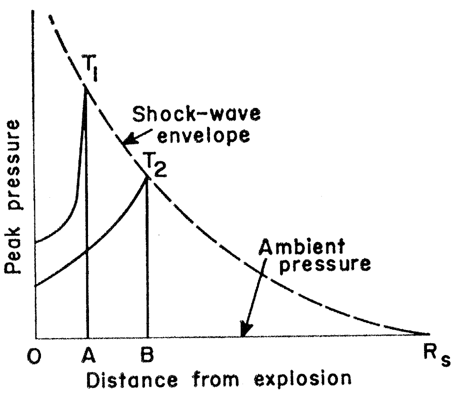

Fig. 5. Diagram of shock-wave propagation (*8*).

and magnitude to reorient the local stress field, thereby creating the second-order shears required to develop the 70° concentrations.

There does not seem to be a relation between the formation of kink-bands and the relation of the direction of the crystallographic axes of the biotite to the direction of shock-wave propagation. Whereas previous workers (*2*) suggested that kink-bands tended to develop preferentially in grains whose [001] axes [that is, normals to (001)] are steeply inclined to the compression axis, the present work indicates no such preference. Rather, in the shock zone of a nuclear explosion, kink-bands in biotite can be formed almost without regard to the crystallographic orientation. One difference in the conditions of the laboratory experiment (*2*) and the nuclear explosion which may account for the indiscriminate development of kink-bands with respect to the crystallographic axes is that the stress produced by the nuclear explosion's shock wave is of such a large magnitude and rapid application that the crystallographic anisotropy of biotite has little influence on kink-band formation.

The mode of failure, under the extremely rapid dynamic loading of a shock wave from a nuclear explosion, may be quite different from the mode of failure under the essentially static loading applied in laboratory experiments. The mode of failure, therefore, may not follow the same preferred crystallographic orientation under shock loading as under static loading.

DAVID CUMMINGS*

U.S. Geological Survey,
Denver, Colorado

References and Notes

1. The radius of the shock zone for the granodiorite, R_s, may be obtained by using the scaling formula $R_s = 75\ W^{1/3}$, where W is the yield of the nuclear explosive in terms of trinitrotoluene equivalents.
2. D. T. Griggs, F. J. Turner, H. C. Heard, *Geol. Soc. Am. Mem.* **79**, 65 (1960).
3. M. S. Paterson and L. E. Weiss, *Nature* **198**, 1046 (1962).
4. J. G. Ramsey, *Geol. Mag.* **99**, 516 (1962).
5. H. E. McKinstry, *Am. J. Sci.* **251**, 401 (1953).
6. J. D. Moody and M. J. Hill, *Bull. Geol. Soc. Am.* **67**, 1207 (1956).
7. "Mathematically, a shock wave is an actual discontinuity propagating with a velocity greater than the local sound velocity," E. H. Freedman and E. F. Green, in *American Institute of Physics Handbook*, D. E. Gray, Ed. (McGraw-Hill, New York, 1957), pp. 2–231.
8. S. Glasstone, Ed., *The Effects of Nuclear Weapons* (U.S. Atomic Energy Commission, Washington, D.C., 1962), p. 104.
9. Work done on behalf of the U.S. Atomic Energy Commission. Publication authorized by the Director, U.S. Geological Survey. I thank the Lawrence Radiation Laboratory for providing the core samples.

* Present address: Astrogeological Branch, U.S. Geological Survey, Flagstaff, Arizona.

8 February 1964

■

Growth Layers on Ammonium Dihydrogen Phosphate

Abstract. *Microscopic observations of growth layers and etch pits on ammonium dihydrogen phosphate crystals reveal screw dislocations on the {100} face generating elliptical spirals that change rapidly but reversibly to rectangular shape when chromium-ion impurity is added. The effects of the impurity on crystal habit are judged to be secondary to changes in the morphology of the growth layers. No sources of growth are observed on the {101} faces; the layers spread inward from the edges and at times are mutually annihilating so that, temporarily, no steps are observed. Similar behavior is recorded for the* $\{10\bar{1}1\}$ *faces of* $NaNO_3$.

