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## SYNTHETIC OVERVIEW OF STRUCTURAL ANALYSIS METHODS FOR METALLIC MATERIALS USING X-RAY DIFFRACTION

BY

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**Abstract.** Identifying the structure of a material is not a trivial investigation process, especially considering the scale at which the investigation should be performed. An important method for such a study is the one known as "X-ray diffraction structure analysis". The initial purpose of X-ray diffraction was and remains to identify the structure of an unknown material. This type of analysis is much more favorable for the analysis of monocrystals of different substances, but for the analysis of polycrystalline materials the variant known as the "powder method" is used. Structural analysis by X-ray diffraction is based on X-ray diffraction by atomic planes inside the crystals. The result of the diffraction is the so-called diffraction pattern which is visualized in graphic form. This "diffraction pattern" has as its main characteristics the "peaks" or "diffraction maxima", which represent the angles (expressed as  $2\theta$ ,  $\theta$  being the angle expressed in degrees) at which the diffraction maxima are obtained.

**Keywords:** X-ray diffraction; peaks; diffraction pattern.

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## 1. Introduction

X-rays are electromagnetic radiation with typical photon energies in the range of 100eV - 100keV. For diffraction applications, only short wavelength X-rays (hard X-rays) in the range of a few angstroms to 0.1 angstroms (1 keV - 120 keV) are used. Because the X-ray wavelength is comparable to the size of atoms, they are suitable for testing the structural arrangement of atoms and molecules in a wide range of materials. Energy X-rays can penetrate deep into materials and provide information about the structure (<https://www.scribd.com/doc/71600712/Indexing-X-Ray-Diffraction-Patterns>).

X-ray crystallography (Fig. 1) determines the arrangement of atoms in a crystalline structure. In essence, we are dealing with an X-ray beam that strikes the studied crystal, and based on the diffraction angles and the intensities of the scattered rays, a three-dimensional image of the electron density inside the crystal is obtained (Bruno *et al.*, 2017).

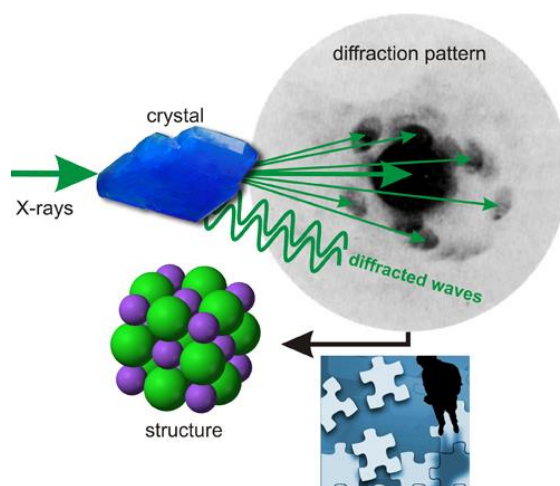


Fig. 1 – Schematic representation of the X-ray crystallography workflow (<https://www.xtal.iqfr.csic.es/Cristalografia/cascara-en.html>).

X-rays are generally produced either by X-ray tubes (Fig. 2) or by synchrotron radiation. In an X-ray tube, which is the primary radiography source used in laboratory radiography instruments, X-rays are generated when an accelerated electron beam focused on a high-voltage field bombards a stationary or rotating solid target. As the electrons collide with the atoms in the target and slow down, a continuous spectrum of X-rays is emitted, which is called Bremsstrahlung radiation. High-energy electrons also expel internal electrons into the shell through atoms through the ionization process. When a free electron fills the shell, an X-ray photon is emitted with energy characteristic of the target

material. Common lenses used in X-ray tubes include Cu and Mo, which emit 8 keV and 14 keV X-rays with corresponding wavelengths of 1.54 Å and 0.8 Å, respectively.

