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Bent-Laue

5.3 Theory

Bent-crystal optics has been proven to have distinct advantages over X-ray mirrors and flat-crystal monochromators. The acceptance of X-rays can be considerably increased because of the much larger Bragg angles \( \theta_b \), while the bending simultaneously provides focusing and broadens the rocking curves. It will be described first how crystal bending can be utilised to produce a broad-energy X-ray band-pass beam with a constant intensity distribution.

5.3.1 Focusing

Focusing of X-rays by bent crystals is based on a change in the Bragg plane orientation. The focal distances \( p \) (source to monochromator) and \( q \) (monochromator to focal spot) are related through

\[
q = -\frac{q_0}{2 - \frac{p_0}{p}} \quad (5-1)
\]

where

\[
p_0 = \rho \gamma_0 = \rho \cos(\chi \pm \theta_b)
q_0 = \rho \gamma_0 = \rho \cos(\chi \mp \theta_b) \quad (5-2)
\]

are those for monochromatic focusing\(^{[3,4]}\). The parameters \( \gamma_0 \) and \( \gamma_0 \) denote the direction cosines of the incident beam and the reflected beams, respectively, and the asymmetry angle \( \chi \) is the angle between the Bragg planes and the surface normal of the crystal. Furthermore, the bending radius \( \rho \) is positive when the beam is incident on the concave crystal surface, and \( p \) is positive for a real source.

5.3.2 Energy dispersion

The bending of the crystal allows the energy dispersion to be increased or decreased for a given divergence \( \Delta \theta \) of the incident beam with respect to the flat-crystal case, i.e. \( \Delta E/E = \Delta \theta \cot \theta_b \). In the focusing Laue-case geometry, the beam impinges on the convex crystal side and the beam...
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...divergence and the angular change of the Bragg planes over the beam footprint have to be added.
For a bent crystal in Laue geometry the energy dispersion is given by

$$\frac{\Delta E}{E} = \cot \theta_n \frac{h_n}{p} \left[ \frac{p}{\rho \gamma_\rho} - 1 \right]$$  \hspace{1cm} (5-3)

where $\gamma_\rho = \cos(x^\pm \theta_n)$ and $h_n$ is the horizontal beam size. Thus, for a given beam-line geometry the energy width or energy dispersion can be modified by adjustment of the width of the horizontal beam size, the asymmetry angle and the bending radius.

5.3.3  Rocking curve width

When the crystal is cylindrically bent the Bragg planes are curved and their spacing and orientation change according to the elastic compliances. Several models exist for the theoretical description of X-ray diffraction in distorted crystals\textsuperscript{5,10}. In the geometrical theory outlined by Penning and Polder\textsuperscript{11}, the propagation of a ray in a crystal where the reciprocal lattice vector changes slowly is treated analogously to the propagation of light in an inhomogeneously diffracting medium. When the deformation is not too strong, the X-ray wave fields adjust themselves to the slowly varying lattice parameters and their propagation can be described locally in terms of the dynamical theory for perfect crystals. Penning and Polder demonstrated that the adjustment of the wave fields to the local lattice parameters is equivalent to a gliding of the tie-points along the dispersion surface. Since each tie-point represents a finite angular deviation from the kinematical Bragg angle, the shift corresponds to a broadening of the rocking curve by an amount given by

$$\Delta \theta_n = \frac{T \sin \chi}{\rho \gamma_\rho \cos \theta_n} \left[ 1 + \frac{1}{2} \left( \cos 2 \chi + \cos 2 \theta_n \left( 1 - \frac{s_2}{s_{11}} - \frac{s_1}{s_{11}} \right) \right) \right]$$  \hspace{1cm} (5-4)

where $T$ is the crystal thickness and $s_\rho$ denotes the elastic compliance for the given crystal material and orientation\textsuperscript{12}. As a further consequence of the tie-point shift, the reflectivity of the bent-Laue crystal increases. In general, it exceeds the value of 0.5 known for thick, flat crystals and is ultimately limited only by anomalous absorption. When the bending radius is further decreased the creation of new wave fields\textsuperscript{13} extracts intensity from the diffracted beam in such a way that for a strongly deformed crystal the integrated reflectivity approaches the kinematical limit.

