Absorption correction for flat specimen with QUATTRO-TRANS

W.-H. Kaps, J. Ihringer, W. Prandl

Project: The Multiple-Detector System for the powder diffractometer at beamline B2

Institut für Kristallographie, Universität Tübingen, Auf der Morgenstelle 10, D-72076 Tübingen www.uni-tuebingen.de/uni/pki

Introduction

For an accurate structure analysis it is of great importance to treat properly the absorption effect, which largely influences the intensities of the diffracted beam. At the beamline B2 an multiple-detectors system (MDS) [1, 2] with four analyser diffractometers is used three of them (detector 2, 3 and 4) are operating under asymmetrical diffraction condition. Using flat specimen an absorption correction of the raw data is necessary. We developed a routine for this correction which is implemented in the program QUATTRO-TRANS [3]. Based on the formula of James [4] the geometrical case of asymmetrical diffraction condition is introduced.

Theory

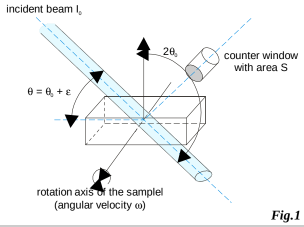


Fig.1

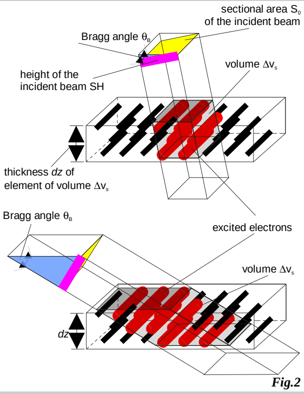


Fig.2

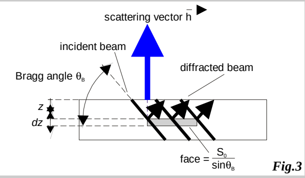


Fig.3

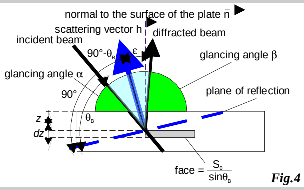


Fig.4

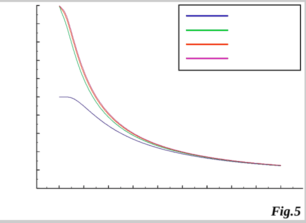


Fig.5

Transmission functions of SiO_2 data. The blue curve shows the reference data set (detector 1). The transmission of the other detectors operating under asymmetrical diffraction geometry is higher at small diffraction angles.

The area S of the counter window is perpendicular to the reflected beam when the angle of reflection is θ_0. The total energy E_s passing through the area *S* which is perpendicular to the reflected beam depends on the intensity of the incident beam I_0 the time $d\varepsilon/\omega$ to rotate through the very small angular range $d\varepsilon$ and the scattering power $R(\varepsilon)$; (I_ε / I_0). According to fig. 1 this gives

$$E_S = I_0 \cdot S \int_{angle} R(\varepsilon)\frac{d\varepsilon}{\omega} \cdot \qquad (1)$$

The integration of the scattering power $R(\varepsilon)$ gives the volume Δv_s (fig.2) and the factor *Q* which describes the scattering power $R(\varepsilon)$ in direction of $2\theta_0$ and dependent on the wavelength λ.

$$\frac{E_S \cdot \omega}{I_0} = \frac{N^2\lambda^3}{\sin 2\theta_B}\,|F|^2 \left(\frac{e^2}{mc^2}\right)^2 \cdot \frac{dz\,S}{\sin\theta_B} \qquad (2)$$

$$\frac{E_S \cdot \omega}{I_0} = Q \cdot \Delta v_S \qquad (3)$$

The sectional area S_0 and the height *SH* of the incident beam are constant and independent on the Bragg angle θ_0. It is significant to note that a small Bragg angle θ_0 extends the volume Δv_s (the number of excited electrons increases) but the density of the electrons does not change. In the case of symmetrical diffraction condition the scattering vector **h** is perpendicular to the surface of the sample plate (fig. 3). The path length of the incident and diffracted beam are equal; $z / \sin\theta_B$. The integrated intensity is given by (4). Particulary for the MDS the asymmetrical diffraction condition (fig. 4) means for the detectors 2, 3 and 4: the incident beam falls with an glancing angle α (θ) on the surface of the sample and the detectors receive the diffracted beams with three different glancing angles β (det. 2, θ+offset 1; det. 3, θ+offset 2; det. 4, θ+offset 3). QUATTRO-TRANS works on the least square routine with a reference data set (raw data of the first detector, symmetrical diffraction geometry) and the data sets of the three detectors in asymmetrical diffraction geometry. The fixed parameter of the fit is the total linear absorption coefficient μ. The scale, the offsets of the three detectors in asymmetrical diffraction geometry and the distance the beam travels through a homogeneous isotropic material are refined by the fit. The integrated intensity is given by (5).

