Week 08

Diffraction & Crystallography

	True	False
1. A systematic absence is a diffraction peak with zero intensity, where one would expect to see a peak based on the prediction from Bragg's law, as a result of the internal symmetry of the basis of the crystal.	☐	☐
2. Laue diffraction uses polychromatic radiation to investigate powder samples	☐	☐
3. Powder diffraction patterns display hexagonal symmetry for crystals with hexagonal crystal structure	☐	☐
4. A systematic absence is a diffraction peak with zero intensity, where one would expect to see a peak based on the prediction from Bragg's law, as a result of the internal symmetry of the basis of the crystal.	☐	☐

Exercise 1

Answer these questions by true or false:

Exercise 2:

Select the correct answer(s) (more than one answer can be correct)

1. X-ray powder diffraction is…
a. A scattering technique
b. Involves elastic processes
c. Involves inelastic and elastic processes
d. Can only be performed at a synchrotron

2. What is a structure factor in crystallography
a. The atomic basis of a crystal
b. The vector sum of the atomic scattering factors from each atom that makes up the basis, when the crystal satisfies Bragg's law. The angles between the vectors are determined by the phases of the incident radiation that they experience

3. What is an atomic form factor

a. It is the Fourier transform of electron density distribution

b. Describes the scattering amplitude of an atom as a function of scattering angle and X-ray energy

c. Has its minimum in the forward direction

4. The Ewald sphere…

a. Is a geometrical construct facilitating the determination of whether a crystal will produce one or more diffraction spot signals

b. Has a radius equal the scattering vector q

c. Always has the direct beam, equal to (000) Bragg peak, on its surface

5. Textured samples…

a. Have a preferred orientation of certain crystallographic directions

b. Show only defined peaks in the 2D scattering pattern

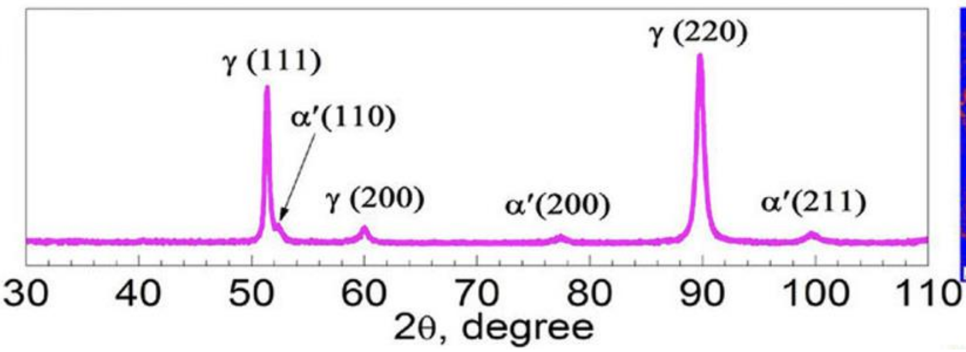
c. Fibre-textured materials are used to make textiles

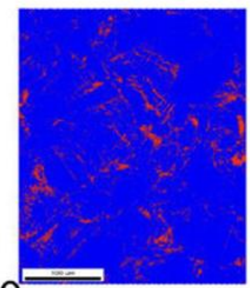
d. Show defined peaks in the 1D scattering curve (Intensity vs. scattering angle/scattering vector)

Exercise 3: Crystal phase identification

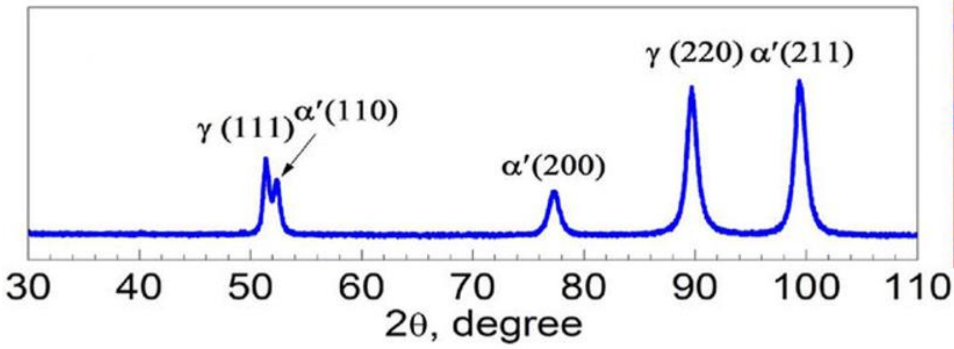
Steel can contain different amounts of Ferrite (α-phase) and Austenite (γ-phase).

