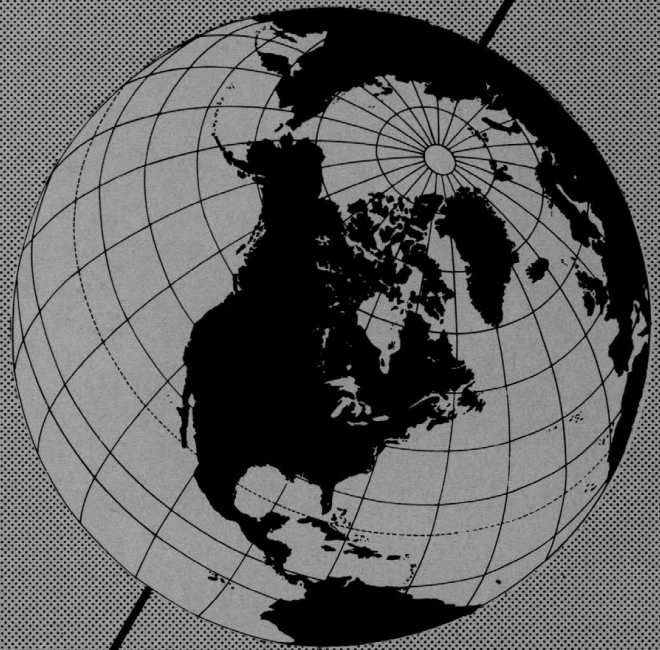


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# Creep of Single Crystals of Ice



**U. S. ARMY  
SNOW ICE AND PERMAFROST  
RESEARCH ESTABLISHMENT**

*Corps of Engineers*

# CREEP OF SINGLE CRYSTALS OF ICE

by  
D. T. GRIGGS AND N. E. COLES

## ABSTRACT

A commercial deep-freeze unit was modified to serve as a laboratory for growing single crystals, cutting specimens of a given orientation, testing these specimens in compressive creep, and studying thin-sections of the results on a universal stage. A method of growing single crystals of adequate size was developed by adapting the Bridgman method. Forty-one creep tests were made at temperatures ranging from  $-1^{\circ}$  to  $-18^{\circ}\text{C}$ . These gave an unexpected form of creep-curve in which the rate of strain continuously increases with time. The mechanism of deformation is dominantly basal translation—consistent with earlier work. The dependence on temperature and stress is expressed empirically.

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## INTRODUCTION

The objectives of this work were:

- a. To learn how to use a deep-freeze unit as a cold laboratory.
- b. To grow moderate-sized single crystals of ice.
- c. To determine the plastic properties of single crystals of ice in constant load "creep" experiments as a function of temperature, crystal orientation, and stress.

The first two occupied so much of the time available that the creep tests were not nearly as comprehensive as had been hoped. The examination of the deformed material has fallen far short of expectations. The material was stored for future examination, when the power line was disconnected and all material melted. Nevertheless, it is felt that the experimental results add considerably to the previously available information, and it is hoped justify the program.

The experiments were conducted at the Institute of Geophysics, University of California, Los Angeles, between December 1950 and June 1952.

### USE OF THE DEEP-FREEZE AS A COLD LABORATORY

This program is the first of its kind known to the authors in which a commercial deep-freeze unit was used as a complete laboratory for growing specimens, preparing them, test-

ing, and examining the deformed material. After one learns the simple precautions that are necessary, such a unit makes a very satisfactory laboratory. Actually, in this program a major part of the time was spent in learning the simple do's and don'ts of life in this only slightly abnormal environment.

The most fundamental difficulty encountered was the sublimation and crystallization over considerable periods of time in the presence of temperature differences, due to the comparatively high vapor pressure of ice at the working temperatures. On one occasion an alcohol-water coolant bath was used. In short order alcohol transported in the vapor phase had permeated all the ice in the freezer, which had to be emptied and thoroughly cleaned. In practice, since it was not feasible to eliminate temperature gradients, movement of  $\text{H}_2\text{O}$  in the vapor phase was minimized by restricting the motion of air with tight bulkheads and placing spare ice near hot spots to maintain high humidity.

Another plaguy condition was the frosting of glass windows. Some standard preventatives were used to no avail. The only really satisfactory method is the use of double windows with dry air gaps. Glass within the chamber, as in microscopes, etc., must be accessible for occasional cleaning, but this can be kept to a minimum if the glass can be placed in a comparative hot spot.

Finally, the necessity of manipulating things inside the chamber is troublesome. In general the operations are not sufficiently repetitive



to justify the construction of remote-control devices, and one resorts to opening the top of the compartment and using gloved hands. After each such disturbance of a temperature-regulated compartment, sufficient time must be allowed for return to thermal equilibrium before measurements are made.

The deep-freeze unit used was a Coldspot 19.4 cubic foot standard domestic model. Its refrigeration unit and temperature regulator were used without modification. When more accurate regulation of temperature was required, insulated chambers with local heating controlled by a precision regulator were used.

The deep-freeze was divided into four compartments as shown in Figure 1. The right hand chamber was used for machining the single crystal specimens and attaching them to the creep test platens, and for general purposes. The second chamber from the right contained the crystal grower. The third chamber housed the small microscope and universal stage for examining sections of the specimens. The fourth chamber was used for the creep testing. It was the only one of the three that was normally closely controlled in temperature. Since it frequently was operated at a considerably higher temperature than the other compartments, it had more insulation on its walls. The inter-compartment bulkheads were half-inch plywood (the plywood must be well bonded and sealed or it separates). The creep test chamber had in addition  $\frac{1}{8}$ -inch masonite with a  $\frac{3}{4}$ -inch dead air space.

Temperature in the creep chamber was regulated by a special thyatron regulator which was on hand, driving a light-bulb heater. The sensing element was a large open grid of hitemco wire. This grid constitutes one arm of a Wheatstone bridge, and a change in its resistance due to temperature change alters the current passed by the thyatron. The heater is thus driven constantly at the power level necessary to balance the heat losses from the compartment. In practice it is possible to maintain the temperature of this compartment constant to  $0.1^{\circ}\text{C}$  for long periods.

#### GROWING SINGLE CRYSTALS

Several methods differing in detail were tried in the process of developing a satisfactory technique of growing large single crystals of ice. These were all modifications of two basic methods developed respectively by Nacken (1915) and Bridgman (1925). In the first, a seed crystal of appropriate orientation is frozen to a cold plate and dipped in water slightly above the freezing point. Heat is re-

moved from the plate by a suitable heat-transfer means, causing the seed crystal to grow in a direction initially normal to the plate. As the crystal grows, the plate is raised at the same rate as crystal growth occurs, forming a cylindrical single crystal.

All our experiments with the Nacken method failed to grow crystals of sufficient size for two reasons: (1) The varying rate of heat transfer from the growing face could not be adequately matched by the elevating mechanism, so that the crystal either increased or decreased in diameter. (2) Nucleation at the boundary of the growing crystal could not be prevented, and the end-product always had crystals of extraneous orientation superimposed on the new growth of the desired orientation.

For the growth of small single crystals, the Nacken method appears to be satisfactory. Our experience leads us to believe, however, that it would be difficult to adapt the Nacken method to grow crystals 1 inch in diameter and 3 inches long, as we desired.

In the conventional Bridgman method for growing single crystals from the melt, a suitable container holding the material is heated above the melting point in a vertical furnace. The container is then slowly lowered from the furnace, causing crystallization initially to an aggregate. The container has a constriction (called a filter) to capillary size. Crystal growth proceeds toward this filter, and if the grain size is large compared to that of the filter, the whole cross-sectional area of the capillary is usually filled by a single crystal. This crystal is then supposed to propagate itself through the filter and expand in growth of the same orientation, filling the second chamber with a single crystal. Precautions have to be taken to insure nearly one-dimensional heat flow, and to prevent nucleation in the second chamber. This method has been used with conspicuous success by Bridgman and many other workers for the growth of single crystals of metals, etc.

