7 Characterization of (bio-)materials

7.1 Electron clouds around a hot filament can be extracted by the application of an electrostatic field. The applied high voltage (usually kV) determines the energy of the electrons.

Determine the velocity of electrons accelerated with a voltage of 40 kV, which is a typical value of μCT for the characterization of bone.

Electrons have a kinetic energy of:

$$mv^2/2 = eU,$$

where:

- U= 40 kV - applied voltage;

- m=9,10938291(40)·10^{-31} kg - mass of electron;

- e=-1,602176565(35) ·10^{-19} kl - charge of electron;

- c=3 · 10^8 m/s - speed of light;

- $v = \sqrt{\frac{2eU}{m}} = 0.396 \cdot c$

Why such a process is an effective way to produce X-rays?

Both of these X-ray production processes are significantly inefficient, with a production efficiency of only about one percent.

But, in comparison to electrons, X-rays have smaller interaction potential, so they can escape from the bulk and not only from surface area.

What is the maximal energy of the generated (X-ray) photons?

The maximum energy of the produced X-ray photon is limited by the energy of the incident electron, which have the kinetic energy of $mV^2/2 = eU$, U is equal to the voltage on the tube, so an 40 kV tube cannot create X-rays with an energy greater than 40 keV.

$\implies U_{max} = 40 \ kV$

$hc = E_{BE} + E_{kin}$, where

- E_{BE} - binding electrons energy;

- h - Plank's constant;

It should be discussed qualitatively:

How the X-ray flux can be increased? What are the advantages and disadvantages for μCT, if the flux is increased?

- Increased voltage $\Longrightarrow$ NO! X-ray flux does not increased;

- Current in the cathode (filament) increased $\Longrightarrow$ X-ray flux increased $\Longrightarrow$ exposure time shorter, but spatial resolution worse;

- Increase exposure time$\Longrightarrow$ X-ray flux increased $\Longrightarrow$ BUT radiation increased.

7.2 The object (2x2 matrix) is given by the following projections (values arbitrary units).

		5
		5
3	7	6

Please determine the values of the matrix in an iterative way. For the first cycle, one may assume constant values in each voxel to be determined from the average of rows and columns. The next step might be the incorporation of the diagonal. The differences between the estimate and the projection value should be the basis of the next cycle. Please compare the actual values of the object (you have to find it really simple) with the ones of the second cycle. Please demonstrate that the finite number of iteration steps results in the actual values for this simple example.

(a) First step:

3/2	7/2
3/2	7/2

Secend step:

+0.5	-
3/2	7/2
3/2	7/2
-	+0.5

If we compare the actual values of the object with the ones of the second cycle and calculate relative error:

25%	16%
50%	12.5%

Third step - actual values:

2	3
1	4

(b) First step:

5/2	5/2
5/2	5/2

Second step:

-1	+1
5/2	5/2
5/2	5/2
-1	+1

Third step:

+0.5	-
1.5	3.5
1.5	3.5
-	+0.5

Forth step - actual values:

2	3
1	4

This algorithm is used in computer tomography.

X-ray computed tomography, also Computed tomography (CT), is a medical imaging method employing tomography created by computer processing.

7.3   **Draw a linear crystal lattice with lattice constant a in real and reciprocal space. Add the vectors, which have double amount (cp. surface reconstructions). Add vectors, which are 100 times larger as the lattice constant, as typical value for atomic terrace widths. Do you understand, the term reciprocal? Discuss the consequences for quantitative diffraction studies.**

- Reciprocal space is also called Fourier space, k- space, or momentum space in contrast to real space or direct space.

- The reciprocal space lattice is a set of imaginary points constructed in such a way that the direction of a vector from one point to another coincides with the direction of a normal to the real space planes and the separation of those points (absolute value of the vector) is equal to the reciprocal of the real interplanar distance.

- The relationship between the period and frequency is similar to that of the reciprocal and the direct lattice. Therefore Fourier transformation is used in the studies of the real lattice to yield the reciprocal lattice in the same fashion as with the studies of any other periodic function, therefore the reciprocal space is also called Fourier space.

- For a periodic object, the diffraction pattern is equivalent to the reciprocal lattice of the object.
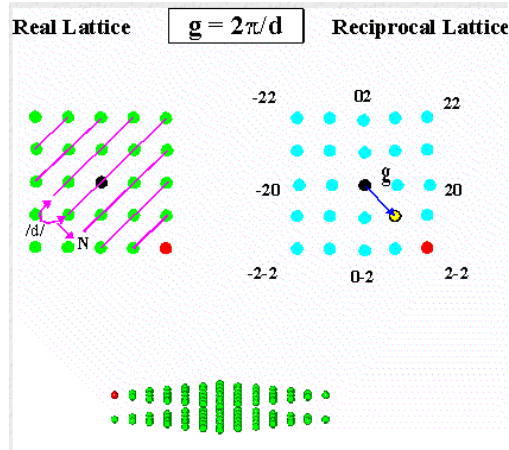

