

BRAGG SCATTERING I

Diffraction patterns from x-ray scattering
 according to Debeye-Sherrers method

1 Theory in pills

1.1 Bragg's law

X-rays with a wavelength of 1\AA ($10^{-10}m$) can be used to analyze the structure of crystalline materials. This is due to the fact that the crystal structure behaves like a three dimensional diffraction lattice. The radiation is scattered from each and every atom, and in certain directions the radiation from different atoms will interact constructively. Such constructive interaction is called Bragg reflection and is achieved when the Bragg's law

$$2d \sin \theta = n\lambda \quad (1)$$

is satisfied. In Eq.(1) d is the distance between consecutive planes in the set of planes responsible for the reflection in question, θ is the angle between the wavevector of the incident plane wave and the lattice planes, n is an integer ($n = 1, 2, 3, \dots$) linked to the order of the reflection, and λ is the wavelength of the x-rays. The Bragg's law is equivalent to the diffraction condition in the reciprocal space and to the Laue equations (see course literature for details). The crystal planes behave almost like partially transparent mirrors, where Bragg reflection is achieved if radiation that is reflected from successive planes interacts constructively. The glancing angle θ is both the angle of approach and the reflection angle of the reflected radiation relative to the crystal planes. The direction of the radiation is changed with a deflection angle of 2θ (see figure 1). Since the crystal pattern repeats in three dimensions forming a 3D diffraction grating, three integers (denoted h, k, l) are required to describe the order of the diffracted waves. These three integers are the Miller indices. They are written (hkl) and define the orientation of the reflecting crystal plane (see course literature for details). Assume that the lattice is *cubic*, i.e. *sc* (simple cubic), *bcc* (bodycenteredcubic) or *fcc* (facecenteredcubic) with lattice constant a . (Note that the reciprocal lattice is also simple cubic and the lattice constant is $2\pi/a$). For this case, the distance between successive crystal planes within a set of planes with reduced Miller indices (h_r, k_r, l_r) (where $h_r = h/n$, $k_r = k/n$ and $l_r = l/n$) is:

$$d = \frac{a}{\sqrt{h_r^2 + k_r^2 + l_r^2}}. \quad (2)$$

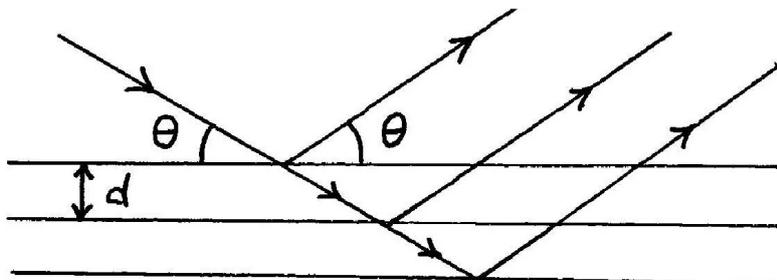


Figure 1: The Geometry of Bragg reflection.

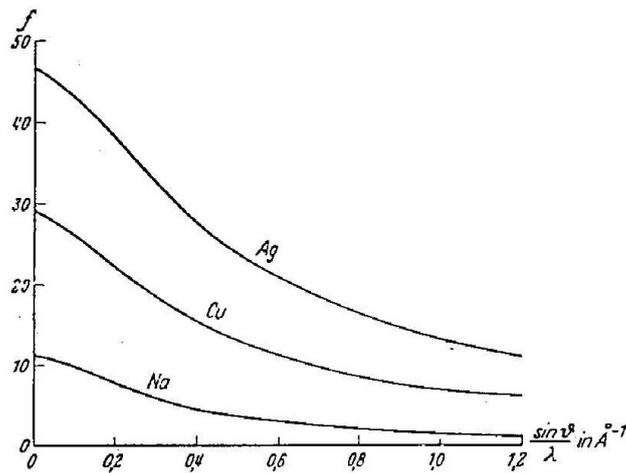


Fig. 42. The atomic scattering factor f as a function of $\sin \theta/\lambda$ in \AA^{-1} for Na, Cu and Ag. Their values for $\theta=0$ are the atomic numbers Z .

Figure 2: (From Encyclopedia of Physics, ed. S. Flügge, Vol. 32).

Combining Eqs. (1) and (2) the Bragg law can be written as

$$\sin \theta = \frac{n\lambda}{2a} \cdot \sqrt{h_r^2 + k_r^2 + l_r^2} \quad (3)$$

that can be rewritten as

$$\sin^2 \theta = \frac{\lambda^2}{4a^2} \cdot (h^2 + k^2 + l^2). \quad (4)$$

1.2 Structure and atomic form factor

The intensity (proportional to the square of the amplitude) of the scattered radiation is proportional to the square of the so called *structural factor* (see course literature for details):

$$S_{hkl} = \sum_j f_j e^{-2\pi i(hx_j + ky_j + lz_j)} \quad (5)$$

where (x_j, y_j, z_j) are the coordinates for the atom j in the unit cell. The coefficient f_j is the scattering power of the atom j , and is called *atomic form factor* for the atom j . It is proportional to the amplitude of the radiation scattered against the atom in question. The factor $\exp[-2\pi i(hx_j + ky_j + lz_j)]$ describes the interaction between waves scattered from different points in the base where the atoms are located. The atomic form factor f_j describes the interaction between waves scattered by different parts of the atom j . For x-rays the atomic form factor (for x-rays) is determined by the distribution of electrons within the atom and depends on both the wavelength of the radiation and the scattering angle, i.e. f_j is actually a function $f_j = f_j(\lambda, \theta)$. In general f_j decreases with increasing θ and decreasing λ (see figure 2). The samples used in this experiment are either of simple fcc or simple bcc type. The word simple means that they contain only one kind of atoms and there is precisely one atom in every primitive lattice point (for example metallic copper, fcc). In other words we only have one atomic form factor $f(\lambda, \theta)$ (losing the subscript j). For these structures the structural factor is:

For **fcc**: $S_{hkl} = 4f$ if (hkl) are all even or odd; otherwise $S_{hkl} = 0$

For **bcc**: $S_{hkl} = 2f$ if $h + k + l = \text{even}$; $S_{hkl} = 0$ if $h + k + l = \text{odd}$.

You should convince yourself that you can get these results from Eq.(5).