There is a seeming paradox in Bridgman's description of this method. It would appear that the orientation of the crystal which happens to fill the capillary would be nearly random, since the aggregate formed in the first chamber contains crystals of nearly random orientation. This would lead one to expect that the orientation of the large single crystal in the second chamber would also be random. Bridgman found, on the contrary, that, if the crystal being grown was characterized by a prominent translation plane, that translation

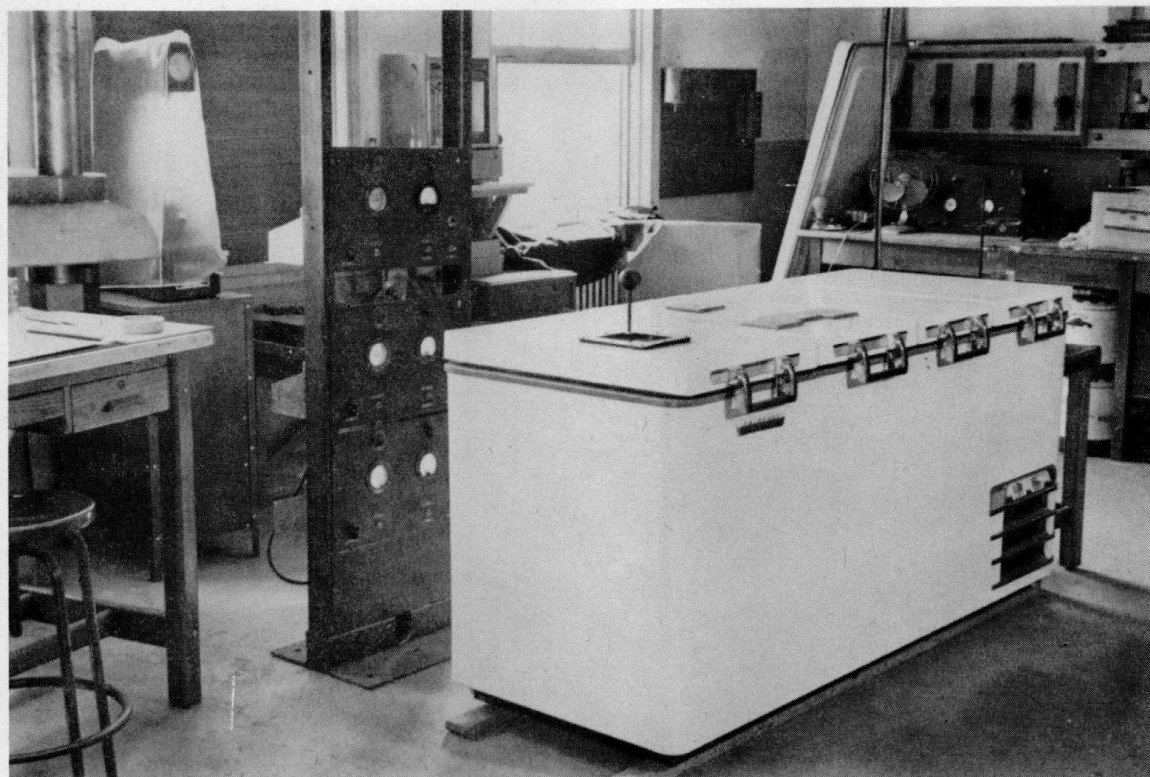


FIGURE 1. DEEP FREEZE, SHOWING FOUR COMPARTMENTS  
Temperature regulator at left.

plane usually was oriented parallel to the axis of the second chamber. As will be seen later, we observed the same effect.

To adapt the Bridgman method to the growth of ice single crystals, the first obviously necessary change is to turn it upside-down, since ice is lighter than water. Accordingly, we built a small furnace and elevator mechanism, constructed suitable two-chamber containers with a capillary constriction between, and put the whole apparatus in the deep-freeze. We found encouraging propagation of single crystals from the filter. It was very difficult, however, to get sufficiently one-dimensional heat flow and prevent nucleation in the second chamber, so that the size of our single crystals was always limited to a small fraction of the second chamber.

The results obtained in several experiments with this apparatus were sufficiently encouraging that we decided to develop a method embodying the Bridgman principles in a static apparatus to permit the growth of several crystals at once. It had become obvious that,

whatever method we tried, growth of large single crystals was going to require several days. The difficulty of matching the rate of elevating with the rate of growth in an apparatus with short thermal time constants seemed to be full of inherent difficulties, to be avoided if possible.

We reasoned that we could achieve the same growth of crystals relative to the crystal filter and chamber if the chamber remained fixed in a vessel of water which gradually cooled from the top. Accordingly, we lagged one compartment of our deep-freeze with about 6 inches of rock wool, and placed a vessel about  $6 \times 6 \times 6$  inches in the center, with its surface exposed to the air, but its sides and bottom well insulated from the walls and bottom of the deep-freeze. To insure nearly one-dimensional heat flow, a small heater grid was placed below the vessel. By trial and error, it was found that, when this heater dissipated five watts, the water in the vessel froze from the top down, with very little growth from the sides.



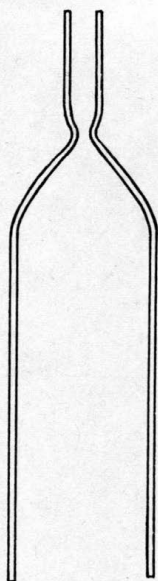


FIGURE 2. CRYSTAL FILTER

Four to six crystal filters were placed upright on the bottom of the vessel, with their tops about 1 inch below the water level. The whole assembly is shown schematically in Figure 3. The shape of the crystal filter is shown in Figure 2. These filters were made of glass. Various sizes of construction were tried, including no constriction. It was found that most satisfactory results were obtained when the constriction was  $\frac{1}{2}$  mm in diameter or smaller. With no constriction, single crystals were seldom attained.

It was found necessary to take considerable precautions against air bubbles and nucleation. The filters were cleaned with a sulfuric acid and potassium dichromate solution and placed in the water vessel, which was filled with boiled water. As the water cooled, it was placed under a bell jar evacuated by an aspirator which was adjusted to keep the absolute pressure only slightly above the vapor pressure of the water.

This method of growing crystals yielded good specimens 1 inch in diameter and 3 inches long. Four days were required for the growth of a batch, with the air in the deep-freeze compartment at  $-16^{\circ}$  to  $-18^{\circ}\text{C}$ . The average yield was 55 percent good single crystals. These were free from bubbles and from optical evidences of inhomogeneity, except as noted below.

The most common imperfections in crystals which were otherwise good were "veils." These irregular planar structures were usually sub-

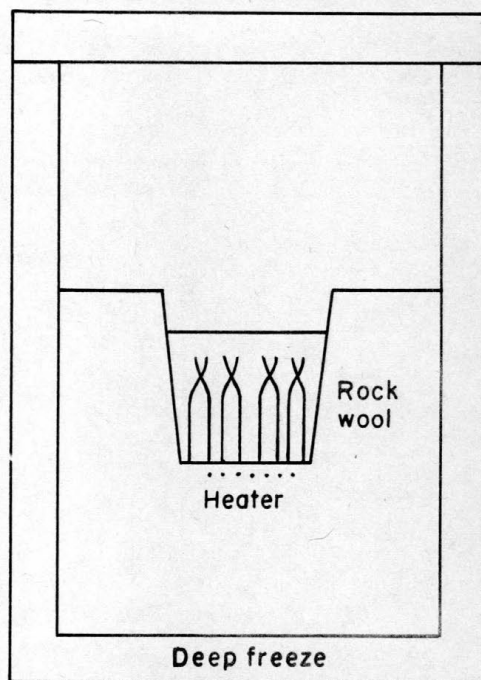


FIGURE 3. CRYSTAL-GROWING CHAMBER

parallel to the axis of the crystal-growing chamber. They gave the appearance of distributed impurities or crystal imperfections which, however, did not show up under crossed polaroids. Such crystals were rejected.

To ascertain whether or not the boule was a single crystal, the whole boule was immersed in a beaker of ice water placed on a stand between crossed polaroids and illuminated from below (Fig. 4). In this test, it would be most desirable to use a liquid which has the same index of refraction as that of ice ( $\epsilon = 1.313$ ,  $\omega = 1.309$ ). No liquid was available which exactly matched this, so ice-water was used ( $\mu = 1.333$ ). The slight difference in index coupled with the thickness and cylindrical shape made it impossible to obtain complete extinction in the specimen, since there was always some variable refraction at the cylindrical boundary. When the boule was rotated so that the *c*-axis of the crystal was horizontal, extinction was nearly perfect, but slight color banding could be seen parallel to the basal plane, which was parallel to the length of the cylindrical boule. The observed amount of birefringence was roughly that to be expected from refraction. It was not possible, however, to exclude the possibility of some growth banding, similar to lineage structures, subparallel to the basal plane.

