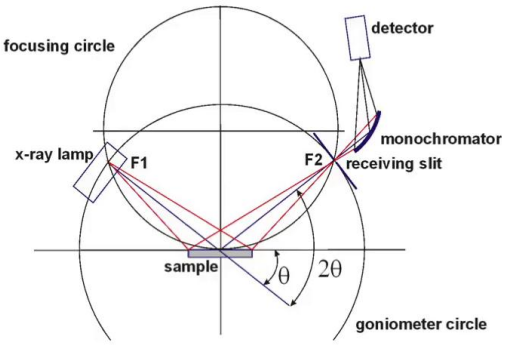
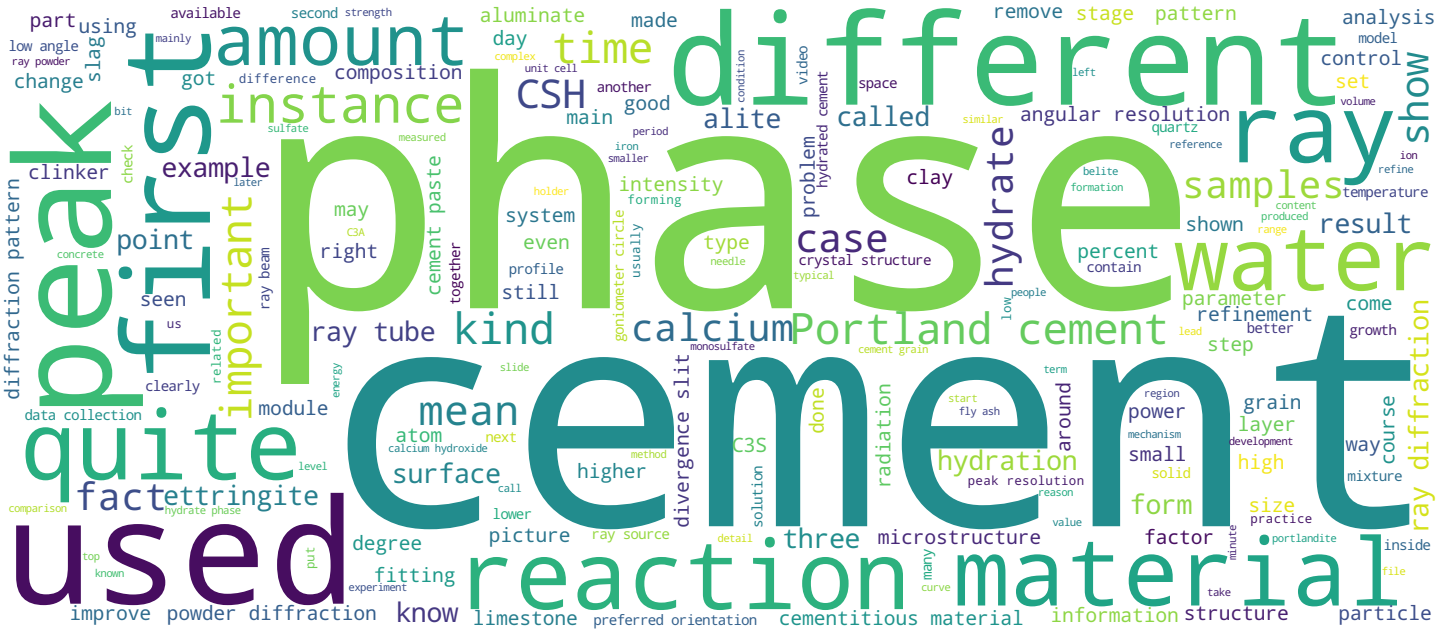


X-ray powder diffraction equipment and data collection

Cement Chemistry and Sustainable Cementitious Materials

Dr. Ruben Snellings



Search MOOC

Video

EPFL

Calcite [$CaCO_3$]

