

DEFECT STRUCTURE OF CaF_2 CRYSTALS

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Calcium fluoride crystals grown by EFG method were studied. The characteristics of the defect structure are analysed on the basis of high resolution microscope images. The crystals have been grown under different conditions - various pulling speeds. Structural defects occurring in these crystals have been studied (especially the dislocations distribution) by selective etching and optical microscopy. The quality of the crystals has been investigated by examining the dislocation distribution upon the $[111]$ cleavage surface.

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The study of CaF_2 crystal growth and properties are important for fundamental understanding of the growth process and for applications.

This short communication aims to give a contribution to the understanding of the crystal growth process by investigating the defect states in the calcium fluoride crystal grown by EFG method under various conditions.

The presence of lattice defects influences many of the crystal properties, especially its mechanical strength and some optical properties [1]. Precipitation of impurities and accumulation of space charges are likely to occur at defect centers causing trapping of light.

For a pulling rate of ≤ 0.5 mm / min the crystals are clear, colorless and have good cleavage planes. For higher growth rate dislocations appear. If the growth takes place in 10^{-3} Torr vacuum or less, the crystals take white colour. Generally speaking, the external crystal surfaces are not flat, at high pulling rates, ridges are observed both along the pulling direction and perpendicularly to it.

The quality of the crystals was studied by examining the dislocation distribution. The crystals were cut into slices upon the cleavage surface $[111]$ and were subjected to etching in aqueous solution using HCl (with different concentrations between 1N and 8N) [2, 3]. Experiments were carried out on the same sample for different periods of etching at different temperatures (between 40°C and 50°C using a temperature controlled water bath for maintaining a constant temperature. The samples were examined under an optical microscope. The dislocation distribution of the crystals for a given cross - section in radial direction has been examined. The formation, multiplication and high mobility of the dislocation in ionic crystals lead not only to high densities of individual dislocations, but also cause arrangements of dislocations into well developed grain subboundaries. These are stable and nearly immobile in contrast to individual dislocations.

Fig. 1 illustrates the dislocations in the central part in CaF_2 crystals grown at a rate of 2 mm/min. Fig. 2 illustrates the cross section of the crystals with individual etch pits which appears especially in crystals grown with a rate of 0.5 mm/min. The density of etch pits is low and fairly uniform through the volume of crystals grown with 0.5 mm / min [4].

Fig. 2 shows the characteristic etch pits produced in a crystal 5 mm diameter rod grown at a rate of 0.5 mm/min. The density of revealed etch pits is independent of exposure time to the etching. The etch pits density changes along the sample and also for a given cross -section in radial direction.

Fig. 3 shows the characteristic etch pits produced in a crystal rod having 5 mm in diameter grown at a growth rate of 2 mm/min. The density of the revealed etch pits is independent of exposure time to the etching. The dislocation density changes along the sample and also for a given cross - section in radial direction. The dislocations can be arranged in subboundaries in CaF_2 crystals grown with a rate of 2 mm/min.

In Fig. 3 and 4 the density of etch pits is as follows: ① $1.088 \cdot 10^5 \text{ cm}^{-2}$; ② $1.32 \cdot 10^5 \text{ cm}^{-2}$ ③ $1.56 \cdot 10^5 \text{ cm}^{-2}$; ④ $1.344 \cdot 10^5 \text{ cm}^{-2}$ ⑤ $0.8 \cdot 10^5 \text{ cm}^{-2}$. [4]

The density of pits and subboundaries is low and fairly uniform through the volume of the crystal grown with the rate of 0.5 mm / min. There are regions in the crystals with individual dislocation that appears especially in crystals grown with a rate of 0.5 mm/min.

