Natural crystals selection process for neutron diffraction applications

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This work aims to show a selection process for natural crystals that considers the major characteristics and performance as gratings for neutron monochromators using the neutron diffraction technique. A total of 19 crystals have been selected and classified regarding their adequacy for use as neutron diffraction devices. Applying special criteria, method and the rocking curve technique, the measurements have been performed and the theoretically available values compared with the experimental results, obtained directly from a neutron diffractometer in operation at the IEA-R1 (5 MW) nuclear research reactor. The choice for natural crystals, expanding the operational range of the neutron diffraction instruments, is related with the purpose of getting monochromatic neutron sources suitable for the applications. The neutron has proved to be the most powerful microscopic testing particle in condensed matter studies and in many other areas of application. © 1998 American Institute of Physics. [S0034-6748(98)01809-7]

I. INTRODUCTION

Neutrons have been used as microscopic probes to study the properties of materials in several fields of applications.1 The neutrons are derived from a variety of sources. A review included some 70 experts in different areas of neutron research, sources and applications.2 However, during the last years the steady state research reactors with thermal neutron fluxes within the usual range of $10^{12}$ – $10^{15}$ neutrons cm$^{-2}$ s$^{-1}$ have been used in most of the investigations. In a steady reactor, the crystal spectrometers and neutron diffractometers are the main instruments for a variety of studies.

The neutron diffraction (ND) technique is appropriate to investigate both structure of solids and neutron inelastic scattering. Techniques to study the dynamics of solids and liquids have traditionally been carried out with steady state sources using spectrometers and diffractometers, because of certain advantages of operating these instruments in a specific manner. Historically, the main application for neutron diffraction was research on condensed matter where the aim was to obtain information about the crystal structure or spin configuration of magnetic materials. It was later found that the inelastic scattering phenomena provides useful information about the elementary excitations in solids, e.g., phonons and magnons.3,4 There is an increasing interest in applying the ND technique to various materials, not only for the basic sciences, chemistry, biology, earth and space material sciences but also for some industrial technologies. Sometimes, the application of neutron diffraction is the sole available technique for obtaining a particular parameter.5,6

The monochromator crystal or analyzer is the most important part of the ND instrument. Conventional artificial crystals of metallic monoelements and those of simple crystalline structure are the most usual according to the following priority rank: pyrolytic graphite (PG), Be, Cu, Pb, Al, Zn and the semiconductors Ge and Si. The Heusler’s ferromagnetic, ternary alloy (Cu$_2$MnAl or Cu$_2$MnSb) is used in experiments concerning magnetism and polarization. The main difficulties concerning the use of artificial crystals include the following: doubtful quality control during the manufacture, limitation of the choice of type and size, quasiperfect formation of the crystalline structure, and operational limitation taking in account the distance between the crystalline planes $<3.5$ Å.7

The quasiperfect crystalline structure of the common mosaic blocks of the artificial crystals shows strong neutron attenuation in the reflectivity due to the primary extinction phenomena. On the contrary, the limited periodicity of the mosaic crystalline blocks alignment makes the “ideally imperfect crystals” showing larger widths on the distribution preferable. With the ideally imperfect crystals occurs the predominance on the less intense phenomena of neutron attenuation called secondary extinction. With the exception of gems, the greatest majority of natural occurring crystals are included in the class of the ideally imperfect mosaic crystals. These types are preferred for use in neutron diffraction experiments.8

II. OBJECTIVE

The purpose of this work lies in the implementation of criteria, methodologies and experimental techniques for the selection and classification of natural crystalline types since they are available from Brazil and other countries’ mineral resources. The natural crystalline types and its main diffraction phenomena are included in the class of the ideally imperfect mosaic crystals.
tion plane families makes them suitable for use as monochromators or analyzers coupled to instruments operating with the ND technique. The verification of the selected performance of the crystals was accomplished with the use of a neutron diffractometer installed close to the 5 MW IEA-R1 nuclear research reactor of the ‘‘Instituto de Pesquisas Energéticas e Nucleares (IPEN/CNEN-SP)’’.

The expected results of this work include the following: to alleviate difficulties due to the exclusive use of the artificial crystals and hence to increase the instrumental operational range; to allow greater flexibility on the suitable choice of the types and crystalline plane families that best fit the intended operational time; and to improve the flexibility of the collimator’s available geometry and the experimental arrangement.