Figure 1: The real lattice is described at the left, the reciprocal lattice is described at the right. g is the reciprocal lattice vector. The absolute value of g is equal to $2\pi/d$ and the direction is that of the normal N to the appropriate set of parallel atomic planes of the real lattice separated by distance d. Two parallel planes of the three-dimensional lattice are shown below.
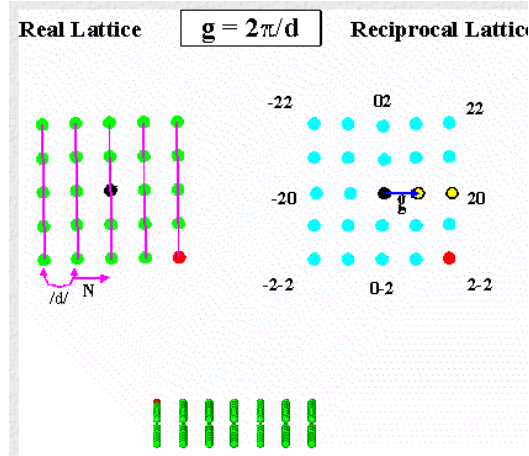

Figure 2: The reciprocal space.

Two-dimensional array of equally spaced atoms consequently produces scattered waves which reinforce along the lines of the cross section of two sets of corresponding cones oriented along the coordinate axes. In three-dimensional case the set of the cones oriented along the third coordinate axes causes the reinforcement of scattered waves (constructive interference) to occur at certain locations. Those locations are the points of cross section of all three sets of cones, oriented along three coordinate axes of the crystal.

W. L. Bragg made the assumption that the sheets of planes of atoms in the crystal behave like perfect reflectors for X-rays, so that interference was caused by multiple reflections from these planes in the same manner as a stack of glass plates produces interference by multiple reflection. Let us consider a set of partially reflecting planes spaced at an interval d.
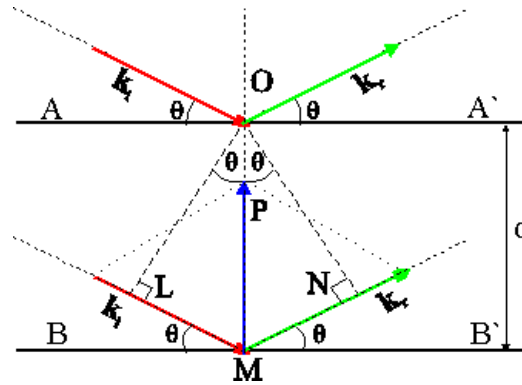

Figure 3:

We wish to know the path difference between two reflected rays:

$$D = LM + MN = 2d \sin\theta$$

In order to get constructive interference, D must be equal to $n\lambda$ (n is the called the order of interference and is equal to $0, \pm1, \pm2$ etc.). Therefore:

$$n\lambda = 2d \sin\theta$$

This is Bragg Law.

On diffraction, part of the energy in the incident beam with wave-vector k_i, will be scattered into a beam marked k_r in the figure. How does k_i differ from k_r? k_r is only the component of k_i, perpendicular to the planes AA'

and BB' which is changed. The change of wave-vector upon diffraction is perpendicular to the planes and its magnitude for the diffraction of the first order is :

$$2(2\pi/\lambda)\sin\theta = 2k\,\sin\theta = 2\pi/d$$

Where wave-vector k = $2\pi/\lambda$, by definition.

Term $2\pi/d$ is equal to the magnitude of the reciprocal lattice vector. Therefore in the X-ray diffraction experiment where we control the magnitude and the direction of incident wave-vector we can directly map the reciprocal lattice of the crystal.

The Ewald Construction

The X-ray diffraction experiment is carried out so that the wavelength and the direction for the incident X-ray beam are known. This information may be put into the reciprocal lattice.
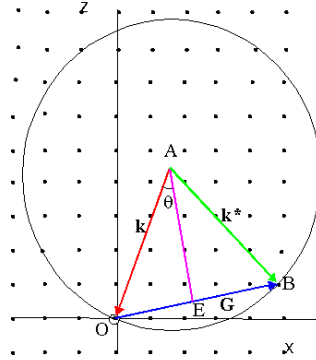

Figure 4:

(a) Chose a point according to the orientation of the specimen with respect to the incident beam.

(b) Draw a vector AO in the incident direction of length $2\pi/\lambda$ terminating at the origin.

(c) Construct a circle of radius $2\pi/\lambda$ with center at A. Note whether this circle passes through any point of the reciprocal lattice.

7

(d) Draw a vector AB to the point of the intersection.

(e) Draw a vector OB to the point of the intersection.

(f) Draw a line AE perpendicular to OB.

(g) Complete the construction to all the intersection points in the same fashion.

We have the following facts:

- Since OB ends on a point in the reciprocal lattice, it is normal to some set of lattice planes and is of length $2\pi/d$, the interplanar spacing for the set. By definition, OB $= 2\pi/d$.

- Since AO $= 2\pi/\lambda$, then OE $= 2\pi/\lambda \sin\theta$ and OB $= 2 \cdot (2\pi/\lambda) \sin\theta$.

