UNIVERSIDADE DE SÃO PAULO

INSTITUTO DE FÍSICA
CAIXA POSTAL 20516
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IRRATIONAL TWINS IN α - $NiSO_4.6H_2O$ CRYSTALS

by

V. Stojanoff and C.A. Pimentel

Instituto de Física, Universidade de São Paulo,
São Paulo, Brasil

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IRRATIONAL TWINS IN α - $NiSO_4.6H_2O$ CRYSTALS*

V.Stojanoff and C.A.Pimentel
Instituto de Física, Universidade de São Paulo, Caixa Postal 20516,
05508 São Paulo, S.P., Brasil

α - $NiSO_4.6H_2O$ crystals were grown from a monocrystalline seed in a saturated solution. X-ray studies has revealed the presence of twins. A possible origin for this twinning is discussed.

α - $NiSO_4.6H_2O$ crystals were grown for acoustic measurements [1,2]. The samples were analysed by X-ray methods and some of them have shown a particular kind of defect. A discussion about this defect is undertaken and we propose a model for their formation.

The tetragonal crystals of α - $NiSO_4.6H_2O$ were grown from a monocrystalline seed suspended in a saturated solution of $NiSO_4$ maintained at (38 ± 1) ^oC [3]. This method has permited to grow large crystals of approximately 2cm³ weighting about 4 g; the α - phase has been verified by the Debye-Scherrer technique. The samples have the form of a four faced topless pyramid presenting a quadratic top - face perpendicular to the direction of growth; this direction corresponds to the cleavage plane (001), as verified by Laue back-reflection photographs.

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The Laue back-reflection method not only allowed the orientation of the crystals but permited to verify their quality. The spots in the Laue diagram were sharp and very definite, typical of crystals of high cristallographic quality. However, about 80% of the samples have presented double Laue sharp spots of nearly equal intensities, distributed preferentially in the diagram (figure 1). In order to study this preferential distribution, various (001) diagrams were obtained from different regions of the cleavage surface of one typical crystal; we assured that the area reached by the X-ray beam was sufficiently small to justify this study. It was observed that only diagrams from the central region of the cleavage surface showed double spots in nearly all crystallographic directions. The diagrams from the other regions presented double spots only for particular directions, which were not the same if the diagrams were from different regions of the (001) surface. The distribution of the double Laue spots in the diagrams is related to a particular region of the cleavage plane, but it was not possible to establish a correlation between them. We suppose that some kind of twin is present in this crystals, since the observed double spots are typical of twins. The existence of twins in α - $NiSO_4.6H_2O$ was already suspected by Jäger and Schaack [4], but these authors could not identify them.

The lateral faces of the α - $NiSO_4.6H_2O$ crystals grow in <101> direction and form 69^o with the cleavage plane. However, the crystals have shown lateral faces formed not only by {101} planes but by two or more {hkl} planes, which affects the crystal habit, as can be seen in figure 2. The angle θ between the directions of these lateral planes and the cleavage plane varied from 51^o to 57^o. An analysis of these angles has shown that these lateral planes have irrational Miller index, except for θ equal to 52^o, 54^o or 57^o which correspond to planes with entire Miller index. This kind of irregular growth appears generally when the crystal is nearly full grown (figure 2). The correlation between this irregularities and the double Laue spots could be verified by cleaving the crystal exactly at the beginning of irregular growth and making Laue diagrams from both parts.

One of the crystals was cleaved in two crystals A and B, perpendicular to the <001> direction, at the intersection of two planes which form a particular lateral face of the crystal. Crystal A (seed end), approximately 2/3 of the original crystal, has its lateral faces formed by {101} planes, while the lateral faces of crystal B (tang end, approximately 1/3 of the original crystal) corresponded to planes with irrational Miller index. Back-reflection and transmission Laue diagrams were made from the central region of the (001) plane on both sides of crystals A and B. The diagrams of crystal A showed only two double spots; yet a few directions presented double spots in the diagrams of crystal B. As can be seen in figure 2b, the intersection of two planes which form a particular lateral face of the crystal did not occur exactly parallel to the (001) plane, being possible that some part of the irregularity remained in crystal A when the original crystal was cleaved. Nevertheless it is not possible to identify a single twin plane in the original crystal so that eventual formation of micro-twins must also be also considered.

A particular kind of mechanical twins due to plastic deformation, called irrational twins [5], has the twin plane with irrational Miller index. Kassen-Neklyudova [6] discuss different models for the origin and occurence of mechanical twinning; in particular they suggest that irrational twins could be due to some internal stresses caused by external forces.

