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X-ray Diffraction

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Welcome

MyScope was developed by Microscopy Australia to provide an online learning environment for those who want to learn about microscopy. The platform provides insights into the fundamental science behind different microscopes, explores what can and cannot be measured by different systems and provides a realistic operating experience on high end microscopes.
We sincerely hope you find our website: www.myscope.training an enjoyable environment. In there you can explore the microscopy space and leave ready to undertake your own exciting experiments.

Module Contents

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Glancing Angle XRD
Glancing angle XRD

Credits

Introduction to XRD - XRD basics

X-ray diffraction (XRD) uses X-rays to investigate and quantify the crystalline nature of materials by measuring the diffraction of X-rays from the planes of atoms within the material. It is sensitive to both the type of and relative position of atoms in the material as well as the length scale over which the crystalline order persists. It can, therefore, be used to measure the crystalline content of materials; identify the crystalline phases present (including the quantification of mixtures in favourable cases); determine the spacing between lattice planes and the length scales over which they persist; and to study preferential ordering and epitaxial growth of crystallites. In essence it probes length scales from approximately sub angstroms to a few nm and is sensitive to ordering over tens of nanometres.

Different disciplines sometimes have different names for this technique:

- Materials science : XRD (X-ray diffraction)
- Chemistry : PXRD (powder XRD)
- Physics : WAXS (wide angle X-ray scattering)

Samples analysed using XRD are typically in the form of finely divided powders, but diffraction can also be obtained from surfaces and bulk specimens. Applications of XRD include:

Pharmaceutical industry

XRD is the key technique for solid-state drug analysis, benefiting all stages of drug development, testing and production. Many pharmaceuticals are crystalline in nature, and XRD is used to unambiguously characterize them. For example, once an active drug has been isolated, an indexed X-ray powder diffraction pattern is used to identify the crystal structure, secure a patent and protect the company's investment. For multi-component formulations, the percentages of the active ingredients in the final dosage form can be accurately quantified, along with the percentage of any amorphous packing ingredients used.

Forensic science

XRD is used mainly in contact trace analysis. Examples of contact traces are paint flakes, hair, glass fragments, stains of any description and loose powdered materials. Identification and comparison of trace quantities of material can help in the conviction or exoneration of a person suspected of involvement in a crime.

Geological applications

XRD is the key tool in mineral exploration. Mineralogists have been amongst the foremost to develop and exploit the field of X-ray crystallography after its discovery. Thus, the advent of XRD has literally revolutionized the geological sciences to such a degree that they have become unthinkable without this tool. Nowadays, any geological group actively involved in mineralogical studies would be lost without XRD to unambiguously characterise the individual crystal structures within a specimen. Each mineral type is defined by a characteristic crystal structure, which will give a unique X-ray diffraction pattern, allowing rapid identification of minerals present within a mineral sample. The XRD data can be quantified to determine the proportion of the different minerals present.

Glass industry

While glasses are X-ray amorphous and do not themselves give X-ray diffraction patterns, there are still manifold uses of XRD in the glass industry. They include identification of crystalline particles which cause tiny faults in bulk glass, and measurements of crystalline coatings for texture, crystallite size and crystallinity.

Materials Science

Diffraction is used in the materials science discipline to examine crystal size, quantify microstrain, determine the dislocation density, measure crystallographic preferred orientation, quantify the volume fraction of phases within a specimen and also to determine the crystal structure of unknown samples such as corrosion products and new materials.

Interesting facts

Did you know that the SEM in the EDX-mode operates as a giant X-ray tube with your sample as the anode material?

Wilhelm Konrad Rontgen discovered X-rays in 1895?

Mayor John Hall Edwards pioneered X-ray photography for medical purposes. He took the first X-ray in Birmingham in 1896.

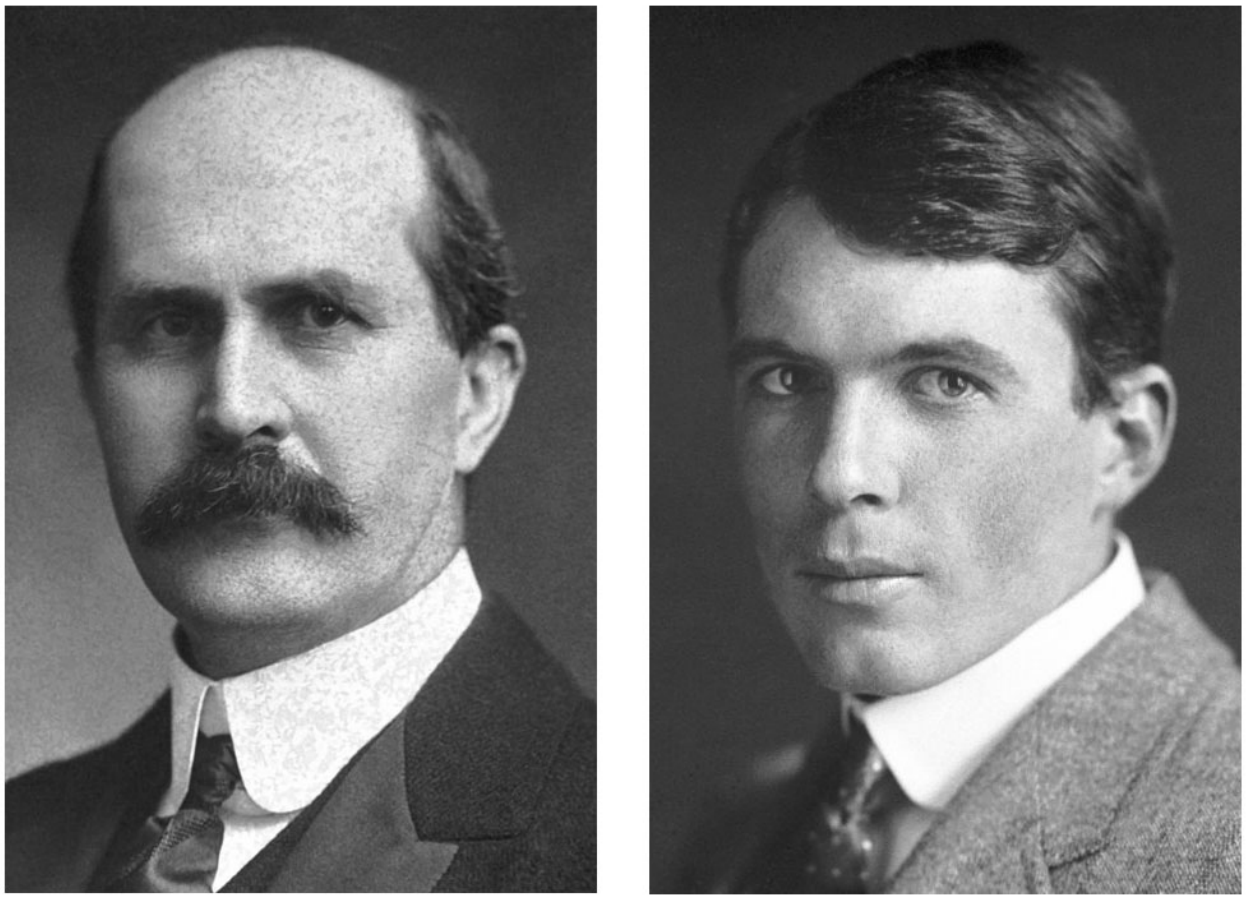
The refractive index of X-rays in most materials is slightly lower than one. A collimating lens for visible light therefore has the opposite effect on X-rays.

Did you know that early X-ray cameras were a popular attraction during parties?

The first crystal structure ever solved by X-ray diffraction was the one of zinc sulfide (W. H. Bragg 1912).

The first crystal structure solution from powder was performed in 1916 by P. Debye and P. Scherrer (LiF).

Sir William Lawrence Bragg was the youngest person ever (25) when he was awarded the Nobel Prize in Physics 1915. He received the prize together with his father Sir William Henry Bragg for “their services in the analysis of crystal structure by means of X-rays”, work carried out at The University of Adelaide.



Sir William Henry Bragg (left) and his son, Sir William Lawrence Bragg (right). Nobel foundation / Public domain.

Nobel prizes for research with X-rays

- 1901 W. C. Roentgen in Physics for the discovery of X-rays.
- 1914 M. von Laue in Physics for X-ray diffraction from crystals.
- 1915 W. H. Bragg and W. L. Bragg in Physics for crystal structure derived from X-ray diffraction.
- 1917 C. G. Barkla in Physics for characteristic radiation of elements.
- 1924 K. M. G. Siegbahn in Physics for X-ray spectroscopy.
- 1927 A. H. Compton in Physics for discovery of wavelength change in diffused X-rays.
- 1936 P. Debye in Chemistry for work on dipole moments and diffraction of X rays and electrons in gases.
- 1985 H. Hauptman and J. Karle in Chemistry for direct methods to determine X-ray structures.

Background information - X-rays Overview

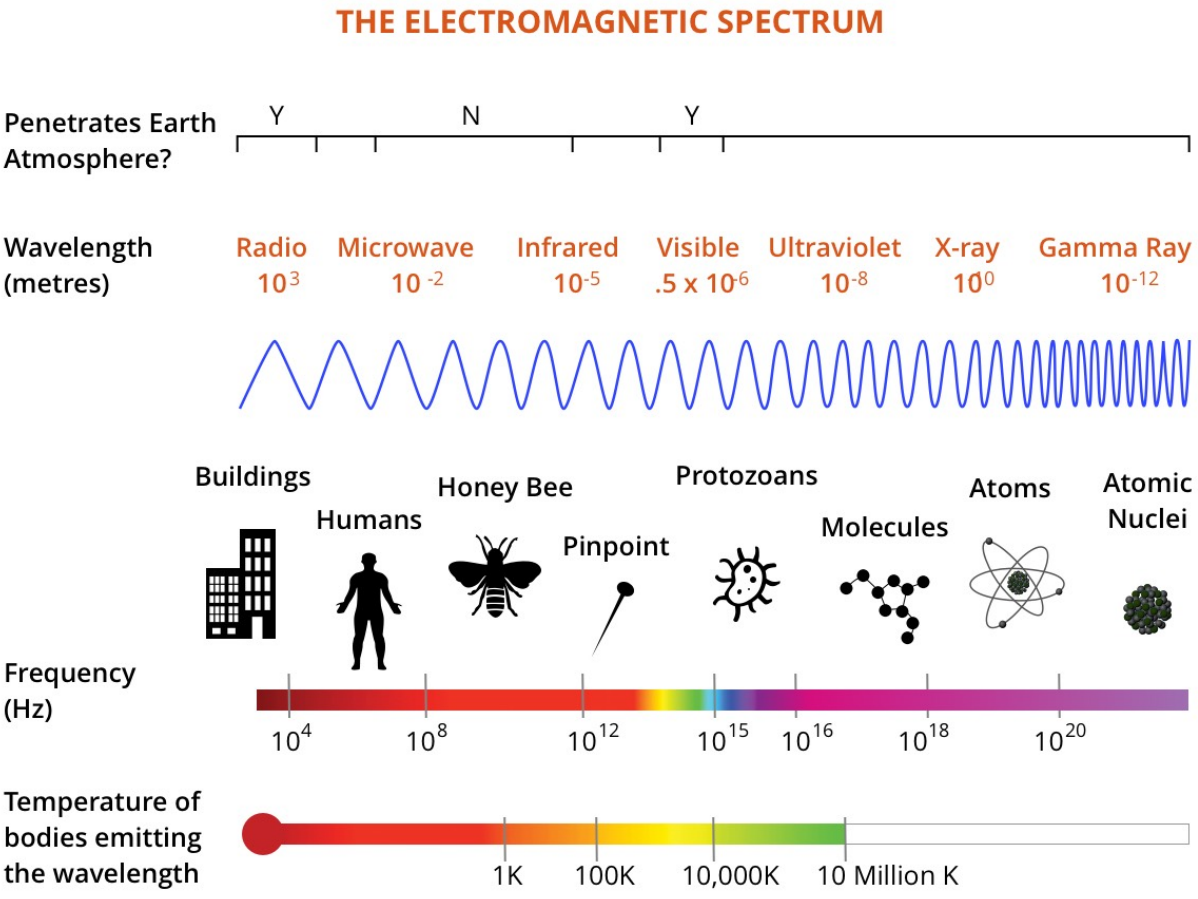
X-ray diffraction is a tool for the investigation of the structure of matter. X-rays are scattered by interaction with the electrons of the atoms in the material being investigated. The technique began when von Laue discovered that crystals diffract X-rays in 1912. Since then it has been applied to chemical analysis, stress and strain measurement, the study of phase equilibria, measurement of particle size, as well as crystal structure.

To describe a material uniquely, generally two pieces of information are required: what are the elements present and how are they arranged. The first question is usually answered by chemical analysis techniques and gives the ratio of the elements present in the material. This is a chemical formula often called the chemical stoichiometry. The chemical formula does not describe how the atoms are arranged in space or with respect to each other. For example, the material calcium carbonate has the formula $CaCO_3$, but can naturally occur with two different atomic arrangements. These atomic arrangements are called crystal structures. The different structures for the same chemical formula are called polymorphs. Calcium carbonate's two most common crystal structures are orthorhombic and trigonal-rhombohedral. The first material is the mineral aragonite and the second is calcite. X-ray diffraction can be used to tell different crystal structures apart.

The following three sections summarise the important background information you need to know to understand XRD: the properties of X-rays; the geometry of crystals; and how X-rays are diffracted by crystal planes.

Properties of X-rays

X-rays were discovered in 1895 by German physicist Roentgen. The typical wavelength of X-rays is 1 x 10^{-10}m (1 angstrom), whereas the wavelength of visible light is typically 1 x 10^{-6}m (1 μm).

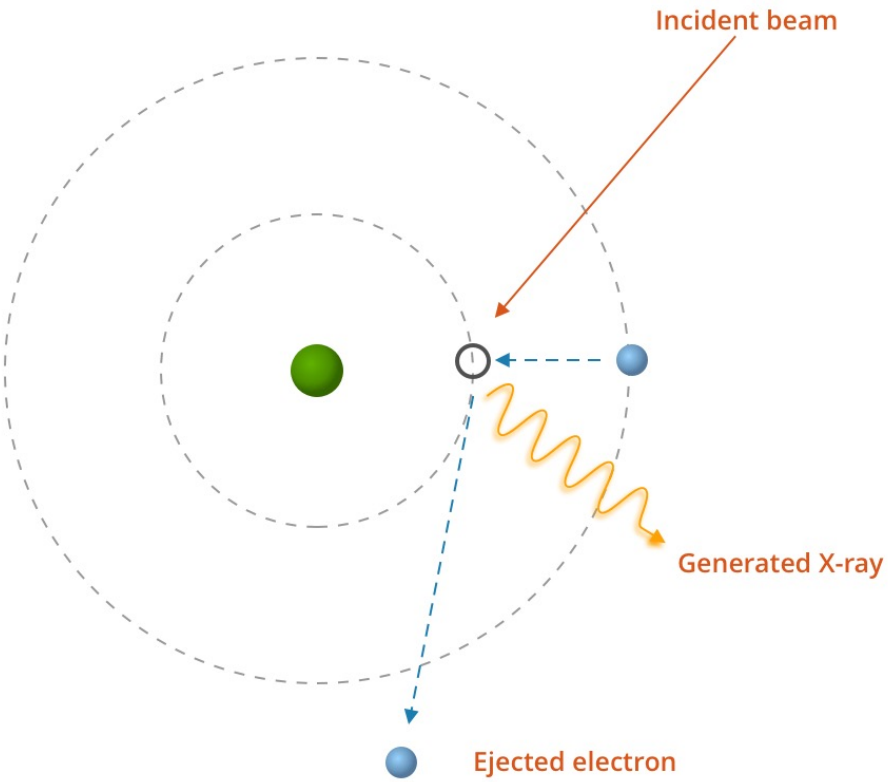


	NEUTRONS	X-RAYS	ELECTRONS
Wavelength range	0.4 - 10 Å	0.1 - 5 Å	0.04 - 0.2 Å
Energy range	0.001 - 0.5 eV	3000 - 100000 eV	6000 - 120000 eV
Cross-section	10^{-2} barns	10^{-25} Z^{-2} barns	~10^{-22} barns
Penetration depth	~ cm	~ µm	~ nm
Typical flux	10^{24} s^{-1} m^{-2}	10^{24} s^{-1} m^{-2}	10^{26} s^{-1} m^{-2}
Beam size	mm-cm	µm-mm	nm-µm
Typical sample	Any bulk sample	Small crystals, powder surfaces	Surfaces, thin films, grains, gases
Techniques	Diffraction Inelastic scattering Reflectivity	Diffraction, Photon absorption, Photoemission, Inelastic scattering	Microscopy, Diffraction, Emission spectroscopy, EELS
Phenomena	Magnetic / crystal structures Collective excitations (phonons, spin waves) Electronic excitations (crystal-field, spin-orbit)	Crystal structures, Electronic transitions (photoemission, absorption)	Microstructures, Crystal structures, Electronic transitions

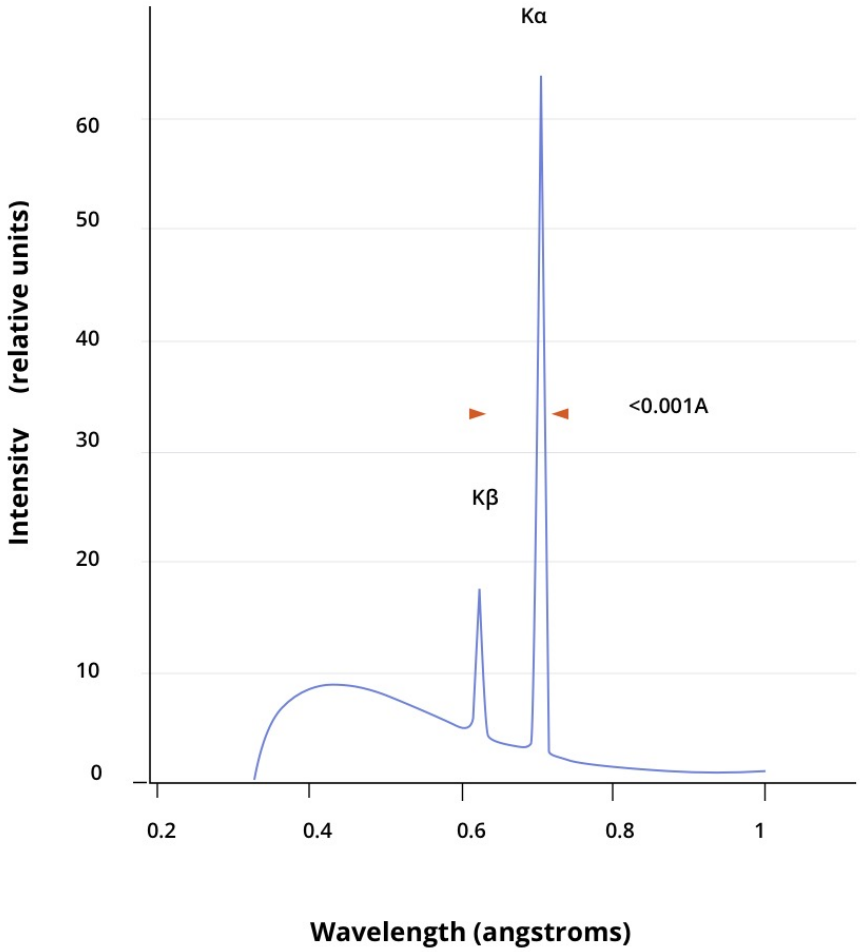
Production of X-rays

X-rays are produced by the deceleration of charged particles, usually electrons. We generate X-rays by bombarding a target material with high energy electrons. The decelerating electrons produce a continuous spectrum of energies when they hit the target called the 'bremsstrahlung' which is German for 'braking radiation' as it is produced by stopping electrons.

If the incident electrons have sufficient kinetic energy, it can knock an electron out of its shell, leaving the atom in a high energy state. One of the outer electrons immediately falls into the lower energy empty shell. Since the falling electron moved from a higher energy shell to a lower energy shell, this difference in energy is emitted as a phonon which has an energy characteristic of the electron shell. This creates what we refer to as the "characteristic spectrum" of the target material.

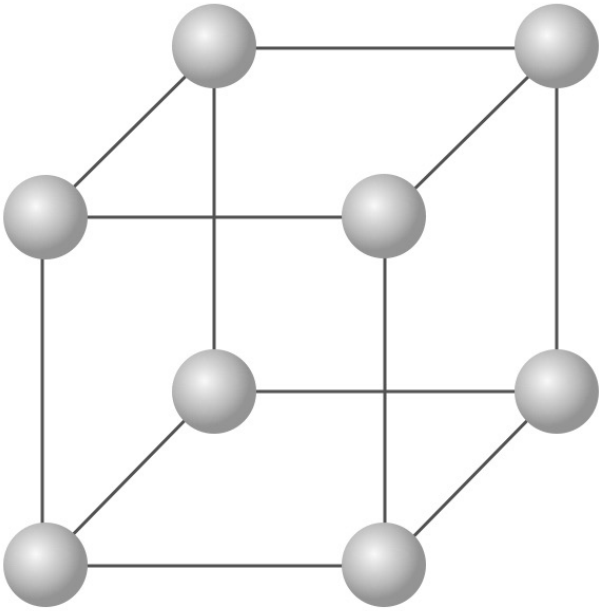


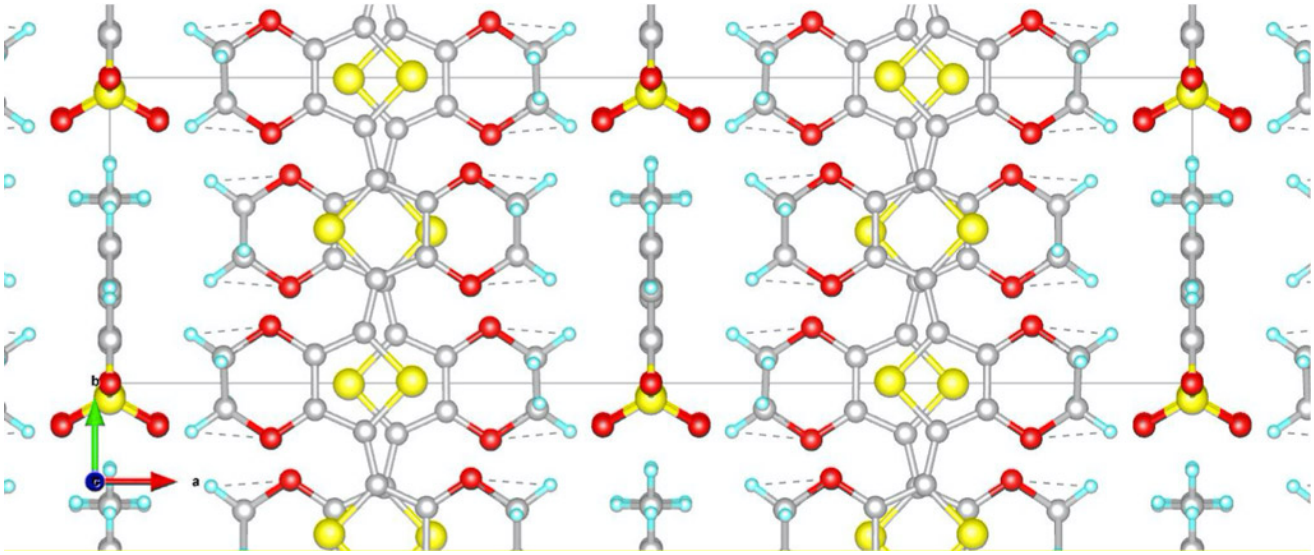
In XRD we usually use the K alpha peak, shown below. Due to the quantum mechanics of electron energies and their shell arrangement, we find a typical shape to the characteristic peaks where the most intense peak is the K alpha 1, and this is accompanied by a smaller K alpha 2 peak which is approximately half the intensity of the K alpha 1.


Kα
60
50
40
30
20
10
0
Intensitv (relative units)
<0.001A
Kβ
0.2
0.4
0.6
0.8
1
Wavelength (angstroms)

The geometry of crystals - Crystal structure

A crystal is a solid composed of atoms arranged in a periodic pattern. Solids that do not contain order, such as glass, are referred to as amorphous. Many polymers and geological specimens are a mixture of crystalline and amorphous phases.





Example of a crystalline polymer. This structure is for a polymer known as PEDOT, and contains carbon, Sulphur, oxygen and iron. Structure courtesy of Reza Mahjoub and Drew Evans, University of South Australia.

It is easier to represent and visualise crystals as an array of imaginary points known as a lattice. There is a limited number of different point lattice constructions possible, and these are known as the seven crystal systems. Within these seven systems, there are additional locations where atoms can be located, and these variants produce the 14 Bravais lattices.