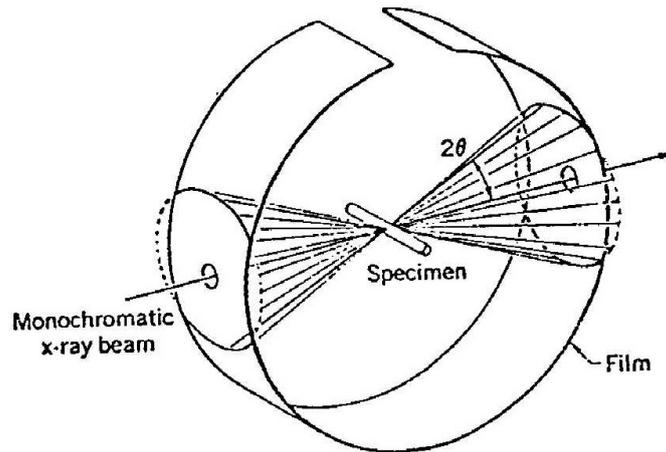


Figure 3: Experimental setup in the Debye-Scherrer method.

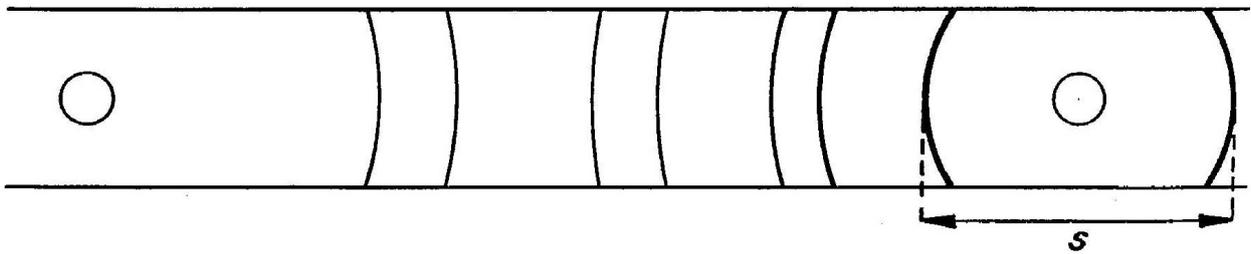


Figure 4: Schematics of a resulting diffraction pattern

2 Experimental method

2.1 Debye-Scherrer method

The spectrum has been measured using the Debye-Scherrer method. The polycrystalline sample (in this case a metal wire) is radiated with x-rays resulting from the bombardment of copper with electrons. The three characteristic wavelengths for copper $K_{\alpha 1}$, $K_{\alpha 2}$ and K_{β} result in three similar spectra (not entirely similar since the relation between θ and λ isn't linear). The sample contains crystals oriented in all directions, and the condition for Bragg reflection with a certain set of miller indices (hkl) , and the corresponding d , n and θ , will be met by many crystals with different orientations relative to the direction of the incoming radiation. The radiation is therefore scattered with the scattering angle 2θ concentrically around the incoming direction and results in a ring (or part of a ring) on the photographic film (see figure 3). The experimental Debye-Scherrer spectrum analyzed in this exercise could be from any of the following metals: Ag (fcc), Al (fcc), Au (fcc), Cu (fcc), W(bcc) and α Fe(bcc). With knowledge of the radius of the film cylinder R_f , the Bragg angle θ of the different reflections can be determined. The relation between the diameter s of the rings and the angles θ_{obs} is:

$$s = R_f \cdot 4\theta_{obs} \quad (6)$$

The values of θ_{obs} have to be corrected for a geometric effect resulting from absorption of x-rays within the sample (see Section 3).

Obs: the characteristic radiation K_{β} has a wavelength $\lambda_{\beta} = 1.39217 \text{ \AA}$. The wavelength of $K_{\alpha 1}$ is $\lambda_{\alpha 1} = 1.54050 \text{ \AA}$ and its intensity is approximately 5 times greater than the intensity of K_{β} . The wavelength of $K_{\alpha 2}$ is $\lambda_{\alpha 2} = 1.54433 \text{ \AA}$ and its intensity is approximately 2 times greater than K_{β} . Due to finite resolution the two K_{α} radiations can sometimes be added and treated as radiation with a wavelength of 1.542 \AA with an intensity of approximately 7 relative to K_{β} .

Table 1: Appearance numbers.

Set of plane hkl	n_{hkl}
h00	6
hh0	12
hk0	24
hhh	8
hhl	24
hkl	48

2.2 Appearance number

The analyzed sample consists of a large number of randomly oriented crystals. The intensity of the Bragg reflections derived from sets of planes with a certain interplanar distance d will be proportional to the probability of such a set having the correct orientation and this probability will be proportional to the number of such sets in the crystal. This number is the appearance number n_{hkl} . Each set of planes with the distance d has a corresponding reciprocal lattice vector with the length $2\pi/d$, and results in Bragg reflection if the vector has an appropriate direction. For this reason n_{hkl} is equal to the number of reciprocal lattice vectors with that length.

Example 1: A set of planes with Miller indices (100). The corresponding reciprocal lattice vector has the coordinates $(1, 0, 0)$. Equivalent reciprocal lattice vectors (i.e. vectors with the same length - see equation 3) are $(0, 1, 0)$, $(0, 0, 1)$, $(1, 0, 0)$, $(0, \bar{1}, 0)$, $(0, 0, \bar{1})$. Thus $n_{100} = 6$.

Example 2: A set of planes with Miller indices (123). We now have 8 different ways to change the sign of one or more of these indices (for example $(1\bar{2}\bar{3})$). We can also permute 123 in $3! = 6$ different ways. Therefore $n_{123} = 6 \cdot 8 = 48$.

The appearance number for cubic structures is summarized in table 1.

According to the above reasoning the intensity of a line in a Debye-Scherrer spectrum can be expected to be proportional to

$$I_{ber} = |S_{hkl}|^2 \cdot n_{hkl} \cdot I_{\lambda} \quad (7)$$

where I_{λ} is the relative intensity of the corresponding incoming x-ray radiation, discussed in section 2.1. The relative intensity of a diffraction ring in the experimental, photographically registered spectrum can roughly be approximated with visually observed relative exposure (exposure per unit area). (See discussion below).

