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A NEW TECHNIQUE IN THERMOLUMINESCENCE PHOTOGRAPHY

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ABSTRACT

A simple and sensitive method has been developed to produce photographs of thermoluminescence in minerals. Placing an X-irradiated mineral in direct contact with a photographic emulsion forms images with a sensitivity comparable to photomultiplier tubes and with resolution of particles 10 μm in size. Some geological and paleontological applications are also discussed.

INTRODUCTION

Photography, in the usual sense, of thermoluminescing minerals is complicated by the typically low light intensity involved. Although the film may be exposed over the entire time a sample is being heated, physical limitations allow only a small fraction of the light produced to enter the camera. The success of investigations (for example Roach, 1968, p. 591; and Fremlin, 1968, p. 407), utilizing the photograph of strongly thermoluminescent materials, indicates the desirability of a method allowing its extension to low intensity cases. The method described below, although restricted to a smaller temperature range, is quite simple and provides sensitivities comparable to that of a photomultiplier tube.

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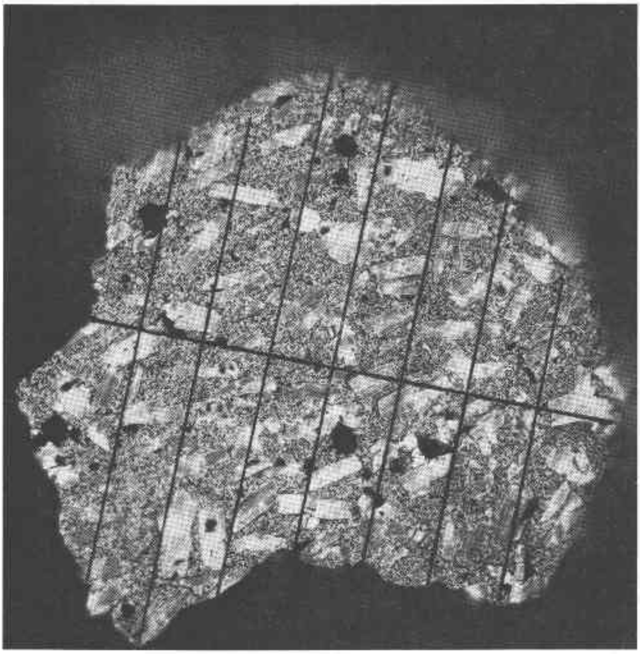

FIG. 1. Image produced by placing an irradiated feldspar porphyry in contact with Polarid ASA 3000 black and white film. The dark grid is the image of a pattern drawn directly on the mineral's surface with India ink. The parallel lines are separated by approximately one centimeter.

After X-ray irradiation a mineral emits light (phosphorescence) which corresponds to the room temperature tail of the thermoluminescence glow curve that would have been obtained in the usual fashion by X-irradiation at liquid nitrogen temperatures followed by warming to room temperature. In nature the radiation dose rate is so low that the light emitted is negligible. However, at the much higher dose rates of artificial irradiation, the light emitted is even visible to the naked eye in some cases.

Surprisingly good photographs were obtained by placing a flat, smooth surface of a freshly irradiated mineral directly in contact with a photographic emulsion. This process produced quite sharp images in which structures about 10 μm in size could be resolved under the microscope. An example is shown in Figure 1. In a survey of more than forty minerals, all exhibited at least a trace of luminescence by this method. The increased sensitivity can be attributed to the film subtending a much larger solid angle than in standard photographic techniques.

After two to five minutes irradiation by 50 kV X rays at currents of two mA (yielding an intensity of approximately 30,000 Roentgens per minute), the sample surface is quickly placed in contact with the film sheet for an exposure time varying from several seconds to several minutes. The intensity of the X-ray beam is more important than its energy since the area of interest is near the mineral's surface, and even the weakest X rays are more than sufficient to excite the electrons.

Polaroid 4×5 film packets in black-and-white (ASA 3000) and color (ASA 75), used in conjunction with a Polaroid 4×5 film holder, proved quite convenient for this method. Polaroid positive/negative film is less sensitive than black-and-white but is preferable when it is desired to view the image through a microscope or measure the variation in luminescence quantitatively with a densitometer. If standard sheet film is used, it should be supported by a smooth, nonreflecting surface.

The method can be modified by performing the process at a temperature above or below room temperature or by chilling the mineral prior to irradiation and then allowing it to warm in contact with the film. The sample could be irradiated by a means other than X-ray, provided sufficient intensities were available.

Results and Possible Applications

The highly carbonaceous fossils of crinoid stems, large barnacles, and turritella produce easily visible orange phosphorescence of manganese centers in calcite. The alkali-halides and alkaline earth halides emit blue of a slightly lower intensity. Other materials examined such as gabbros, granites, volcanics, porphyries, and basalts produce less intense luminescence.

In an effort to identify the components responsible for the very low-intensity thermoluminescence of basalt, a sample was cut to the dimensions of a thin section, except with a thickness of several millimeters for adequate X-ray absorption. A reference grid was drawn on the surface with India ink. After an image was made, the emitting surface was cemented to a glass slide and lapped to thin section thickness. The luminescing areas were then located from the image and identified in the thin section. All of the areas located appeared to be grains of altered feldspar.

In some fossil specimens the differentiation of minerals produces a variation in thermoluminescent properties which makes structures easily visible in the photographic image that were indistinct when viewed in normal light. The resolution available makes this method a possible aid in micropaleontology.

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A VARIABLE-VOLUME SILVER LINER FOR HIGH PRESSURE HYDROTHERMAL CRYSTAL GROWTH

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ABSTRACT

The design and use of a variable-volume silver liner for hydrothermal crystal growth is described.

We wish to report details of the design, construction and use of a variable-volume silver liner for high pressure autoclaves. It has proved especially suitable for hydrothermal growth of crystals such as zinc oxide and potassium zinc phosphate. They are best grown from a basic medium like aqueous potassium hydroxide which is extremely corrosive to steel at elevated temperature. Although the use of noble metal liners or plating in autoclaves is an established practice (Ballman, 1963) it has been attended with serious problems including fabrication, sealing and pressure equalization.

The liner illustrated in Figure 1 was designed for use with the high pressure autoclave shown there and described in the experimental section. Silver bellows segments are formed from dead-soft cup stock by hydraulic pressure applied internally. As the tube expands outward into a die, it is folded longitudinally, forming bellows. Sealing of both tube ends is accomplished with a silver disc flared to conform to the flared end of the tube. A circular