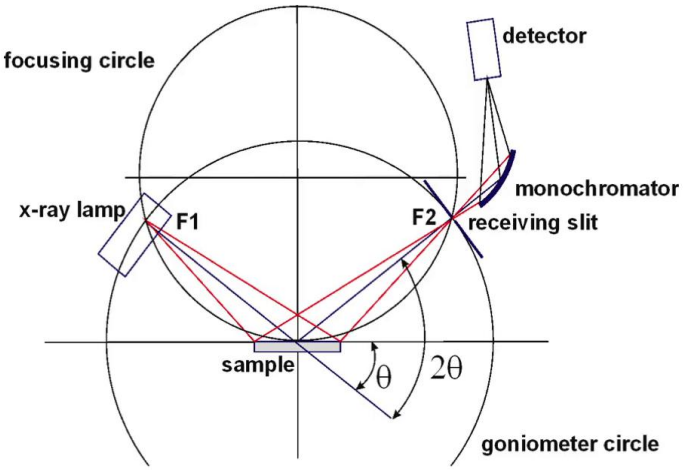
Portland cement

X-ray diffraction methods measure the interference pattern produced by the coherent scattering of an incoming monochromatic X-ray by a three dimensional periodic created material, here the crystal lattice. For a specific crystal lattice, constructive interference, peaks of scattered X-rays and the diffraction pattern takes place only at angles defined by the specific repeat distances of the lattice. As seen in the previous lecture Bragg law defines this relationship. X-ray diffraction experiments can be conducted on single crystals but also on powders. Powder diffraction has the advantage that mixtures of phases can be investigated relatively easily and no- relatively rare well formed crystals are necessary. Instead in powder diffraction, the original material is ground very finely and mounted on a sample holder. The powder amount consist ideally of crystalline particles in completely random orientation. If the orientation is truly random then for each characteristic repeat distance d there are many particles that satisfy the Bragg law.

Notes

Summary

0m 04s

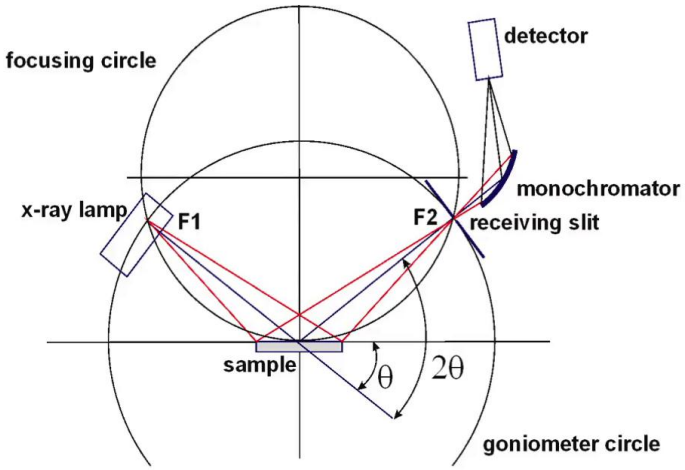


Flat powder mounts are usually measured in reflection or so-called Bragg-Brentano parafocussing geometry.