Moreover, the natural crystals show other qualities essential to the experiments; namely, they provide optimal dimensions and formats with various cleavage planes and partitions, voluntary choice of impurities according to the origin, greater distances between the crystalline planes, and a relatively low cost.

The main obstacles to the selection of the natural crystalline types which were overcome are the following: complexity of the chemical formulation, different crystalline systems and lack of atomic-nuclear parameters essential to the crystals neutron reflectivity calculations. The last difficulty was overcome with the organization of the information and data available in the specialized literature. After that, the essential parameters of natural crystals reflectivity and their main families of neutron diffraction planes were determined, because a data file also extensible to the main artificial monochromators was set up.

In order to evaluate the neutron reflectivity power for the different natural crystalline types, first formulations from literature were applied, these formulations are usually applied for artificial conventional crystals. A more comprehensive formula was also implemented that provides a parameter called crystalline Merit Figures that best fit the selection process of the other natural crystals. The application allowed the pre-classification of 15 naturally occurring crystals and identification of main diffraction planes families. Afterwards, the experimental corroboration of 12 of them was done through the experimental determination of the intensity distributions around the monochromatic neutron beam emerging from the neutron diffractometer, called a ‘‘rocking curve’’.

III. THEORETICAL BACKGROUND

The principle of the diffraction instrument’s operation is based on the Bragg relation: \( n\lambda = 2d_{hkl}\sin\theta \), where: \( n = 1,2,3,\ldots \), \( \lambda \) = wavelength associated, \( d_{hkl} \) is the distance between the crystalline planes, \( (h,k,l) \) are the Miller’s or Miller–Bravais’ \((H,K,L)\) indexes and \( \theta \) is the incidence angle of the polychromatic neutron beam. The value of \( \lambda = h/mv = 0.286/\sqrt{E(eu)} \) Å (Broglie’s hypothesis), and where \( h \) depicts the Planck’s constant; \( m \), \( v \) and \( E \) are, respectively, the mass, velocity and kinetic energy of the neutron.\(^7\)

The commonly used crystals for neutron diffraction have mosaic blocks small enough (<5000 Å) so that the primary extinction shall be considered negligible. The reduction of neutron beam intensity inside the crystal depends on the angular distribution \( W(\Delta) \), relative to the position of the blocks’ normal lines, assuming an isotropic and normalized Gaussian function, described by the expression

\[
W(\Delta) = \frac{1}{\sqrt{2\pi}} \exp \left(-\left(\frac{\Delta^2}{2\sigma^2}\right)\right),
\]

where \( \Delta \) refer to the normal lines angular deviations from the distribution mean direction, being the width of the crystal’s mosaic \( \sigma = 0.42661 \beta \), i.e., characterized by the crystalline parameter \( \beta \) directly derived from the measurement of angular full width at half maximum (FWHM) on the rocking curve.\(^10\)

The diffracted monochromatic radiation intensity by an ideal small crystal is equal to \( Q \delta V \), when it is rotated around the Bragg’s position on the rocking curve considering \( \delta V \) the crystal’s element of volume and \( Q \) the crystallographic quantity defined by Zachariasen,\(^11\) i.e., the reflectivity integrated by volume unit:

\[
Q = \lambda^3 N^2_c F^2_{hkl} / \sin 2\theta,
\]

where \( N_c \) is the number of cells per volume unit and \( F_{hkl} \) represents the structure factor of the crystalline planes family.

Bacon and Lowde\(^12,13\) assumed the crystal ideally imperfect mosaic as neutron not absorbing with the value \( \Delta = 0 \) in \( W(\Delta) \), being the maximum reflectivity of the diffracted monochromatic beam described by the expression is

\[
R^\text{max} = \left[1 + \frac{1}{\sqrt{2\pi}}(Q t_0 / \eta \sin \theta)\right] / \left[1 + 1/\sqrt{2\pi}(Q t_0 / \eta \sin \theta)\right],
\]

where \( t_0 \) is the crystal thickness.