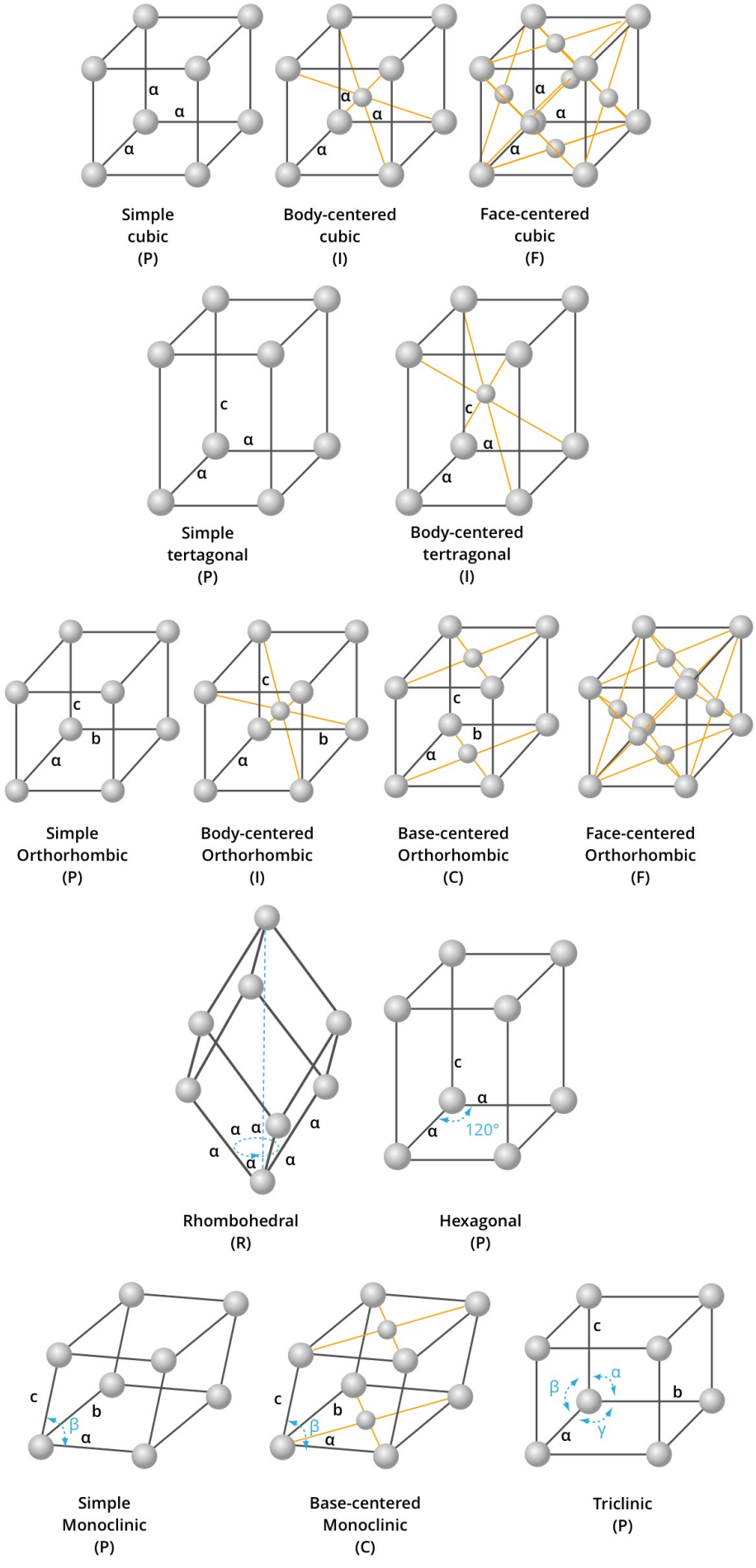


Figure of the 14 Bravais lattices.

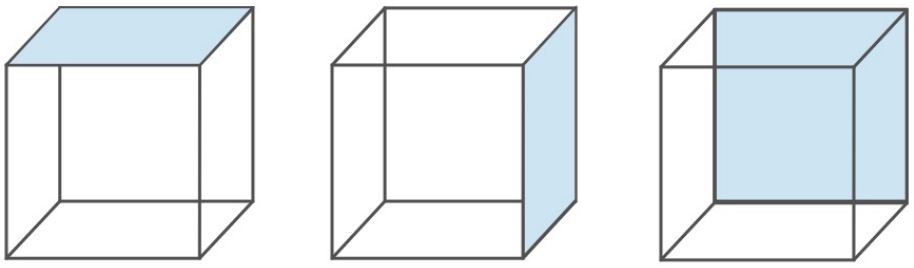
SYSTEM	AXIALS LENGTHS AND ANGLES	BRAVAIS LATTICE	LATTICE SYMBOL
Cubic	Three equal axes at right angles $a = b = c,\ \alpha = \beta = \gamma = 90°$	Simple Body-centered Face-centered	P I F
Tetragonal	Three axes at right angles, two equal $a = b \neq c,\ \alpha = \beta = \gamma = 90°$	Simple Body-centered	P I
Orthorhombic	Three unequal axes at right angles $a \neq b \neq c,\ \alpha = \beta = \gamma = 90°$	Simple Body-centered Bace-centered Face-centered	P I C F
Rhombohedral*	Three equal axes, equally inclined $a = b = c,\ \alpha = \beta = \gamma = 90°$	Simple	R
Hexagonal	Two equal coplanar axes at 120°, third axis at right angles $a = b \neq c,\ \alpha = \beta = 90°,\ \gamma = 120°$	Simple	P
Monoclinic	Three unequal axes, one pair not at right angles $a \neq b \neq c,\ \alpha = \gamma = 90° \neq \beta$	Simple Bace-centered	P C
Triclinic	Three unequal axes, unequally inclined and non at right angles $a \neq b \neq c,\ \alpha \neq \beta \neq \gamma \neq 90°$	Simple	P

Table of the 7 crystal structures and 14 Bravais lattices.

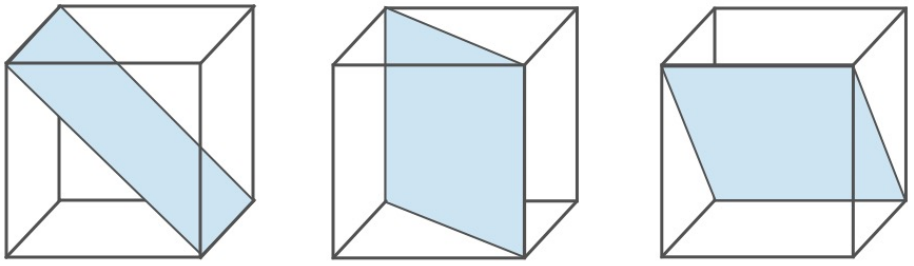
Miller Indices

To discuss the seven crystal systems in detail, we have to establish some understanding of crystal geometry. The notation used to describe crystals is known as “Miller indices”. Before William H. Miller (1801-1880) devised this mathematical system for describing any crystal face or group of similar faces (forms), there was a considerable amount of confusion due to the many different descriptive systems. Miller applied relatively simple mathematics to the problem, and his notation has become the universal language for all crystallography.

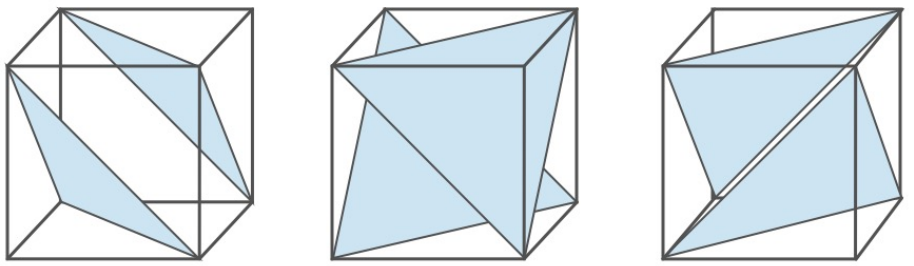
The Miller indices of a cubic structure are shown below, and it can be seen that the planes are described by three whole numbers, fractions are not allowed. To determine the Miller indices of a plane, we first determine where the plane crosses each intercept. We take the reciprocal of each, and convert them to whole numbers. Negative indices are described by a bar on top of the integer, and planes are described as three integers surrounded by round brackets. Families of planes, for example, (100) (001) and (010) can be described collectively by using a different type of bracket {100}.



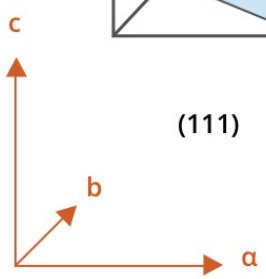
(001) (100) (010)

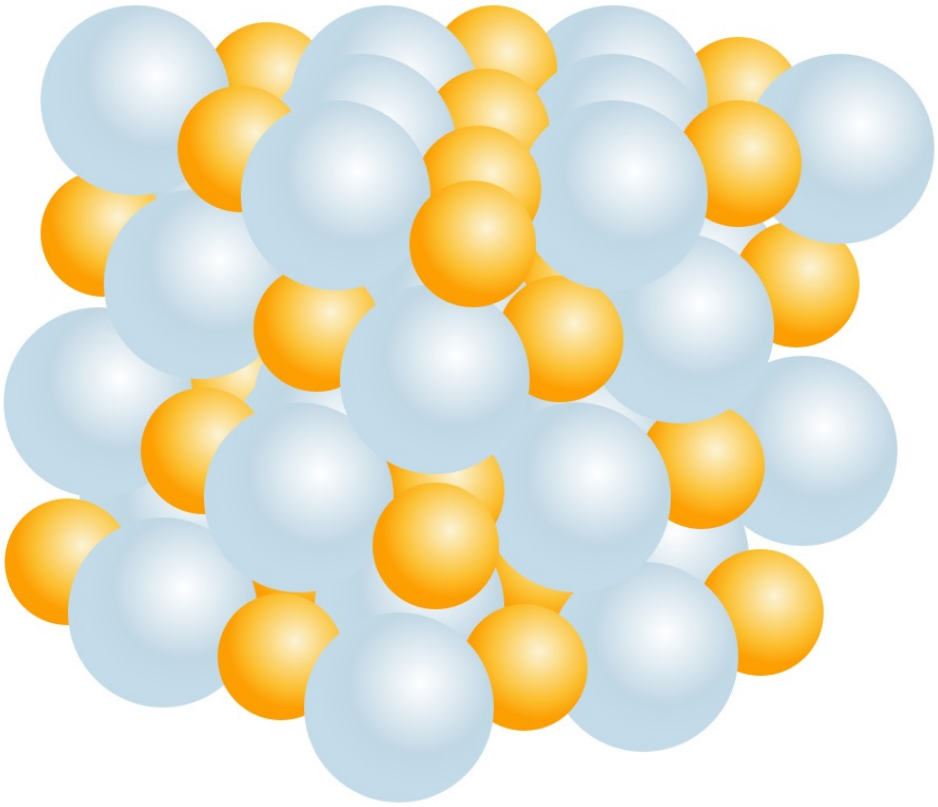


(101) (110) (011)

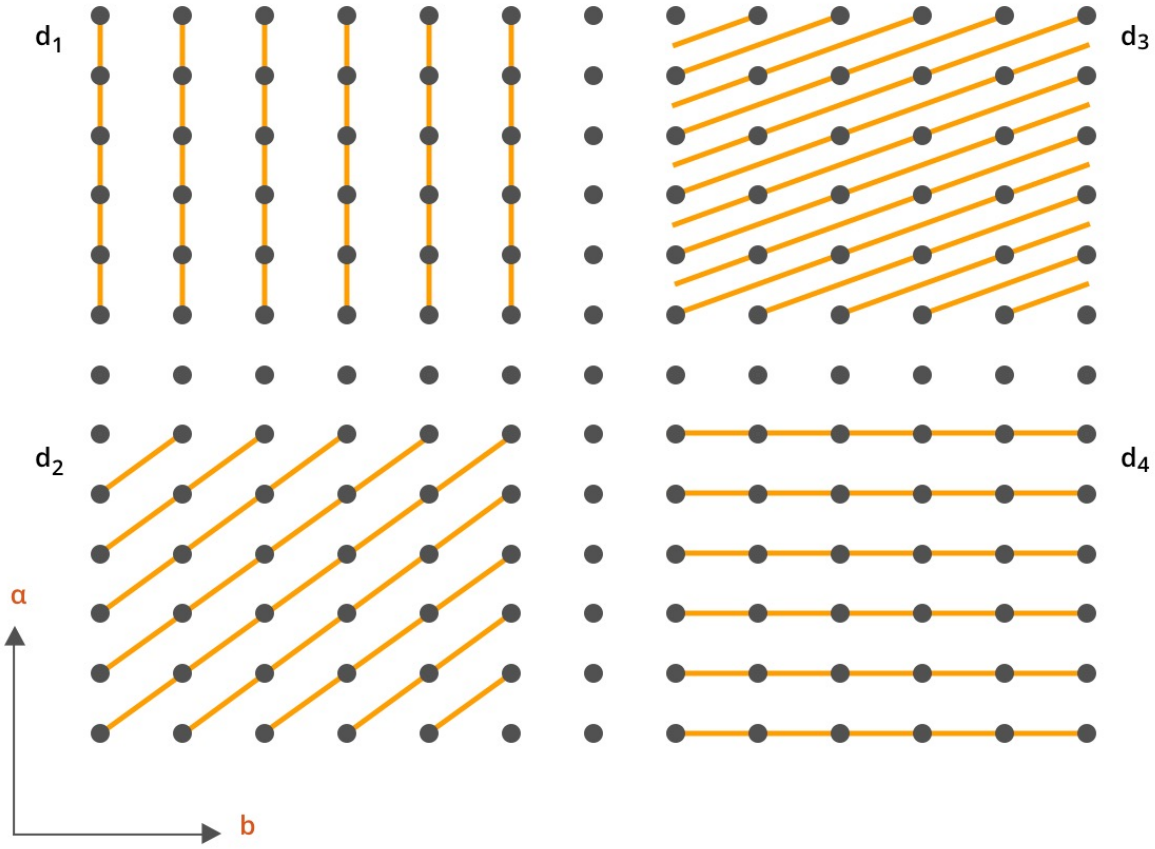


(111) ($1\bar{1}1$) ($\bar{1}11$)



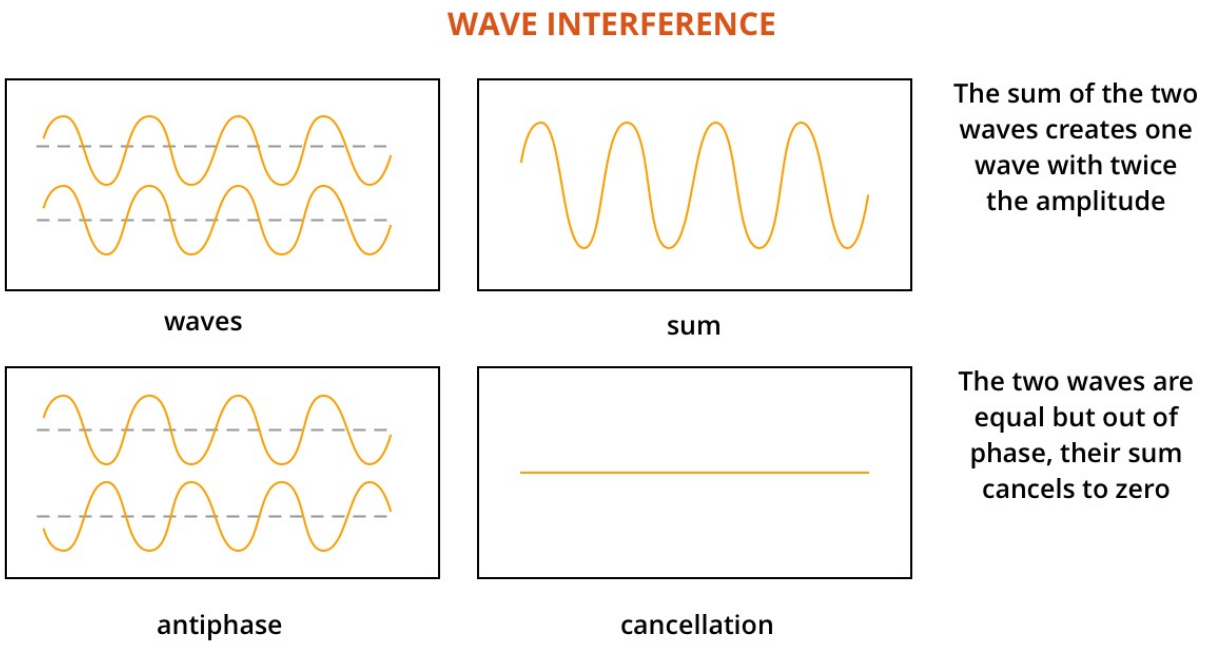


Diffracting planes in a stylized 2-D crystal lattice

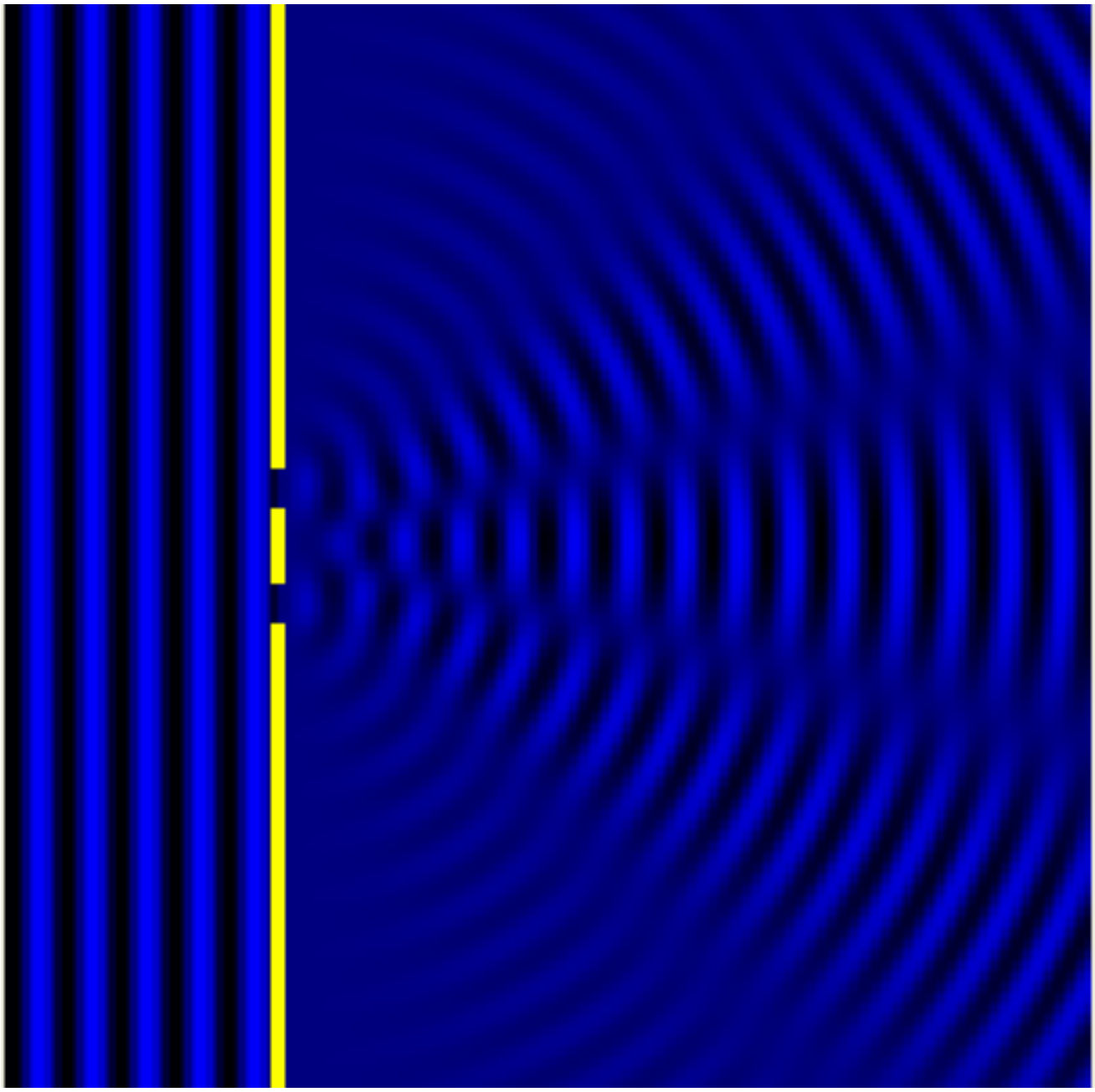


Principles of diffraction - Wave structure

The origin of diffraction is the constructive and destructive interference of waves. Two waves are said to be “in phase” if their peaks and troughs are at the same location. If two waves are in phase, they can sum to make one wave with a larger amplitude. This is called constructive interference. If the waves are not in phase they will sum to make a wave of smaller amplitude, and this is called destructive interference.



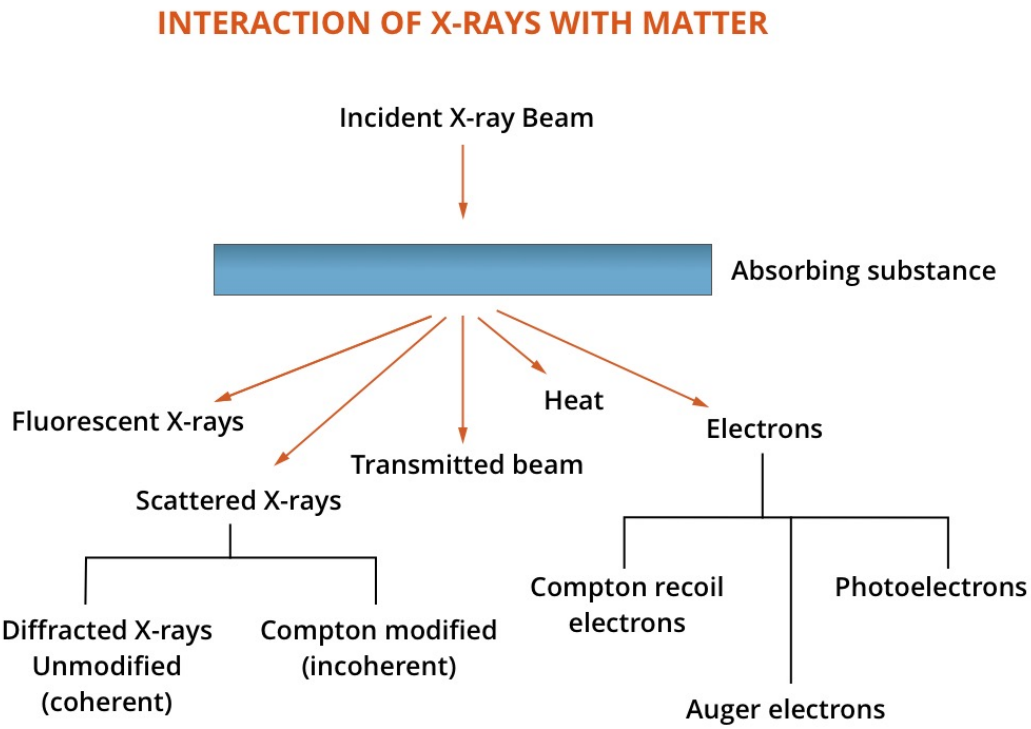
This type of waveform interaction can be seen in all wave systems. The image to the right shows a simulated wave pattern where the lattice is placed on its side and two slits are present. The interfering wave propagation can be seen amplifying three waves. A crystal lattice can be thought of as a source of multiple tiny slits, where the X-ray beam is acting like the waveform in the image.



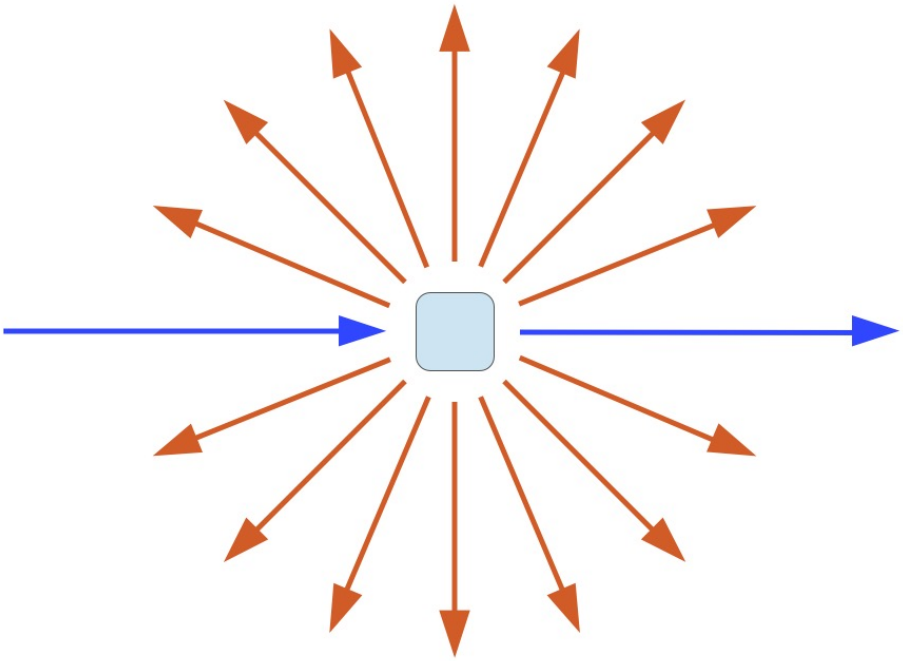
Generation of an interference pattern from two-slit diffraction. Image by Lookang.

Interaction of X-rays with matter

Just like electrons, when an X-ray hits the specimen, a number of interactions are possible. For X-ray diffraction, the wavelength of the incoming incident beam and the diffracted beam must have the same wavelength for constructive interference (diffraction) to occur. Therefore, only the elastic interactions, those which conserve energy, contribute to the diffracted signal that we use in XRD. However, there are many other interactions of the X-ray beam with the specimen. Inelastic scattering such as fluorescence and Compton scattering can be picked up by X-ray detectors, and in this way they contribute to the background that is observed in all lab-scale diffraction measurements.

