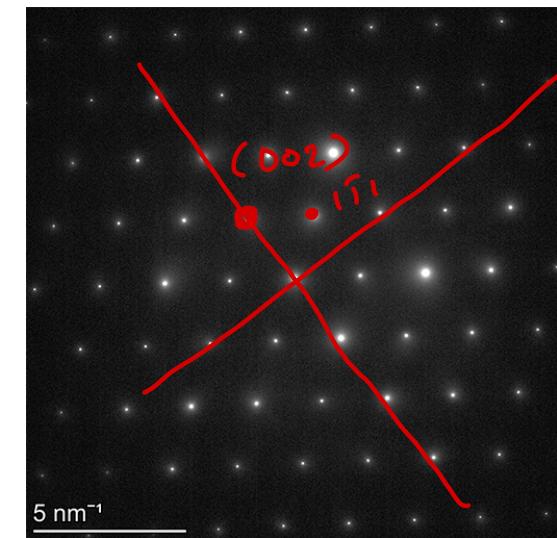
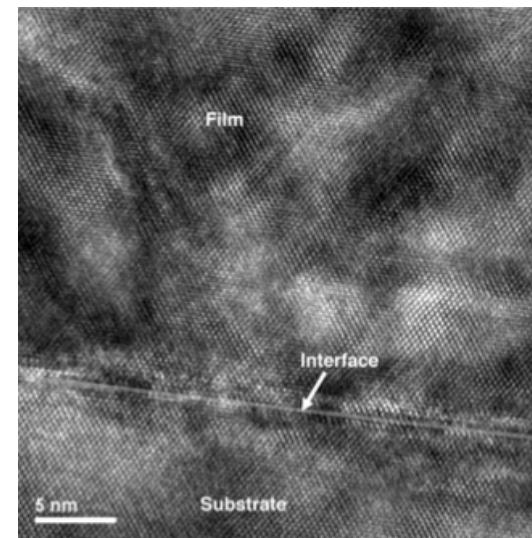
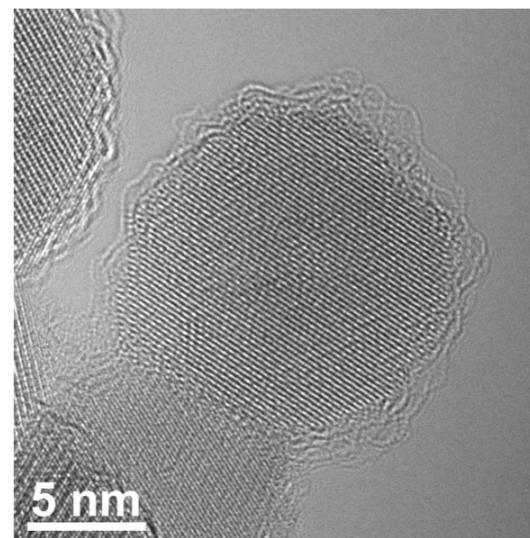
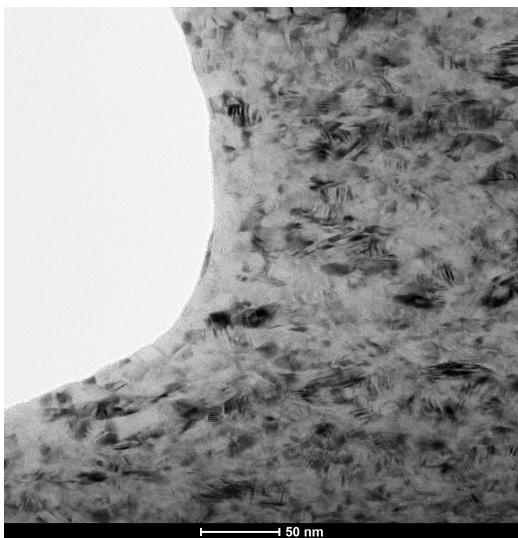
Electron Microscopy: Advanced Methods (Week 0)

Duncan Alexander

EPFL-IPHYS-LSME

EPFL Course objectives

Give you a deep understanding of the most important modes of operation of a modern transmission electron microscope. We will focus on the instrument operation as well as on the essential electron–matter interactions that we exploit, in order to explain the results obtained using these techniques



EPFL Organisation

- Nominally: 2 hours lecture / 1 hour exercises and demos at the microscope
- 8/13 weeks in form of MOOCs. Follow course before the class-room “lecture”
- Class-room time will be used for Q&A, discussions, quizzes, and interactive sessions at the TEM
- Aim: interactive course. **Be participative!**
Use EdDiscussion for questions and discussions
- Prerequisites:
 - Crystallography, solid state physics
 - SEM & TEM lecture at Bachelor level
 - Geometrical optics (convergent lenses)

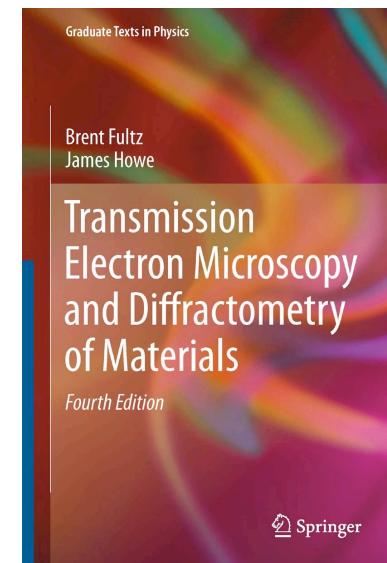
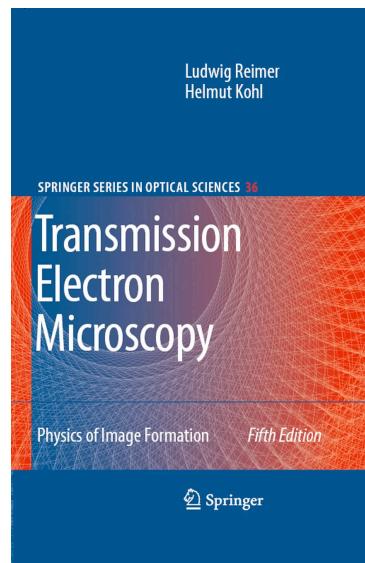
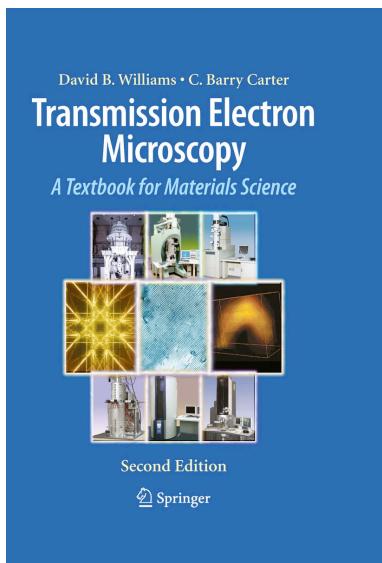
EPFL Lecture material

EPFL Platform

- Open on-line course – ~~Coursera~~ – you will be emailed sign-up link for private session
- Lecture slides
- Books (Williams & Carter, Fultz & Howe, Reimer & Kohl...)

$$|\vec{k}| = \frac{1}{\lambda}$$

$$\left(\frac{2\pi}{\lambda} \right)$$



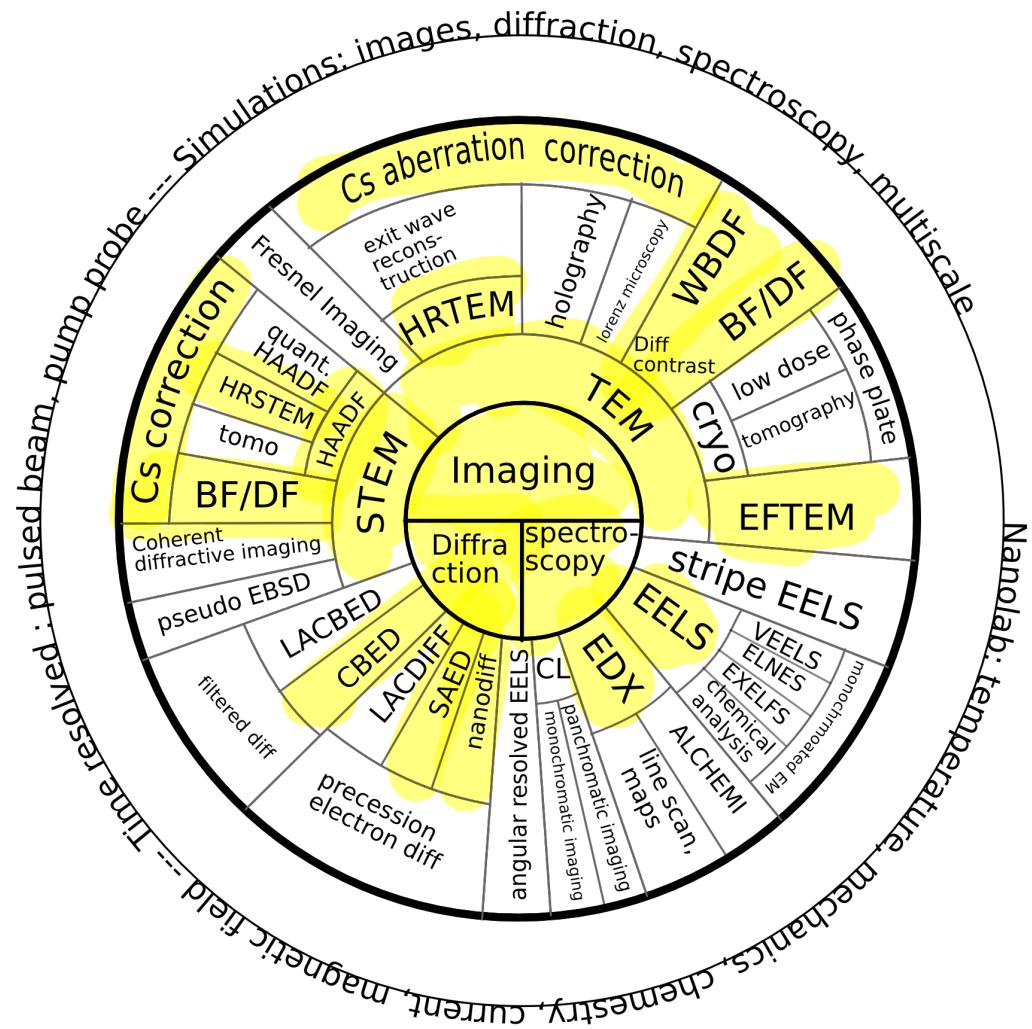
EPFL Course layout

- Week 0: course introduction, “refresher” on crystallography and ray optics
- Weeks 1–2: introduction to TEM (Coursera)
- Weeks 3–7: TEM diffraction and imaging. 3–6 on Coursera (with in-class supplements), week 7 conventional lecture
- Weeks 8–9: phase contrast TEM (Coursera)
- Weeks 10–11: scanning TEM (STEM); conventional lectures
- Weeks 12–13: analytical TEM; conventional lectures

EPFL Course evaluation

- Project-based evaluation with one individual report + oral evaluation during the exam period
- You will receive one (or max. 2) paper(s) with TEM images and data. You will be asked to write a short report explaining the techniques used for acquiring those images and data
- Each student will be given an individual interview based on their report. The questions will only be on the TEM-related techniques, but can encompass all parts of the course
- The grade will be 50% written report 50% oral exam

The TEM: a very versatile instrument

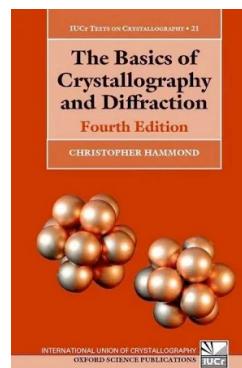


EPFL Chapter 0: background prerequisites

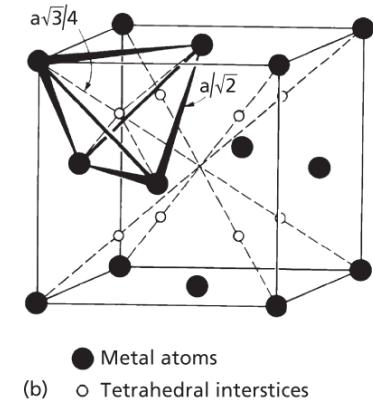
- Aim: provide short recap (or introduction?) of previous lecture courses you have taken
 1. Crystallography: real and reciprocal space (week 0)
 2. Geometrical optics: ray diagrams for converging lens (weeks 0–2)
 3. TEM basics: weeks 1–2 (Coursera)
- Your duty: identify the domain(s) where your background is less solid and catch up during the next 2 weeks!

EPFL The crystalline specimen

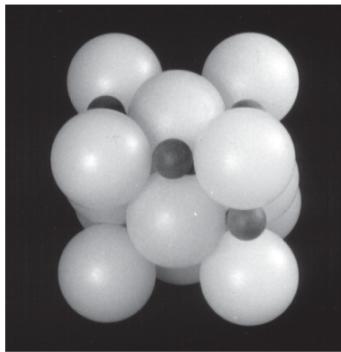
- A large part of the samples investigated by TEM are crystals (even sometimes in biology: e.g. proteins can be crystallised!)
- Many imaging techniques make specific use of the interaction of the electron beam with the crystal: high resolution TEM, diffraction, dark-field imaging, weak-beam imaging, convergent beam electron diffraction, high resolution scanning TEM...
- Therefore we need to be familiar with the basics of crystallography to understand TEM



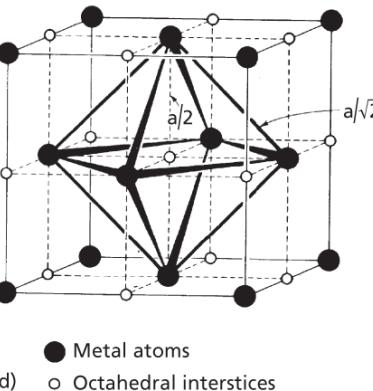
(a)



(b) Metal atoms
Tetrahedral interstices



(c)

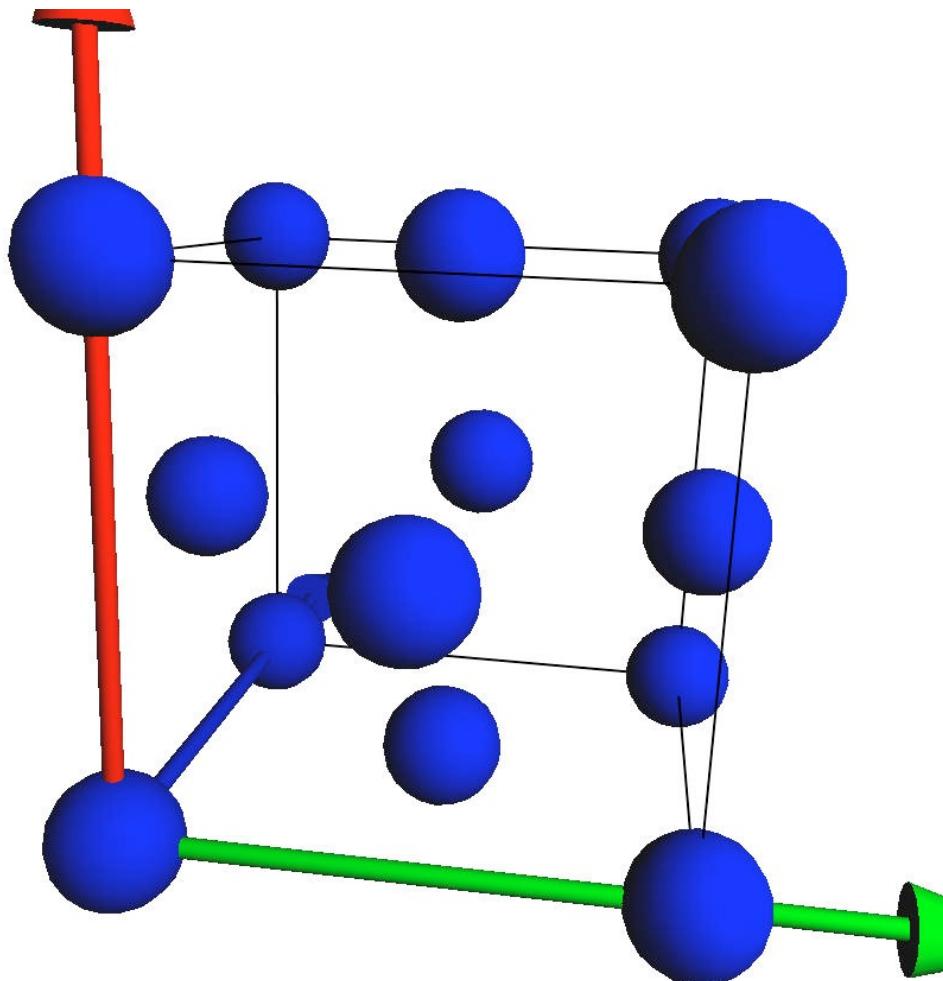


(d) Metal atoms
Octahedral interstices

<https://academic.oup.com/book/43705>

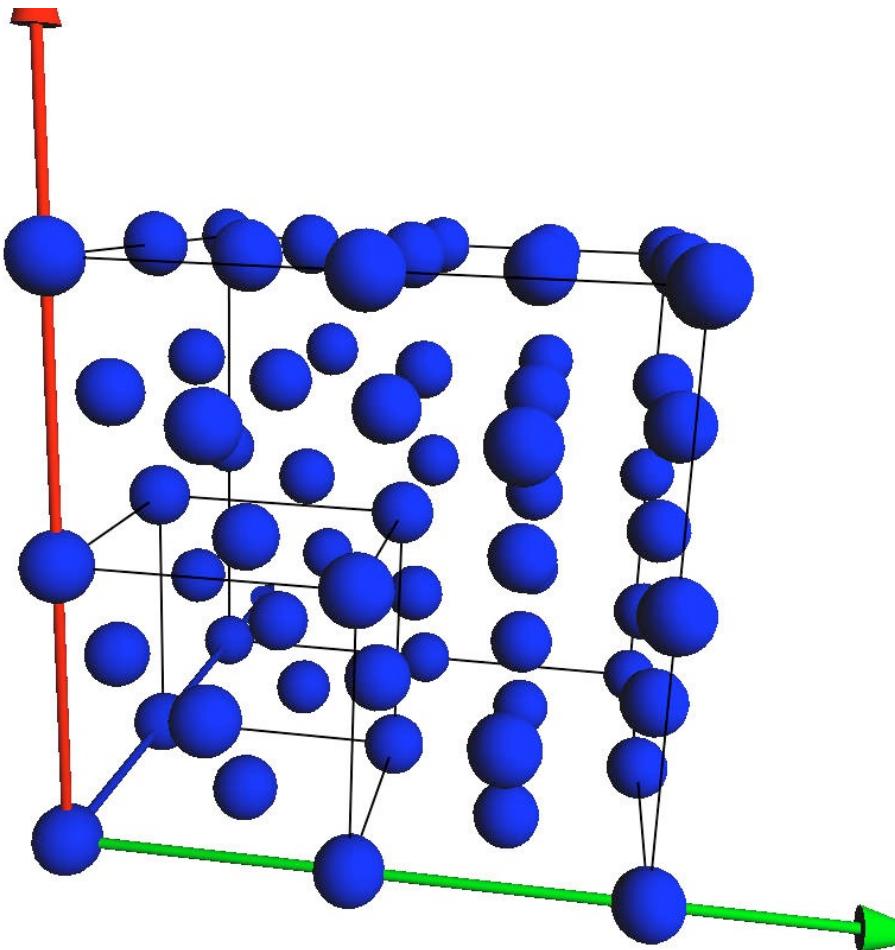
EPFL Crystalline nature

- Crystals defined as atomic structures with translational periodicity & symmetry



