Monochromator 611 Manual

(HUBER G670 Guinier Camera)

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1 General

This manual should help users to install the HUBER Guinier monochromator housing 611 and to adjust the monochromator crystal series 615xx and 616xx. This manual refers to users who have been trained by a HUBER specialist before.
Many of the critical illustrations of this manual are movies. Please refer to the electronic version of this manual therefore.

PLEASE BE AWARE OF THE RADIATION WHEN WORKING WITH OPEN X-RAY SHUTTER.

2 Introduction

The HUBER Guinier Monochromator crystals 615xxx and 616xx are asymmetrically grounded and bent (curved) crystals made of quartz (SiO2), silicon (Si) or germanium (Ge). The X-ray radiation starting from the fine line focus of the X-ray tube strikes onto the monochromator crystal as a divergent bundle. In the crystal, a narrow band of wavelengths is separated from the polychromatic broad band of radiation by diffraction according to Bragg's law: $n = 2d \sin\alpha$. The diffracted radiation leaves the crystal under the glancing angle as a convergent bundle according to Fig. 1. By installing the HUBER focussing Johansson monochromator on X-ray tubes of small focal spot width, X-ray diffractograms of highest resolution can be produced. The intense spectral line $K_{\alpha 1}$ is clearly separated and used. All other characteristic emission lines and the Bremsstrahlung are completely eliminated. This leads to a remarkable decrease of background scattering in the diffraction diagram. It sets a number of new tasks to the fine-structure examination and improves the resolution of classical procedures as: Crystal powder diffraction and structural refinement, phase analysis, texture analysis, small-angle scattering, examination of separation processes, structural phase transitions as well as disarrangements in metals and minerals.

3 HUBER Guinier Monochromator Crystals

Huber No.	Rad	$K\alpha1$ [A]	Crystal	h	k	l	A [mm]	B [mm]
615002	Cu	1,54060	Ge	1	1	1	120	220
615004	Cr	2,28962	Ge	1	1	1	120	220
615006	Fe	1,93597	Ge	1	1	1	120	220
615008	Co	1,78892	Ge	1	1	1	120	220
615010	Mo	0,70926	Ge	2	2	0	120	220
615012	Ag	0,55936	Ge	2	2	0	120	220
616002	Cu	1,54060	Ge	1	1	1	120	360
616004	Cr	2,28962	Ge	1	1	1	120	360
616006	Fe	1,93597	Ge	1	1	1	120	360
616008	Co	1,78892	Ge	1	1	1	120	360
616010	Mo	0,70926	Ge	2	2	0	120	360
616012	Ag	0,55936	Ge	2	2	0	120	360

$K_{\alpha1}$ wavelengths as per International Tables for Crystallography, Vol. C, 177ff (1995).

Crystals for different wavelength and focal distances are available on request.

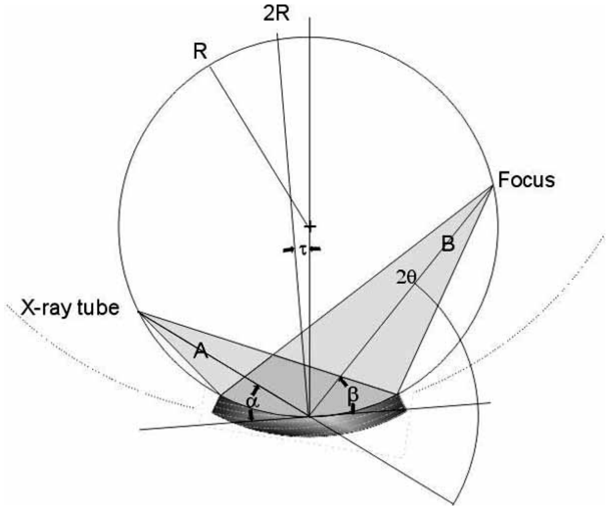

Fig. 1 Johansson Monochromator Geometry

The monochromator crystals are sawn, grounded and polished in such a way that the crystal lattice planes are inclined against the crystal surface at an angle τ. The crystal plates are then grounded with a cylinder of radius 2R and finally bent over fitting areas with radius R. This is the asymmetric Guinier-version of a focussing Johansson-monochromator (which has $\tau=0$). A consequence of this procedure is that, by an adequate selection of τ, the distances between the line focus of the X-ray tube and the centre of the crystal as well as between the centre of the crystal and the focal line can be kept constant for all common X-ray wavelengths.
It is easily possible to exchange the X-ray tube with one of a different wavelength. Only the monochromator crystal has also to be changed. Major alterations of the diffractometer equipment are not necessary.

4 Monochromator Housing 611

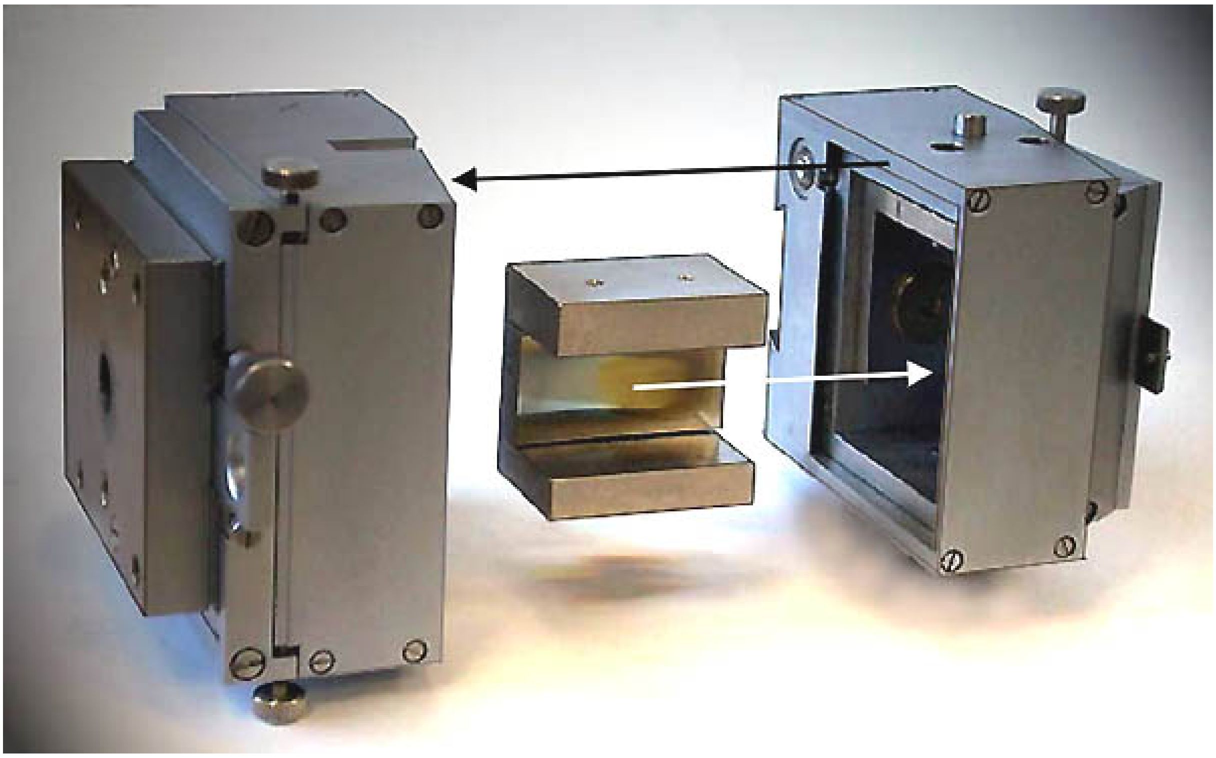

Fig. 2 Monochromator housing and monochromator crystal assembly

The monochromator housing 611 is designed in such a way that the crystal 615xxx/616xxx can be adjusted by several degrees of freedom with regard to the focal line spot of the X-ray tube. The crystal is completely hidden in the housing and only strictly monochromatic X-ray radiation diffracted by the crystal emerges, proper alignment assumed. The degrees of freedom are:

- Movement parallel to the X-ray tube axis.
- Movement vertical to the X-ray tube axis
- Rotation of the crystal around a central axis of it cylinder shape, i.e.
- Tilt around the X-ray beam axis.
- Tilt vertical to the beam axis.