We suppose that the weight of the α-$NiSO_4.6H_2O$ crystals (~ 4 g)can act like a traction force inducing internal

stresses in the crystal; this plastic deformation would be responsible form mechanical twinning. The traction force would account for the existence of four slip planes (equivalent or not), like the model shown in figure 3, which would create the observed breakdown in the lateral faces. We consider that the so formed lateral planes correspond to lateral faces of two or more superposed and malajusted crystals, responsible for the preferential distribution of the double spots in the Laue diagrams.

We concluded that large α-$NiSO_4.6H_2O$ crystals of approximately 2 cm^3, grown from a seed in a saturated solution, show very frequently irrational twins desoriented about some degrees. We propose that these twins are originated by plastic deformation due to crystal weight. This defect may influence some properties that depend strongly on crystallographic directions and experimental measurements [4] that take into account the whole crystal.

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FIGURE CAPTIONS

FIGURE 1 - Laue back-reflection diagram from central region of the (001) cleavage plane of a α- $NiSO_4.6H_2O$ crystal.

FIGURE 2 - α-$NiSO_4.6H_2O$ crystal showing the breakdown in lateral faces: a) fotography; b) diagram of sections to evidenciate irregular growth of lateral faces (planes with irrational Miller index (*)).

FIGURE 3 - Model of formation of irrational twins in α-$NiSO_4.6H_2O$ crystals due to internal stresses ((*) slip planes).

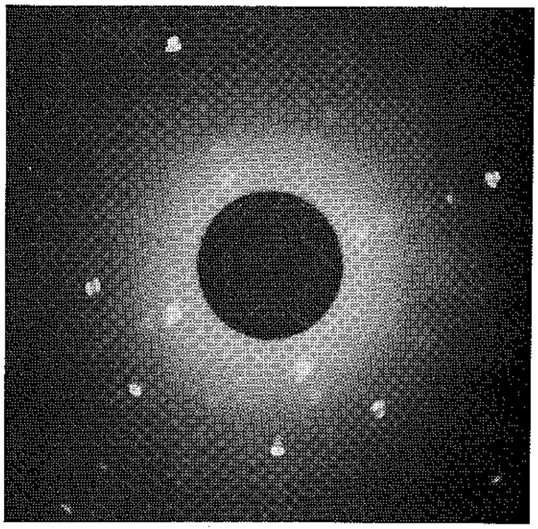
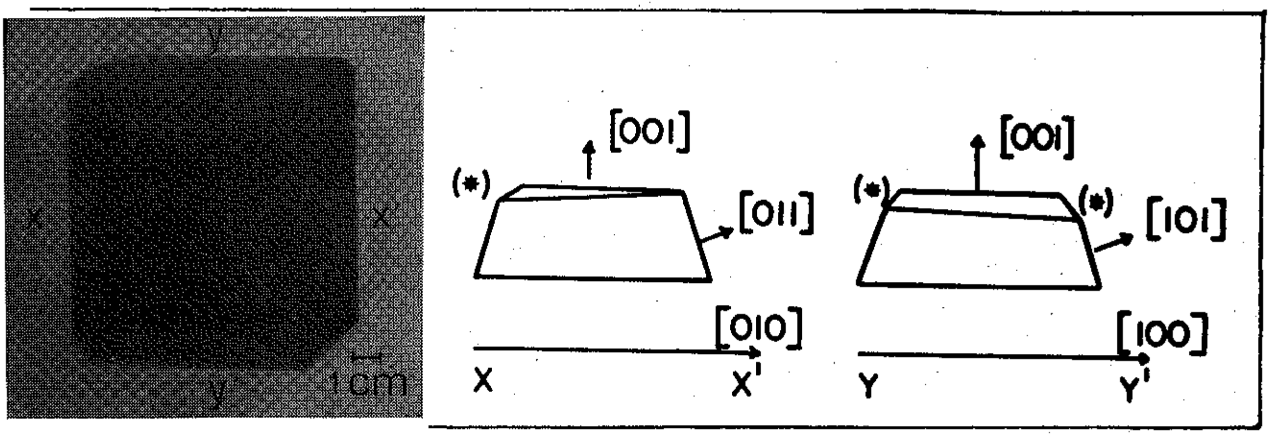


Fig. 1:



(a) (b)

Fig. 2:

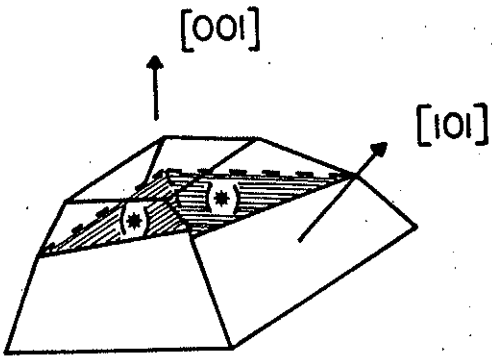


Fig. 3: