

tions are not necessary when the purpose is only to produce good crystal faces for other experiments.

With negative crystal growth, surface contamination is automatically avoided, as is the need for elaborate pressure of temperature control. Ideally the surfaces would be the same as those produced by positive crystal growth.

CHARLES A. KNIGHT

NANCY C. KNIGHT

National Center for Atmospheric Research, Boulder, Colorado

References and Notes

1. Z. Yosida *Low Temp. Sci. Ser. A* **12**, 143 (1954); T. Nei, *Nature* **192**, 1177 (1961).
2. H. Kramers and S. Stemerding, *Appl. Sci. Res. Sect. A* **3**, 73 (1951); R. F. Strickland-Constable and E. W. Bruce, *Trans. Inst. Chem. Engrs. (London)* **32**, 192 (1954).
3. K. Higuchi, *Acta Met.* **6**, 636 (1958).
4. D. Kuroiwa and W. L. Hamilton, in *Ice and Snow*, W. D. Kingery, Ed. (M.I.T. Press, Cambridge, 1963), p. 34.
5. U. Nakaya, "Properties of single crystals of ice, revealed by internal melting," *Snow, Ice, Permafrost Res. Estab. Res. Pap.* **13** (1956).
6. The crystals were collected during an expedition from the University of Hokkaido in 1964, led by Prof. A. Higashi, who furnished the crystals used in this work and measured their dislocation density.
7. Work done at the Ice Research Laboratory, Department of Applied Physics, Hokkaido University, Japan and supported partially by NSF and the Japan Society for the Promotion of Science, as part of the Japan-U.S. Cooperative Science Program. Primary support was from the National Center for Atmospheric Research. Criticism and help from A. Higashi, co-researcher on the cooperative program, members of his laboratory, and members of the Institute of Low Temperature Science are gratefully acknowledged.

25 August 1965

Autoradiography: Technique for Drastic Reduction of Exposure Time to Alpha Particles

Abstract. *High-speed, gross, alpha autoradiographs can be made if silver-activated zinc sulfide is used as an intensifier in conjunction with high-speed film. The intensifier is interposed between the sample and film. This technique requires about 1/1000 of the exposure time required with Kodak NTB plates. The gross autoradiographs have greater contrast but slightly less resolution than conventional plates.*

High-speed autoradiographic technique is sought when exposure time is the prime consideration or when the radiation level is too low for conventional methods to be practical. We describe the use of silver-activated ZnS as an intensifier to reduce exposure time for gross alpha autoradiography.

When a 5-Mev alpha particle strikes a nuclear film, probably no more than 0.1 percent of the energy is utilized by the emulsion to produce the useful latent image. Since no more than 90 ev are required to produce a developable silver halide grain (1), and since alpha tracks rarely have more than 50 silver grains, over 99 percent of the alpha energy is ineffective in formation of a latent image. Use of phosphorescent ZnS crystals may reduce the direct effects of alpha particles on the film, but the radiation of light, which is efficient in producing a latent image compensates for the reduction. Thus, when a ZnS-augmented autoradiograph is made by an alpha emitter, the film may not receive the alpha radiation, but will receive a large quantity of photons from the excited ZnS crystals; consequently exposure time to attain a given grain density is reduced.

Silver-activated, microcrystalline ZnS is superior in radiation amplification to other combinations such as copper-activated ZnS or a mixture of ZnS and CdS. The phosphor finally selected was ZnS:Ag P7-920B (2) which is an efficient intensifier; it has desirable characteristics, such as wettability and uniformity of grain size.

About 25 g of phosphor was added to 100 ml of 2 percent gelatin solution. The coarse particles were allowed to settle out from the warmed solution for 1 to 2 minutes. The suspension was then decanted and coated directly onto the mounted specimen or deposited by sedimentation on a double-sided, aluminum-coated Mylar film (3) or thin plastic (such as Saran wrap). A uniform deposition of 12 to 14 mg/cm² was found to be optimum. Although the aluminum-coated Mylar film absorbs more alpha energy than one without backing, the absorption loss was offset by the reflection of light from the aluminum coating. The dried film was sandwiched between the photoemulsion and the specimen (Fig. 1, top) and exposed for a suitable period. By arranging the ZnS:Ag and photographic film on aluminum-coated Mylar film into a single light-tight unit, exposure can be carried out in full light. The ZnS:Ag on an aluminum-coated Mylar film can be made light-tight by taping it to the Polaroid film holder with the ZnS:Ag side adjacent to the photographic film (Fig. 1, bottom). Such a unit can be repeatedly used as long as the ZnS and