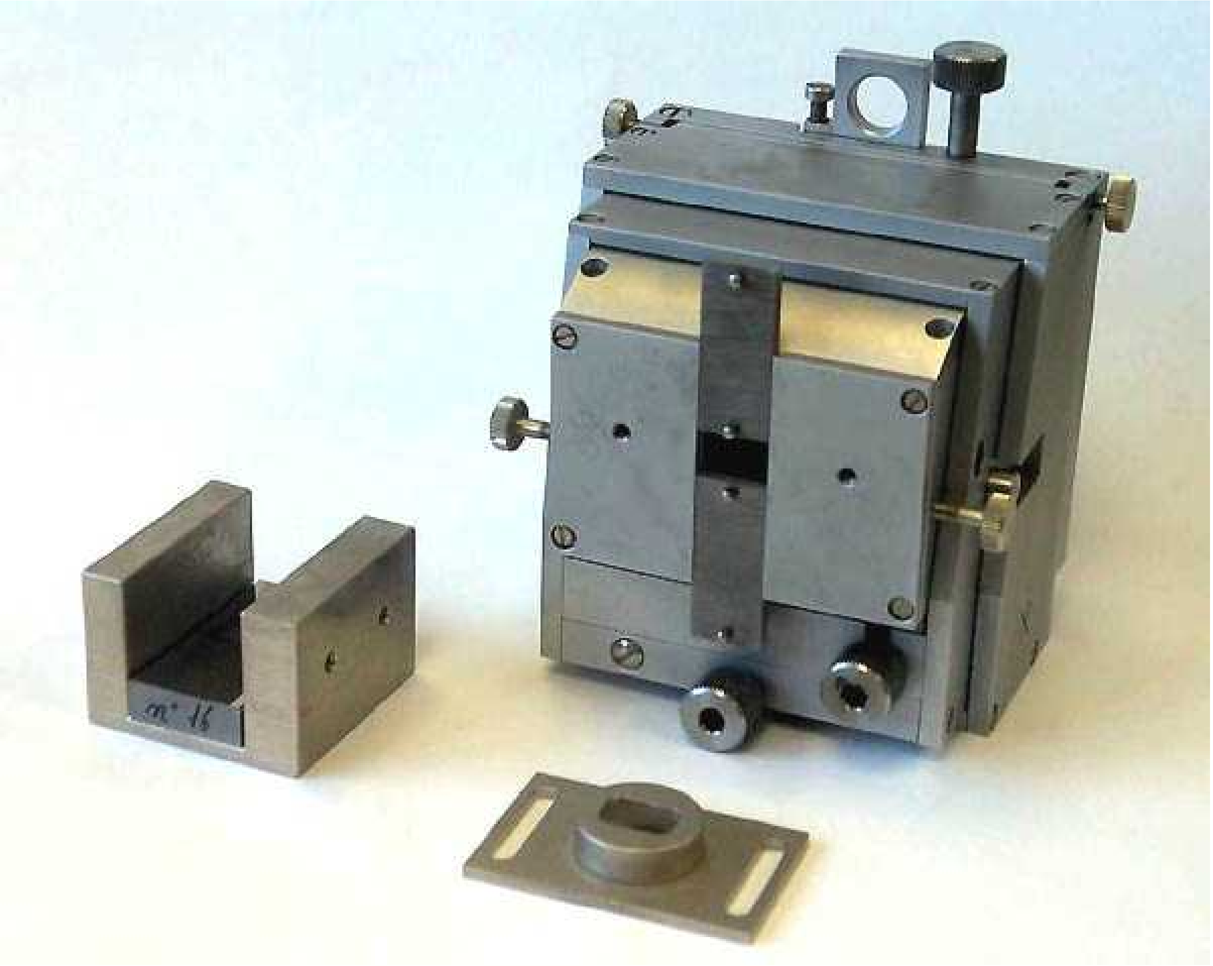

Fig. 3 Monochromator housing parts

5 Assembly and Adjustment of the Monochromator 611-15xxx and 16xxx

In the following paragraph we refer to a horizontal X-ray tube shield with the focus line vertical to the table top and to the monochromator, when seen in the longitudinal direction of the tube (from the HV cable connector) on the right side (Fig. 3).
For a monochromator on the other tube side, all instructions have to be mirrored, respectively.
For the mounting of the monochromator housing 611 onto the various brands of X-ray tube shields, adapter plates are available: 0.72 for Philips model PW 1316/90 or 91, 0.73 for Seifert model V4/V14, 0.74 for Enraf-Nonius FR561, 0.76 for Bruker AXS (Siemens) Type S, etc. For other options, please contact Huber.

1. Before assembling the monochromator housing 611, the adapter plate has to be mounted onto the exit window of the tube shield. **When the window is obscured by β -filter, remove it (refer to x-ray tube housing manual).**

2. Remove the fastening plate from the 611. Loosen the fine adjustment screw y of the monochromators longitudinal shift, pull out the fastening plate from the dovetail guide. Fix the fastening plate together with a spacer plate to the tube shield by means of the screws supplied along with the equipment, as shown in Fig. 4. Push the monochromator onto the fastening dovetail plate. Place it in a central position by moving the fine adjustment screw parallel to the tube axis. Place the tilt screws (Rx and Ry see Fig. 5) in a central position.

3. Open all diaphragms on the 611, put the fluorescent screen (with its vertical side) in front of the 611s exit opening and open the X-ray shutter. To avoid too much hazardous radiation exposure, turn the current and voltage setting of the X-ray generator to its minimum. By turning the Rz-screw, find out the parallel-to-main-beam position of the crystal surface, i.e. when the shadow of the crystal inverts its movement see Fig. 6.

4. Push the 2°-screen diaphragm in and adjust the centre of the beam area to the crystals surface (Fig. 7).

5. Now close the output shutter of the 611 which is at the back side of the curved crystal until it just stops the direct beam.

6. Set the X-ray generator to its common long term current and voltage, e.g. 30 mA and 40 kV, when using a 1500 W Cu fine focus tube.

7. The focal length A is fixed to 120 mm, per definition. However, this specification may vary individually by a few mm. Adjust the crystals focal distance A roughly to that value shown on the sticker on the 611 housing, using a ruler. The centre of the monochromator crystal is defined evidently by the short visible shaft of the Rz-rotation on the housing. The tubes focal spot is at the centre line of the X-ray tube shield 21 mm apart from the tubes head surface (when having a short version! 34 mm for a long one.). When the Monochromator crystal tilt is carefully increased by turning R_z clockwise, at first, a weaker diffraction reflex of the K_β line will come up. Further increase of the Bragg-glancing angle will result in a bright reflex of both, $K_{\alpha 1}$ and $K_{\alpha 2}$. Now, observe the appearance and disappearance of the reflex on the fluorescent screen by slowly turning the R_z –screw back and forth (Fig. 8).

8. When varying the glancing angle from smaller to larger values, the image of the Bragg reflex should "breathe" on fluorescent screen area similar to an expanding soap bubble. This is a good indication of having the correct A distance.

9. When the crystal is not in the specified A, the reflex looks more like Debye-Scherrer rings and migrates horizontally over the screen when varying R_z. Then A has to be corrected carefully, until that breathing feature is visible.

10. In case that the reflex does not migrate horizontally, but from top to bottom or even diagonally, then one of the tilts (R_x, R_y) has to be varied.

11. It should be take care during all phases of adjustment that, when rotating the monochromator crystal from a small to a bigger glancing angle, the reflex appears suddenly in the centre of the fluorescent screen and disappears at the same rate to the top as well as to the bottom. In this case, the distance A is correct and the crystal axis is parallel to the line focus of the X-ray tube. The monochromator is ready for operation, now.