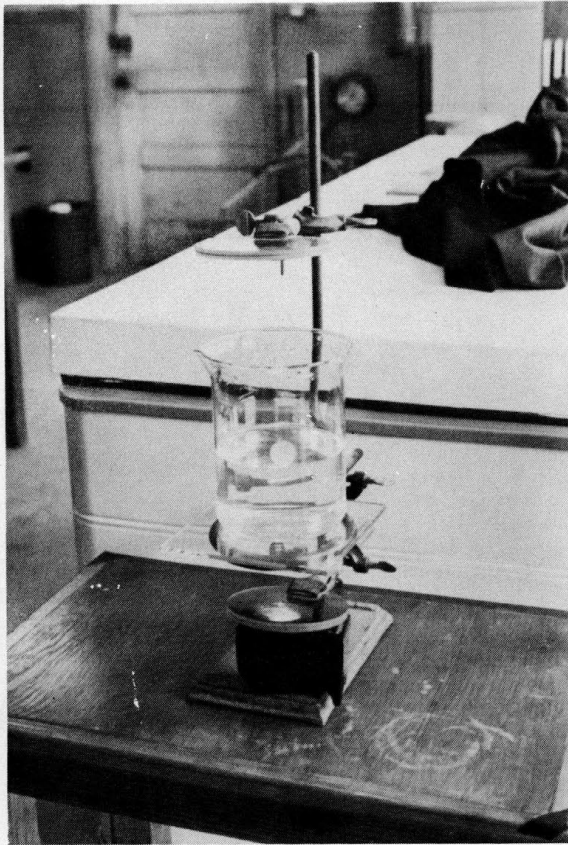


FIGURE 4. SET-UP FOR TESTING OPTICAL CHARACTER OF SINGLE CRYSTALS

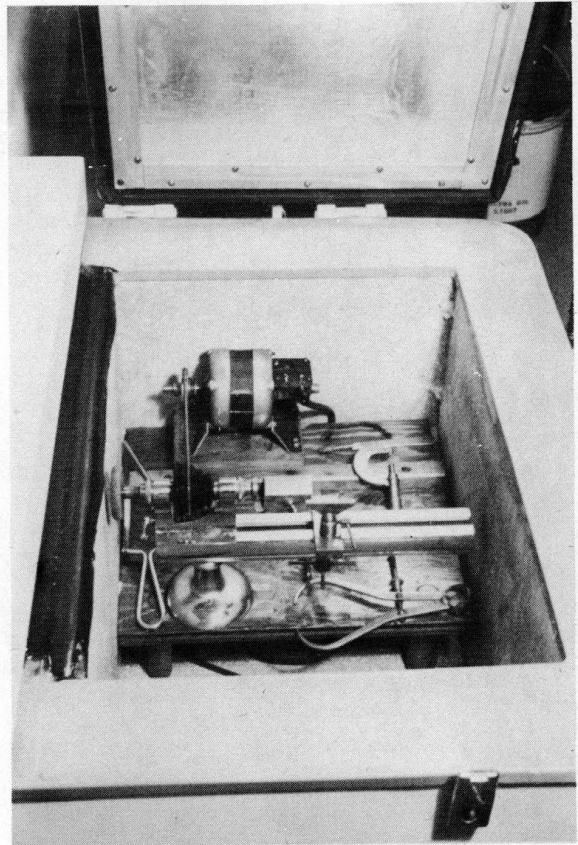


FIGURE 5. SPECIMEN-MACHINING COMPARTMENT

The electron microscope has resolved some structure in ice single crystals. E. J. Workman and F. K. Truby have found a roughly hexagonal prismatic structure of about 1 micron diameter parallel to the  $c$  axis of pure ice. Truby took electron photomicrographs of our single crystals and also of a sample of the large Alaskan glacier single crystals supplied to us by SIPRE. Both kinds showed a similar structure parallel to the  $c$  axis.

The orientation of our single crystals relative to the container is of interest. The ice that formed on the surface of the large vessel crystallized into individuals about an inch in diameter. The  $c$  axis of these crystals was usually normal to the water surface. One such crystal would grow into the small upper end of the crystal chamber, maintaining its orientation normal to the water surface in most cases. The single crystal that grew in the larger lower chamber, however, was invariably oriented with its  $c$  axis horizontal. We found from our creep tests that the primary trans-

lation plane in these single crystals was the basal plane. Hence, our observations are consistent with those of Bridgeman—that the single crystals grown in this manner are oriented with the primary translation plane parallel to the axis of the growth chamber.

It seems possible that the stresses resulting from unequal pressures through the capillary can cause translation and consequent rotation in the growing crystal. It is also possible that the stresses and pressures can favor the growth of the final orientation so strongly that this change takes place essentially discontinuously. There may be other possible explanations for this change in orientation. The observations in these experiments were not sufficient to prove or disprove these hypotheses.

It is necessary that the vessel containing the filters be sufficiently plastic that the expansion of the ice on freezing does not break it. We used a vessel of pliable vinyl plastic. At the end of the 4-day growing period, the



solidly frozen cake of ice was pushed out of this vessel. The crystal filters were chipped out with an ice pick. The single-crystal boules were removed by holding the filters under a water faucet. The boules themselves must not be exposed directly to the water for any extended period of time, for they are still cold and will crack due to thermal shock. Before immersing the boules in the ice water for the optical check, it was found necessary to allow the boules to warm slowly in a part of the deep-freeze just below  $0^{\circ}\text{C}$ . In the ice water, a pair of wooden tongs was used to orient the crystals so as to check for single crystals, and to find the location of the  $c$  axis. The crystal was appropriately marked.

The test specimen was machined from the boule on the jeweler's lathe in the deep-freeze (Fig. 5). The boule was first cut with a saw, normal to the desired axis of the cylinder, and shaved with a knife to the rough shape. The sawed face was then frozen to the faceplate of the lathe, and the cylinder was turned to final dimensions and cut off, using hand tools. Machining the specimens presented no problem. All operations were done in the open quick-freeze compartment of the deep-freeze, at a temperature of  $-10^{\circ}$  to  $-15^{\circ}\text{C}$ . Thin nylon gloves were worn to keep the hands from sticking and to prevent heat transfer from the hands to the specimen. The single crystals machined readily.

Before cutting off the test cylinder, a disk was cut off the end and filed for later comparison with the deformed specimen. Although most of these disks were lost through a power failure before the ultimate microscopic check had been made, a number were used to check the orientation of the test cylinder.

An ordinary petrographic universal stage was mounted on a specially built stage for study of thin sections in polarized light (Fig. 6). A four-power magnifier could be swung into or out of the line of sight. The upper polaroid could also be swung in or out. Light was provided from an external source, to avoid heating the chamber.

When using the stage for any prolonged study, it was necessary to close the lid of the compartment, work through two openings for the gloved hands, and look through a third opening. Light was introduced through a fourth opening (Fig. 1). Determinations were made in the same way as with an ordinary petrographic microscope and universal stage.

### CREEP TESTS

The single-crystal test specimens were first accurately centered on the test platens and



FIGURE 6. UNIVERSAL STAGE MOUNT

then frozen in place with a little water applied with an eye-dropper. A mounted specimen is shown in Figure 7. The mounted specimen was then placed on the dead-weight tester (Fig. 8). The cylinders were tested in compression. The load was applied by the V-shaped yoke on which the weights were hung by a chain. The lever at the top raises or lowers the small three-pronged platform immediately below the weights, so as to remove or apply the weight on the yoke. The yoke was centered on the specimen by a ball, shown in Figure 9 on a deformed specimen. The shortening of the specimen was measured by a Federal dial gauge reading .0001 inch/division, and with a calibrated accuracy of nearly one hundred-thousandth of an inch. This gauge was fastened directly to the stout steel support on which the lower platen rested. An invar bar was used for this connection to minimize the effect of any possible temperature change. The gauge was mounted on the centerline of the specimen. Leveling screws were provided in the base to level the whole assembly, so that the center of gravity of the weights was directly below the center of the specimen.