Notes

Summary

1m 21s

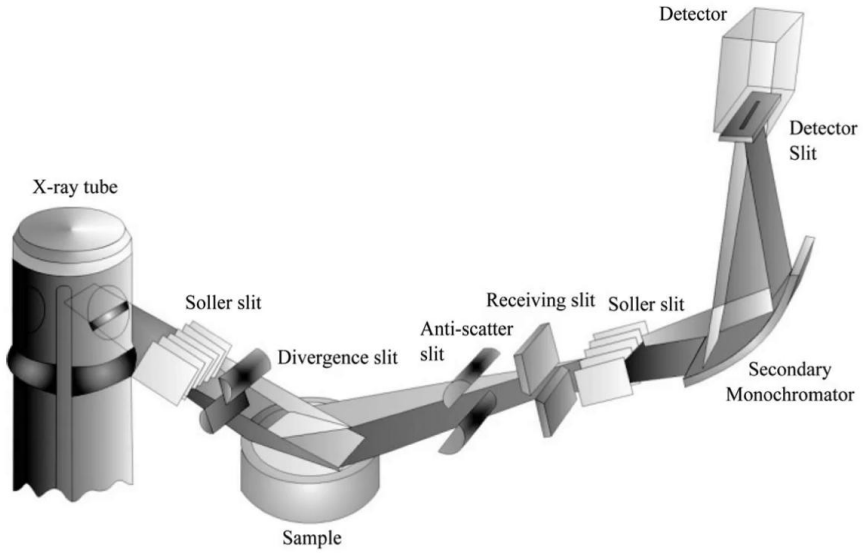


In this geometry, the incident beam from the line focus of the X-ray tube diverges until it is reflected by the sample, the diffracted beam then converts from the sample and is focused at the point where the focusing circle and the goniometer circle intersect. Here, a receiving slit is positioned to filter out any unfocussed radiation. A secondary monochromater can be used to ensure that only elastically scattered radiation, this is radiation that has the same wavelength as the source, is allowed to enter the detector device. The letter measures considerably improve the angular resolution. This will make peaks become narrower thus showing less overlap with other reflection peaks. Various Bragg-Brentano configurations exist. In the theta-theta system, both the X-ray source and the detector system rotate simultaneously along the goniometer circle, the sample stage in the center of the goniometer circle is fixed. Thus if the angle between the surface normal and the incident beam has changed by theta in fact the angle between the incident and the diffracted beam has changed by 2 theta.

Notes

Summary

1m 31s

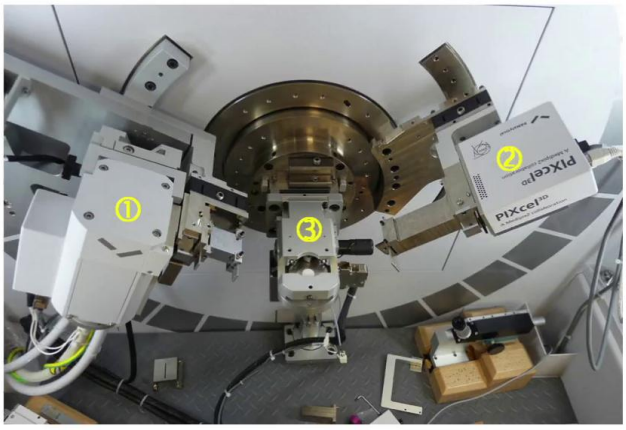


The peak to background intensity and the peak resolution are the most important parameters to assess the quality of a diffraction scan. Increasing the peak to background ratio can be done by increasing measurement times, increasing X-ray source intensity or decreasing background scattering intensity. To improve the quality of the XRD data, several so-called optical devices are used to focus, collimate and filter the X-ray beam. Soller slits, divergence slits, anti-scatter slits and receiving slits are all used to condition the beam and remove background noise. Increasing the peak resolution often involves cutting a part of the incoming reflected intensity that leads to peak broadening and thus reduces overall peak intensities. Thus in practice, a compromise needs to be found among different data quality measures. Also taking into account practical considerations such as the available time for measuring and the acceptable sample exposure time.

Notes

Summary

2m 51s



1. X-ray tube
2. Detector
3. Sample stage

Finally in practice if you look at the X-ray powder diffractometer, it looks a lot like in these pictures. On the left side you can recognize the X-ray source or the tube. In the middle, the sample stage and on the right hand side you see the detector. In between source and sample and sample and detector optical devices are positioned. The X-ray tube requires cooling and is usually connected to an external cooling circuit. The high voltage generator for the X-ray tube is usually positioned underneath. During the experiments the X-rays are shielded off by leathered casing or lead glass to protect the operator.