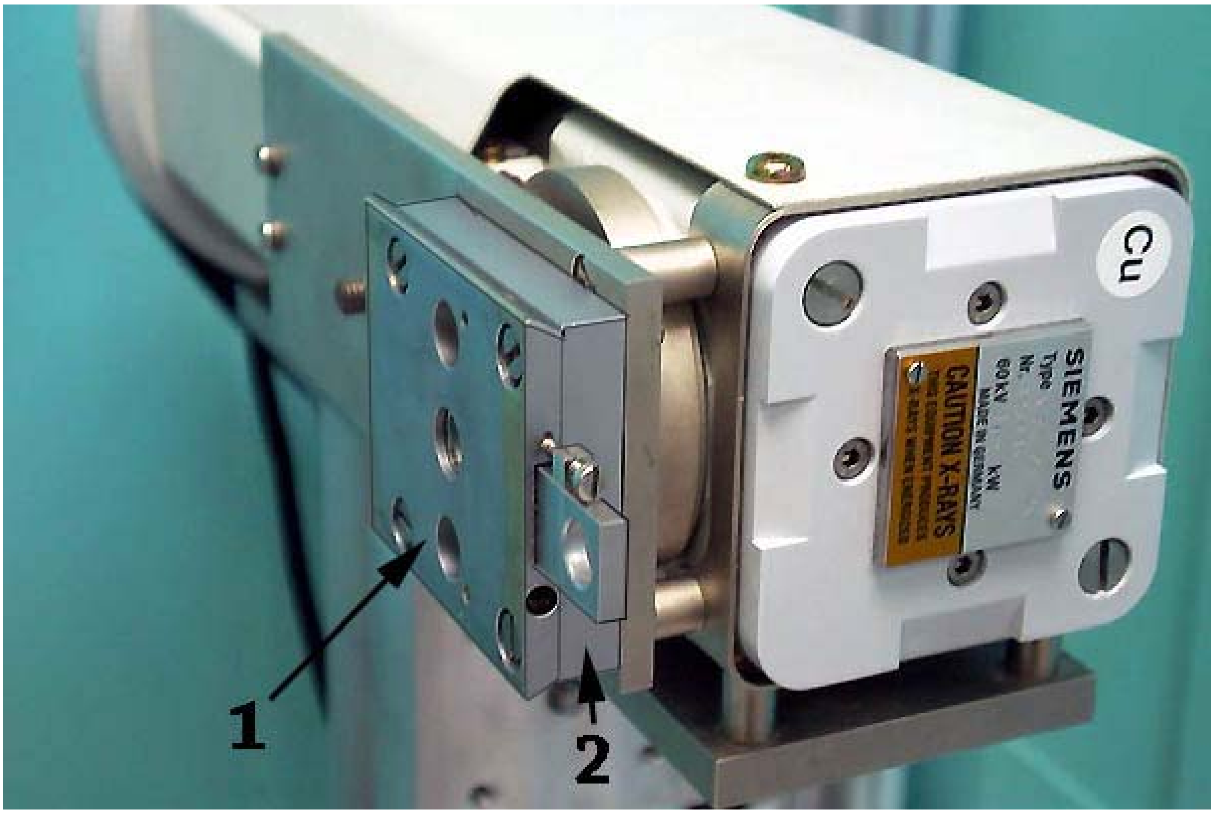

Fig. 4 Mounting plate for monochromator housing (1 fastening plate, 2 spacer plate)

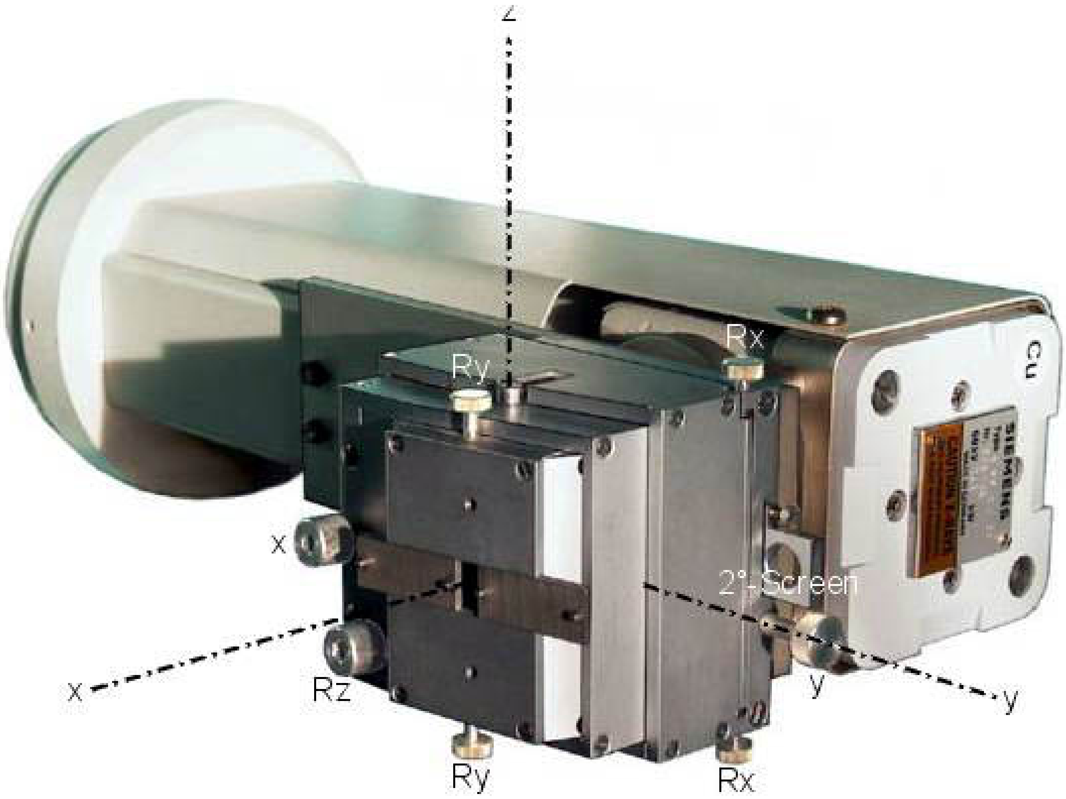

Fig. 5 Monochromator mounted on X-Ray Tube Housing; Degrees of freedom

Fig. 6 Crystal shadow (Movie)

Fig. 7 Crystal shadow 2 (Movie)

Fig. 8 Adjustment of theta (Movie)

6 Fine Tuning to K_{a1} and Alignment of the Base 601

In the previous chapter, the monochromator crystal has been installed and aligned to operational conditions against the focal target of the X-ray tube. Now fine tuning of the diffracted beam to $K_{\alpha 1}$ is necessary. Afterwards the exact position of the base 601 against focal line of the monochromator crystal at the distance B has to be found. This will again be done by the help of the adjustment bridge together with the three fluorescent screens, supplied with the 601. The adjustment base is mounted next to the horizontally arranged X-ray tube in such a way that the foot of the base with the adjusting pin is located beneath the rotation centre of pre-adjusted monochromator crystal (for the long B distance monochromators 616xxx this might not be possible). The projection of the X-ray beam diffracted by the monochromator should according to Fig. 9 approximately bisect the circular segment formed by the adjustment base 601.