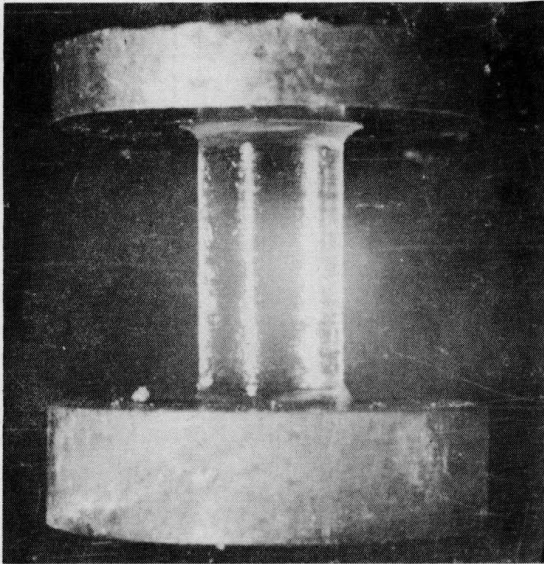


FIGURE 7. SINGLE CRYSTAL MOUNTED ON PLATENS

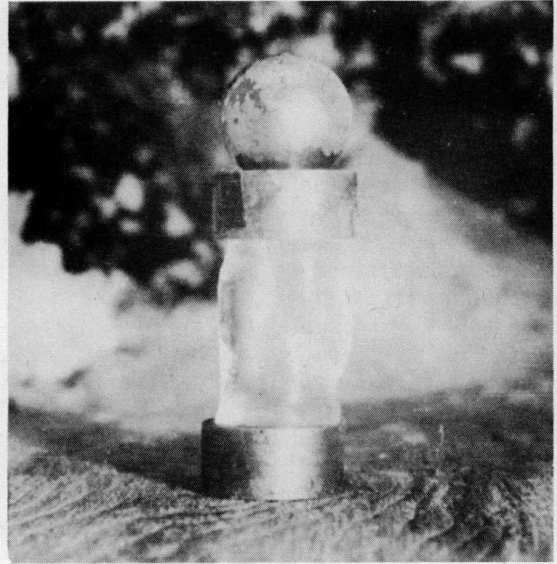


FIGURE 9. EXPERIMENT 29  
21% shortening at  $-1^{\circ}\text{C}$ ,  $1.4 \text{ kg/cm}^2$ .

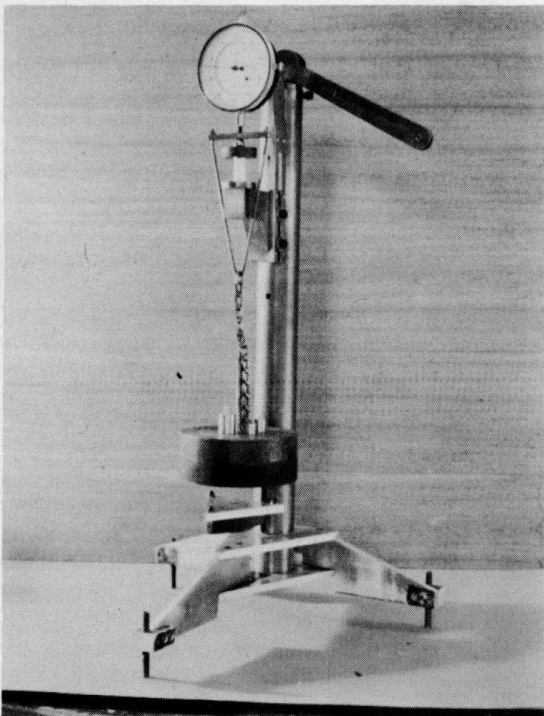


FIGURE 8. DEAD-WEIGHT CREEP TESTER

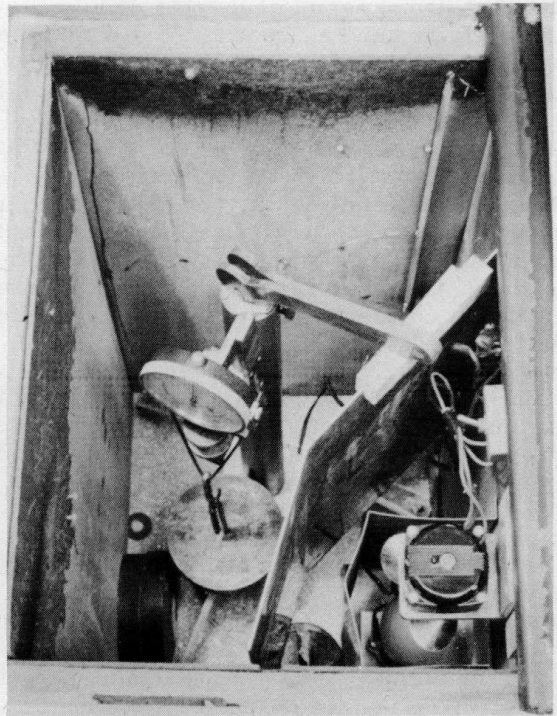


FIGURE 10. CREEP TESTER IN OPERATION



The dead-weight tester was kept in the left-hand compartment of the deep-freeze (Fig. 10). This compartment was doubly insulated and heated to the desired temperature by a light bulb whose power was regulated by an electronic temperature regulator. A fan immediately above the lamp (lower right corner) stirred the air in the compartment rapidly. It was necessary to maintain high humidity in this compartment to avoid sublimation of the specimen. This was accomplished by a small pile of ice immediately below the lamp. The hot air driven down by the fan sublimed enough of this ice to maintain the humidity at nearly 100%. Radiation shields of sheet aluminum were placed between the lamp and the apparatus (Fig. 10).

The control grid for the thyatron regulator was suspended on the back of the second radiation shield (not shown in Figure 10). The temperature of the compartment was measured by mercury bulb thermometers—one shown on the back wall, and one (not shown) suspended with its bulb close to the specimen. The dial gauge was read through a double glass window in the lid of the compartment, with the aid of mirrors mounted on an adjustable handle. The mirrors also allowed the specimen to be viewed from two angles, 90° apart.

In running an experiment, first the specimen was mounted on the creep-tester as described above. Its diameter had been measured, and the required number of weights for the particular run were placed on the weight pan, which was elevated so that only the weight of the yoke was on the specimen. The lid of the compartment was then closed and the temperature regulator was set to the desired value. A period of several hours was allowed for the attainment of thermal equilibrium. The lid was then briefly lifted to uncage the weights, and dial gauge, time, and temperature readings were made.

Eight preliminary creep tests were made with cylinders cut normal to the  $c$  axis (parallel to the length of the boule). These gave very erratic results. A few deformed specimens had surface markings parallel to the basal plane, suggesting basal translation, as had been found in short-term tests by earlier workers. The erratic results in these first experiments are completely explicable by the hypothesis that translation on the base is the dominant mechanism of deformation. In a perfectly oriented crystal loaded normal to the  $c$  axis, the resolved shear stress on the basal plane would be zero. Since our specimens were never perfectly oriented, the load

required to initiate creep would depend critically on the inclination of the basal plane to the axis of the specimen. As deformation proceeded, the translation would cause rotation of the basal plane so as to increase the resolved shear stress, and hence the rate of deformation. This is consistent with our observations. These preliminary experiments (Table II, Nos. 1-8) will not be further described, since they provide no other information.

In the remainder of the tests, cylinders cut at about 45° to the  $c$  axis were used. Thirty-two tests were made (excluding Experiment 21), 24 on our artificial single crystals, and 8 on specimens cut from large single crystals of glacier ice supplied to us by SIPRE.

The creep curves are shown in Figures 11-16. It will be immediately noticed that the curves are all of the same kind—concave upward throughout each test. This kind of a creep relation has never been observed by the senior writer in similar creep tests on a variety of different materials. The normal creep curve is initially concave downward—creep rate decreasing inversely with time; then, a nearly constant rate of creep; followed by a rapid increase in creep rate leading to fracture. The writers know of no similar creep curves, except for the results of Andrade (Andrade and Jolliffe, 1952) on the creep of lead in pure shear. He found a somewhat similar creep curve when the lead was warm enough for recrystallization to be important. Glen's (1952) published results on the creep of polycrystalline ice in compression show the normal type of creep curve<sup>1</sup>.

The next most obvious generalization from our data is that the creep rate at a constant stress diminishes with decreasing temperature. For example, the creep curve for 3.0 kg/cm<sup>2</sup> applied compressive stress at -1°C is similar to that for 10 kg/cm<sup>2</sup> at -18°C.