For the integrated reflectivity \( R^\theta \) of the mosaic crystal rotating around the vertical axis, i.e., the considered angular interval under which the crystal fully reflects the monochromatic neutron beam, the formula

\[
R^\theta \approx 0.96(\eta Q t_0 / \sin \theta)^{1/2}
\]

is used. The nondimensional constant \( \gamma \) was introduced by Holm,\(^14\) and its magnitude indicates the neutron reflectivity power of crystal, according to the following expression:

\[
\gamma = (8d^3_{hkl}t_0 \eta^2 N^2_c F^2_{hkl}) / (\sqrt{2\pi} \eta \lambda^3).
\]

IV. SELECTION PROCESS FOR NATURAL CRYSTALS

Starting from a list of the natural crystals available on the mineralogical literature,\(^15–19\) in a preliminary procedure to the selection process, the basic criteria were considered: simplicity on the chemical composition and on the crystalline spatial group, compatibility of physical state, available dimensions, preferential aggregation, difficulties on the acquisition of samples, etc. After that, the selective process itself was implemented, using four stages targeting the main crystal operational characteristics, namely:

(i) Basic characteristics: analysis of physical–chemical, mechanical–metallurgical and crystallographic prop-
TABLE I. Results from the selection process of 19 available natural crystals for the application of the ND technique.

<table>
<thead>
<tr>
<th>Crystal and plane ((h,k,l)) or ((H,K,I,L))</th>
<th>Crystalline system and spatial group</th>
<th>Crystalline parameters (a_0, b_0, c_0) (Å) or (\beta_0(°))</th>
<th>(V_c) ((10^{-24} \text{ cm}^3))</th>
<th>(d_{\text{slit}}) or (d_{\text{HKIL}}) (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beryl (10(\overline{1})0)</td>
<td>Hexagonal (beryl)-P(6\overline{3}mg)</td>
<td>8.215; 9.192</td>
<td>675.98</td>
<td>7.9804</td>
</tr>
<tr>
<td>Calcite (10(\overline{1})1)</td>
<td>Trigonal-R (calcite)-R(3\overline{1}c)</td>
<td>4.9899; 17.064</td>
<td>121.90</td>
<td>3.0348</td>
</tr>
<tr>
<td>Corundum (0002)</td>
<td>Trigonal-R (corundum)-R(3\overline{1}c)</td>
<td>4.7591; 12.9894</td>
<td>129.75</td>
<td>6.4947</td>
</tr>
<tr>
<td>Diamond (111)</td>
<td>Isometric (diamond)-F(d)3(m)</td>
<td>5.3670</td>
<td>45.39</td>
<td>2.0594</td>
</tr>
<tr>
<td>Fluorite (111)</td>
<td>Isometric (fluorite)-F(m)3(m)</td>
<td>5.4638</td>
<td>163.41</td>
<td>3.1545</td>
</tr>
<tr>
<td>Galena (200)</td>
<td>Isometric (halite)-F(m)3(m)</td>
<td>5.9360</td>
<td>209.16</td>
<td>2.7680</td>
</tr>
<tr>
<td>Gypsum (020)</td>
<td>Monoclinic-C(2\overline{1}c)</td>
<td>5.68; 15.18; 6.29; 113.83°</td>
<td>496.10</td>
<td>7.5910</td>
</tr>
<tr>
<td>Graphite (0002)</td>
<td>Hexagonal (graphite)-P(6\overline{3}mcc)</td>
<td>2.4612; 6.7079</td>
<td>35.19</td>
<td>3.3511</td>
</tr>
<tr>
<td>Halite (200)</td>
<td>Isometric (halite)-F(m)3(m)</td>
<td>5.6402</td>
<td>179.43</td>
<td>2.8138</td>
</tr>
<tr>
<td>Hematite (0002)</td>
<td>Trigonal-R (corundum)-R(3\overline{1}c)</td>
<td>5.0329; 13.7492</td>
<td>301.61</td>
<td>6.8746</td>
</tr>
<tr>
<td>Lepidolite (0002)</td>
<td>Monoclinic (mica)-C(2\overline{1}c)</td>
<td>9.2; 5.3; 20.0; 98.0°</td>
<td>965.70</td>
<td>9.9903</td>
</tr>
<tr>
<td>Magneteite (111)</td>
<td>Isometric (spinel)-P(3\overline{2}1)</td>
<td>8.3940</td>
<td>591.43</td>
<td>4.8463</td>
</tr>
<tr>
<td>Muscovite (0002)</td>
<td>Monoclinic (mica)-C(2\overline{1}c)</td>
<td>5.203; 8.995; 20.030; 94.47°</td>
<td>934.57</td>
<td>9.6614</td>
</tr>
<tr>
<td>Periclase (200)</td>
<td>Isometric (halite)-F(m)3(m)</td>
<td>4.2117</td>
<td>74.71</td>
<td>2.1059</td>
</tr>
<tr>
<td>Pyrite (200)</td>
<td>Isometric (pyrite)-P(a)3</td>
<td>5.4175</td>
<td>159.00</td>
<td>2.7088</td>
</tr>
<tr>
<td>Quartz (10(\overline{1})0)</td>
<td>Trigonal-R-P(3\overline{2}1)</td>
<td>4.91304; 5.40463</td>
<td>113.01</td>
<td>4.2550</td>
</tr>
<tr>
<td>Scheelite (101)</td>
<td>Tetragonal (scheelite)-(14_1/a)</td>
<td>5.242; 11.372</td>
<td>312.49</td>
<td>4.7610</td>
</tr>
<tr>
<td>Silvite (200)</td>
<td>Isometric (halite)-F(m)3(m)</td>
<td>6.2951</td>
<td>249.23</td>
<td>3.7419</td>
</tr>
<tr>
<td>Topaz (303)</td>
<td>Orthorhombic-P(mm)</td>
<td>8.394; 8.792; 4.649</td>
<td>343.10</td>
<td>1.3560</td>
</tr>
</tbody>
</table>