Bunn and Emmett (*1*) stimulated interest in the formation of growth layers at the same time that Frank (*2*) suggested that crystal dislocations could provide the sources of steps required for continuous crystal growth. Albon and Dunning (*3*) developed a particularly fine experimental technique for the observation of layer morphology and growth kinetics of sucrose. Their methods have been emulated and extended in our investigation of the mechanism of growth of ammonium dihydrogen phosphate crystals from aqueous solution. In particular, we studied the nature of the deposition process itself, whether by surface nucleation or as initiated at dislocation sites, and as influenced by impurities.

In experiments with crystals that are nucleated more readily than sucrose, the chief difficulty arises from the occurrence of spontaneous growth in tubes connecting the storage vessel to the growth cell. Undue heating of these leads necessarily interferes with precise temperature control in the cell.

The crystals were grown in a thin (3-mm) cell consisting of top and bottom (black) glass plates of optical quality, secured in a gold-plated brass block. Inlet and outlet tubes allowed the circulation of salt solution from an external, carefully lagged, storage thermostat (40°C) by means of a variable peristaltic pump. Housed in a poly-(methyl methacrylate) container, the cell could be rotated about the axis of the microscope tube and given some degree of tilt so that a particular face of a crystal could be brought into position to be strongly illuminated by a focused beam of light. Photomicrographs were taken with a 35-mm single-lens reflex camera and with fine-grain film. For cine-film recording we used a 16-mm camera and reversal film.

Heat was supplied to or withdrawn from the growth cell by means of a Peltier junction attached to the bottom of the cell. Thus the temperature of the cell could be raised or lowered to increase super- or undersaturation as desired. While unintentional temperature fluctuations in the cell block amounted to a few thousandths of a degree Celsius, as measured with a platinum resistance thermometer, it was suspected that fluctuations in the solutions tended to be somewhat greater.

We found that growth on the {100} face of ammonium dihydrogen phosphate crystals proceeds by a screw dislocation mechanism. The spirals are roughly elliptical in shape (eccentricity about 0.86) and oriented with the short axis of the ellipse parallel to the [001] axis of the crystal. Growth proceeds by the movement of steps across the crystal face. The steps are of varying height, visibility being roughly proportional to the step height. Occasionally we found single spirals as in Fig. 1. However, most frequently two cooperating spirals of opposite hand (a Frank-Read source) come together to give closed loops, and the steps spread outward across the face of the crystal; this can be seen in Fig. 2. By counting the steps and noting their position with respect to the edges of the face, the progression of the steps can be followed. The step velocity is of the order of 10^{-3} cm/min.

We invariably found a number of dislocation spirals active on {100} faces, but usually one spiral was dominant; its growth fronts annihilate the fronts of weaker spirals. The indications are that the growth rate is, to a first approximation, independent of the number of dislocation sites and that a group of dislocations is as strong as its most active member.

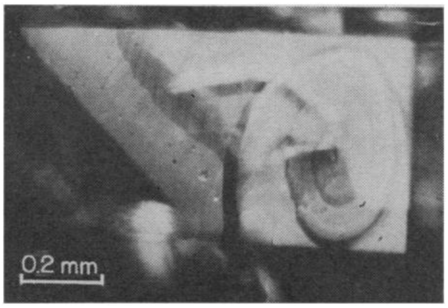

Fig. 1. Single spiral of growth layers, emanating from a screw dislocation, on a {100} face of ammonium dihydrogen phosphate.