Next, it is clear that our reproducibility is poor, both with the artificial and natural crystals. In two cases, three identical experiments were run: at -1°C, 3.0 kg/cm<sup>2</sup> on artificial

<sup>1</sup>Since this was written, two articles on the deformation of ice single crystals have appeared: J. W. Glen and M. F. Perutz, *Journal of Glaciology*, vol. 2, no. 16, p. 397-402, 1954; and S. Steinemann, *Jour. Glac.*, vol. 2, no. 16, p. 404-412, 1954. Both give creep curves similar in form to ours. For small strains, our data compare reasonably well with those of Steinemann. For large strains, our method gives misleading results, due to inhomogeneity of strain and lack of constancy of stress.

crystals (Exps. 25, 26, 27); and at  $-2.9^{\circ}\text{C}$ ,  $5.3\text{ kg/cm}^2$  on natural crystals (Exps. 2-N, 4-N, 5N). The average departure from the mean is 30% in the former and 16% in the latter, both measured with respect to time at 3% strain. It will further be noticed that the creep curves at a given temperature are not always in the order of the stresses, which is also an evidence of unreproducibility.

The causes for this lack of reproducibility cannot be identified with certainty, but the most probable causes are:

1. Indeterminate variations in the resolved shear stress coefficient on the basal plane and in the translation direction, due to our inability to locate the  $a$  crystal axis prior to deformation. This can cause a maximum error of  $\pm 7^{\circ}$  and a probable error of about 4%.

2. Lack of perfect orientation of the test cylinder with respect to the  $c$  axis. The error here is  $\pm 2^{\circ}$ , corresponding to a variation in resolved shear stress of about  $\pm 1\%$ .

3. Eccentric loading of the specimen. The error so introduced is indeterminate in our experiments, but may have been large in some cases (e.g. Fig. 22). The shape of the deformed specimens suggests that in general the eccentricity was not important, since they seem to exhibit pure translation on the basal plane. If we were doing these experiments again, it would seem desirable to mount the specimen on the platen before machining, so that the specimen could be turned true in the lathe.

4. Change in the stress due to sublimation of the specimen. Again, this is hard to estimate because it was not possible to measure the specimen during a run, and afterward it was so deformed that measurements were not very meaningful. A case in which sublimation appears to have been important is shown in Figure 23. This specimen, however, is one which behaved as though it had a lower applied stress than that computed—just the opposite of what would be expected if sublimation had reduced its cross-sectional area.

5. Variations in the single crystals. The principal argument against this being important is the fact that the glacier ice crystals tested showed essentially the same behavior as our artificial crystals.

The creep curves plotted on log-log scales are shown in Figures 17-20. An attempt was made to find an empirical equation which would represent all the data. An equation of the form

$$\epsilon = [a (\sigma + b + cT + dT^2) t]^n \quad (1)$$

seemed to offer the most plausible possibility of a fit. Here  $\epsilon$  = strain, in percent,  $\sigma$  =

applied initial stress in  $\text{kg/cm}^2$ ,  $T$  = temperature in degrees C,  $t$  = time in hours,  $a, b, c, d, n$  are disposable constants. It was found statistically that the constants  $b$  and  $c$  were not sensibly different from zero, and that  $n = 2$  provided as good a fit as any. The empirical equation, fitted at 1, 3, and 5% strain, is:

$$\epsilon = [.62 (\sigma - .02T^2) t]^2. \quad (2)$$

Sample values from this equation are shown in Figure 21. Comparison with Figures 17-20 indicates that these curves are about as good as the data. The average deviation from the mean at 1, 3, and 5% is 26% for all experiments, 25% for the artificial crystals, and 29% for the natural crystals. It should be emphasized that this equation is purely empirical and has no theoretical basis. Since we have not a clue, physically, as to why the rate of strain should continuously increase in these experiments, we have no basis for theory.

The equivalent viscosity can be derived from equation (2) as follows: First put equation (2) in c.g.s. units:

$$\epsilon = 3.1 \times 10^{-20} (\sigma - 2 \times 10^4 T^2)^2 t^2$$

where  $\epsilon$  is still in percent,  $\sigma$  in dynes/cm<sup>2</sup> and  $t$  in seconds. It has been shown (Griggs, 1939, p. 230) that the equivalent viscosity may be calculated for a compressive creep test by:

$$\eta = \frac{\sigma}{300 \frac{d\epsilon}{dt}} = 0.55 \times 10^{21} \frac{\sigma}{(\sigma - 2 \times 10^4 T^2) t} \quad (3)$$

Expressing  $t$  in terms of  $\epsilon$ , this becomes:

$$\eta = \frac{.96 \times 10^{11}}{\left(1 - \frac{2 \times 10^4 T^2}{\sigma}\right) \sqrt{\epsilon}} \quad (4)$$

Table I gives illustrative values.

TABLE I. EQUIVALENT VISCOSITY OF ICE SINGLE CRYSTALS AT 1% STRAIN;  $\eta$ ,  $10^{12}$  POISE

$T, ^{\circ}\text{C}$	$\sigma, \text{kg/cm}^2$			
	1	2	10	20
-1	0.10	0.10	0.10	0.10
-3	0.12	0.10	0.10	0.10
-10	---	0.30	0.12	0.11
-20	---	---	0.53	0.16

The viscosities are comparable to those found by Hess (1902) for river ice, but are much lower than the values quoted by Dorsey (1940, p. 456) for glacier ice.



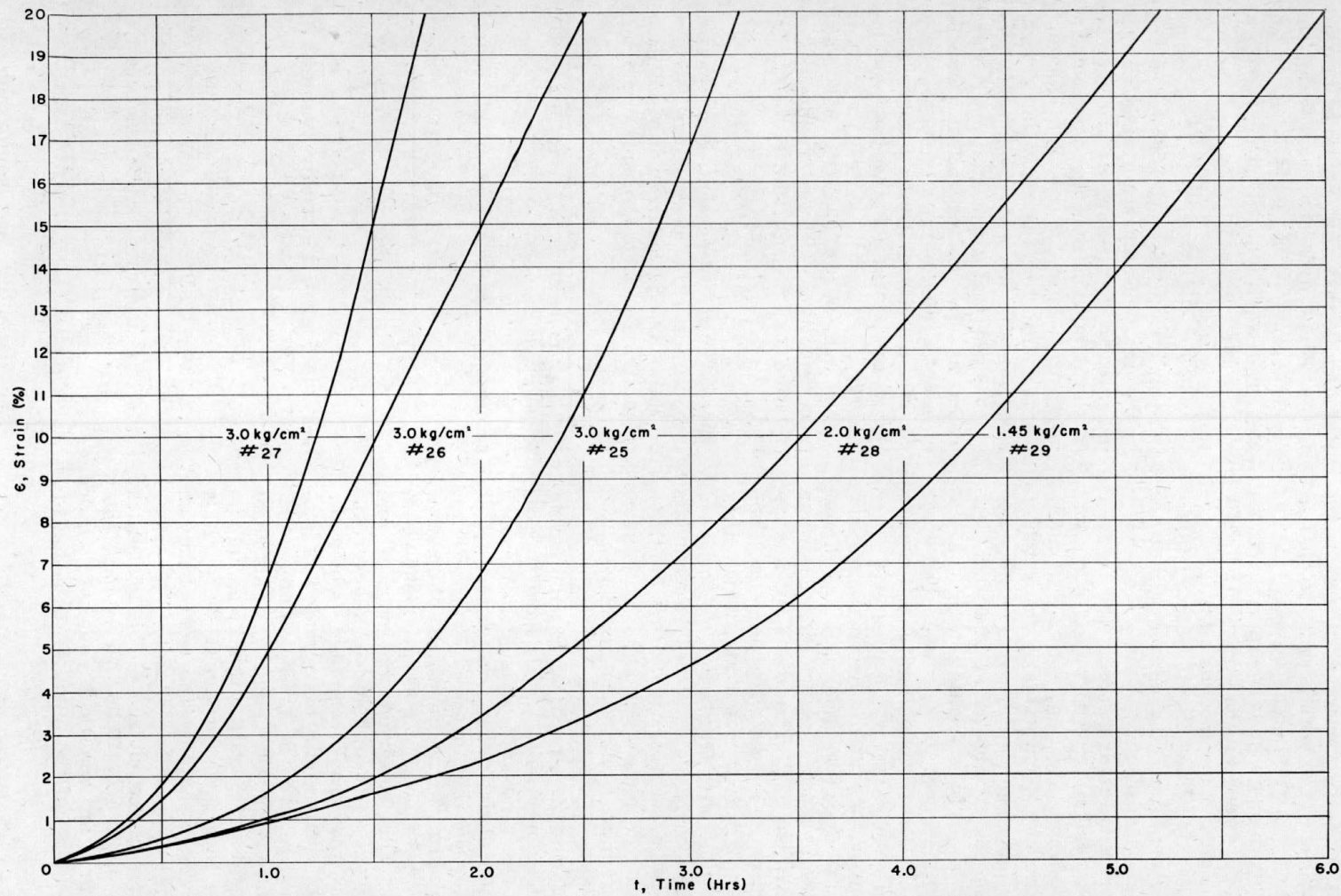


FIGURE 11. CREEP OF ARTIFICIAL ICE CRYSTALS, AT  $-1.0^{\circ}\text{C}$   
Specimens inclined  $45^{\circ}$  to  $c$  axis.

