Lawrence Berkeley National Laboratory

LBL Publications

Title

Techniques for Handling and Processing Emulsion Stacks

Permalink

https://escholarship.org/uc/item/2244s8fw

Authors

Birge, Robert W Kerth, Leroy T Richman, Chaim <u>et al.</u>

Publication Date

1954-09-01

UCRL 2690 UNCLASSIFIED

UNIVERSITY OF CALIFORNIA

Radiation Laboratory

TWO-WEEK LOAN COPY

This is a Library Circulating Copy which may be borrowed for two weeks. For a personal retention copy, call Tech. Info. Division, Ext. 5545

BERKELEY, CALIFORNIA

DISCLAIMER

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor the Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or the Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof or the Regents of the University of California.

UCRL-2690 Unclassified Instrumentation

UNIVERSITY OF CALIFORNIA

Radiation Laboratory

Contract No. W-7405-eng-48

TECHNIQUES FOR HANDLING AND PROCESSING EMULSION STACKS

Robert W. Birge, Leroy T. Kerth, Chaim Richman, Donald H. Stork, and Stanley L. Whetstone

September 8, 1954

Berkeley, California

TECHNIQUES FOR HANDLING AND PROCESSING EMULSION STACKS

Robert W. Birge, Leroy T. Kerth, Chaim Richman, Donald H. Stork, and Stanley L. Whetstone

Radiation Laboratory, Department of Physics, University of California, Berkeley, California

September 8, 1954

ABSTRACT

The techniques for assembling, processing and handling large nuclear-emulsion stacks are discussed. Results of experiments .varying the development procedure are presented.

-3-

TECHNIQUES FOR HANDLING AND PROCESSING EMULSION STACKS

Robert W. Birge, Leroy T. Kerth, Chaim Richman, Donald H. Stork, and Stanley L. Whetstone

Radiation Laboratory, Department of Physics, University of California, Berkeley, California

September 8, 1954

The Richman group has been developing techniques for processing, bandling and lining up nuclear-emulsion stacks. Because many different articles have appeared on this and related subjects, we are writing mainly a review of the various techniques that have been found most satisfactory. Certain stages of the operations, however, are different from those used elsewhere and seem worth comment. In general the techniques are those of the Bristol group,¹ with modifications due to Brussels,² Bombay,³, the Naval Research Laboratory⁴ at Washington, D.C., Mr. C. Waller of Ilford Ltd., and others.

I. PREDEVELOPMENT PROCEDURES

The emulsions as received from Ilford are G-5 pellicles, 600 microns thick, mounted on a so-called temporary glass backing suitable for stripping. We eradicate the emulsions to clear background tracks because the schedule for running time on the Bevatron is uncertain. When the machine is on a standard operating schedule, this step may be omitted.

Eradication is done at 33° - 36° C for 5-7 days. These variations in time and temperature seem to have little effect on eradication. All plates, however, lose sensitivity, as shown in Section IV. After the

UCRL-2690

emulsions have been allowed to come to equilibrium with about 60% relative humidity in a closed container, they are stripped from the glass by the method proposed by Mr. Waller, viz., running a knife around the edge and peeling the emulsion from the glass. The pellicles have their rough edges trimmed with scissors, holes punched in each corner, then are stacked together (with no separators) between 1/2-inch-thick black bakelite sheets, and clamped tight with screws which pass through the holes. The completed stack is machined to size in daylight with a milling machine. The edges exposed to light in this way are blackened less than 200 microns in from the edge. The machined stack is X-rayed on the edges through slits at eight positions about 1 inch from each corner, leaving a faint line 50 µ wide penetrating about 1/4 inch into each emulsion.

After the stack has been exposed and is to be developed, the emulsions are separated, a procedure that is fairly simple if the pellicles were dry before stacking. The Bristol method of wiping the emulsion and treated glass plates with chamois skin in 5° C water with 1% glycerine and rolling with an ordinary wringer has proved quite satisfactory for both large and small pellicles. A heavy load on the wringer appears to be necessary. Seven lbs, per linear inch of emulsion is satisfactory. The mounted pellicles are then blotted with filter paper and left overnight to "set".

II. DEVELOPMENT PROCEDURE

The developing procedure is essentially that of Bristol,¹ everything done at 5° C except for the 2 1/4-hour dry hot stage at 16° C. (Complete grain count versus time, temperature, and formula is discussed in Section IV.)