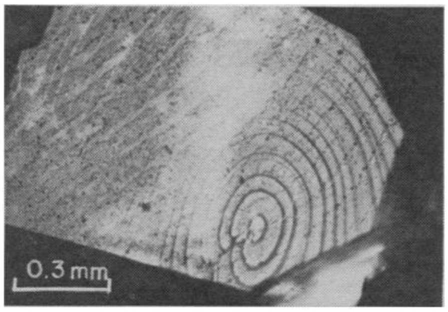

Fig. 2. Screw dislocations of opposite hand (Frank-Read source) generate elliptical growth layers on the {100} face of ammonium dihydrogen phosphate.

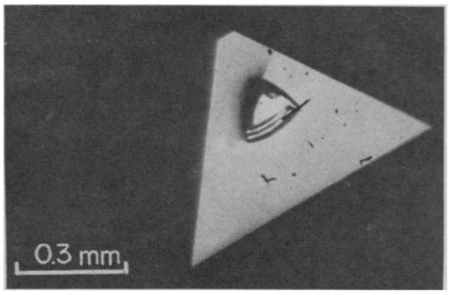

Fig. 3. Growth layers on the {101} face of ammonium dihydrogen phosphate converge from the face edges toward the center; here they are near annihilation.

Growth on the {101} face proceeds by the movement of layers from the edges toward the center of the face. The growth layers become apparent only at some distance from the edge. It may be that we were not able to resolve finer layers that actually start at the edges. This phenomenon is shown in Fig. 3. In accordance with the observations and conclusions of Bunn and Emmett (*1*), it is held that these layers on the {101} face have their origin in the spiral growth on the {100} face. This carries with it the corollary that steps on the {101} face are higher, a feature that we were not in a position to measure. We have not been able to establish exactly what happens to the growth layers on the {100} face when they reach the edge of the face. It is conceivable that the layers distribute themselves along the edges of the {101} face and then proceed to build up layers on that face. We found no evidence of screw dislocation sites on the {101} face of ammonium dihydrogen phosphate, so the layers must have their origin at some other source. The layers move toward the middle of the face from all three edges of the face—that is, also from the edges with the same crystallographic form, where they presumably have not originated. The step height from all edges appears to be the same within our ability to observe. The stepped pit (Fig. 3) onto which the layers converge is often annihilated, leaving momentarily a face without observable steps.

We found a similar but even more striking example of this growth-step morphology when examining the $\{10\bar{1}1\}$ faces of sodium nitrate (Fig. 4). Growth of crystals from the edges and corners is not uncommon. Such growth is often the cause of inclusions of mother liquor. A hollow is formed in the center of the face; this hollow subsequently closes over, trapping some of the liquor and causing opacity in the crystals. If such cavities are not closed over, the final crystals have hollow faces and often there is a stepped structure in each hollow, reminiscent of hopper crystals (*4*).

It is well known that chromium changes the habit of ammonium dihydrogen phosphate crystals (*5*). The predominant prism faces are replaced by curved, tapering faces converging toward the points of emergence of the fourfold axis. The faces are vicinal and commonly marred by distortions. We have now found that chromium also has a profound effect on both the shape and behavior of the growth spirals on the {100} face.

Crystals were grown from a solution containing 0.025 percent chromium (weight-percentage based on the amount of solids in the solution) in the form of chromic chloride. The growth spirals assumed an almost rectangular shape, again with the short dimension of the rectangle parallel with the [001] axis of the crystal and with a ratio of long to short dimension close to 2.0.

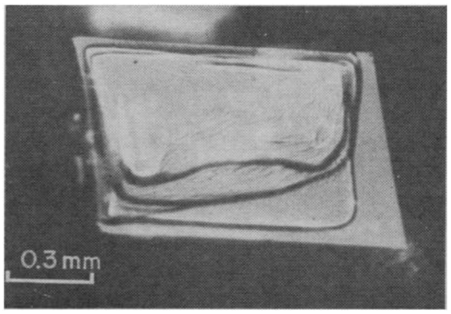

Fig. 4. Converging growth layers on the $\{10\bar{1}1\}$ face of $NaNO_3$.

