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THE OBSERVATION OF REVERSIBLE ELASTIC DAUPHINÉ TWINNING IN α -QUARTZ

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Résumé. — Dans un cristal de quartz une contrainte uniaxiale parallèle à l'axe polaire (X_1) crée des macles du Dauphiné. Des mesures de charges de polarisation ainsi que des observations optiques montrent que les macles disparaissent lors de la suppression de la contrainte mécanique.

Abstract. — Under uniaxial load applied in the direction of the polar X_1 -axis Dauphiné twins are formed in α -quartz. By measuring the net polarization charge of the stressed crystal as well as by direct visualization of the twins it was observed that these twins have a pronounced tendency to disappear on removal of the load.

1. **Introduction.** — In the terminology introduced by Aizu [1, 2] and Newnham [3, 4, 5] α -quartz is a ferrobielastic crystal. Its ferrobielastic domains are called Dauphiné twins. They are related by a 180° rotation about the optical axis resulting in a reversal of sign of the polar X_1 -axis. Atomic displacements involved in the formation of such twins are small and do not require the breaking of Si-O bonds [6].

Secondary twinning may occur only if it results in a decrease of the Gibbs free energy. Denoting the density of the Gibbs free energy in thermodynamic equilibrium in the reference orientation by $G(+)$ and in the corresponding twin orientation by $G(-)$ a necessary condition for twin formation is

$$\Delta G = G(+) - G(-) > 0. \quad (1)$$

For α -quartz according to [2] this condition may be written as

$$\Delta G = -2 s_{14}(T_1 T_4 - T_2 T_4 + 2 T_5 T_6) \quad (2)$$

where T_λ are the stresses in matrix notation ($\lambda = 1 \dots 6$), s_{14} is the pertinent elastic compliance and where it is assumed that the electric field is zero. Under uniaxial stress (only $T_1 \neq 0$) in α -quartz $G(+)$ equals $G(-)$. Therefore no twinning should occur under these conditions. However in compression experiments under uniaxial load as well as in force transducers employing X_1 -cut plates twinning is observed at mean stresses ranging from 0.5 to 0.9 GPa at room temperature. This observation is not surprising in view of the fact that even with uniaxial application of the load

it is practically impossible to produce truly uniaxial stress in the crystal specimen.

On the basis of relations (1) and (2) stress orientations relative to the crystallographic axes have been determined [6, 7] in which twinning is strongly favored and others which may actually be used for detwinning. The disappearance of twins after removal of the external load was observed for instance in calcite [8] but has not yet to our knowledge been described in the literature for Dauphiné twins in α -quartz.

2. **Experiment.** — Figure 1 shows schematically the experimental arrangement used in this investigation. The specimen Q is located between two steel pistons P_1 , P_2 with carefully machined and ground end faces. They serve as electrodes for the measurement of the polarization charge. The normal force (created by a hydraulic press) applied through them to the specimen is measured by a calibrated piezoelectric force transducer FT. Its response is recorded along the X -axis of an XY -recorder while the polarization charge of the specimen is plotted along the Y -direction after preamplification by means of charge amplifiers. Since the electrodes are identical with the pistons applying the load to the specimen such a plot may be regarded as representing the electrical flux density D_1 as a function of the average mechanical stress T_1 .

Synthetic right-handed α -quartz specimens were used. Their geometry and the orientation of crystallographic axes are illustrated in figure 2. Their side faces were polished optically flat.

FIG. 1. — Experimental set-up (schematic).

FIG. 2. — Specimen geometry: Specimen dimensions are $10 \times 10 \times 5 \text{ mm}^3$.