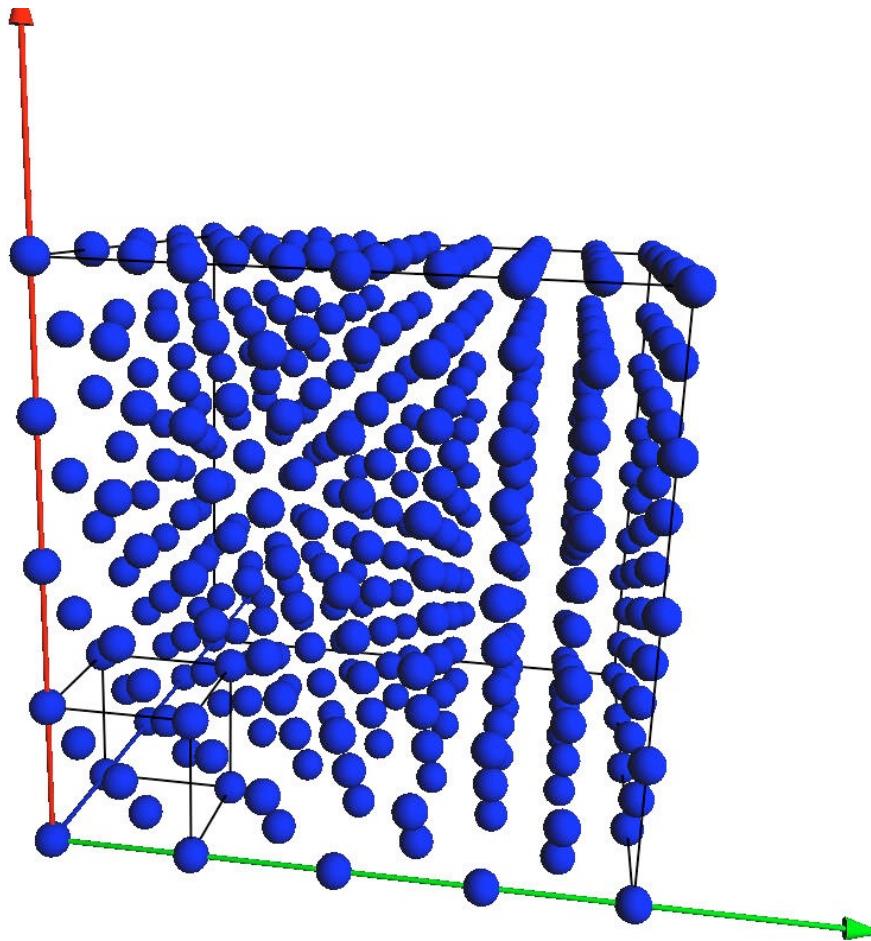
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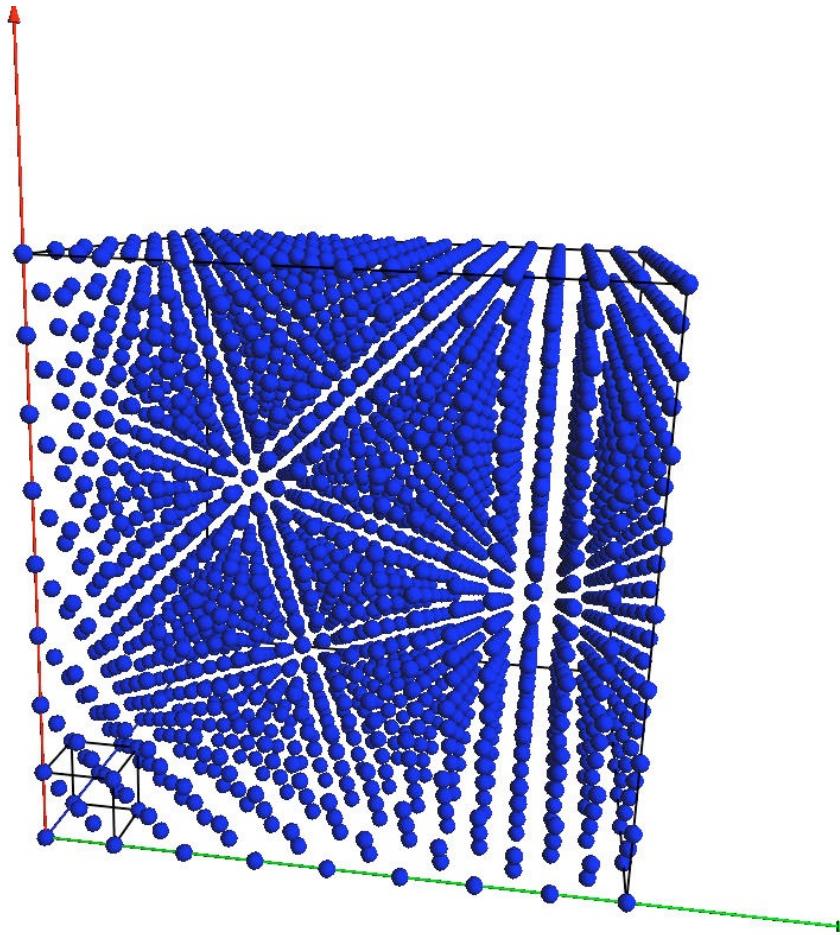
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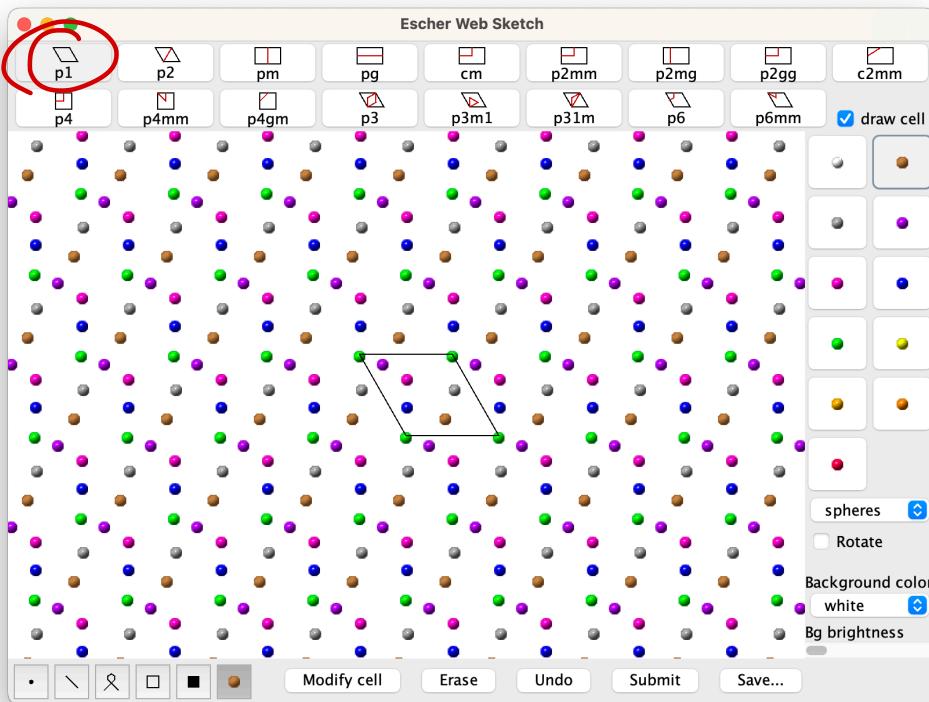
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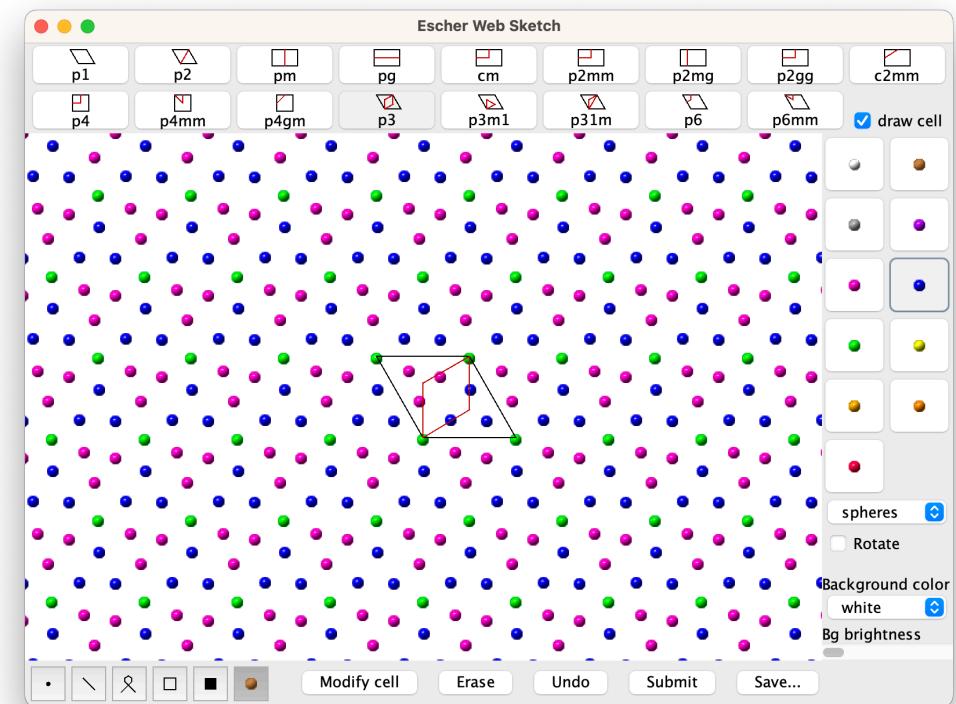
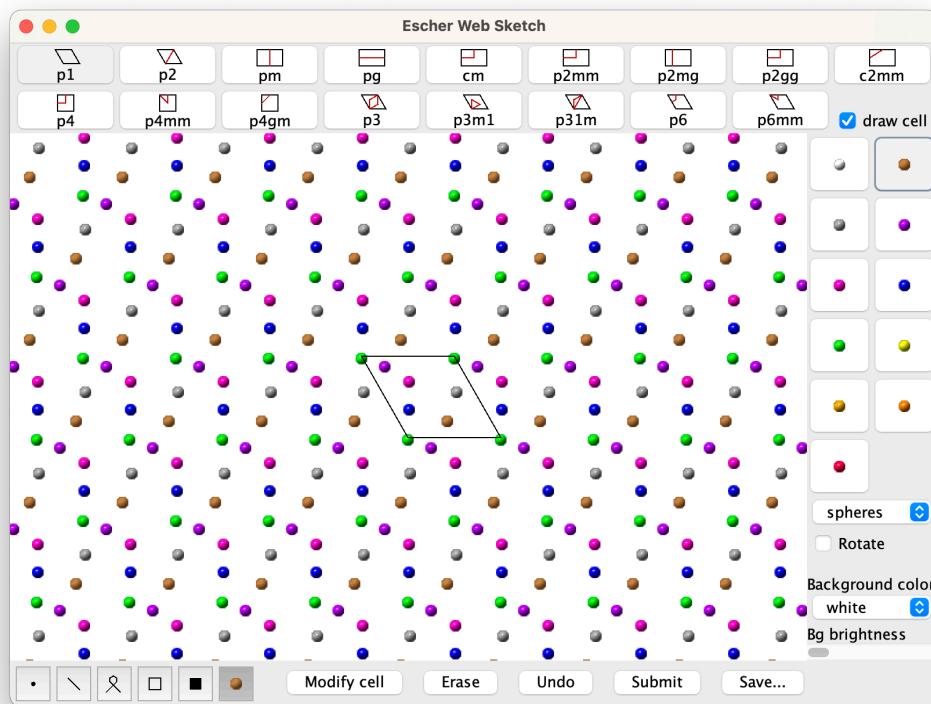
EPFL Crystallography

- Identify the (smallest) repeating pattern
- Determine the symmetries involved in repeating the pattern
- Java app for exploring symmetries in 2D:
<https://www.epfl.ch/schools/sb/research/iphys/teaching/crystallography/escher-web-sketch/>



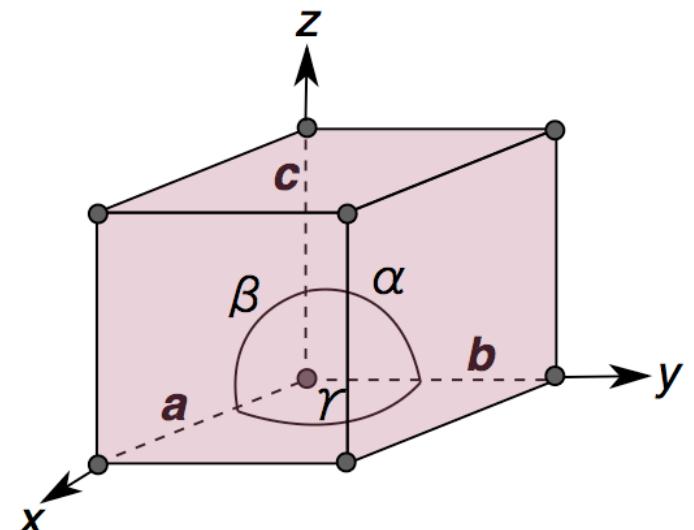
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EPFL Crystallography: the unit cell

- Unit cell is the (usually) smallest repeating unit of the crystal lattice
- Has a lattice point on each corner (and perhaps more elsewhere)
- Defined by:
 - lattice parameters a, b, c along axes x, y, z
 - angles between crystallographic basis vectors:
 $\gamma = \mathbf{a} \angle \mathbf{b}$; $\alpha = \mathbf{b} \angle \mathbf{c}$; $\beta = \mathbf{a} \angle \mathbf{c}$



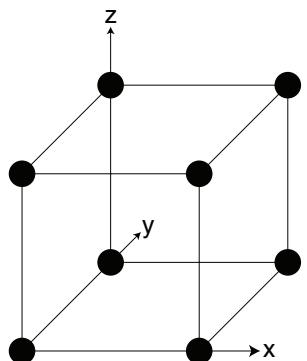
Cubic $a = b = c$
 $\gamma = \alpha = \beta = 90^\circ$

Hexagonal: $a = b \neq c$ $\gamma = 120^\circ$

EPFL Building a crystal structure

Use example of CuZn brass

Choose the unit cell - for CuZn: primitive cubic (lattice point on each corner)



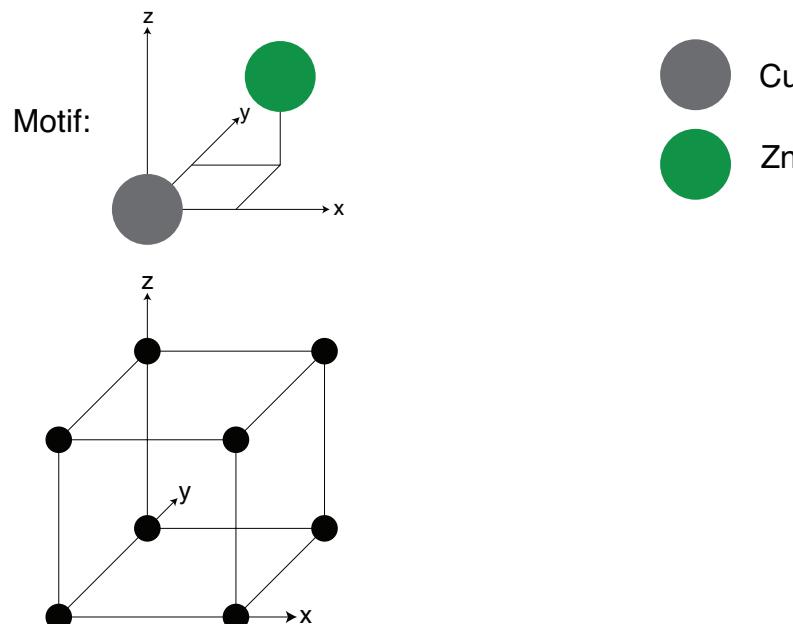
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Choose the motif – Cu: 0, 0, 0; Zn: $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$

*arrangement
x unit
cell
parametr* \leftarrow *fixed to each
lattice point*



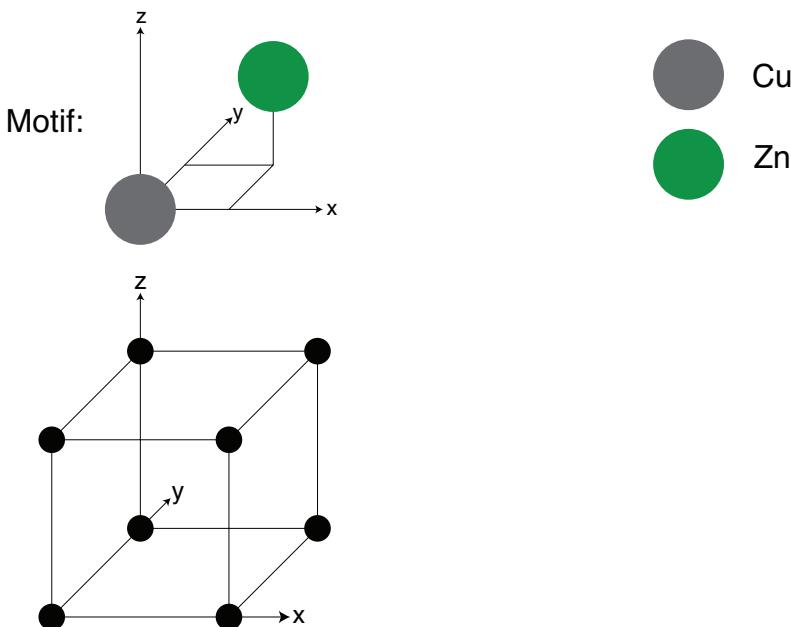
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Structure = lattice +motif \Rightarrow Start applying motif to each lattice point