3 Corrections

Most of the x-rays are absorbed by the sample (the metal thread). Absorption is caused by the photo-electric effect, i.e. the photon is annihilated and its energy excites an electron bound inside the metal (see Section 3.1). There is then inelastic scattering due to the Compton effect (scattering against loosely bound electrons), and elastic scattering against harder bound electrons (so called Rayleigh-scattering). It is the latter which, when it is coherent, results in Bragg scattering. The observed Bragg scattering mainly originates from the surface of the wire. Since the wire has a radius not equal to zero, this results in a slight change of the geometry of the experiment, and thus in the need to correct Eq.(4). We will also try to estimate the width of the diffraction rings in the experimental spectrum and we will discuss the intensity Eq.(7).

3.1 Photo-electric effect: absorption of x-rays in the sample

In order to estimate the radiations penetration depth in the sample we can discard the effects of Compton scattering and Rayleigh scattering, and we will only consider the photo-electric effect. Due to the photo-electric effect the intensity I of

Table 2: Photo-electric absorption coefficient and penetration depth of the radiation for several common metals.

	$\mu(cm^2/g)$	$\delta(mm)$
Al	46.8	0.24
Fe	300.	0.013
Cu	50.	0.067
Ag	207.	0.014
W	164.	0.0095
Au	203.	0.0077

the radiation decreases proportionally to the distance x that the radiation travels through the absorbing material according to the following equation:

$$I(x) = I(0)e^{-\mu\rho x} \quad (8)$$

where μ is the photo-electric absorption coefficient (cm^2/g) and ρ is the density of the material (g/cm^3). For Cu K_α -radiation, with a wavelength of $\approx 1.54 \text{ \AA}$, we can get the absorption coefficient from *Atomic data and Nuclear data tables vol. 54 (1993)*. As an example we can then calculate the distance at which the intensity decreases to 5% of the initial value, and can call this length the penetration depth δ of the radiation. The results are presented in the table 2.

3.2 An approximated method for the localization of the Bragg scattering within the sample

The sample (metal wire) has a radius R_r , whose exact value is unknown. It should however be approximately 0.25 mm. This is (with exception of Al and Cu) considerably larger than δ as shown in Table 2. It should therefore be possible to assume that the observed Bragg scattering is from the surface of the wire. We make the following simplified assumptions:

- 1) Bragg scattering only happens on the surface of the wire.
- 2) The part of the surface contributing to a certain diffraction line is the one that can be seen from the area where the line has been exposed on the film and at the same time is directly exposed to the incoming radiation.
- 3) Since the film is relatively narrow, we can assume that the path of the radiation is limited to a plane perpendicular to the metal wire.

Figure 5 illustrates the geometry of the model where $\theta < \pi/4$ and $\theta > \pi/4$ (all possible values are of course $0 < \theta < \pi/2$). It is apparent in the figure that the scattering surface comprise a total angle of 2θ , i.e. it represents a ratio of θ/π of the wire's total surface area. We can therefore expect that the diffuse background (caused by Compton scattering and incoherent Rayleigh scattering) will be larger at larger angles θ . Figure 5 also indicates that the width of the diffraction rings should increase with increasing θ .

3.3 Corrections on the measured deflection angle

We will now make a simple approximation of the relation between the deflection angle 2θ that should be used in the Bragg Eq.(4) and the observed deflection angle $2\theta_{obs}$, derived from Eq.(6). In order to do this we make one more assumption:

- 4) When making an approximate calculation of θ_{obs} , the radiation scattered by the sample can be assumed to be scattered from an infinitely narrow wire, parallel to the sample placed in the middle of the scattering part of the samples surface, i.e. in the point P in figure 5.

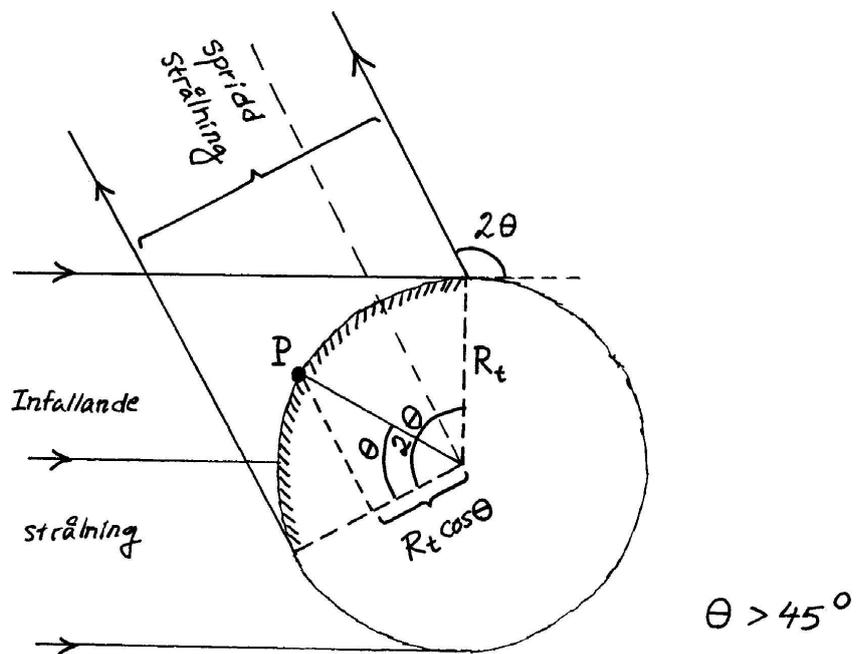
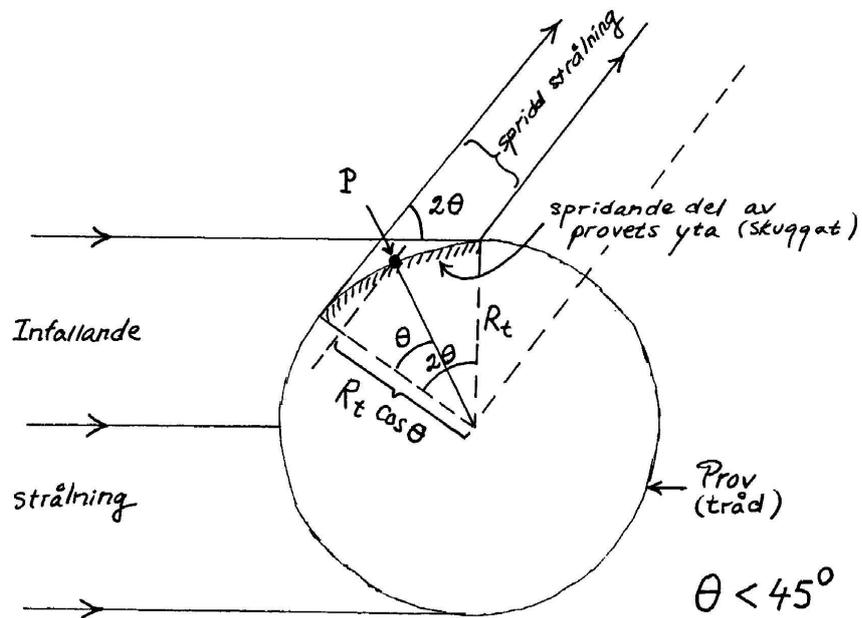