5.3.4  Energy band pass

Both the energy band-pass due to divergence of the incident beam and the rocking curve of the bent crystal are, in first approximation, box shaped. The resulting energy spectrum is therefore flat topped and its FWHM is given by the larger of the two terms, see Figure 5-4.

The convolution of the two components contributing to the energy width helps to achieve a flat spectrum as it averages spatially over intensity inhomogeneities of the incident beam as well as over thickness variations or other distortions of the crystal.
Figure 5-4: Convolution of a bent-Laue crystal rocking curve (to the left) with an energy band of 1 keV (vertical lines) as defined by Equation 5-3. Parameters: Si(111), $E=40$ keV, $p=42.13$ m, $q=-2.475$ m, $\chi=35.26^\circ$, $\rho=6.2$ m and $T=1$ mm. Note that the rocking curve width of 235 µrad exceeds that of the flat Laue crystal by a factor 35.

In the convolution shown in Figure 5-4 it was assumed that the spectrum delivered from a bending-magnet or wiggler would be perfectly flat and that the integrated reflectivity of the bent crystal would be energy independent. In view of the very small differences of the integrated intensities that have to be detected ($\Delta I/I = 0.1\%$), this approximation is not fully justified.

As stated in Chapter 2, the intensity distribution of the radiation fan emitted by a bending-magnet or wiggler is determined by the electron-beam energy, the photon energy, the magnetic field strength and the magnetic period of the wiggler as well as the angle of observation\[^{14,15}\]. Figure 5-5a depicts the theoretical flux delivered by the wiggler installed at the ESRF Materials Science beam-line\[^{16}\] through a pinhole of 0.1x0.2 mm\(^2\) as a function of the energy and the horizontal position (upper abscissa). Energy and position are related through Equation 5-3. The second curve in the plot shows the integrated reflectivity of the bent-Laue crystal as function of energy. The convolution of these two curves yields the intensity distribution of the beam as reflected by the Laue monochromator, which is shown in Figure 5-5b. The spectrum is not perfectly flat. However, between 39 and 40 keV the maximum intensity difference amounts to only 0.1\% ($\sigma_{\text{rms}}=0.031\%$). The remaining variation can be compensated for by means of an absorber of appropriate thickness profile. Figure 5-5b shows the thickness profile of an Al absorber with an average transmission of 85\%, which would yield a perfectly smooth spectrum.
Figure 5-5: a: Flux delivered by the ESRF ID11 wiggler through a pinhole of 0.1x0.2 mm$^2$ as a function of energy and horizontal position (A) as well as integrated reflectivity of the Laue crystal monochromator (B) (same parameters as in Fig. 5-4), b: Intensity distribution of the beam diffracted by the Laue crystal (C) together with the thickness profile of an Al absorber (D) which compensates for the variation of the former distribution.

5.4 Optics Set-up

Optical elements can be used to select, reflect, focus and collimate X-ray beams. They are often made from Si crystals for its high purity. In mechanical sense, their robustness makes them easy to process, i.e. to cut and polish. Three Si crystal wafers with a different cut and shape where used as monochromators in the bent-Laue experiments.
5.4.1 Triangular-shaped Si crystal wafer

Obtaining a broad-energy X-ray band-pass requires a perfectly bent monochromator crystal, which implies that the bending should be cylindrically. For this a triangular-shaped crystal, shown in Figure 5-6a, was chosen. A force transmitted onto the tip of the triangle causes a moment and results, when the crystal end is fixed, into a bending of the crystal. Since the moment is linearly distributed, the local height of the crystal compensates for the resistance of the local moment so that a cylindrical bending will be accomplished.