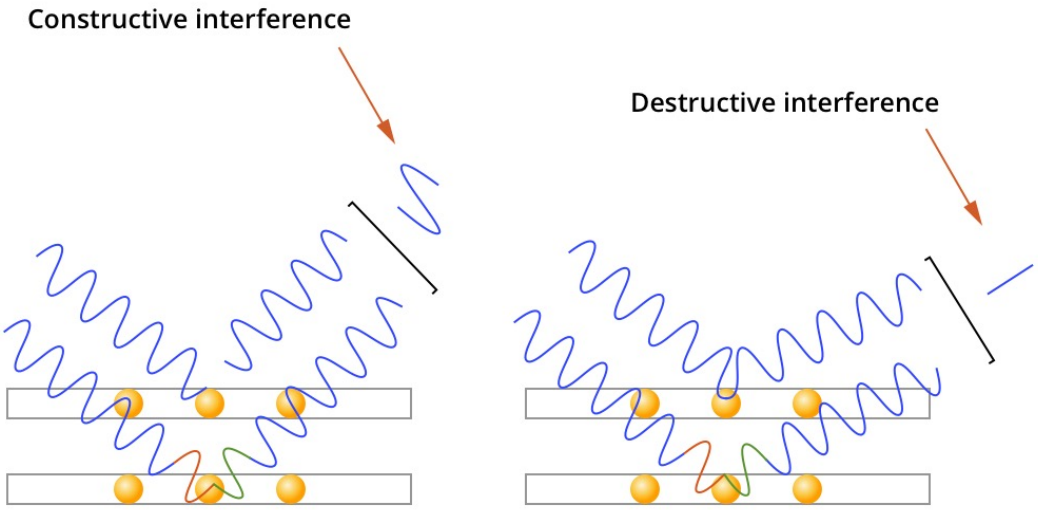


The scattering of X-rays comes from the electron cloud, and scattering occurs in all directions.



Diffraction of X-rays by a crystal

When an X-ray hits an atom, the X-rays interact with the electron cloud and become scattered in all directions. The construction below shows that the wave scattered by the lower plane has travelled a longer distance than the parallel wave scattered by the upper plane. Depending on the angle and the difference in distance travelled, these waves will sum to give either constructive of destructive interference.

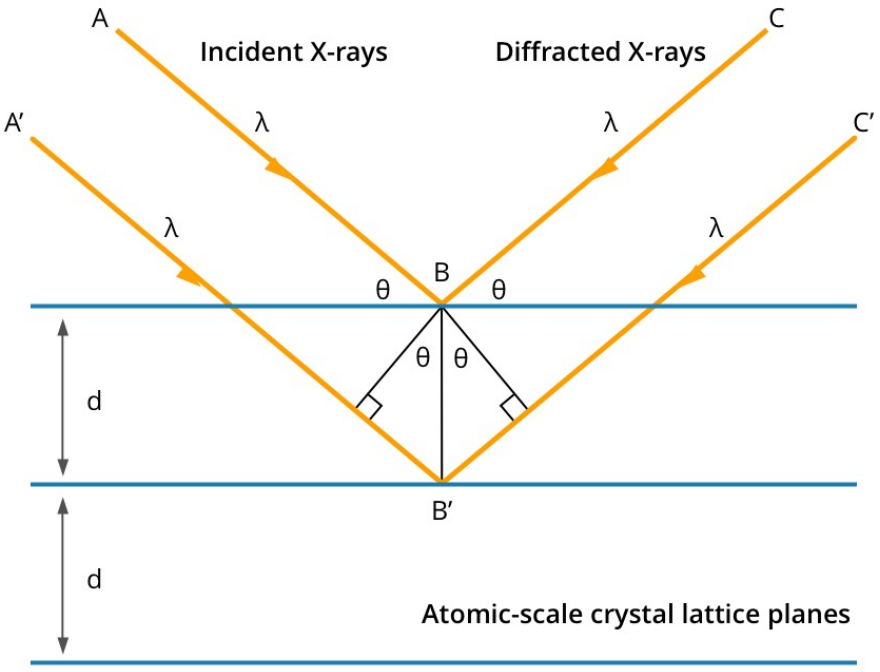


At certain angles to a crystal plane, the X-rays will constructively interfere (diffract) and produce a diffracted beam with a much higher intensity that at other angles. This is described by Bragg's Law:

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

Where λ = wavelength of x-rays
d = spacing between the crystal planes
θ = angle of incident rays

where n is an integer, λ is the wavelength of the X-rays (in our case 1.54Å for a copper tube source), d is the spacing between planes in the atomic lattice of the sample, and θ is the diffraction angle in degrees.



Bragg's law can be easily derived through simple geometry, and is based on determining at what angle the X-rays will be in phase, and produce a constructive wave. It is clear from Bragg's law that to study plane spacing, we need a single wavelength, and this is why lab-based instruments are usually monochromated.

Penetration depth

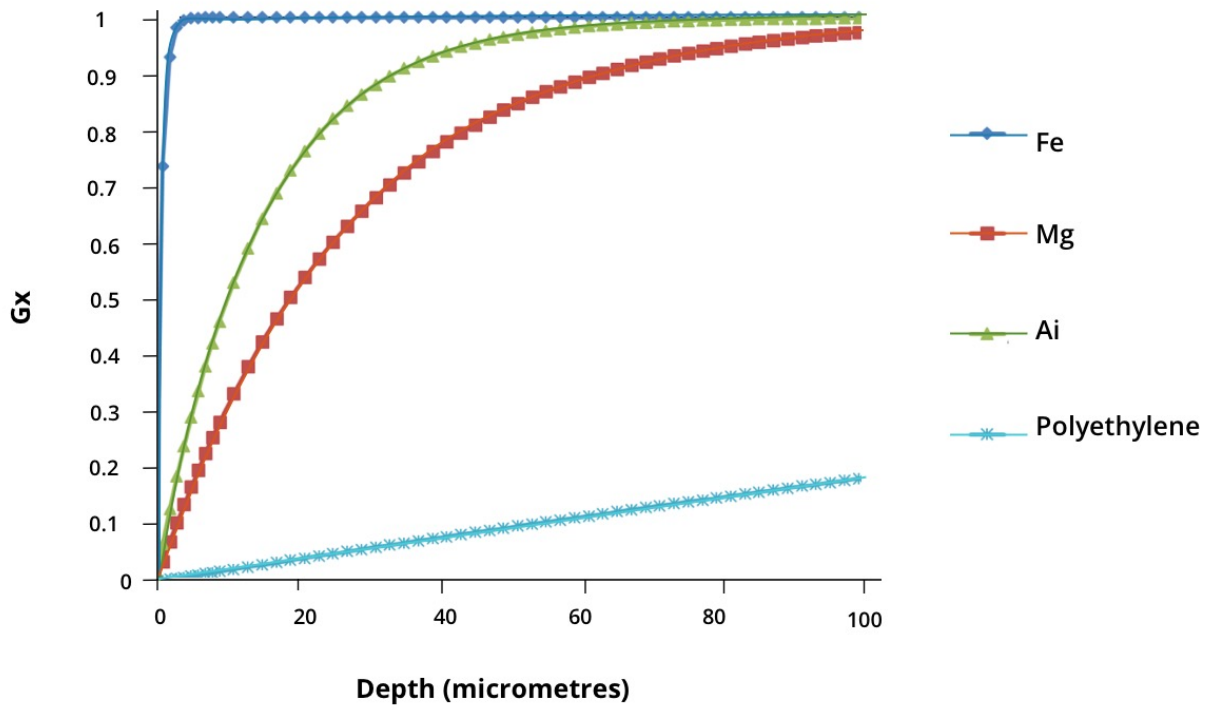
In many solid samples, particularly metallurgical ones, the X-rays are readily absorbed by the specimen. Therefore, there is only a thin layer at the top of the specimen from which diffraction can occur. It is therefore useful to know from what depth the diffracted X-ray information is coning. The depth from which we can get diffraction information is a function of the linear absorption coefficient (μ) and the angle of incidence of the X-ray beam with the surface. The diffracted intensity, G_x, can readily be calculated for different materials using the following relationship:

$$G = (1^x - e^{-2\mu x/\sin\theta})$$

Where x = distance
θ = angle of incidence of the X-rays beams in radians

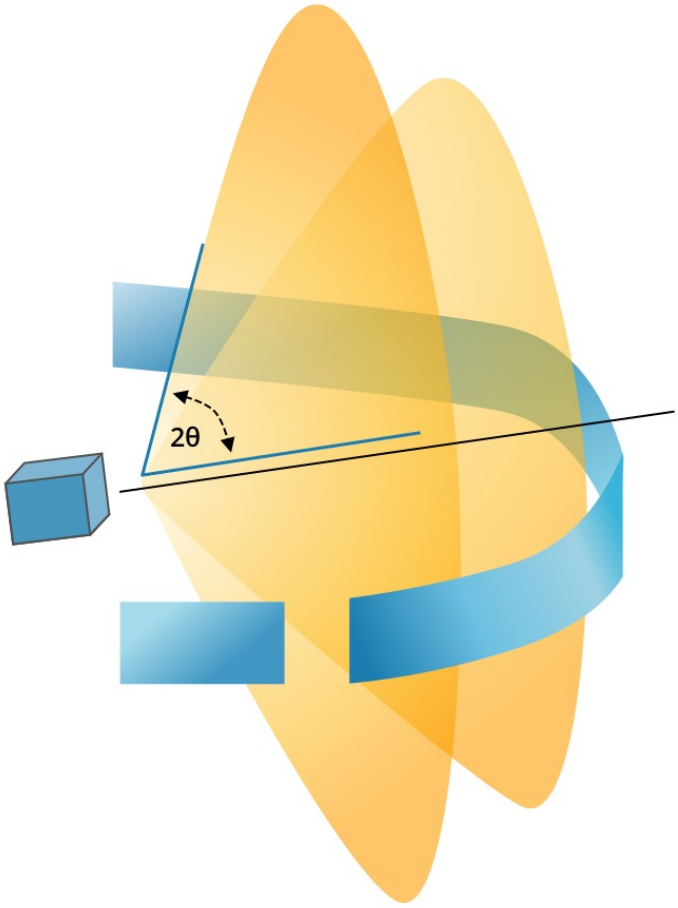
In this equation x is the distance, and θ is the angle of incidence of the X-ray beam in radians. The linear absorption co-efficient is measured in units of cm^{-1}, and can be found in reference books for many materials. Note that μ is a function of the energy of the incoming beam, and this must be taken into consideration when looking up reference tables for such values.

This equation has been used to plot the diffracted intensity for a number of different materials in the figure below. As can be seen, diffraction from steel occurs within just a few micrometres of the surface, while we can obtain diffraction information from polymers for tens of micrometres below the surface.



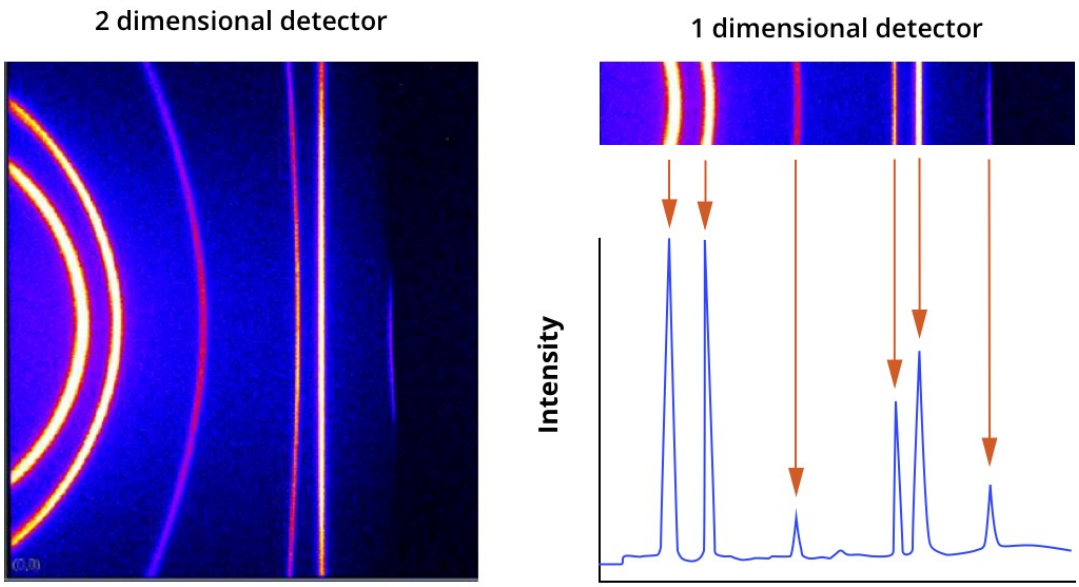
Diffraction measurements

As we saw in the previous section, the X-rays are scattered in all directions by the electron cloud. The constructive interference of these scattered rays collectively produce the diffracted beam. When considered in all three dimensions, the diffracted volume can be best visualised as a cone shape, and diffraction will occur in all directions where the Bragg law is satisfied.

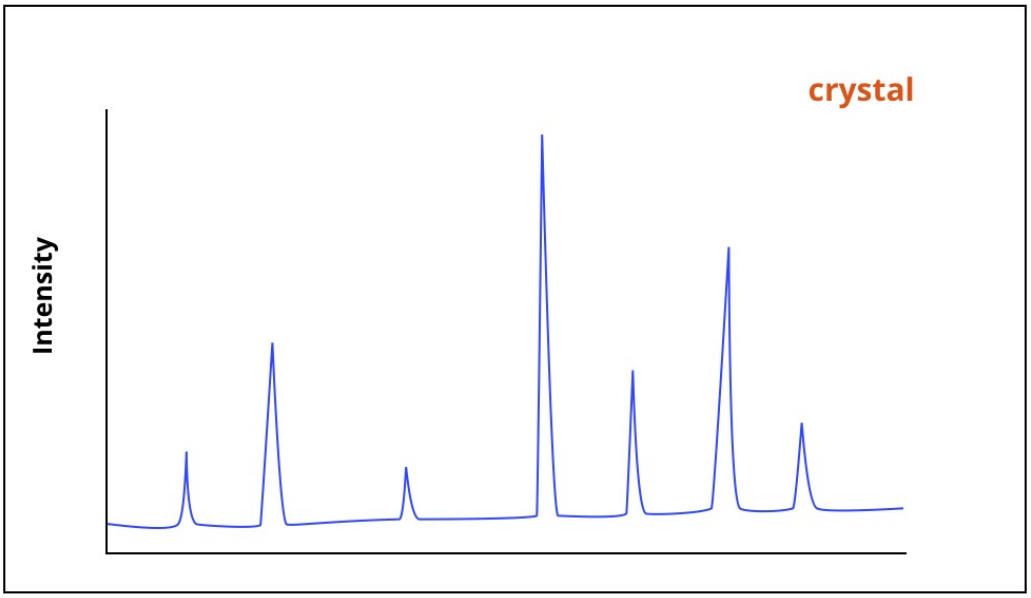


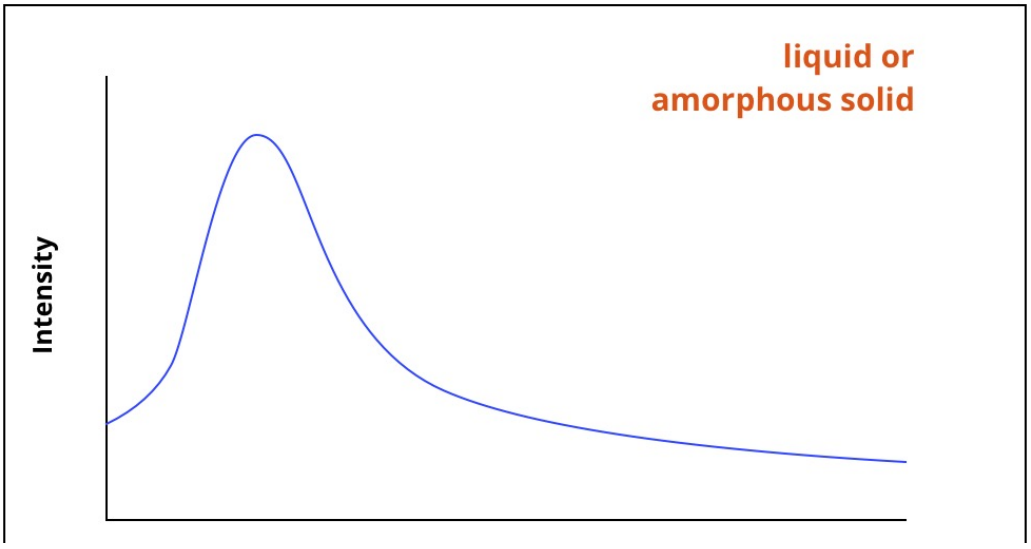
In some specialised lab-scale instruments, and in most synchrotrons and neutron diffraction facilities, the entire cone of diffraction is measured using a 2-dimensional detector.

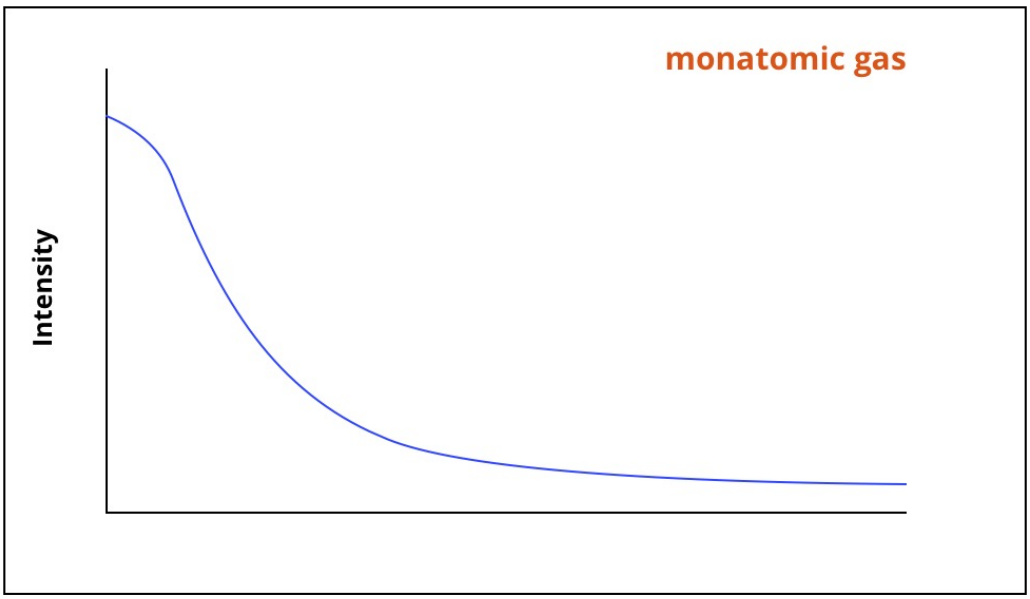
However, a majority of lab-scale instruments use a 1-dimensional line detector, and the data is viewed as a plot of angle versus intensity.

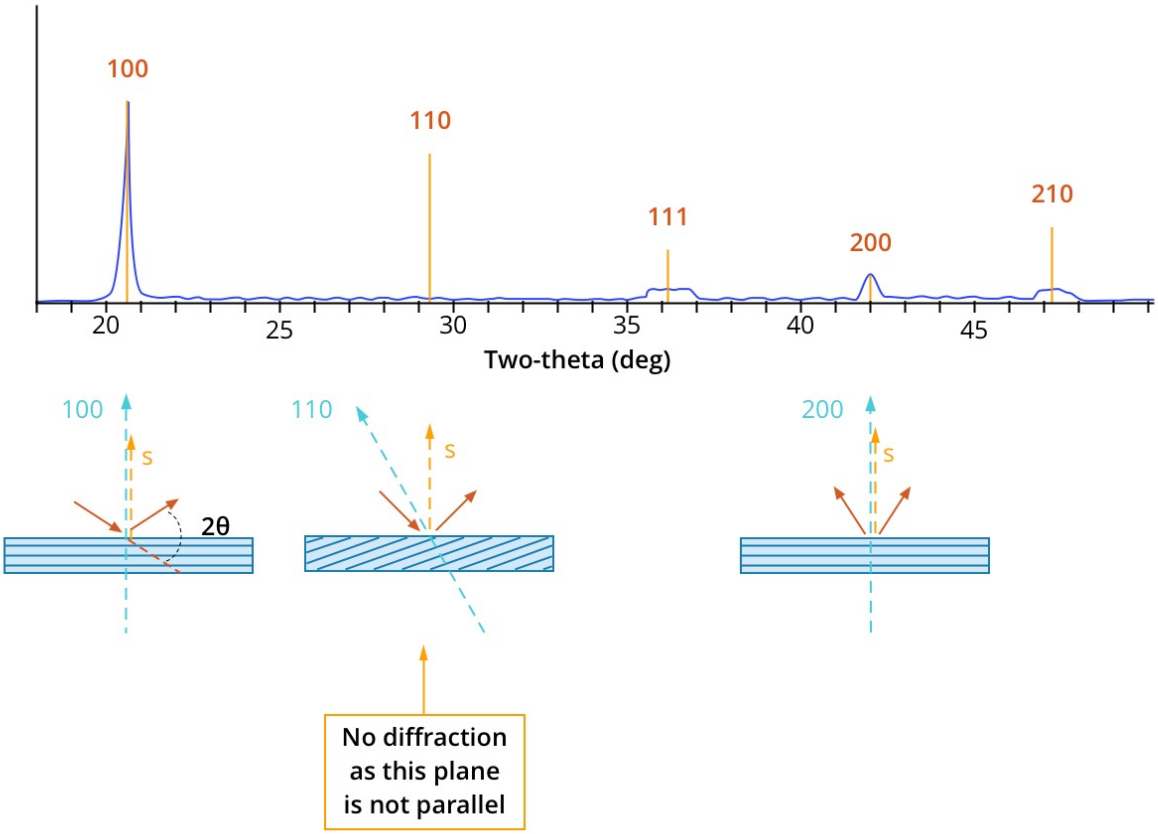


The XRD data is typically plotted as a line trace with the angle in degrees on the x-axis, and X-ray intensity on the vertical axis. Typical X-ray diffraction spectra are given below.



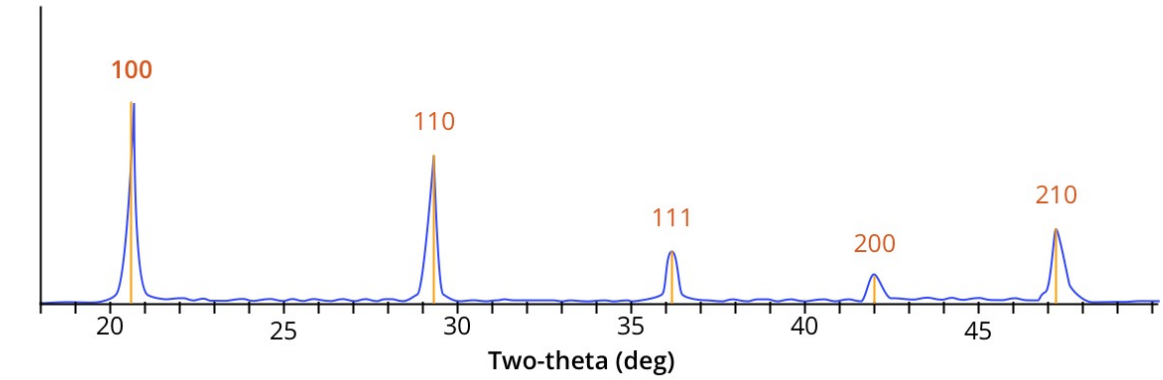


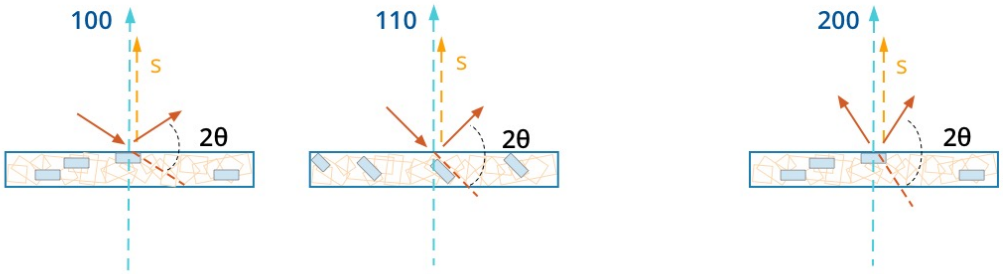




In polycrystalline samples, or powders, there are many grains on the sample surface. Some of these will be properly oriented for diffraction.

In highly textured materials (such as rolled metals) the relative height of the peaks can be very different to what we expect because some grains have rotated away from being parallel to the sample surface. The more planes that are parallel to the sample surface, the more they will diffract, producing a more intense peak.