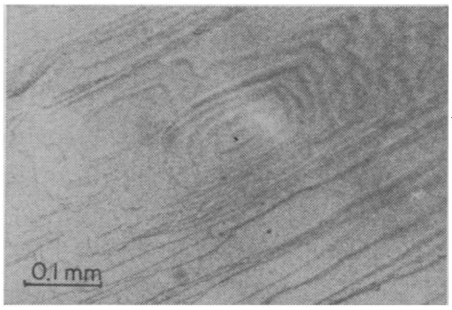

Fig. 5. Rectangular growth spiral (influence of chromic-ion impurity) on the {100} face of ammonium dihydrogen phosphate.

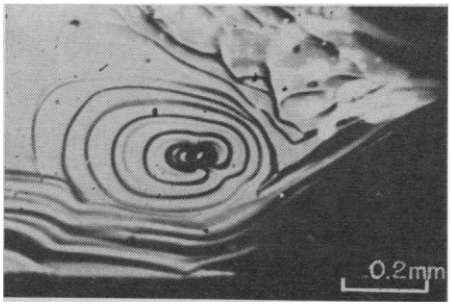

Fig. 6. Dissolution etch pit at a Frank-Read source on the {100} face of ammonium dihydrogen phosphate.

At approximately the same supersaturation, the growth steps moved much faster than they did in crystals grown from pure ammonium dihydrogen phosphate solutions, so much so that it was difficult to photograph the spirals with the light available. As would be expected, the steps appeared to be shallower than those observed under other conditions. Figure 6 shows an enlargement made from 16-mm cine film. At the same supersaturation the number of dislocation sites was markedly increased. The number of single spirals was greater than with crystals grown from pure solution, although some combinations of spirals of opposite hands, forming closed loops, were found. The step heights were more uniform than were those in crystals grown from pure solution, and there was less dominance by one particular spiral. The observation of more dislocation sites in the chromium-contaminated crystals agrees with our observation of higher dislocation densities, as revealed by x-ray diffraction topography in crystal portions contaminated with chromium (*6*).

We also investigated the effect of growing pure ammonium dihydrogen phosphate crystals, observing the typical elliptical growth spirals, and then suddenly introducing a solution contaminated with chromium. This had a startling effect. The surface momentarily became inactive and then was quickly reactivated, with rectangular spirals. This suggests that the habit modification caused by the chromium proceeds by selective adsorption along the one-dimensional growth fronts rather than by adsorption on certain crystal planes, and, further, that the changes in crystal habit are secondary to the changes in the morphology of the growth spirals. If the contaminated solution was replaced by pure solution, the spirals reverted to their original elliptical shape.

By slightly raising the temperature of the cell so that the solution becomes unsaturated, etch pits can be observed and recorded photographically. Etch pits on the {100} face of ammonium dihydrogen phosphate are elliptical in shape, the short axis of the ellipse being parallel to the *c*-axis of the crystal. Therefore, they have a distinct relation to the spiral growth form on that face. Figure 6 illustrates dissolution at a Frank-Read source. The etch pattern is strikingly similar to the growth pattern of Fig. 2. Fewer etch pits are produced on the {101} face; again they have a distinctive pattern resembling that of the growth morphology, being triangular in shape.

JOHN L. TORGESEN
RONALD W. JACKSON*
National Bureau of Standards,
Washington, D.C.

References and Notes

1. C. W. Bunn and H. Emmett, *Discussions Faraday Soc. No. 5,* 119 (1949).
2. F. C. Frank, *ibid.*, p. 48.
3. N. Albon and W. J. Dunning, *Acta Cryst.* **12**, 219 (1959).
4. H. E. Buckley, *Crystal Growth* (Wiley, New York, 1951); R. G. Lang and E. S. Wadja, in *The Art and Science of Growing Crystals* (Wiley, New York, 1963), p. 141.
5. J. Jaffe and B. R. F. Kjellgren, *Discussions Faraday Soc. No. 5,* 319 (1949); J. L. Torgesen and A. T. Horton, *Natl. Bur. Std. U.S. Tech. Note 174,* 5 (1963).
6. R. A. Deslattes *et al.*, in *Advances in X-ray Analysis*, W. T. Muller, Ed. (Plenum Press, New York, in press).
7. Supported by the Advanced Research Projects Agency.