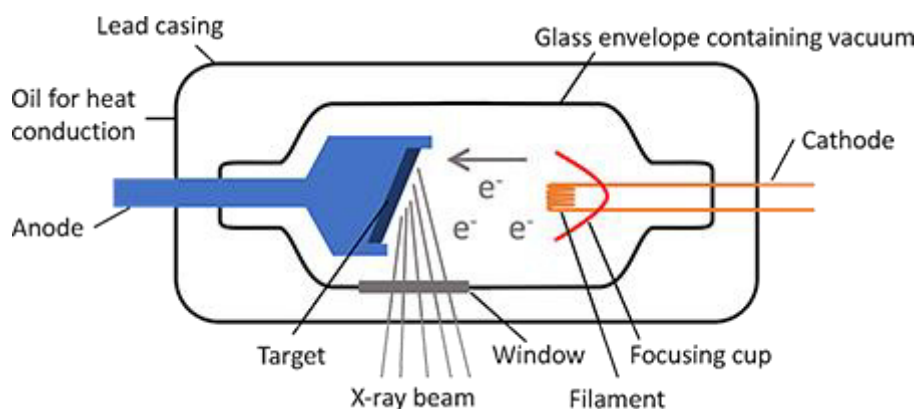


Fig. 2 – Schematic of an X-ray tube (<https://www.radiologycafe.com/radiology-trainees/frcr-physics-notes/production-of-x-rays>).

Over the past decade, synchrotron installations have become widely used as preferred sources for X-ray diffraction measurements. Synchrotron radiation is emitted by electrons or positrons traveling at speeds close to light in a circular storage ring. These powerful sources, which are thousands to millions of times more intense than laboratory radiography tubes, have become indispensable tools for a wide range of structural investigations and have made progress in many fields of science and technology.

X-rays interact primarily with electrons in atoms. When X-ray photons collide with electrons, some photons in the incident beam will be deflected away from the direction they originally traveled, just like billiard balls that jump on top of each other. If the wavelength of these scattered X-rays has not changed (meaning that X-ray photons have not lost any energy), the process is called elastic scattering (Thompson Scattering) in that only the process has been transferred to the scattering process. These are the X-rays we measure in diffraction experiments, because scattered X-rays carry information about the distribution of electrons in materials. On the other hand, in the process of inelastic scattering (Compton Scattering), the X-rays transfer some of their energy to the electrons, and the scattered X-rays will have a different wavelength than the incident X-rays.

Diffracted waves from different atoms can interfere with each other, and the resulting intensity distribution is strongly modulated by this interaction. If the atoms are arranged periodically, as in crystals, the diffracted waves will consist of maximum interferences (peaks) with the same symmetry as in the distribution

of atoms. The measurement of the diffraction pattern allows us, therefore, to deduce the distribution of atoms in a material.

The peaks in an X-ray diffraction pattern are directly related to the atomic distances. Consider an incident X-ray beam that interacts with the atoms arranged periodically, as shown in 2 dimensions in the following illustrations. Atoms, represented as green spheres in the graph, can be seen forming different sets of crystal planes (colored lines in the graph on the left). For a given set of atomic planes with a distance between planes of  $d$ , the condition for a diffraction (peak) to occur can simply be written as  $2d\sin\theta = n\lambda$  which is known as Bragg's law (Fig. 3), after WI Bragg, who first proposed it. In the equation,  $\lambda$  is the wavelength of the radiograph,  $\theta$  the scattering angle and  $n$  is an integer number representing the order of the diffraction peak. Bragg's law is one of the most important laws used to interpret X-ray diffraction data.

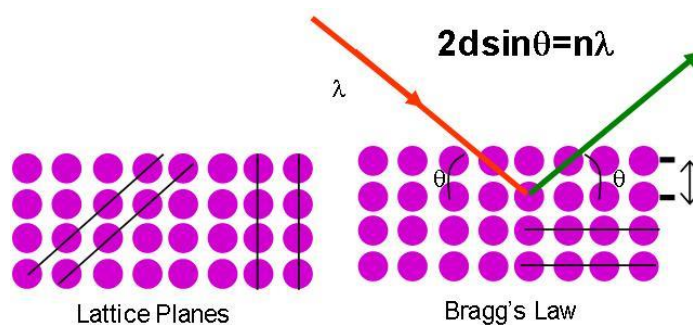


Fig. 3 – Bragg's law

([https://chem.libretexts.org/Bookshelves/Analytical\\_Chemistry/Supplemental\\_Modules\\_\(Analytical\\_Chemistry\)/Analytical\\_Sciences\\_Digital\\_Library/JASDL/Courseware/Introduction\\_to\\_X-ray\\_Diffraction\\_\(XRD\)/03\\_Basic\\_Theory/02\\_Diffraction\\_and\\_Bragg%E2%80%99s\\_Law](https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Supplemental_Modules_(Analytical_Chemistry)/Analytical_Sciences_Digital_Library/JASDL/Courseware/Introduction_to_X-ray_Diffraction_(XRD)/03_Basic_Theory/02_Diffraction_and_Bragg%E2%80%99s_Law)).

