ON THE IDENTIFICATION OF ETCH FIGURES ON TRACKS FROM FISSION FRAGMENTS IN IONIC CRYSTALS

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ON THE IDENTIFICATION OF ETCH FIGURES ON TRACKS FROM FISSION FRAGMENTS IN IONIC CRYSTALS

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I. In studying the structure and physical properties of defects in crystals of radiation origin, in particular tracks arising from fission fragments, the problem of identifying these defects is of fundamental interest. As applied to the technique of chemical etching, such a problem arises in the study of tracks from fragments of spontaneous or induced fission of uranium nuclei in those natural crystals where, as a rule, the track density is negligibly small in comparison with the dislocation density. This problem becomes all the more difficult because, by external features, etch figures on tracks and on dislocations are practically indistinguishable.

In the present article we set forth certain considerations and the results of experiments carried out with the aim of establishing distinctive features by which, using the etching technique, tracks can be distinguished from dislocations.

Crystals of NaCl, KCl, LiF, and $CaCO_3$ were chosen as the objects of investigation; for these crystals the problem of track identification had previously practically not been considered,* although it is of considerable scientific interest and is important from an applied point of view.

II. In undertaking the experiments, we were guided by the following ideas concerning the physical characteristics of a track.

1. In contrast to a dislocation, a track may terminate at an arbitrary point of the crystal.
2. On the basis of general ideas about the structure of a track and of known experimental facts obtained in experiments with micas (^{4}), one may expect that tracks in the crystals investigated differ from dislocations by lower thermal stability, and should anneal at lower temperatures and in shorter times.
3. With respect to the investigated surface of the crystals, a track may be oriented at an arbitrary angle, in contrast to dislocations, which, being

oriented at a small angle to the crystal surface, will leave it; and thus only dislocations oriented at an angle exceeding some limiting value will remain in the crystal.

III. In order to be certain that the specimens we studied, along with dislocations, also certainly contained tracks, in all experiments carried out with the purpose of artificially producing tracks the natural cleavage surface of the crystals was bombarded with fission fragments from a Cm^{244} source.

Two methods were used for studying etch figures. The first consisted in comparing the etch figures on two surfaces of mirror cleavages, one of which, after cleavage, was subjected to irradiation. The second method consisted in successive double etching of one–

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* Separate information on the etching of LiF crystals is contained in (^{1}), and on $CaCO_3$ crystals and certain other crystals in (^{2,3}).

of one and the same area of the surface; between etchings, this area was subjected to irradiation. Both of these procedures made it possible to distinguish unambiguously the etch figures on dislocations from the etch figures on artificially produced tracks (Fig. 1).

In accordance with the three expected distinguishing features of tracks described above, we carried out three series of experiments.

Series 1. The surfaces of all the crystals studied were subjected to repeated polishing with a solution that was at the same time an etchant. The following solutions were used: for LiF crystals, a saturated solution of $FeCl_3$ in water; for NaCl and KCl, glacial acetic acid saturated with zinc ions; for $CaCO_3$, 30% citric acid.

The main result of this series of experiments is that, as the surface layer was polished away, the etch figures belonging to tracks successively became flat-bottomed, whereas the figures belonging to dislocations retained a pointed shape (Fig. 2). This circumstance unambiguously indicates the correctness of the earlier assumption that a track is a defect that terminates not at some surface but within the volume of the crystal. After a preliminary calibration of the polishing kinetics, the maximum extents of the tracks were determined in the experiments described. These values, which are underestimated because of the partial decrease in the energy of the fragment during its passage through the source layer and through air, proved to be as follows: for LiF, 2.3 μ; for NaCl, 1.9 μ; for KCl, 1.6 μ; for $CaCO_3$, 3.3 μ.

Fig. 4. Diagram of the arrangement of the apices of the pyramids of etch figures on dislocations (circles) and tracks (black dots)

Series 2. The principal experiments on the thermal stability of tracks were performed on LiF single crystals, which were annealed over a wide range of relatively low temperatures (100–300°), at which dislocations are practically not annealed

Fig. 4. Diagram of the arrangement of the apices of the pyramids of etch figures on dislocations (circles) and tracks (black dots).

Figure 1: Fig. 4. Diagram of the arrangement of the apices of the pyramids of etch figures on dislocations (circles) and tracks (black dots).

out, as can be judged from the transformation of a pointed pit into a flat-bottomed one. Flat-bottomed etch figures on individual tracks appeared already at a temperature of $ 100°, *andpracticallyalletchfiguresontracksbecameflat−bottomedatanannealingtemperatureof*300°*for*1*hour*; *atthesametime*, *theetchfiguresondislocationsremaine* crystals.

Series 3. Semiquantitatively, the orientation of a track and a dislocation can be characterized by the position of the apex of the pyramid of the etch figure relative to its square perimeter. From measurements of a large number ($\sim 10^3$) of etch figures on dislocations and tracks, diagrams were constructed showing the position of the pyramid apex. As Fig. 4 indicates, the scatter of points corresponding to etch figures on tracks is substantially greater than on dislocations; in the latter case the points do not extend beyond the limiting circle, whose radius is determined by the limiting angle of orientation of the dislocation with respect to the surface under study (100).

In view of the fact that tracks can form very small angles with the surface of natural cleavage, the etch figures on some of them have not square outlines but the shape of a rectangle.

Fig. 1. Etch figures on mirror-cleavage surfaces of a LiF crystal subjected to twofold etching.
a —unirradiated surface; *b* —surface irradiated by fission fragments

Fig. 2. Etch figures appearing at successive stages of polishing off the surface layer of an irradiated LiF crystal. Thickness of the polished-off layer *l*:
a —0.1 μ; *b* —0.4 μ; *c* —1.0 μ; *d* —2.5 μ; *e* —4.0 μ

Fig. 3. Etch figures on the surface of a LiF crystal subjected to irradiation by fission fragments and annealing.
a —unirradiated surface; *b* —irradiated surface; *c* —surface of the irradiated crystal after annealing at 300° for 1 hour

Taken together, the experiments described above attest to the soundness of the ideas concerning the characteristics of tracks in ionic crystals. These ideas, in particular, may serve as a basis for techniques for determining track density in the crystals studied, which is of considerable interest for various aspects of the problem of geochronology and for determining very small ($\sim 10^{-10}$ wt.%) concentrations of uranium in natural crystals.

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Note: Figure translations are in progress. See original paper for figures.