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PHOTOMICROGRAPHIC TECHNIQUE FOR MEASURING GRAIN DENSITIES OF HIGHLY IONIZED PARTICLE TRACKS IN NUCLEAR EMULSIONS

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This report covers a facet of the work authorized during FY 1961 and 1962 by the Bureau of Ships under RDT&E Subproject S-RO07 II 01, Task 0549, titled “Effects of Space Radiation.” Details of this work are found in the U. S. Naval Radiological Defense Laboratory FY 1962 Technical Program as Program D-1, Problem 3, entitled “Emulsion Studies for Evaluating Potential Radiation Hazards from Heavy Cosmic Ray Particles,” the objective of which is to conduct basic dosimetric studies aimed at determining the rate of energy loss (REL) of primary cosmic rays in tissue. Funds to support this work during FY 1963 were provided by the Bureau of Ships on Budget Project 10, Allotment 178/63.

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ABSTRACT

A technique for rapid and accurate measurement of gap lengths of nearly saturated particle tracks in nuclear emulsions is reported. The method consists of obtaining photomicrographs of particle tracks with a superimposed image of a calibrated eyepiece-micrometer disc. The gap lengths are measured to an accuracy of 1/4 micron.
SUMMARY

This report describes a technique developed at NRDL for measuring grain densities of highly ionized particle tracks in nuclear emulsions. The method consists of using a microscope to obtain photomicrographs that yield the desired information.
INTRODUCTION

Nuclear emulsions are designed primarily to respond to charged particles. They differ from ordinary photographic emulsions in several ways. The important differences are: (1) they are much thicker, i.e., thicknesses vary from 10 to 3000 microns; and (2) the silver bromide to gelatin ratio is much higher in nuclear emulsions (4:1 by weight). As a charged particle traverses the emulsion, it loses its energy by the processes of excitation and ionization of atoms in the emulsion and, for electrons, by radiation of electromagnetic energy. For massive, highly charged ions, we are concerned only with the energy loss due to the ionization of atoms.

The number of grains per unit length of track that become developable is a function of the rate of energy loss of the particle. The energy can come from the original particle or from the secondary electrons which are produced in the process of ionization. Upon development, these emulsion grains become chiefly metallic silver while the other crystals of silver bromide are dissolved. A charged particle track in a nuclear emulsion is a series of roughly collinear, spherical grains. If the rate of energy loss of the particle is such that not every grain is made developable, then the distribution of distances between developed grains (gaps) is exponential, and is of the form:

\[ N(\ell) = N_0 e^{-\lambda \ell} \]

where \( N(\ell) \) is the number of gaps greater than some \( \ell \), \( N_0 \) is the total number of gaps and \( \bar{\ell} \) is the mean gap length.

The grain density of a track in emulsion (the number of developed grains per unit length) can yield information with regard to the rate of energy loss of the particle which produced this track. This information can, in turn, be used to determine the velocity of the particle and, if its residual range is known, its charge (mass).
Grain density can seldom be measured directly by counting the developed grains. Usually it is necessary to obtain estimates of the true grain density indirectly.\(^3\)

One good estimate of grain density is known as \(g_1 = 1/\bar{L}\), where \(\bar{L}\) is the mean gap length.\(^2\) Another way to measure \(\bar{L}\) is to find the least-squares solution for the slope of the gap-length distribution, Eq (1). In cases where the rate of energy loss of the particle is very large, the mean gap length can be the order of 1/2 micron and less. The accurate measure of small distances done in a reasonable length of time becomes a problem which must be overcome.

**DESCRIPTION OF EXPERIMENT**

Insensitive Ilford emulsions (K.O, K minus 1, and K minus 2) were used in this experiment. The emulsions were 100 microns thick, 1 x 3-in. glass-backed plates. They were exposed to artificially accelerated beams of heavy ions of \(^{12}\text{C}\), \(^{16}\text{O}\), \(^{20}\text{Ne}\), \(^{40}\text{A}\) at the Lawrence Radiation Laboratory's Heavy Ion Linear Accelerator. Energies of \(10.4 \pm 0.2\) Mev/nucleon were obtained. The 3-in. dimension of each plate was positioned in such a way that the entering ions made a 5-deg angle with the plane of the emulsions. Exposed in this manner, all ions recorded on a single plate had essentially the same momentum. The track lengths varied from 100 microns for \(^{40}\text{A}\) to 200 microns for \(^{12}\text{C}\). Each track was divided into 10-micron intervals. This was done so that the rate of energy loss could be considered as essentially constant over that region of track. In actual practice the change of \(dE/dx\) over the interval from its median value was almost always less than 10 percent. Because of the exponential nature of the gap-length distribution, only gaps over certain lengths had to be measured; this length was chosen to be 1/4 micron, the limitation being the resolution limit of the optical system.