3. **Results and discussion.** — A typical plot of D_1 vs T_1 is reproduced in figure 3. Up to point A the curve is a straight line and follows the equation

$$D_1 = d_{11}^*(0) T_1 \quad (3)$$

where d_{11}^* is the effective piezoelectric coefficient at $T_1 = 0$. It differs from d_{11} due to the fact that the lateral expansion of the specimen is partly impeded by the pressing pistons. Point A marks the onset of twinning. The reorientation of the twin region under stress implies a reversal of its polarization. As a consequence the net polarization charge is reduced. The domain formed at A remains stable upon further

FIG. 3. — D_1 as a function of average stress T_1 for a full loading cycle resulting in elastic twin escape.

increase of T_1 between points B and C. In this region the piezoelectric polarization again increases linearly with the stress. The slope of BC however is smaller than that of OA reflecting the reduced effective piezoelectric coefficient. At point C another twin domain is formed reducing further the effective piezoelectric coefficient to a value $d_{11}^*(D)$.

With reduction of the load the piezoelectric polarization first diminishes linearly with T_1 , the slope of the recorder trace being determined by $d_{11}^*(D)$. At point E however, a new effect is observed. The effective piezoelectric coefficient increases again and at point F finally reaches the original value $d_{11}^*(0)$. We interpret this as signalling the disappearance of the previously formed twin domains. Below point F the specimen behaves in the same way as in the original untwinned state. At renewed loading the original slope corresponding to $d_{11}^*(0)$ is observed. Twins are formed again at roughly the same load as in figure 3. The details of the measured curves between points A and D may vary from run to run. The main features are closely reproducible. In one instance a crystal slab was subjected to more than 50 loading cycles each one leading to twinning without resulting in a permanent decrease of $d_{11}^*(0)$.

Points E and F shift to slightly lower stress values if several loading cycles (each of a few minutes duration) are performed in rapid sequence. If on the other hand the maximum stress is maintained for periods from one to three hours (extending section CD to C'D' involving the growth of the existing twins or additional twinning) the shape of the return trace is altered. A curve of this type is shown schematically in figure 4. (A direct recording would show additional changes which are due solely to charge leakage during the

FIG. 4. — Schematic representation of D_1 vs T_1 for a loading cycle during which maximum load is maintained for several hours resulting in the formation of permanent twins.

holding period and thus would lead to erroneous conclusions.) The transition region $E'F'$ extends over a broader range of T_1 values and point E' therefore usually is difficult to localize. Point F' then falls below the upward trace. This indicates that in this case not all the twin domains formed between A' and D' disappear upon removing the load. Indeed a subsequent measurement then shows a reduced value of $d_{11}^*(0)$ and the presence of permanent Dauphiné twins may be revealed by etching the surfaces perpendicular to the X_1 direction.

As an independent check of our conclusions we have observed the formation of twins directly by an optical method (based on the schlieren technique) which will be described in detail elsewhere. These observations were carried out simultaneously with the

recording of the piezoelectric response of the specimen. It was thereby confirmed that twins did form exactly at points A and C in figure 3. The twin domains form either at a surface in contact with the pressing piston or in highly stressed regions where previously some surface chips had cracked off. The domains usually first grow in the X_1 -direction. They extend throughout the crystal thickness only when the boundary starts to move laterally in the X_3 -direction. This boundary propagation appears to proceed as a sequence of small steps. Overall the average propagation speed does not seem to exceed the order of mm/s.

The complete absence of previously formed twins is clearly seen during the following loading cycles where twin formation occurs in an almost identical manner. As a rule twins form in the same regions and their boundaries reach the same positions if the final load is kept at the same value in every cycle.

It should be emphasized that the effect of elastic twin escape is not a consequence of the specific specimen geometry described above. The same features as those shown in figures 3 and 4 were also observed in experiments using circular geometry (discs and rings). An optical confirmation using our method was not possible with this geometry.

According to (2) the occurrence of twinning implies the presence of T_4 and/or of T_5 and T_6 . This is not unlikely considering the differences in mechanical boundary conditions and the elastic properties of the pressing pistons and the anisotropic crystal specimen. If local slipping or plastic deformation of piston and specimen faces should occur it would be conceivable that certain stress components at the same value of T_1 are different at increasing and decreasing load such that at decreasing load detwinning is favored energetically.

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