* Present address: 90 Hayes Chase, West Wickham, England.

25 March 1965

"Trinitite": Cobalt-60, Cesium-137, and Europium-152

Abstract. *Cobalt-60, cesium-137, and europium-152 have been identified in "trinitite," an artifical mineral produced in the first nuclear explosion at Alamagordo, New Mexico, in 1945. Cesium-137 is an expected fission product, but the origin of the other two radionuclides is not certain.*

Operation Trinity, the first nuclear detonation, was a 19-kiloton plutonium device set off on a 30-m steel tower at Alamagordo, New Mexico, 16 July 1945 (*1*). The debris found around the site, consisting of fused soil, materials of construction, and parts of the device, has been named "trinitite." Larson and co-workers reported on investigations of plutonium and fission products in "trinitite" and other environmental materials at the test site

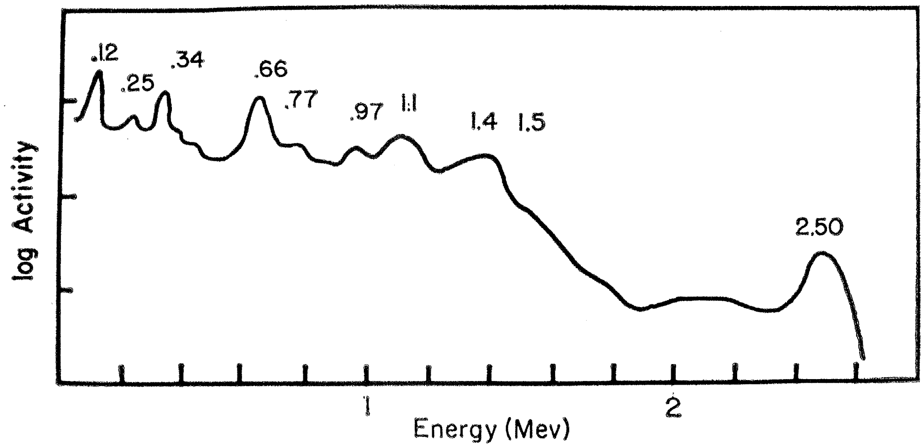

Fig. 1. Gamma-emission spectrum of trinitite.

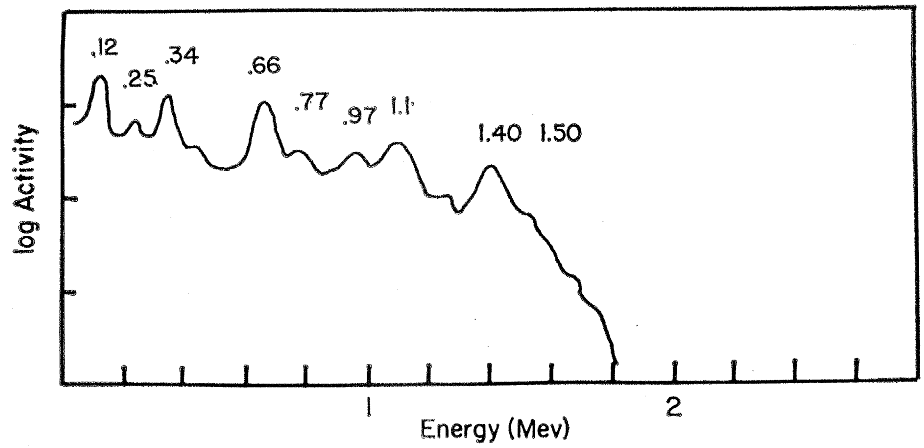

Fig. 2. Gamma-emission spectrum of trinitite with cobalt-60 subtracted.