-4-

In our work the emulsions are never moved during the processing. The various solutions flow in and out of the tanks by gravity feed and the temperature is controlled by cooling coils soldered to the outside of the tanks. A thermocouple connected to a recorder registers the temperature. Two such tanks, with a total area of 10 square feet, are in operation. After development and stop bath have been finished, the tank is filled with fixer, and a timing system then lets fresh fixer into the tank for 10 seconds every 3 minutes. The old fixer goes out a drain in the bottom of the tank to an overflow. The flow changes one tank volume each day. We have also recirculated the fixer through a large storage tank. Following a suggestion of Dr. Gerson Goldhaber, we now use a 30% hypo solution (defined as 300 grams of hypo and 22.5 grams of sodium bisulfite in a total volume of 100 cc. of solution) which clears the emulsions in three days. With a 40% solution, clearing takes seven days, and with a 20% solution the time is intermediate between the two.* The plates are left in the fixer for one day after clearing, and then water dilution and washing are started. The dilution is done slowly, with the same timing system as used for fixing, to keep down the formation of bubbles. The fluid is drawn out of any occasional bubbles with a hypodermic needle as soon as they are observed. When the plates are completely free of hypo, a plasticizing solution of 4% glycerine in water is used and 4% glycerine in alcohol is added at a rate increasing logarithmically with time until about two volume changes have occurred. (This gives an alcohol concentration of about 87% in two days.) The solution is then drained and the plates finish drying in room air and at room temperature.

We now start at 40% and dilute to 30% during clearing time. This procedure minimizes bubbles and the plates clear as rapidly as with 30% directly.

-5-

UCRL-2690

III. POSTDEVELOPMENT

-6-

The emulsions are wiped off with alcohol to remove surface grains and the excess glass is broken off, with the aid of a diamond scribe, with reference to the X-ray marks. The accuracy of this method allows lineup to about $\frac{1}{2}$ 200 µ. Following this the plates are dipped in thinned Duco cement (E. I. du Pont de Nemours and Co., Inc., Wilmington, Delaware) for protection. For more accurate lineup to speed up follow-through work, we glue brass tabs across the four corners with the aid of a jig and then sand these tabs down with reference to the X-ray marks, using a microscope under 210 x magnification. A stack of 20 emulsions takes about four hours to line up to $\frac{1}{2}$ 15 µ. This number includes misalignment due to distortion in the emulsion, since alignment was measured by using diving tracks of 340-Mev protons. Mounted uneradicated pellicles developed at 16^o C or eradicated pellicles developed at 25^o C show distortion amounting to 35 Covans.

IV. RELATED PROBLEMS

A. Variation in Grain Count as a Function of Development

(1) Of the nearly infinite number of variables that determine the the final grain count in nuclear emulsions, we have varied a few, and present the results here.

The time and temperature for the "dry stage" were varied for "Bristol" and "Brussels" developers, using 600 u Ilford G-5 nuclear emulsions. (See Appendix for composition of developers.) The water presoak and cold developer soak were 2 hours and 3 hours respectively at 5° C in all cases. For the particular dry stage done at 5° C, some of the emulsions were left in the developer during the entire "dry stage" time and appear as a separate curve. These data are shown in Figs. 1 through 4.

A simple analysis, based on the assumption that the rate of the chemical reaction is proportional to the number of undeveloped developable grains multiplied by a function of temperature, gives a relation for the contribution to the grain count from the dry stage of the form

$$G_{D} = G_{R} (1-e^{-\lambda(T)t}),$$

where G_D is the contribution to the grain count by the dry stage, G_R is the number of grains rendered developable by the passage of a charged particle (function of the velocity only), $\lambda(T)$ is a function of the temperature T,

t is time in dry stage.

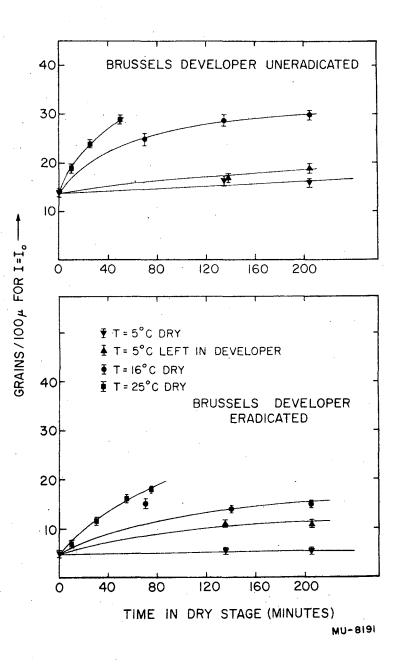
Then the total grain count is given by

$$G = G_{CS} + C_{D},$$

where G_{CS} is the contribution to grain count by cold presoak.