Figure 5: Model for the localization of the scattering on the sample surface.

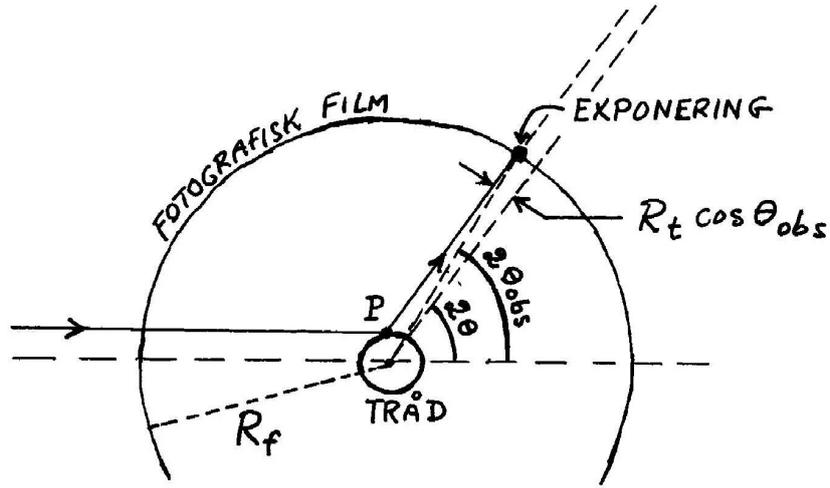


Figure 6: Model for the correction of the measured deflection angle.

It can be seen in figure 5 that P is at a distance $R_t \cos \theta$ from a line through the center of the sample going in the same direction as the incoming radiation. This results in the construction of figure 6, where R_f is the radius of the film, i.e. its distance to the center of the sample. In figure 6 we can see that

$$R_f \cdot (2\theta_{obs} - 2\theta) \approx R_t \cos \theta \quad (9)$$

Since θ is practically very close to being equal to θ_{obs} we can substitute the term $R_t \cos \theta$ with $R_t \cos \theta_{obs}$ and thus get

$$2\theta \approx 2\theta_{obs} - \frac{R_t}{R_f} \cos \theta_{obs} \quad (10)$$

This is an approximate equation to correct the deflection angle that we cannot use since we don't know the ratio R_t/R_f . We can however observe that the deviation of the observed angle decreases with increasing angle $2\theta_{obs}$ and reaches zero when $2\theta_{obs} = 180^\circ$.

3.4 Correction of the calculation of the lattice constant a

If the difference between θ and θ_{obs} can be neglected we can calculate the lattice constant a from Eq.4, which is then appropriately written as

$$a^2 = \frac{\lambda^2}{4 \sin^2 \theta} \cdot (h^2 + k^2 + l^2). \quad (11)$$

If we measure the deflection angle 2θ for a certain diffraction ring, and manage to determine its Miller indices (hkl) and the wavelength λ , we can then calculate the lattice constant from equation (11). If the determined a doesn't need to be corrected, then all diffraction rings should give the same value a (within the error limits). If θ_{obs} is used without any correction in equation (11), then we get:

$$a'^2 = \frac{\lambda^2}{4 \sin^2 \theta_{obs}} \cdot (h^2 + k^2 + l^2) \quad (12)$$

where we denote the lattice constant with a' to mark that this is a non-corrected value. Correction of θ can be included in the following way. From equation (10) we get

$$\sin \theta \approx \sin \theta_{obs} \cos \left(\frac{R_t}{2R_f} \cos \theta_{obs} \right) - \cos \theta_{obs} \sin \left(\frac{R_t}{2R_f} \cos \theta_{obs} \right) \approx \sin \theta_{obs} - \frac{R_t}{2R_f} \cos^2 \theta_{obs} \quad (13)$$

The last approximation is possible since $R_t/R_f \ll 1$ and using the properties $\cos \alpha \approx 1$ and $\sin \alpha \approx \alpha$ if α is an angle $\ll 1$ rad. Equation (13) gives:

$$\sin^2 \theta \approx \sin^2 \theta_{obs} - \frac{R_t}{R_f} \sin \theta_{obs} \cos^2 \theta_{obs} = \sin^2 \theta_{obs} \cdot \left[1 - \frac{R_t \cos^2 \theta_{obs}}{R_f \sin \theta_{obs}} \right] \quad (14)$$

where we once again have used the fact that $(R_t/R_f) \ll 1$. From Eqs. (11) and (12) and using Eq. (14) we get:

$$\frac{a'^2}{a^2} = 1 - \frac{R_t \cos^2 \theta_{obs}}{R_f \sin \theta_{obs}} \quad (15)$$

This can be written as

$$a'^2 = a^2 - \frac{a^2 R_t}{R_f} \cdot \frac{\cos^2 \theta_{obs}}{\sin \theta_{obs}} \quad (16)$$

Making a diagram a'^2 as a function of $(\cos^2 \theta_{obs} / \sin \theta_{obs})$, one should get a straight line whose intersection with the ordinate (i.e. $\theta_{obs} = \pi/2$) gives the correct value of the square of the lattice constant a^2 .

3.5 Estimation of the width of the lines

The width B of the diffraction lines on the photographic film could be the sum of several different contributions. The contribution from the thickness of the wire, according to figure 5, should be

$$B_t = R_t \cdot (1 - \cos 2\theta) \quad (17)$$

This contribution apparently increases as θ increases.