Two triangular-shaped monochromator crystals were developed. The first crystal was a Si(111) wafer with a thickness of 1 mm and an asymmetry angle of 35.3°, whereas the second crystal was a 1 mm thick Si(311) wafer with an asymmetry angle of 59.8°. For both crystals the base and height of the triangle was 40 mm and 90 mm, respectively. The bending mechanism consisted of a translation stage, which transmitted a force, via a screw, onto the crystal. The crystal and the crystal mount are depicted in Figures 5-6a and 5-6b, respectively.

5.4.2 Rectangular-shaped Si crystal wafer

Since a triangular crystal does not allow proper cooling by an InGa bath, a rectangular-shaped monochromator was also developed which implies a more complex bending. Since the width of a rectangular crystal is always the same, a non-cylindrical bending would be obtained when a force is applied to one side of the crystal. A solution to this is the application of a torque. This can be obtained by applying a force to a Si-rod (Fig. 5-7), which is connected mechanically to the monochromator. In contrast to the triangular type, this junction does not move and the monochromator will be bent cylindrically.
The rectangular-shaped monochromator crystal used was an optically polished Si(311) crystal wafer with dimensions of 1x40x90 mm\(^3\) and an asymmetry angle of 25.24°. The bending mechanism consisted of one translation stage, which transmits a force onto a Si-rod via a piece of Ag-foil. The crystal and the crystal mount are depicted in Figures 5-7a and 5-7b, respectively.

![Figure 5-7: a: The rectangular Si(311) crystal wafer with holder for the bender, b: Crystal mount and bending system.](image)

5.4.3 Monochromator bender set-up

The monochromator set-up is shown in Figure 5-8. A monochromator crystal is fixed at one side to the bender set-up. The other side of the crystal is mechanically connected to a motor-controlled translation stage. In the case of the triangular-shaped crystal the connection is a screw whereas for the rectangular crystal the connection consists of a rod-type Si crystal and a strip of silver foil. Both connection systems are used to transmit the bending force and torque, respectively. A reservoir for cooling by means of an InGa bath can be used. Two channels through the aluminium bottom piece of the bender set-up can be used for additional cooling by water.

5.5 Samples

Several different samples were used to test the broad-energy X-ray band-pass technique. Some of them were used as analyser crystals, whereas others were used for electric field experiments. The sample preparation for electric field experiments is identical to that discussed in §3.3. Crystals of Si(100), Si(110), AgGaS\(_2\) and LiNbO\(_3\) were used as analyser crystals and the latter two samples were also used for electric field experiments.
Figure 5-8: The monochromator bending set-up; a: For the triangular-shaped crystal and b: For the rectangular-shaped crystal.
5.6 Electric Field and Gating System

A lock-in amplifier (Stanford Research Systems, model SR850) gives the signal for the high-voltage switches and receives the voltage output of the detector. The liquid-nitrogen-cooled high-purity germanium diode (Chapter 4) was used as a detector. A two-step modulation (Chapter 3) of the electric field was applied with a frequency of 33 Hz. The generation of the electric field and gating system is shown in Figure 5-9.

![Figure 5-9: The set-up used for applying an electric field and for synchronous measurement of the changes in the diffracted signal.](image)

It should be noted that the response of the Ge-detector depends on the energy of the X-rays. Therefore, all rocking curves presented in this work were corrected for the energy dependence of the detector.

5.7 Experimental Stations

The fact that a white beam is needed to create a broad-energy X-ray band beam limits the number of beam-lines where experimental work can be carried out. This is due to the scientific research purpose of most beam-lines at the ESRF where a white beam in the experimental hutch is not needed and thus not allowed by the ESRF safety regulations. However, a few beam-lines such as the Materials Science beam-line, the High-Energy X-ray Scattering beam-line and the Optics beam-line are allowed to have a white beam. A discussion of the implementation of the broad-energy X-ray band technique in these beam-lines will be given in the following sections.

5.7.1 Materials Science beam-line

The properties of the wiggler source of the Materials Science beam-line, which are important for the generation of the broad-energy X-ray band beam, are given in Table 5-2.
The beam-line was used in the white beam mode, which means that only the beam-line built-in absorbers are in the beam. Furthermore, two pairs of slits were used to set the size of the beam onto the Laue-crystal. A horizontal beam of 7 mm was selected by the slits. A special Al absorber, similar to the one as is described in §5.3.4, was inserted just before the monochromator to compensate for the wiggler spectrum in the horizontal plane. For the monochromator, the triangular-shaped Si(111) Laue crystal was placed in the experimental hutch, 42.88 m from the wiggler source. The monochromator was mounted on an $XYZ\theta$ stage for positioning. No $\chi$ or $\psi$ adjustment was available. The crystal was cylindrically bent by application of a force on the tip of the triangle so that the focal spot was at a vertically scattering four-circle diffractometer in Kappa geometry\(^{[17]}\). The $\theta$ angle of the monochromator was calibrated using the absorption edge of Gd at 50.239 keV and the energy was set to 50 keV for the actual experiment. The Ge-detector (Chapter 4), a pair of slits and a photo diode were placed on the $2\theta$-arm of the diffractometer. A Pb beam-stop of 2 mm thickness was placed between the detector and the diffractometer into the direct beam of the broad-energy X-ray band-pass. The bent-Laue set-up was fully shielded by a lead castle to lower the background radiation. A beam-stop consisting of large pieces of Pb and Cu was placed directly after the Laue crystal into the direct beam of the wiggler. A schematic overview of the experimental set-up is shown in Figure 5-10.

**Results and discussion**

A rocking curve scan of the (12,0,0) reflection of the Si(100) analyser crystal is shown in Figure 5-11 revealing the profile of the beam. The FWHM is 0.6°, giving a resolution $\Delta\omega/\omega=\Delta\lambda/\lambda=3.9\%$, corresponding to a band-width of 2 keV. This is in good agreement with the result that can be obtained from Equations 5-3 and 5-4 for a horizontal beam of 7 mm. The dip in the middle of the flat part of the profile is due to a glitch caused by the monochromator crystal. This glitch could have been removed by adding an $\psi$ adjustment table to the monochromator set-up.
The resulting fine structure at the top of the curve is not related to counting statistical fluctuations, which are much smaller, but due to structure in the incoming beam, induced by phase contrast of the Be windows and C absorber in the front-end\textsuperscript{[18]}. An attempt to eliminate this phase contrast by insertion of a random phase shifter, here a spinning wooden wheel just in front of the monochromator\textsuperscript{[18]}, showed some influence but no real improvement was achieved. Possible solutions to reduce the fine structure of the incoming beam are:

1. Polishing all Be windows and replacing the front-end C absorber,
2. Insertion of a random phase shifter as far upstream as possible, close to the first Be window and front-end absorber and
3. Using a different reflection and asymmetry angle in order to achieve a larger averaging over the energy band of the incoming beam as given by Equations 5-3 and 5-4, with the penalty of a larger focal spot\textsuperscript{[12]}.

Unfortunately, the first two solutions could not be applied since the beam-line components (vacuum tubes, mirror vessels etc.) and front-end are not accessible for any changes. However, the third solution was applied at the Optics beam-line (§5.7.2).

The general slope of the curve can easily be controlled by a combination of the wiggler gap, vertical slit size and absorber material or thickness, see Figure 5-5b.

Subsequently, a piezoelectric AgGaS\textsubscript{2} crystal (Chapter 3) was mounted on the diffractometer and a rocking curve scan of the (6,6,12) reflection was measured (Fig. 5-12a). The dip at the left side of the peak is again due to the glitch of the monochromator (Fig. 5-11) and the structure at the top of the peak is due to the structure of the incoming white beam. This time a smaller horizontal beam was used. The influence of the structure in the incoming beam on the difference profile was measured for the (6,6,12) reflection for a field of $3.3 \times 10^6 \text{ Vm}^{-1}$. In Figure 5-12b, the difference

\begin{figure}
\centering
\includegraphics[width=\textwidth]{figure5-10}
\caption{Experimental set-up at ID11 for producing a broad-energy X-ray band beam. Note the energy distribution of the beam between monochromator and sample. Dark gray denotes high X-ray energies whereas light grey denotes low X-ray energies.}
\end{figure}
profiles for five consecutive scans are given. The influence on the glitch is clearly visible, as well as the other structure in the incoming beam, which gives rise to a high-frequency structure in the difference profile. The excellent reproducibility of all details shows that this structure is not related to counting statistics. The influence of the high-frequency fluctuations can be reduced or eliminated in the data treatment by smoothing or Fourier-filtering techniques. Figure 5-13 shows the central part of the difference curve at various applied fields. Again, fine structure can be observed.

Figure 5-11: Rocking curve of the (12,0,0) reflection of the Si(100) analyser crystal at the ID11 beam-line, using a triangular-shaped Si(111) Laue monochromator.

Figure 5-14 shows the difference curve of Figure 5-13 together with a plot of the measured phase signal (φ in Fig. 4-7b). The figure reveals clearly that the phase does not remain constant during a scan. The phase is maximal on the flanks of the rocking curve (Fig. 5-12a), and is about zero at the flat part of the rocking curve. A hypothesis that this effect stems from peak deformation can be deduced from the following. The experimental conditions, i.e. electric field, X-rays and detector, are the same for each setting of θ, which indicates that the crystal causes the effect. As the application of an electric field induces a change in integrated intensity and Bragg angle, both are assumed to be instantaneous as compared to the 30 Hz modulation frequency. The third effect caused by the application of an electric field is a change in the mosaicity that affects the peak shape. Two cases of θ settings needs to be considered. Firstly, when the mosaic spread of the reflection is completely within the Ewald shell, no distinction can be made between either a change in mosaicity or Δθ, since only changes in integrated intensities can be observed by the application of the electric field, see Figure 5-15a.
Figure 5-12: a: Rocking curve of the AgGaS$_2$ (6,6,12) reflection, b: Difference curves induced by an external electric field of $3.3\times10^6$ Vm$^{-1}$ for five repeated scans.
**Figure 5-13:** The central part of the difference curve in Figure 5-14b at different voltages.

**Figure 5-14:** Plot of difference curve (solid line) and corresponding phase signal with an external electric field of $3.3 \times 10^6$ Vm$^{-1}$.
Figure 5-15: Change in integrated intensity caused by a change in mosaicity. a: The change in integrated intensity can not be observed due to its centered position in the Ewald shell, b: Change in integrated in intensity caused by a change in mosaicity when falling out the Ewald shell.

However, in the second case where the mosaic spread is near the edge of the Ewald shell, an additional change in integrated intensity caused by peak deformation can occur, see Figure 5-15b. Let it be assumed that initially no peak deformation is present for the mosaic spread belonging to the positive state of the electric field. When the electric field changes to the negative field the mosaic spread will shift (by $\Delta \theta$) towards the Ewald shell edge. If no peak deformation occurs, the spread will still be in total reflection. However, when the mosaic spread is controlled by a peak deformation, a part of the mosaic spread may fall outside the Ewald shell. As a result, partial reflection occurs and the absolute integrated intensity decreases. Since the LIA makes use of the absolute intensity signal coming form the Ge-detector to detect $\Delta I$ and $\phi$, a change in $\phi$ would not be observed when the peak deformation was on the same time-scale as the other electric-field-induced effects. Yet, the fact that the $\phi$ signal is not constant indicates that the peak deformation occurs at a larger time-scale, for example because a change in mosaicity is induced by a slow build-up of space and surface charges.