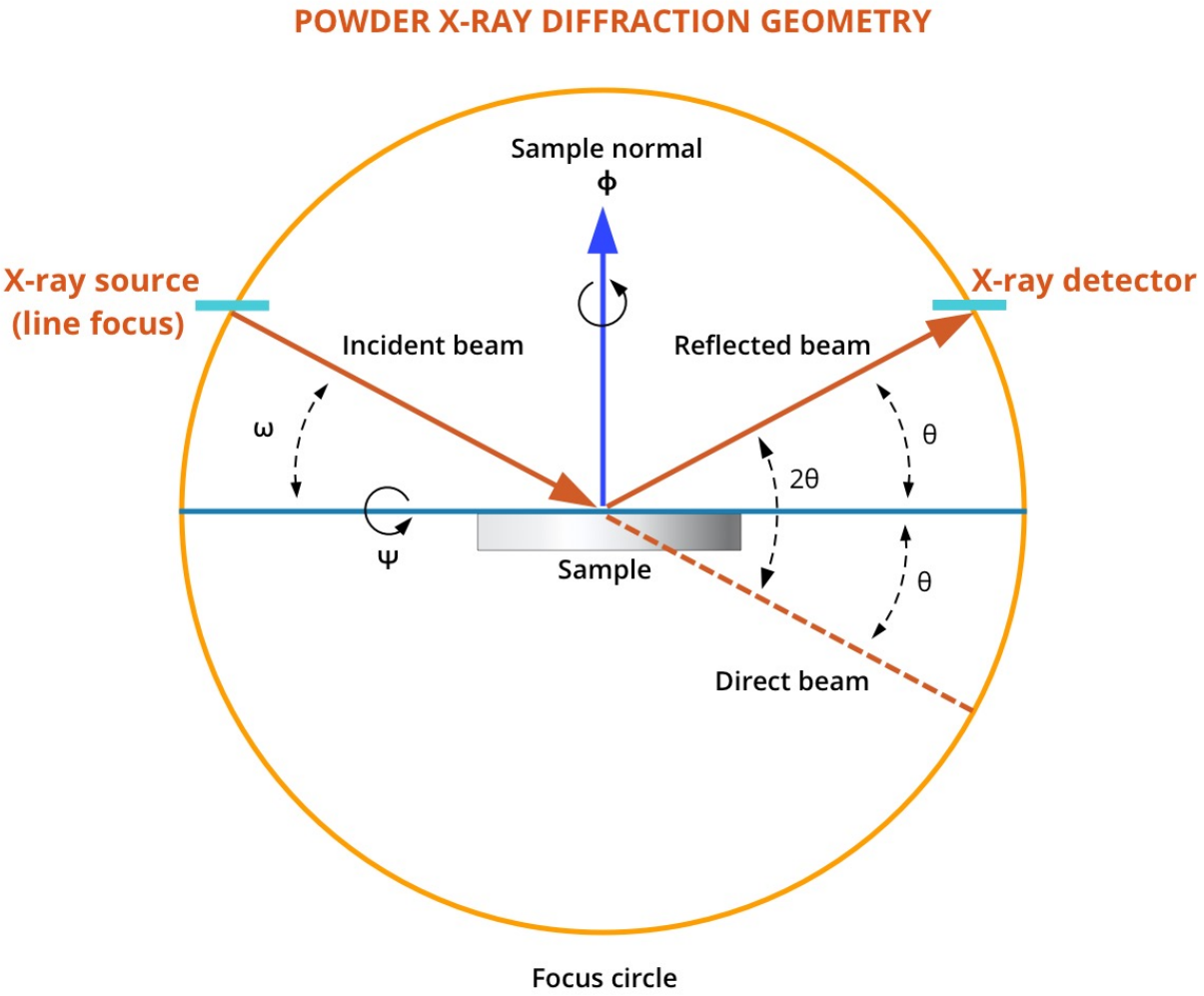

100
110
111
200
210
20
25
30
35
40
45
Two-theta (deg)


100
s
2θ
110
s
2θ
200
s
2θ

XRD in practice -
Anatomy of an X-ray diffractometer - Intro

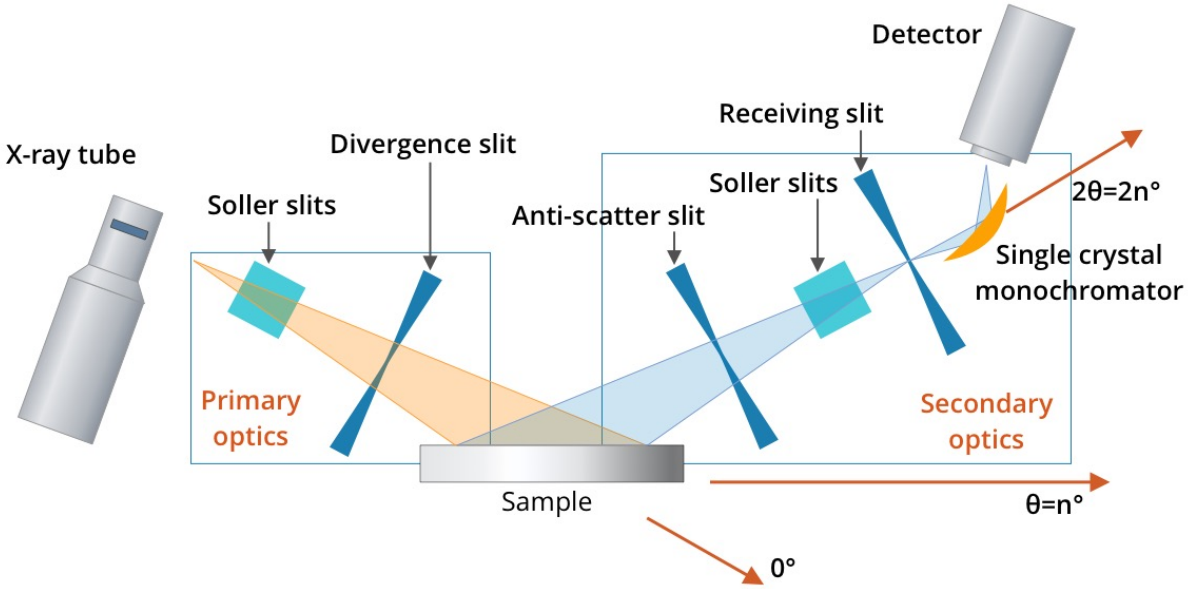
Most diffractometers in materials science are based on the reflection geometry where the X-ray source and detector are on the same side of the sample; the scattered X-rays from the source are reflected off the sample and onto the detector. It is common language to call this reflection geometry, but it is important to remember that the X-rays we examine diffract from the surface, they do not bounce or reflect. This is known as the Bragg-Brentano type geometry and is the dominant geometry found in most laboratories. This requires a rather complex movement of sample and detector or source and detector.

A powder diffractometer in Bragg-Brentano geometry operates with a divergent, beam which broadens until it reaches the specimen. From there it is focused onto the receiving slit, which is either followed by the detector or some secondary monochromators. In the case of secondary monochromators, the beam is refocused onto the detector.



There are several different types of instrument design. In the θ–2θ geometry, the X-ray source and primary optics are fixed, while the sample holder moves around θ and the secondary optics and detector moves around 2θ. In the θ–θ geometry the sample position is fixed while the X-ray source and primary optics, and the secondary optics and detector both move around θ°. Regardless of which components are fixed and which rotate, the essential characteristics of both geometries remain the same: the relationship between θ (the angle between the incident X-ray beam and the sample surface) and 2θ (the angle between the incident X-ray beam and the receiving slit-detector) is maintained throughout the analysis; the distance between the X-ray source and the sample, and the sample and the detector is fixed and equal and define the diffractometer circle in which the sample is always at the centre.

Bragg-brentano type diffractometer



Parts of the machine

The main parts of a powder diffractometer include:

- The source
- The primary optics
- The sample holder & sample stage
- The secondary optics
- The detector

Source

The source produces the X-rays used for analysing samples with X-ray diffraction. Typically, the source is an X-ray tube. It consists of an evacuated ceramic or glass vessel that contains a tungsten filament as the cathode which emits electrons, and an anode onto which these electrons are accelerated with a potential of several tens of thousands of volts.

Several processes lead to the emission of electromagnetic radiation in the X-ray range as the electrons hit the anode target. First, the electrons are quickly decelerated by passing the nuclear cores. This gives the so-called Bremsstrahlung or white radiation effect.

Second, the accelerated electrons hit inner shell electrons from atoms of the target material, remove them and leave holes behind. These holes are quickly filled from higher level electrons of the same atom. On falling down to the lower energy level the atoms emit characteristic radiation, which corresponds to the energy levels of the anode material.

The most intense characteristic lines are the Kα1 and Kα2 radiation and the Kβ. The first two are usually used in diffraction experiments. All other types of radiation (including the Bremstahlung) are unwanted by-products. The wavelengths of each of these characteristic lines are summarised in the table below for two of the most common anode elements.

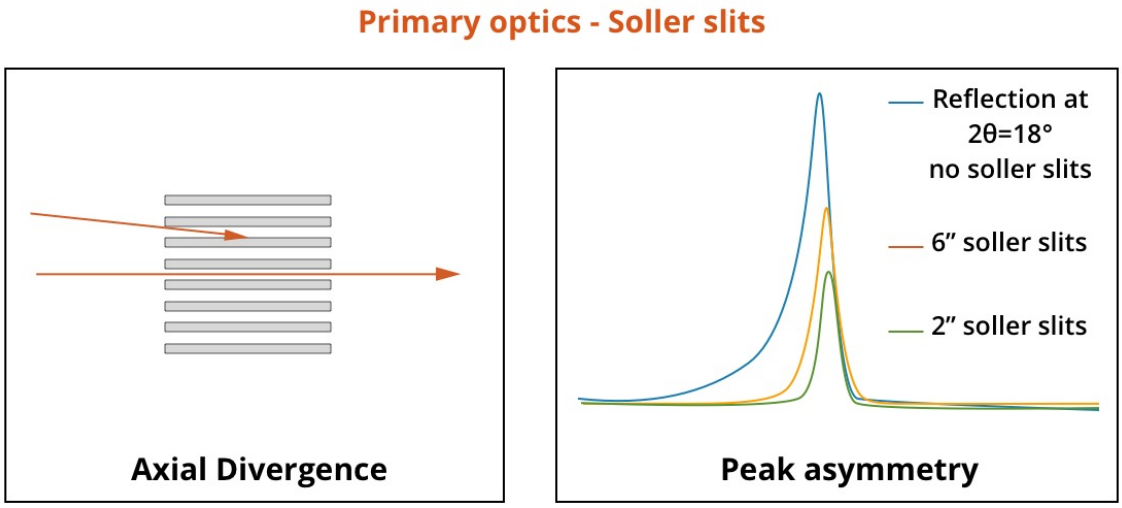
ELEMENT	Kα(av.)	Kα1	Kα2	Kβ
Cobalt (Co)	1.7902758	1.789961	1.7928351	1.6208263
Copper (Cu)	1.5418710	1.5405929	1.5444274	1.3922340

Copper is the most common anode material in lab-based XRD. However, if a sample shows a fluorescence effect then the sample will simply absorb and re-radiate X-rays from the copper anode, leading to large amounts of noise in the output. This is the case with samples containing large amount of iron. This can be solved by using the cobalt anode, which provides less signal intensity but will also remove the noise caused by the fluorescence background.

Primary optics

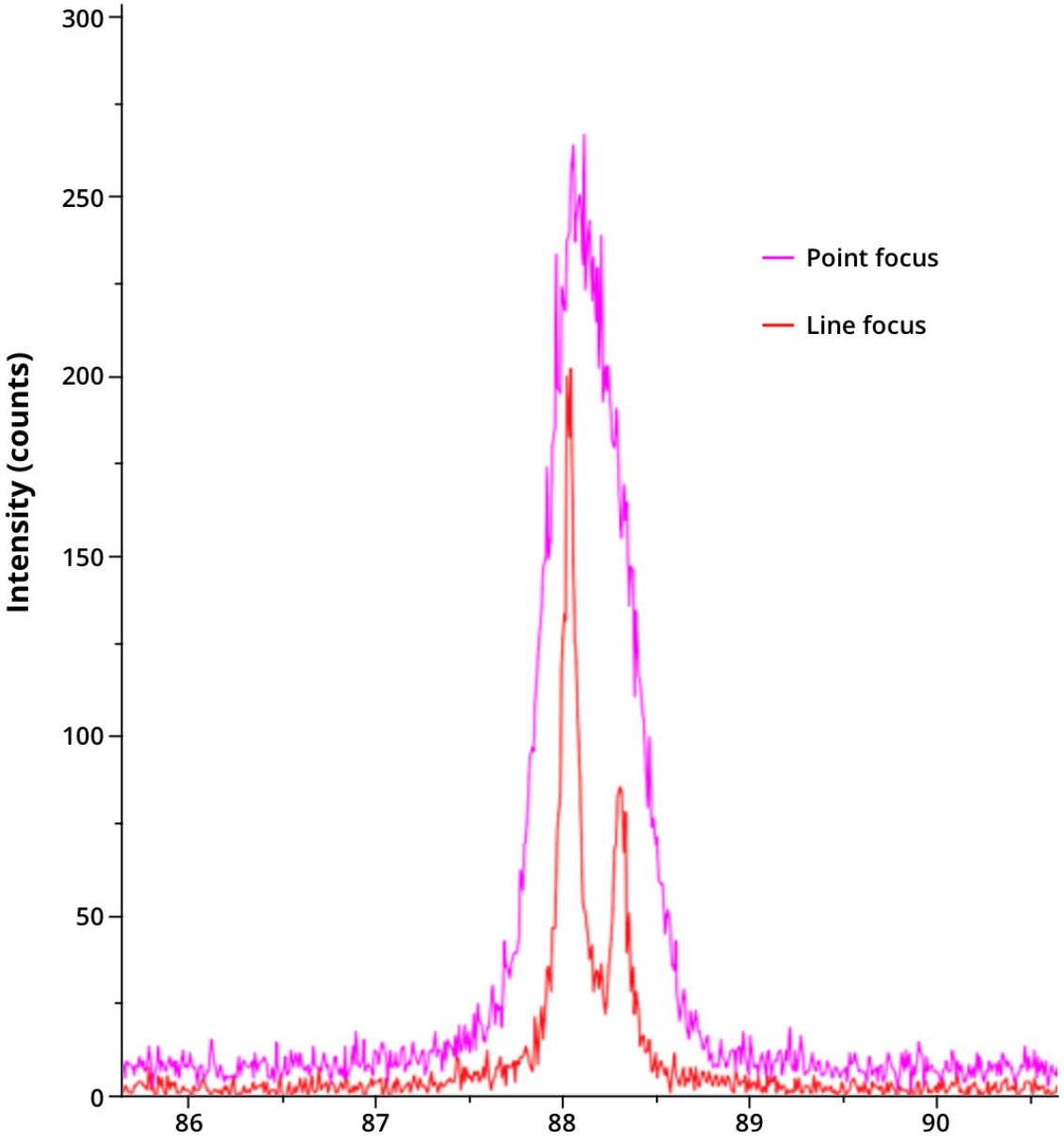
The primary optics controls the beam produced by the X-ray source and manipulates it into forms more useful for diffraction experiments. Optics can be either line focus or point focus. Line focus is the more common form of XRD optics, and produces narrow peaks but is very sensitive to sample height and roughness. There are three main components to the line focus optics:

- The soller slits reduce the axial divergence of the X-ray beam to less than 6° (in some instruments even 4° or even 2°). Reducing the axial divergence will reduce the peak asymmetry in the output.



- The divergence slit reduces the height divergence (beam spread). This narrows the peaks and increases the resolution of the output.

Point focus optics are used for those specimens that have high roughness, and is also used in measurements such as residual stress where the sample is tilted from being parallel to the analysis plane. The diagram below shows the difference between point and line focus optics, and the improved peak width is evident for the line focus. In some instruments a monochromator is put on the primary optics, but more commonly is located on the detector side of the instrument.


Intensity (counts)
300
250
200
150
100
50
0
86
87
88
89
90
Point focus
Line focus

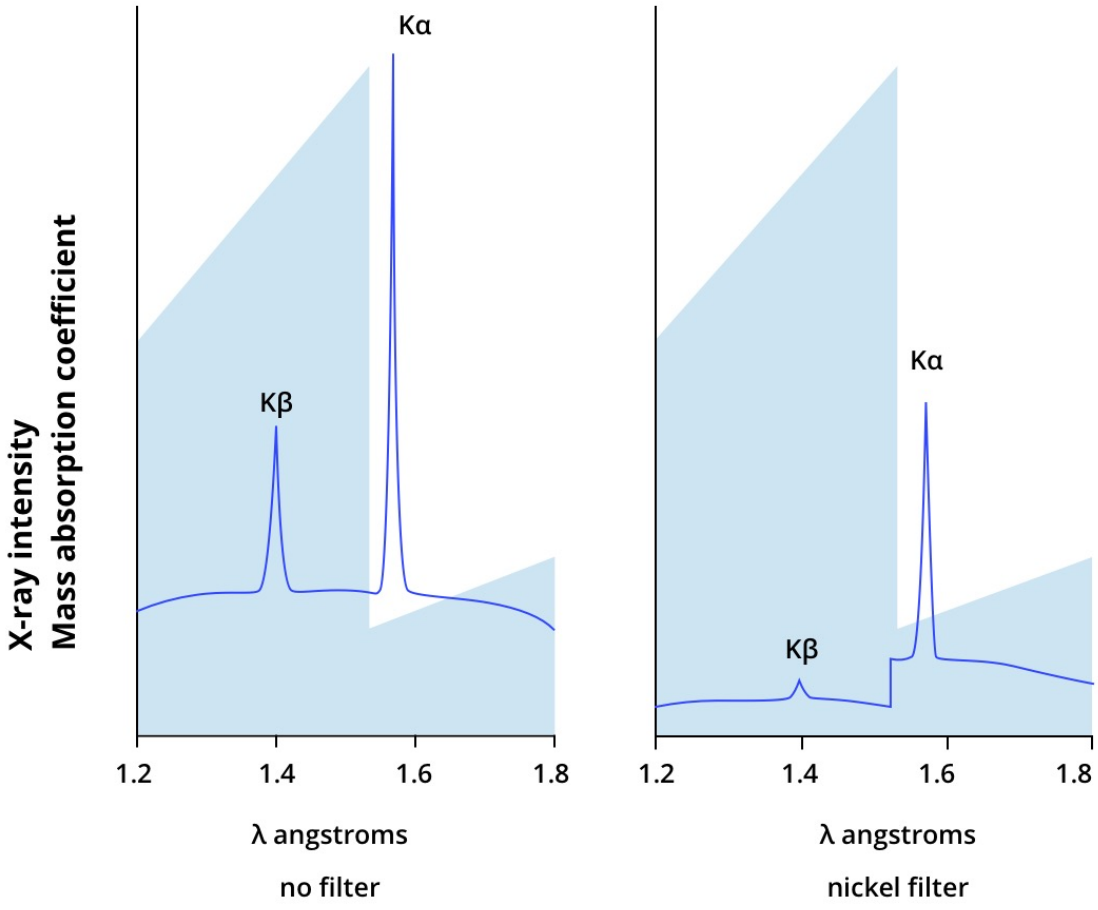
Sample holder & stage

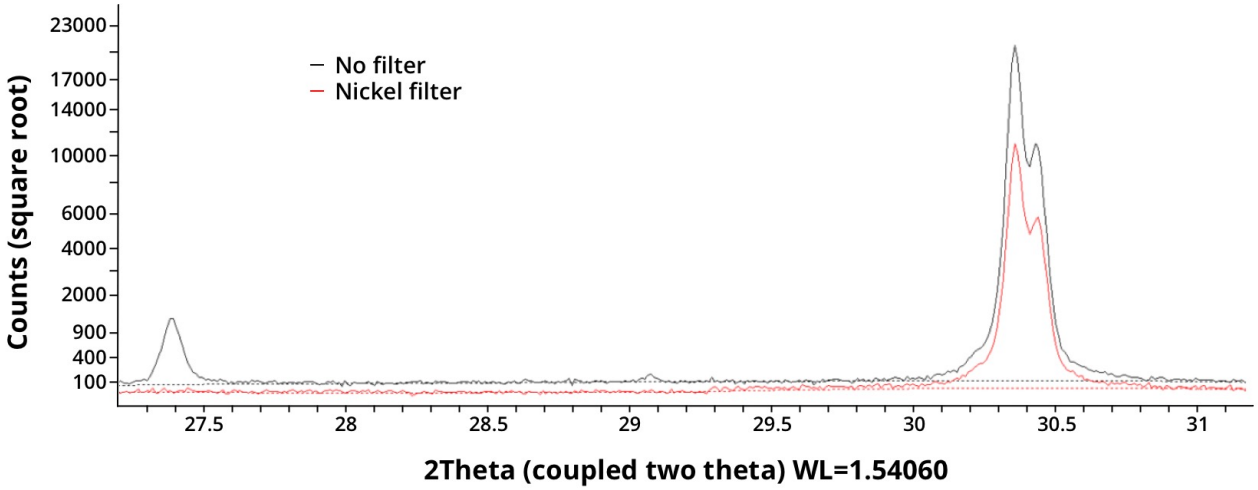
Most diffractometers are supplied with several kinds of sample holders for different types of specimens such as powders, bulk samples and thin films. The best results are obtained with rotating sample holders which considerably improve the measurement statistics, but they are not available for all machines. The most severe error during sample preparation is to fill the sample holder too high or low. Both result in a significant shift of peak positions which can make the interpretation difficult.

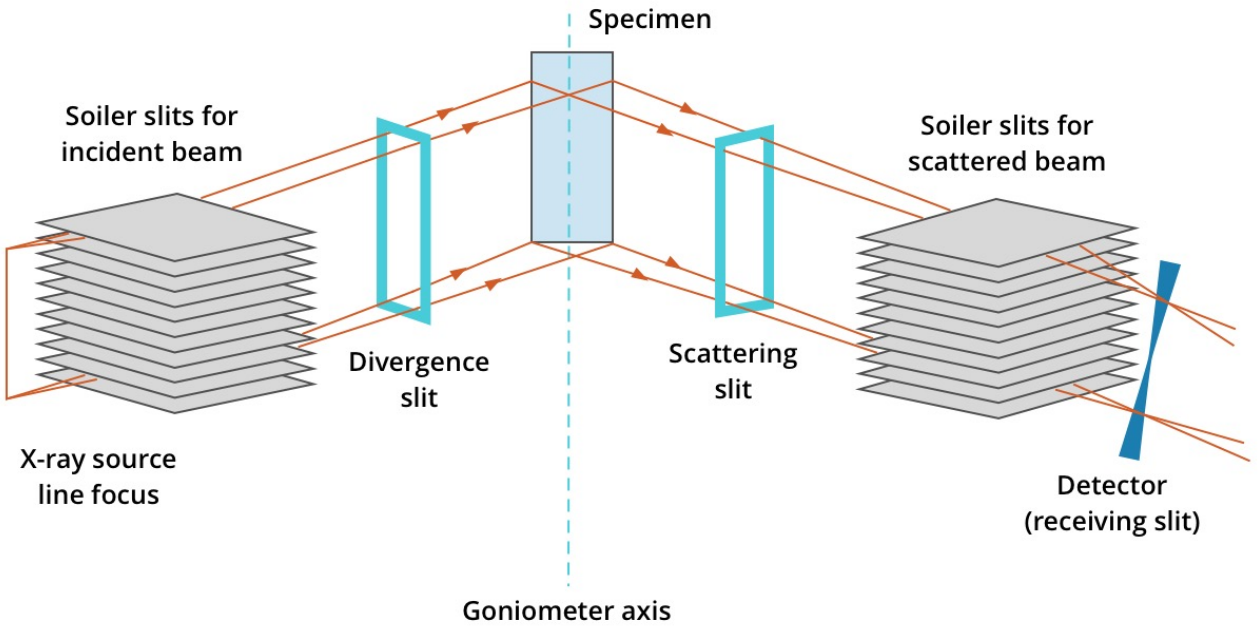
Care must be taken when choosing the sample holder or substrate. In particular, for those researchers using XRD to quantify coatings on substrates, or analyse small volumes of powders dispersed onto a glass slide, care must be taken to ensure that the diffracted signal from the substrate is taken into consideration. In particular, researchers using XRD to quantify the volume fraction of amorphous and crystalline phases within their specimens need to ensure that the substrate does not affect their data analysis, and in these cases, zero diffraction plates are recommended if possible.

Secondary optics

The secondary optics receive the diffracted X-rays from the sample. The first component is a slit called the anti-scatter slit, which reduces the height divergence but also reduces diffusely scattered X-rays that are due to amorphous or air scattering. This is followed by a set of soller slits which prevent axially diverged X-rays entering the detector. In most instruments a monochromator is put between the soller slits and the detector. The monochromator(s) can be fitted either to the incident beam, or to the diffracted beam. Sometimes monochromators are fitted to both sides. There are three common methods of monochromating the beam. The first is to add a filter which removes most of the X-rays with energies below the Bragg edge. The second method used in some newer diffractometers is an energy sensitive detector that only “counts” those X-ray energies within a certain energy range. The third method is to use a large single crystal (usually graphite or silicon) to carry out a second diffraction process on the beam coming from the sample. The second diffraction event ensures that only the wavelength of interest meets the Bragg condition, and only this is directed to the detector. All other wavelengths are excluded.




No filter
Nickel filter
Counts (square root)
23000
17000
14000
10000
6000
4000
2000
900
400
100
27.5
28
28.5
29
29.5
30
30.5
31
2Theta (coupled two theta) WL=1.54060

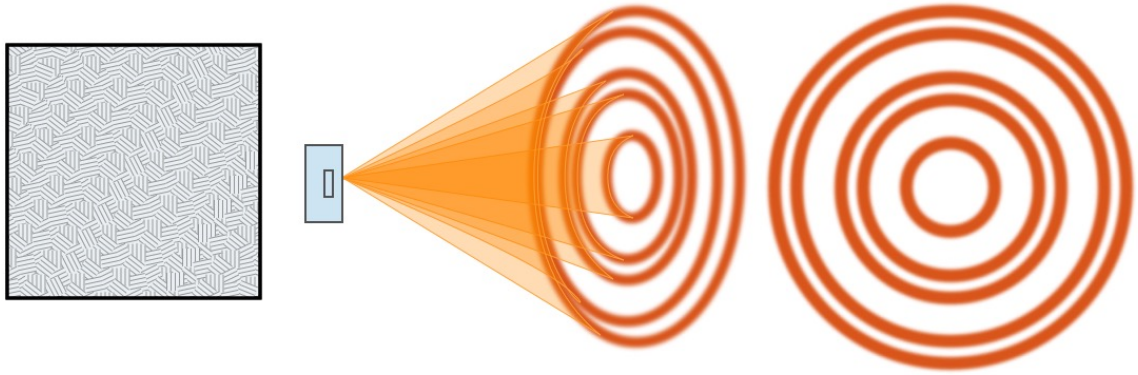

Specimen
Soiler slits for incident beam
Soiler slits for scattered beam
Divergence slit
Scattering slit
X-ray source line focus
Detector (receiving slit)
Goniometer axis

Sample preparation - Types of samples

In both qualitative and quantitative XRD, counting statistics are vital. Since only those crystal planes parallel to the diffracting plane will be detected, it is imperative to ensure that you are obtaining diffraction from many thousands of grains during the measurement. Diffraction from an ideal powder gives a strong diffracted signal from all planes, and the diffracted intensity is uniform around the diffraction ring.