The contribution from the natural width of line $d\lambda$ for example for $K_{\alpha 1}$ radiation can be calculated in the following way. Equation (4) can be written as

$$\sin \theta = \frac{\lambda}{2a} (h^2 + k^2 + l^2)^{1/2} \quad (18)$$

Differentiation gives

$$\cos \theta \cdot d\theta = \frac{d\lambda}{2a} \cdot (h^2 + k^2 + l^2)^{1/2} \quad (19)$$

Combining equations (18) and (19) we get

$$\frac{d\theta}{\tan \theta} = \frac{d\lambda}{\lambda} \quad (20)$$

The contribution to the line width is thus:

$$B_\lambda = R_f \cdot d\theta = R_f \cdot \tan \theta \cdot \frac{d\lambda}{\lambda} \quad (21)$$

The total width is then $B = B_t + B_\lambda$. Further contributions may appear, for example if the incoming rays are somewhat divergent. The literature values (from Atomic data and Nuclear tables vol 18 page 233) suggests that $(d\lambda/\lambda) \approx 4 \cdot 10^{-4}$. If R_t is 0.25-0.5 mm we get that B_t is one order of magnitude larger than B_λ , i.e. the effect from the wires thickness B_t seems to be the dominating reason for the line width.

3.6 Discussion of the intensity equation

The *total* amount of radiation that results in a diffraction ring should be proportional to a factor $Y(\theta)$ that in turn is proportional to the part of the surface of the sample that scatters the radiation, and should increase with increasing angle θ (just like the diffuse background). As a rough estimation of $Y(\theta)$ we can use the projection of the scattering part of the samples surface on a plane perpendicular to the incoming radiation. From figure 5 we get:

$$Y(\theta) = \frac{R_t - R_t \cos 2\theta}{2R_t} \quad (22)$$

To get the exposure of the ring (darkness) we should divide with the width B of the ring. Since $B \approx B_t$ which is proportional to $(1 - \cos 2\theta)$, the angle dependence of Y will apparently not influence the intensity. However it should be observed that the structural factor S_{hkl} includes the atomic form factor $f(\lambda, \theta)$, which *decreases* with increasing θ . Some examples are shown in figure 2. There might also some other angle dependent effects. The estimation of the intensity is so coarse that there is hardly any point in performing any detailed calculations. Rather we can use Eq.(7) directly where the angular dependence of f can be neglected. Equation (7) should however be fairly reliable when only comparing rings close to each other. In this case the θ dependence of $|f|^2$ and other geometrical effects are less noticeable.

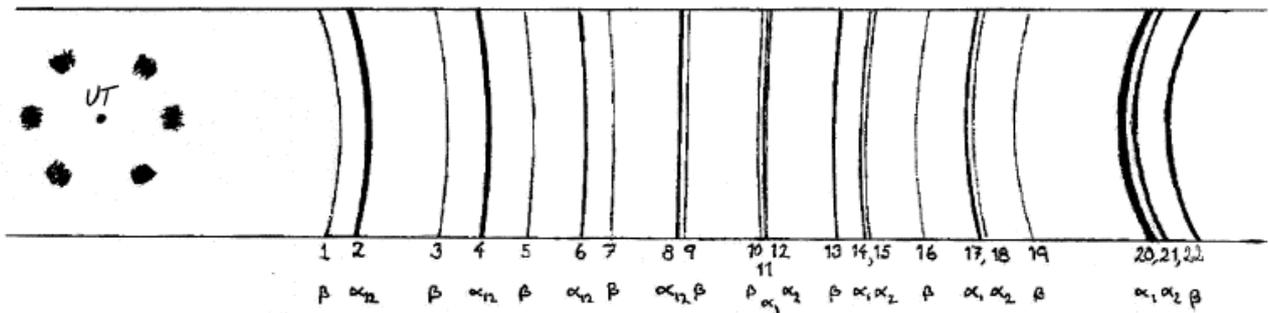


Figure 7: Picture of an experimental Debye-Scherrer spectrum. The symbol α_{12} denotes the lines from α_1 and α_2 radiation in cases where they blur together. Each ring is identified by the numbers 1 to 22.

4 Assignment

The assignment consists in determining the type of lattice (fcc or bcc) and the metal that generated the diffraction spectrum in Fig. 7.

Table 3: Measured values of s and observed intensities of each line I_{obs} . The s values are affected by an error that was estimated to be $\Delta s = 0.4mm$.

Ring nr	s (mm)	I_{obs}
1	73.1	2
2	81.0	4
3	105.1	1
4	117.1	2
5	130.8	2
6	147.2	5
7	154.2	1
8	174.4	3
9	176.8	2
10	199.8	<1
11	201.6	4
12	202.4	2
13	222.0	3
14	229.8	1
15	231.2	1
16	246.8	<1
17	262.8	7
18	264.0	5
19	276.0	4
20	307.4	4
21	309.6	2
22	318.8	4

Such spectrum is impressed on a transparent film and can be analysed using a sort of light table with an incorporated ruler. In such a way one can measure the diameter of all the rings (s) and observe their intensities (I_{obs}) which are estimated on a continuous scale (1-7). These values, together with the wavelengths of the incoming radiation are the raw data that should be analyzed. In figure 7 the characteristic wavelength for each diffraction ring is given, and in table 3 you find the previously measured values of s and I_{obs} . The distance between entrance and exit holes has been measured as $l=(180.0\pm 0.1)$ mm. The radius of the film cylinder R_f is given by $R_f = l/\pi$ (look at Fig. 3 to convince yourself). Notice that all the measured values which are given to you are affected by an error because they are the result of actual measurements done in the past. The I_{obs} shouldn't be considered perfect either since they are the subjective evaluation of the lines intensity; these values should be used as a help and should show some trend when compared to I_{ber} (point 5 of this assignment). Using the key-formulas given in the earlier sections and the information in Fig. 7 and table 3, you have to:

1. determine the type of lattice (fcc or bcc);
2. determine the **corrected** value of the lattice constant a ;
3. compare the calculated value of a with the literature value. In this way it will be possible to determine the sample material.
4. If the result is correct the number of atoms in a unit cell should be an integer. Calculate the number of atoms n in a unit cell to verify your data using the following equation

$$n = a^3 \cdot \rho \cdot N_a / A$$

Where ρ is the literature value for the density of the material, N_a is Avogadro's number and A is the atomic mass.