The conventional technique of measuring gaps in nuclear tracks involves the use of a filar micrometer, an instrument consisting of a special eyepiece to which is attached a micrometer-screw mechanism that moves a hairline over a fixed reticle scale. This technique proved to be inadequate for the following reasons: (1) the image of the hairline often appeared wider than the distance to be measured, (2) the process was extremely slow since the micrometer reading had to be recorded before and after each measurement, and later converted to microns,
(3) a companion eyepiece could not be found for use on a binocular-body microscope, and (4) there was no convenient way of subdividing the track into 10-micron intervals.

EQUIPMENT

Figures 1 and 2 show the equipment used. The equipment consisted of a Cooke, Troughton and Simms Universal microscope used in conjunction with a special tilt superstage, a variable length monocular tube, a Leitz Micro-Ilso photomicrographic attachment, two Leitz cameras and calibrated illumination system.

Microscope

A standard Cooke Universal microscope stand was used. It rested on a 1/4-in. thick lead plate placed on a 3-in. thick piece of foam plastic. The padding was used to minimize vibrations normally occurring in any building and those due to the operator. Since each movement is magnified several hundred times at the image plane, any vibration renders photomicrography almost impossible. The above arrangement was found very satisfactory chiefly because of the fact that the foam plastic isolated the microscope from outside vibrations and dampened internal noise, such as the shutter movement. It is preferable to mount the microscope in such a way that it moves as a single unit. The original monocular tube of the microscope was replaced by one whose tube length was variable. The optical tube length could be adjusted to any required value, making it possible to use various objectives.

Stage

A specially designed superstage was mounted on the regular precision stage of the microscope. This device was capable of rotation about the Z axis and, at the same time, permitted the plane of the emulsion to be tilted so that the track appeared flat in the focal plane of the objective. (Fig. 3.) Owing to the shrinkage in thickness of the emulsion on processing, it was possible to bring tracks with dip angles up to 6 - 7 deg into the focal plane of the objective.
Optics

Oil immersion optics of high numerical aperture were used throughout. Leitz aplanatic-achromatic N.A. 1.4 and a Bausch and Lomb achromatic N.A. 1.4 condensers were used and both gave satisfactory results. A Leitz 100X plano apochromat N.A. 1.32 and a Zeiss 100X plano achromat N.A. 1.25 objectives were found to be excellent for this purpose. Their chief limitation in emulsion work is that of rather short working distance (270 microns for the Leitz objective). However, since the original thickness of our emulsion was 100 microns, and the final thickness of the processed emulsions was approximately 40 microns, this limitation did not affect our work. Taking the shrinkage factor into account, the Leitz objective can be used satisfactorily with emulsions of original thicknesses of up to 600 microns. This is assuming, of course, that the plane of the emulsion would not be tilted.

Photomicrographic Attachment

A standard Leitz Micro-Tbso photomicrographic attachment was mounted on a bracket supported by a column attached to the microscope. (Figs. 1 and 2) By releasing a clamp on the support column, the Micro-Tbso could be swung into or out of position. The photographic eyepiece was a Leitz 10X periplan eyepiece in which was mounted a specially designed eyepiece micrometer disc (reticle). The eyepiece diaphragm was critically adjusted so that the image of the reticle was focused upon the image of the track. (Fig. 4.) The reticle was cut on a machine similar to the one used at LRL in Berkeley, California. The ruling was done on a sheet of optical quality No. 3 Corning coverglass. The scale ruling of 200 divisions was so designed that the image of the ruled lines had the spacing of 0.5 micron between lines at a magnification of 1460 diameters. A separate reticle had to be cut for each objective because of the deviation of actual magnification from that specified by the manufacturer. The calibration of the reticles was checked and verified by using a Bausch and Lomb precision stage micrometer.

Illumination

A standard Cooke light source was used, powered by a Kepco Model PR 15-10M regulated D.C. power supply (regulated to 1 percent). Because of the fact that the area of the image is a function of exposure, the light intensity at the film plane had to be constantly monitored. This was done with the use of a Photovolt M501 lightmeter. The Photovolt itself was kept drift-free by the use of a standard lamp.
Maximum resolution with given optics was obtained with the use of monochromatic green light.

Cameras and Film

A 35-mm Leica and a Leitz 9 x 12-cm Makam camera were used. The Leica was equipped with a standard 1/3-reduction lens. Fine grain Kodak Panatomic X film was used. The Makam camera had a special graphic back adapted to it so that either standard 4 x 5-in. holders or 4 x 5-in. packets of Polaroid film could be used.