## 2. X-ray diffraction

X-ray diffraction allows researchers to examine single crystals and polycrystalline materials (Fig. 4). An X-ray beam is projected into the sample and how the beam scatters the atoms by radiography is examined. Bragg's law enables the observation of scattered X-ray interference ( $2d\sin\theta = n\lambda$ ). In general, measurements are made in Angstroms (1 Angstrom = 0.1 nm), so X-ray diffraction is a primary characterization tool for obtaining information about critical characteristics, such as crystal structure, identification of phases of crystal structure, crystal size (information about the size of the unit cells) and deformation. XRD spectra determine the crystalline nature of silver nanoparticles by the total oxidation state of the particles as a function of time.

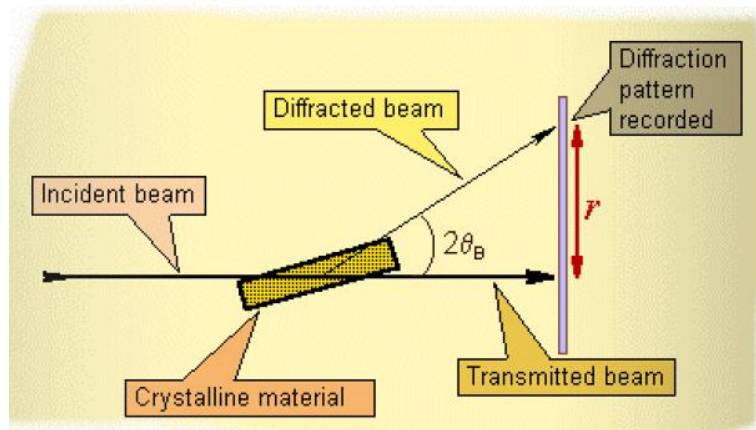


Fig. 4 – Operational principles of X-ray diffraction  
(<https://web.pdx.edu/~pmoeck/phy381/Topic5a-XRD.pdf>).

Figure 5 shows the diffraction of an X-ray beam on a family of crystalline planes with interplanar distance  $d$ ; the diffraction angle  $2\theta$  represents the angle formed by the incident and diffracted X-rays. The condition for forming a maximum diffraction is given by Bragg's relation:  $2d\sin\theta = \lambda$ , where  $\lambda$  is the wavelength of the X-ray. The intensity of the diffracted X-rays is recorded as a function of the diffraction angle.

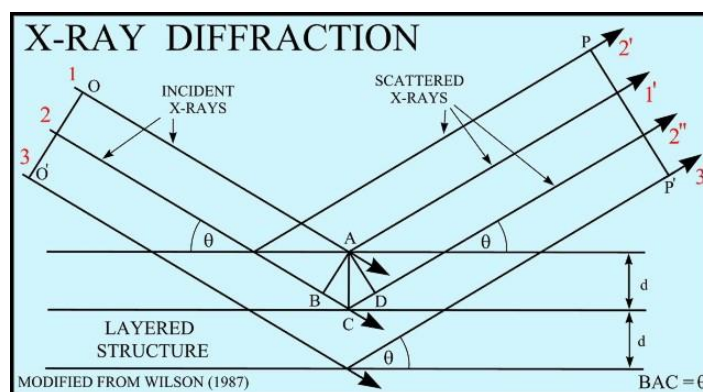


Fig. 5 – Diffraction scheme of an X-ray beam  
(<https://chme.nmsu.edu/elective-course-on-x-ray-diffraction/>).

The parafocus diffractometer (or Bragg-Brentano) is the most common geometry for diffraction instruments.

This geometry offers the advantages of high resolution and beam intensity analysis at the cost of very precise alignment requirements and carefully

prepared samples. In addition, this geometry requires that the source-sample distance be constant and equal to the sample-detector distance. Alignment errors often lead to difficulties in identifying phases and improper quantification. An incorrectly positioned sample can lead to unacceptable errors. The flatness of the sample, the roughness and the positioning constraints prevent the measurement of the sample in line. In addition, traditional XRD systems are often based on bulky equipment with high power requirements and use high-power X-ray sources to increase the flow of X-rays on the sample, thus increasing the diffraction signals detected in the sample. These sources also have large areas of agitation, which are often disadvantageous for the diffraction analysis of small samples or small sample characteristics.