5. Calculate the expected intensity I_{ber} using Eq.(7) and compare it with the observed intensity I_{obs} . Comments?

You will probably find that when analysing a spectrum both systematic work as well as some guesses are necessary. Hints:

1. The type of lattice (fcc or bcc) can be determined looking at the Miller indices. Why?
2. You will need to use Eq.(4) and a *trial and test method* to determine the Miller indices. Think of the wavelength that generated each ring!
3. The **corrected** value of a^2 is obtained by plotting a'^2 as a function of $\cos^2 \theta_{obs} / \sin \theta_{obs}$.

4.1 The report

Your report should contain a description of the analysis. You do not have to describe the theory unless you think it is necessary, but your calculated values and method of analysis should be clearly described. You should also justify each formula that you decide to use.

DL-96/ST-10/AR-12

BRAGG SCATTERING II

Diffraction patterns from x-ray scattering according to Laues method

5 Introduction

Laues method for x-ray diffraction is done according to figure 8. The x-rays, distributed over a continuous spectrum of wavelengths λ , originates from the x-ray tube R , inside which electrons which have been accelerated by a high voltage of the order of kV bombard an anode. The lowest wavelength λ_{min} is determined from

$$E = \frac{hc}{\lambda_{min}} \quad (23)$$

where h is Planck's constant, c is the speed of light and E is the kinetic energy of the electrons when they hit the anode. The radiation is collimated (using led plates P) into a narrow beam, and then hits a mono crystal. Photographic plates are mounted in both the forward (F) and backward (B) directions. Through Bragg reflection the beams are partially deflected in certain directions, which results in a pattern of small spots (diffraction spots) on both plates. Figure 9 shows what such a diffraction pattern can look like. The purpose with this exercise is to give a better insight into the correlation between this pattern and the crystal structure.

6 Theory

First let us consider x-ray diffraction in a simple cubic (sc) lattice with the lattice constant a . The Bravais lattice is defined by the three fundamental vectors $\mathbf{a}, \mathbf{b}, \mathbf{c}$ (each with the length a) which are the base vectors in a right oriented Cartesian coordinate system. The reciprocal lattice has the lattice constant $2\pi/a$ and is defined by the three fundamental vectors:

$$\mathbf{A} = \frac{2\pi}{a} \hat{\mathbf{a}} \quad (2)$$

$$\mathbf{B} = \frac{2\pi}{a} \hat{\mathbf{b}}$$

$$\mathbf{C} = \frac{2\pi}{a} \hat{\mathbf{c}}$$

Where $\hat{\mathbf{a}}$ is the unit vector \mathbf{a}/a , $\hat{\mathbf{b}} = \mathbf{b}/a$ and so on. A general vector in the reciprocal lattice is thus given by

$$\mathbf{G} = h\mathbf{A} + k\mathbf{B} + l\mathbf{C} = \mathbf{G}(hkl) \quad (3)$$

Where h, k, l (the Miller index) are all integers. We now introduce the following denotations

\mathbf{k}_i = wave vector of the incoming radiation

\mathbf{k}_r = wave vector of the reflected radiation

$k_\lambda = |\mathbf{k}_i| = |\mathbf{k}_r| = 2\pi/\lambda$

θ = the Bragg angle

2θ = deflection angle. The scattering geometry, according to the well known condition $\Delta\mathbf{k} = \mathbf{G}$ for Bragg reflection, can be seen in figure 10. From the figure we get

$$\frac{1}{2}G = k_{\lambda} \sin(\theta) \quad (4)$$

where $G = |\mathbf{G}|$.

We now assume that the incoming radiation has the direction $-\hat{\mathbf{a}}$, i.e. \mathbf{k}_i is parallel to $[\bar{1}00]$ and thus perpendicular to a surface of the cube. (It isn't hard to generalize the equations for radiation from any direction). We have (see figure 10):

$$\sin(\theta) = \cos(\alpha) = (\mathbf{G}/G) \cdot \hat{\mathbf{a}} \quad (5)$$

From which we get (you should control this!)

$$\sin(\theta) = \frac{h}{\sqrt{h^2 + k^2 + l^2}} \quad (6)$$

Using Eq. 4 we get (you should control this!)

$$\lambda = 2a \cdot \frac{h}{h^2 + k^2 + l^2} \quad (7)$$

Please observe that for every set of Miller indexes (hkl) there is a certain wavelength λ . It has been noted that if Laue diffraction could be done with visible light the spots would have different colors. You might note that if Eqs. 6 and 7 are combined we get:

$$n\lambda = 2d \sin(\theta) \quad (8)$$

where d is the distance between the crystal planes responsible for the reflection and n is a common integer of h, k and l , i.e. the Bragg's law.

The deflection angle is 2θ (see figure 10). We are trying to determine the location of a diffraction spot on the photographic plate F. If the distance between K and F is L , we get that the distance r from the plates center (the point where the non deflected ray hits the plate, this part of the plate is often cut away) to where the diffraction spot is

$$r = L \tan(2\theta) \quad (9)$$

Finally we seek the x - and y - coordinates for the diffraction spot in a coordinate system where the axis are parallel to the fundamental vectors \mathbf{b} and \mathbf{c} . Since the components of \mathbf{k}_r , in the plane of the plate, are the same as the components as the plane \mathbf{G} (i.e. $k\mathbf{B}$ as well as $l\mathbf{C}$), and thus is proportional to k and l , we get the coordinates

$$x = r \cdot \frac{k}{\sqrt{k^2 + l^2}} \quad (10a)$$

$$y = r \cdot \frac{l}{\sqrt{k^2 + l^2}} \quad (10b)$$

(As an alternative we can use that the diffraction spot is at a distance r from the center of the plate in such a direction that $y : x = l : k$).

7 Symmetries

Please note that the Miller index in this case is not reduced to smallest integer. Furthermore $h > 0$, which can be seen in the geometry of figure 10 (make sure that you understand this), while k and l can be both positive and negative.

From the previously derived equations we can see the following:

- if $(h', k', l') = n \cdot (h, k, l)$ then the diffraction spots corresponding to Bragg reflections with Miller index $(h'k'l')$ and (hkl) will overlap. For example $(20\bar{4})$ and $(10\bar{2})$.