Notes

Summary

4m 02s

Data collection checklist

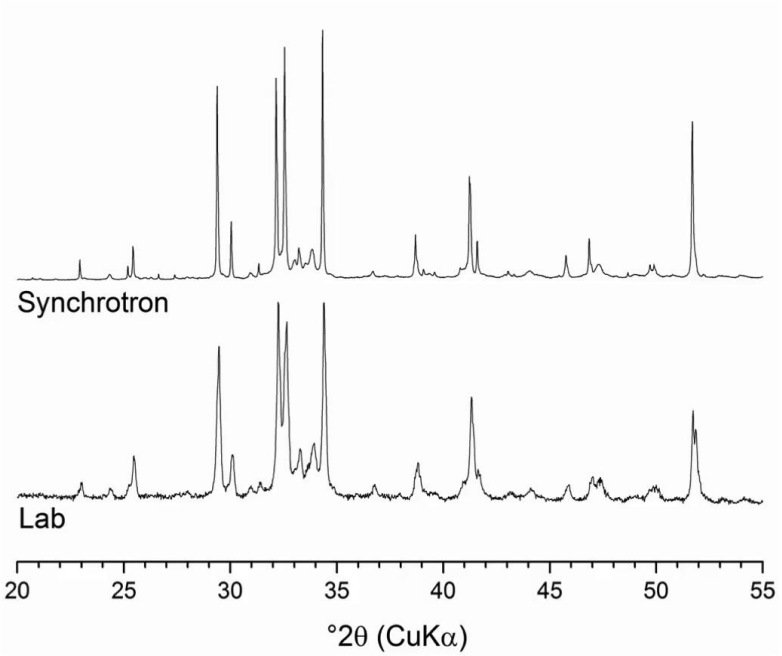
- ✓ Alignment of diffractometer
- ✓ Measurement configuration (optics, sample dimensions)
- ✓ Angular range of data collection (2θ)
- ✓ Angular step size
- ✓ Counting time per step
- ✓ Sample exposure

Before you start data collection, it is important to check if the diffractometer system is well aligned and that a suitable measurement configuration is chosen. In case of a flat plate reflection geometry, a correct combination of optic elements and sample size should ensure that neither sample transparency nor beam overflow at low angles occur. Data collection strategies should aim at collecting a maximum of useful information from the sample. Therefore the data collection range for Portland cements is usually chosen to be within let's say 5 to 70 degrees 2 theta for copper K-alpha radiation but it can be wider or more restrictive depending on the problem. The 5 to 70 degrees range provides key information on hydrate phase reflections at low angles and covers the most intense reflections of both anhydrous and hydrate phases. For most in-house diffractometers a step size of about 0.02 degree 2 theta is used and the counting time per step is usually a compromise between the optimal signal to noise ratio and practical considerations such as the available time for measurements or acceptable sample exposure.