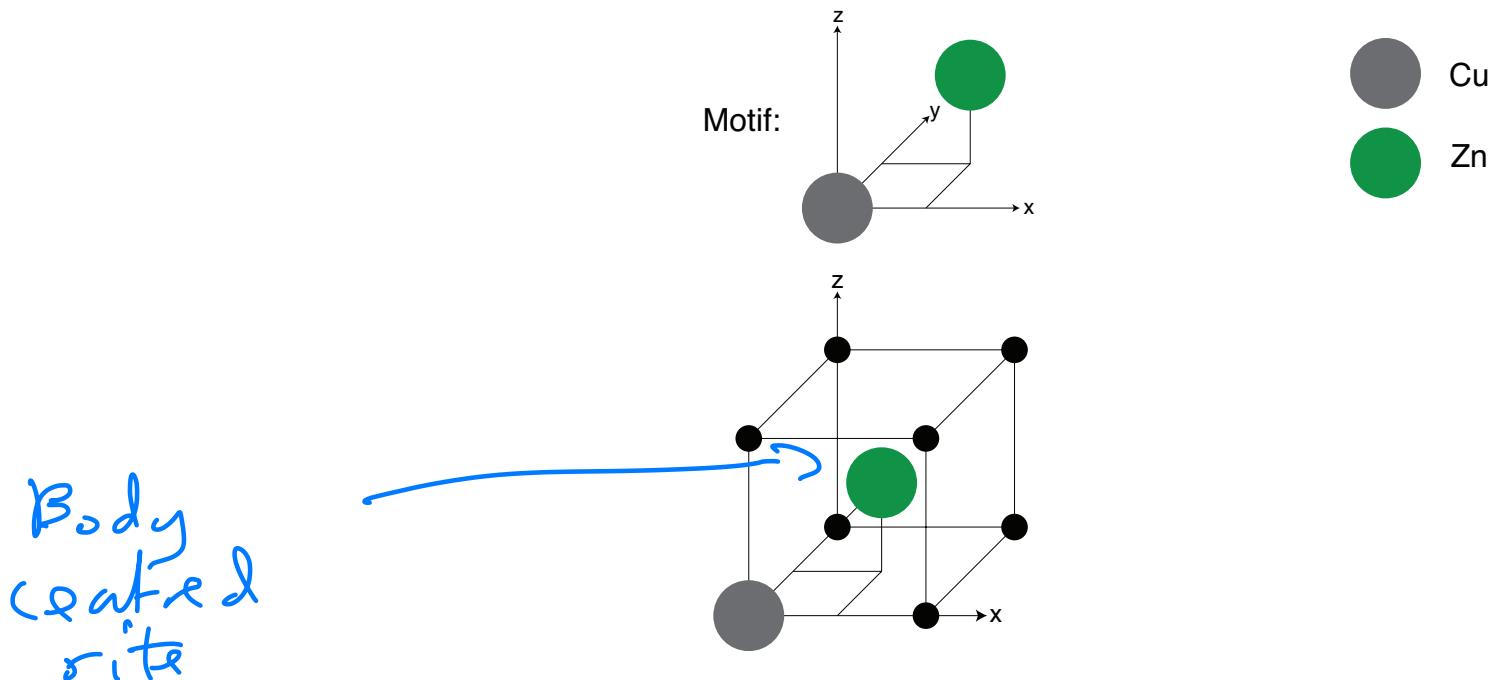
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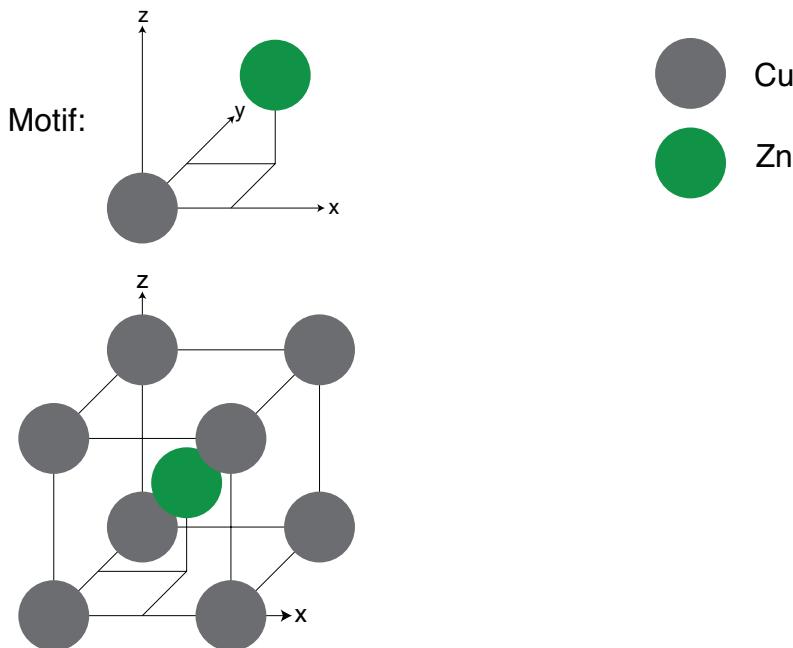
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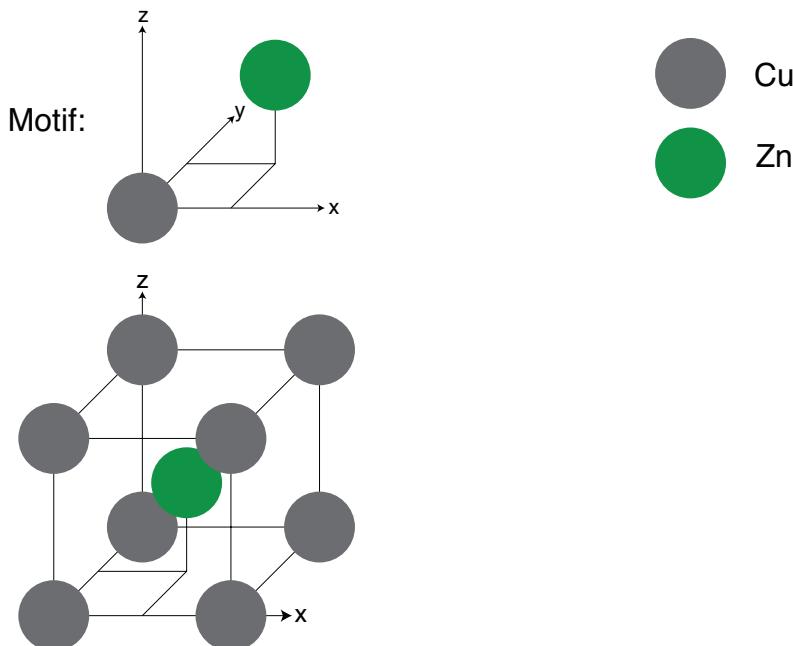
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Extend lattice further in to space



EPFL Building a crystal structure

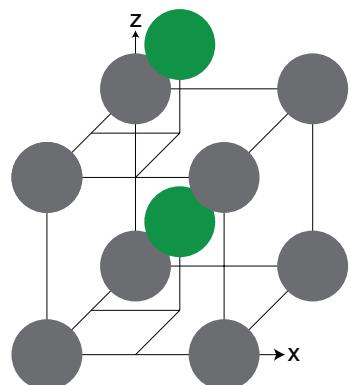
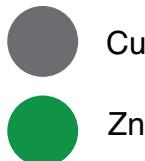
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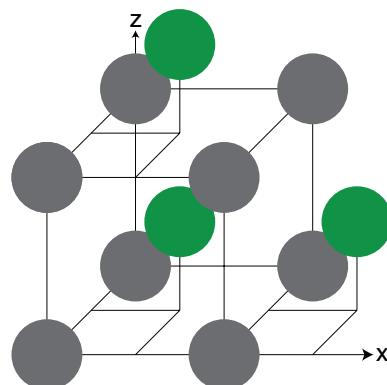
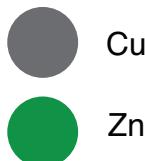
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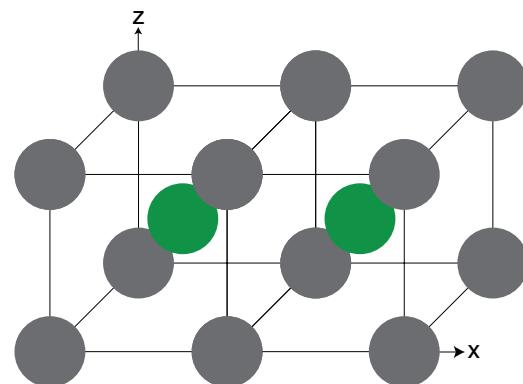
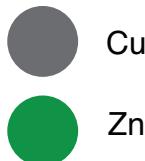
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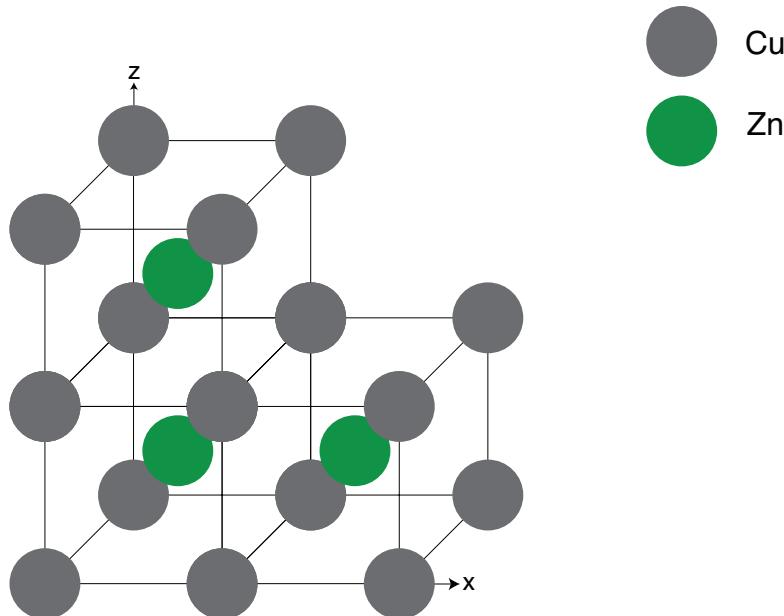
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Extend lattice further in to space



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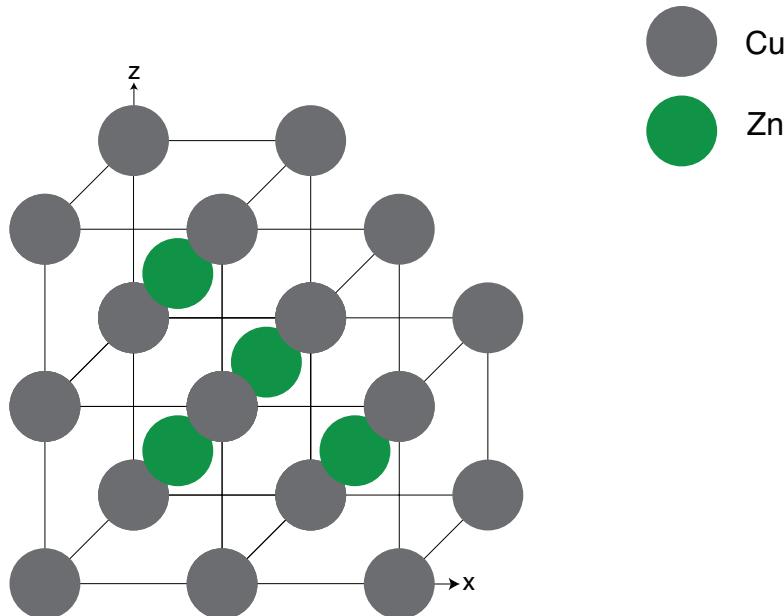
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Extend lattice further in to space



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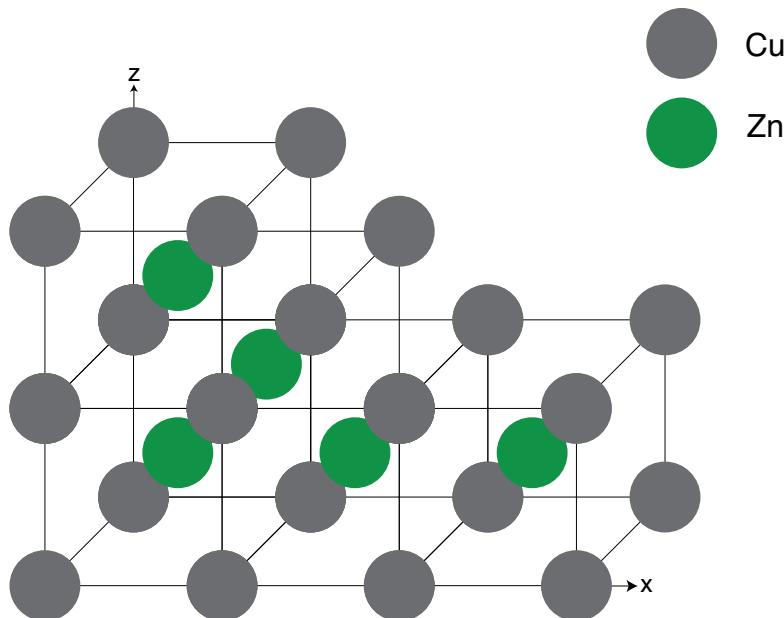
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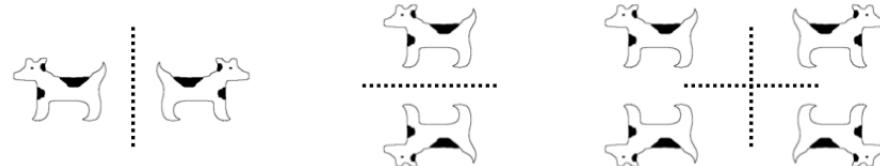
Extend lattice further in to space



EPFL Symmetry elements

- As well as having translational symmetry, nearly all crystals obey other symmetries – i.e. can reflect or rotate structure without changing atom positions

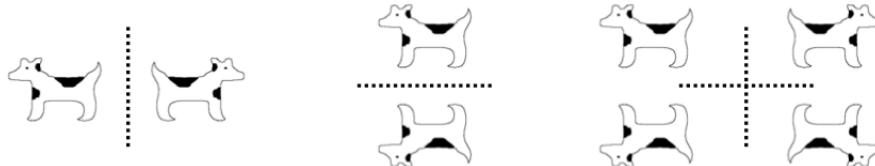
Mirror planes:



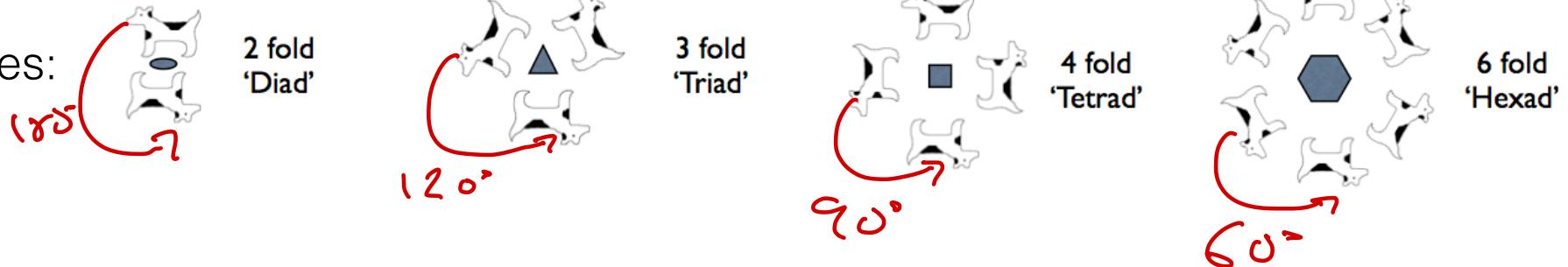
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Mirror planes:



Rotation axes:

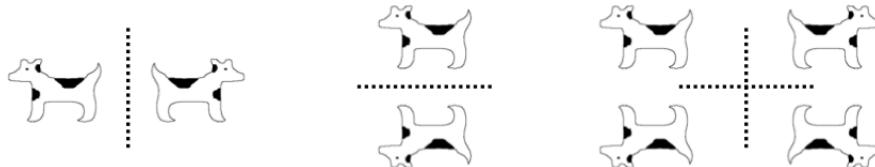


No 5-fold: no translational symmetry
↳ "Quasicrystals"

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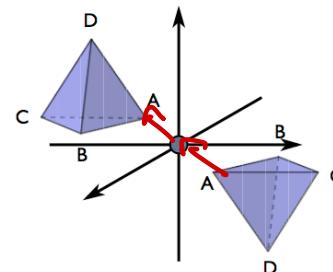
Mirror planes:



Rotation axes:



Centre of symmetry or inversion centre:

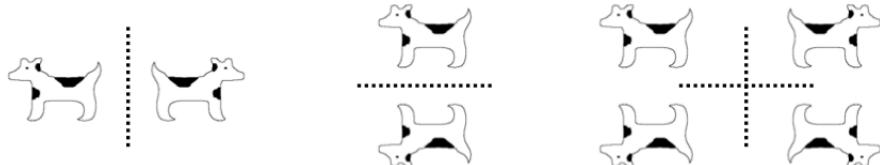


Invert through point

EPFL Symmetry elements

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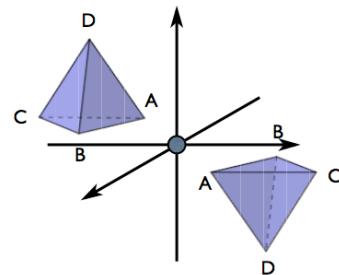
Mirror planes:



Rotation axes:



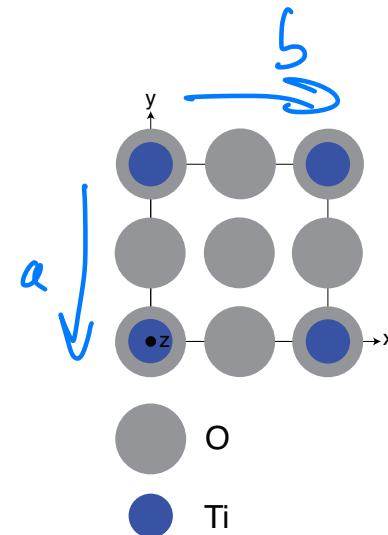
Centre of symmetry or inversion centre:



Inversion axes: combination of rotation axis with centre of symmetry

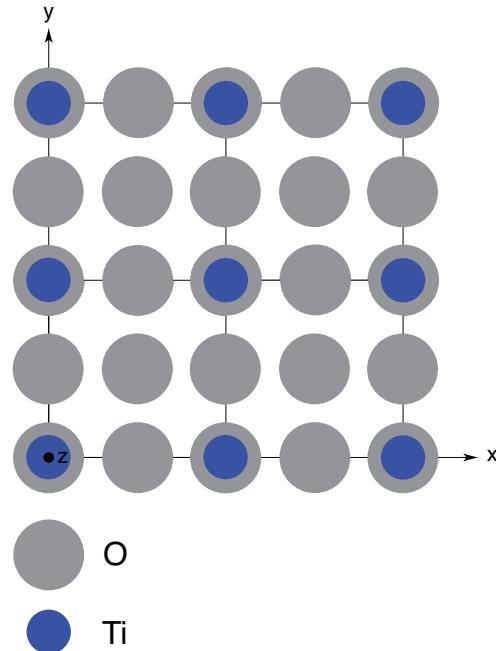
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- Example – tetragonal lattice:
 $a = b \neq c; \alpha = \beta = \gamma = 90^\circ$
- Anatase TiO_2 (body-centred lattice) view down $[0 0 1]$ (z -axis):



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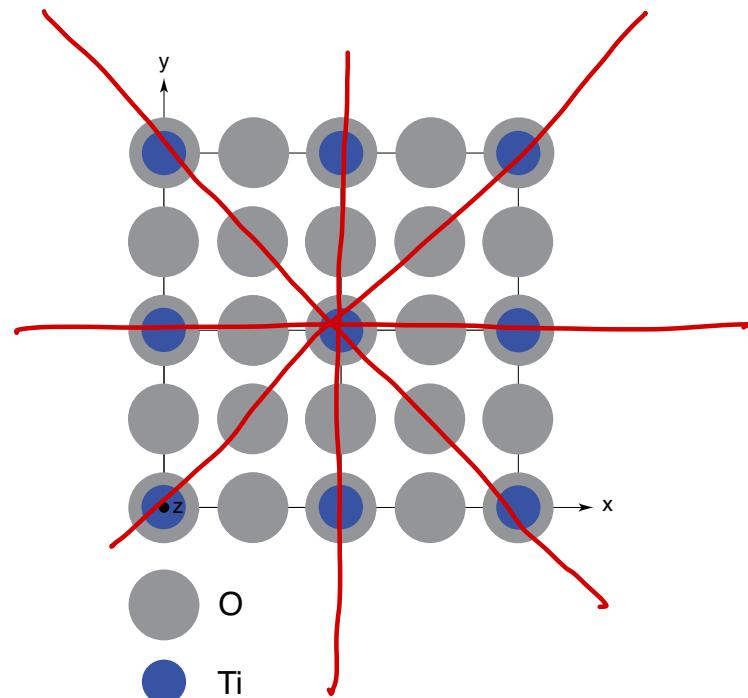