Diffraction of an ideal power

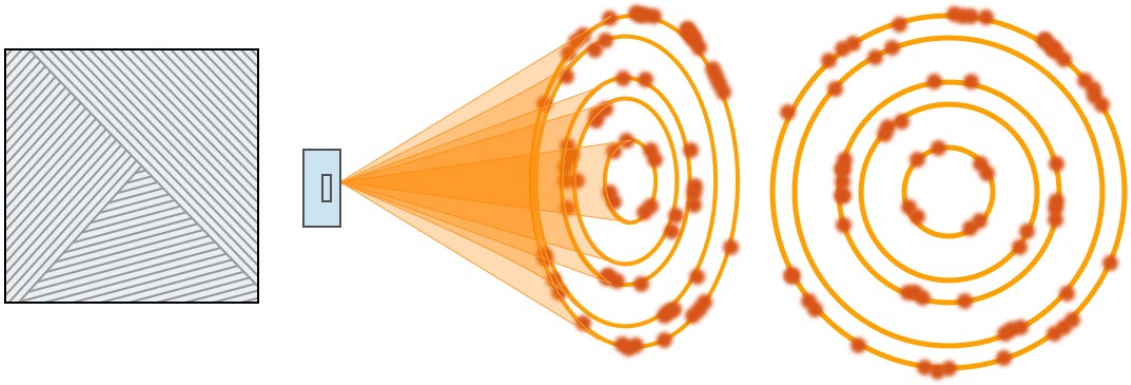
Uniform intensity around diffraction rings



However, if we take a measurement from a smaller number of larger grains, we will get diffraction at some locations around the ring, but not others. This leads to the situation where some peaks may be missed altogether. This will make it impossible to identify, index or quantify your spectrum.

Diffraction of a small number of crystallites
"spottiness effect"

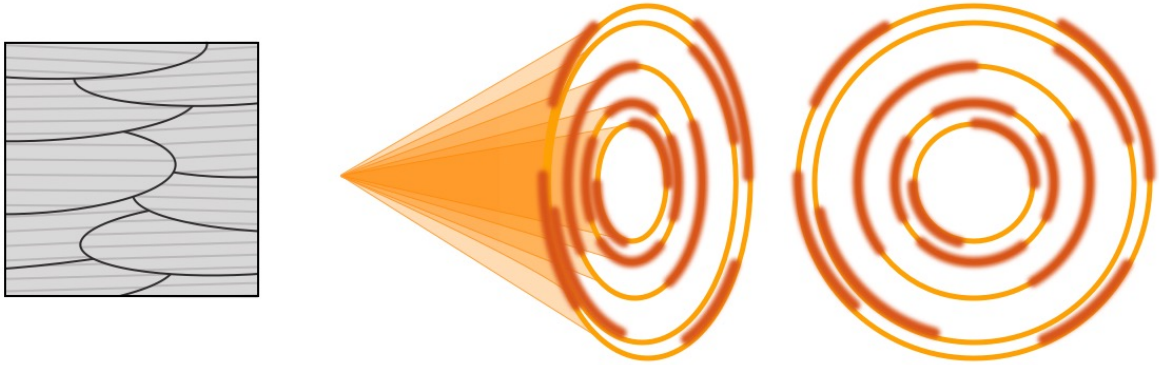
No correction possible

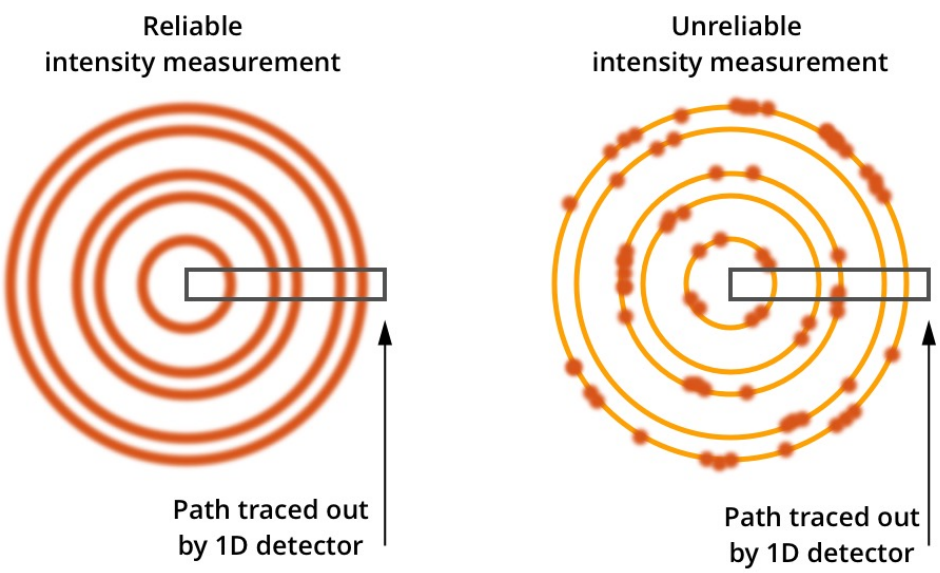


In many solid samples, the orientation of the grains or crystals will not be uniform. Most solids take up some preferred orientation of the crystals within the microstructure. Consequently, the diffraction ring shows a non-uniform intensity. Those orientations that are more common give a larger diffracted signal.

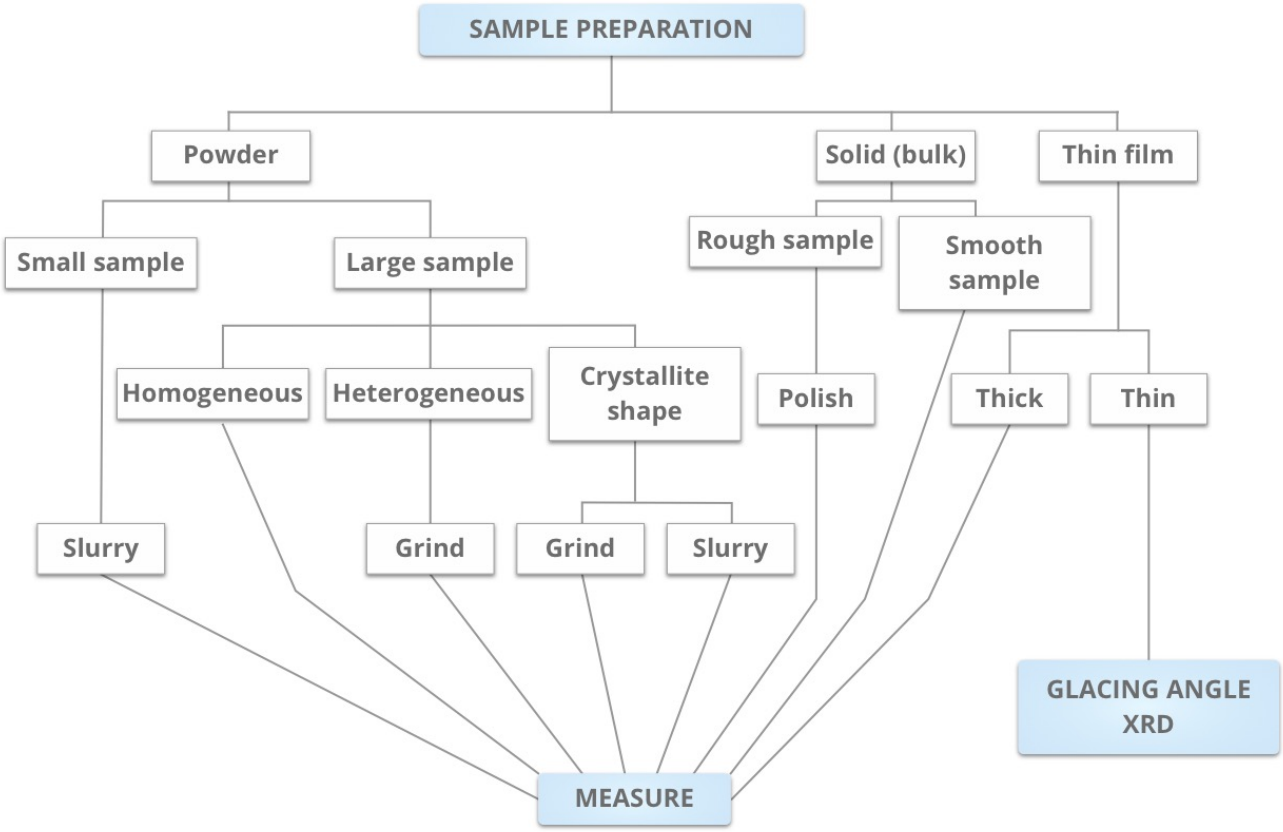
Diffraction of textured materials

Preferred orientation can be corrected to some extent.
Degree of orientation can be extracted



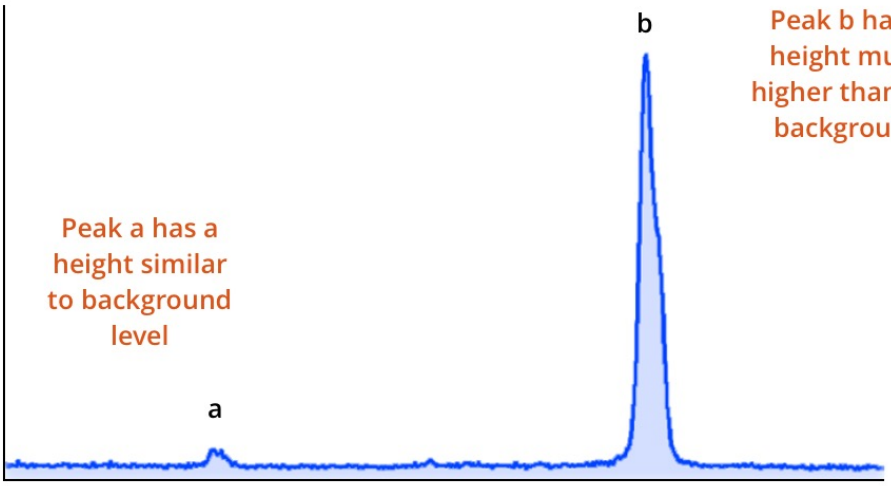


Below is a flowchart which details the steps in preparing a sample of many different types using various techniques.


SAMPLE PREPARATION
Powder
Solid (bulk)
Thin film
Small sample
Large sample
Rough sample
Smooth sample
Homogeneous
Heterogeneous
Crystallite shape
Polish
Thick
Thin
Slurry
Grind
Grind
Slurry
GLACING ANGLE XRD
MEASURE

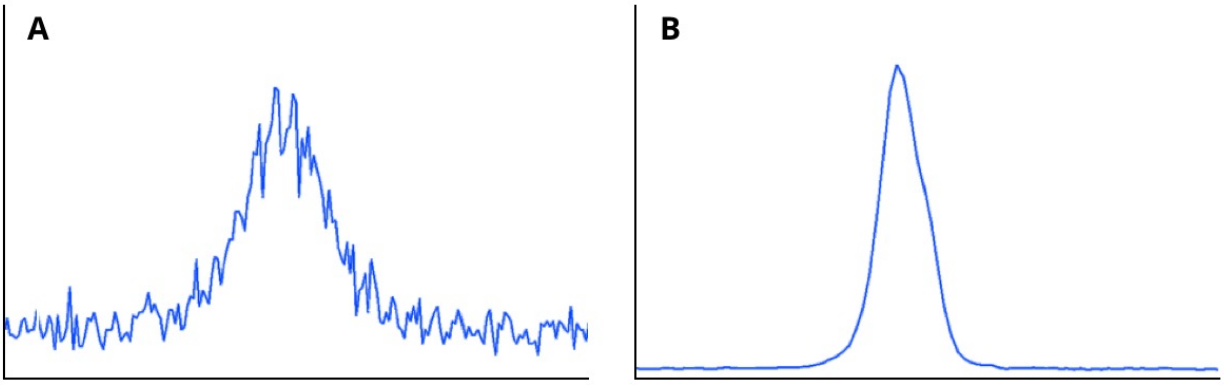
What is good data?

Peak to background ratio



Signal to noise ratio

Peak A clearly has more noise than peak B in relation to its maximum height.

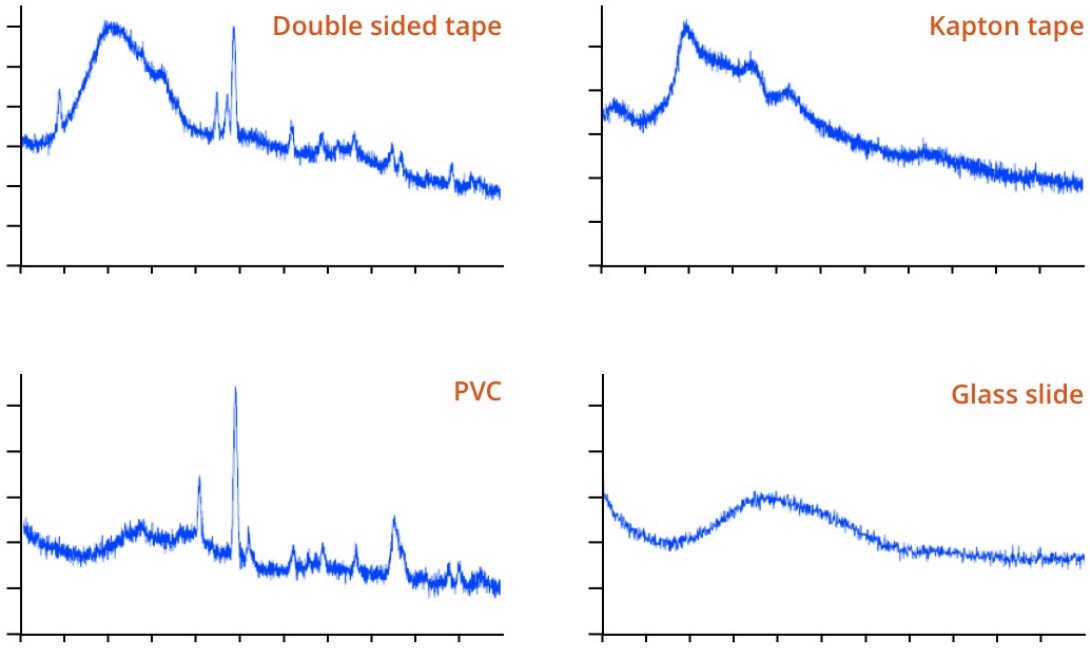


The signal-to-noise ratio can be improved by:

- Scanning longer
- Decreasing the step size (but not the scan rate)
- Irradiating a larger area if possible

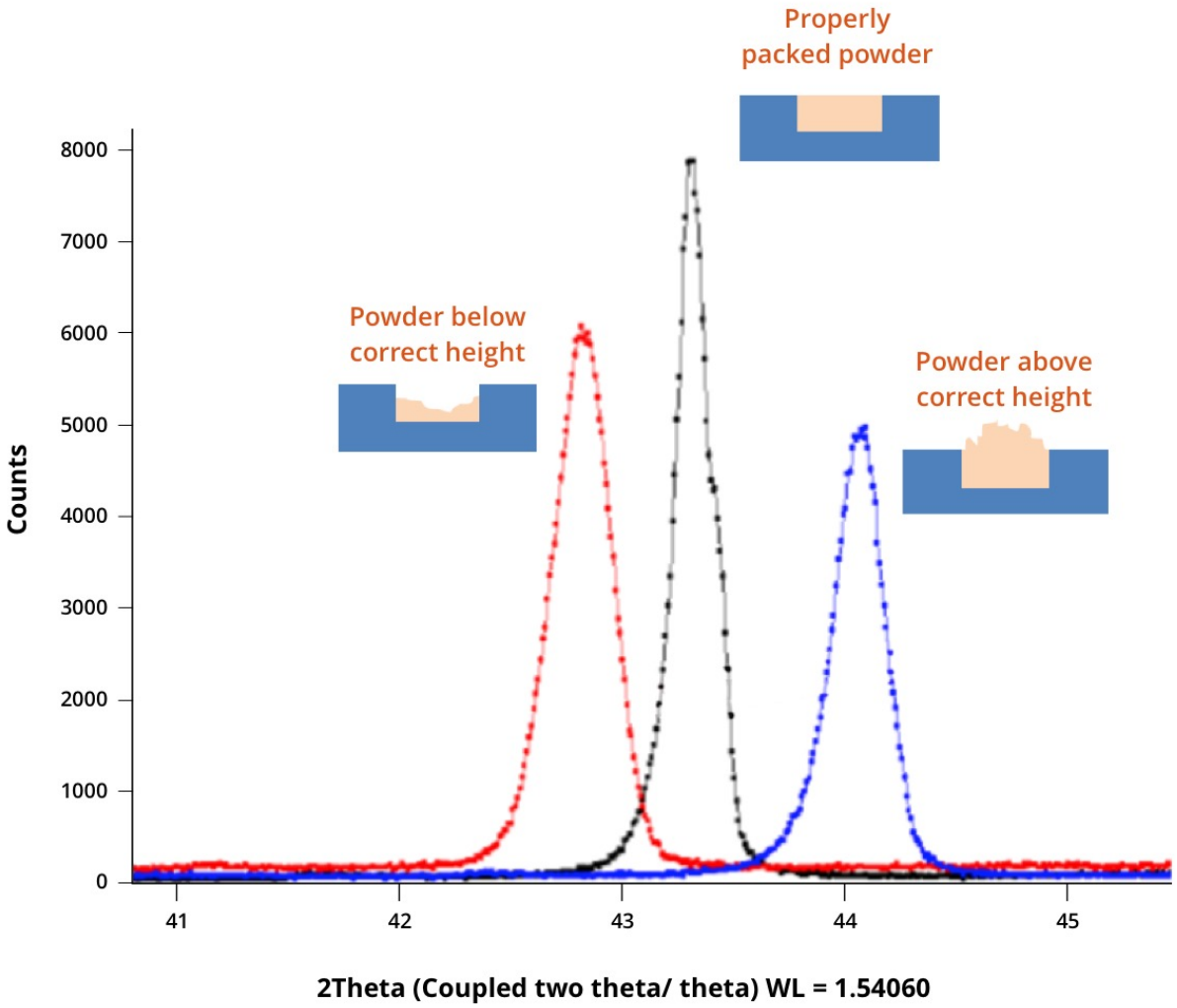
Stray peaks

Choose your substrate wisely



The importance of specimen height

It is critical for powder XRD (line focus) that the powder is with in a few hundred microns of the correct height. This is only the thickness of a human hair. Heaping powder above the correct height, or not sufficiently filling the sample holder cavity, will lead to extremely large errors in the measured angle of the diffracted peak. At the incorrect height the angles can be out by more than 0.5 degree, and the intensities are halved.



Safety

There are a number of safety guidelines and factors to consider when using the equipment for X-ray diffraction.

X-ray equipment and radiation hazards

X-rays are highly energetic ionizing radiation and as such are hazardous to human health. They can trigger a lot of chemical reactions in the human body that ultimately lead to cancer and death. Precautions to prevent exposure to X-rays are therefore essential for any XRD laboratory. These include sufficient shielding of all X-ray equipment, limited access to the laboratory, measuring and recording of all radiation exposure of staff and students with dosimeters. Under normal circumstances direct exposure to X-rays in a laboratory is very unlikely. However, users need to be aware of the risks associated with the equipment since all precautions, in principle, can fail.

Radiation hazards

Analytical X-ray equipment makes use of very narrow collimated X-ray beams of high intensity. Exposure of the eyes or the skin of the body to the primary X-ray beam may result in severe localised radiation burns in a matter of seconds. These burns heal poorly, and on rare occasions have required amputation of fingers.

Hazards also exist from exposure to scattered radiation. Scattered radiation is produced when the primary beam strikes collimators, samples, beam stops or shielding. The intensity of the scattered radiation is several orders of magnitude less than that of the primary beam. However, these scattered radiation fields may still result in exposures, which exceed regulatory limits.

Preventative measures

All the instruments are inside radiation housings, which reduce radiation leakage below detection limit. Whenever a door of the enclosure is opened, lead shutters cut off the X-ray beam and interrupt the measurement. With some equipment, even the generator may be shut down and you will have to ask an operator for assistance.

To prevent exposures: to record possible malfunctioning of equipment you are compelled to carry dosimeters whilst working with the X-ray equipment. This will not prevent radiation damage from occurring, but it does enable immediate medical action to minimise damage and to replace faulty equipment. The physical measure for radiation is the energy dose. It is equal to the energy deposited per unit mass of medium and therefore has the SI unit J/kg = Gy, Gray in honour of the English physicist L. H. Gray (1905-1965). This, however, is only of limited use to determine the actual hazard associated with the radiation. A lot depends on the kind of radiation. The notion that the harder (the shorter the wavelength) the radiation, the greater the hazard is a myth. Actually, copper radiation (λ=1.54187 A) used in powder diffraction is ten times as dangerous as the much harder silver radiation (λ=0.56087 A) used in medical equipment. The reason lies in the different absorption coefficient. While the harder X-rays can potentially cause more damage, they are very much less likely to interact with body tissue and therefore actually cause less damage. To correct for these effects, the so-called equivalent dose is used which multiplies the energy dose with a dimensionless factor Q that depends on the nature of the radiation used. This unit is named Sv or Sievert in honour of the Swedish medical physicist Rolf Maximilian Sievert (1896-1966). This unit is used in our personal dosimeters. The X-ray intensity rapidly decreases with the distance from the X-ray tube. The following table should give you a rough idea about the hazards.

Estimated radiation doses at analytical X-ray equipment:

LABORATORY	DOSE RATE
Primary beam at tube port	Several tens of grays per second
Primary beam at the end of primary optics	Several tens of grays per minute
Scattered radiation near sample	Several milligrays per hour
Scattered radiation near table edge (inside housing)	1 milligray per hour

Essentially, users are safe as long as they remain outside the interlocked instrument enclosure and all safety measures are properly in place.

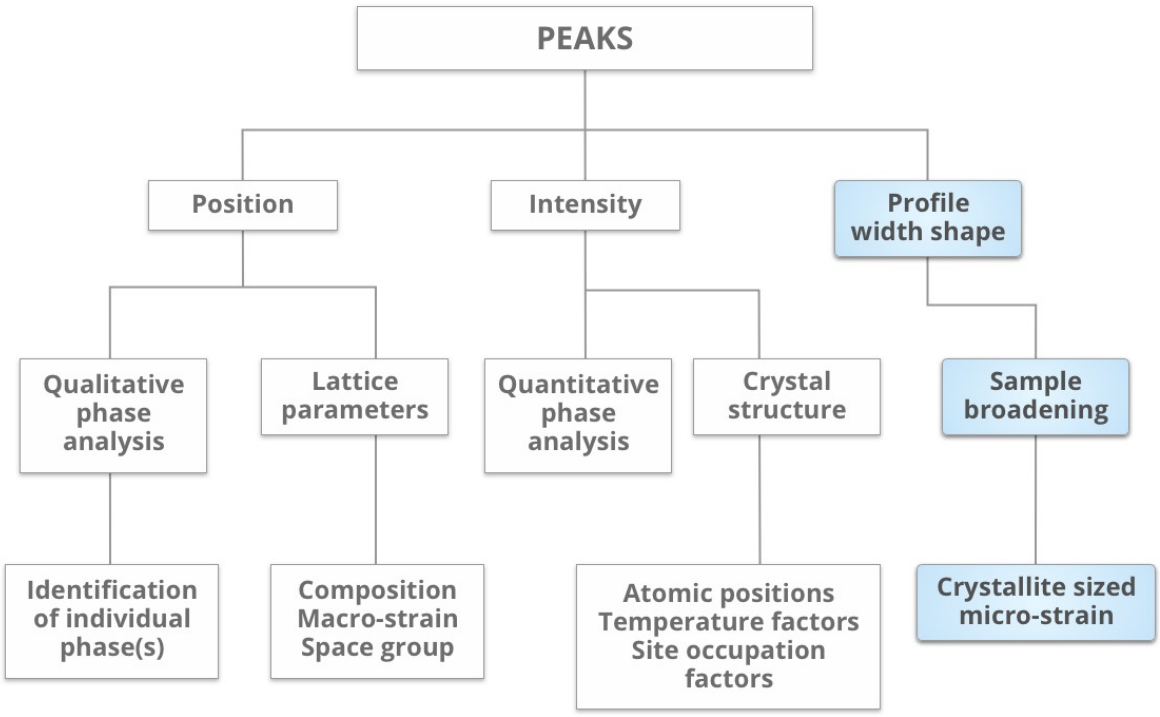
Essentially, users are safe as long as they remain outside the interlocked instrument enclosure and all safety measures are properly in place.

Please alert the instrument manager immediately if you note one of the following:

- The "X-ray On" light is on even though the instrument door is open.
- Your dosimeter shows an unexpected rise after any experiment.

Analysis of data - What the data tells you

What information is actually contained in the diffraction spectra?



Phase identification

What is Phase ID? Phase identification is the name we give to the process of deciding which phases are in your specimen by analysis of the XRD spectrum. This is sometimes referred to as qualitative analysis because you only determine what is present in your sample, but not how much. Phase ID uses the position of peaks, and sometimes their intensity, to determine what is present. This is done by matching peak positions with a known library of spectra. Most researchers are very familiar with their specimens, are are likely to know the few possible phases that may be present. However, if you have an unknown sample, or cannot find what you expect, then you must search a database to try and find a match.

There are three main sources of diffraction spectra that you can use for matching against your experimental measurement.

1. Commercially purchased databases. The most common and most up to date is the database made by the International Centre for Diffraction Data (ICDD). Their database is quite common in most labs, and the diffraction data is provided as a Powder diffractions file, PDF, with a unique identifier. This is particularly useful for publications because you can reference where your crystallographic data came from. This is considered a very reputable source of information. **icdd.com**.