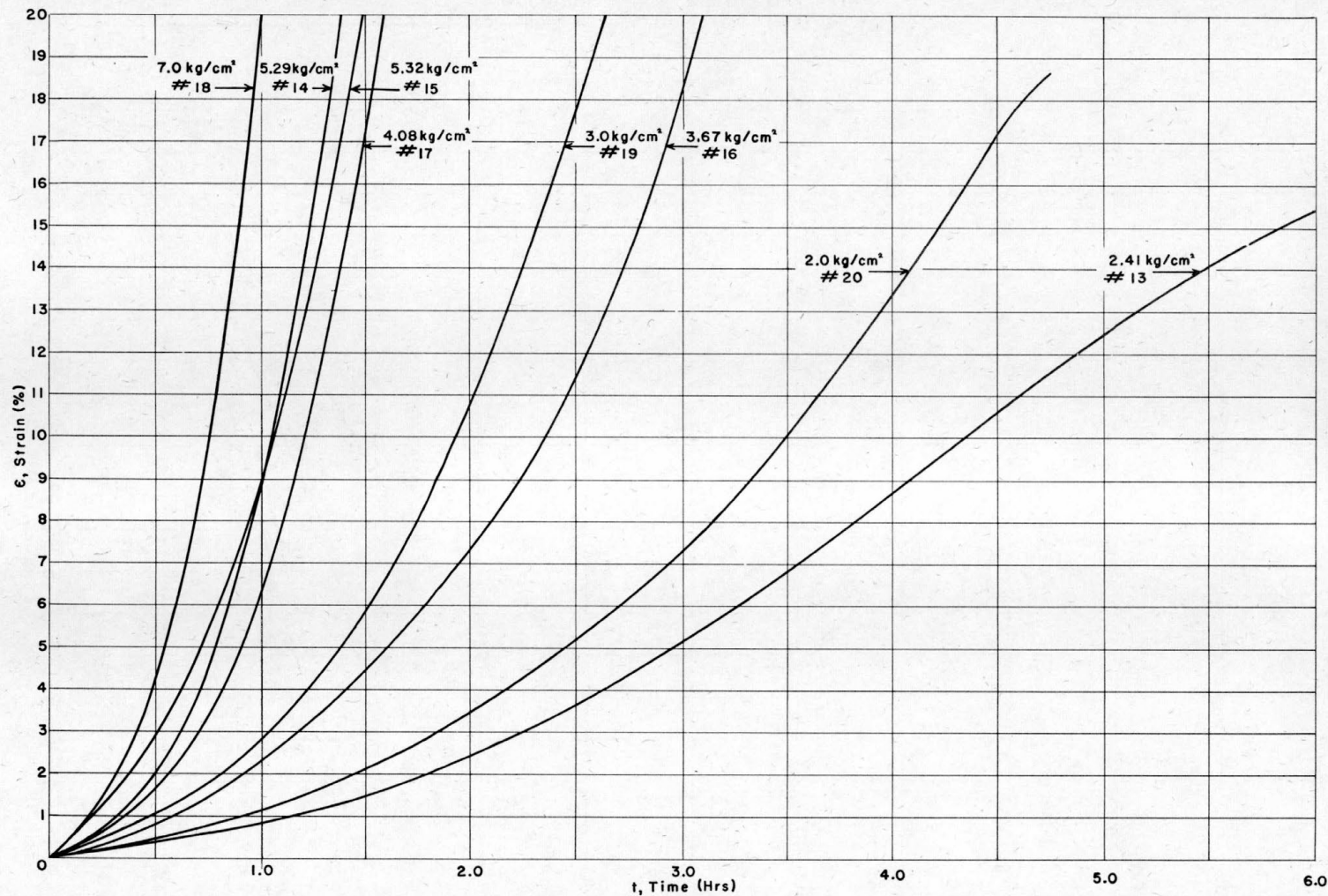


FIGURE 12. CREEP OF ARTIFICIAL ICE CRYSTALS, AT 2.9°C  
Specimens inclined 45° to *c* axis.



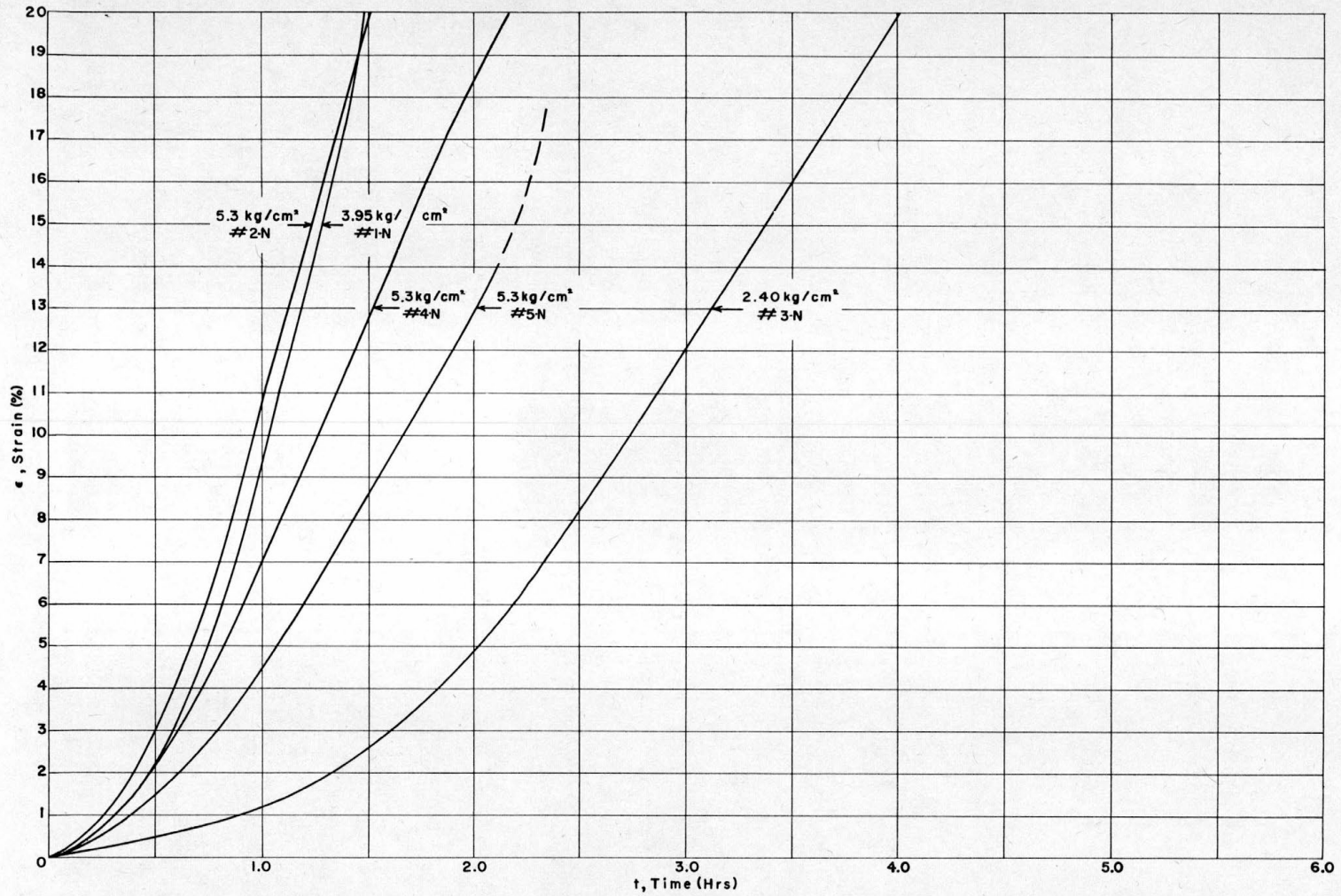


FIGURE 13. CREEP OF NATURAL ICE CRYSTALS, AT  $-2.9^{\circ}\text{C}$   
Specimens inclined  $45^{\circ}$  to  $c$  axis.