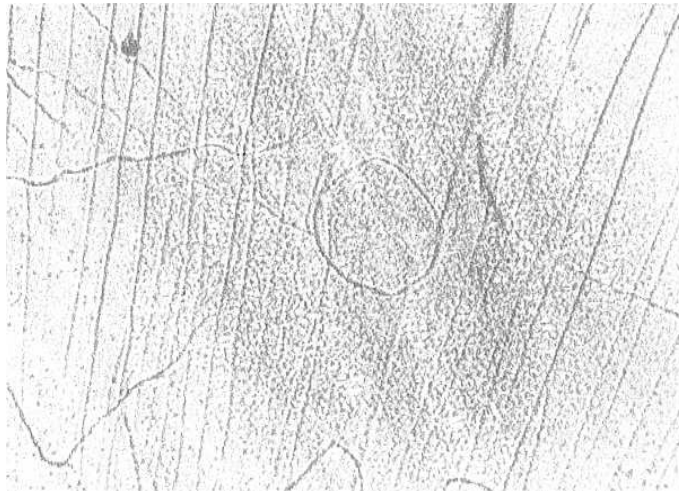


Fig. 1. Dislocations in the central part in CaF_2 crystals grown with a rate of 2 mm/min [4].

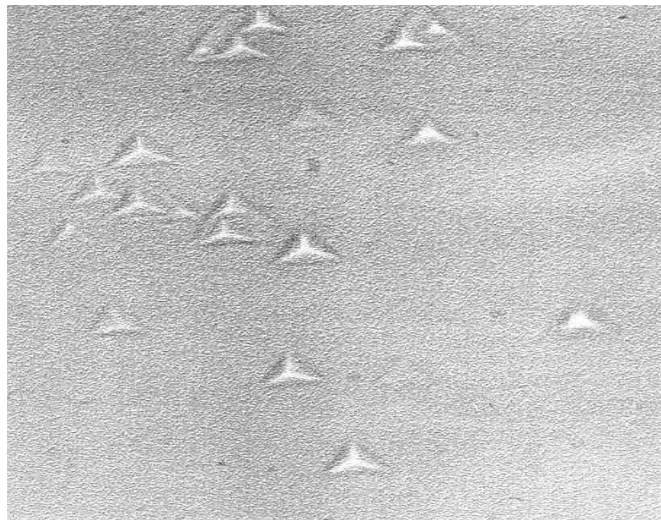


Fig. 2. Cross section of the CaF_2 crystals with individual dislocations that appear especially at a growth rate of 0.5 mm/min [4].