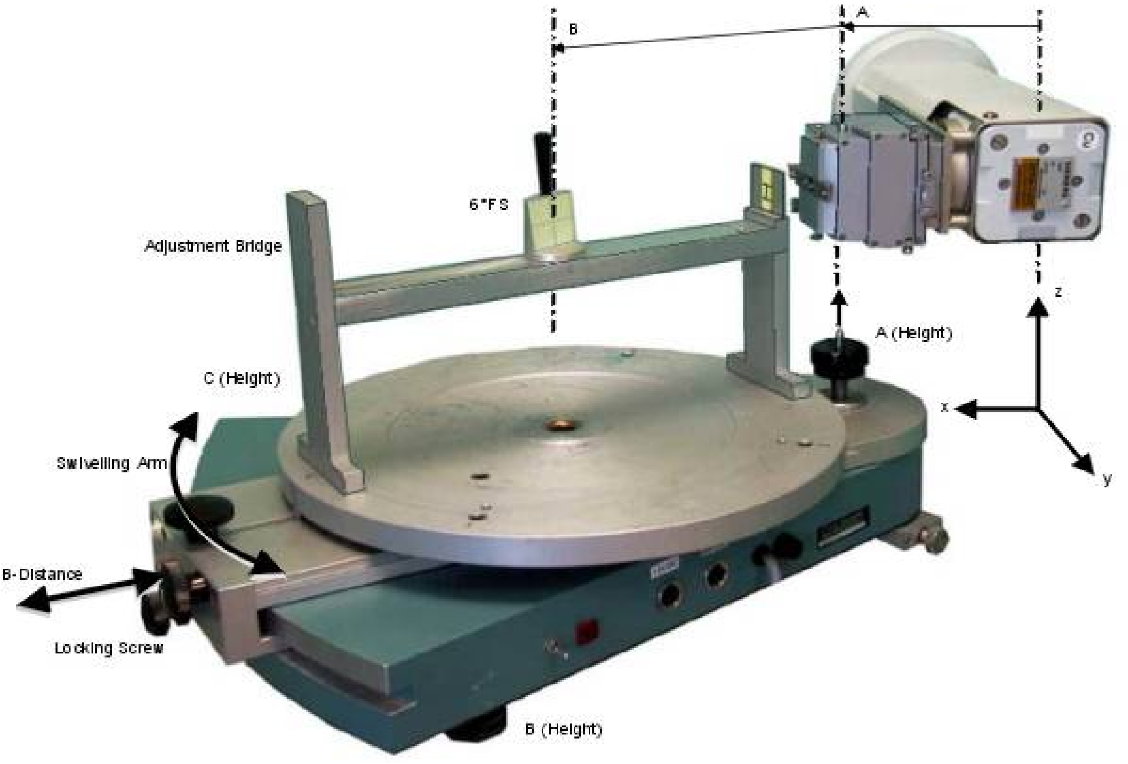

Fig. 9 Adjustment Base with adjustment bridge and the three fluorescent screens

1. The adjustment bridge has to be mounted on the circular base plate of the 601. By moving the swivelling arm of the 601, it is aligned into the X-ray beam diffracted by the monochromator with simultaneous observation of the front- bridge fluorescent screen (Fig. 10). The height of the base and its parallel position to the X-ray beam is then regulated by adjusting the three screw feet (Height A, B, C in Fig. 9).

2. The distance B between the centre of the monochromator crystal and the base plate centre have to be aligned. There is a scale on the swivelling arm, where the distance is to be set to the specified value of 220* mm, when using a 615xxx crystal. When using the 616xx crystal, the distance B=360* mm has to be set by using a ruler. Both adjustments are correct if the X-ray beam appears in a central position, equally wide and parallel to the vertical cross wire lines on the front and rear fluorescent screen, see Fig. 9. (* values may vary for some mm)

3. Fine adjustment is effected in two stages with the 6°-fluorescent screen (6°FS) in the centre position:

    a. 6°FS **perpendicular** to the incident beam: Adjustment of the parallel position of the focal line and the base plate normal.
    b. 6°FS **oblique** to the incident beam (gracing incidence): Exact alignment of the focal distance B, see Fig. 13.

4. When turning the Bragg angle from lower to higher values in the movie Fig. 12, in very small steps, one can see clearly the well separated positions for $K_{\alpha1}$ and $K_{\alpha2}$, see also Fig. 13.
    The focal line with the higher intensity is the correct one. So, one has to adjust the glancing angle of the crystal in such a way that the focal beam waist of $K_{\alpha1}$ appears at the brightest intensity while $K_{\alpha2}$ remains invisible.
    Note: The monochromators for Mo and Ag radiation diffract at relatively low glancing angles of some 5° to 6°. Therefore, $K_{\alpha2}$ cannot be suppressed completely, but a remainder of some 16% of the $K_{\alpha1}$ peak intensity is still there.

Fig. 10 Adjustment of Theta. Observation with front fluorescent screen (Movie)

Fig. 11 Adjustment of Theta. Observation with rear fluorescent screen (Movie)

Fig. 12 Fine Tuning of the adjustment (Movie)

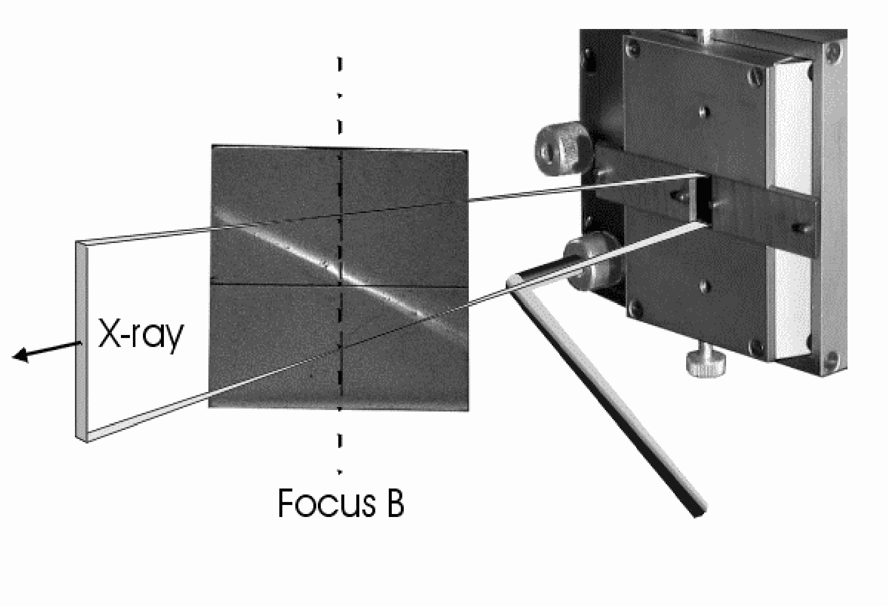

Fig. 13 Fine Tuning (Movie)

7 Exchange of the Monochromator Crystal 615/616

When the wavelength changes, it is necessary to exchange the crystal insert. The crystal inserts available at present are listed in Chapter 3. With decreasing intensity and a loss of resolution monochromators can be used conditionally for wave bands which are adjacent to the wave-length for which the crystal and the crystal inserted were designed, e.g. CoKα instead of CuKα and CrKα instead of FeKα. Crystal inserts of new series can be built into the monochromator holder without any preparatory work. Do not touch the polished surface of the crystal!

Building-in of the crystal insert (see also Fig. 2). Move fine adjustment of the horizontal shift (vertical to the tube) until the inner part of the holder can be slipped off its dovetail guide in the direction of the beam. Set the bragg angle adjustment to a medium position until the two fastening screws are clearly visible. After the countersunk screws have been loosened, the crystal insert can be taken out of the monochromator holder. The crystal insert is built in and the assembly of the monochromator is affected in the reverse order: The crystal insert is put into the inner part of the monochromator holder. The crystal insert is fastened by means of the two lateral fastening screws. The inner part of the holder is pushed into the outer part. The crystal is readjusted as described in Chap. 5.

8 Geometry of the Guinier System 600

The Guinier powder camera 621, the step scanning diffractometers 642, 644, 645, 653, and the imaging plate camera 670 constitute, in conjunction with the precision monochromator 611/615/616, an X-ray diffraction system of high resolution and wide applicability to produce X-ray powder diffractograms. A polychromatic radiation diverging from the fine line focus of an X-ray tube hits the monochromator crystal and is diffracted as a convergent beam containing the wavelength $K_{\alpha 1}$, only. The diffraction arrangements of all devices following the monochromator are based on the focussing principle of Seemann-Bohlin. An essential part of these diffraction arrangements is a cylinder on the inner diameter of which an X-ray film is mounted in the case of the Guinier chamber or on the diameter of which the detector slit is moved parallel to the axis in the case of the Guinier counter tube attachment 642 up. Focussing on this cylinder occurs when the focal line of the monochromator of the cylinder axis is parallel to the cylinder diameter and the plane powder specimen is tangent to the focussing cylinder, Fig. 14.