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Identify mirror planes

Identify rotation axis



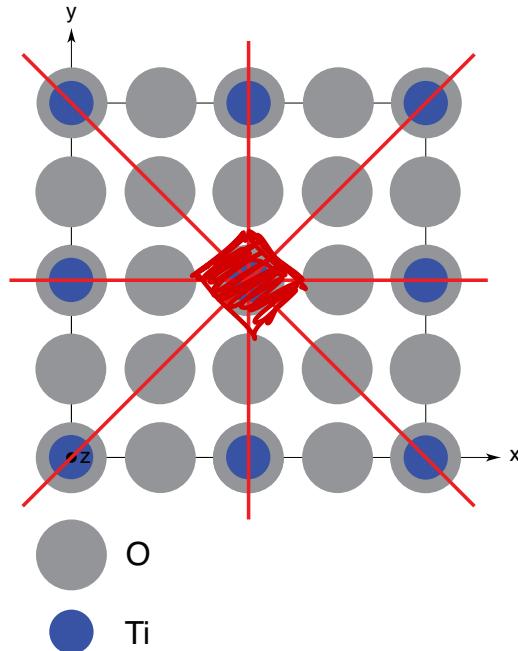
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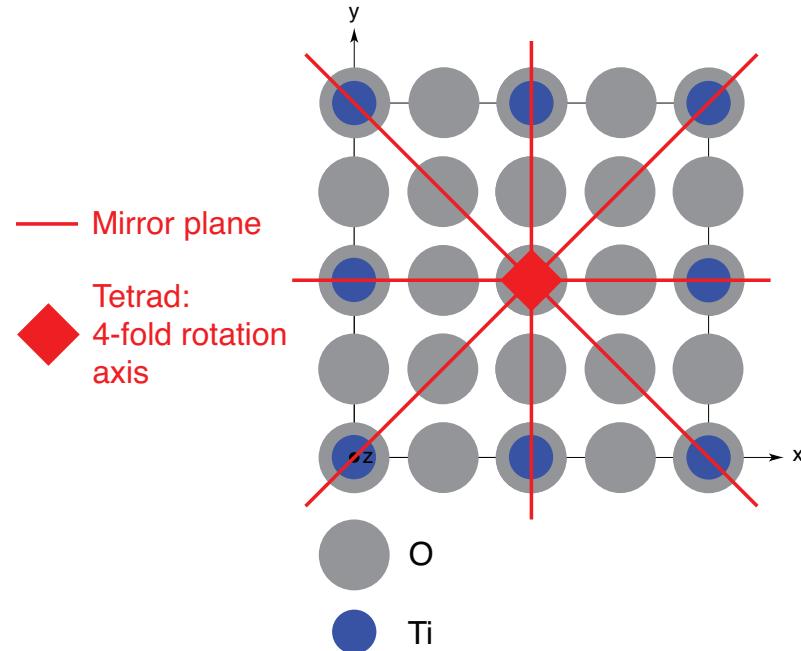
4-fold "tetrad"



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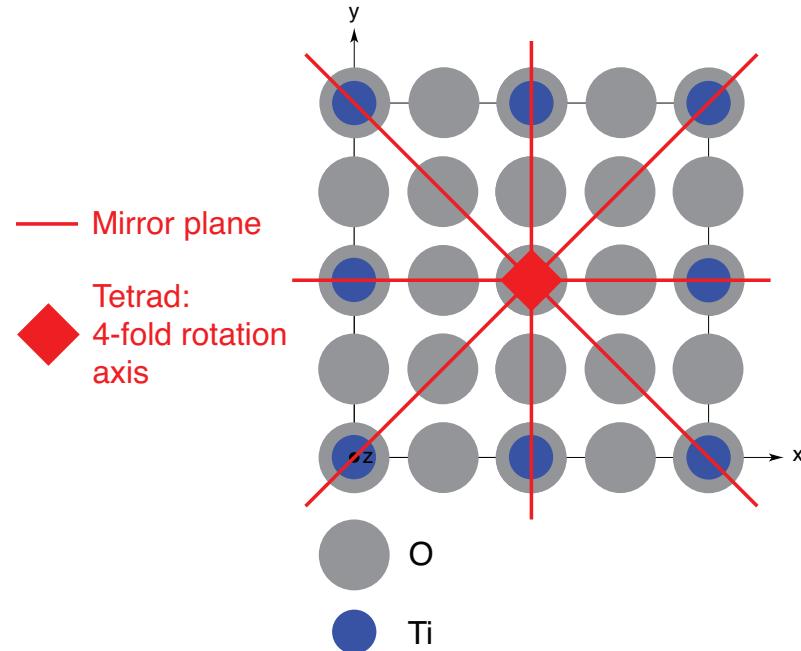


Identify rotation axis

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Identify mirror planes



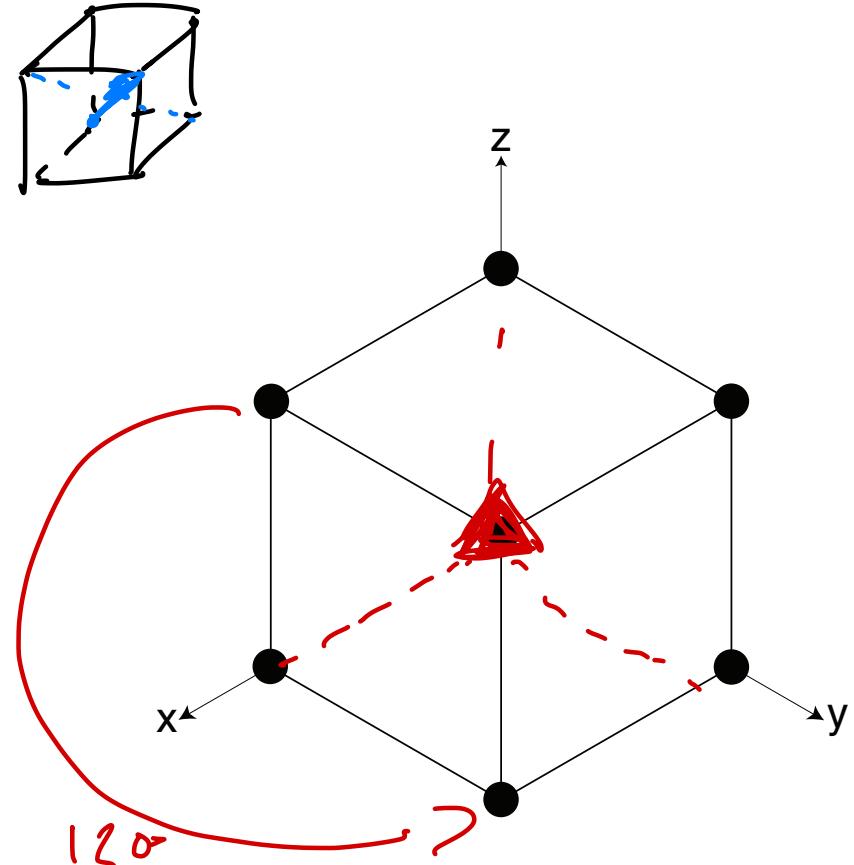
Identify rotation axis: 4-fold = defining symmetry of tetragonal lattice!

EPFL More defining symmetry elements

- Cubic crystal system:
 $a = b = c; \alpha = \beta = \gamma = 90^\circ$
- View down body diagonal (i.e. [1 1 1] axis)
- Choose Primitive cell
(lattice point on each corner)

Identify rotation axis:

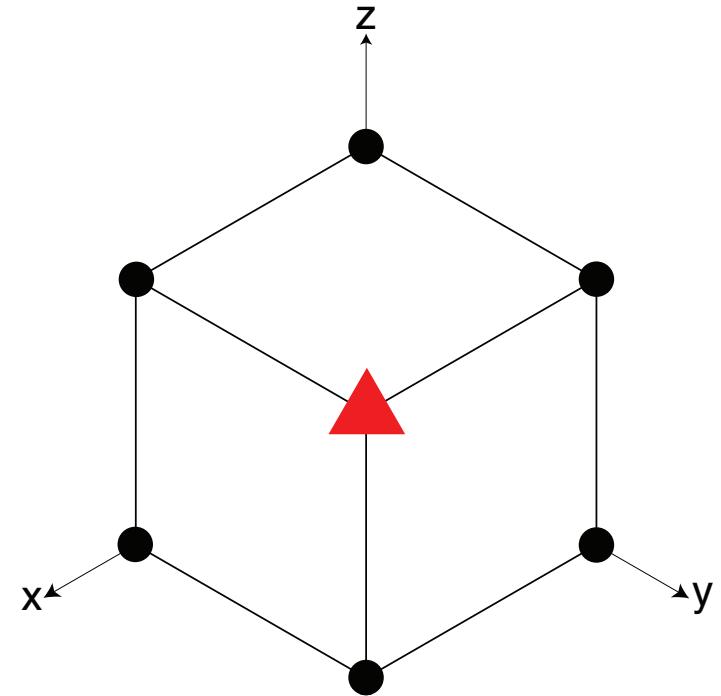
3-fold axis
(Triad)



EPFL More defining symmetry elements

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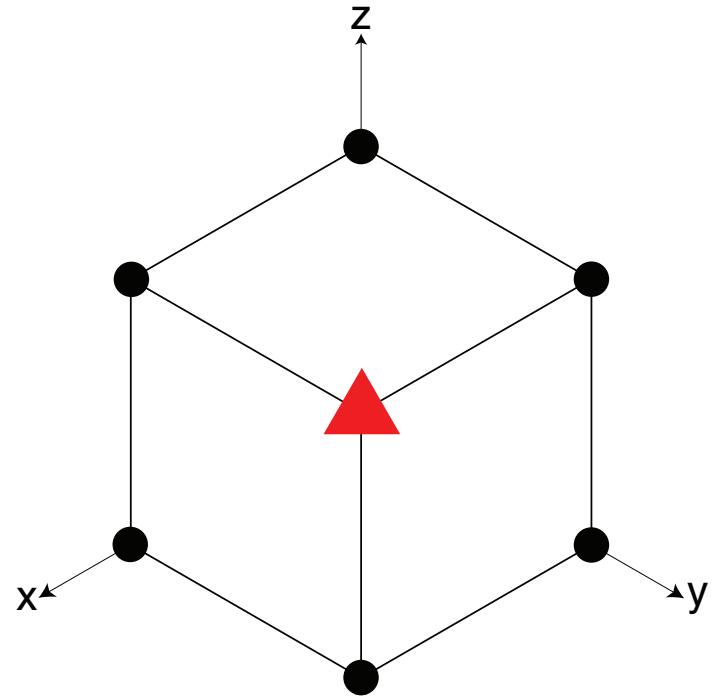
Identify rotation axis:



EPFL More defining symmetry elements

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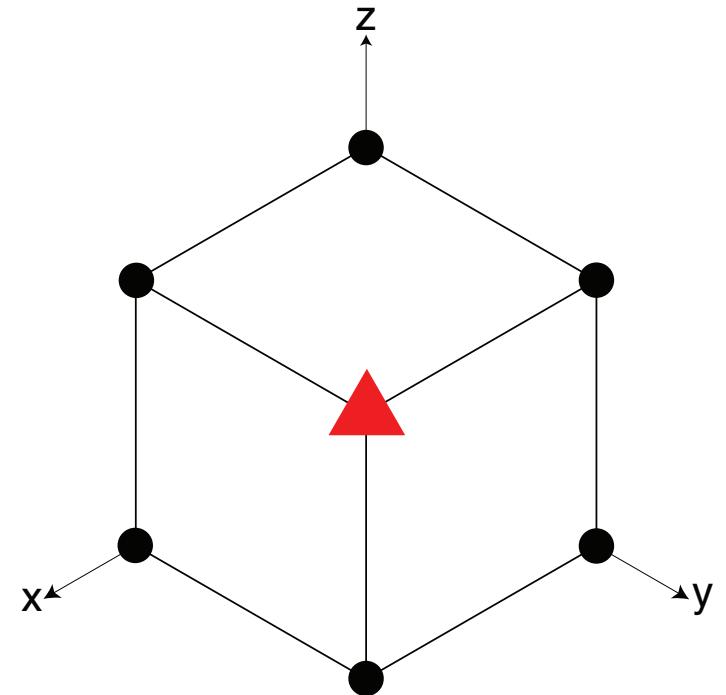
Identify rotation axis: 3-fold (triad)



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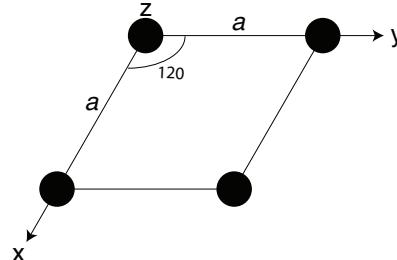
Identify rotation axis: 3-fold (triad)



Defining symmetry of cube: four 3-fold rotation axes (not 4-fold rotation axes!)

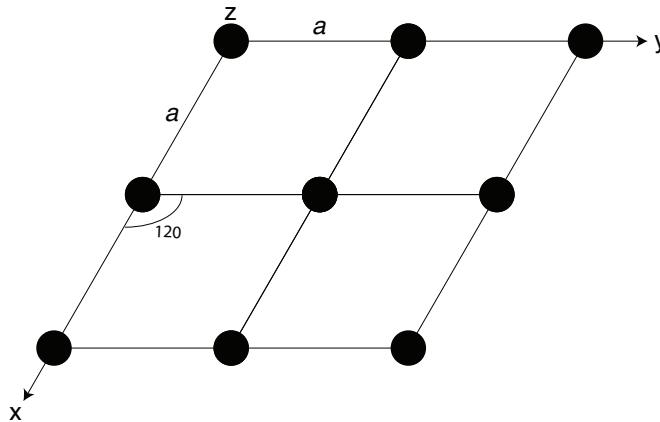
EPFL More defining symmetry elements

- Hexagonal crystal system:
 $a = b \neq c$;
 $\alpha = \beta = 90^\circ, \gamma = 120^\circ$
- Primitive cell, lattice points on each corner; view down z-axis – i.e. $[0 0 1]$



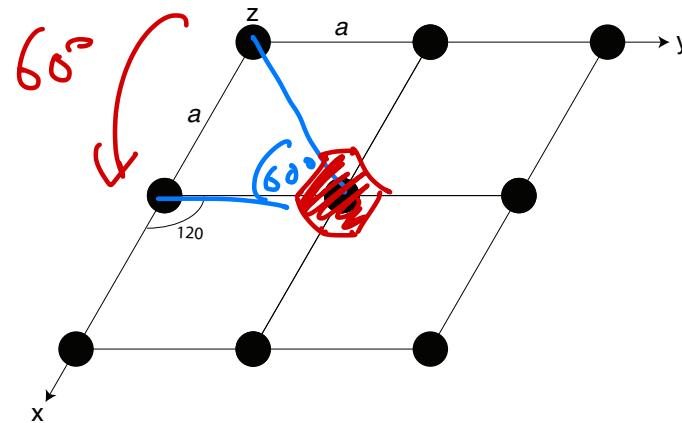
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 $a = b \neq c$;
 $\alpha = \beta = 90^\circ, \gamma = 120^\circ$
- Primitive cell, lattice points on each corner; view down z-axis – i.e. $[0 0 1]$
- Draw 2×2 unit cells



EPFL More defining symmetry elements

- Hexagonal crystal system:
 $a = b \neq c$;
 $\alpha = \beta = 90^\circ, \gamma = 120^\circ$
- Primitive cell, lattice points on each corner; view down z-axis – i.e. $[0 0 1]$
- Draw 2×2 unit cells

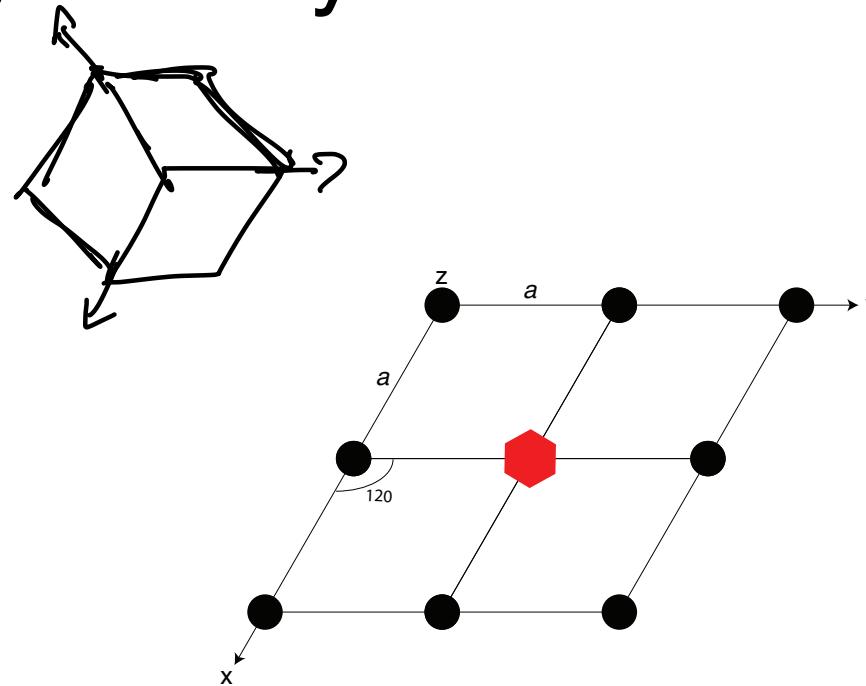


Identify rotation axis:

$6-h(d)$ (Hexad)

EPFL More defining symmetry elements

- Hexagonal crystal system:
 $a = b \neq c$;
 $\alpha = \beta = 90^\circ, \gamma = 120^\circ$
- Primitive cell, lattice points on each corner; view down z-axis – i.e. $[0 0 1]$
- Draw 2×2 unit cells



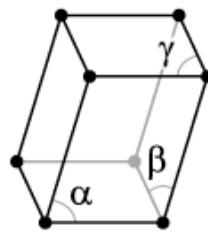
Identify rotation axis: 6-fold (hexad) – defining symmetry of hexagonal lattice

EPFL The seven crystal systems

- 7 possible unit cell shapes with different symmetries that can be repeated by translation in 3 dimensions
⇒ 7 crystal systems each defined by symmetry

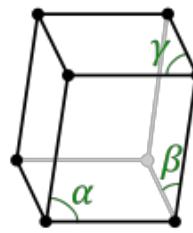
Triclinic

$$\alpha, \beta, \gamma \neq 90^\circ$$



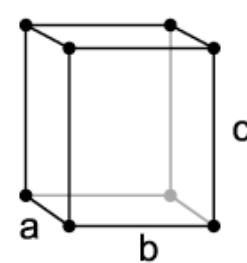
Monoclinic

$$\alpha \neq 90^\circ \\ \beta, \gamma = 90^\circ$$



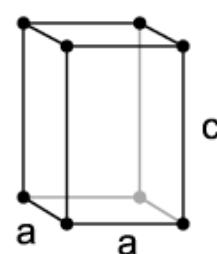
Orthorhombic

$$a \neq b \neq c$$



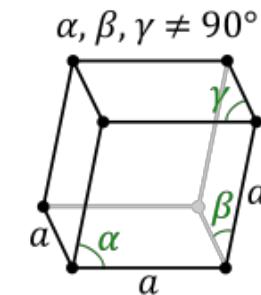
Tetragonal

$$a \neq c$$

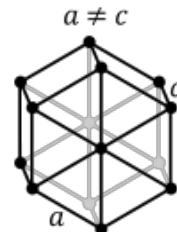


Rhombohedral

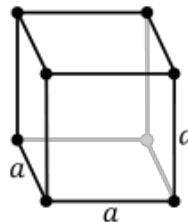
$$\alpha, \beta, \gamma \neq 90^\circ$$



Hexagonal

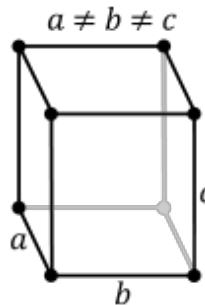


Cubic

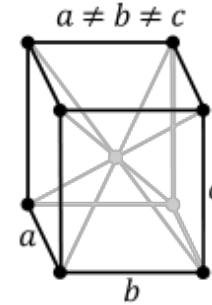


EPFL Four possible lattice centerings

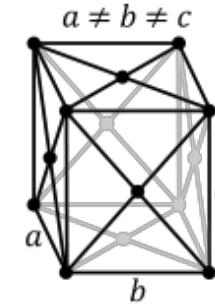
P: Primitive - lattice points on cell corners



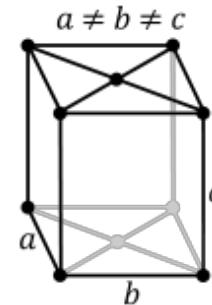
I: Body-centred - additional lattice point at cell centre



F: Face-centred - one additional lattice point at centre of each face



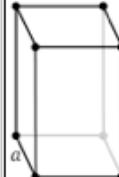
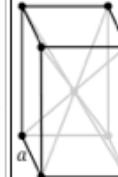
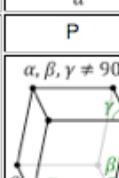
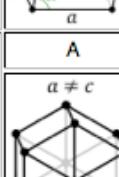
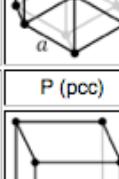
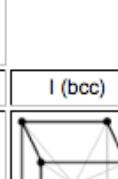
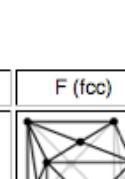
A/B/C: Centred on a single face - one additional lattice point centred on A, B or C face