A second piezoelectric crystal, LiNbO$_3$ (Chapter 3), was mounted onto the diffractometer and the rocking curve of the (0,0,30) reflection is shown in Figure 5-16a. In this case, the band-pass was further reduced by closing the slits in front of the monochromator (see Fig. 5-10) in order to eliminate the glitch at the left and the structure at the right, visible in Figure 5-11. A slightly larger
vertical beam size was used. The profile of Figure 5-16a shows a sloping top, giving a 2% intensity decrease over 0.15°, which is due to an incorrect correction for the response function of the detector during the experiment. The programming error responsible for this was only noticed after completion of the experiment. The influence of the remaining slope of the profile was calculated by simulating a difference profile from two shifted but undeformed peaks. The induced intensity effect due to the non flatness of the peak proved to be negligible for shifts up to 4000 times the experimentally observed shifts. Therefore, the slope, although undesirable, has no significant influence on the experimentally determined difference curves. The solid curve in Figure 5-16b gives the measured difference profile for the (0,0,30) reflection for a field of 1·10⁵ Vm⁻¹. The dashed curve in the same figure is a simulated difference profile obtained by taking the difference between the original peak of Figure 5-16a and a peak shifted by 1.2·10⁻⁶° and decreased in intensity by 1·10⁻⁶%. It is seen that the correspondence is generally very good. The largest differences occur at the sides of the peak, which is related to the fact that no peak deformation is taken into account in the simulation. Such a peak deformation would have no effect at the central part, where the full mosaic spread is within the Ewald shell, but only at the edges. Peak deformation can have an indirect effect on the intensity via modification of extinction effects. Because of the high correlation between the peak shift and peak deformation at the sides, no accurate value can be obtained for these. The change in integrated intensity, however, is given fully by the flat part in the middle.

The relative change in intensity can be determined by either averaging over all points on the flat part or by a least-squares fit of the difference curve to the unperturbed profile using a shift and a change in intensity as refineable parameters. However, in the least-squares procedure, the intensity effect will be influenced by the signal at the slopes and is thus correlated with both peak deformation and peak shifts. The more accurate value is thus given by the average. A third solution, even much faster, is to take a single reading at the flat part instead of scanning the whole profile. In order to test the accuracy of the various methods, the field across the crystal was varied between 5·10⁴ and 4.3·10⁶ Vm⁻¹, and the response curve of the (0,0,30) reflection measured. Figure 5-17 gives the intensity effect as a function of the applied field. The dash-dotted curve (stars) gives the intensity effect determined by the least-squares fit. The dotted curve (crosses) is the intensity effect obtained by averaging over the flat part. It is seen that the qualitative agreement between the least-squares and the result by averaging is excellent. The least-squares result is, however, systematically lower than the result by averaging. This is due to the correlation between the shift and the intensity effect at the edges. The dashed curve (squares) is obtained by performing a second voltage scan but now taking only a single reading at the centre of the flat part. The correspondence between this curve and the other two is good.

It can be seen that the reaction of the crystal to the applied field is linear up to 2.7·10⁶ Vm⁻¹, after which it saturates at ΔH/H = 0.045 (5)%%. This value is about half the value obtained by Fujimoto, who found 0.12 (6)% for an applied field of 5.15·10⁵ Vm⁻¹. However, the standard deviation in the last experiment is too large to make a direct comparison. The curve is reproducible over multiple voltage scans. The intensity effect for the smallest field of 5·10⁴ Vm⁻¹ is only 0.001% and still measurable.
Figure 5-16: a: Rocking curve of the LiNbO₃ (0,0,30) reflection, b: Difference curve induced by an external electric field of $1 \times 10^4$ Vm⁻¹. The solid line is the measured curve, the dashed line is the simulated curve.
Figure 5-17: The induced relative intensity change for the LiNbO$_3$ (0,0,30) reflection as a function of the applied electric-field. The dash-dotted-line (stars) corresponds to a least-squares fit of an intensity effect plus a shift, the dotted-line (crosses) corresponds to an averaging over the central part of the differences curve, the dashed curve (squares) corresponds to a single reading at the centre of the rocking curve.