Experimental Procedure

Upon alignment of the microscope, the 1 x 3-in. nuclear emulsion was placed into the holder of the tilting stage. The tilt of the stage was varied until the image of the track appeared flat in the field of view. At this point either the stage or the photographic eyepiece was rotated in such a way that the image of the reticle was superimposed on the image of the track. (Fig. 5.) Each of the above adjustments was simple to execute since most of the tracks appeared as approximately parallel lines. (Deviations caused by multiple scattering are usually small for heavy ions.) Before photographing, the operator checked that perfect Kohler illumination existed, that the numerical apertures of the condenser and the objective were approximately equal, and that the light intensity was correct.6,7,8

The shutter mechanism of the Micro-1bso was used exclusively. When used in conjunction with a Leica camera, the focal plane shutter of the camera was opened and remained so during the exposure. Focal plane shutters are not useful in photomicrography because of the vibrations which they create.

Each track was photographed in several sections. At the magnification of 1500 diameters, approximately 60 microns of track appeared on each photomicrograph with an overlap of from 5 to 10 microns on each end to permit interidentification between corresponding points of the track. (Fig. 5.) Two photomicrographs were necessary to record a complete Argon or Neon track and four for a complete track of Carbon. With this technique, the thickness of the reticle lines superimposed on the track images was reduced to the point where they no longer obscured the gaps to be measured and yet were clearly visible.
The advantages of this method are: (1) the calibration of the reticle remains constant and independent of shrinkage or expansion of film and the prints themselves; (2) in the process of developing the film and/or the prints, whatever happens to the image of the track also happens to the calibrated reticle lines; and (3) the speed with which grain density can be measured is increased significantly.

In this manner it was possible to take many thousands of photomicrographs and process them in a relatively short time.

EXPERIMENTAL PROBLEMS

Upon examination of Fig. 5, it is observed that the image of the reticle is sharp at the center of the field of view but becomes steadily more distorted toward the edge of the photograph. At the same time the primary image appears sharp at all points. This photomicrograph was taken with a 1/3 X reduction lens using the 35-mm Leica camera. The distortion of the reticle image did not appear when the Makem camera was used. (Fig. 6.) In this example both the image of the track and that of the reticle appear in focus throughout the field of view. Several different 1/3 X reduction lenses were tested, but all showed this peculiarity. This made the task of taking data off film somewhat more difficult and also reduced its accuracy.

During the investigation it was found that the sensitivity of the films used varied considerably from batch to batch. The variation was such for No. 52 Polaroid film that the reported ASA number had very little significance. This was less true of the Kodak Panatomic X film. It was found advisable, however, to run a calibration on each different batch of film.

DISCUSSION

In practice, the technique described herein proved both fast and reliable. It was found that a well trained operator using the 35-mm
Leica camera could average thirty photomicrographs per hour. This is assuming, of course, that the track density is high so that a minimum amount of time is spent in searching for an event.

The errors encountered in measuring $g_1$ can be separated into two categories: those arising from the statistical fluctuations in the gap-length distributions, and the errors introduced by the subjectivity of different readers. It was found that the statistical errors were usually smaller than reader errors. When grain density was of the order of 2 grains/micron and less accuracy was of the order of 10 percent, between 2 and $\frac{1}{4}$ grains/micron accuracy varied from 10 to 20 percent. Beyond $\frac{1}{4}$ grains/micron accuracy deteriorated rapidly.

As mentioned, a disturbing feature of this technique is the fact that when the 35-mm camera is used, the quality of the image of the reticle deteriorates steadily toward the edges of the exposure. On the other hand, the Makam camera offers the advantage of sharp clear images at all points, but has the disadvantage of slow speed due to the fact that cut film is used. It seems advisable to combine the desirable features of both cameras. With this in mind, a new camera is being built which will fit the back of the Makam and will use 100-ft rolls of 35-mm movie film. This will enable two hundred exposures to be made on a single roll with no further magnification necessary, and will offer a wide variety in the types of film (both positive and negative) which may be used.
REFERENCES


Fig. 1  Cooke, Troughton and Simms microscope with a Leitz Micro-Ibso photomicrographic attachment and a 35-mm Leica camera.
Fig. 2 Cooke, Troughton and Simms microscope with a Leitz Micro-Isao photomicrographic attachment and a Leitz 4 x 5-in. Makam camera.
Fig. 3 Tilting superstage.
Fig. 4 Measuring reticle for a Leitz periplanatic 10X eyepiece. Scale rulings of 200 divisions at 52 microns. Width of individual rulings is approximately 5 microns.
Fig. 5 A typical photomicrograph used in measurement of grain densities. Print made from a 35-mm Panotomic X exposure.

Fig. 6 A photomicrograph taken with Leitz Makam camera showing a sharp clear image of the reticle.
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Imposed image of a calibrated eyepiece-micrometer disc. The gap lengths are measured to an accuracy of \( \frac{1}{4} \) micron.

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