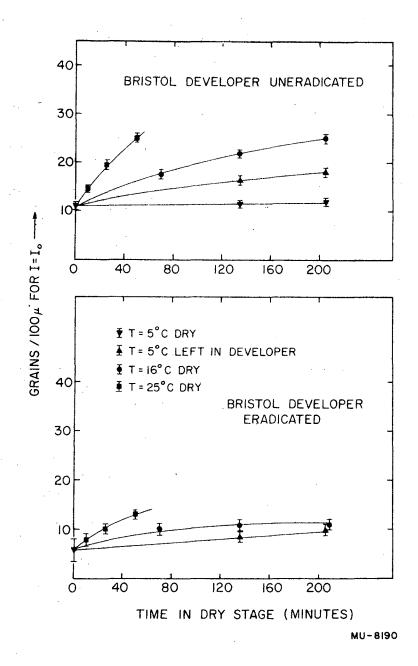
Such a simple analysis gives a surprising fit to the data. The inclusion of the variation in the concentration of the developer as grains are formed (i.e., the "using up" of the developer) would alter the curves in the proper direction to give a better fit.

It is surprising that such a large fraction of the grain count is given by the presoak, for the supposed function of the presoak is to give the developer a chance to penetrate the emulsion while little development takes place.



Figs. 1 and 2.

-8-



Figs. 3 and 4.

As can be seen from Figs. 1, 2, 3, and 4, eradication reduces the grain count to about 50% the uneradicated value. This is a little greater loss than other workers have reported.

The random grain background was examined for emulsions of equal grain count for the two developers used, and found to be very nearly the same. We have found that extending the time for 25° C Brussels development to 75 minutes increased the random grains to an almost intolerable level.

(2) A typical plot of grain count as a function of depth in 600 µ emulsions shows uniform sensitivity with depth until within 30 µ to 50 µ of the surface. At this depth the grain count starts to fall off, and is only about half at the very surface. We have made little or no investigation of the cause of the surface fall-off, and have not come to any conclusion about how to correct it. (With 30% fixer, the grain count no longer

falls off at the surface) <u>B. Discoloration</u>

When 200 μ emulsions are processed by the Bristol formula, they sometimes turn a vivid pink. This always happens when they have been eradicated. The cause is oxidation of the amidol, and the effect may be cured by increasing the Na₂SO₃ in the developer by 30% to 50%, or by using a nitrogen atmosphere during the hot stage, or by leaving a film of developer on the plates in the hot stage instead of wiping them off. In addition to showing the pink stain, the emulsions have almost no sensitivity. Increasing the amidol without proportionate increase in Na₂SO₃ makes the plates nearly opaque. The oxidation has never occurred with 600 μ plates.

-10-

C. Eradication and Soak-up

(1) It was suggested to us by Dora Sherman of this laboratory that eradicated plates, when soaked in water, take up less water than uneradicated plates. Tests were run on both 200 µ and 600 µ plates to determine whether this effect caused the decreased in grain count in eradicated plates. Although the eradicated emulsion takes up only 1/3 as much water as does an uneradicated emulsion, changing the soaking time by a suitable amount to cause either normal or eradicated plates to soak up only 1/3 as much developer caused no change in their respective grain counts.

(2) A separate series of tests was run to determine the amount of water soaked up by emulsions as a function of temperature. After 6 hours, the 5° C emulsion was transferred to 21° C water, but no further significant increase in weight occurred (see Fig. 5). Thus, emulsions that are cooled before immersing in the cold prescak absorb less water and developer than those put in at room temperature. No change in grain count occurs, however, as a result of this change in procedure.