Properties: stability to the external agents; available geometry and dimensions, cleavage and partition; reasonable costs, etc;

(ii) **Macroscopic and atomic characteristics**: preferential composition of light atoms; minimum volume of the unit cell; high structure factor for the main families of diffraction planes; distances between planes within the range from 0.5 to 10 Å; high Debye temperature of the solid; minimum percentages of characteristic impurities; limited crystalline defects and inclusions.

(iii) **Microscopic and nuclear characteristics**: Coherent or Bragg scattering cross section for neutrons greater then as compared to the sum of the parasitic absorption and scattering cross sections (incoherent, inelastic, diffuse, residual, etc.); substitute impurities compatible with the primary elements.

(iv) **Experimental comparison on the neutron diffractometer**: Examination of the several planes families with potential for diffraction in natural crystals; rocking curves relatively narrower and having a Gaussian format; diffracted maximum intensities compatible with the theoretical forecasts; low scattering of the background; physical–chemical and thermal–mechanical capabilities for acceptance of special techniques for the crystalline mosaic widening and following substantial increase of the diffracted maximum intensity; effective reproducibility of the experimental conditions.

After the conclusion of the process stage (iii) 19 crystalline types (shown in Table I) were selected, presenting the primary characteristics for the neutron diffraction. Included are the diamond and natural occurring graphite crystals, considered as the best neutron monochromators, but only used as references on the performed calculations. The latter two crystals are difficult to obtain, respectively, in suitable size and quality for the neutron diffraction purposes and they are also considered as having high cost or being rare in the natural reserves.

Figure 1 shows a consolidated listing of the 19 selected natural crystals and 8 types of the usual artificial ones, as a function of their respective distances between their main families of neutron diffraction planes. Noticeable is the enlargement of the operational interval up to 10 Å provided to the instruments operating with the ND technique, through use and complementation with the natural types.

V. METHODOLOGY OF SELECTION

The natural crystals are more complex compounds having constituent elements or impurities that sometimes exhibit high neutron cross sections, hence conditions are very different from those of the artificial crystals constituted by a single element that may have a low neutron absorption ability.

In order to establish a more complete and suitable method for the selection and pre-qualification processes of natural crystals, the expression called crystalline Merit Figures \((F_M)\) is here presented, derived from Holm’s original \(\gamma\) formula [Eq. (5)].