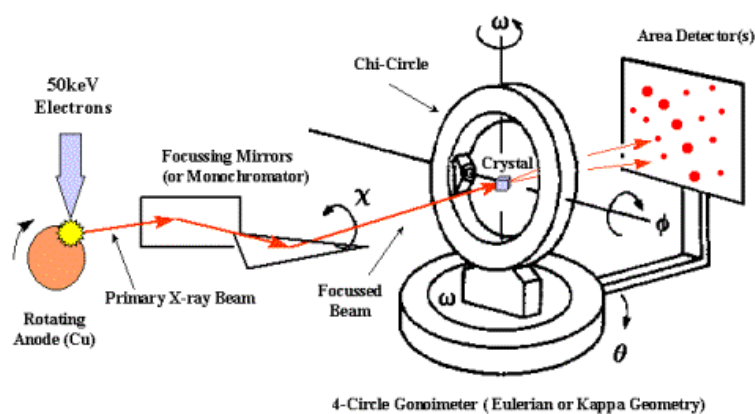


Fig. 6 – The atomic planes of a crystal cause an incident X-ray beam and mix with each other when they leave the crystal.

The setup of an X-ray detector

(<http://pruffle.mit.edu/atomiccontrol/education/xray/xray-diff.php>).

Polycapillary X-ray optics can enhance XRD applications by eliminating many of these challenges. These optics can be used to create the geometry of a parallel beam XRD instrument (Fig. 7), which reduces or eliminates many sources of peak position errors and intensity inherent in parafocusing geometry, such as position, shape, roughness, flatness, and transparency. Polycapillary focusing optics collect X-rays from a divergent X-ray source and direct them into a small beam focused on the sample surface. This beam has a diameter of tens of micrometers, making it ideal for small sample X-ray diffraction applications or small features. Both types of polycapillary optics direct very high X-ray intensities onto the sample surface. Thus, XRD systems that use these optics can use low-power X-ray sources, which reduces instrument size, cost, and power requirements.

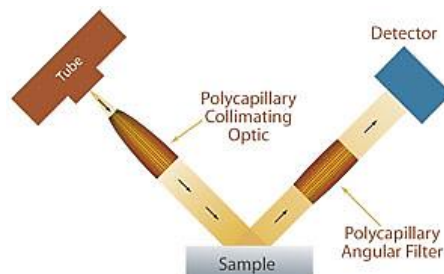


Fig. 7 – Parallel Beam XRD (<https://www.xos.com/Parallel-Beam-XRD>).

X-ray diffraction using X-ray optics has been applied to many different types of applications, including thin film analysis, sample texture evaluation, phase and crystal structure monitoring, and investigation of sample voltages.

### 3. Structural analysis by X-ray diffraction

The diffraction pattern obtained by the powder method mainly provides useful data on:

1. the shape and dimensions of the unit cell, which can be deduced from the position of the diffraction lines (diffraction maxima) indicated by the value  $2\theta$ ; the unit cell defines the symmetry and structure of the entire crystal lattice, a crystal lattice that is constructed by the repetitive translation of the unit cell along its main axes; the lengths of the main axes of the unit cell and the angles between them are the network constants, also called the network parameters; the symmetry properties of crystals are described by the concept of space group; all possible symmetrical arrangements of particles in three-dimensional space can be described by the 230 spatial groups.

2. the arrangement of the atoms in the unit cell, which is deduced from the relative intensities of the diffraction lines; the positions of the atoms inside the unit cell are described by the set of atomic positions  $(x_i, y_i, z_i)$  measured from a reference point of the network. Typically, atomic positions are represented in terms of fractional coordinates, relative to the lengths of elementary cells (Cullity, 1956).

Several methods or a combination of methods can be used to interpret an X-ray diffraction pattern. Special problems arise in the interpretation of diffractograms (diffraction patterns in graphic form) of new materials that may have in the structure new crystalline phases or phases derived from known phases. Beginning in 1913, when W.L. Bragg described the crystal structure of NaCl, the work of determining the crystal structure of inorganic and organic crystals is ongoing without interruption until now because new unknown structures appear in various fields of activity. For a known structure the diffraction pattern can be calculated directly, but the inverse problem, the direct calculation of the network

parameters in the diffraction pattern, does not have a well-defined solution. In fact, the current methodology is essentially a "trial and error" one (Sfetcu, 2019).