- Since OB is normal to a lattice plane, AE is the lattice plane, and θ is the angle between the incident wave AO and the lattice plane. It is equal to the angle between the lattice plane AE and reflected wave AB.

- Since $2\pi/d = 2 \times (2\pi/\lambda)\sin\theta$, we obtain $\lambda = 2d\sin\theta$; the Bragg law is satisfied.

Thus, knowing the direction and wavelength of the incident wave, we have been able to determine which plane will diffract it.

If the circle had passed through no points, that would indicate that the particular wavelength in question would not be diffracted by that crystal in that orientation. Further, if the magnitude of the vector $|AO| \prec 1/2(2\pi/a)$, the circle could not pass through any point, showing that X-ray diffraction cannot occur if the wavelength exceeds 2a. We note also that the longer the vector AO (the shorter the wavelength), the greater is the likelihood of the circle's intersecting a point, and hence of diffraction.

The periodic terraces of simple crystalline surfaces result in a broadening of the diffraction spots. Measuring the spot profiles, one can extract periodicities much larger than atomic distance.

7.4 Porosity is an important quantity for scaffolds in tissue engineering and for implants. How the interconnected and total porosity can be determined?

Porosity is a way to characterize material's porous nature, such as its diameter, surface area, etc.

Porosity is the volume fraction of a material that can in empty.

- Total porosity = all pore space expressed as a percentage or a fraction of the materials volume;

- Effective porosity =interconnected porosity=connected pore space. The portion of pores that can be filled from outside.

Several methods have been developed for determining the surface area and the pore size distribution in porous systems. The operations of these different methods generally are based on different physical principles. It should be expected, therefore, that they effectively represent probes of different sizes and, hence, that the pore size ranges in which they are most reliable are necessarily different.
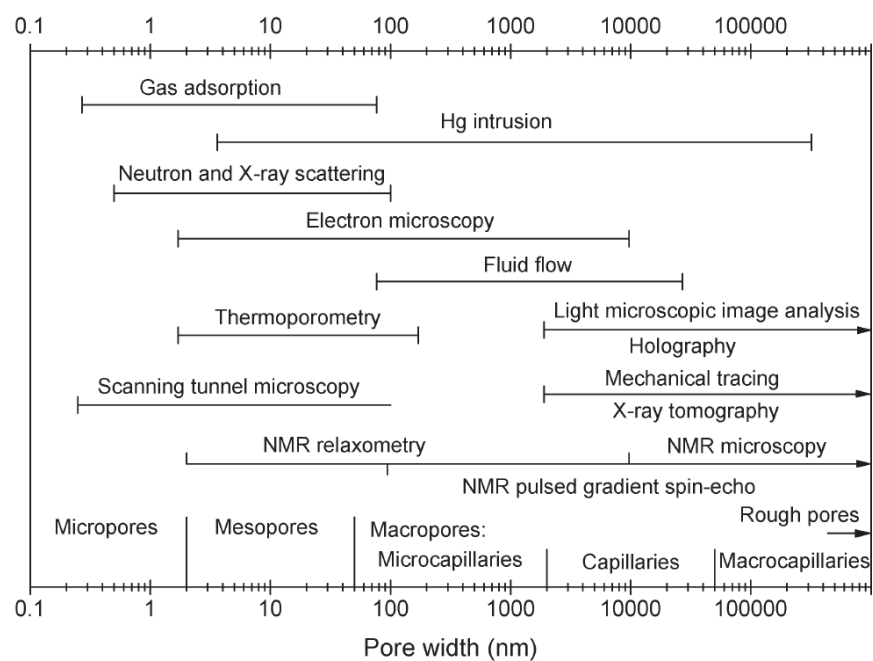
Figure 5: Measuring ranges of methods for pore size determination.

Procedure	Range of pore sizes	Assumption	Advantages	Disadvantages
Xylene and water impregnation	d > 100 nm		Simple method, short time	No pore size distribution, specimens with small diameter
Liquid metal impregnation			True pore network	No measurable pore size distribution, carbon burn-off
Air/He penetration	A few nanometers depending upon gas applied		Very short time method, about 1 min, good reproducibility, no influence on sample	No pore size distribution
N_2 adsorption	d < 50 nm		Correlation with BET surface area	Complicated evaluation program
Mercury intrusion	4 nm < d ≤ 60 µm	Cylindrical pores	Much in use, comparable data, extended range of pore sizes	Danger of breaking pore walls, large pores are filled at atmospheric pressure

Figure 6: Methods for the determination of open porosity.

- **Mercury porosimeter**
  Mercury porosimetry characterizes the scaffolds porosity. The technique involves the intrusion of a non-wetting liquid (mercury) at pressure or vacuum into a material through the use of a porosimeter. One intentionally changes the pressure (vacuum) acting on the scaffold immersed in mercury. The pore size can be determined based on the external pressure needed to force the liquid into a pore against the opposing of the liquid's surface tension. The pressure required to intrude mercury into the pores is inversely proportional to the size of the pores. The method works well for pore diameters well below 10 μm.

- Analysis of topmography data

- Gravimetry (measurement of weight and volume)