2. Online open access databases. There is a growing amount of open access scientific information being published online, and XRD is no exception. The main open access website for crystallographic and XRD data is the Crystallography Open database, COD. This website has been operating for about twenty years and has recently received funding by the European Union's Horizon 2020 program. **crystallography.net/cod**.

3. Literature. In many cases, very newly developed materials will not yet appear in these databases, the best place to find these spectra is in the original publication. Most publications show the full XRD spectrum, so you have the ability to critically examine if you agree with the authors conclusion before you proceed.

   Researchers who are familiar with their specimens are usually able to visually compare the main peaks from the possible phases against their experimentally measured data. This is often sufficient to decide which phases are present, and which are not. However, if you have a very complex spectrum, a more sophisticated approach is required. In this case, the full database (such as the PDF for example) will need to be searched by a matching algorithm. Most XRD software will have this function, but you will need to link this to your database of choice.

Quantitative powder diffraction - Quantitative analysis

Quantitative analysis refers to the use of the XRD spectrum to extract quantitative data such as the volume fraction of phases present, the grain size of the sample, or the lattice parameters.

Factors affecting peak intensity - Intro

In XRD, peak intensity refers to the area under the peak, not the peak height. The intensity of a diffracted peak is determined by the following factors:

- The structure factor
- Multiplicity of the crystal plane
- Lorentz and polarisation factors
- Temperature factor

Each of these four factors will be discussed in the following pages. You don't have to necessarily have a strong understanding of the mathematics behind the calculation, but having an understanding of how it all works is important to help you analyse your own experimental data, and understand what it means.

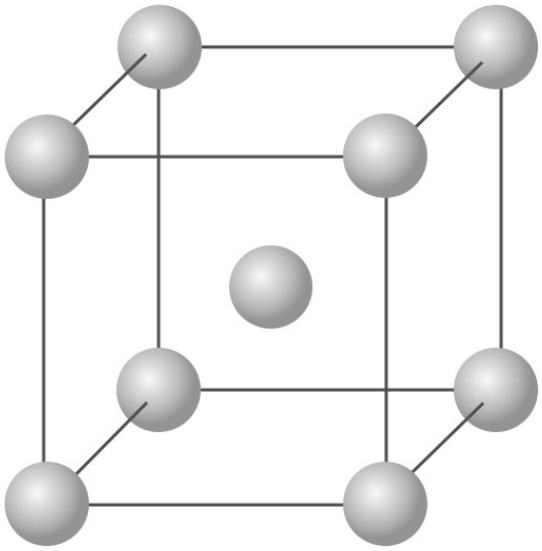
Structure Factor

The structure factor incorporates information about atomic locations within the crystal and determines if their collective interference will produce a peak for each of the different crystal planes or not.

$$S(hkl) = \Sigma_j f_j \exp\{-2\pi(hx_j + ky_j + lz_j)\}$$

Where f_j = form factor for the j^{th} atom
h, k & l = Miller indices of the hkl reflection
x_j, y_j & z_j = fractional coordinates of the j atom

If we consider the case for body centred cubic structures, the structure factor will be zero for those planes where the Miller indices sum (h+k+l) to give an odd number but are a positive integer when h+k+l is even. This means that for the BCC phase, there are systematic absences in the diffraction pattern, and this is the usual telltale sign by which we identify BCC systems.



S(100) = 0

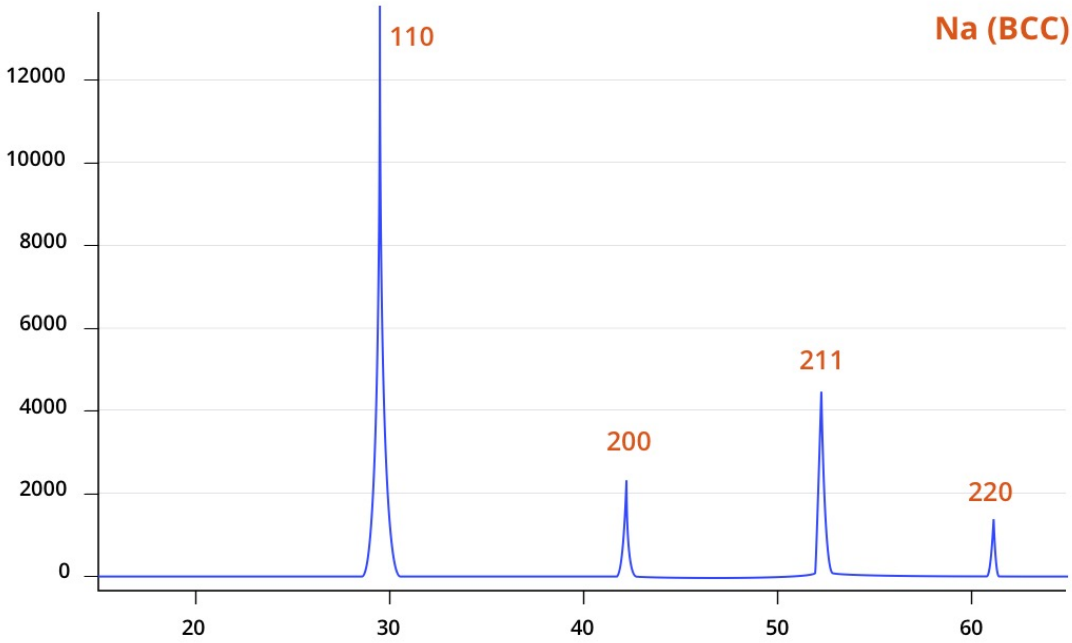
S(111) = 0

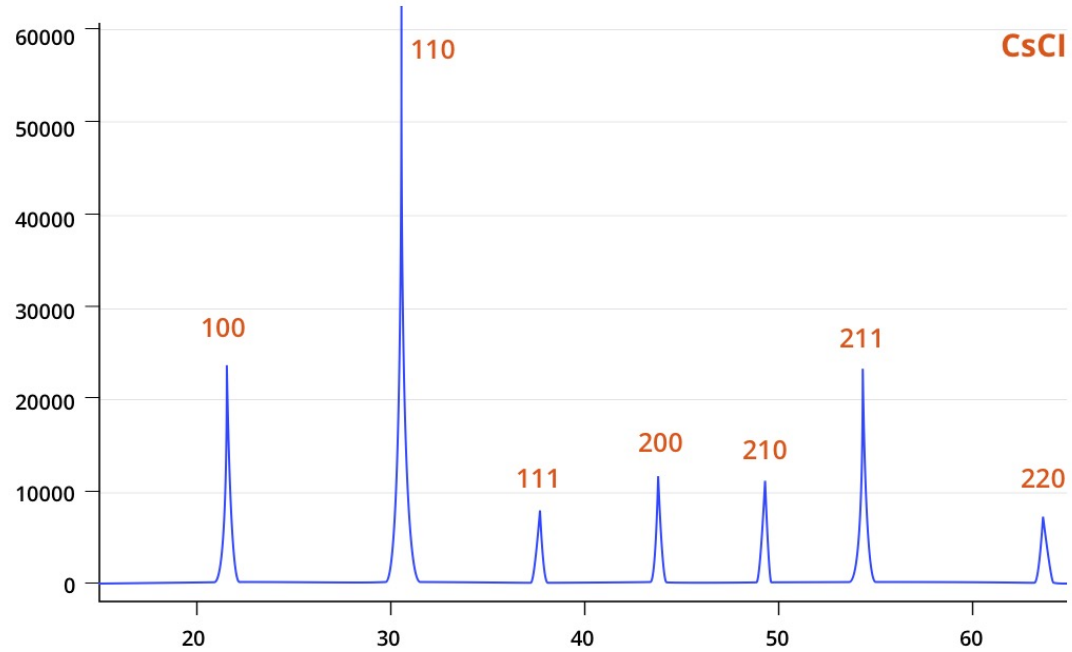
s(210) = 0

S(110) =2f

S(200) = 2f

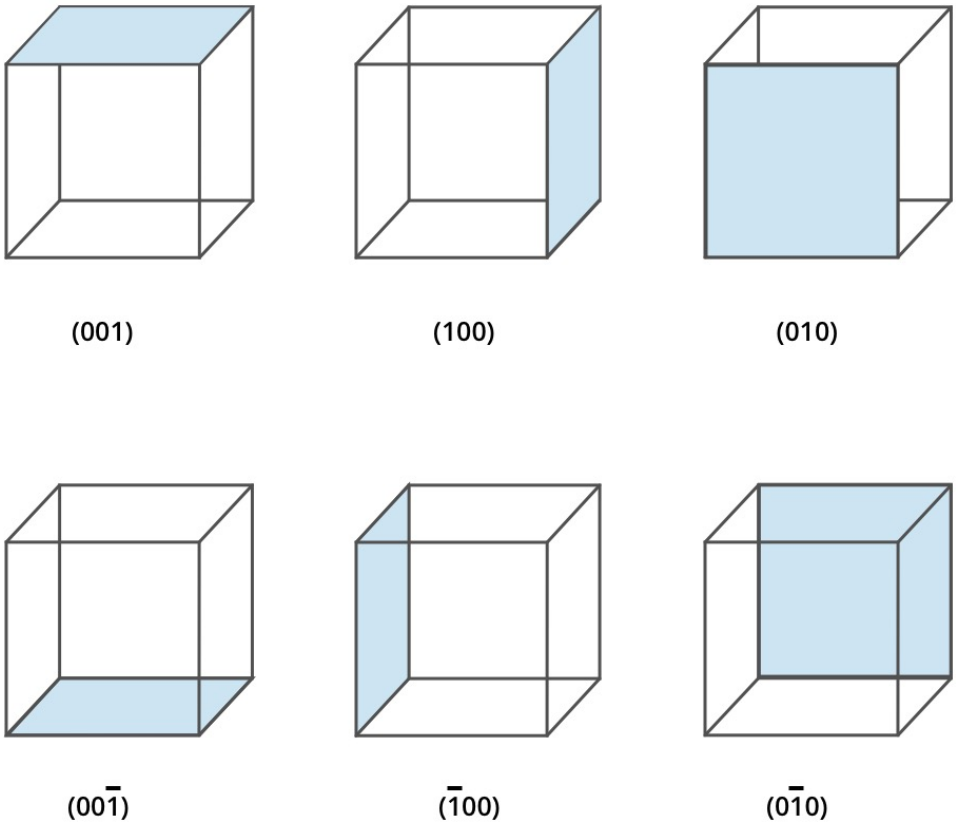
S(211) + 2f





Multiplicity factor

For many crystal structures, particularly those with simple unit cells, there will be more than one plane in the system with the same crystallography (in other words, if you look at the crystal from a different direction, it will look the same). A picture of this was shown in the crystallography section. For example, in a simple cubic system there will be six equivalent planes in the {100} family, just like there are six equivalent faces on a cube. In crystal notation, these equivalent planes have the Miller Indices (100) (010) (001) ($\bar{1}$00) (0$\bar{1}$0) (00$\bar{1}$). Thus, in the cubic system the multiplicity factor will be 6 for the {100} family of planes.



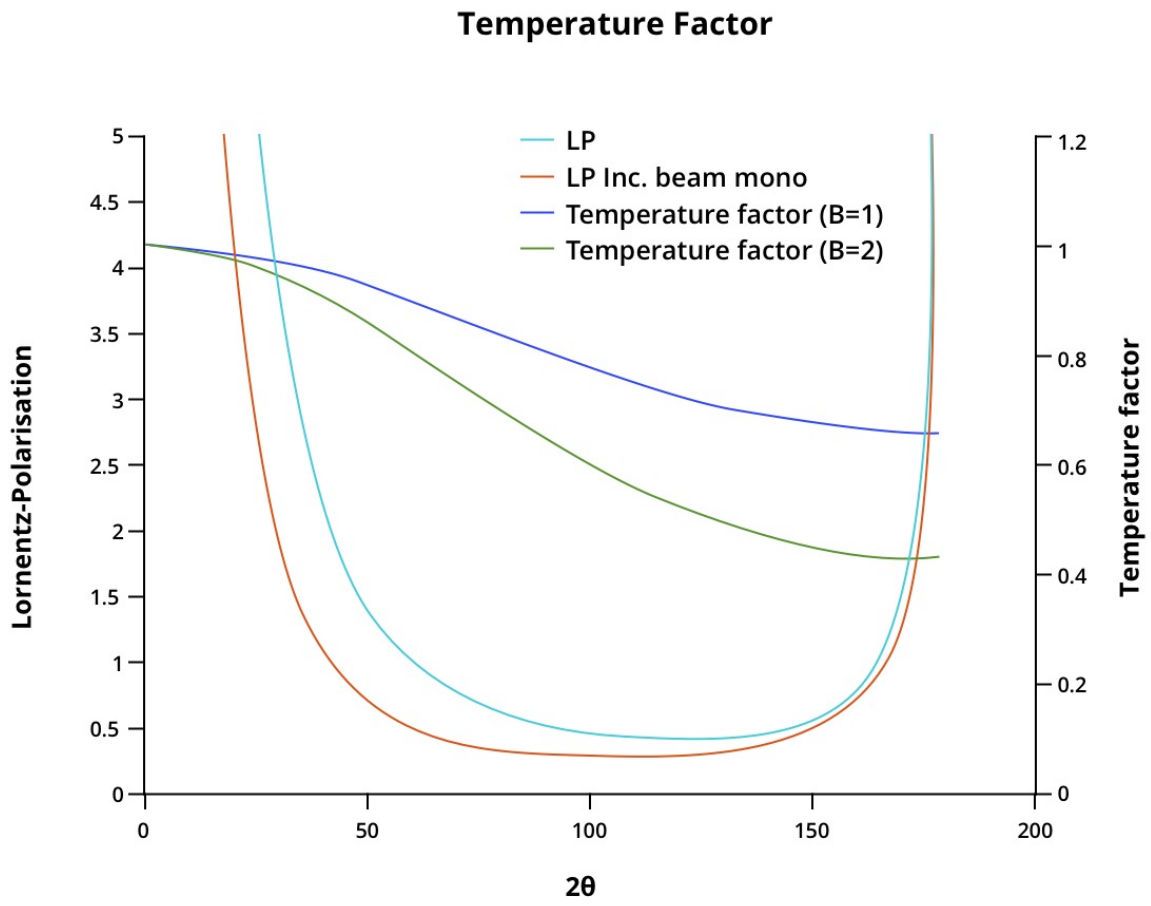
For the {110} family in the cubic system, there are 12 crystallographically equivalent planes (110) ($\bar{1}$10) ($\bar{1}\bar{1}$0) (10$\bar{1}$) (10$\bar{1}$) ($\bar{1}$0$\bar{1}$) (101) (011) (0$\bar{1}$1) (01$\bar{1}$) (0$\bar{1}\bar{1}$) , so the multiplicity factor will be 12.

Multiplicities are lower in low symmetry crystals such as hexagonal and trigonal crystals, and in these cases, it is important to note that, for example, (100) is not crystallographically equivalent to (010) or (001).

Lorentz polarisation factors

The Lorentz and the polarisation factors refer to the summation of reasons that the diffracted intensity of any set of planes is dependent on the angle 2θ. These include geometric considerations such as the fact that the number of crystals orientated to satisfy Bragg's law are largest at small angles, and the fraction of the diffraction cone that can intersect the detector is largest at low angles. The polarisation refers to the directional dependence of scattering intensity. Typically this is largest in the direction of the incident beam. The Lorentz and the polarisation factors are commonly summed into the Lorentz-Polarisation factor:

$$I \alpha (1 + \cos^2 2\theta)/(8\sin^2 \theta \cos\theta)$$



Temperature Factor

Atomic vibrations decrease the scattered intensity because the atoms within each plane are no longer in their "perfect" position. The temperature factor is usually not the largest contributor to changes in peak intensity, but pattern fitting software will often take this into consideration. In the equation below, B refers to the temperature factor, and is usually between 0.5 and 1.5. It is also evident that the temperature factor has the most effect at large angles of 2θ and small wavelengths.

$$TF(\theta) = \exp\{B(\sin\theta/\lambda)^2\}$$

Summation of factors effecting peak intensity

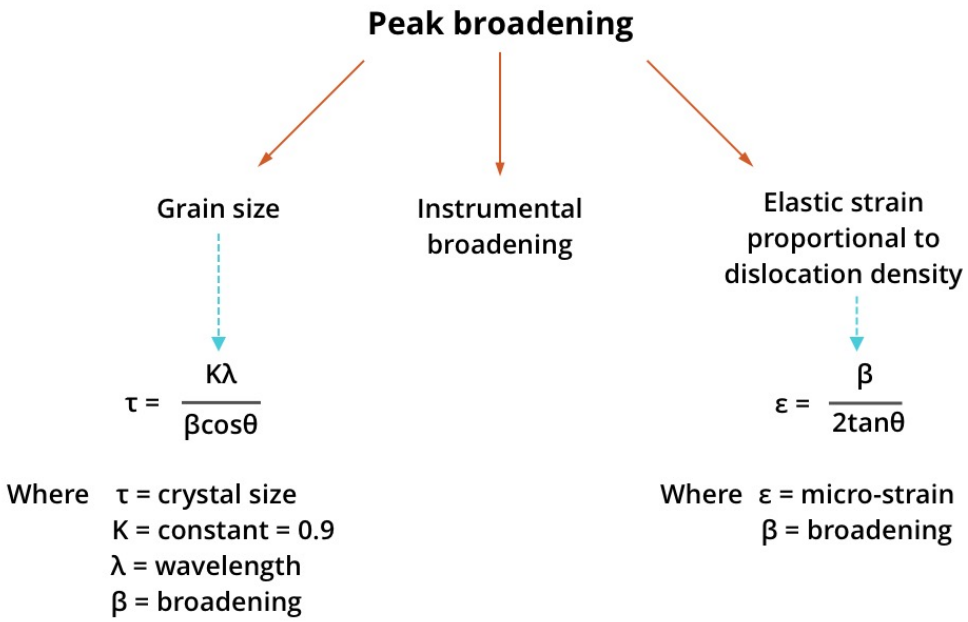
$$I(hkl) = |S(hkl)|^2 \times M_{hkl} \times LP(\theta) \times TF(\theta)$$

Where S(hkl) = Structure Factor
M_{hkl} = Multiplicity
LP(θ) = Lorentz & Polarisation Factors
TP(θ) = Temperature factor (more correctly referred to as the displacement parameter)

Factors effecting peak width

There are three contributions to peak broadening in the XRD machine:

- Instrumental broadening
- Grain-size broadening
- Micro-strain broadening (proportional to dislocation density)



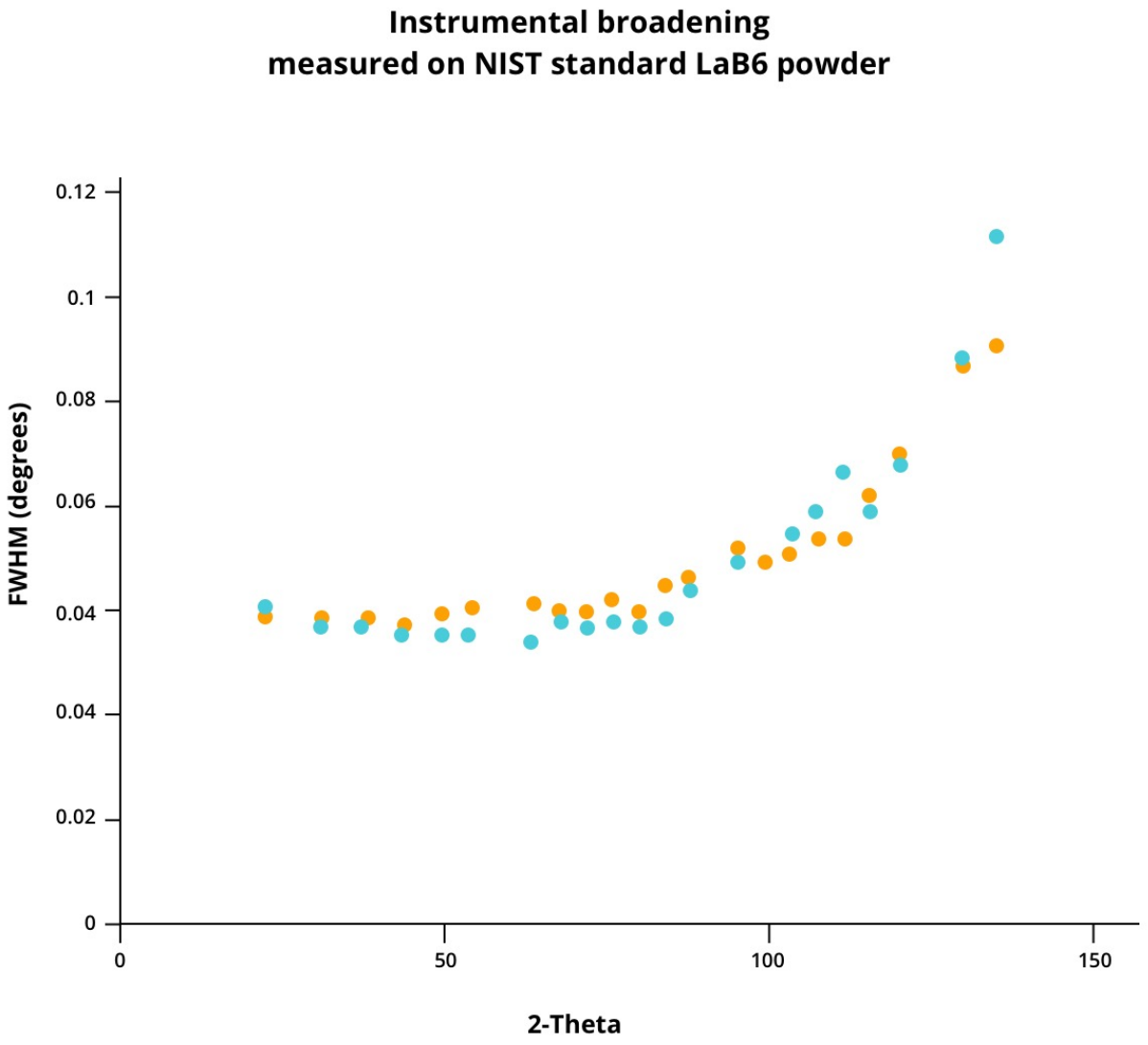
Instrumental broadening is easily measured and simply subtracted from the peak width measurement. However, if you have both strain and crystal size broadening it may be difficult to separate these effects. There are methods to separate these effects, but they are not always effective.

Instrumental broadening

Sources of instrumental broadening include:

- Non-ideal optics
- Wavelength dispersion
- Sample transparency
- Axial divergence
- Detector resolution

The breadth of a peak is usually expressed as the full width at half maximum, the FWHM. Generally speaking, the instrumental broadening increases with 2θ, and the instrumental broadening from a typical lab-scale instrument is shown in the figure.



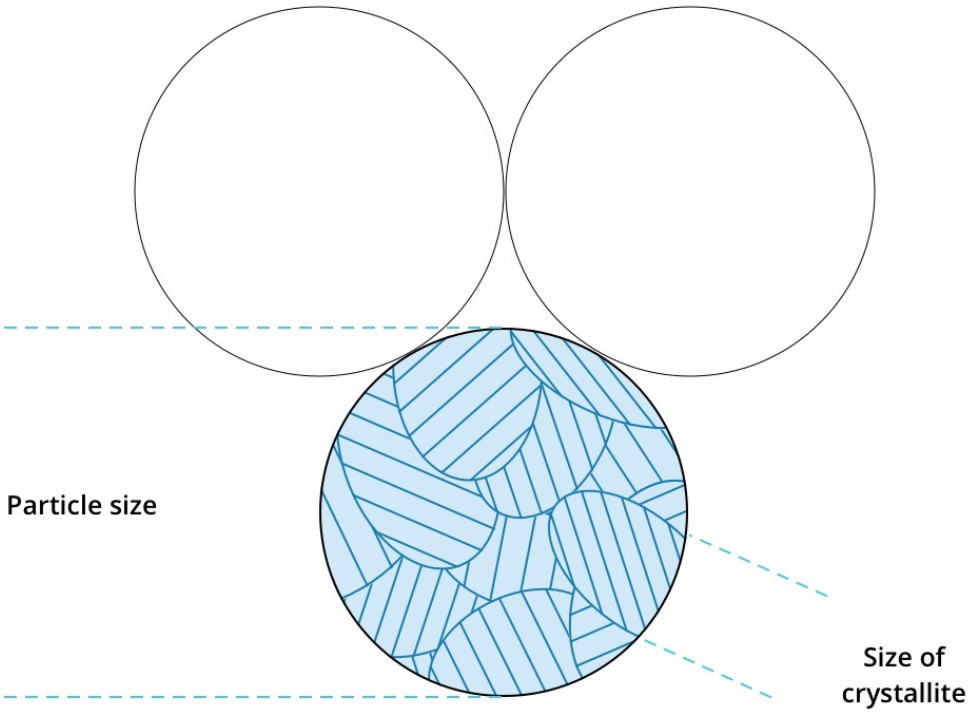
Instrumental broadening measured on NIST standard LaB_6 powder

If you choose to make instrumental broadening corrections manually, it is usually a good idea to fit a curve to the measured broadening. The most common function is a polynomial called a Caglioti function:

$$FWHM = (U.tan_2\theta + V.tan\ \theta + W)_{1/2}$$

To measure the instrumental broadening is a simple matter of carrying out a high quality scan on a certified standard reference material. LaB_6 and Si are common choices. A note of caution though, instrumental broadening changes with X-ray optics. You therefore need to measure the reference material and your samples using the exact same optic and detector settings to be sure that the instrumental broadening that your measured on the reference material matches exactly the broadening from your experimental measurement.