The characterization of an unknown structure is carried out in three main stages:

(1) the shape and size of the unit cell are deduced from the angular positions of the diffraction lines. In the first phase, one of the seven crystal systems is chosen - when there is a priori information on the possible structure of the crystalline material, this choice being relatively easier to make - and then, based on this hypothesis, the corresponding Miller indices are assigned to each diffraction line.

(2) The number of atoms per unit cell is then calculated from the shape and size of the unit cell, the chemical composition of the material and the measured density of the material.

(3) Finally, the positions of the atoms inside the unit cell are deduced from the relative intensities of the diffraction lines.

When the three stages have been completed, it can be said that the structure has been fully characterized. The third stage is generally the most difficult to complete, which is why there are many materials (inorganic or organic substances) whose structure is partially completely known, in the sense that this final stage has not been completed. However, knowing the shape and size of the unit cell, without having any data on the position of the atom, is of great value in many applications.

Since the problem of determining the structure is one of finding the structure that will take into account all the lines that make up the model, both in terms of position and intensity, the analyst must ensure from the beginning that the experimentally obtained diffraction pattern does not contain lines from other sources. The ideal diffraction pattern contains lines formed by the diffraction of X-rays of a single wavelength (monochromatic radiation) and which were generated only by the interaction with the analyzed substance. It follows from the above that there are two sources of diffraction lines in addition to those of the investigated substance:

(1) lack of monochromatic radiation, ie radiation with a single wavelength; usually a filtered radiation is used, the main component being  $K_\alpha$ , but it is possible that the filtration does not ensure the total removal of the  $K_\beta$  component, so that additional diffraction lines may occur; however, the presence of diffraction lines due to  $K_\beta$  radiation can be identified by calculation as the diffraction pattern is formed by interaction with the same substance. It follows from the application of Bragg's law that:

$$\left(\frac{\lambda_{K_\alpha}^2}{\lambda_{K_\beta}^2}\right) \sin^2 \theta_\beta = \sin^2 \theta_\alpha \quad (1)$$

where the ratio of wavelengths is close to 1.2 for most radiation sources. If a particular line is suspected to be due to  $K_\beta$  radiation, multiplying the  $\sin(\theta_\beta)^2$  value by the value of the above ratio will lead to a value equal to or nearly equal to the  $\sin(\theta_\alpha)^2$  value for some lines corresponding to the radiation  $K_\alpha$ , unless it exceeds the unit. Another source is the characteristic L radiation, which can be tested and eliminated by the same method. However, it should be noted that the above equation establishes the possibility that a certain line may be due to additional radiation, but this is not sufficient evidence.

(2) the overlap with the diffraction pattern generated by substances other than the one analyzed. These substances are usually 'impurities' or the sample carrier or substance. For this reason, it is necessary to carry out a good preparation of the sample and a careful realization of the experiment of recording the diffraction pattern.

*Indexing of diffraction patterns of crystals in the cubic system* (Gheorghies, 1990). A crystal belonging to the cubic system is characterized by a diffraction pattern containing lines whose values for  $\sin(\theta)^2$  verify the following equation, which was obtained by combining Bragg's law and the equation of interplanar distance for the cubic system:

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

Since the sum  $s = (h^2 + k^2 + l^2)$  is always an integer, and  $\lambda^2/4a^2$  is a constant for any diffraction pattern (for a single phase), the problem of indexing the diffraction pattern of a crystallized substance in the cubic system is one to find a set of integers "s" that will determine a constant coefficient when dividing the values  $\sin(\theta)^2$  in turn. It should be noted that certain values, such as 7, 15, 23, 28, 31, etc., are impossible to obtain due to the fact that they cannot be formed by summing three integers squared. Once the appropriate values for s have been found, the *hkl* indices corresponding to each diffraction line can be identified by testing or from tabulated values.

Each of the four types of cubic networks is recognized by a characteristic sequence of diffraction lines, and these in turn can be described by the sequential values of the size s:

Simple cubic: 1, 2, 3, 4, 5, 6, 8, 9, 10, 11, 12, 13, 14, 16, ....

Cubic with centered volume: 2, 4, 6, 8, 10, 12, .....

Cubic with centered faces: 3, 4, 8, 11, 12, 16, .....