Notes

Summary

4m 46s

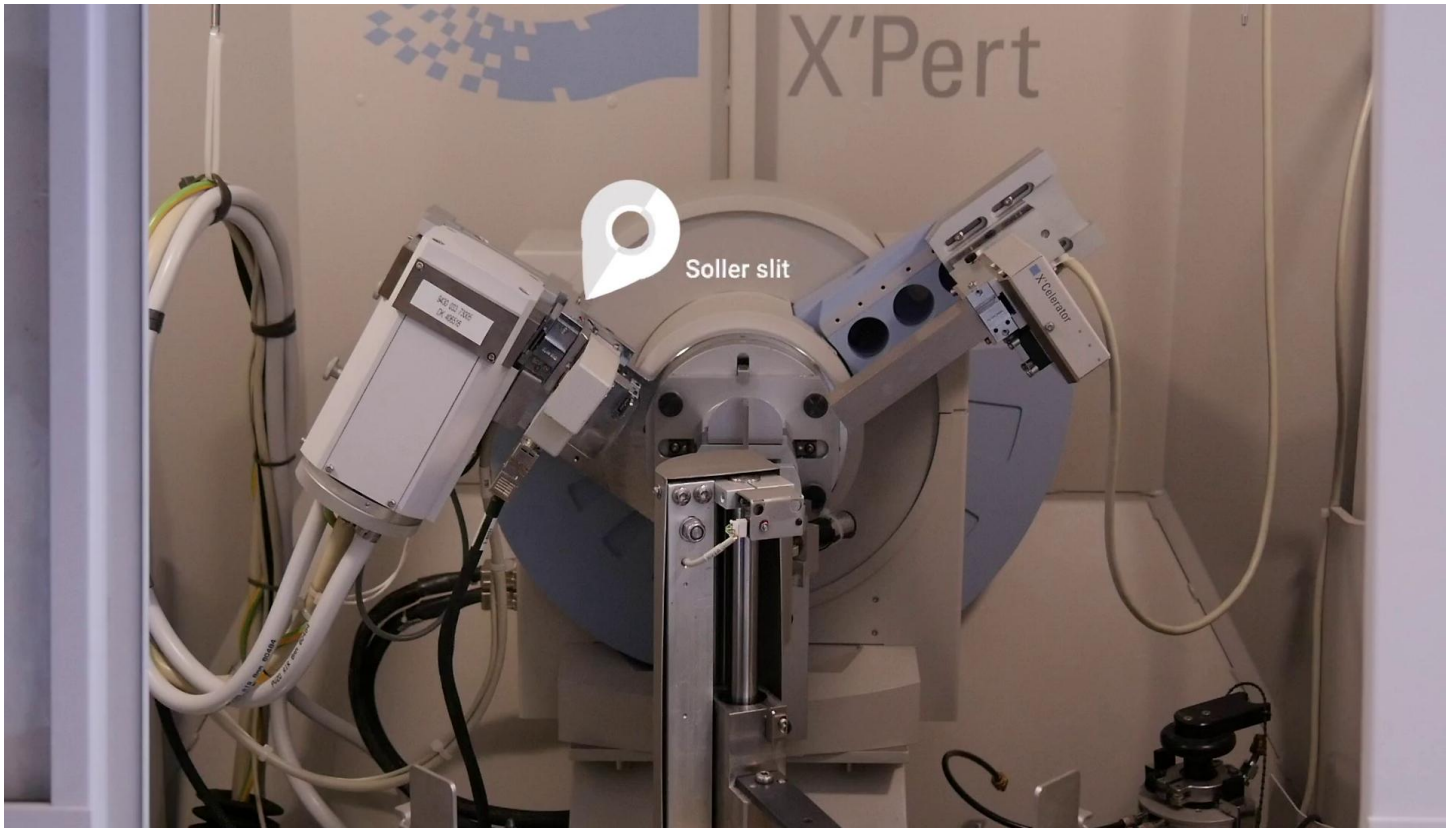


The development of high performance X-ray detector systems in the last decades has allowed to actually drastically reduce measurement times and thus to minimize exposure times of hydrated samples to a couple of minutes. It is advisable also to spin your sample in the horizontal plane to improve the particle statistics of the measurements when doing powder diffraction. This figure gives us an example of the dependence of data quality on the type of radiation that you use. Here a comparison is made between the diffraction patterns of two different Portland cement, one measured by a lab diffractometer and the other by a synchrotron XRD. The main advantage of using a synchrotron is the much higher monochromaticity and the intensity of the X-ray radiation. This greatly improved peak resolution enables much shorter data collects and times and this is useful for instance for early age cement hydration experiments where you want to have very short experiments measurements for instance a couple of minutes.