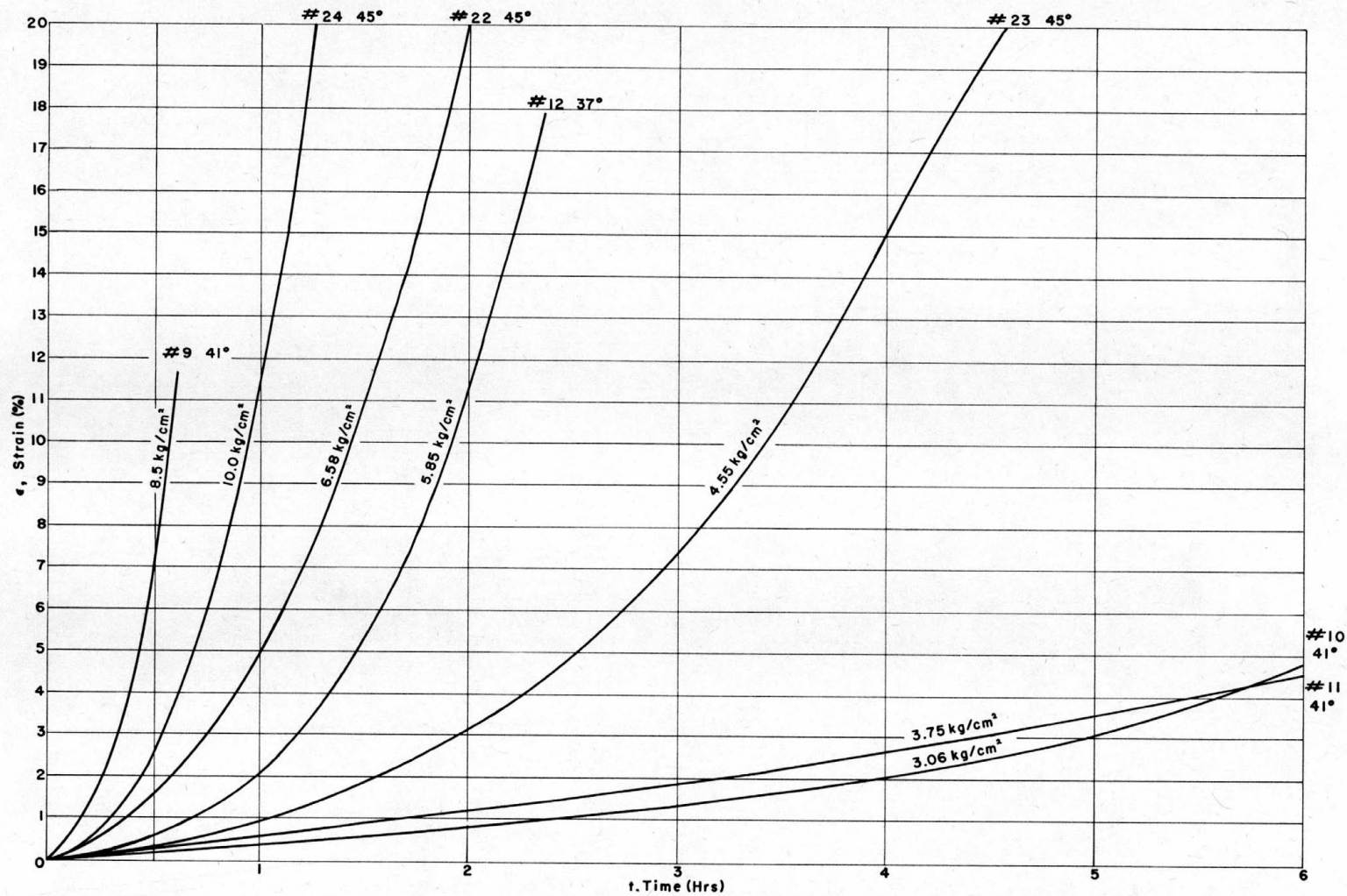


FIGURE 14. CREEP OF ARTIFICIAL ICE CRYSTALS, AT  $-10^{\circ}\text{C}$  TO  $-11^{\circ}\text{C}$   
Inclination to  $c$  axis indicated for each specimen.



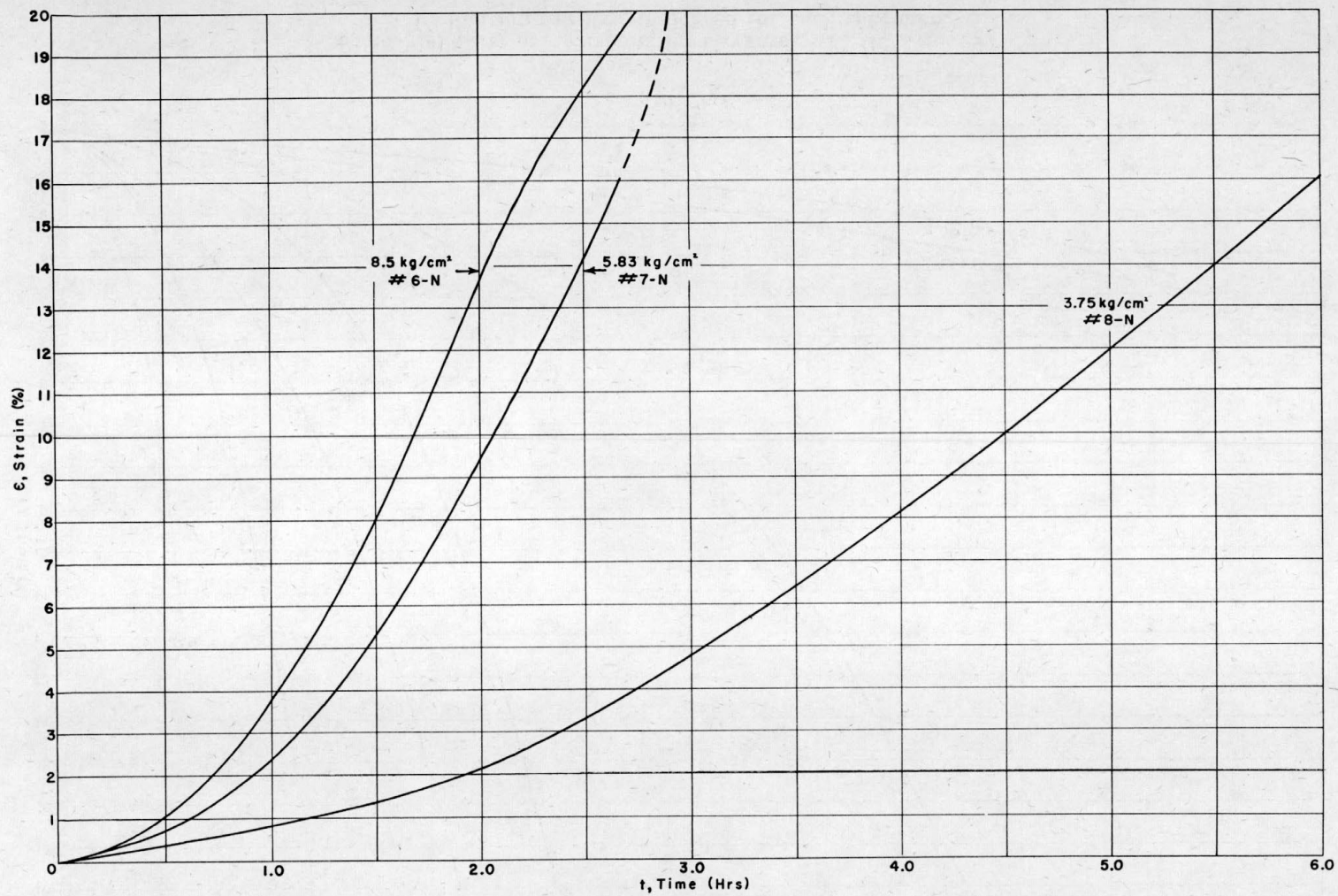


FIGURE 15. CREEP OF NATURAL ICE CRYSTALS, AT  $-11.0^{\circ}\text{C}$   
Specimens inclined  $45^{\circ}$  to  $c$  axis.