EPFL 14 Bravais lattices

- Combinations of crystal systems and lattice point centering that describe all possible crystals – equivalent system/centring combinations eliminated $\Rightarrow 14$ (not $7 \times 4 = 28$)

The 7 Crystal systems		The 14 Bravais lattices															
triclinic		P	$\alpha, \beta, \gamma \neq 90^\circ$														
monoclinic		P	$\alpha \neq 90^\circ$	$\beta, \gamma = 90^\circ$													
		C	$\alpha \neq 90^\circ$	$\beta, \gamma = 90^\circ$													
orthorhombic		P	$a \neq b \neq c$	C	I	F	$a \neq b \neq c$	$a \neq b \neq c$	$a \neq b \neq c$								
			a	b	c		a	b	c		a	b	c		a	b	c

The 7 Crystal systems		The 14 Bravais lattices		
	tetragonal	P $a \neq c$ 	I $a \neq c$ 	
	rhombohedral (trigonal)	P $\alpha, \beta, \gamma \neq 90^\circ$ 		
	hexagonal	A $a \neq c$ 		
cubic		P (pcc) 	I (bcc) 	F (fcc) 

Tetragonal
body centered
 \equiv face centered

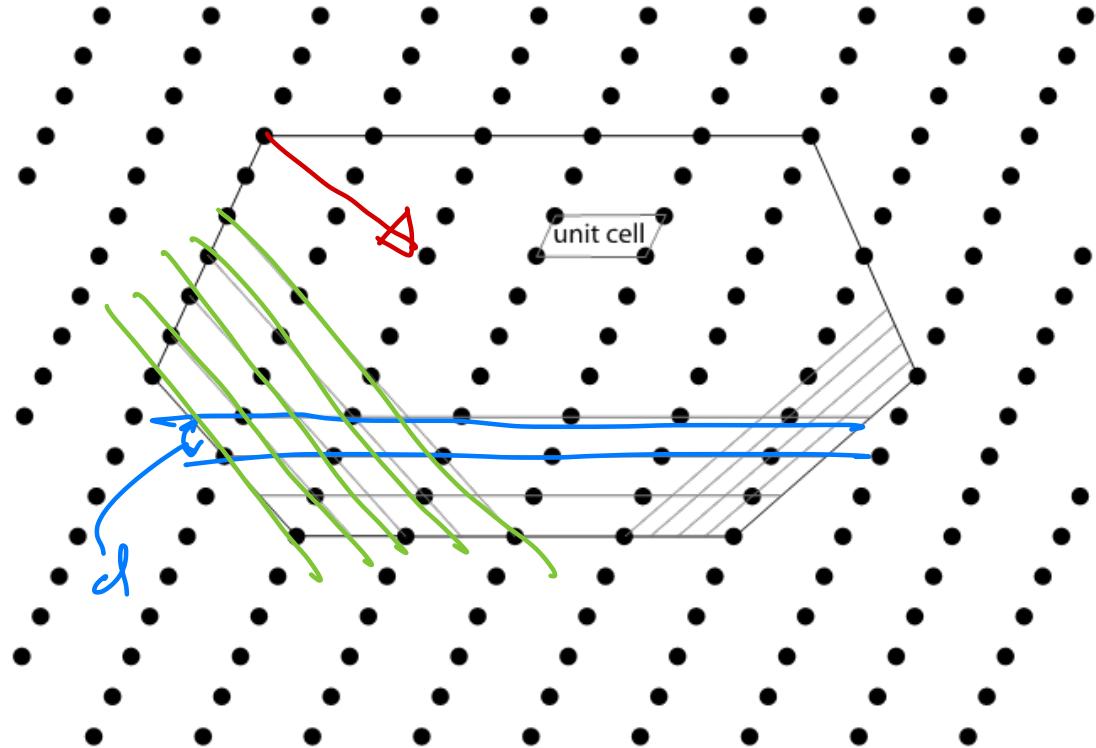
I: body-centred
F: face-centred

Crystal System	Defining Symmetry (rotation or inversion)	Conventional Unit Cell	Conventional Lattice Types
Cubic	4 triads	$a = b = c$ $\alpha = \beta = \gamma = 90^\circ$	P, I, F
Hexagonal	1 hexad	$a = b \neq c$ $\alpha = \beta = 90^\circ, \gamma = 120^\circ$	P
Trigonal	1 triad	$a = b \neq c$ $\alpha = \beta = 90^\circ, \gamma = 120^\circ$	P, R
Tetragonal	1 tetrad	$a = b \neq c$ $\alpha = \beta = \gamma = 90^\circ$	P, I
Orthorhombic	3 diads	$a \neq b \neq c$ $\alpha = \beta = \gamma = 90^\circ$	P, C, I, F
Monoclinic	1 diad	$a \neq b \neq c$ $\alpha = \gamma = 90^\circ, \beta \geq 90^\circ$	P, C
Triclinic	-	$a \neq b \neq c$ $\alpha \neq \beta \neq \gamma$	P

- Combine crystal systems and lattice types with different compatible symmetry element combinations \Rightarrow 230 space groups

EPFL Crystal lattice planes and vectors

- In 2D, we can draw families of lines parallel to each other, containing the same linear arrangement of atoms
- In 3D these are called **lattice planes**
- Lattice planes are defined using **Miller indices**
- We can also define vectorial directions – how do we label these?

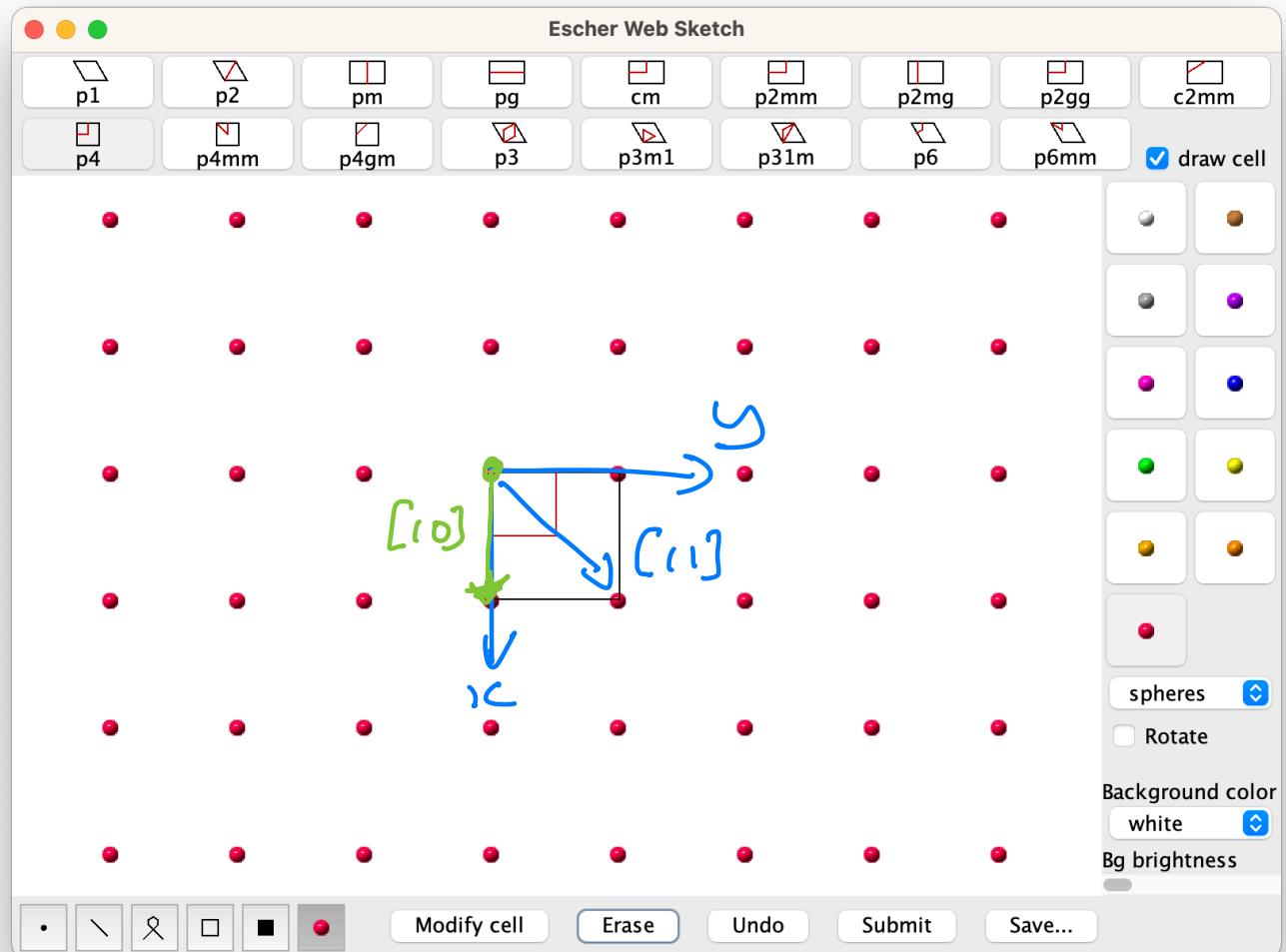


EPFL Lattice vectors

- Lattice vector: a vector joining any two lattice points
- Written as linear combination of unit cell vectors \mathbf{a} , \mathbf{b} , \mathbf{c} :
$$\mathbf{t} = U\mathbf{a} + V\mathbf{b} + W\mathbf{c}$$
- Write in square brackets: $\mathbf{t} = [U \ V \ W]$

EPFL Lattice vectors (2D)

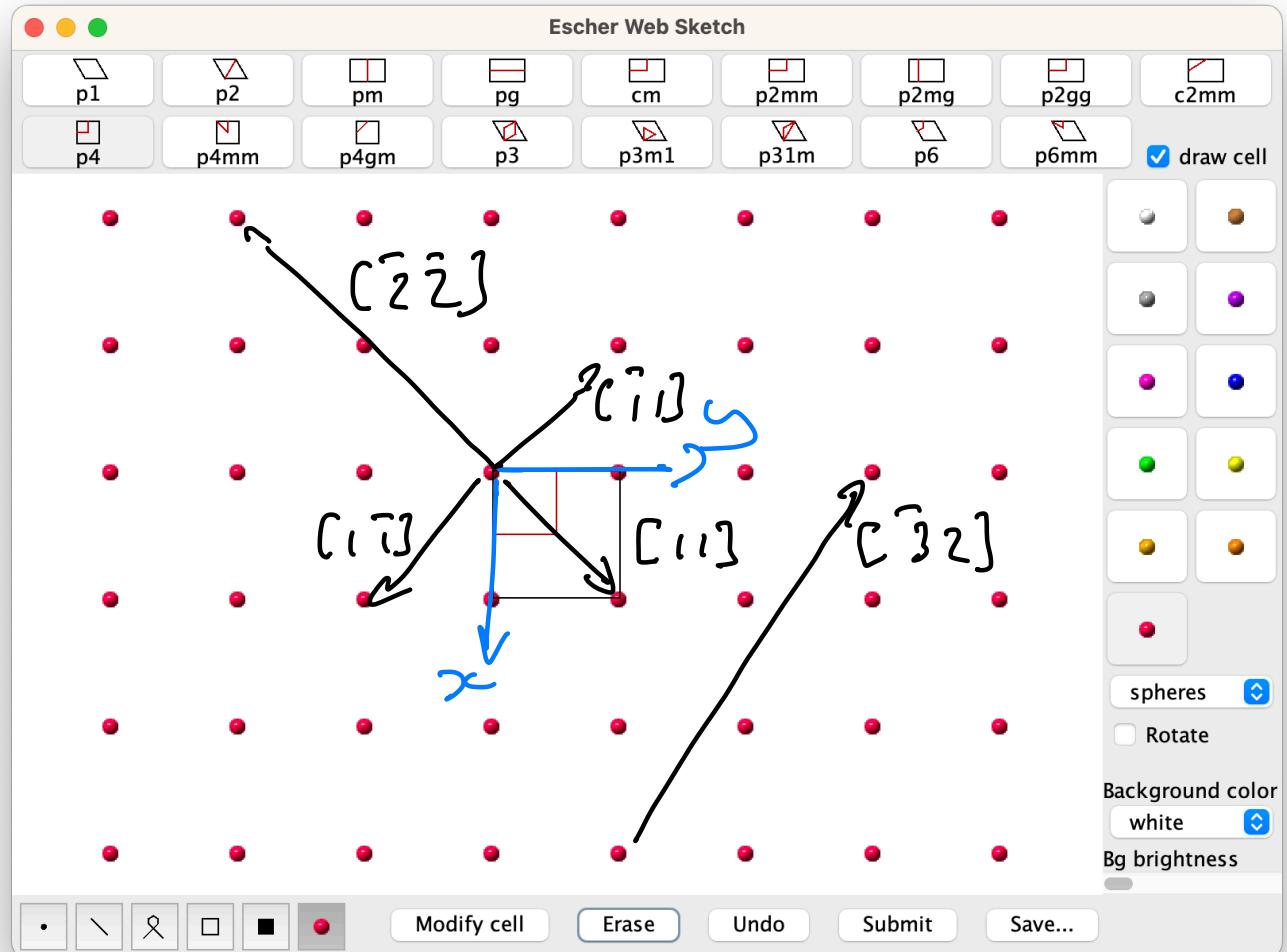
- For 2D lattice on right, draw lattice vectors:
 - Parallel to $[1 \ 0]$
 - On the diagonal $[1 \ 1]$



EPFL Lattice vectors (2D)

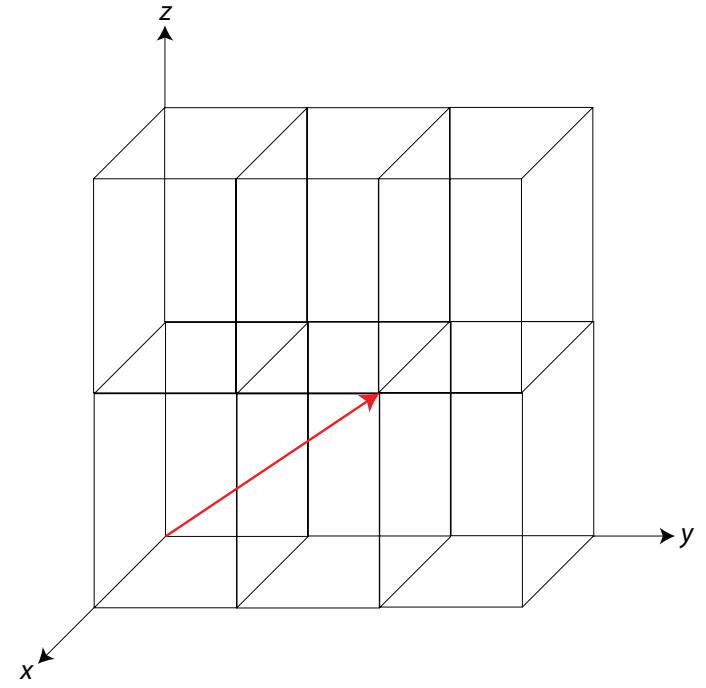
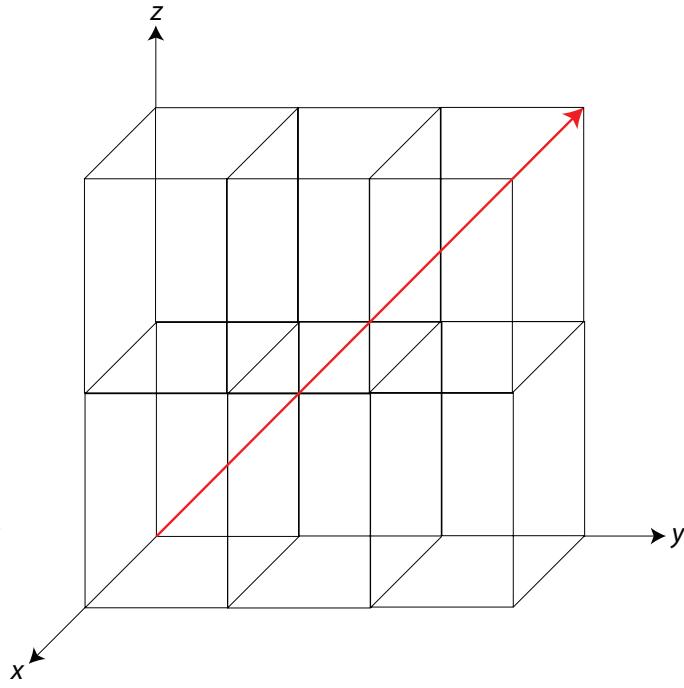
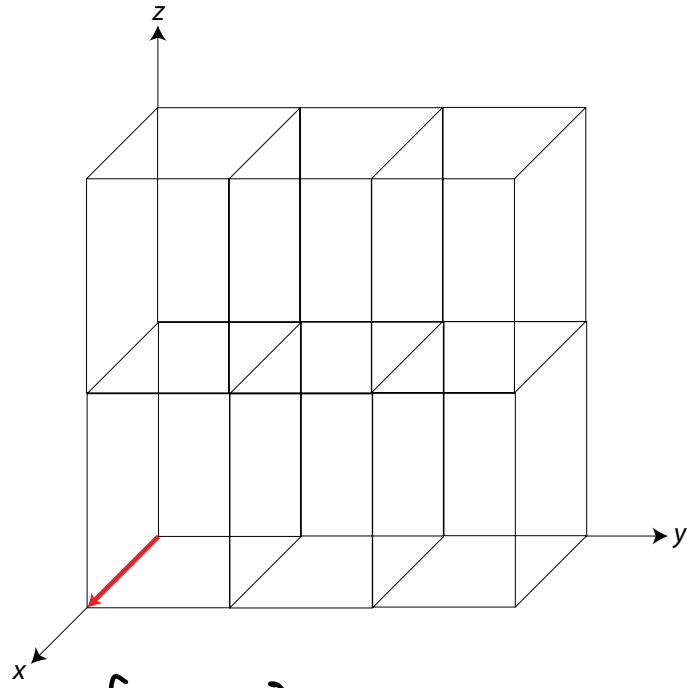
- Now draw:
 - $[1 \ 1]$
 - $[1 \ \bar{1}]$
 - $[\bar{1} \ 1]$
 - $[\bar{3} \ 2]$
 - $[\bar{2} \ \bar{2}]$

Families of similar directions are given in angular brackets
 $\langle U \ V \ W \rangle$