Schematic representation of the difference between a particle and a crystal



Crystal (grain) size broadening

Crystal size is a measure of a coherently diffracting domain, therefore a particle may be different to a crystal (see below). Many powders are agglomerates of smaller crystals, and in XRD we measure the individual crystals.

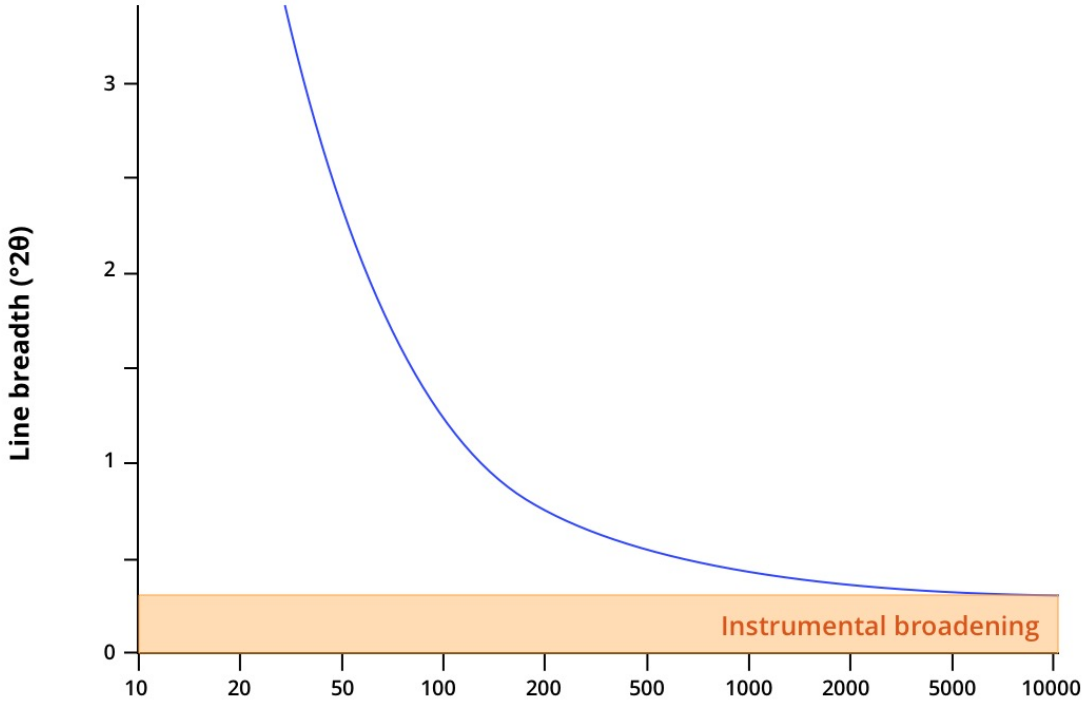
For crystallites of large size (i.e., thousands of unit cells), diffraction peaks will be produced only at the precise location of the Bragg angle. This occurs because of the strong coherent scattering within the structure and the cancelling of other diffractions by incoherent scattering within the large crystal structure. If the particle size is smaller (such that there are insufficient lattice planes to effectively cancel all incoherent scattering at angles close to the Bragg angle) the net result will be a broadening of the diffraction peak around the Bragg angle. Basically, the larger the volume of diffraction, the more perfect the peak. The peak width can be related quantitatively to the mean crystallite dimension by the Scherrer equation:

$$\tau = \frac{K\lambda}{\beta_\tau cos\theta}$$

Where β = peak broadening in radians (measured at the FWHM)
λ = wavelength
θ = Bragg angle
K = a shape factor usually assumed to be 0.9

Where β is the peak broadening in radians (measured at the FWHM), λ is the wavelength, θ is the Bragg angle and K is a shape factor usually assumed to be 0.9. A modified equation similar to this one but based on the integral breadth, rather than the FWHM, is more commonly used now because the constant in that case is independent of crystal shape.

From the Scherrer equation we can estimate the peak broadening for different crystal sizes. It can be seen that crystal size broadening is only measurable for grain sizes less than ~0.5 μm. For crystal sizes larger than this the crystal size broadening can be ignored.



Peak broadening due to crystal size.

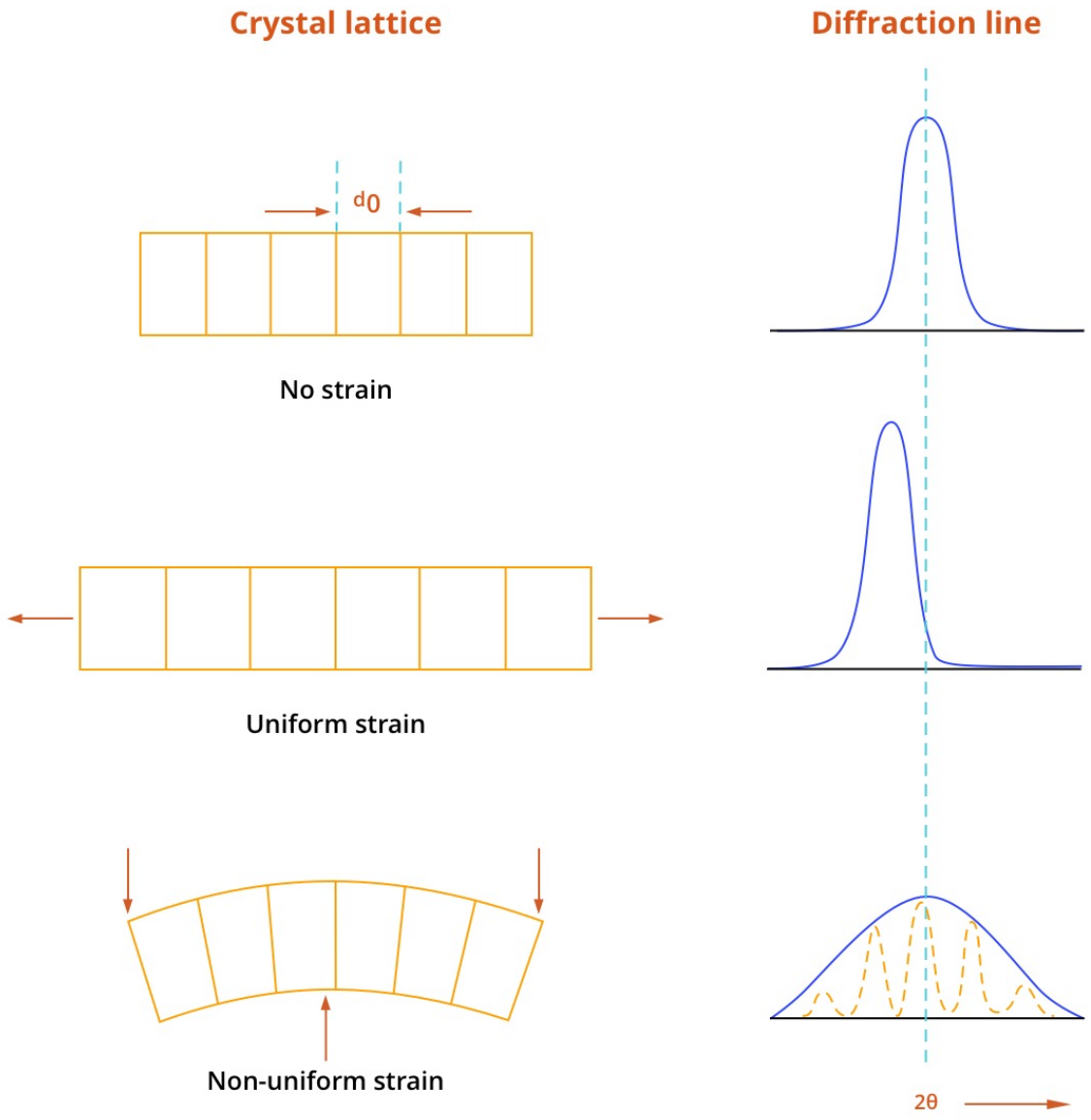
Dislocation density from microstrain

XRD measured the distance between diffracting planes. If we compress these planes, the spacing between them is reduced and the position of the diffracted peak will change accordingly. The same is true in tension, when the diffracted planes are separated and their spacing becomes larger, the diffracted peak will be shifted to a lower value of 2θ. This is shown below. However, if there are both tensile and compressive strains within the diffracting crystals, then some regions will be shifted to higher values of 2θ, while others will be shifted to lower values. The net result in this case is peak broadening.

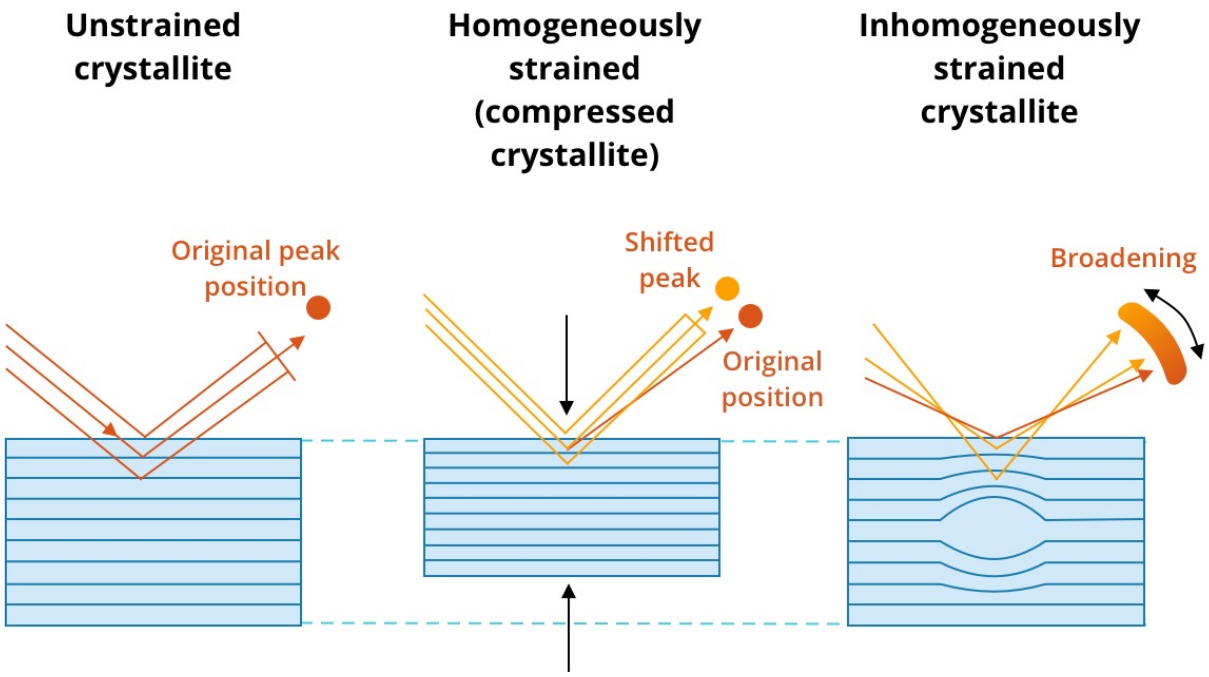
The microstrain is related to the peak broadening by the following equation:

$$\varepsilon = \frac{\beta}{2tan\theta}$$

Where β = peak broadening in radians
θ = Bragg angle of the peak



Effect of lattice strain on the peak position and peak widths observed during XRD



Schematic illustration of inhomogenous strain within a crystal

Converting microstrain to dislocation density

To use microstrain broadening to estimate the dislocation density you need to make some assumptions about dislocation arrangement. For cubic systems the Burgers vector only has one value, but for other crystal structures you may need to make assumptions about which dislocations are contributing to the broadening. Accurate assessment of dislocation density can only be made on samples where the crystal size broadening is negligible. Note that this refers not only to the grain size, but also to the subgrains, which may contribute to crystal size broadening.

The microstrain can be converted to a dislocation density by the following equation:

$$\varepsilon_o^2 = \frac{1}{4\pi} \rho C b^2 \ln \frac{R_c}{L}$$

Where ε_o= microstrain (proportional to peak width)
r = dislocation density
C = dislocation contrast factor
b = Burgers vector

For cubic systems the calculation is quite simple:

$$\rho = \frac{k\varepsilon_o^2}{b2}$$

Where ρ = dislocation density
ε_o= microstrain
b = Burgers vector
k = 16.1 for FCC metals
& 14.4 for BCC metals

For further information go to G. K. Williamson & R. E. Smallman Philosophical Magazine, 1:1, 34-46

Rietveld Refinement - What is Rietveld refinement?

Rietveld refinement is a technique devised by Hugo Rietveld for use in the quantification of crystalline phases from diffraction spectra. The Rietveld method uses a least squares approach to refine a theoretical line profile until it matches the measured profile. It includes an approximation of peak shape, peak width, preferred orientation, and of course all of the intensity predictions discussed in the previous section. It sounds complicated, but you don't have to write your own code, there are tools available for you to use that have already been developed such as programs like TOPAS and Siroquant.

Rietveld,H.M.(1969). J. Appl. Crystallogr.,2,65-71.

Broadly speaking there are two different approaches to quantitative Reitveld refinement:

- Traditional Rietveld refinement
  - assumes all phases are crystalline
  - is probably the easiest method
  - can be used on bulk solid or powder
  - cannot accurately quantify amorphous percentages unless additional measurements are made on standard specimens to accurately quantify the background.

- Internal standard method
  - only works on powders
  - you add a known amount of a known phase to your sample and mix thoroughly
  - can be used to quantify amorphous percentages very accurately.

Summary of analysis cues

Peak positions show:
- Crystal system
- Space group symmetry
- Translational symmetry
- Unit cell dimensions
- Qualitative phase information

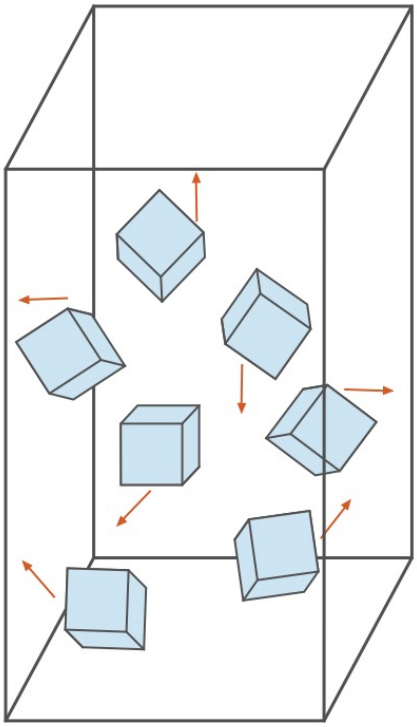
Peak intensities show:
- Unit cell contents
- Point symmetry
- Quantitative phase fractions

Peak shapes and widths show:
- Crystallite size
- Non-uniform microstrain
- Extended defects (stacking faults, antiphase boundaries etc.)

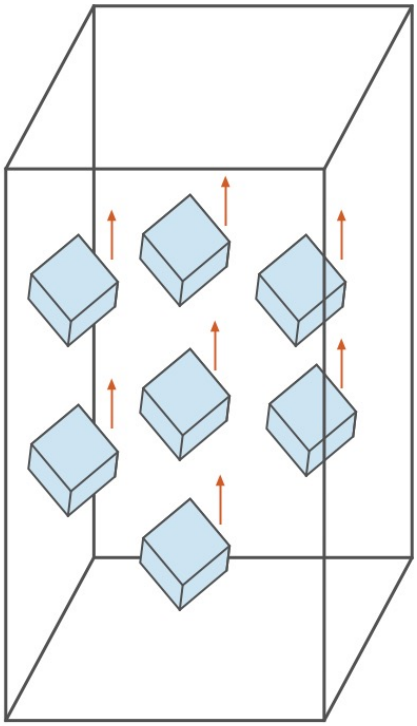
Limitations of powder diffraction:
- Single crystal techniques utilise mathematical algorithms and accurate peak intensities to solve structures
- The 3D collection of spots and intensities of a single crystal experiment are reduced to a 1D pattern. This causes peak overlap and makes accurate peak intensities problematic.
- Crystal symmetry is not obvious from the pattern.
- Multiphase mixtures complicate the issue.
- Preferred orientation leads to inaccurate peak intensities.
- One important thing to note about powder diffraction is that sample preparation must be done carefully and precisely to get a good result.

Specialist techniques using XRD - Texture - What is texture?

Texture is the word materials scientists use to describe the crystallographic preferred orientation that develops in many materials. This simply means that some crystals prefer to point in a certain direction, for example, if a plate is rolled, some planes might prefer to be aligned with the rolling direction. In geology, the term fibre is sometimes used instead of texture. Texture is a statistical measure of the way in which the crystals are oriented, so care must be taken to ensure a large number of crystals are measured. Many diffraction techniques can be used to measure texture, X-ray diffraction, neutron diffraction, and electron diffraction using the technique called electron backscattered diffraction **EBSD**.



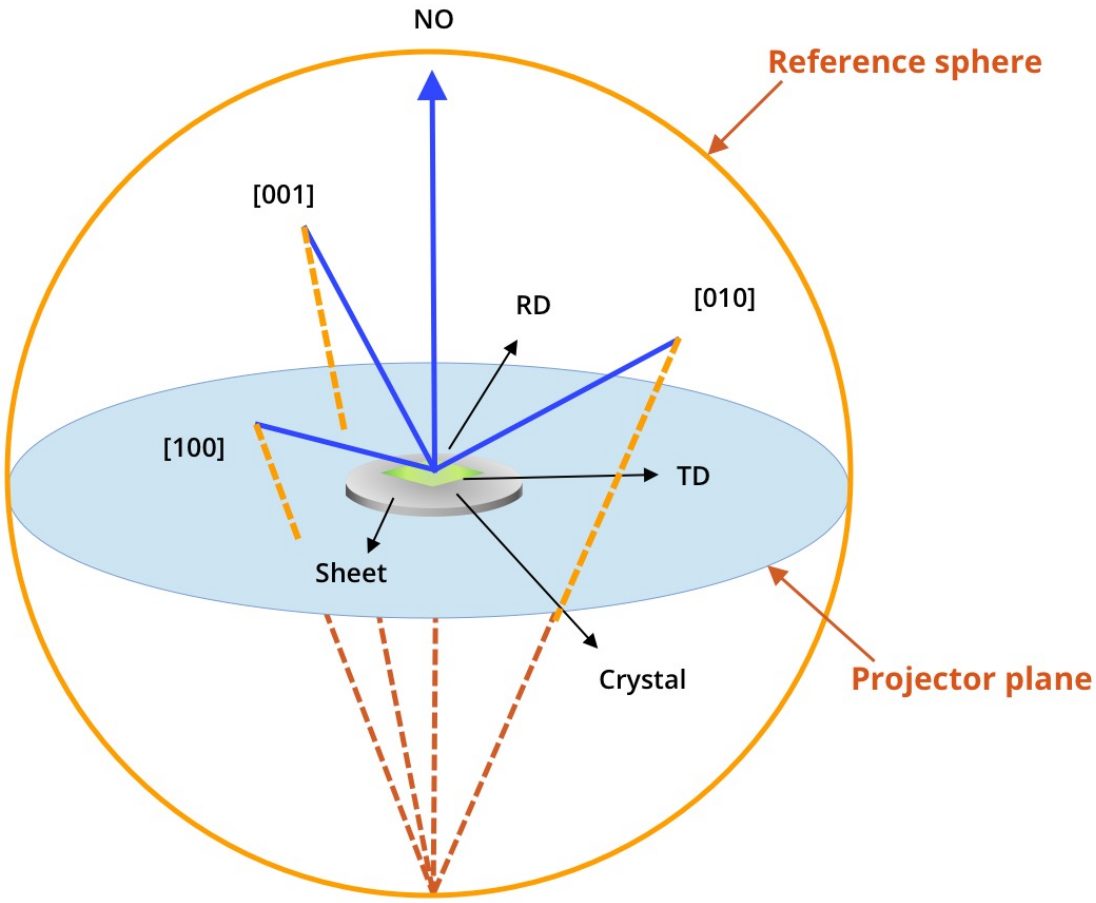
Random orientations



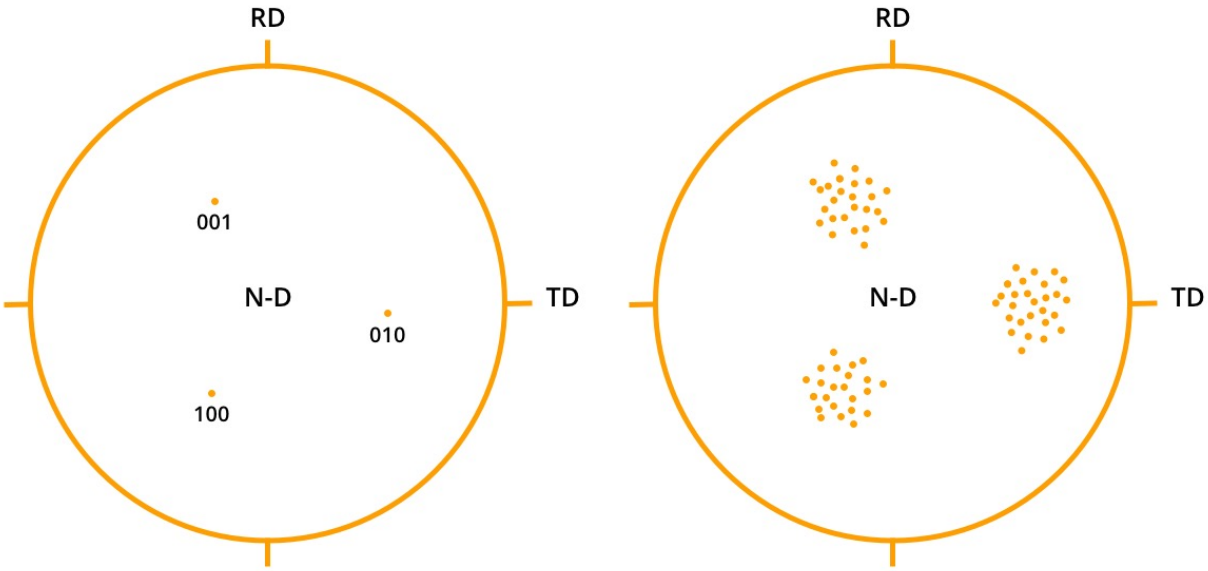
Preferred orientations

Displaying texture

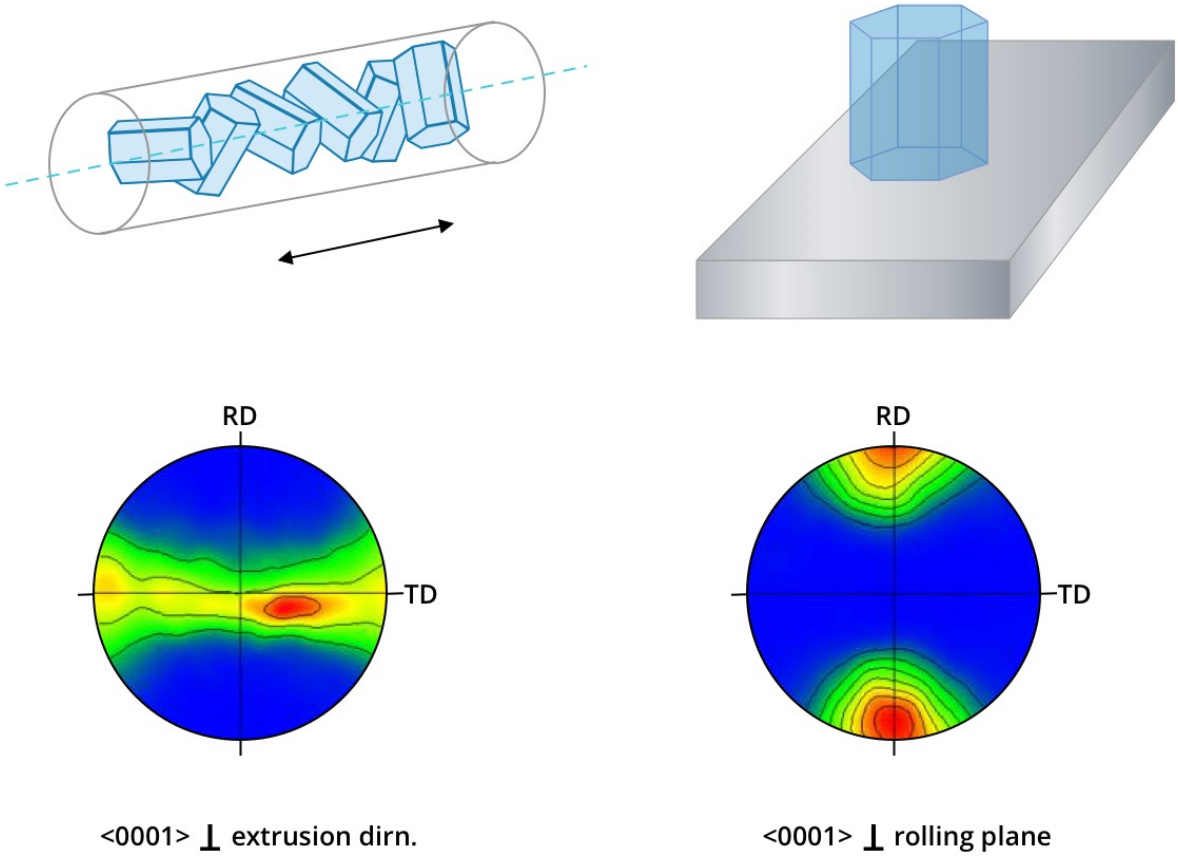
There are three main ways in which we display texture: pole figures, inverse pole figures, and orientation distribution functions. Pole figures are probably the easiest for people to visualise, and remain the most common way of displaying both texture and individual orientations from EBSD and XRD data. The pole figure is based on a spherical reference frame, and an imaginary line is drawn perpendicular from the crystal plane onto the sphere. A projection from this point on the sphere down to the flat projection plane completes the plot of this one point onto the pole figure.