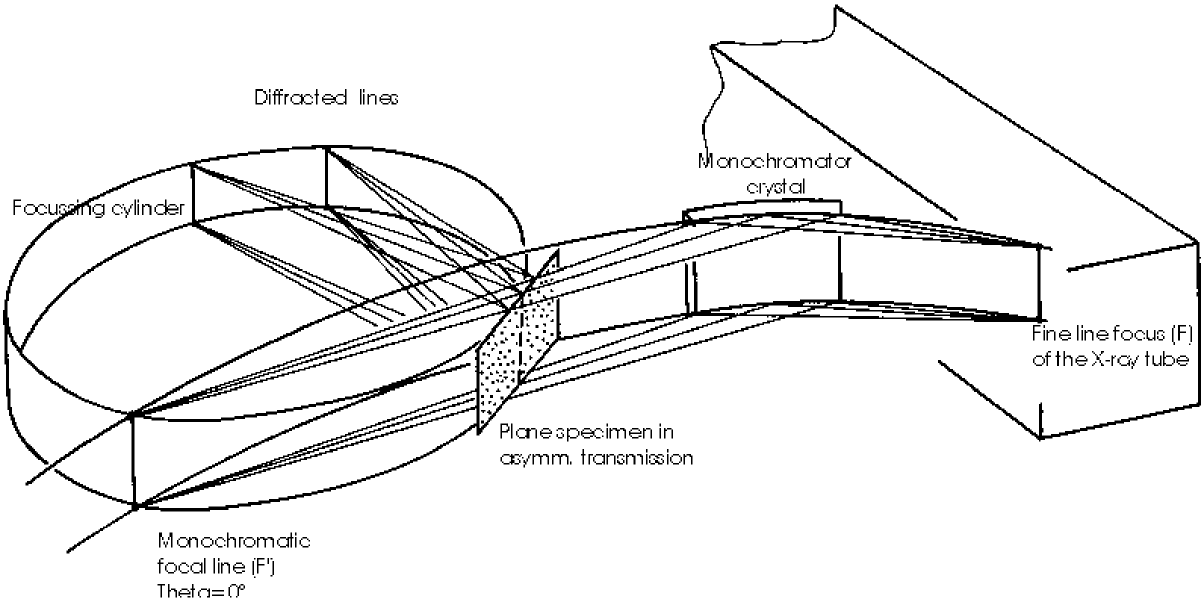

Fig. 14 Guinier geometry

The focussing cylinder is pivotally attached round the focal line; varying with the position and the glancing angle of the primary beam with regard to the specimen, the diffraction lines from various angular ranges are projected according to Fig. 15.

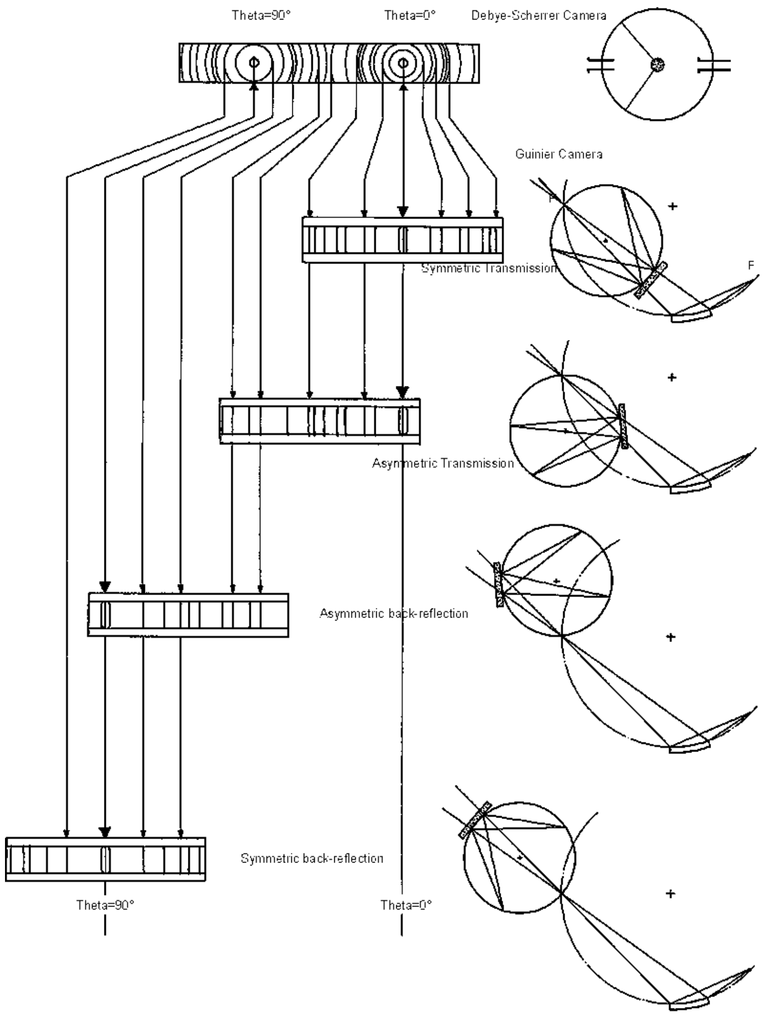

Fig. 15 Diffraction geometries

In contrast to the non-focussing Debye-Scherrer method, the diffraction diagram of (theoretical) 0° <2Theta < 180° can no longer be covered by one geometrical arrangement. However, regarding this drawback, the radiation is strictly monochromatic (except Mo and Ag radiation) and, with the same camera radius, there are considerable gains in resolution due to focussing with a shortened time of exposure. In order to obtain maximum resolution and minimum exposure time, it is recommended to operate the Guinier system with fine line focus X-ray tubes. When using other tubes having a wider focal spot, the complete separation of $K_{\alpha 1}$and $K_{\alpha 2}$ cannot longer be insured.

9 Resolution

The resolution of a Guinier system is determined by the focal width of the X-ray tube, by the quality of the monochromator crystal and the perfection of its workmanship, furthermore by the diameter of the focussing cylinder of the camera, which is limited by the focal length of the monochromator. The focal length of the precision monochromator 611/615/616 is around 220 and around 360 mm varying a few mm, with the monochromator type and the wavelength used. The diameter of the focussing cylinder is 114.6 mm for all instruments of the series 600, except the model 670 (180 mm). This gives an angular dispersion 4 mm or 6.28 mm per 1° of Theta, respectively. For the film camera 621, when using commercially available double sided X-ray films, only the front side of the film should be developed (development of one layer). In addition, the thickness of the specimen, the size of the particles in the specimen as well as the interaction of chromatic dispersion of the monochromator and the specimen have an effect on the width of the diffraction lines and, as a consequence, on the resolution of the camera. These influences are to be evaluated and discussed below with regard to their effect on the resolution of the camera. Provided that the treatment of the crystal, mounting and adjustment are correct, the monochromator makes essentially three contributions to the line width.

10 Structural Defects of the Monochromator Crystal

The orientation of a crystal lattice plane varies as a consequence of growth defects - especially in natural crystals. This range of variation is $\Delta\tau=\pm$ 20" for the SiO2 crystal-lattice plane (101). This results in a line width contribution where

(1) $L\tau=\Delta\tau/\tau\ B/\cos(\psi)$

with B=focal distance crystal centre - focal line, ψ=angle of incidence of the beam on the powder specimen, perpendicular incidence ψ =0°, oblique incidence ψ=45°.

The line width contribution $L\tau$ depends on the wavelength and the glancing angle. For $\Delta\tau=\pm$ 20" and B=210 mm for quartz (101) and ψ=45° (asymmetric position) 0.0575 mm for ψ=0° (symmetric position) 0.0407 mm, measured on the focussing cylinder of the camera.

11 Penetration Depth Effects

As a consequence absorption and extinction, X-ray radiation penetrates into the monochromator crystal only to a determined depth, it is thus diffracted from a volume of finite thickness. For a first evaluation of the resulting effect in the thickness of the layer or in the "particle size", the thickness of the half-value absorption layer t may be taken for the penetration depth, considering the oblique incidence of the X-ray beam (glancing angle θ_k) and the oblique grinding of the monochromator crystal (cutting angle τ).

Extinction may be neglected in a first approximation.

The line width contribution in the X-ray beam diffracted by the monochromator crystal follows as:

(2) $L_s=t\cos(\theta_k+\tau)$,

with t=half-value absorption layer, θ_k=glancing angle at the monochromator and τ=grinding angle (in monochromators according to the Johansson-Guinier principle).

For $CuK\alpha_l$=1.54 Å, (t=0.0067 mm, θ quartz (101)=13° 20', τ=6° 04') one can calculate

L_s=0.006 mm.