$$\frac{E_S \cdot \omega}{I_0 \cdot S_0} = \frac{Q}{2\mu} \cdot \left[1 - \exp^{-2\frac{\mu \cdot D_S}{\sin\theta_B}}\right] \qquad (4)$$

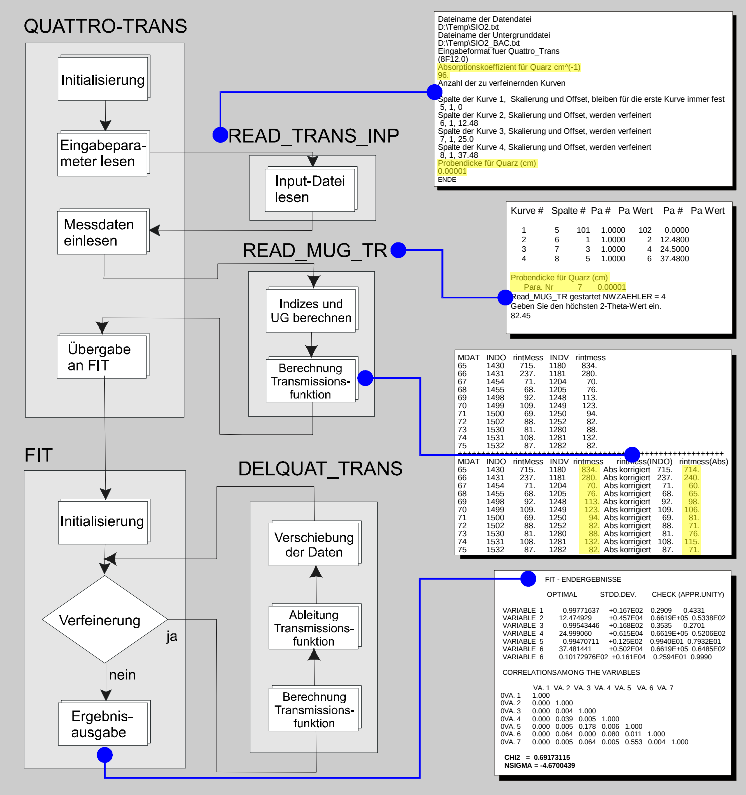
$$\frac{E_S \cdot \omega}{I_0 \cdot S_0} = \frac{Q}{\mu \cdot \left(1 + \frac{\sin\alpha}{\sin\beta}\right)} \cdot \left[1 - \exp^{-\mu\left(\frac{1}{\sin\alpha} + \frac{1}{\sin\beta}\right)\cdot D}\right] \qquad (5)$$

For each data set a transmission function is calculated. In fig. 5 the transmission functions of SiO_2 are shown ($\mu = 96$ 1/cm, D = 0.001 cm). The reference data set (detector 1; TRA, (6)) differs from the other data sets (TRV, (7)) caused by the offsets.

$$TRA = \frac{1}{2\mu} \cdot \left[1 - \exp^{-\mu D_S\left(\frac{2}{\sin\alpha}\right)}\right] \qquad (6)$$

$$TRV = \frac{1}{\mu\left[1 + \frac{\sin\alpha}{\sin\beta}\right]} \cdot \left[1 - \exp^{-\mu D_S\left(\frac{1}{\sin\alpha} + \frac{1}{\sin\beta}\right)}\right] \qquad (7)$$