Notes

Summary

6m 01s

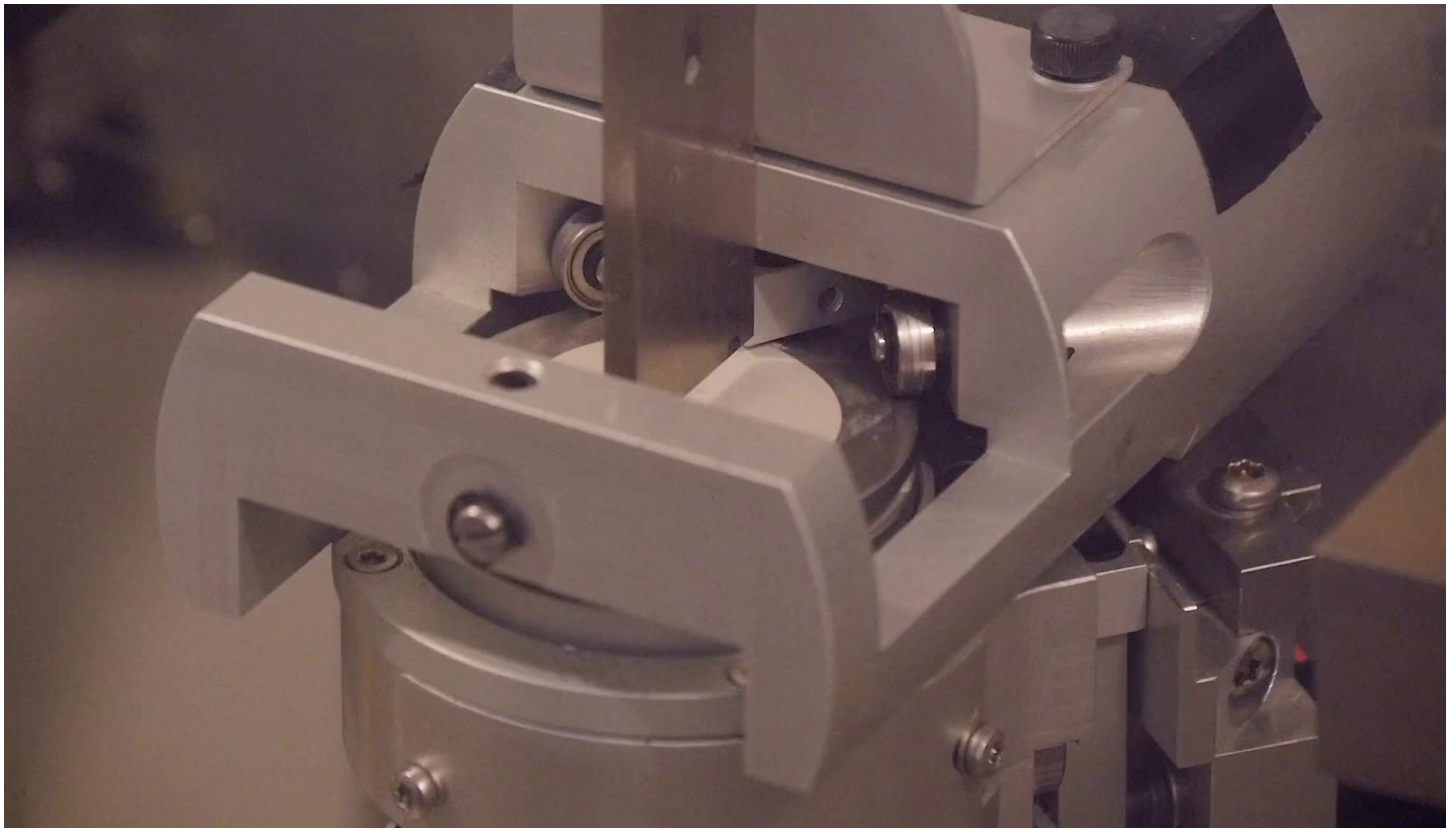


You can discover the insides of a real lap diffractometer in this demonstration video. The video will also show you the operation of the machine during an X-ray powder diffraction measurement. So please have a look at the demo. Hello I am Dr. Xuerun Li and as you know, I will do the demo videos. So here is our PANalytical X'Pert XRD diffractometer. The X-ray was generated by X-ray tube. Here we are going to use the characteristic radiation from copper. The X-ray tube is cooled, we use cooling water. The power of the X-ray diffractometer was determined by the power of the tube and the precision of the detector. Ideally the more powerful, the better. A typical laboratory X-ray tube is working at 45 kV and 40 mA. Unfortunately the age of the tube is limited because of the decay of the intensity, typically 10 percent per year. The X-ray passed through a set of opticals onto the samples, then was collected by the detector. the K beta filter, namely the Nickle filter, is used to remove the K beta radiation. The set of metal plates called the soller slits removed the most diverging X-rays, letting the more parallel X-ray pass. A mask is used to control the length of the X-ray beams which is related to the size of the sample holder.