This type of information is also found in graphical form in Fig. 8. From the diffraction lines it is possible to calculate the values of the size s using equation 1, values that are systematized in Fig. 8.

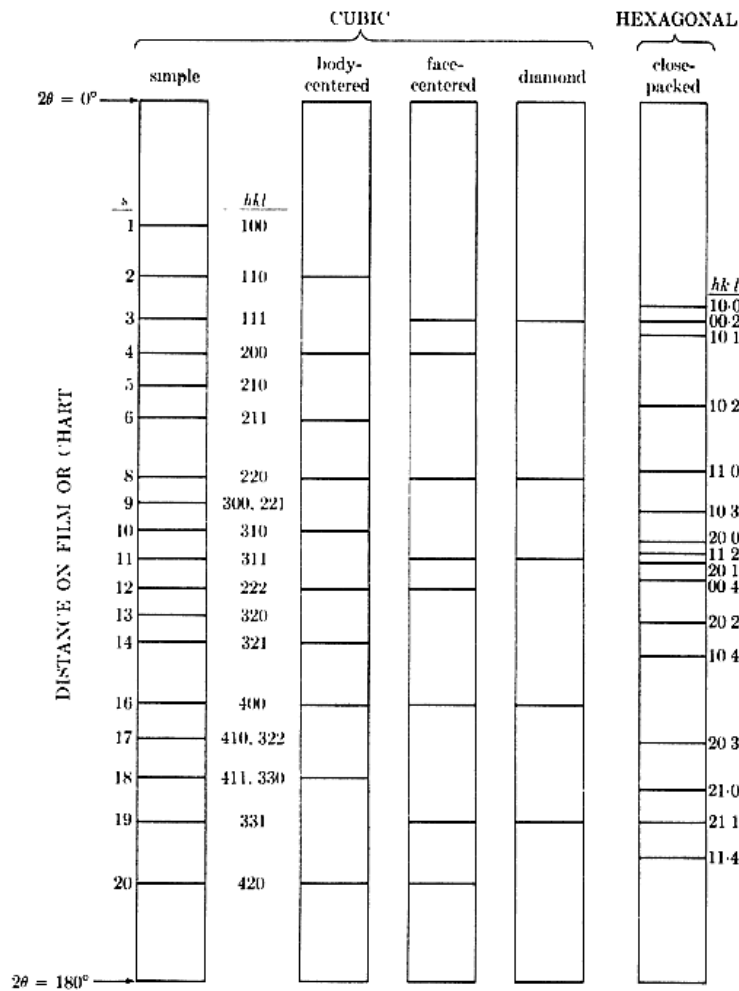


Fig. 8 – Graphically indicated diffraction patterns for some crystal lattice (Li *et al.*, 2007).

In addition to using this indexing method, the following two can be used, which applied to crystallized substances in the cubic system will use the sequential values of size  $s$ , namely: 1) the mathematical method; 2) analytical method (Luca *et al.*, 1985).

*Mathematical method.* Since  $\lambda^2/4a^2$  has a constant value for any diffraction pattern, for any two planes that determine a diffraction line the following relation can be written:

$$\frac{\sin^2 \theta_1}{\sin^2 \theta_2} = \frac{\left(\frac{\lambda^2}{4a^2}\right)(h_1^2 + k_1^2 + l_1^2)}{\left(\frac{\lambda^2}{4a^2}\right)(h_2^2 + k_2^2 + l_2^2)} \quad \text{or} \quad \frac{\sin^2 \theta_1}{\sin^2 \theta_2} = \frac{(h_1^2 + k_1^2 + l_1^2)}{(h_2^2 + k_2^2 + l_2^2)} \quad (3)$$

that is, the values of the ratio between the values of  $\sin(\theta)^2$  are equal to the values of the ratios between the values of the size  $s$ .

In the case of the cubic system, the first diffraction line in the diffraction pattern is due to the planes with the smallest Miller indices, which are the planes with compact packing: simple cubic, (100) with  $s = 1$ , cube with centered volume, (110) with  $s = 1$ , and cube with centered faces, (111) with  $s = 3$ . Since the indices  $h, k, l$  are always integers, values for  $s$  can be obtained by dividing the values  $\sin(\theta)^2$  for all diffraction lines by the smallest value of  $\sin(\theta)^2$  in the diffraction pattern and multiplying this ratio by an appropriate integer (either 1, 2 or 3). This algorithm should generate a list of integers that represent values of size  $s$ . The corresponding Bravais network can be recognized by comparison with the characteristic sequence of diffraction lines presented above for the cubic system.