The \(\gamma\) expression was considered basic due to the implication of most the crystallographic and atomic parameters which are more representative for a given crystal and its families of planes for neutron diffraction. To the Holm’s original expression was initially added three parameters, namely: \(\lambda\) according to the Bragg relation; \(\beta\) (measured directly from the rocking curve); isotropic temperature factor \(\exp(-M)\), where \(M = \beta(\sin^2 \theta h)\), with \(\beta = 8 \pi^2 \beta^2\) and \(\beta\) describing the quadratic mean displacement of the atoms referring to their mean positions, in the direction perpendicular to the family of neutron diffraction planes.20 The modified Holm’s expression takes the new form

\[
\gamma^* = \left[3.758 d_{hkI}^2 \lambda t g N^2 (e^{-M F_{hkI}})^2\right] / B n^2 \sin \theta.
\] (6)
The new expression is more complete, but it still remains under the dependence of the parameter related to the geometrical, crystallographic and atomic properties of the material.

We would also like to consider the nuclear properties of the crystalline material. In this sense, the introduction of a correcting factor is proposed, defined by the ratio between the macroscopic elastic coherent scattering cross section \( \Sigma_{\text{coher.}} \) and the sum of the absorption macroscopic cross section \( \Sigma_a \), with another competitive one from parasite interaction processes, represented by the macroscopic scattering cross sections \( \Sigma_{\text{paras.}} \), where

\[
\Sigma_{\text{paras.}} = \Sigma_{\text{inc.}} + \Sigma_{\text{inel.}} + \Sigma_{\text{residual}},
\]

i.e., macroscopic scattering cross sections incoherent, inelastic, and residual, respectively. The value of the \( \Sigma_{\text{coher.}} \) should prevail over the \( \Sigma_a \) and \( \Sigma_{\text{paras.}} \). Applying this corrective factor, the \( F_M \) formula is denoted by

\[
F_M = \frac{\gamma}{\Sigma_a + \Sigma_{\text{paras.}}} [\Sigma_{\text{coher.}}].
\]  

(7)

The application of the \( F_M \) formula operates as a final stage before the experimental evidence in the selection and classification processes of natural crystals. It also acts to evaluate the performance of a given variety of crystalline type as a function of the neutron diffracted intensity.

VI. EXPERIMENTAL RESULTS

The experiments performed for the corroboration and final classification of the natural crystal and their main families of neutron diffraction planes were carried out using the conventional neutron diffractometer, installed close the horizontal channel for experiments on the research nuclear reactor IEA-R1. A total of 62 samples of 12 selected natural crystals were examined through the respective plotting of the rocking curves.

The artificial monochromator of Cu (220) was used on the neutron diffractometer, aligned under the angle \( \theta \) which allowed us to obtain a monochromatic beam of \( \lambda = 1.137 \text{ Å} \). The rocking curve was determined through the rotation of the crystalline sample examined around its vertical axis, after the respective alignment on the geometrical table, keeping fixed the position of the neutron detector (BF3) under \( 2 \theta \). Several rocking curves were obtained for each sample and its main crystalline plane. Thus, more then a hundred of such curves were determined.

The more representative distribution, i.e., a rocking curve having a form close to the Gaussian shape was selected for each crystalline type with a reasonable mosaic width and having the best neutron diffracted maximum intensity. To the obtained experimental set of points for each rocking distribution, a curve formed by one or various Gaussian distributions was adjusted by the minimum squares method with the aid of a mathematical computer program.

In Table II are shown the comparative results obtained with the application of the \( F_M \) formula, relative maximum intensities per unit of volume and respective \( \beta \)'s, determined for the 12 samples of the main natural crystals selected for the corroboration of the applied selection method. It can be observed that, with the application of the \( F_M \) formula compared with the relative intensity, there are small discrepancies in the positioning between the intermediary crystals, imputed to the presence of impurities with high neutron absorption cross sections in the crystalline samples.

In a comparison among the classification of the 12 main crystalline types and the respective families of diffraction
VII. DISCUSSION

The natural crystals allow the use of greater values $d_{hkl}$, up to 10 Å, hitherto limited in 3 Å by the usually employed artificial crystals, providing measurements in the subthermal region by use of the ND technique.

