Short Communications
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Observations on the phase transition in quartz by synchrotron-radiation X-ray topography. By A. ZARCA, Laboratoire de Minéralogie-Cristallographie, Université P. et M. Curie, 4 place Jussieu, 75230 Paris CEDEX 05, France

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Abstract

By use of a high-temperature camera designed for \textit{in situ} synchrotron-radiation X-ray topography the \( \alpha - \beta \) transition was investigated in a Z-cut synthetic-quartz sample. Preliminary dynamical observations on movement of the boundaries between \( \alpha \) and \( \beta \) phases and \( \alpha_1 \) and \( \alpha_2 \) domains are reported.

Quartz undergoes a phase transition at about 846 K. The low-temperature phase has symmetry 32 and the high-temperature \( \beta \) phase presents symmetry 622 (Frondel, 1945). In the Dauphiné twin the part \( \alpha_1 \) is rotated by 180° about the \( c \) axis with respect to \( \alpha_2 \). Different phases (\( \alpha - \beta \)) and twinned regions (\( \alpha_1 - \alpha_2 \)) satisfy simultaneously the Bragg condition for all lattice planes. However, there are differences in atomic positions in the unit cell, so that the respective structure factors differ for some reflections (Young, 1962). For example, the structure factors \( F_{3031} = 25 \) and \( F_{3031} = 1.5 \) differ for the \( \alpha_1 \) and \( \alpha_2 \) of a twinned sample at low temperature while \( F_{3031} = F_{3031} = 15 \) in the \( \beta \) phase.

By taking X-ray topographs of 3031 or 3031 reflections, \( \alpha \) and \( \beta \) phases at the transition temperature, or \( \alpha_1 \) and \( \alpha_2 \) twinned regions at low temperatures, can be identified by differences in intensity (Lang, 1965). Therefore, high-temperature X-ray topographic studies can give direct information on phase transition and twin formation (Inoue, Iida & Kohra, 1974; Kume & Kato, 1974).

By use of the UHV (ultra high vacuum) system designed by Gastaldi, Jourdan, Marzo, Allasia & Jullien (1982) for \textit{in situ} synchrotron-radiation X-ray topography, associated with a TV camera and a video recording system (Sauvage & Petroff, 1980), it is now possible to follow directly the \( \alpha - \beta \) transition and the \( \alpha_1 - \alpha_2 \) twinning in quartz samples.

Fig. 1. X-ray transmission topograph of the Z-cut quartz sample obtained with a conventional X-ray tube. Ilford L4 nuclear plate. 110 reflexion. Mo K\(_\alpha\). Room temperature. Two growth sectors \( Z \) and \( X \) separated by a growth-sector boundary \( H \) are present. Note the dislocations \( D \) in zone \( Z \) and the growth bands \( G \) in zone \( X \).

Fig. 2. Synchrotron-radiation X-ray topograph of the same sample obtained after the third cycle. 3031 reflexion. Kodak R-type film. Exposure time: 20 s. Temperature ca 833 K. The \( \alpha_1 \) and \( \alpha_2 \) domains have already appeared and developed. The topograph corresponds to the final stage at a temperature well below the transition point. Note an \( \alpha_1 \) domain located around the crack \( L \) and the needle-like structures of the twin boundaries. The \( A \) area at bottom right is an artefact due to the superposition of another weak diffraction spot.
For preliminary investigations, a Z-cut plate of 10 x 10 x 1 mm was cut from a synthetic bar (Zarka, Liu Lin & Buisson, 1981) in a zone where lattice defects like dislocations and growth bands were present. This was done in order to observe possible interactions of the phase and domain boundaries with these defects. In Fig. 1 we present a conventional X-ray topograph of this sample, obtained before the transition study. We can note at D the dislocations located in the Z zone, at H the growth-sector boundary between the Z and X regions and at G the growth bands in the X zone. The sample was mounted on the sample holder of the heating unit of the UHV system and the white-beam topographic technique (Tuomi, Naukkarinen & Rabe, 1974) was used. After indexation of the Laue pattern the first two cycles through the transition temperature were followed on the 2111 diffraction spot with the TV camera, whereas the third and fourth cycles were recorded with the 3011 spot. Thus it was possible to follow continuously the features associated with the transition during the heating or the cooling of the sample by direct viewing on the TV monitor and storage on a video-tape recorder. Some static topographs were also recorded on Kodak R-type film.

The preliminary results have shown the following.
(i) The α-β phase boundary movement was observed first on the 2131 diffraction spot (during the heating of the sample through the transition point). Its velocity was about 0.5 cm min^{-1} for a temperature variation of about 1 K min^{-1}. Important deformations (Dolino & Bachheimer, 1982) in the whole sample were detected during the transition and during the second cycle a crack was even seen to propagate in the crystal. However, no evidence for the intermediary zone between α and β phases, already observed by some authors (Van Tendeloo, Van Landuyt & Amelinckx, 1976; Dolino, 1979), has been obtained yet.
(ii) The development of the α1 and α2 domains from the β phase and the reverse process were clearly detected on the 3031 diffraction spot during the third and fourth cycles. We present in Fig. 2 the final configuration of these domains obtained after the third cycle. The different stages of the α1-α2 domain development from the β phase are shown in Fig. 3. These α1 and α2 domains appear first in the lower part of the sample (below the crack L) and in a second stage in the upper part of the crystal. The whole transformation process takes place over 4 min for a temperature-variation rate of about 1 K min^{-1}.
(iii) Contrary to other observations (Inoue et al., 1974), quite large α1 and α2 domains are observed in this Z-cut sample. Moreover, they present boundaries with needle-like structures (Fig. 2) like those already observed in an X-cut sample (Inoue et al., 1974).
(iv) The α1-α2 domains present different configurations after each of the four cycles. Between the first cycle (Fig. 4a) and the other three (Figs. 4b, c, d) the crack L has taken place and, further on, a particular α1 domain has developed around this crack. However, the development of the twinning is not exactly reproducible although the different configurations are very similar (Figs. 4a, b, c, d).
(v) The role of the dislocations during the transition is not yet clearly established. However, the strains associated with the planar defects (the growth-sector boundary H and the growth bands G) seem to play an important role. We can note (Figs. 4a, b, c, d) some α1 and α2 domains on these defects after the different cycles. More generally, local strains (growth bands) or free surfaces (cracks) seem to have a
significant influence in the development of the different phases (α→β) and the movement of the phase boundary during the temperature transition.

This report shows that the UHV camera associated with the direct observation system (TV camera and video-tape recorder) is an excellent technique to study details concerning the α→β transition in quartz and can also be used for other phase-transition studies. Further dynamical studies of the α→β transition in quartz are at present in development.

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References