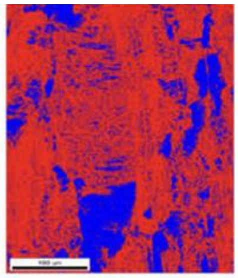
Sample A:





Sample B:





What is the crystallographic structure of Ferrite and Austenite? And which one is the blue phase?

Exercise 4: Powder diffraction

A copper crystal powder (face-centered cubic, a = 3.596 Å) is measured with 16 keV radiation.

a) Determine the scattering angle of the fourth reflection of copper (face-centered cubic, a = 3.596 Å) in a powder diffraction pattern.

b) Where does the peak appear in a powder diffraction plot with Intensity vs. scattering vector q

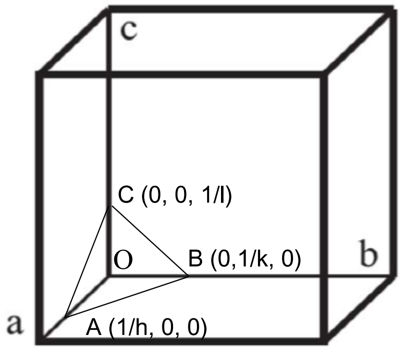
c) What is the scattering angle and scattering vector q if measured with 10 keV instead?

d) What is the scattering vector q if measured with thermal neutrons?

e) Assume that the powder is crystallographically perfect and unstrained nanoparticles and the diffraction pattern I vs. 2θ is corrected for instrument broadening. The (311) peak measured at 16 keV has a width of 0.25°. What is the dimension of the nanoparticles?

EXTRA EXERCISES TO PART I: CRYSTALLOGRAPHY

Exercise 5 : Family of crystal planes



We consider a family of plans $\{hkl\}$ in a primitive cubic lattice structure of edge a, with the basis (O, **a**, **b**, **c**) as shown in the schematic. One of these planes is the one intercepting the points A, B and C on the schematic. To simplify the visualization, we consider without loss of generality that h, k and l are strictly positive integers (as in the schematic) that are co-prime (greatest common divisor is 1)

5a. The plan defined by the points A, B and C is indeed a lattice plane, with equation in the orthonormal basis:

$$\mathcal{P}_1^{(hkl)} = \left\{ M = \begin{pmatrix} x \\ y \\ z \end{pmatrix}, hx + ky + lz = a \right\}$$

(i) Is the plane parallel to $\mathcal{P}_1^{(hkl)}$ and passing through the lattice point defined as the origin O, a lattice plane ?

(ii) Show that its equation is given by: $\mathcal{P}_0^{(hkl)} = \{(x, y, z) \in \mathbb{R}^3, hx + ky + lz = 0\}$

5b. In class, we calculated the distance between the origin (the plane $\mathcal{P}_0^{(hkl)}$) and $\mathcal{P}_1^{(hkl)}$, see week 4, slide 26. However, we didn't explicitly verify that there is no plane in the $\{hkl\}$ family that is in between these two planes, i.e. that intercepts the axis **a**, **b** and **c** closer to the origin O at points A'(a/H,*0*,*0*), B'(*0*,a/K,*0*) and C' *0*,*0*,a/L) with (H,K,L) integers and $H \geq h, K \geq k, L \geq l$. In other words, we translate $\mathcal{P}_0^{(hkl)}$along its normal $[hkl]$, and see if we intercept a crystal plan of the $\{hkl\}$ family before $\mathcal{P}_1^{(hkl)}$:

(i) Show that one equation of this plan containing A', B' and C', is given by:

$$\mathcal{P}^{(HKL)} = \left\{ (x, y, z) \in \mathbb{R}^3, hx + ky + lz = \frac{ah}{H} \right\}$$

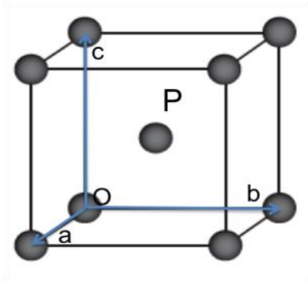
(ii) Since we supposed that $\mathcal{P}^{(HKL)}$ is a crystal plane, show that

$$\exists (n_1, n_2, n_3) \in \mathbb{N}^3, H \times (hn_1 + kn_2 + ln_3) = h$$

(iii) Conclude that necessarily, $H = h$ and so that $\mathcal{P}^{(HKL)} = \mathcal{P}_1^{(hkl)}$, and there is no $\{hkl\}$ plane between $\mathcal{P}_0^{(hkl)}$ and $\mathcal{P}_1^{(hkl)}$ so what we calculated was indeed the distance between two closest planes.