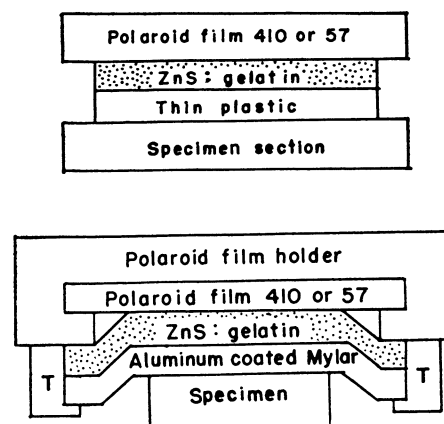


Fig. 1. (Top) Phosphor-film arrangement. (Bottom) Arrangement of a light-tight unit for daylight exposure. T, light-tight tape.

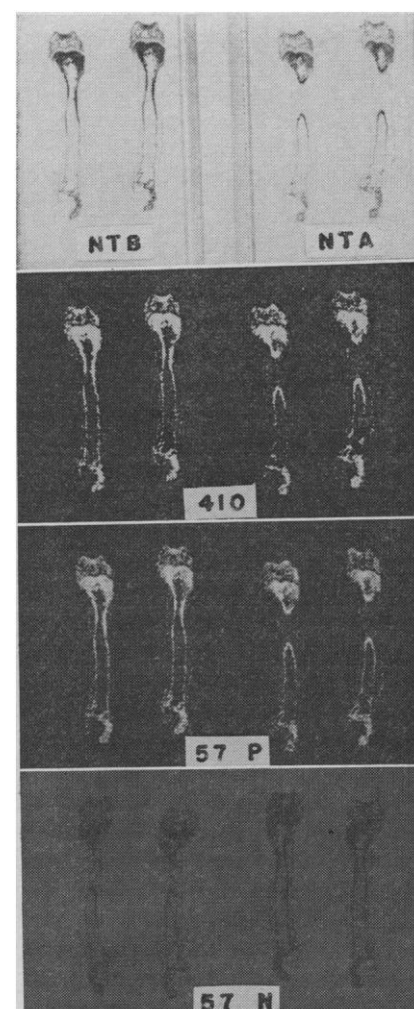


Fig. 2. Rat femur sections with ²³⁹Pu deposition were autoradiographed with NTB and NTA plates by conventional techniques. Then the same femur sections were used in the new technique. The exposure times were: 265 hours for NTB, 361 hours for NTA, 10 minutes for Polaroid Polaroscope 410, 15 minutes for Polaroid Land 57 positive, and 15 minutes for Polaroid Land 57 negative (developer removed with Polaroid coating fluid).

aluminum coatings are in good condition.

High-speed Polaroid Polascope 410 and Polaroid Land type 57 film were chosen for the experiment because of their sensitivity, convenience, and speed of development. Polascope 410 film required about 200 times longer exposure than the same film used with the help of ZnS intensifier.

Figure 2 shows typical autoradiographs obtained with standard NTA or NTB film as compared with autoradiographs of the same material using amplification and photographic film. The specimen was obtained from a rat injected intravenously with ^{239}Pu . Sections of femur 10 microns thick were attached to glass slides with double-coated cellophane tape, and the phosphor (13 mg/cm^2) on aluminum-coated Mylar film was sandwiched between the photographic film and the sections for exposure. The quality of the high-speed gross autoradiographs produced with the aid of ZnS is close to that obtained with standard emulsion, and the exposure time needed is sharply reduced.

Using the same intensifier technique to locate a sliver of plutonium in

excised human skin required an exposure time of less than a minute. Even a trace of plutonium rubbed off as a sliver penetrates would be adequate to identify location if deeper penetration occurred. The very short time required for exposure and the ease of development suggest that this technique might have application during surgical probing to remove imbedded slivers of Pu or other alpha emitters. Likewise, this technique has shown some promise in examining plates of ^{239}Pu prepared from urine samples and used in routine bioassay. Because of its speed and convenience, this new technique of autoradiography may find other areas of application.