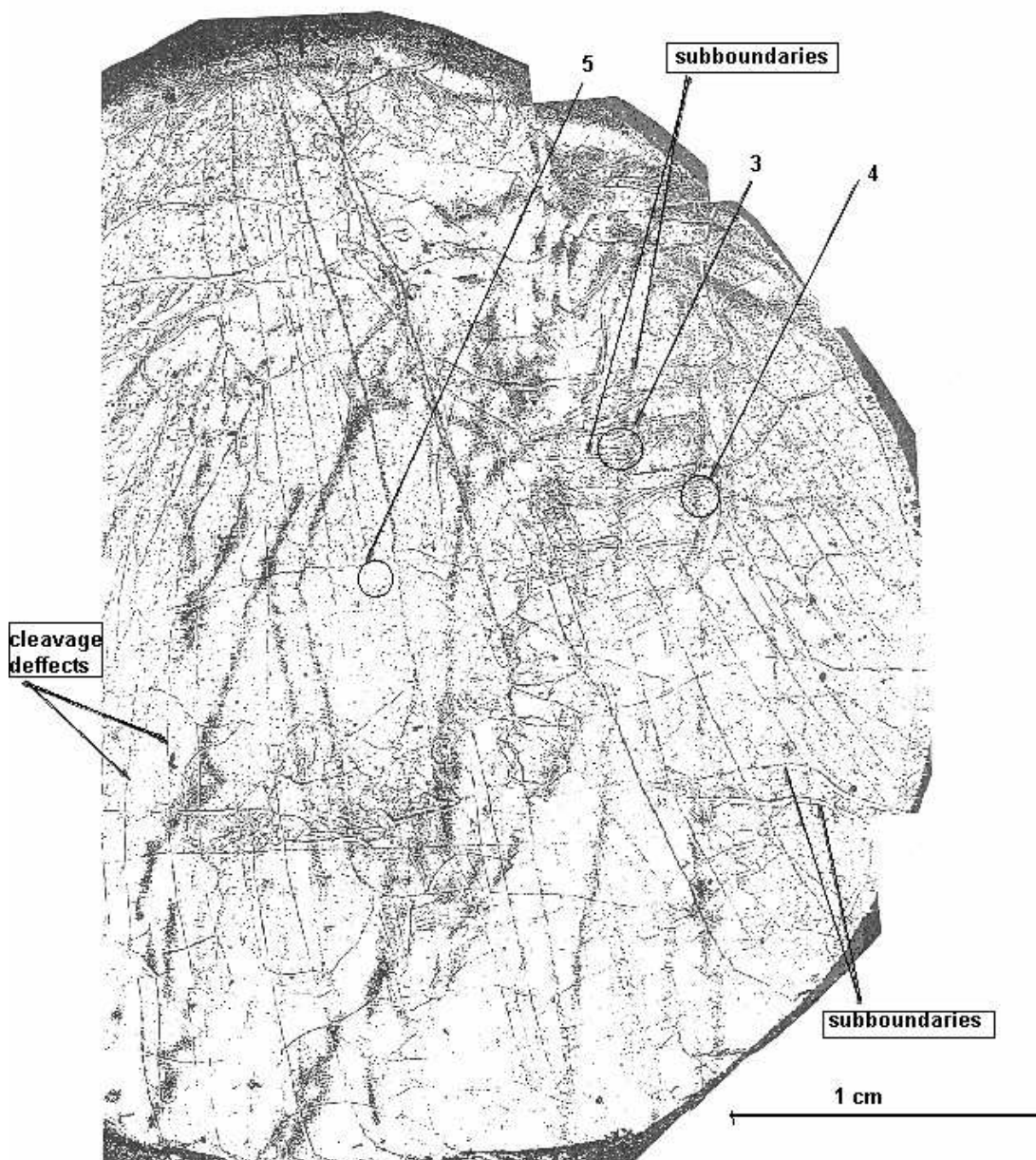


Fig. 3. Characteristic etch pits for CaF_2 crystal grown at a rate of 2 mm/min. In this image the calculated etch pits density is: ③ 1.56^5 cm^{-2} ; ④ $1.344 \cdot 10^5 \text{ cm}^{-2}$; ⑤ $0.8 \cdot 10^5 \text{ cm}^{-2}$. Figs. 3 and 4 give together the full image of the crystal cleavage surface.

4. Conclusions

Comparing the distribution of etch pits for the cases considered in this report it can be concluded that the following characteristics are typical:

1. the etch pits distribution exhibits high value when the growth rate is high;
2. high values of etch pit density are present near the central and marginal part of the crystal;
3. low values of etch pit density are present at $1 \div 2 \text{ mm}$ from the marginal part of the crystal.