*Analytical method.* This method is the result of an alternative approach that will lead to a result similar to the mathematical method. It follows from relation 2 that:

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

For the cubic system, regardless of the crystal lattice, the possible values of the size  $s$  correspond to the following sequence of numbers: 1, 2, 3, 4, 5, 6, 8, 9, 10, 11 .... If for each diffraction line calculate  $\sin(\theta)^2$  and the values are divided by the integers 2, 3, 4, 5, 6, 8, ... we can obtain the value of a common constant, which is the value of  $K$  that corresponds to the value  $s = 1$ . Knowing the value of  $K$ , by dividing the values of  $\sin(\theta)^2$  by the value of  $K$  we obtain the values of  $s$ , which can be used to identify the characteristic sequence of diffraction lines.

#### 4. Conclusions

The following ideas arise from summarising the arguments presented:

- With the development of the field of phase analysis, a priori information regarding a large number of phases regarding the crystal structure is known. There are several databases, both commercial and open access, for either inorganic or organic substances. These include: Powder Diffraction File (PDF) managed by the International Center for Diffraction Data, Inorganic Crystal Structure Database (ICSD) managed by FIZ Karlsruhe-Leibniz Institute for Information Infrastructure, Crystallography Open Database managed by the Department of Chemistry of the University of Cambridge, which contains data on both organic and inorganic substances except biopolymers (<https://icsd.products.fiz-karlsruhe.de/>).

- The structural analysis of metal alloys is based on phase diagrams which were also built on data accumulated over time, and today there is specialized software for calculating phase diagrams, for example ThermoCalc Software produced by the company of the same name. Until the advent of computer programs, phase diagrams were described in various printed collections, for example ASM Handbook Volume 3: Alloys Phase diagrams.

- The methods described above are applicable when the diffraction pattern is obtained for a single phase. When the material contains at least two phases, which differ at least in the size of the unit cell, things become more complicated. This type of investigation is specific to metal alloys, which often contain at least two crystalline phases. In this case another method is frequently used, namely that by which the experimentally obtained diffraction pattern is compared with known patterns of possible phases to form in a given alloy. Depending on the chemical composition of the alloy, the existing databases identify diffraction patterns of possible phases and assign them to a certain sequence of diffraction lines in the diffraction pattern of the alloy. This is done until all diffraction lines can be mapped to the diffraction pattern of a phase in the database. After identifying the phases, the corresponding diffraction patterns will be indexed.

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## PREZENTARE GENERALĂ ASUPRA METODELOR DE ANALIZĂ STRUCTURALĂ A MATERIALELOR METALICE UTILIZÂND DIFRAȚIA CU RAZE X

(Rezumat)

Alături de compoziția chimică, structura cristalină și morfologia fazelor determină în mare măsură comportamentul (răspunsul) materialelor la diferite „solicitări” externe, care pot fi mecanice, electrice, magnetice, chimice. Majoritatea materialelor de uz industrial (metal, ceramică, plastic) nu sunt utilizate sub forma unui singur cristal ci sub formă de agregate policristaline, formate din multe cristale mici, de obicei mai mici de 1 milimetru, dar care pot ajunge până la dimensiuni nanometrice. Identificarea structurii unui material nu este un proces banal de investigație, mai ales având în vedere amploarea la care ar trebui efectuată investigația. O metodă importantă pentru un astfel de studiu este cea cunoscută sub numele de „analiza structurii difracției cu raze X”. Scopul inițial al difracției cu raze X a fost și rămâne să identifice structura unui material necunoscut. Acest tip de analiză este mult mai favorabil pentru analiza monocristalelor diferitelor substanțe, dar pentru analiza materialelor policristaline se folosește varianta cunoscută sub numele de „metoda pulberii”. Analiza structurală prin difracție de raze X se bazează pe difracția de raze X de către planurile atomice din interiorul cristalelor. Rezultatul difracției este așa-numitul model de difracție care este vizualizat sub formă grafică. Acest „model de difracție” are ca principale caracteristici „vârfurile” sau „maxima difracției”, care reprezintă unghiurile (exprimate ca  $2\theta$ ,  $\theta$  fiind unghiul exprimat în grade) la care se obțin maximele de difracție.