According to P. Scherrer, the following holds for the angular widening b of an X-ray interference by the linear particle size:

(3) $\beta = K \lambda / (G \cos \theta)$

With G=particle size, K=shape factor (~1).

In the monochromator crystal, the thickness of the half-value absorption layer t takes the place of the particle size G. For the contribution of the particle size of the monochromator measured on the focussing cylinder of the camera, we receive:

(4) $L_{GM} = (B \lambda \cos \tau) / (\tau \cos\theta_k \cos \psi)$

When using CuKα_l: (B=210 mm, τ=6°04', q=13°20' for quartz (101))

then L_{GM}=0.0047 (mm) for ψ=45° and L_{GM}=0.0033(mm) for ψ=0°.

This line width contribution is independent of the size of the chamber and the glancing angle θ of the radiation diffracted at the specimen.

12 Chromatic Line Width

The characteristic radiation of the X-ray tube has a finite resolution. $\Delta \lambda / \lambda$ is approximately 0.0006 Å for CuKα_l. The following holds for the contribution of this finite line width for the widening of the lines:

(5) $L_\lambda = B \, \mathrm{tg}\theta_k \, \Delta \lambda / \lambda \; 1/\cos\psi$ (mm),

which results for CuKα_l (B, θ_k see above) and ψ=0° to 0.02 mm, for ψ=45° to 0.0283 mm.

The quality, i.e. resolution and intensity of an X-ray monochromator, is influenced decisively by crystal treatment (cutting of the angle τ, grinding, polishing and bending). According to data given by E.G. Hofmann and H. Jagodzinski as well, as according to the data given by the manufacturer of the monochromator crystals, the working tolerances of quartz are to be within the defects of the crystal structure of $\Delta\tau = \pm 20''$.

13 Contribution of the Monochromator to the Line Width

The contribution of the monochromator to the line width of a Guinier system can be calculated by folding:

(6) $L_M = L_\tau * L_{GM} * L_\lambda$,

or as a good approximation by a simple addition:

For ψ=0°: $L_\tau + L_{GM} + L_\lambda = 0.0407 + 0.0033 + 0.02 = 0.064$ (mm)

For ψ=45°: $L_\tau + L_{GM} + L_\lambda = 0.0575 + 0.0047 + 0{,}0283 = 0.090$ (mm)

The contributions to the line width given in mm apply to diffraction angles. With a camera diameter of 114.6 mm, a distance of 4 mm on the film corresponds to a diffraction angle θ of 1°, giving the monochromator contributions to 0.016° and 0.023°, respectively. The numbers reduce by some 30%, when using the model 670, due to the larger focal circle, correspondingly.

14 Influence of the Specimen Thickness

The contribution L_D to the diffracted line width due to the thickness D of a two-dimensional powder specimen located in the focussing cylinder also depends, as shown in Fig. 16, on the angle of incidence of the X-ray beam to the specimen ψ and on the glancing angle θ.

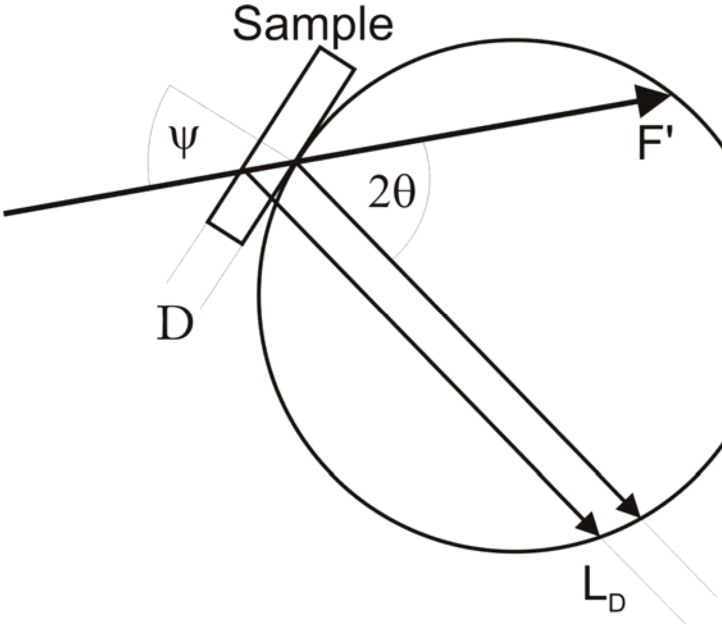

Fig. 16 Influence of the sample thickness to the line width

Without taking into consideration any further properties of the specimen, the following holds for the transmission case (focal line behind the specimen):

(7a) $LD=2\ D/(\sin2y + (\cos2y + 1)\ \mathrm{ctg}2q)$

for the back-reflection case (focal line in front of the specimen):

(7b) $LD=2\ D/(\sin2y - (\cos2y + 1)\ \mathrm{ctg}2q)$

A numerical evaluation of this relation shows that optimal values for LD are obtained with the angle of incidence y=30° and small glancing angles q. Contrary to this, there are essentially more unfavourable conditions for y=30° and q > 45°. For y=45°, maximum divergence is only approx. 15%. An evaluation shows that the use of fixed angles of incidence of y=0° (perpendicular incidence, symmetric position of the camera) and 45° (oblique incidence, asymmetric position of the camera) is generally sufficient, except special cases, such as the examination of thin layers in back-reflection.

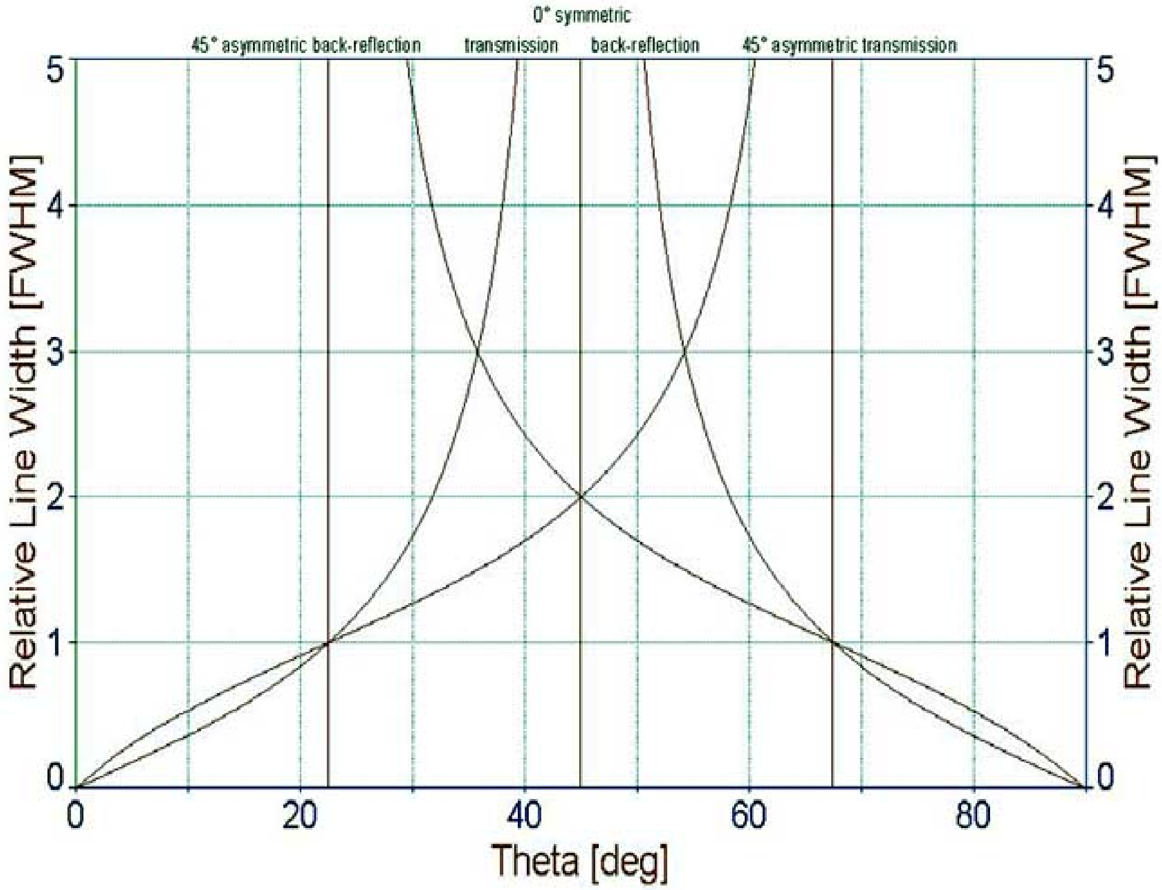

Fig. 17 FWHM

In Fig. 17, the relative line width contribution D/L_D was plotted for $\psi=0°$and for $\psi=45°$ as a function of the glancing angle θ. It can be seen from Fig. 1102 that especially for symmetric Guinier photographs ($\psi=0°$) and for θ between 30° and 60° , the specimen thickness contribution to the interference width gets high values. This can be counteracted conditionally by reducing the thickness of the specimen, however it is more expedient to photograph this angular range in an asymmetric position of the camera ($\psi=45°$).