QUATTRO-TRANS



```
Dateiname der Datendatei
D:\Temp\SIO2.txt
Dateiname der Untergrunddatei
D:\Temp\SIO2_BAC.txt
Eingabeformat fuer Quattro_Trans
(8F12.0)
Absorptionskoeffizient für Quarz cm^(-1)
96.
Anzahl der zu verfeinernden Kurven

Spalte der Kurve 1,  Skalierung und Offset, bleiben für die erste Kurve immer fest
 5, 1, 0
Spalte der Kurve 2, Skalierung und Offset, werden verfeinert
 6, 1, 12.48
Spalte der Kurve 3, Skalierung und Offset, werden verfeinert
 7, 1, 25.0
Spalte der Kurve 4, Skalierung und Offset, werden verfeinert
 8, 1, 37.48
Probendicke für Quarz (cm)
0.00001
ENDE
```

```
Kurve #   Spalte #  Pa #   Pa Wert    Pa #   Pa Wert

   1         5       101    1.0000     102    0.0000
   2         6         1    1.0000       2   12.4800
   3         7         3    1.0000       4   24.5000
   4         8         5    1.0000       6   37.4800

Probendicke für Quarz (cm)
     Para. Nr        7      0.00001
Read_MUG_TR gestartet NWZAEHLER = 4
Geben Sie den höchsten 2-Theta-Wert ein.
82.45
```

```
MDAT   INDO   rintMess   INDV   rintmess
65     1430     715.     1180     834.
66     1431     237.     1181     280.
67     1454      71.     1204      70.
68     1455      68.     1205      76.
69     1498      92.     1248     113.
70     1499     109.     1249     123.
71     1500      69.     1250      94.
72     1502      88.     1252      82.
73     1530      81.     1280      88.
74     1531     108.     1281     132.
75     1532      87.     1282      82.
++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
MDAT   INDO   rintMess   INDV   rintmess       rintmess(INDO)   rintmess(Abs)
65     1430     715.     1180     834.  Abs korrigiert  715.    714.
66     1431     237.     1181     280.  Abs korrigiert  237.    240.
67     1454      71.     1204      70.  Abs korrigiert   71.     60.
68     1455      68.     1205      76.  Abs korrigiert   68.     65.
69     1498      92.     1248     113.  Abs korrigiert   92.     98.
70     1499     109.     1249     123.  Abs korrigiert  109.    106.
71     1500      69.     1250      94.  Abs korrigiert   69.     81.
72     1502      88.     1252      82.  Abs korrigiert   88.     71.
73     1530      81.     1280      88.  Abs korrigiert   81.     76.
74     1531     108.     1281     132.  Abs korrigiert  108.    115.
75     1532      87.     1282      82.  Abs korrigiert   87.     71.
```

```
              FIT - ENDERGEBNISSE

              OPTIMAL         STDD.DEV.     CHECK (APPR.UNITY)

VARIABLE  1     0.99771637    +0.167E02   0.2909      0.4331
VARIABLE  2    12.474929      +0.457E04   0.6619E+05  0.5338E02
VARIABLE  3     0.99543446    +0.168E02   0.3535      0.2701
VARIABLE  4    24.999060      +0.615E04   0.6619E+05  0.5206E02
VARIABLE  5     0.99470711    +0.125E02   0.9940E01   0.7932E01
VARIABLE  6    37.481441      +0.502E04   0.6619E+05  0.6485E02
VARIABLE  6    0.10172976E02  +0.161E04   0.2594E01   0.9990

CORRELATIONSAMONG THE VARIABLES

          VA. 1  VA. 2  VA. 3  VA. 4  VA. 5  VA. 6  VA. 7
0VA. 1    1.000
0VA. 2    0.000  1.000
0VA. 3    0.000  0.004  1.000
0VA. 4    0.000  0.039  0.005  1.000
0VA. 5    0.000  0.005  0.178  0.006  1.000
0VA. 6    0.000  0.064  0.000  0.080  0.011  1.000
0VA. 7    0.000  0.005  0.064  0.005  0.553  0.004  1.000

 CHI2   =  0.69173115
 NSIGMA = -4.6700439
```

Raw data and synthetic data

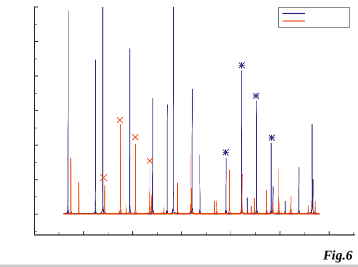


Fig.6

Raw data of LaB_6 + Si. The blue curve shows the reference data set (detector 1) and the red curve the data of detector 3. Themarked peaks are equal reflex positions. Compare the maximum cps of these peaks with those in fig. 7.

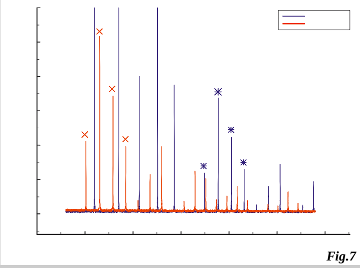


Fig.7

Synthetic data of LaB_6 + Si (blue curve det. 1, red curve det. 3). The maximum cps of themarked peaks are changed compared with those in fig. 6. This is caused by the transmission function used to generate the synthetic data.

References

[1] W.-H.Kaps, J.Ihringer, W.Prandl, M.Schilling, The Multiple-Detector System for the powder diffractometer at beamline B2 (HASYLAB), XVIIIth IUCr Congress Glasgow, P13.01.016, (1999), S.239

[2] W.-H.Kaps, H.Ehrenberg, J.Ihringer, W.Prandl, M.Schilling, The Multiple-Detector System at beamline B2: The modular system of the analyser units, HASYLAB Nutzertreffen, Hamburg, 28.01.2000

[3] W.-H.Kaps, Technisches Handbuch: Multi-Detektor-System, Universität Tübingen, Inst. f. Kristallographie, 2000

[4] R.W. James, “The Optical Principles of the Diffraction of X-Rays” London: Bell, 1967 (The Crystallin State - VOL II), S. 46 ff.

[5] J. Ihringer, A quantitative measure for the goodness of fit in profile refinements with more than 20 degrees of freedom, J. Appl. Cryst. (1995), 28, 618-619

Acknowledgement

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