The performance presented by the $F_M$ formula was considered effective in accounting for the nuclear properties of the composition of the crystalline types. Another advantage of the use of the $F_M$ formula for the same crystal type is that it allows for the choice of a suitable variety according to a given experiment with the available experimental arrangement, optimizing the diffracted intensity and providing the intended resolution.

Besides the natural occurring crystals diamond(111) and graphite(0002), 17 other types and the respective main families of planes were selected for application with the ND techniques, from which 12 of them had corroborated their theoretical–experimental performance. The calcite crystal(1011) is indicated for most experiments including uses in reactors with low neutron fluxes. The quartz crystal(1010) is recommended for experiments that require better resolutions and low $\lambda$. The magnetite or ferrite(111) are useful in experiments with magnetism and neutron polarization, as an eventual substitute for the Heuler’s alloy. The crystals pyrite, galena, halite or silvite, oriented on the families of planes (200) are useful for general experiments because of their common isometric structure. The fluorite(111) replaces the conventional semiconductors Ge and Si in the suppression of second order contamination. The crystals beryl(1010), hematite(0002), gypsum(020), muscovite(0002) and lepidolite(0002) are indicated for use with neutrons with greater $\lambda$, including the subthermal and cold neutrons. The crystals periclase(200), topaz(303) and scheelite(101) are also indicated for use with the ND technique with the prominence of the first one. The sapphire or corundum are recommended for use as a high order neutron contamination filter.

### Table III. Potentiality of the $F_M$ formula in the comparison process of ten samples of calcite crystal(1011), from different sources, as a function of the mosaic width parameter and of the results from relative maximum intensity per unit volume, obtained from the rocking curve, with the neutron diffractometer—IPEN.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Main characteristics</th>
<th>Mosaic $\beta$ parameter $\times 10^{-3}$ (rd)</th>
<th>$F_M$</th>
<th>Relative maximum intensity</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>MG$^a$</td>
<td>1.149</td>
<td>2.36988</td>
<td>1.79±0.01</td>
</tr>
<tr>
<td>II</td>
<td>SP$^b$</td>
<td>1.154</td>
<td>2.36021</td>
<td>1.59±0.01</td>
</tr>
<tr>
<td>III</td>
<td>MG$^a$</td>
<td>1.265</td>
<td>2.15363</td>
<td>2.46±0.01</td>
</tr>
<tr>
<td>IV</td>
<td>SP$^b$</td>
<td>1.276</td>
<td>2.13375</td>
<td>2.41±0.01</td>
</tr>
<tr>
<td>V</td>
<td>CE$^c$</td>
<td>1.300</td>
<td>2.09512</td>
<td>1.43±0.01</td>
</tr>
<tr>
<td>VI</td>
<td>MG$^a$</td>
<td>1.333</td>
<td>2.04329</td>
<td>0.77±0.01</td>
</tr>
<tr>
<td>VII</td>
<td>CE$^c$</td>
<td>3.181</td>
<td>856.03</td>
<td>2.45±0.01</td>
</tr>
<tr>
<td>VIII</td>
<td>SP$^b$</td>
<td>3.222</td>
<td>845.40</td>
<td>2.10±0.01</td>
</tr>
<tr>
<td>IX</td>
<td>CE$^c$</td>
<td>3.947</td>
<td>690.04</td>
<td>3.65±0.02</td>
</tr>
<tr>
<td>X</td>
<td>RJ$^d$</td>
<td>4.015</td>
<td>678.30</td>
<td>8.07±0.05</td>
</tr>
</tbody>
</table>

$^a$MG: Minas Gerais.
$^b$SP: São Paulo, Brazil.
$^c$CE: Ceará.
$^d$RJ: Rio de Janeiro.
Several techniques that allow the increase of the diffracted intensity can be applied to the natural crystals: a special cut of Fankuchen (calcite and beryl); magnetic field (magnetite, ferrites and hematites); ultrasound (quartz); high temperature (fluorite); mechanical–plastic damage (pyrite, galena and hematite–specularite); beam focusing (gypsum, muscovite and lepidolite); impurities (halite and silvite); compositions of several microcrystal (quasiperfect crystals and gems, in general); fission or nuclear reactions product inclusions (muscovite, lepidolite, topaz, beryl, i.e., crystals that have in the composition elements with special nuclear properties, such as: U, B, Be, Li, etc.).

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