- Changing the sign of k or l does not change the distance r . Furthermore the spots (hkl) and $(h\bar{k}l)$ have the same distance r and are spaced symmetrically around a line forming the angle 45° with the x -axis. From this we get that the diffraction pattern have a 8 folded symmetry, which is used when calculating the position of the spots.

- A small hint: the characteristic "flower like" pattern in figure 2 can be understood by studying sequence of the type

$$(h0l), (h1l), (h2l), \dots, (hll).$$

8 Exercise

Do the following assignments.

1. Answer the following questions:

1a Why don't we get a diffraction pattern on either the F or B plate corresponding to miller index (101)?

1b Which Miller indexes (hkl) corresponds to scattering in 180° ?

2. Assume as above that the incoming radiation is in the direction $[\bar{1}00]$. Furthermore assume that the crystal is of sc type with $a = 4\text{\AA}$ and that the radiation is distributed over a continuous spectrum of wavelengths where the shortest wavelength is $\lambda_{min} = 1\text{\AA}$. Calculate the Laue-diffraction pattern on plate F for deflection angles $2\theta < 70^\circ$. The distance from the crystal to the plate is assumed to be $L = 5\text{cm}$.

Present your results as a picture of the diffraction pattern on a mm-paper (or a computer generated picture where the scale of x and y clearly shown so the coordinates can be checked). Mark the miller index (hkl) by each spot. Also mark the wavelength for each spot. Append your method of calculation. (Hint: find the maximum value for the Miller indices (hkl) using the above conditions, investigate the results for the possible Miller indices (hkl), possibly using a computer program).

3. Assume that the crystal is instead of bcc type and the other assumptions and conditions as in assignment 2. Present the new diffraction pattern and a short description of your calculations.

4. Repeat assignment 3 but assume fcc structure instead.

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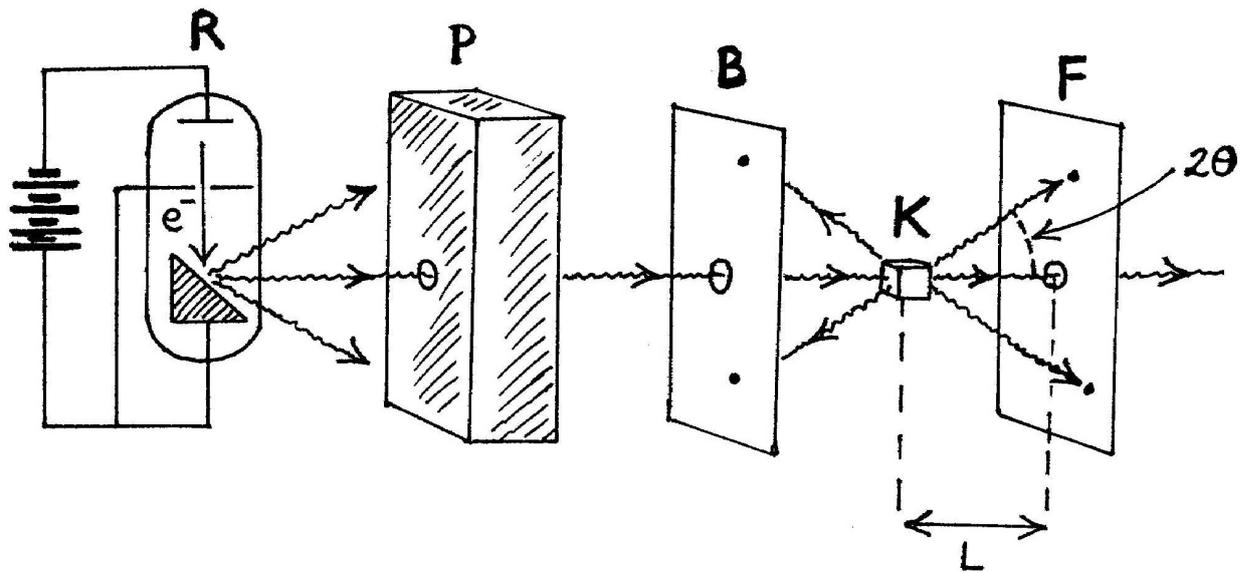


Figure 8: Schematic picture of the experimental setup of x-ray diffraction using Laues method.

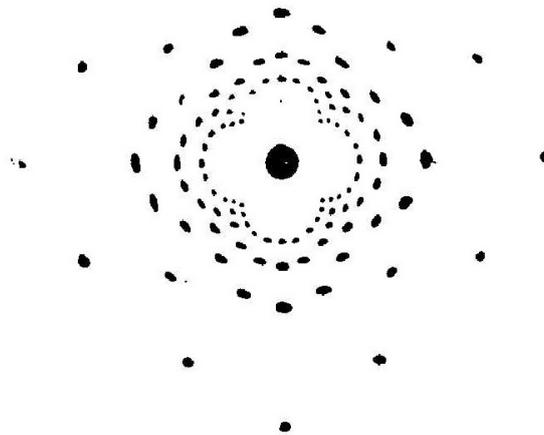


Figure 9: Laue photograph of a simple cubic crystal. The radiation inclines perpendicular to a surface of the cube. Picture from R.W. James and O.W. Richardson, X-Ray crystallography, Methuen & Co 1945

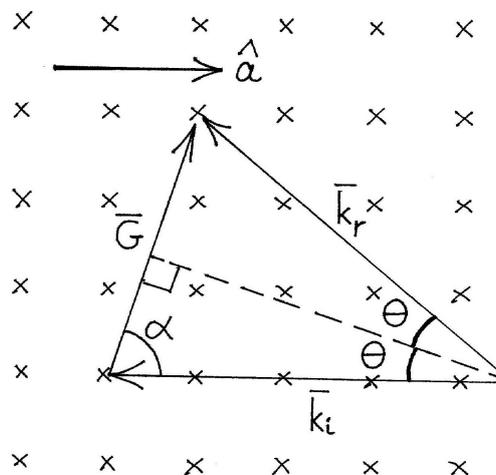


Figure 10: Scattering geometry. Each x marks a reciprocal lattice point.

Table 1.1 Crystal structure of the elements

Elements not listed have less common and in general more complex structures. As and Bi share a trigonal structure, Se and Te another trigonal structure. The graphite form of carbon has a hexagonal structure. None of the three possible structures of Mn is close-packed, but they have cubic or tetragonal symmetry. S and Se have a variety of structures. Structure data from R. W. G. Wyckoff, "Crystal Structures," Interscience Publishers, Inc., New York, 1948-1960; specific-density values, for room temperature, from "Periodic Table of the Elements," Central Scientific Co., Chicago, 1962.