D. Locating events

In order to relocate events in our emulsions, we have had printed (at the suggestion of Mr. Erb of Erb and Gray Co.) a rectangular matrix of 150 x 150 squares, each square having a cross in the center and two 3-digit numbers giving the x and y coordinates of the square. This matrix has been photographically reduced by Mr. Phil Carnahan from 36 in. x 36 in. to 6 in. x 6 in. and 3 in. x 3 in. Although we originally planned to print only a few of these on glass plates, we have found it

-11-

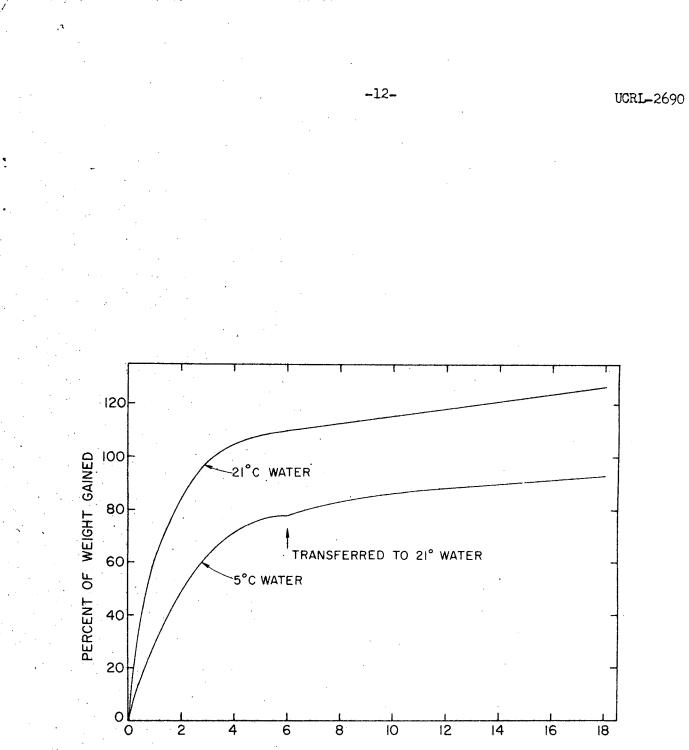


Fig. 5.

possible to mount pellicles, in the usual way, directly onto either Eastman Kodalith or Ilford Formalith plates that have previously had the matrix photographed onto them and been processed. (Ilford will supply these Formalith plates in any size and of the same thickness as their treated glass plates.) We believe this method to be as easy and reliable as that recently suggested by Vanderhaege (gluing film to the surface of finished plates), and furthermore, it does not limit the working distance of the objective. (We now use 10 micron G-5 for our grids)

Dr. Harry Heckman of this laboratory is making prints of these matrices directly on the pellicles themselves, and has obtained satisfactory results. However, mistakes in exposure cannot be rectified, and there is the possibility of losing a track along a line of the matrix.

ACKNOWLEDGMENTS

The calibration tracks for all this work were from 300- to 340-Mev protons from the 184-inch cyclotron. Fortunately there has been ample opportunity to obtain high-energy protons of the right intensity, owing to the almost continuous operation of the cyclotron for experiments with the polarized proton beam. The cooperation of Messrs. Chamberlain, Segre, Tripp, Wiegand, and Ypsilantis is greatly appreciated.

We are grateful for the aid of Mrs. Beverly Baldridge and Mrs. Marilynn Harbert in making the grain counts.

Thanks are due also to Dr. Jack Merritt, Mr. Roy Haddock, and Mr. Jack Sandweiss.

The discussions with Dr. Donald Perkins and Dr. Peter Fowler from Bristol were of great help in our early work.

-13-

à

APPENDIX

"Brussels" developer (2,3)		
Distilled water	1000	cc
Boric acid	35	g
Sodium sulphite (anhydrous)	18	g
Potassium bromide (10% solution)	8	cc
Amidol	4.5	g

"Bristol" developer (1)

Distilled water	930 cc
Sodium sulphite (anhydrous)	6.7 g
Sodium bisulphite (saturated solution)	1.4 cc
Potassium bromide (10% solution)	8.0 cc
Amidol	3.0 g

REFERENCES

- (1) A. D. Dainton, A. R. Gattiker, and W. O. Lock, Phil. Mag. <u>42</u>, 396 (1951)
- (2) C. C. Dilworth, G. Occhialini, and L. Vermaesen, Bulletin du Centre de Physique Nucleaire de L'Universite Libre de Bruxelles, Note N^O 13a, Février 1950
- (3) D. Lal, Y. Pal, B. Peters, Proc. Indian Acad. Sci. 38, 277 (1953)
- (4) B. Stiller, M. M. Shapiro, F. W. O'Dell, Rev. Sci. Instr. 25, 340 (1954)