The line width contribution in FWHM (θ) is shown for the special case of the G670 in Fig. 18.

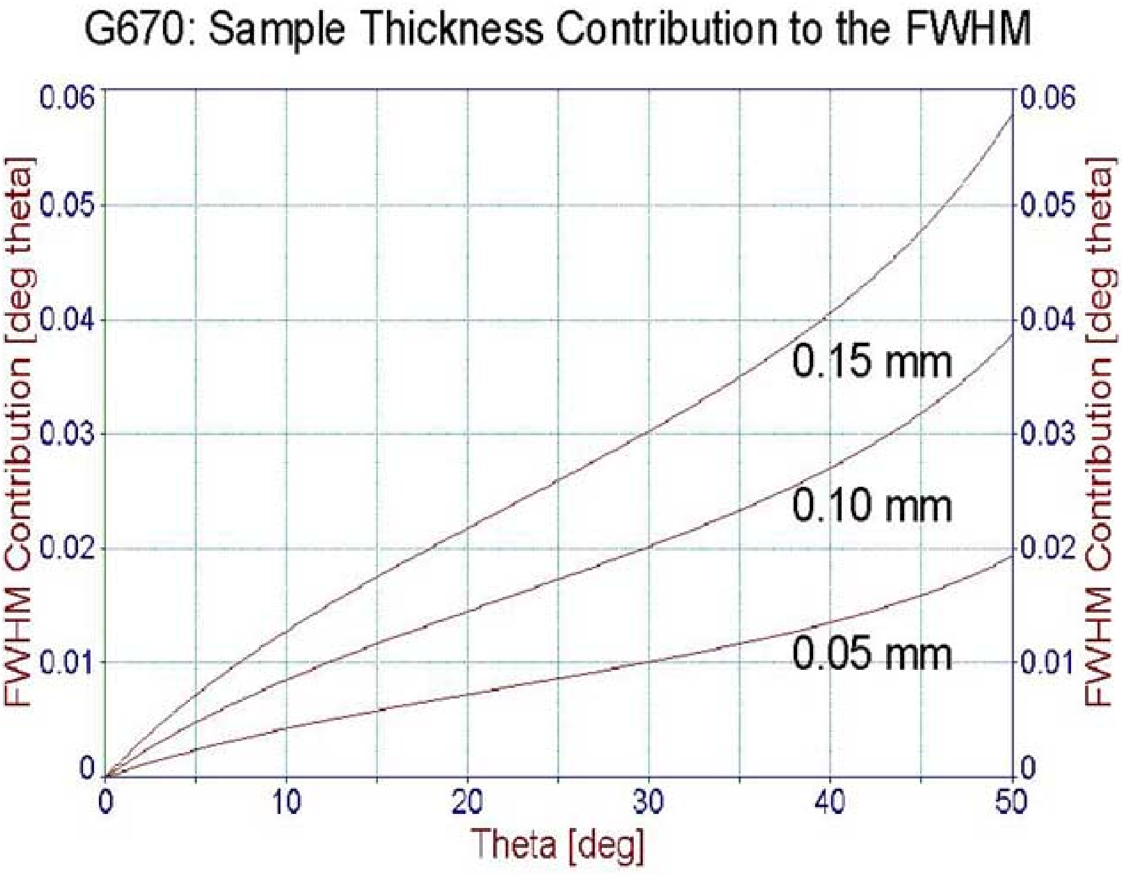

Fig. 18 FHWM depending on sample thickness

With ψ=45°, absorption correction remains below 10%, it may, therefore, be neglected for a first approximation. The optimal specimen thicknesses are approx. 0.1 mm for inorganic specimens, metal specimens should be thinner, organic substances thicker in order to obtain favourable exposure times.

By a suitable selection of the photographing positions, it is possible to obtain interference lines with a minimal widening effect in each angular range. As has already been mentioned, the said relations strictly apply to cylindrical specimens.

For plane specimens lying tangentially to the focussing cylinder, a slight correction of the thickness of the specimen (D + ΔD) becomes necessary. The following holds:

(8) $\Delta D = 2r \sin^2(\varepsilon/2)$

For the camera diameter 2r=114.6 mm and an angle of aperture of the monochromator at the side of the specimen of ε=2°, ΔD=0.035 mm. By reducing the angle of aperture, ΔD can further be reduced, however at the expense of intensity.

15 Influence of the Specimen Particle Size

Equations (7a) and (7b) do not depend on the radius r of the camera, the increase of which must logically, lead to higher resolution. There are, however, limits to this resolution due to the effect of particle size of the powder specimen. The following holds for the full width of half maximum FWHM of an X-ray diffraction line:

(9) $L_G=K\ \lambda\ 2r/(G\cos\theta)$ (mm)

Even film blackening requires a grain size of $G < 5\cdot10^{-4}$ mm for an unmoved specimen.

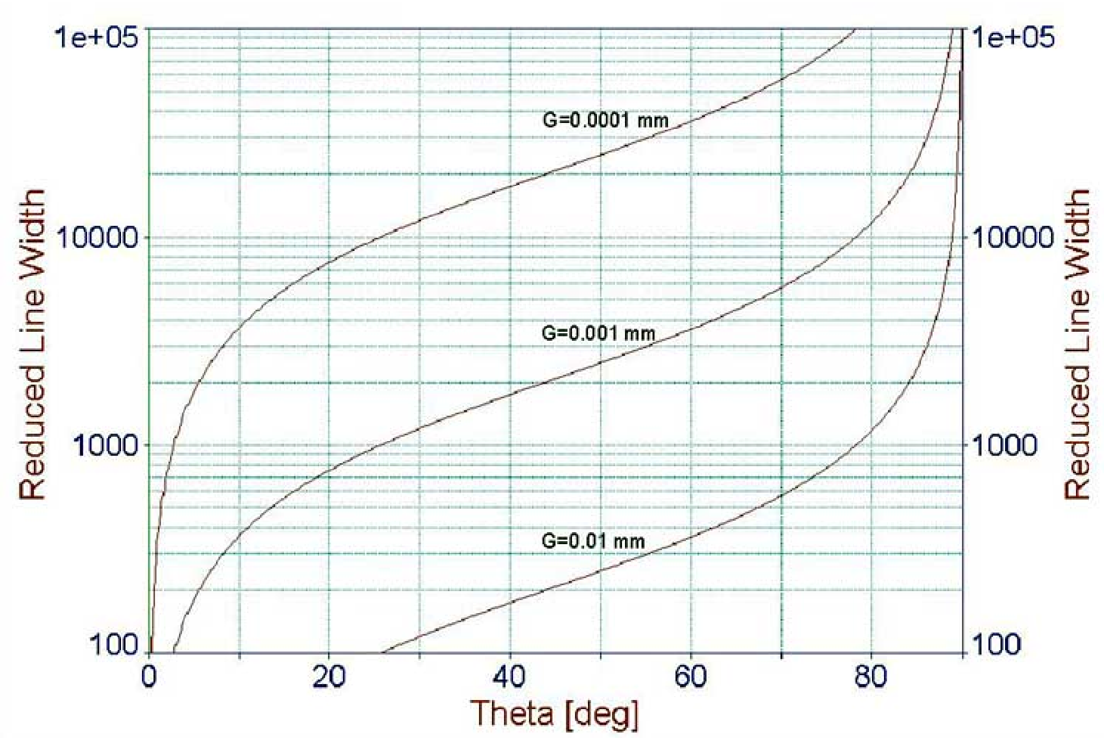

Fig. 19 Influence of the specimen particle size

With a camera radius of 2r=114.6 mm, L_G just reaches the extent of the specimen thickness effect in the case of small diffraction angles. The camera has maximum resolution in this case. An increase in the camera diameter does not give any advantages because of the particle size effect prevailing. Fig. 19 shows, for CuKα-radiation and 2r=114.6 mm, the line width contribution L_G for different particle sizes G as a function of the glancing angle θ, in addition the reduced line width

(10) $L_G\ G/(K\ \lambda)=2r/\cos\theta$

In order to keep the particle size contribution low even for larger glancing angles, the particle size of the powder specimen must be increased to approx. 10^{-2} mm. In order to receive evenly blackened X-ray interferences with this essentially increased particle size, the specimens must be moved tangential to the focussing cylinder.

