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ON THE POSSIBILITY OF DETERMINING THE ODD PART OF THE ORIENTATION DISTRIBUTION FUNCTION IN QUARTZ-LIKE STRUCTURES USING ANOMALOUS SCATTERING

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INTRODUCTION

At present one of the most puzzling problems in texture analysis is the loss of information on the orientation distribution function (ODF) in pole figure measurements, i.e., the problem of the so-called ghost phenomena. Matthies and Helming/1/ have discussed and classified all possible crystal structures with respect to this problem, taking into account anomalous scattering to be in principle an experimental tool for the determination of the so-called unreduced pole figures, which are a premise to calculate ghost-free ODFs. In this context it seems to be favourable to evaluate the practical possibility of measuring unreduced pole figures by diffraction methods using anomalous scattering.

MATHEMATICAL BACKGROUND

In any common diffraction experiment the structure factor

$$|F_{\vec{h}_i}|^2 = \Big|\sum_{j=1}^{r} b_j e^{2\pi i \vec{h}_i \cdot \vec{\rho}_j}\Big|^2 \qquad (1)$$

is independent of the sign of $\vec{h}_i$, given in Miller indexes. The position of the j-th atom in the basis of r atoms is described by $\vec{\rho}_j$, b_j is the atomic scattering factor or scattering length of the j-th atom. In this case the $\vec{h}_i$ reduced pole figure can be written/2/

$$\frac{P_{\vec{h}_i} + P_{-\vec{h}_i}}{2} = \tilde{P}_{\vec{h}_i}(\vec{y}) = 4\pi \sum_{\ell=0}^{\infty}\sum_{\mu=1}^{M(\ell)}\sum_{\nu=1}^{N(\ell)} \frac{c_\ell^{\mu\nu}}{2\ell+1}\,\frac{1+(-1)^\ell}{2}\,k_\ell^{\mu}(\vec{h}_i)\,k_\ell^{\nu}(\vec{y}), \qquad (2)$$

using the series expansion method. In the formula $c_\ell^{\mu\nu}$ are series expansion coefficients, $k_\ell^{\mu}(\vec{h}_i)$ and $k_\ell^{\nu}(\vec{y})$ are the symmetry-adapted spherical harmonics for crystal and sample orientation, respectively. $M(\ell)$ and $N(\ell)$ are the numbers of linearly independent spherical harmonics at given ℓ for crystal and specimen symmetry (see ref./1/).

If ℓ is odd, the right-hand side of equation (2) will become zero, so it is impossible to determine the coefficients $c_\ell^{\mu\nu}$ for this ℓ. That loss of information in pole figure measurements leads to the known ghost effects.

If the radiation used for the diffraction experiment is highly absorbed by the sample atoms, the scattering length b_j becomes complex and Friedel's law

$$|F_{\vec{h}_i}|^2 = |F_{-\vec{h}_i}|^2 \qquad (3)$$

is violated for some reflexes in a sample containing one kind of enantiomorphic crystals. In this case in place of equation (2) we write:

$$P_{\vec{h}_i}(\vec{y}) = 4\pi \sum_{\ell}\sum_{\mu}\sum_{\nu} \frac{c_\ell^{\mu\nu}}{2\ell+1}\,[a_{\vec{h}_i} + a_{-\vec{h}_i}(-1)^\ell]\,k_\ell^{\mu}(\vec{h}_i)\,k_\ell^{\nu}(\vec{y}), \qquad (4)$$

where $a_{\vec{h}_i}$ is proportional to the structure factor and $a_{\vec{h}_i} + a_{-\vec{h}_i} = 1$. If $a_{\vec{h}_i} \neq a_{-\vec{h}_i}$ the right-hand side of equation (4) doesn't vanish. In this way it is possible to determine $c_\ell^{\mu\nu}$ for odd ℓ also.

DISCUSSION

The most of common crystal structures possess inversion and/or mirror symmetry elements. In the Matthies-Helming classification they belong to types II or III. In these cases even anomalous scattering cannot avoid some loss of information in ODF determinations, leading to the occurrence of ghost effects. No loss of information in anomalous scattering experiments is expected in type I crystals having neither inversion nor mirror symmetry elements. Prominent examples for this type are quartz-like structures (symmetry class D_3). Because of the interest of geological fabric analysis in prefered orientation of quartz rocks, the possibility of avoiding ghost phenomena is worth to discuss for this class of structures. Unfortunately no imaginary part of a scattering length of silicon and oxygen is known in all the range of thermal neutron wavelengths. In this way anomalous scattering experiments in quartz are not possible using neutron diffraction. On the other hand anomalous scattering of X-rays in quartz is a well-known phenomenon/3/. Therefore the latter technique can be used to look for ghost effects in this substance.

The isotope ^{10}B is a notable neutron absorber. Because there are quartz-like forms of the compounds BPO_4 and $BAsO_4$ where silicon is substituted alternatively by boron and phosphorous or arsenic/6/, the scattering of neutrons by BPO_4 was considered taking into account isotope ^{10}B only and natural boron, which contains 20 percents of ^{10}B.