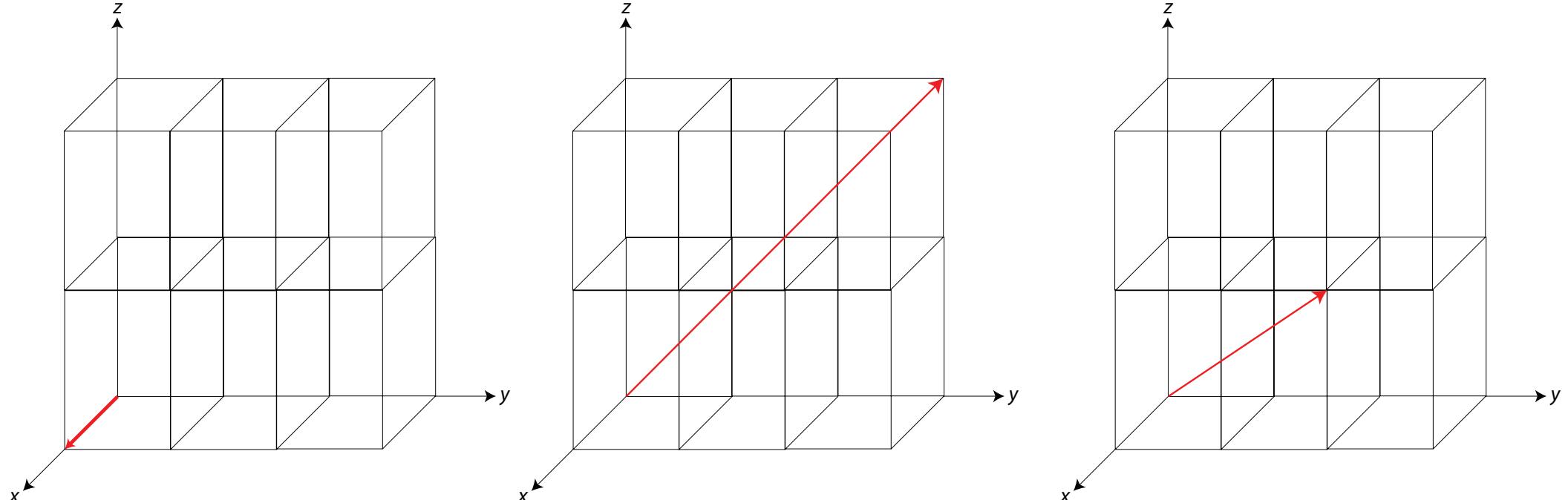
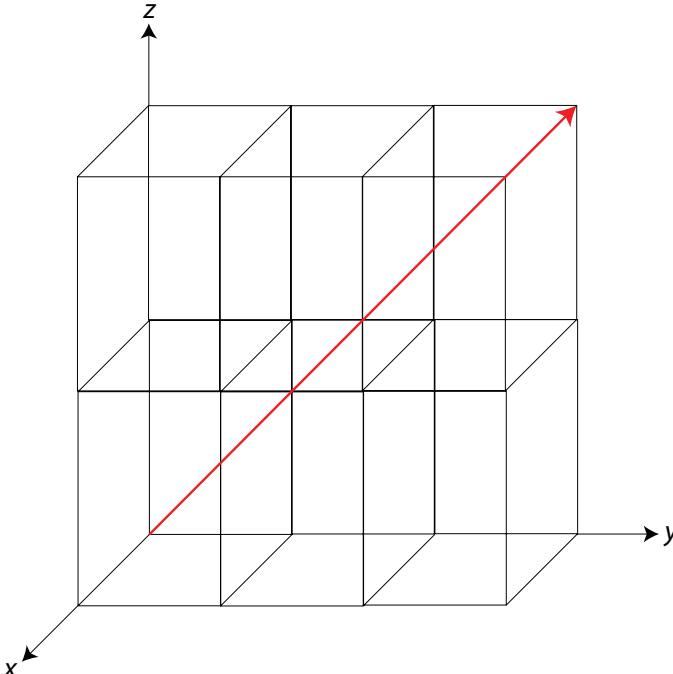
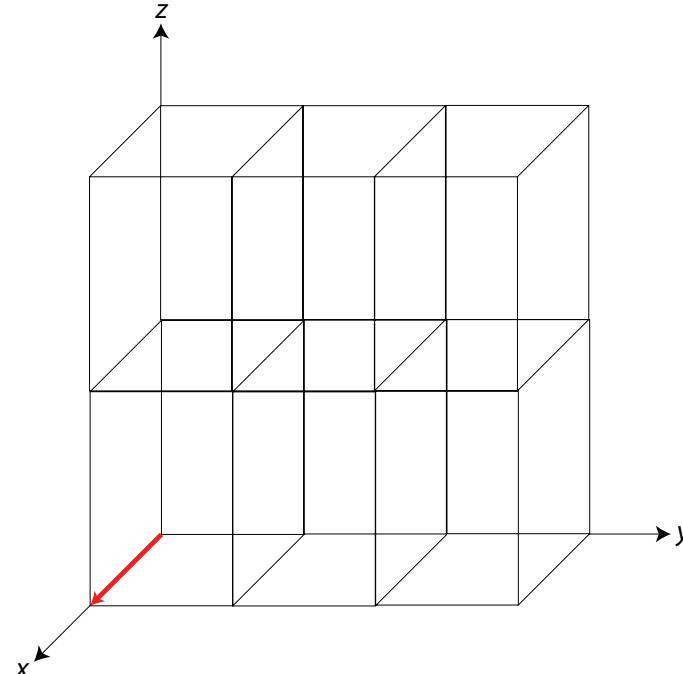
EPFL Lattice vectors (3D)

- Now look at examples in 3D:



EPFL Lattice vectors (3D)

- Now look at examples in 3D:



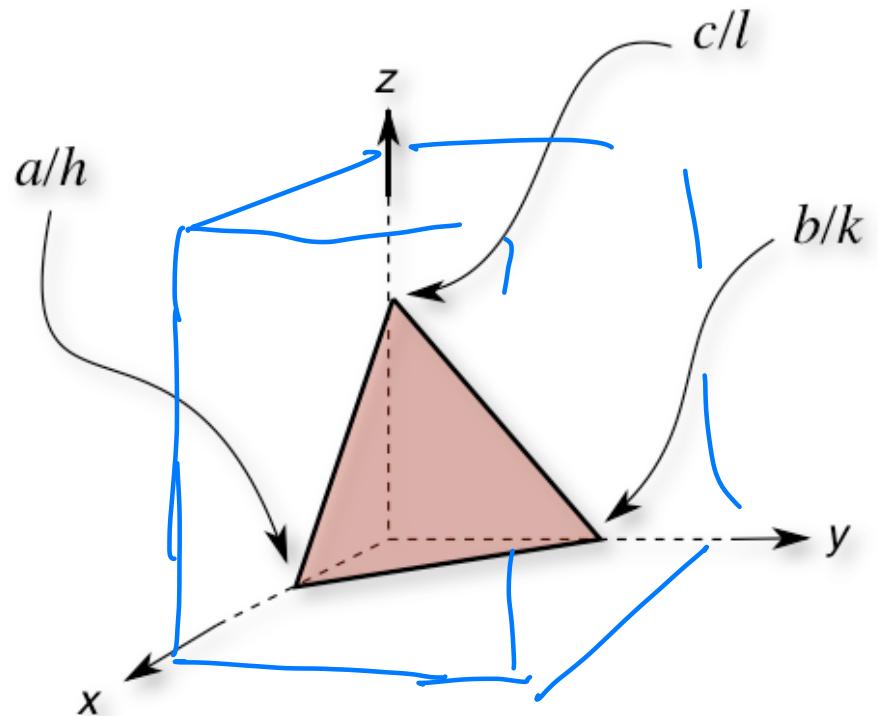
$[100]$
 $\langle 100 \rangle$ - family of dir's
e.g. cubic $\hookrightarrow [100], [010], [001]$
 $[\bar{1}00], [0\bar{1}0], [00\bar{1}]$

Important in TEM diffraction because we “look” down the lattice vectors (“zone axes”)

EPFL Lattice planes

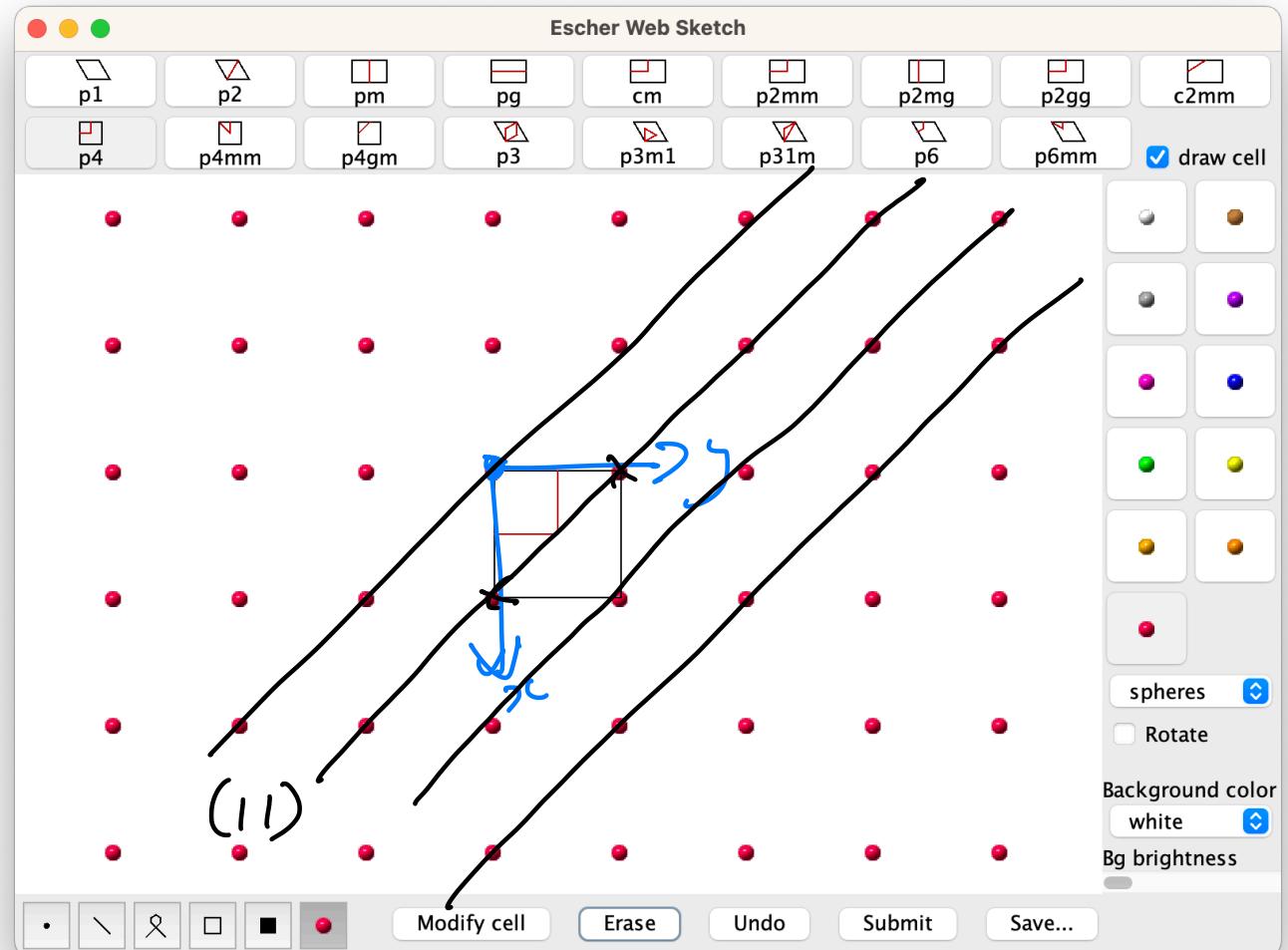
- Lattice plane is a plane which passes through any 3 lattice points which are not in a straight line
- Described using Miller indices $(h \ k \ l)$ where the first plane away from the origin intersects the x , y , z axes at:

a/h on the x axis
 b/k on the y axis
 c/l on the z axis



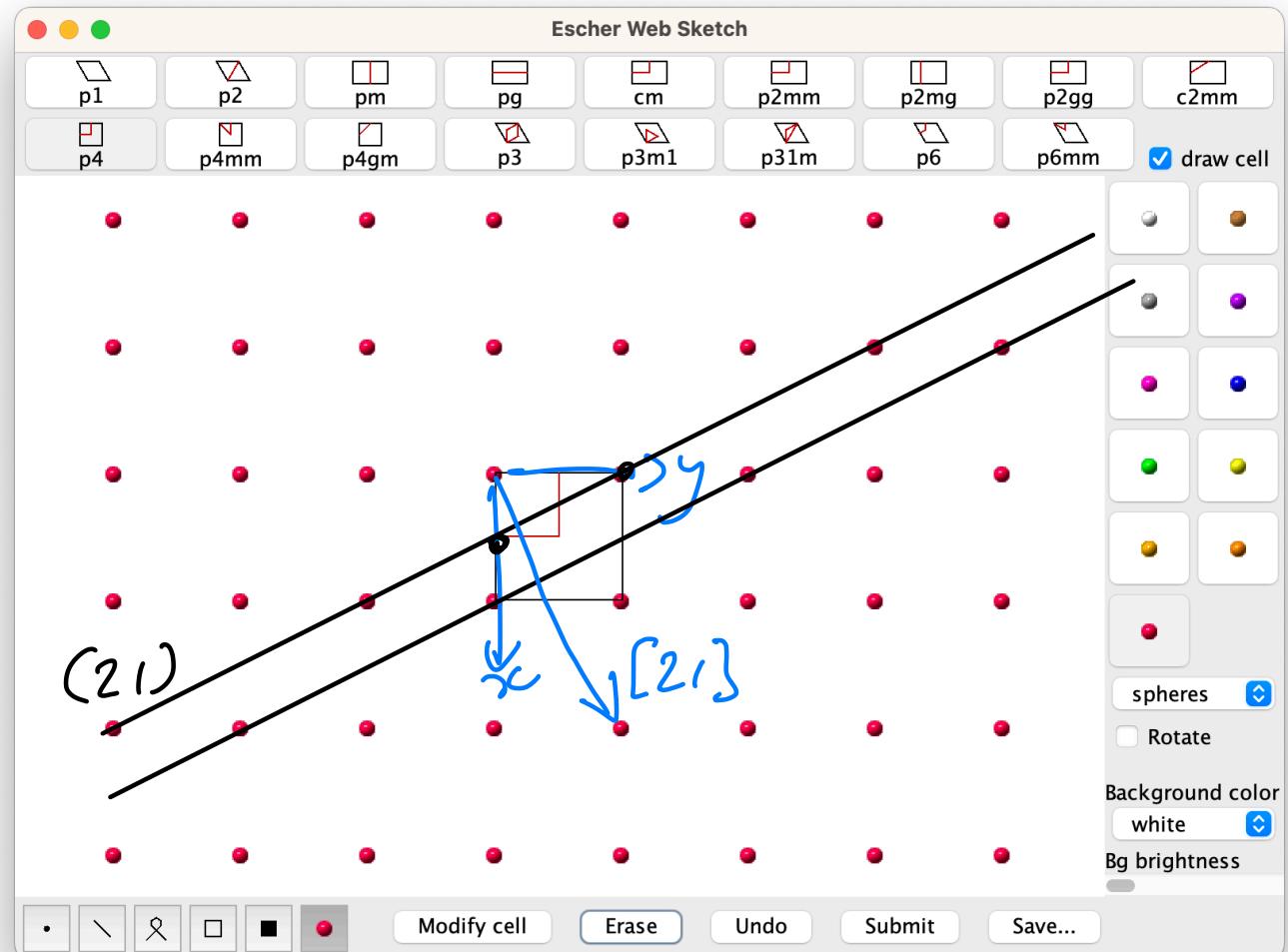
EPFL Lattice “planes” (2D)

- In 2D correspond to lattice *lines*
- Draw the $(1 \ 1)$ lattice line:



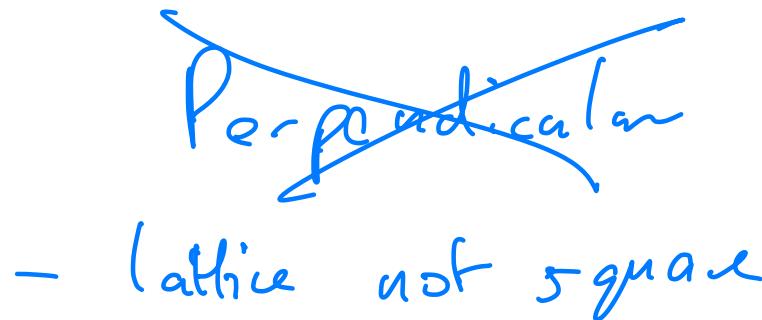
EPFL Lattice “planes” (2D)

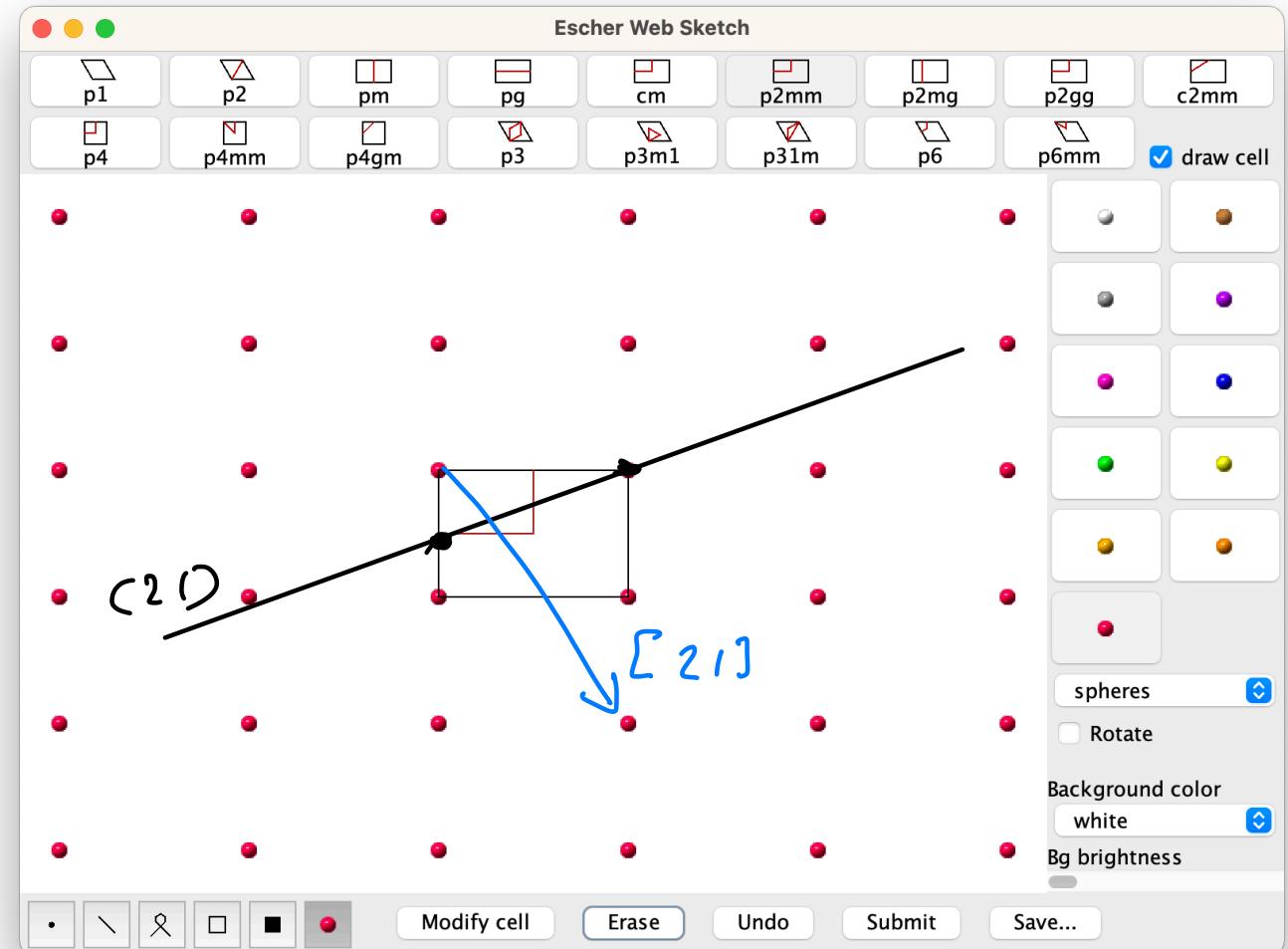
- In 2D correspond to lattice *lines*
- Draw the $(2\ 1)$ lattice line and $[2\ 1]$ lattice vector



EPFL Lattice “planes” (2D)