When many points are placed onto the one pole figure, a clear preference for certain locations can be seen.



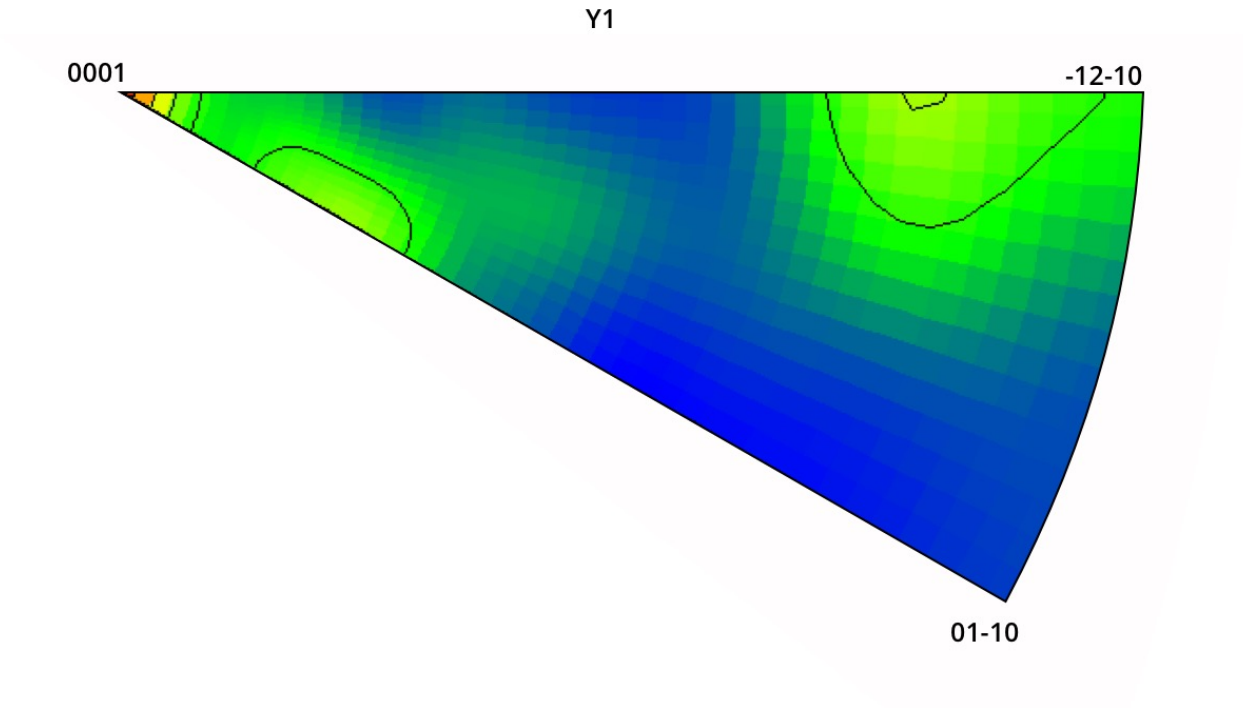
If we have measured sufficient data, a mathematical method known as spherical harmonics can be used to create a statistical fit of the individual datum points into a statistically correct curve. The pole figure can be considered to be a map of crystal orientations with reference to the sample geometry. It shows how one crystal plane prefers to be aligned within the specimen geometry.



<0001> ⊥ extrusion dirn. <0001> ⊥ rolling plane

An inverse pole figure, as the name implies, plots data as a mirror of the pole figure. The inverse pole figure is a map of the specimen direction or geometry with respect to the crystal directions. The IPF shows which of the different crystal orientations are preferred by the specimen geometry. Usually we plot one direction like the rolling direction, or the direction of solidification. The IPF has a triangular shape with one curved edge, and each edge of the IPF represents one of the main crystal planes.

Inverse pole figure



Another method of describing the direction a crystal is pointing is to use a co-ordinate system known as Euler angles. This is also used in other fields such as astronomy. The three angles, Euler 1, 2 and 3, can be plotted in the same way as we would plot any other set of three Cartesian co-ordinates. Just like the pole figure, the regions where there are many points in a cluster indicate those regions that are preferred in the specimen. The data can be contoured by spherical harmonics just like the other two methods of showing texture. Some researchers prefer this description, but it is apparent that obtaining any reference between the ODF and the specimen geometry requires extensive experience.

Measuring texture with X-rays

Measuring texture on a lab-scale instrument is relatively straightforward, but additional rotation axes are required on the instrument. This is sometimes referred to as a texture cradle, or texture goniometer. The goniometer allows the specimen to be rotated to ensure that the different directions get measured. Unlike conventional 2θ scanning where the specimen is stationary and the detector and X-ray tube move, to measure texture we set the Bragg condition, but leave these optics stationary. Only the specimen is rotated to look at the intensity change of one peak in all directions. This kind of measurement uses different optics, the Bragg-Brentano setup uses line focus optics, but for measurements that involve specimen tilting, or extremely rough specimens, point focus optics are used.

Before plotting the data, we do three corrections.

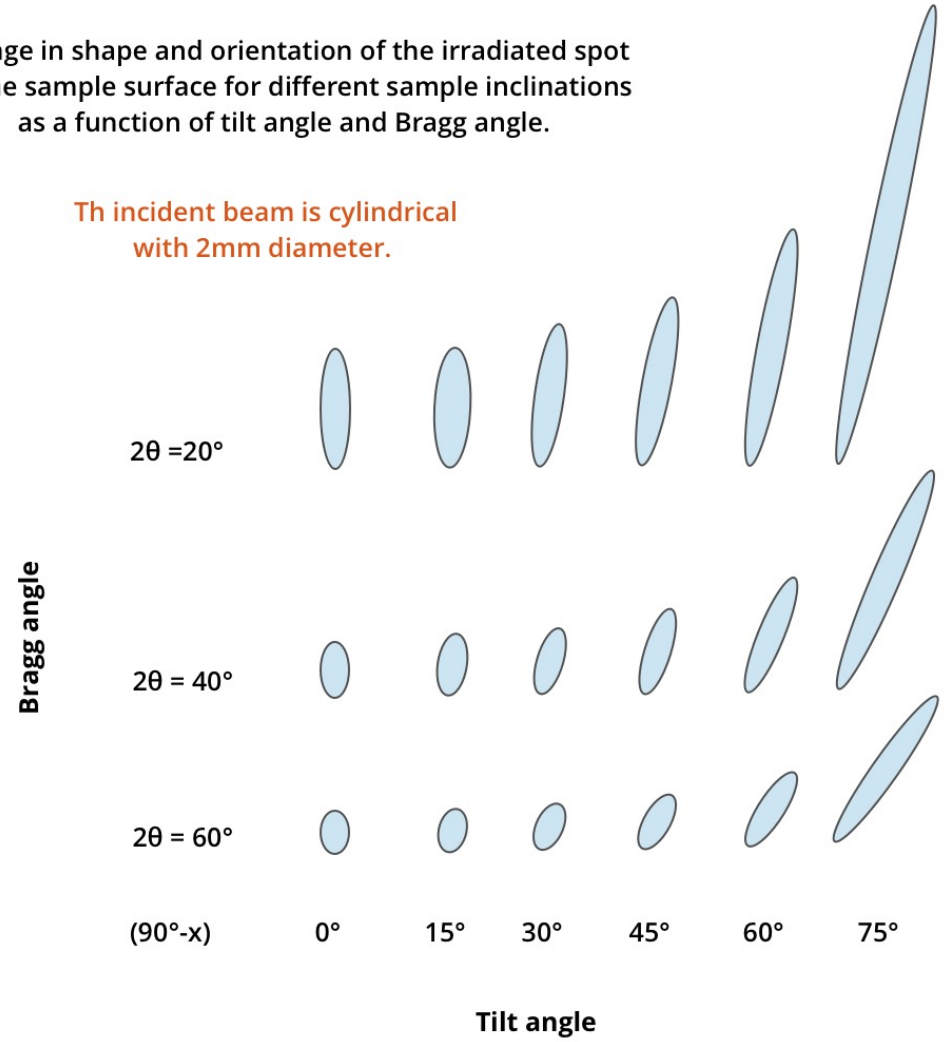
Background correction

The background is removed from the peak of interest using data from either side of the peak.

Defocussing correction

As the sample is tilted to high angles, the beam illuminates an increasingly large area, and the detected intensity drops for most instruments above a tilt angle of about 60 degrees. The drop in intensity at high tilt angles is measured on a random reference of the same material as that being measured. These can be purchased commercially, but for unusual materials, your own random reference can be made by simply mixing a powder of the material with enough epoxy to bind it into a solid. Standard sample prep techniques can then be applied. Once the random reference material has been measured, the defocusing correction can be applied. The method of converting the measurement into a correction is slightly different for different software packages, some apply the reference measurement directly to your experimental data, others fit a mathematical curve to the reference measurement and then use this on their data. Either way, the defocusing correction must be measured individually for each different set of planes being measured.

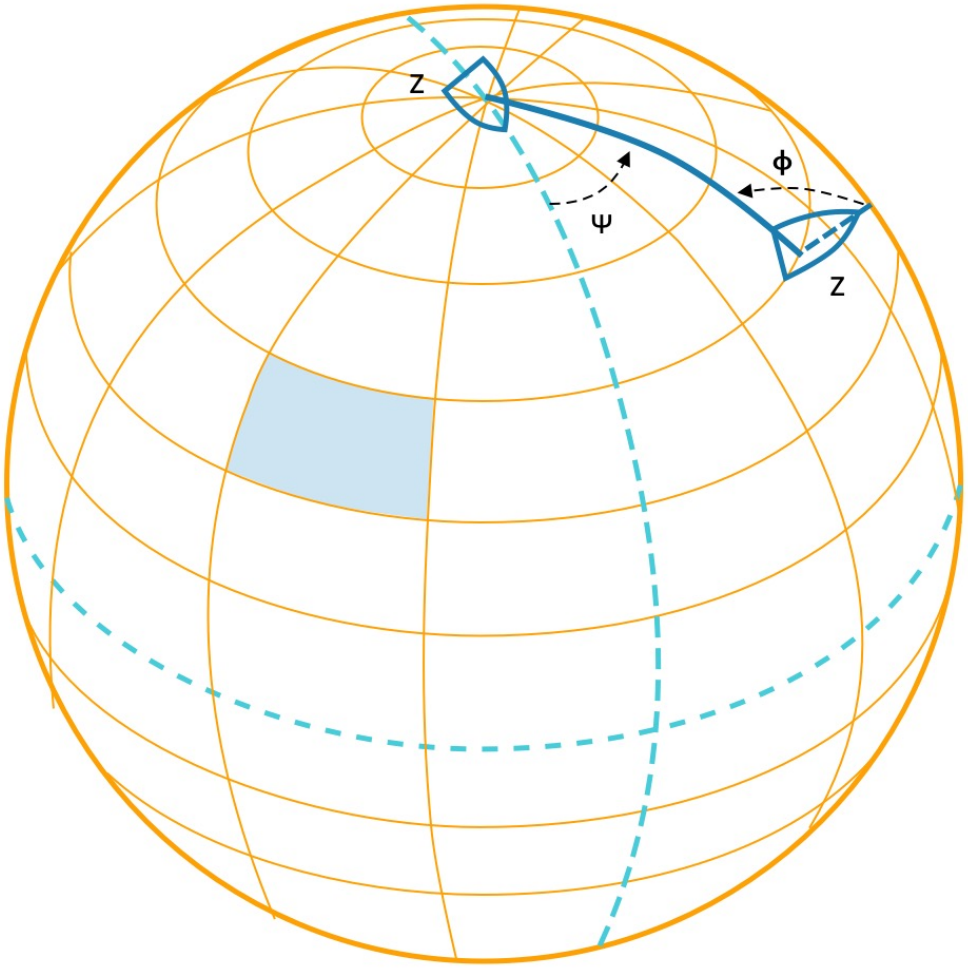
Change in shape and orientation of the irradiated spot on the sample surface for different sample inclinations as a function of tilt angle and Bragg angle.



[Kocks]

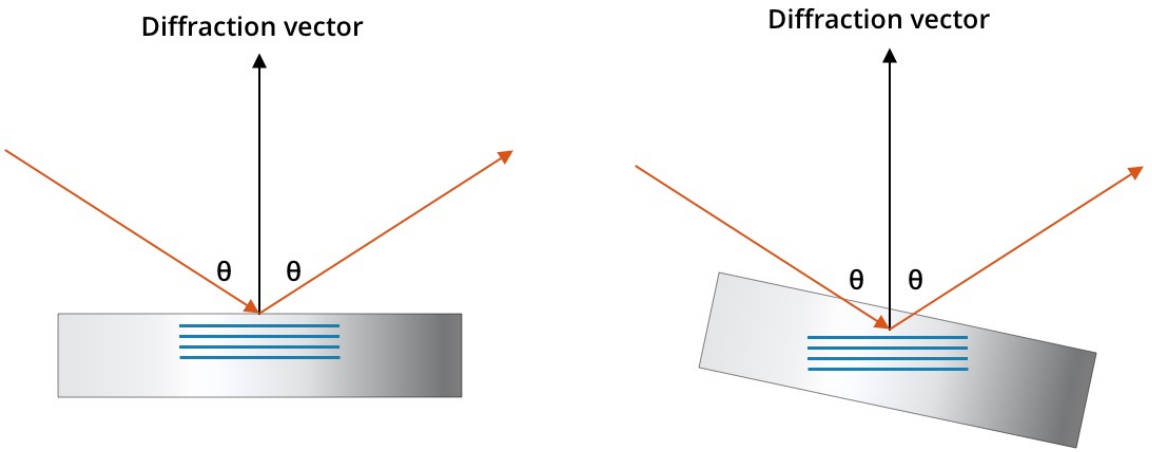
Normalisations

Normalisation is the mathematical way in which we take a direct intensity measurement and convert it into the “times random intensity”, a normalisation procedure when ensures that a truly random sample will have an intensity of one. Most plotting software will do this correction for you, and is simply a matter of summing all intensity, dividing each measurement by this sum, and applying an area correction. The area correction takes into account the different areas on the surface of a sphere depending on the latitude (in other words, the area of the boxes at the top of the sphere are smaller than the ones close to the hemisphere)



Residual stress

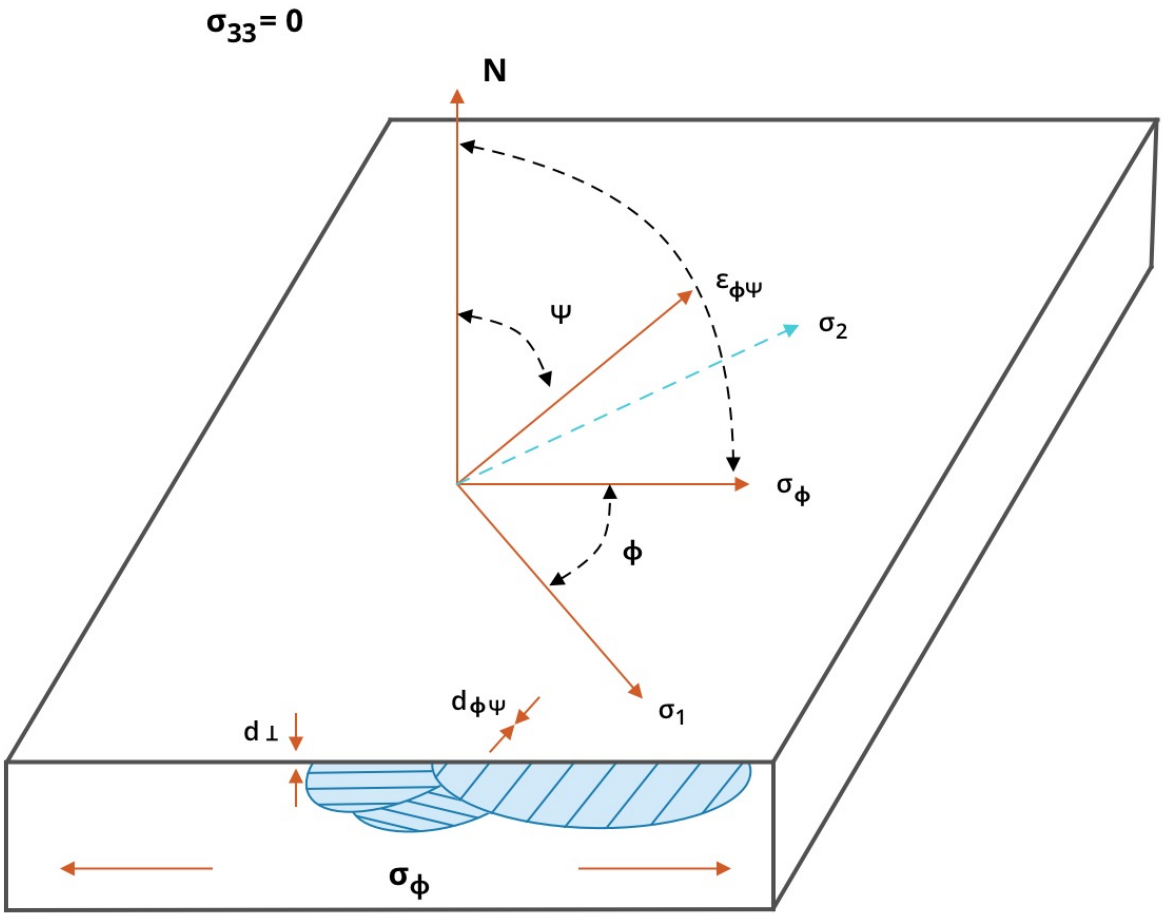
Residual stress is usually measured on the same instrument as texture measurement, those that can allow tilting of the specimen. The residual stress measurement is based on the notion that the crystal planes parallel to the free surface (the one being measured) are free of stress, and therefore have a d-spacing indicative of zero stress. We usually measure diffracted peaks parallel to the specimen surface.



If the specimen is now tilted, the same family of planes is still being measured, because we are still at the same Bragg angle, but these planes are now not parallel to the free surface, and may therefore be under stress. A typical change in peak position as a function of tilt angle is shown here for rolled aluminium.

The complex geometry of the residual stress measurement necessitates more complex nomenclature than the figure shown above, and the stress normal to the plane is denoted σ33, the rotation around this plane is termed ϕ , and the specimen tilt termed ψ.

The strain in the lattice is determined by the usual way we determine strain in any system, simply the change in length divided by initial length, but in the case of this measurement we use the lattice spacing as our measurements: ε


σ33= 0
N
ε ϕψ
ψ
σ2
σϕ
ϕ
σ1
d ϕψ
d ⊥
σϕ

The full 3-dimensional description of residual stress can be derived as:

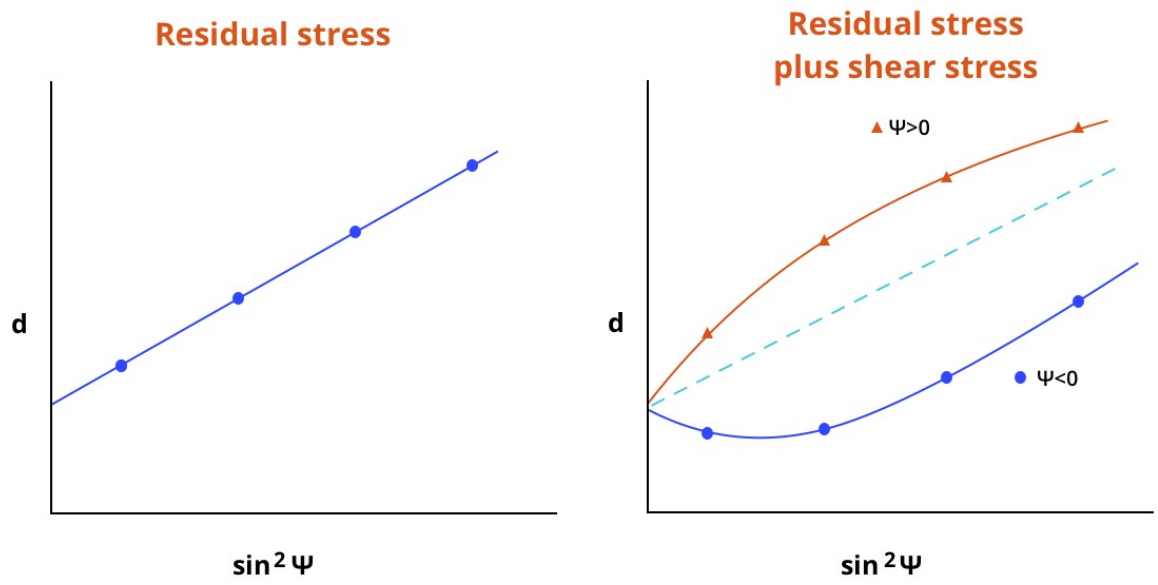
$$\varepsilon_{\psi\phi} = \varepsilon_{33} + [\varepsilon_{11}\cos^2\phi + \varepsilon_{12}\sin 2\phi + \varepsilon_{22}\sin^2\phi - \varepsilon_{33}]\sin^2\psi + [\varepsilon_{13}\cos\phi + \varepsilon_{23}\sin\phi]\sin 2\psi$$

Where ε_{33} = 0 because the free surface is not constrained
ϕ = 0° (unless multiple measurements are made at different sample rotations)

Therefore the equation reduces to:

$$\varepsilon_{\psi\phi} = [\varepsilon_{11}]\sin^2\psi + [\varepsilon_{13}]\sin(2\psi)$$

If we assume firstly that there is no shear stress, we can simply plot $\sin^2\varepsilon$ as a function of d-spacing. If this is a straight line, the slope = the residual strain. To convert to stress, multiply by Youngs modulus. Use a modulus from literature, don't try and measure it yourself because elastic moduli are notoriously difficult to measure and need special equipment.

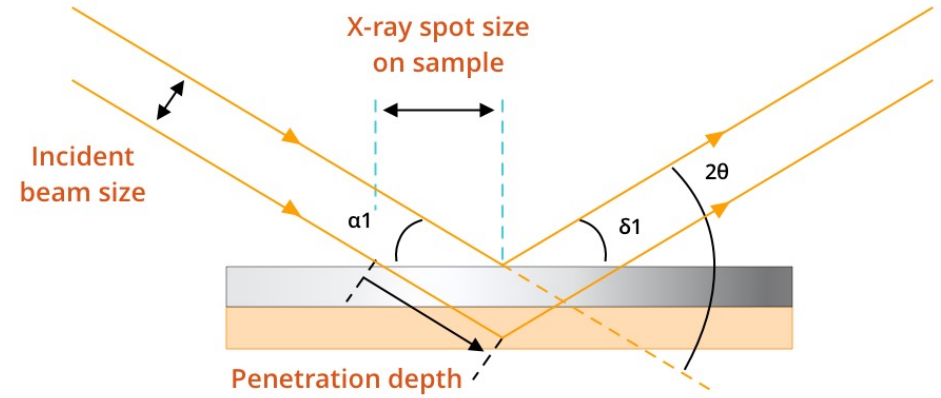


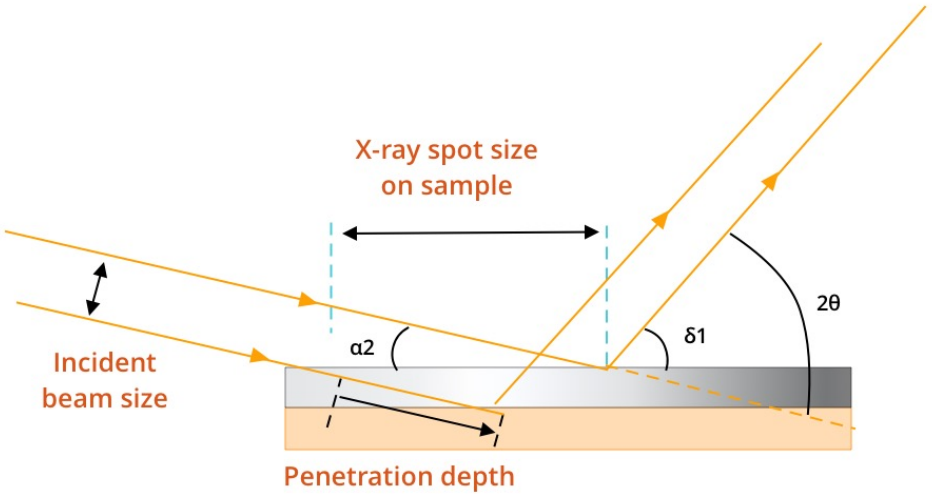
If your data shows psi splitting it means you have a shear component. You will not get both sides of the split, just one, but you can measure the other by rotating your sample by 180° and measuring the residual stress again (However, this is typically not required). Using the same plot you will be able to solve for ε_{11} and ε_{13}.

You will notice that at high tilt angles the intensity of the peak decreases due to the defocusing issued discussed in the texture section. This is not a concern of residual stress measurement because it is only the peak position that is

Glancing angle XRD

We saw in the section on penetration depth that at higher angles of 2θ, the X-ray diffraction signal came from deeper within the specimen. In these cases where we have a thin film such as a coating, it is advantageous to remain at a low angle so that more of the diffracted signal comes from the surface. This is called glancing angle XRD, and can be carried out on some lab-scale instruments, although most of this work is increasingly being carried out at synchrotrons. The physics of the experiment is the same: the incident beam is kept at a low (glancing) angle, and only the detector is moved through the full range of 2θ required for the experiment. For those instruments with a 2-dimensional detector the experiment is simply a case of setting up the beam at a glancing angle with the specimen, and measuring the full 2-D diffractogram.





Credits

Microscopy Australia acknowledges the huge input of time and expertise by the many staff members and associates who have contributed to the development of MyScope over the years.

For the XRD module we thank: Nikki Stanford, Kevin Jack, Matt Foley, Ruth Williams, Aaron Dodd, Anya Yago, Philip Francis.