16 Influence of the Chromatic Dispersion

From the focal spot of an X-ray tube with a width of the line focus of 1 mm (visually ~0.1 mm), a wavelength spectrum $\lambda+\Delta\lambda$ is diffracted by the monochromator crystal, which - when adjusting the monochromator to the wavelength $K\alpha_1$- also contains wavelength $K\alpha_2$ (chromatic dispersion). In analogy with the monochromator, chromatic dispersion also occurs in the specimen. According to Fig. 6a and 6b varying with the position of the crystal-lattice planes of the monochromator and the specimen with regard to each other, both effects may either be intensified (" addition position ") or weakened (" subtraction position ").

Fig. 7 shows the various possible positions of the monochromator, the specimen and the focussing cylinder of the camera as well as their combined effect with regard to chromatic dispersion. The following holds, where the first term in the brackets is due to the monochromator, the second due to the sample:

(11) $L_{\lambda}=r\ \Delta\lambda/\lambda\ (B/(r\cos\psi)\ tg\theta_{k} \pm 4\ tg\theta_{p})$

With (according to Fig. 7) + addition position, - subtraction position, r=radius of the camera, ψ=angle of incidence X-ray beam-specimen, θ_K=glancing angle at the monochromator, θ_p=glancing angle at the specimen, B=focal distance.

In order to avoid the troublesome double structure of the diffraction diagram - $K\alpha_1$ and $K\alpha_2$ appear next to one another for each interference - such structure must be compensated, in accordance with equation (11), by an adequate selection of ψ.

Complete compensation of the dispersion effect is in each case only possible for narrow ranges of θ_p by a variation of ψ. The angle ψ must, therefore, be freely selectable. In order to avoid the dispersion effect over the range $0<\theta<90°$, numerous photographs are required. When using a fine-focus tube with a narrow focus (<50µm), this is not necessary, as in this case only the spectral line $K\alpha_1$, is brought to "reflection" in the diffracted beam in a good monochromator crystal. The diffraction arrangement may - under this condition - be constructed with a fixed angle of incidence ψ=0°, ψ=+ 45°, ψ=-45°.

However, the chromatic influence on the interference width itself must always be taken into consideration. For the spectral line $CuK\alpha_1$ and a camera with a diameter 2r of 114.6 mm, this chromatic line contribution is represented in Fig. 8. This effect influences above all diffraction angles θ>45°. Because of the proportionality of the dispersion effect to the camera radius r, it is not expedient to increase the latter to more than 57.3 mm. An increase in resolution seems possible only when a wavelength smaller than the FWHM of the spectral line $K\alpha_1$ can be brought to reflection out of an X-ray tube with an extremely sharp focal spot with an ideal crystal monochromator.

17 Evaluation of the Camera Resolution under Experimental Conditions

Assuming the correct adjustment of the camera, the FWHM of an X-ray diffraction line is determined by

(12) $L=L_{\tau} * L_{GM} * L_{\lambda} * L_{D+\Delta}D * L_{G}$

L are delta-functions and the * means folding. The first three terms were discussed as the contributions of the monochromator properties in Chap. 11 and evaluated for $CuK\alpha_1$ and a quartz monochromator. This resulted in the following approximation:

(6) $L_M=L_\tau * L_{GM} * L_\lambda$

becoming 0,065 (mm) for $\psi=0°$ and 0,09 (mm) for $\psi=45°$.

For the estimation of the diffraction line width of the whole camera system, the particle size G of the specimen is 10 µm for $\theta=30°$, the angle of aperture of the monochromatic beam ε is 2° and $\psi=45°$ in subtraction geometry. So, the following formula

(13) $L=L_M * L_{D+\Delta D} * L_G$

results by way of approximation L=0.065 + 0.15 + 0.002 roughly into L=0.2 mm. which corresponds to an FWHM of approx. 0.05° (θ) in a Guinier camera with 2R=114.6 mm. When an amount slightly over double the interference width is defined as resolution of the camera system, two interferences with a θ distance of approx. 0.11 would still just appear to be separate. This is quite evident from Fig. 20 showing the "Three Fingers" diffraction triplet of α-Quartz around 34° θ. These are the well known "Five Fingers of Quartz", when using a standard Bragg-Brentano diffractometer without monochromator.

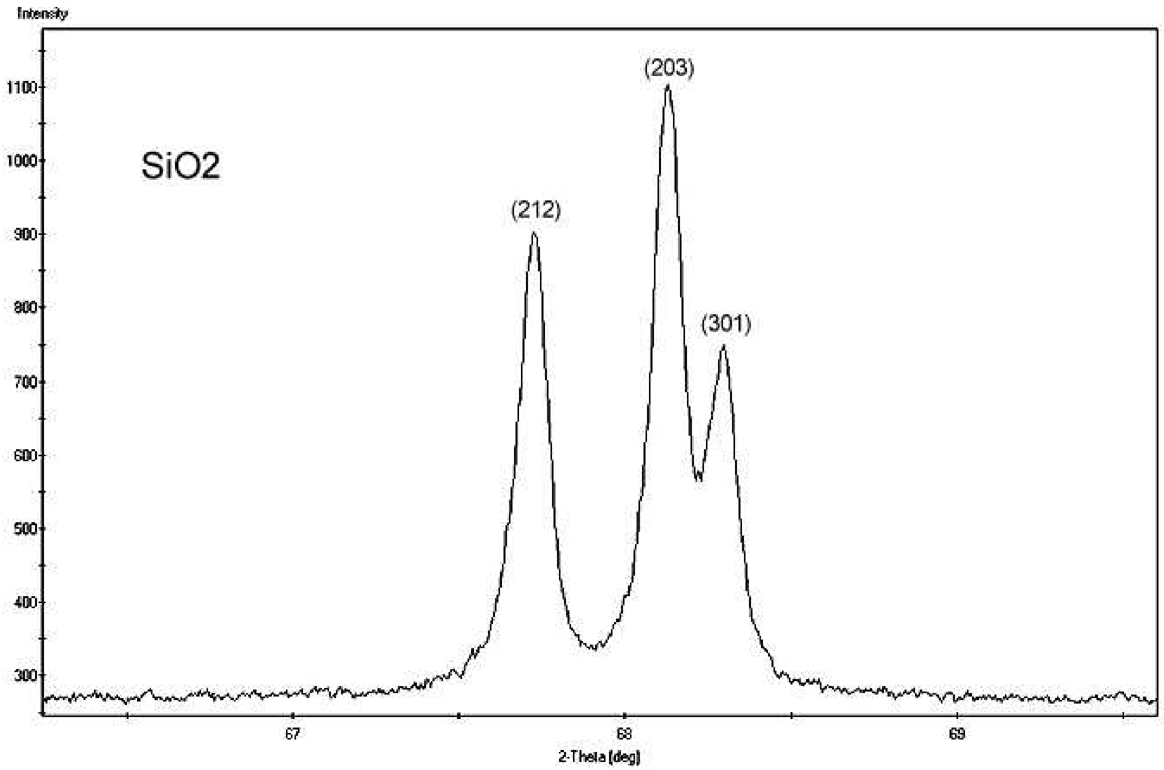

Fig. 20 The three fingers of quarz

18 References

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