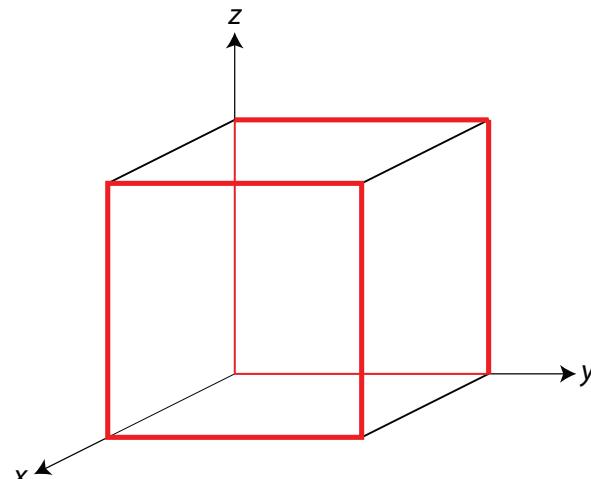
- In 2D correspond to lattice *lines*
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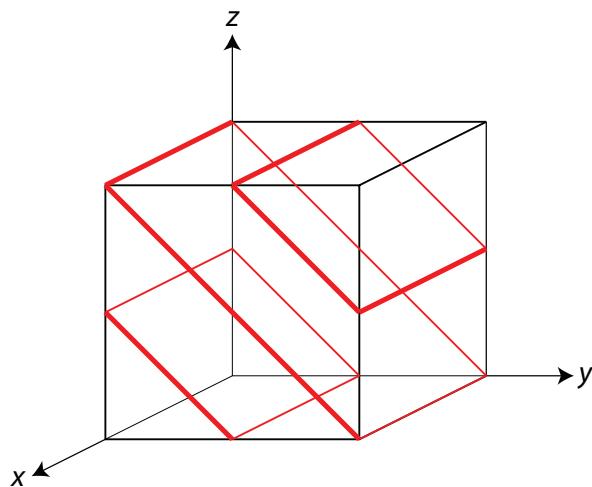


EPFL Lattice planes (3D)

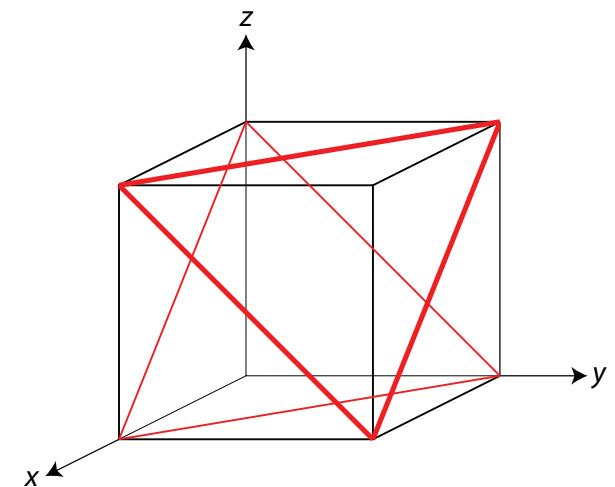
- Some examples to “solve”:



(100)



(022)

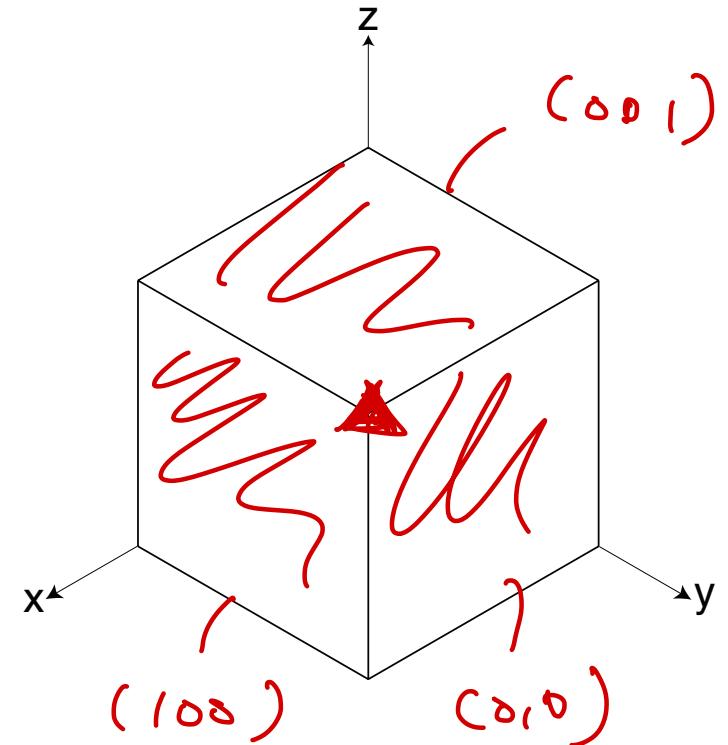


(111)

EPFL Lattice planes and symmetry

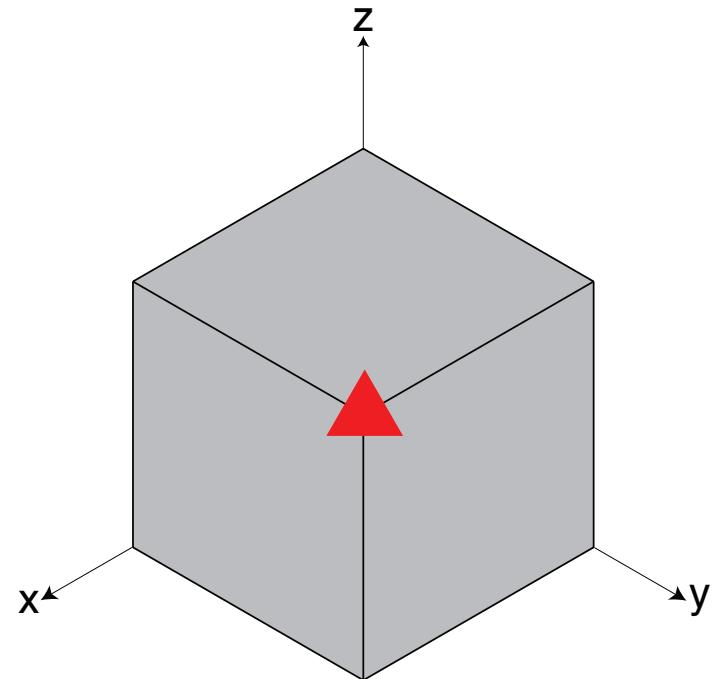
- Families of lattice planes are equivalent lattice planes related to each other by the crystal symmetry
- E.g. in cubic lattices the 3-fold rotation axis on the $[1\ 1\ 1]$ body diagonal relates the planes $(1\ 0\ 0), (0\ 1\ 0), (0\ 0\ 1)$:

$$(1\ 0\ 0), (0\ 1\ 0), (0\ 0\ 1) \xrightarrow{\quad} \{100\}$$



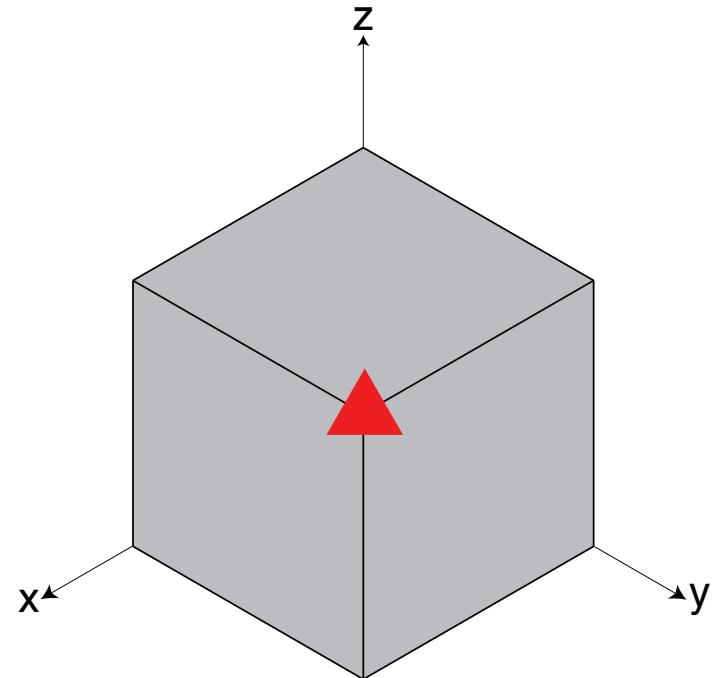
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EPFL Lattice planes and symmetry

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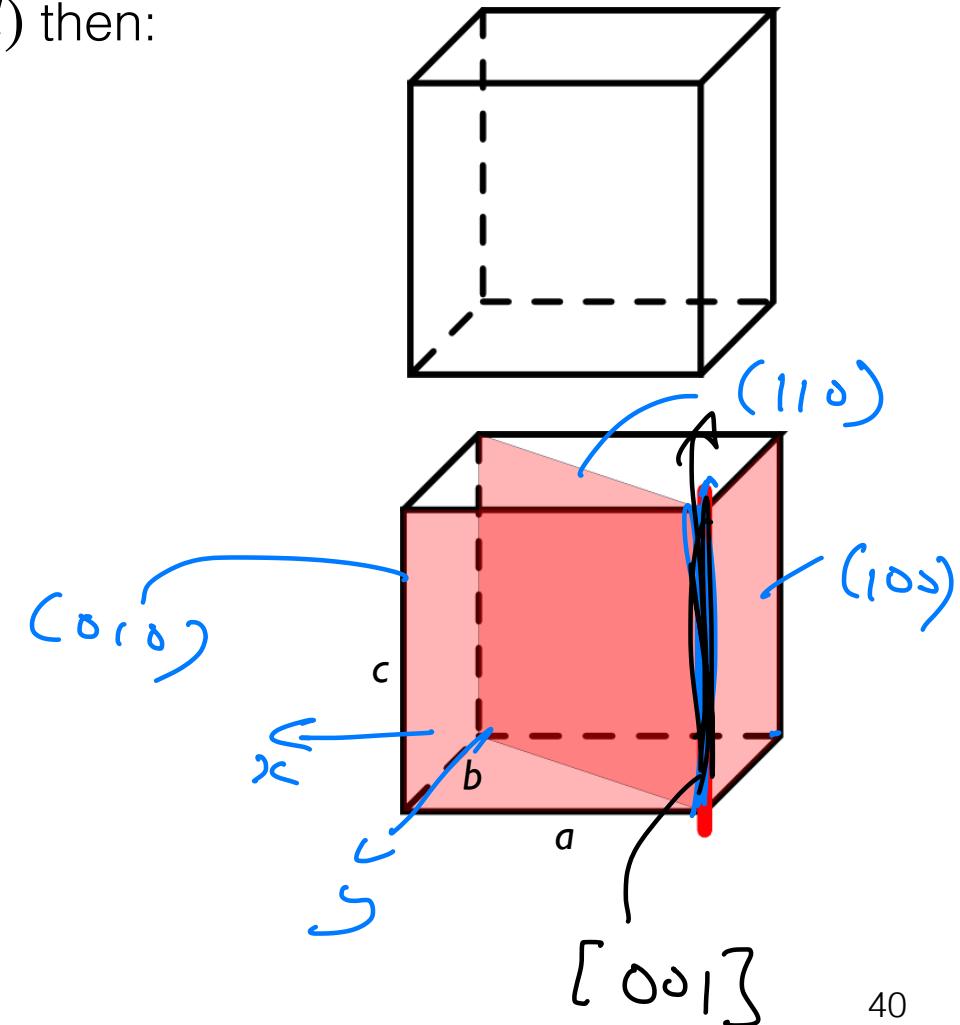


Family of planes given by curly brackets, e.g. for cubic:
 $\{1\ 0\ 0\} = (1\ 0\ 0), (0\ 1\ 0), (0\ 0\ 1), (\bar{1}\ 0\ 0), (0\ \bar{1}\ 0), (0\ 0\ \bar{1})$

EPFL Weiss Zone Law

If the lattice vector $[U \ V \ W]$ lies in the plane $(h \ k \ l)$ then:

$$hU + kV + lW = 0$$



EPFL Weiss Zone Law

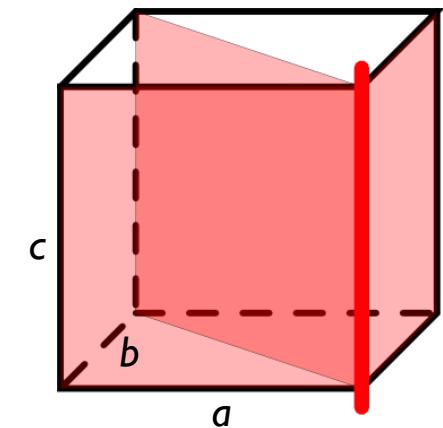
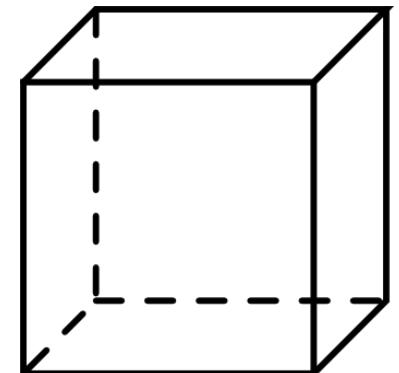
If the lattice vector $[U \ V \ W]$ lies in the plane $(h \ k \ l)$ then:

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Electron diffraction:

Electron beam oriented parallel to lattice vector called the **zone axis**

Diffracting planes must be parallel to electron beam
– **therefore they obey the Weiss Zone law***



EPFL Weiss Zone Law

If the lattice vector $[U \ V \ W]$ lies in the plane $(h \ k \ l)$ then:

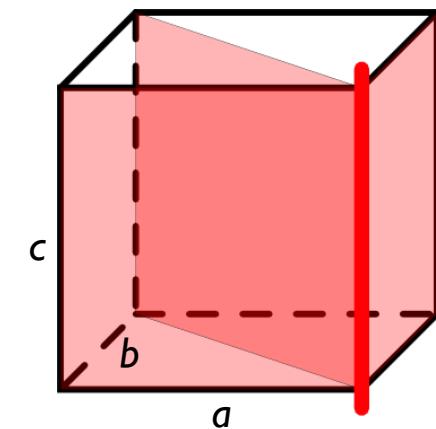
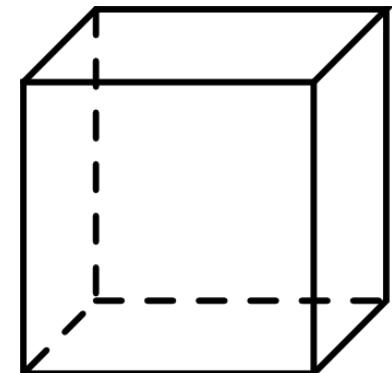
$$hU + kV + lW = 0$$

Electron diffraction:

Electron beam oriented parallel to lattice vector called the **zone axis**

Diffracting planes must be parallel to electron beam
– **therefore they obey the Weiss Zone law***

(*at least for zero-order Laue zone)



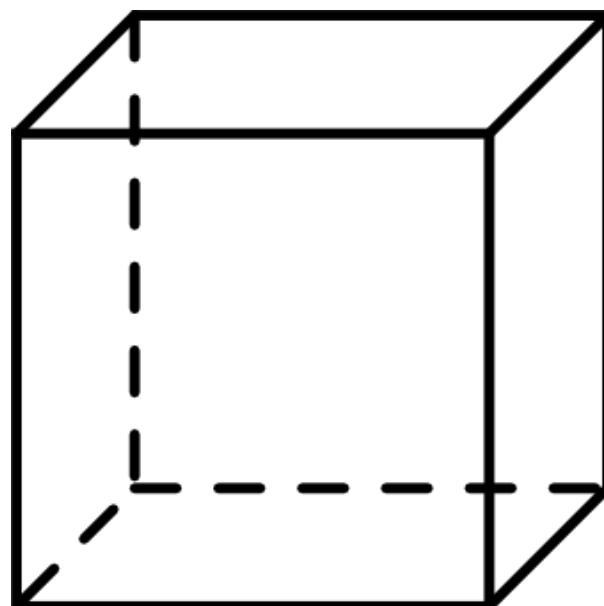
EPFL Weiss Zone Law

- To obtain the zone axis defined by two non-parallel planes $(h_1 k_1 l_1)$ and $(h_2 k_2 l_2)$ calculate the cross-product:

$$U = k_1 l_2 - k_2 l_1$$

$$V = l_1 h_2 - l_2 h_1$$

$$W = h_1 k_2 - h_2 k_1$$



e.g. for planes $(1 0 0)$ and $(0 1 0)$:

$$\begin{pmatrix} 1 \\ 0 \\ 0 \end{pmatrix} \otimes \begin{pmatrix} 0 \\ 1 \\ 0 \end{pmatrix} = \begin{pmatrix} 0 \\ 0 \\ 1 \end{pmatrix}$$

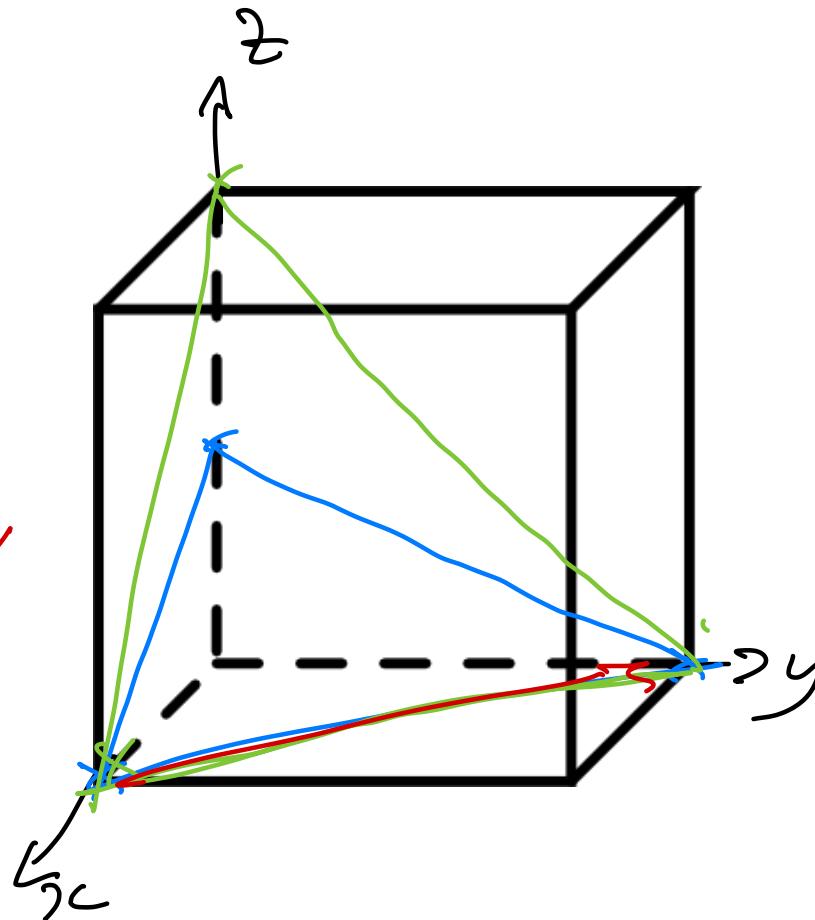
They define the $[0 0 1]$ zone axis

EPFL Weiss Zone Law

- Draw the $(1\ 1\ 2)$ and $(1\ 1\ 1)$ planes
- What zone axis do they define?