Face-centered cubic structure			
Element	Name	Cube edge a_0 , angstroms	Specific density, g/cm ³
Ag	Silver	4.0862	10.5
Al	Aluminum	4.04934 (25°C)	2.71
Ar	Argon	5.43 (-253°C)	
Au	Gold	4.07864 (25°C)	19.3
Ca	Calcium	5.576	1.55
Ce	Cerium	5.150	6.9
β -Co	Cobalt	3.548 ^a	8.75
Cu	Copper	3.61496 (18°C)	8.9
γ -Fe	Iron	3.5910 (22°C)	8.02
Ir	Iridium	3.8394 (26°C)	22.4
Kr	Krypton	5.705	
La	Lanthanum	5.296	6.15
Li	Lithium	4.404 (-195°C)	0.53
		4.379 (-194°C)	
Ne	Neon	4.52 (-253°C)	
Ni	Nickel	3.52387 (25°C)	8.9
Pb	Lead	4.9505	11.34
Pd	Palladium	3.8898 (25°C)	12.0
Pr	Praseodymium	5.161	6.5
Pt	Platinum	3.9231 (25°C)	21.45
δ -Pu	Plutonium	4.6370 (320°C)	19
Rh	Rhodium	3.8031 (25°C)	12.5
Sc	Scandium	4.541	3.1
Sr	Strontium	6.0847 (25°C)	2.6
Th	Thorium	5.05	11.7
Xe	Xenon	6.25	
Yb	Ytterbium	5.460	7.01

Diamond structure			
Element	Name	Cube edge a_0 , angstroms	Specific density, g/cm ³
C	Carbon (diamond)	3.56679 (20°C)	3.51
Ge	Germanium	5.65748 (20°C)	5.36
Si	Silicon	5.43086 ^d (20°C)	2.33
α -Sn	Tin	6.4912	5.75

^a In liquid N₂, $a_0 = 3.550$ A; in H₂, $a_0 = 3.557$ A.
^b Above values *in vacuo*. In N₂, $a_0 = 2.508$ A, $c_0 = 4.068$ A; in H₂, $a_0 = 2.512$ A, $c_0 = 4.072$ A.
^c At -183°C, $a_0 = 3.4832$ A.
^d At 1300°C, $a_0 = 5.445$ A.
^e In an ideal hcp structure, $c_0 = 2\sqrt{\frac{2}{3}}a_0$, but atomic asymmetry often alters this dimension. See Fig. 1.8 for definitions.

Body-centered cubic structure			
Element	Name	Cube edge a_0 , angstroms	Specific density, g/cm ³
Ba	Barium	5.025 (26°C)	3.59
Cr	Chromium	2.8839 (25°C)	7.1
Cs	Cesium	6.05 (-185°C)	1.9
α -Fe	Iron	2.8665 (25°C)	7.86
β -Fe	Iron	2.91 (800°C)	
δ -Fe	Iron	2.94 (1425°C)	
K	Potassium	5.21 (-150°C)	0.86
Li	Lithium	3.5093 (20°C) ^c	0.53
Mo	Molybdenum	3.1473 (25°C)	10.2
Na	Sodium	4.2906 (20°C)	0.97
Nb	Niobium (columbium)	3.3004 (18°C)	8.4
γ -Np	Neptunium	3.52 (ca. 600°C)	19.5
ϵ -Pu	Plutonium	3.638 (500°C)	
Rb	Rubidium	5.63 (-185°C)	1.53
γ -Sr	Strontium	4.85 (614°C)	
Ta	Tantalum	3.3058 (25°C)	16.6
β -Ti	Titanium	3.3065 (900°C)	
Tl	Thallium	3.882	11.85
γ -U	Uranium	3.474	19.05
V	Vanadium	3.040	5.96
W	Tungsten (wolfram)	3.16469 (25°C)	19.3
Zr	Zirconium	3.62 (850°C)	

Hexagonal close-packed structure				
Element	Name	Planar spacing a_0 , angstroms	c_0 , angstroms ^e	Specific density, g/cm ³
Be	Beryllium	2.2860	3.5843	1.86
Ca	Calcium	3.98	6.52 (450°C)	1.55
Cd	Cadmium	2.97887	5.61765 (26°C)	8.6
Ce	Cerium	3.65	5.96	6.9
α -Co	Cobalt	2.501	4.066 ^b	8.9
Cr	Chromium	2.722	4.427	7.1
Dy	Dysprosium	3.584	5.668 (49°K)	8.56
		3.596	5.649 (30°K)	
Er	Erbium	3.558	5.590 (43°K)	9.16
		3.562	5.602 (301°K)	
Gd	Gadolinium	3.629	5.796 (106°K)	
		3.639	5.777 (349°K)	7.95
He	Helium	3.57	5.83 (-271°C)	
Hf	Hafnium	3.1967	5.0578 (26°C)	13.3
Ho	Holmium	3.564	5.630	8.76
La	Lanthanum	3.75	6.07	6.15
Li	Lithium	3.09	4.83 (-195°C)	0.53
Lu	Lutecium	3.516	3.566	
Mg	Magnesium	3.20927	5.21033 (25°C)	1.74
Nd	Neodymium	3.657	5.902	7.0
Ni	Nickel	2.65	4.33	8.9
Os	Osmium	2.7352	4.3190 (20°C)	22.5
Pr	Praseodymium	3.669	5.920	6.5
Rh	Rhenium	2.7608	4.4582 (25°C)	21.0
Ru	Ruthenium	2.70389	4.28168 (20°C)	12.4
Sc	Scandium	3.309	5.255	3.1
β -Sr	Strontium	4.32	7.06 (248°C)	2.6
Tb	Terbium	3.592	5.673	8.33
Ti	Titanium	2.950	4.686 (25°C)	4.5
Tl	Thallium	3.456	5.525	11.8
Tu	Thulium	3.530	5.575	9.35
Y	Yttrium	3.636	5.761	4.34
Zn	Zinc	2.6648	4.9467 (25°C)	7.14
α -Zr	Zirconium	3.232	5.147 (25°C)	6.4

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