CBECIMAT NATAL 2002
CONGRESSO BRASILEIRO DE ENGENHARIA E CIÊNCIA DOS MATERIAIS
NATAL, 09 A 13 DE NOVEMBRO DE 2002
Universidade Federal do Rio Grande do Norte
Centro de Ciências Exatas e da Terra
Departamento de Química
Departamento de Física Teórica e Experimental

DETERMINATION OF THE β-QUARTZ CELL PARAMETERS USING NMD AZIMUTHAL ANGULAR DIFFERENCES

L. C. Campos[1], C. B. R. Parente[1], V. L. Mazzocchi[1], O. Helene[2]
CP 11049 Pinheiros, São Paulo/SP, CEP 05422-970 – lccampos@curiango.ipen.br
[1] Instituto de Pesquisas Energéticas e Nucleares – IPEN-CNEN/SP
[2] Instituto de Física – Universidade de São Paulo - USP

ABSTRACT

This work presents a method for the determination of accurate lattice parameters of hexagonal cells, using neutron multiple diffraction (NMD) patterns. For this purpose, the authors used a 00.1 β-quartz NMD Umweg pattern obtained in the IPEN-CNEN/SP neutron diffractometer. To measure the β-quartz neutron multiple diffraction pattern, the crystal was heated to 1003 K. Taking the azimuthal distances between pairs of experimental peaks and comparing them with the variable distances between corresponding curves in appropriate indexing diagrams, a series of values of the parameter in question is obtained. The weighted mean of these values gives a value for the parameter. An iterative process was used in order to obtain more precise results. In this process, a newly determined parameter is used for the determination of a new value for the other parameter. After 8 cycles of iteration, the process converged to a = 4.99638 ± 0.00057 Å and c = 5.46119 ± 0.00044 Å.

Key-words: Neutron multiple diffraction, β-quartz, hexagonal cell parameters.

INTRODUCTION

The main purpose of this work is to develop a method for the determination of the parameters *a* and *c* of the β-quartz hexagonal cell at 1003 K, using neutron multiple diffraction (NMD) data. To attain this purpose, the authors used a 00.1 β-quartz NMD Umweg pattern measured by Mazzocchi & Parente [(1)] in the IPEN multipurpose neutron diffractometer. Figure 1 shows this pattern. The pattern was measured in an experimental arrangement which included a special collimator in order to obtain NMD patterns with good resolution. Although not mentioned in the work, the pattern was measured after a careful orientation of the crystal in the neutron beam, in order to obtain the primary reflection.

This work deals first with a selection of the experimental peaks according to their intrinsic qualities: shape, positions and signal-to-noise ratio and with an unambiguous identification of the secondary reflections, responsible for the formation of peaks in the experimental pattern. Identification is followed by a selection and a classification of the peaks, according to how sensitive are their angular azimuthal positions (ϕ) to the variation of *a* and *c* cell parameters. Such a classification has permitted the employment of an iterative process in the determination of the cell parameters.

EXPERIMENTAL

At approximately 846 K, α-quartz, one of the polimorphs of silica (SiO_2), undergoes a reversible transition to β-quartz. In this high-temperature structural transition, the structure of quartz changes from a trigonal to a hexagonal symmetry. The hexagonal β-phase remains unaltered till approximately 1143 K [(2)].

To obtain the 00.1 β-quartz NMD pattern, Mazzocchi & Parente [(1)] used a natural quartz crystal shaped into a cylinder of size 5 cm diameter x 5 cm height with the [00.1] crystalline direction