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

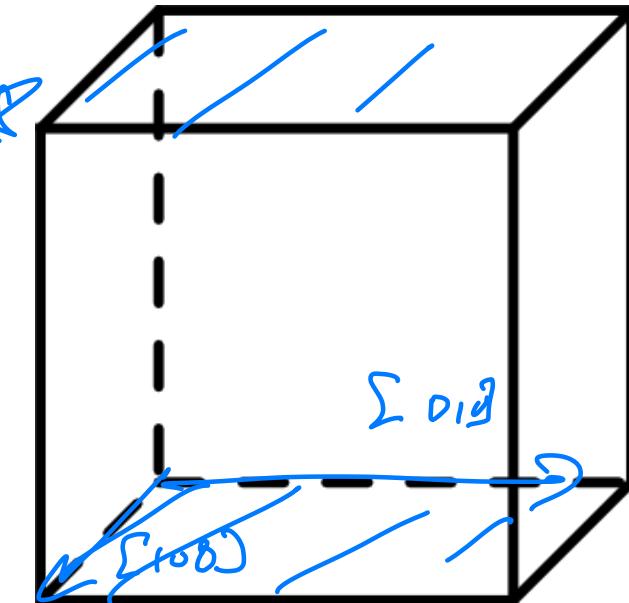
$$h_u + h_v + h_w = 0 \checkmark$$



EPFL Lattice vectors defining planes

- Similarly, two non-parallel lattice vectors $[U_1 \ V_1 \ W_1]$ and $[U_2 \ V_2 \ W_2]$ define a lattice plane as their cross-product
- e.g. the plane containing the lattice vectors $[1 \ 0 \ 0]$ and $[0 \ 1 \ 0]$ is:

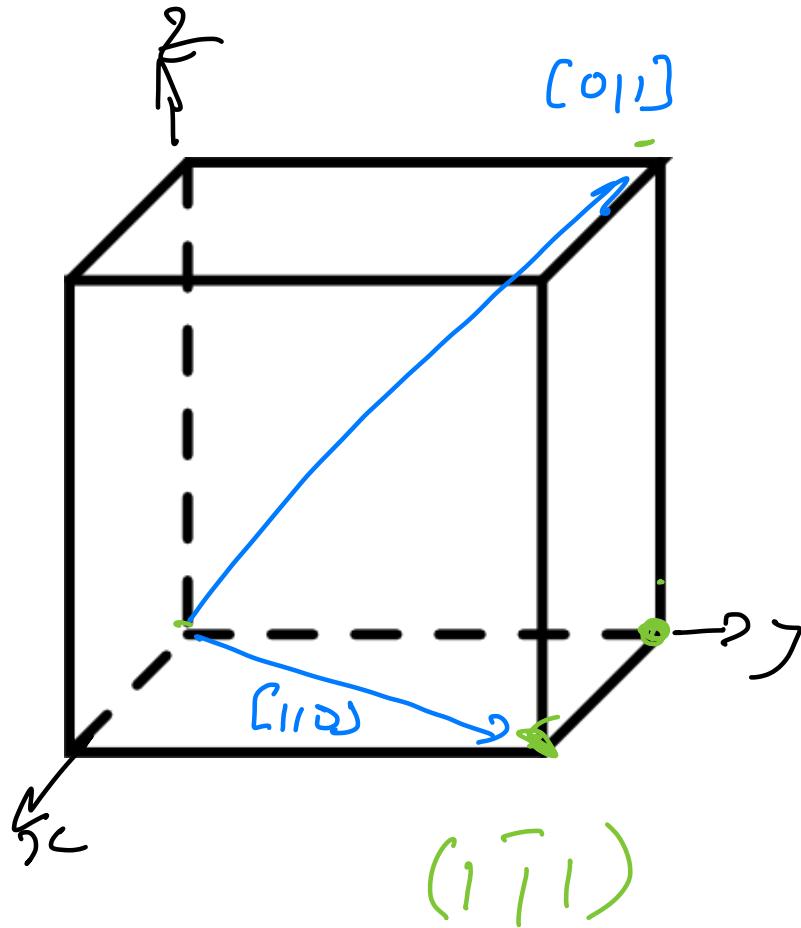
$(0 \ 0 \ 1)$

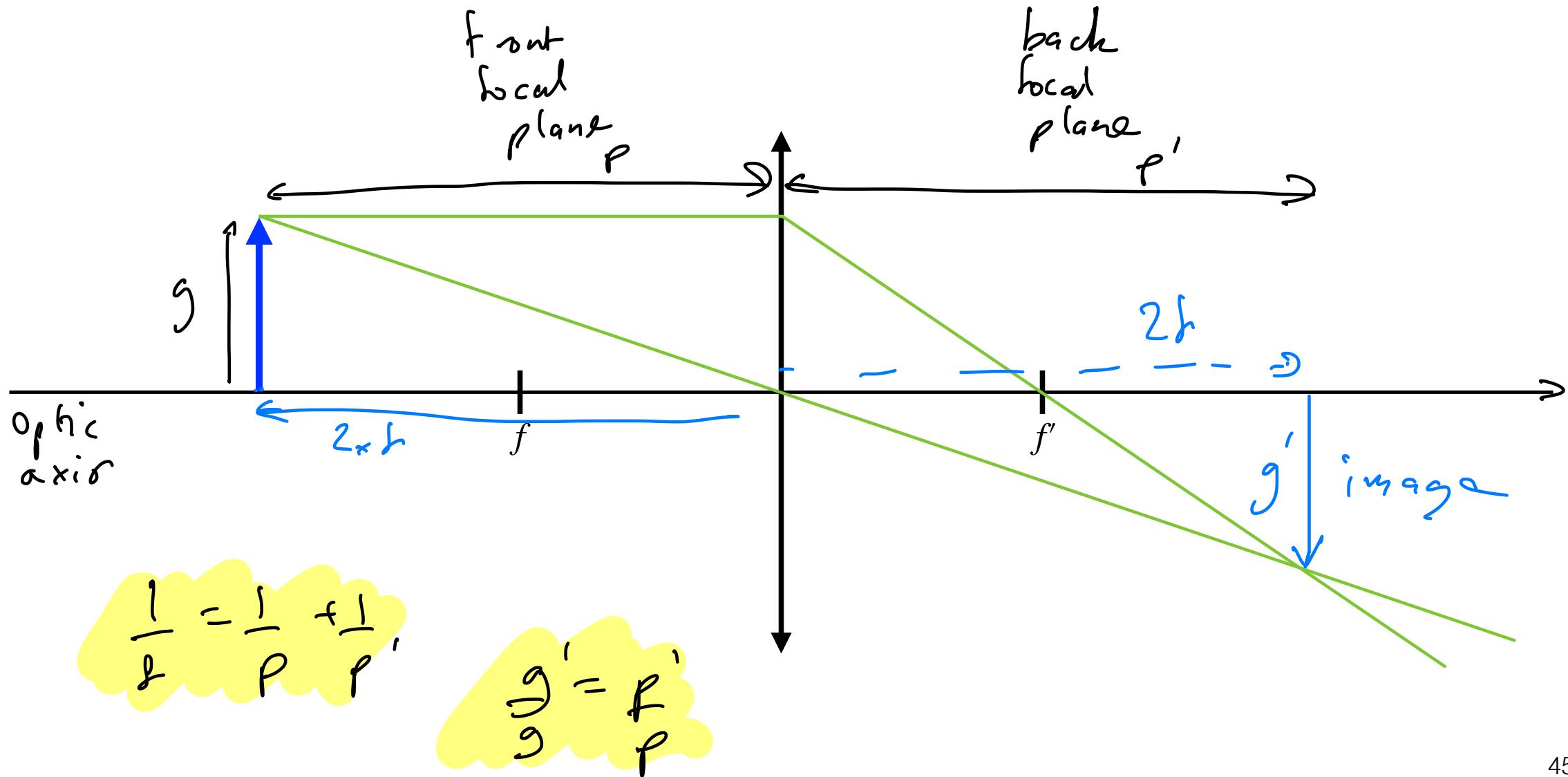


- Identify the plane containing the lattice vectors:

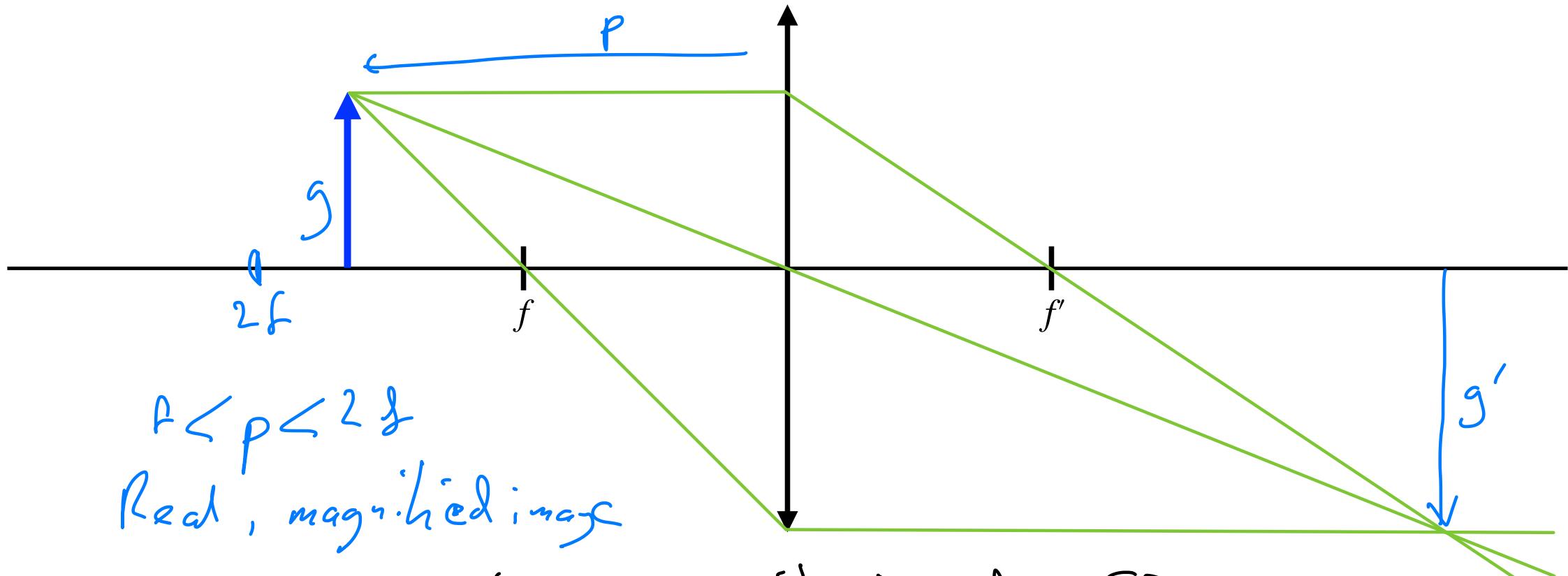
[1 1 0] and [0 1 1]

$$(1\overline{1}1)$$



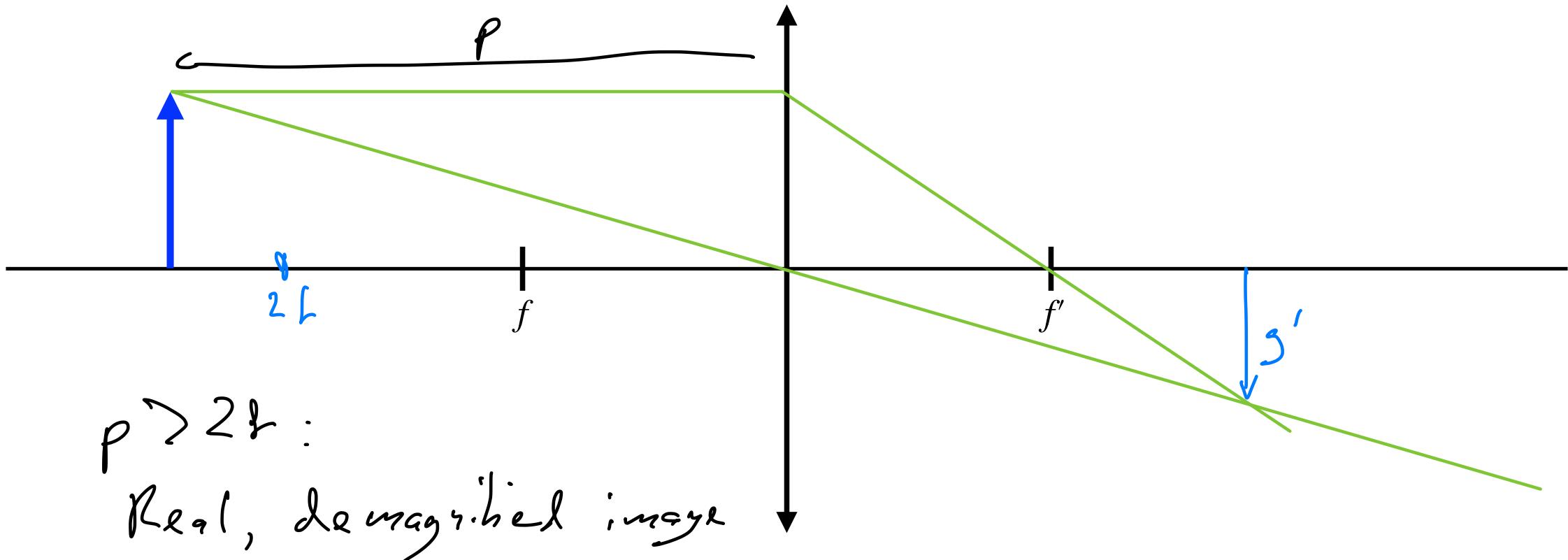


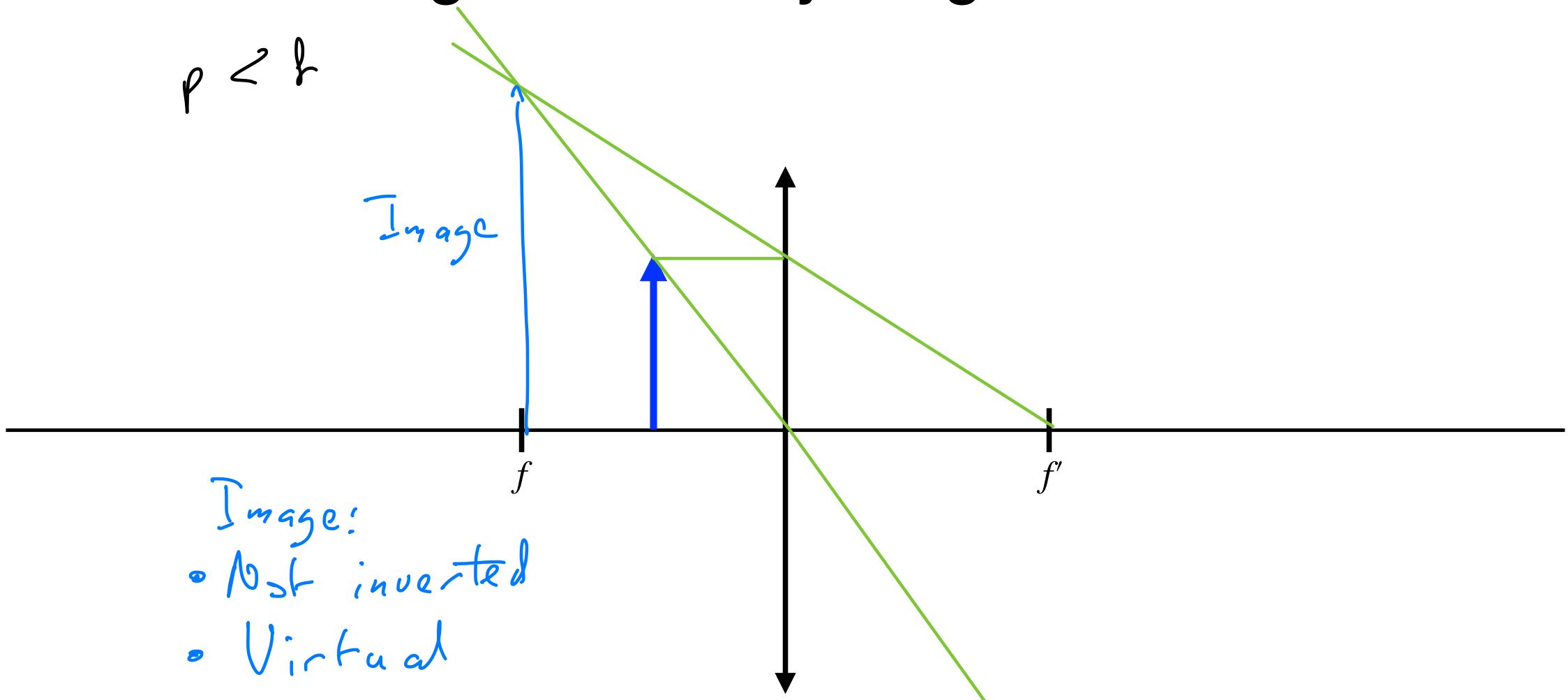
EPFL Convergent lens ray diagram



$f < p < 2f$
Real, magnified image

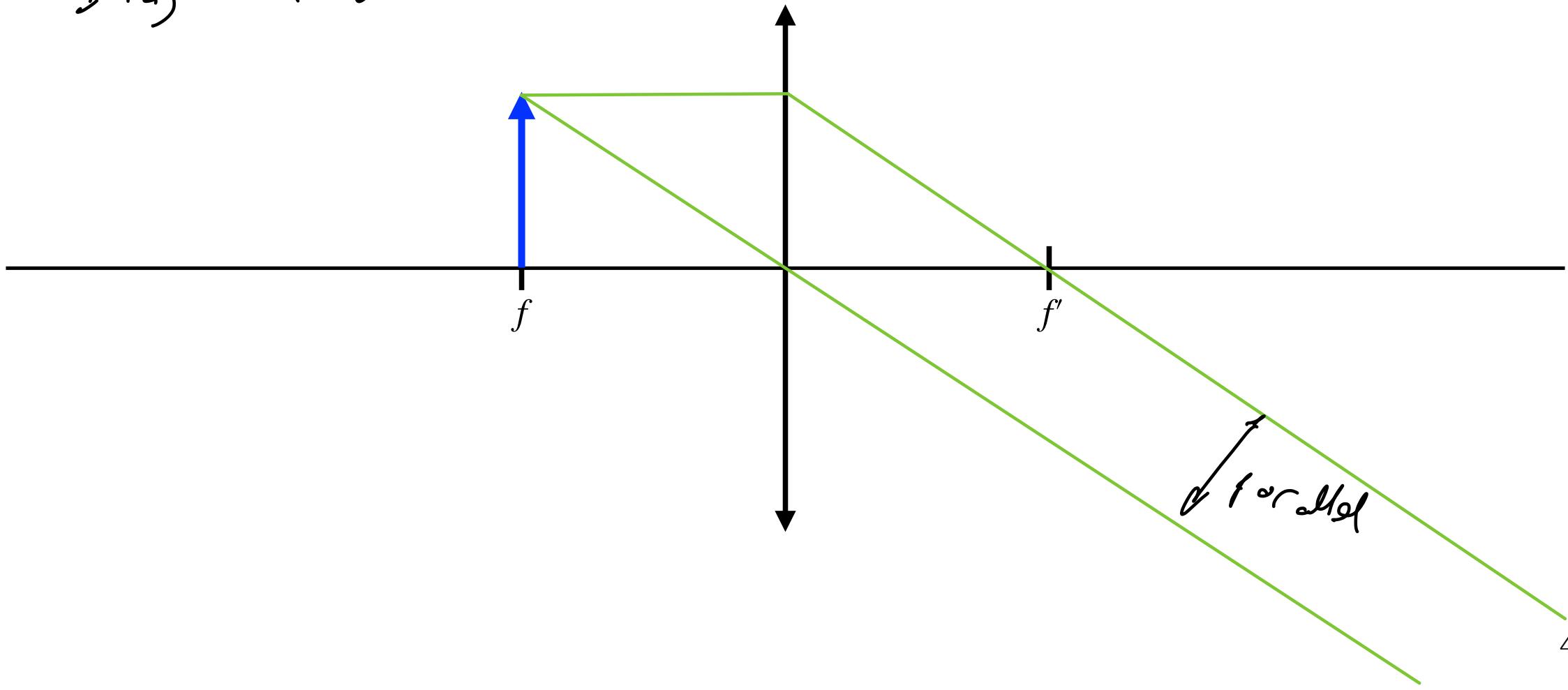
TEM objective lens: magnification of $\sim 50x$





EPFL Convergent lens ray diagram

It object is at f
Image at ∞ !



EPFL Resources

- Transmission Electron Microscopy: A Textbook for Materials Science
Williams & Carter
<https://link.springer.com/book/10.1007/978-0-387-76501-3>
- Transmission Electron Microscopy and Diffractometry of Materials
Fultz & Howe
<https://link.springer.com/book/10.1007/978-3-642-29761-8>
- Transmission Electron Microscopy: Physics of Image Formation
Reimer and Kohl
<https://link.springer.com/book/10.1007/978-0-387-40093-8>
- The basics of crystallography and diffraction
Hammond
<https://academic.oup.com/book/43705>