The data points for a single reading at the centre are obtained in 10 s. To put this into perspective, if an intense synchrotron beam is used together with the conventional scanning method (Chapter 3), approximately 20 min per data point are needed. If a similar curve were to be produced on a rotating anode, the measuring time would be several years per data point.

5.7.2 Optics beam-line

In order to reduce the fine structure of the broad-energy X-ray band (§5.7.1), a different monochromator reflection and asymmetry angle was used and applied at the Optics beam-line. The experimental set-up was as follows.

The triangular Si(311) monochromator crystal mounted on $XY\theta\phi\psi$ stage 40.5 m from the source. An energy of 57.45 keV was selected after an initial calibration of the $\theta$ angle by means of the absorption edge of W at 68.5 keV. The triangular crystal was cylindrically bent by applying a force onto the tip, so that the focal spot was 2.86 m behind the monochromator. A two-circle diffractometer with a horizontal plane geometry was placed at the focal spot. It should be noted that the Optics beam-line is situated at a bending magnet, giving thus less flux than the wiggler of the Materials Science beam-line.
Results and discussion

A rocking curve scan of the (660) reflection of a Si(110) analyser crystal was performed and is shown in Figure 5-18. This figure shows a perfectly flat spectrum at the top, with the fine structure now being due to pure photon-counting statistics.

![Figure 5-18: Rocking curve of Si(660) analyser crystal at the BM5 beam-line, using a triangular-shaped Si(311) Laue monochromator.](image)

5.7.3 High-Energy X-ray Scattering beam-line

Another broad-energy X-ray band experiment was performed at the High-Energy X-ray Scattering beam-line. Here, the same experimental set-up, as discussed in §3.4.4 was used. The monochromator of the beam-line is a Laue crystal made of Si(001) with an asymmetry angle of 2.5°. A broad-energy X-ray band beam with a mean energy of 40 keV was selected using the (111) reflection and directed into the experimental hutch by fine-tuning of the Bragg angle. Furthermore, a specially shaped Al absorber with a thickness of 3 mm was placed between the Be-window and the first slits, both situated in the experiments hutch.

Results and Discussion

A two-step modulation of the electric field with three different frequencies, 1000, 490 and 33 Hz was applied to a KD$_2$PO$_4$ (DKDP) crystal (§3.3) with an electric field of $1.33\times10^6$ Vm$^{-1}$. Figure 5-19a shows the rocking curves of the (2-6-2) reflection for different modulation frequencies. The figure indicates that no significant changes occur when the frequency of the electric field modulation is changed. The glitch coming from the monochromator is reproduced in each of the rocking curves.
Figure 5-19: Rocking curves for the (2-6-2) reflection at different frequencies for KD$_2$PO$_4$ with an electric field of $1.33 \times 10^6$ Vm$^{-1}$. a: Rocking curves measured for 33, 490 and 1000 Hz, b: Corresponding difference curves and c: Phase curves.
Similar observations can be made from the measured difference curves at different modulation frequencies, see Figure 5-19b. However, different phase ($\varphi$) curves were obtained as can be seen in Figure 5-19c.

As explained earlier in this Chapter, the change in $\varphi$ is a result of peak deformation. This is confirmed by the application of different modulation frequencies. These results indicate that at the flanks of the curve, where the mosaic spread is partly inside and partly outside the Ewald shell, the total signal ($\Delta h$) needs a certain time ($\Delta t$) to stabilise. Figure 5-19c shows that this time is constant and independent of the frequency $v$ of the applied perturbation. The phase is defined as:

$$\Delta \varphi = \frac{\Delta t}{\tau}$$

where $\tau$ equals to $v^{-1}$. Since $\Delta \varphi$ for 1 kHz is twice as large as for 0.49 kHz it indicates that $\Delta t$ is constant.