Table

Diffraction parameters of quartz-like crystals, with anomalous scattering contributions

h k i l	SiO_2 · X-Rays (Cr K α) d(Å)	F^2	a	BPO_4 · Neutrons (λ = Å) d(Å)	Natural B F^2	Natural B a	Isotope ^{10}B F^2	Isotope ^{10}B a
$1\,1\,\bar{2}\,0$	2.455	385.6	0.536	2.235	11.79	0.508	12.31	0.544
$\bar{1}\,\bar{1}\,2\,0$		334.1	0.464		11.40	0.492	10.31	0.456
$1\,1\,\bar{2}\,1$	2.235	147.1	0.588	2.038	6.11	0.515	8.95	0.558
$\bar{1}\,\bar{1}\,2\,\bar{1}$		103.2	0.412		5.75	0.485	7.09	0.442
$1\,1\,\bar{2}\,2$	1.816	519.6	0.505	1.661	13.16	0.502	8.60	0.517
$\bar{1}\,\bar{1}\,2\,\bar{2}$		508.9	0.495		13.06	0.498	8.05	0.483
$1\,1\,\bar{2}\,3$	1.451	166.9	0.479	1.329	6.15	0.492	4.05	0.445
$\bar{1}\,\bar{1}\,2\,\bar{3}$		181.6	0.521		6.34	0.507	5.06	0.555
$2\,2\,\bar{4}\,0$	1.227	329.1	0.477	1.117	10.04	0.488	5.70	0.410
$\bar{2}\,\bar{2}\,4\,0$		361.0	0.523		10.51	0.512	8.19	0.590
$2\,2\,\bar{4}\,1$	1.197	138.7	0.482	1.090	7.46	0.494	5.21	0.460
$\bar{2}\,\bar{2}\,4\,\bar{1}$		149.2	0.518		7.63	0.506	6.10	0.540

The structure factors for X-ray diffraction in quartz and for neutron diffraction in BPO_4 were investigated for the first 22 low-index Bragg reflections. The structure of quartz has been taken from /4/ and the complex atomic scattering factors from /5/. The data for BPO_4 are found in /5,6/.

Only six reflections show the wanted effect. According to /1/ a maximum series expansion degree $\ell = 19$ can be approached. The structure factors for the above-mentioned reflections are presented in the Table (the pairs $11\bar{2}0/\bar{1}\bar{1}20$ and $22\bar{4}0/\bar{2}\bar{2}40$ are structurally equivalent, but differ in their anomalous scattering part).

The weight factors $a_{\vec{h}_i}$ from equation (4) are also given in the Table. In general the largest differences between $a_{\vec{h}_i}$ and $a_{-\vec{h}_i}$ are found for $^{10}BPO_4$, but this material can only posses the character of a model. For natural BPO_4 the necessary experimental accuracy has to be very high to see significant effects over all pole figures, taking into account inevitable corrections for irradiated volume and strong beam weakening, which are changing during the measurements. Besides of Li, Gd and some rare earth elements, ^{10}B is one of the most neutron absorbing materials, so the effect of anomalous scattering of neutrons for other substances is expected to be much lower than that given in the Table.

X-ray investigations of quartz seem to be more optimistic. The anomalous scattering effect should be seen in $11\bar{2}0$ and $11\bar{2}1$ reflections without essential troubles. To analyze the other reflections, the experimental accuracy has to be also high. Besides of the above-mentioned corrections the mean difficulty in this method is the high linear absorption factor $\mu \sim 340$ cm^{-1}. It permits only a sample thickness of some 10^{-2} mm for transmission diffraction technique, necessary for a complete pole figure measurement. Especially in natural rocks this specimen dimension is too small to give a representative grain statistics of the most cases. The previous considerations are valid for one kind of enantiomorphic crystals, i.e., for left-handed or right-handed ones. In general natural rocks will contain both forms. In this way, two independent ODFs have to be determined from the available data simultaneously, leading to a lower possible series expansion degree.

The discussion of this paper has shown the principal possibility of low degree ghost corrections in quartz-like structures by X-ray anomalous scattering, supposing a high experimental accuracy. The use of neutron diffraction for the odd part determination of the ODF is only possible with selected elements.

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Фельдман К., Фуентес Л. E14-83-697
О возможности измерений нечетной части ФРО
в кристаллах со структурой кварца
с использованием аномального рассеяния

Обсуждается возможность измерений неприведенных полюсных фигур для текстурного анализа в кристаллах со структурой кварца с использованием аномального рассеяния рентгеновских лучей и тепловых нейтронов. Приведены необходимые формулы и расчетные данные для структурных факторов различных полюсных фигур. Показана принципиальная возможность поправок на "духи" функции распределения ориентации, при этом предполагается высокая экспериментальная точность.

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On the Possibility of Determining the Odd Part
of the Orientation Distribution Function
in Quartz-Like Structures Using Anomalous Scattering

The practical possibility of measuring unreduced pole figures in texture analysis of quartz-like structures using anomalous scattering of X-rays and neutrons is evaluated. The corresponding formulae and numerical values of the determining factors are given. From these it is shown the principal possibility of low-degree "ghost corrections" in natural and Boron-substituted quartz supposing a high experimental accuracy.

The investigation has been performed at the Laboratory of Neutron Physics, JINR.

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