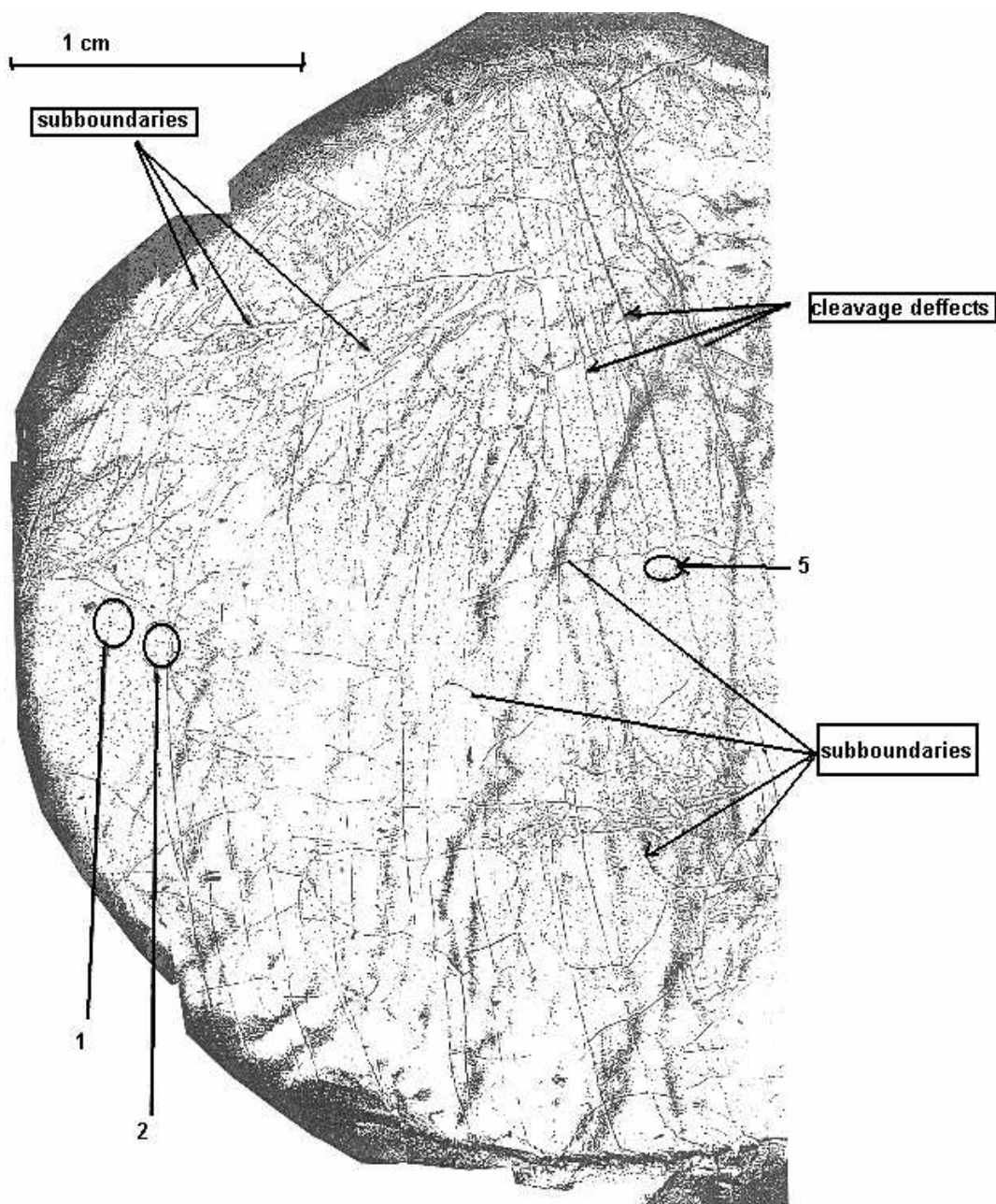


Fig. 4. Characteristics etch pits CaF_2 crystal grown at a rate of 2 mm/min. In this figure etch pits density calculated was: ① $1.088 \cdot 10^5 \text{ cm}^{-2}$; ② $1.32 \cdot 10^5 \text{ cm}^{-2}$; ⑤ $0.8 \cdot 10^5 \text{ cm}^{-2}$. Figs. 3 and 4 give together the full image of the cleavage surface of the crystal.

References

- [1] K. Sangwal, Etching of Crystals, Theory, Experiment and Applications, North Holland, 1987.
- [2] I. Nicoara, D. Nicoara, O. F. G. Aczel, Crystal Research and Technology, **22**, 1139-1144 (1987).
- [3] D. Nicoara, I. Nicoara, Journal of Crystal Growth, **82**, 95 (1987).
- [4] M. Nicolov, Ph. D Thesis, National Institute for Physics of Materials, Bucharest, 1999.