Exercise 6 : Primitive cell of the BCC structure

Consider the body centered-cubic unit cell shown to the right with the origin marked as O and the three orthogonal axis of length the cube edge a.



6a.Verify that the formula for the volume of the cell defined by the basis vectors, i.e. $V = \boldsymbol{a}.(\boldsymbol{b} \times \boldsymbol{c})$, gives $V = a^3$.

6b. In the basis (O, **a**, **b**, **c**), what are the coordinate of the point P at the center du cube ? Is this basis a Bravais lattice for the BCC cubic structure ?

6c. We consider the same origin O and define three new vectors **a'**, **b'** and **c'** such that :

$$\vec{a}' = \frac{1}{2}\left(-\vec{a} + \vec{b} + \vec{c}\right) ;\ \vec{b}' = \frac{1}{2}\left(\vec{a} - \vec{b} + \vec{c}\right) ; \vec{c}' = \frac{1}{2}\left(\vec{a} + \vec{b} - \vec{c}\right)$$

(i) Express the vectors **a**, **b** and **c** as a function of **a'**, **b'** and **c'**.

(ii) Show that for all point M of the BCC lattice, one can find relative integers n, p and q, such that:

$\overrightarrow{OM} = \frac{n}{2}\vec{a} + \frac{p}{2}\vec{b} + \frac{q}{2}\vec{c}$, with *(n,p,q)* either all even numbers, or all odd numbers.

(iii) Using i), express the **OM** vector in the (O, **a'**, **b'**, **c'**) basis.

(iv) Conclude that this basis is indeed a primitive basis for the BCC structure.

6d. Using the same formula as in 6a, and the expression found in of the primitive lattice vectors given in 6c, calculate the volume occupy by the BCC primitive cell.

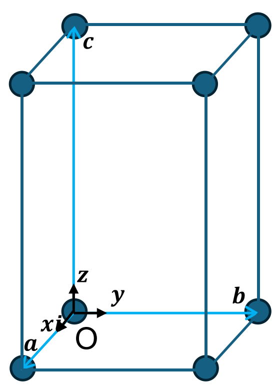
6e. How many motifs do the conventional and primitive cell contain ? Justify your answer relating to the respective volume of these two cells.

6f. Following the same approach, demonstrate that the Bravais Lattice for the face-centered cubic lattice is:

$$\vec{a}' = \frac{1}{2}\left(\vec{b} + \vec{c}\right) ; \vec{b}' = \frac{1}{2}(\vec{a} + \vec{c}) ; \vec{c}' = \frac{1}{2}\left(\vec{a} + \vec{b}\right)$$

Exercise 7: Rotation and mirror symmetry

We consider the tetragonal primitive structure as shown to the right (as we have seen in exercise 2 of week 5). We represent the origin, the orthonormal basis $\mathcal{B}_{(O,x,y,z)}$, and the orthogonal basis $\mathcal{B}_{(O,a,b,c)}$, with: $\boldsymbol{a} = a\boldsymbol{x},\ \boldsymbol{b} = a\boldsymbol{y}, \boldsymbol{c} = c\boldsymbol{z}$, and $a \neq c$.



7a.

(i) What is the new vector $\boldsymbol{c}' = \mathcal{R}_{\left(b,\frac{\pi}{2}\right)}(\boldsymbol{c})$ under a rotation around the axis **b** and of angle $\frac{\pi}{2}$?

(ii) Find a condition on a and c so that $\boldsymbol{c}'$ belongs to the Bravais Lattice.

7b.

(i) What angle of rotation around the **b** axis leaves the lattice invariant ?

(ii) How about around the **c** axis?

7c.

(i) Is the plane (110) a plane of symmetry for the tetragonal structure?

(ii) How about the plane (101) ?