JACK J. C. HSIEH

F. P. HUNGATE

S. A. WILSON

Biology Department, Pacific Northwest Laboratory, Battelle Memorial Institute, Richland, Washington

References and Notes

1. C. E. K. Mees, *The Theory of the Photographic Process* (Macmillan, New York, 1954), p. 309.
 2. U.S. Radium Corp.
 3. Alexander Vacuum Co., about 1 mg/cm^2 .
- 18 November 1965

Ice-Rafted Detritus as a Climatic Indicator in Antarctic Deep-Sea Cores

Abstract. *Ice-rafted detritus is readily identified in sediment cores raised from the deep ocean floor around Antarctica. A few cores have reached a depth below which no ice-rafted material is found. This depth is interpreted as indicating the establishment of earliest Pleistocene glaciation in the Southern Hemisphere. It is just below a depth where there is a change in assemblages of Radiolaria which Hays associates with the Pliocene-Pleistocene boundary. The presence of ice-rafted material throughout the upper zone in cores taken south of the Polar Front indicates continuity of glaciation in Antarctica. Further north, near 45°S in the Argentine Basin, zonation of the ice-rafted detritus can be used to delineate glacial stages of the Pleistocene.*

It is well known that there is a wide zone (320 to 960 km wide) of ice-rafted or glacial marine sediments on the sea floor surrounding Antarctica (1). Lizitzin's description (2) of the sediments that cover the continental shelf and slope and extend into the adjacent oceanic areas around Antarctica indicates that most ice-borne sediment is released south of 40° to 50°S . The glacial marine sediments intergrade with a zone of diatom ooze (up to 1300 km wide) whose northern boundary is slightly north of the Antarctic Polar Front. North of the zone of

diatom ooze the pelagic sediments are generally calcareous ooze in depths less than 4100 to 4700 m and red clays in greater depths. Hays has summarized the literature on these sediments (3); the zones shown in Fig. 1 are based on his work (4).

Hays studied cores raised in a program of sediment-coring in high southern latitudes that was undertaken by Lamont Geological Observatory in 1956. The purpose of the program was to compare the history of Pleistocene glaciation in the Southern and Northern Hemispheres.

Several sediment cores from south of the Polar Front show a marked depositional change from red clay to overlying diatom ooze. Hays showed (3) that this change correlates with a horizon marking the extinction of Tertiary Radiolaria and that it is related to the transition in Foraminifera and *Discoaster* which has been studied in other deep-sea cores in connection with the problem of the Pliocene-Pleistocene boundary (4, 5).

The sand fraction from five of the cores used by Hays (Fig. 1, Table 1) has been examined at intervals of 20 to 30 cm down the cores, and the occurrence and abundance of ice-rafted detritus have been estimated (Fig. 2A). Three of the cores (V16-59, V16-116, and V16-132) consist of 5 to 9 m of diatom ooze which overlies red clay. The fourth, V17-88, was taken north of the Polar Front near Tierra del Fuego; it has gray lutite overlying red clay. In three of these cores the boundary between ooze and red clay is gradational, but in the remaining core of diatom ooze (V16-116) this boundary is sharp (Fig. 2A). The fifth core consists of 11 m of foraminiferal ooze and foraminiferal clay. It also was raised from north of the Polar Front.

The diatom oozes contain 2 to 10 percent sand but the red clay generally contains less than 1 percent. The percentage of sand-size material in the core of foraminiferal ooze decreases from 50 percent at the top to 20 percent at the bottom of the core. The sand-size sediment in the oozes consists mainly of tests of radiolarians or foraminiferans mixed with ice-rafted and volcanic detritus. The ice-rafted detritus is easily identified, being poorly sorted, angular, and consisting of quartz, feldspar, garnet, and fragments of granitic, sedimentary, and metamorphic rocks of all sizes, from very fine sand to pebbles as large as the diameter of the coring tube (64 mm). The ice-rafted material is mixed with pumice, lapilli, glass shards, fresh plagioclase, and volcanic rock fragments that presumably are derived from local volcanoes (2).

The amount of ice-rafted sand and pebbles varies from just a few grains per gram of sediment to 50 percent of the total sand fraction. Thin sections of the sand fraction from core V16-132 consist essentially of 20 to 40 percent quartz, 20 to 30 percent potassic feldspar, plagioclase, granitic