Notes

Summary

7m 09s

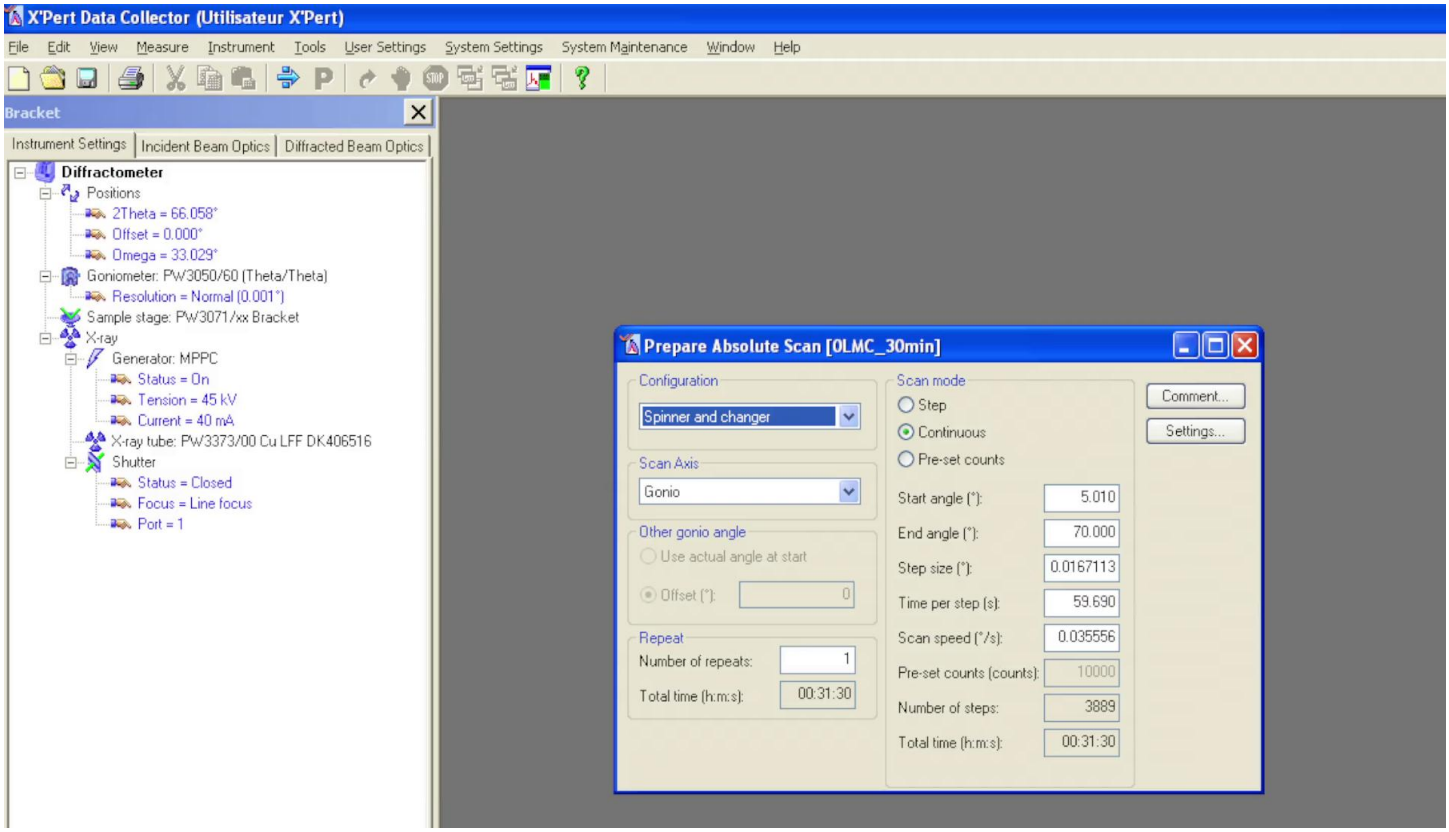


Another important slit, called the divergence slit, is used to control the width of the beam which is related to the angular resolution of the pattern. The smaller the divergence slit we used, the higher the angular resolution. However the intensity of the diffracted X-rays will be decreased when smaller divergence slit is applied. Sometimes the beam knife just on top of the samples will be applied to remove the scattering of the X-rays at lower angles which is important to the sample presenting peaks at lower angles. Some of the sample stage are equipped with the sample spinner which rotates the sample during the data acquisition. Whenever the spinner is available, it is recommended to spin the sample to improve the statistics of the particles.

Notes

Summary

8m 51s



The configuration of the XRD is mainly to optimize the X-ray beam to give the highest intensity and the required angular resolution. At the same time the beam overflow and the scattering at low angle should be avoided. A range of 7 to 70 degree of 2 theta using copper radiation for the data acquisition is usually enough to cover most of the peaks for some cement related samples. The scan speed can vary from lab to lab, depending on the power of the XRD. Typically 30 minutes of scanning within a range of 7 to 70 degrees is good enough to perform the Rietveld analysis. While for fresh disks, the scan should be acquired within 15 minutes to prevent the drying and the carbonation of the fresh samples. For some specific cases such as the comparison of polymorphous tricalcium silicate, enhanced angular resolution and peak intensity for selected range of the pattern can be more useful instead of the full pattern.

Notes

Summary

9m 46s