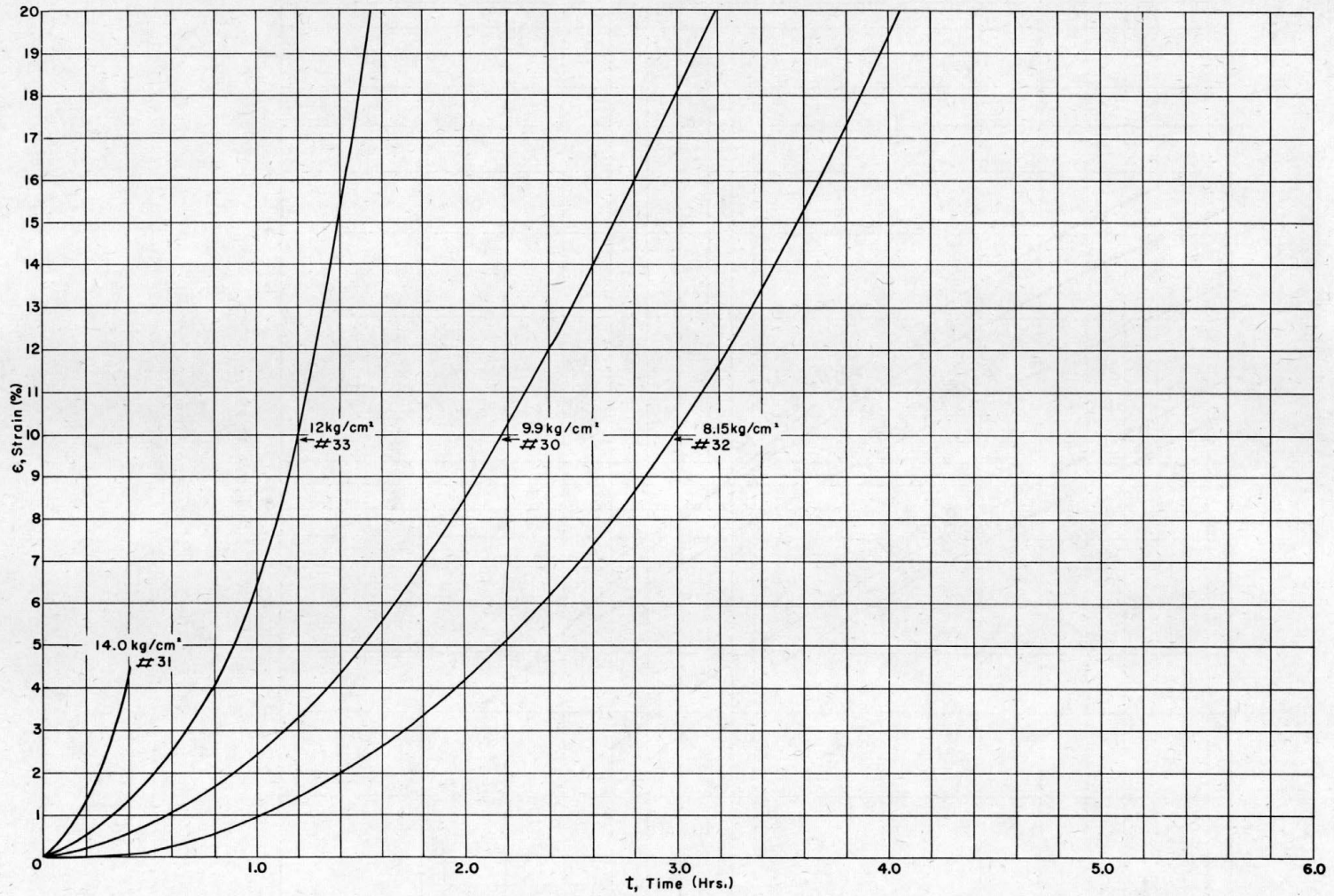


FIGURE 16. CREEP OF ARTIFICIAL ICE CRYSTALS, AT  $-18.0^{\circ}\text{C}$   
 Specimens inclined  $45^{\circ}$  to  $c$  axis.



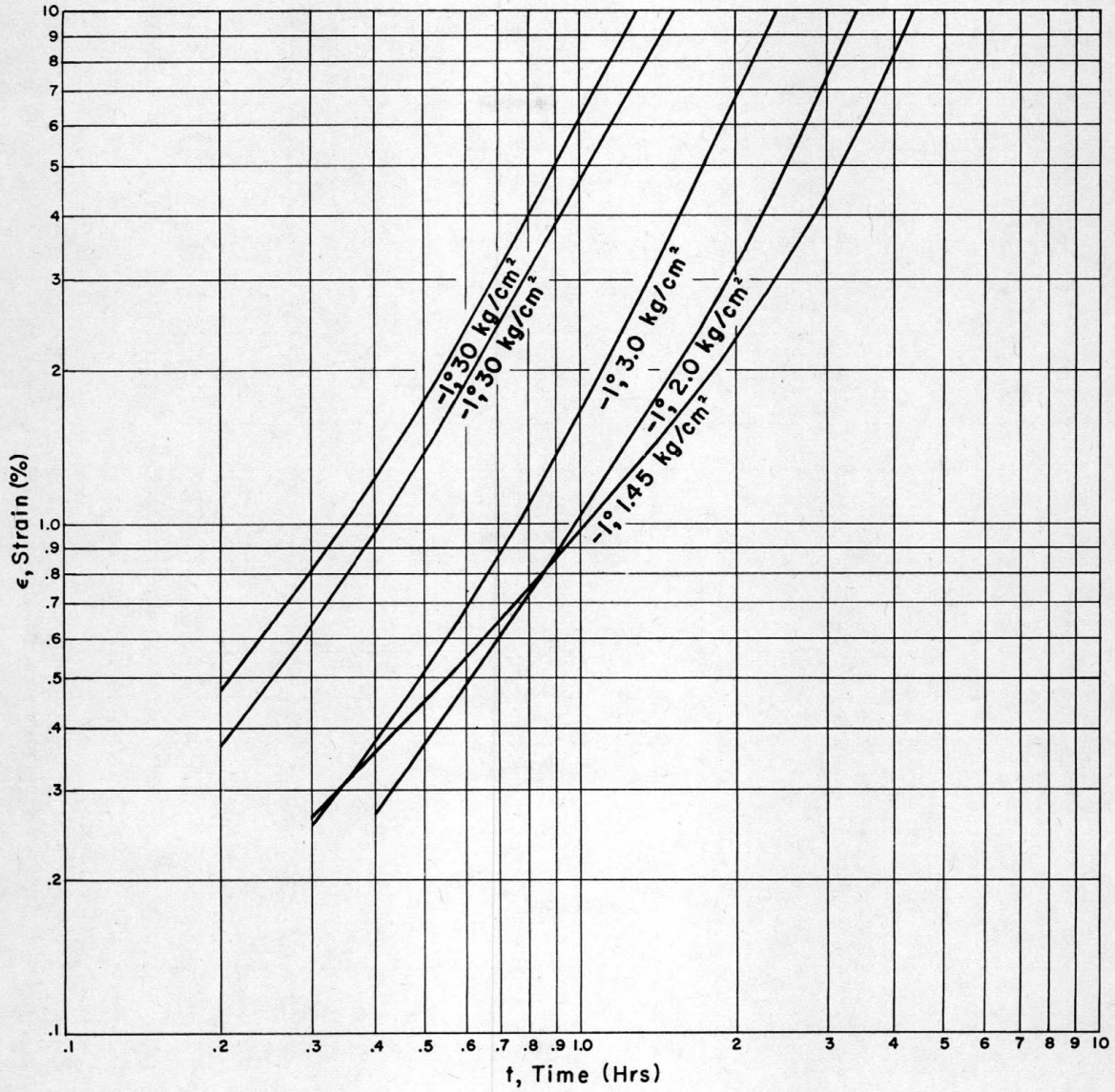


FIGURE 17. CREEP CURVES

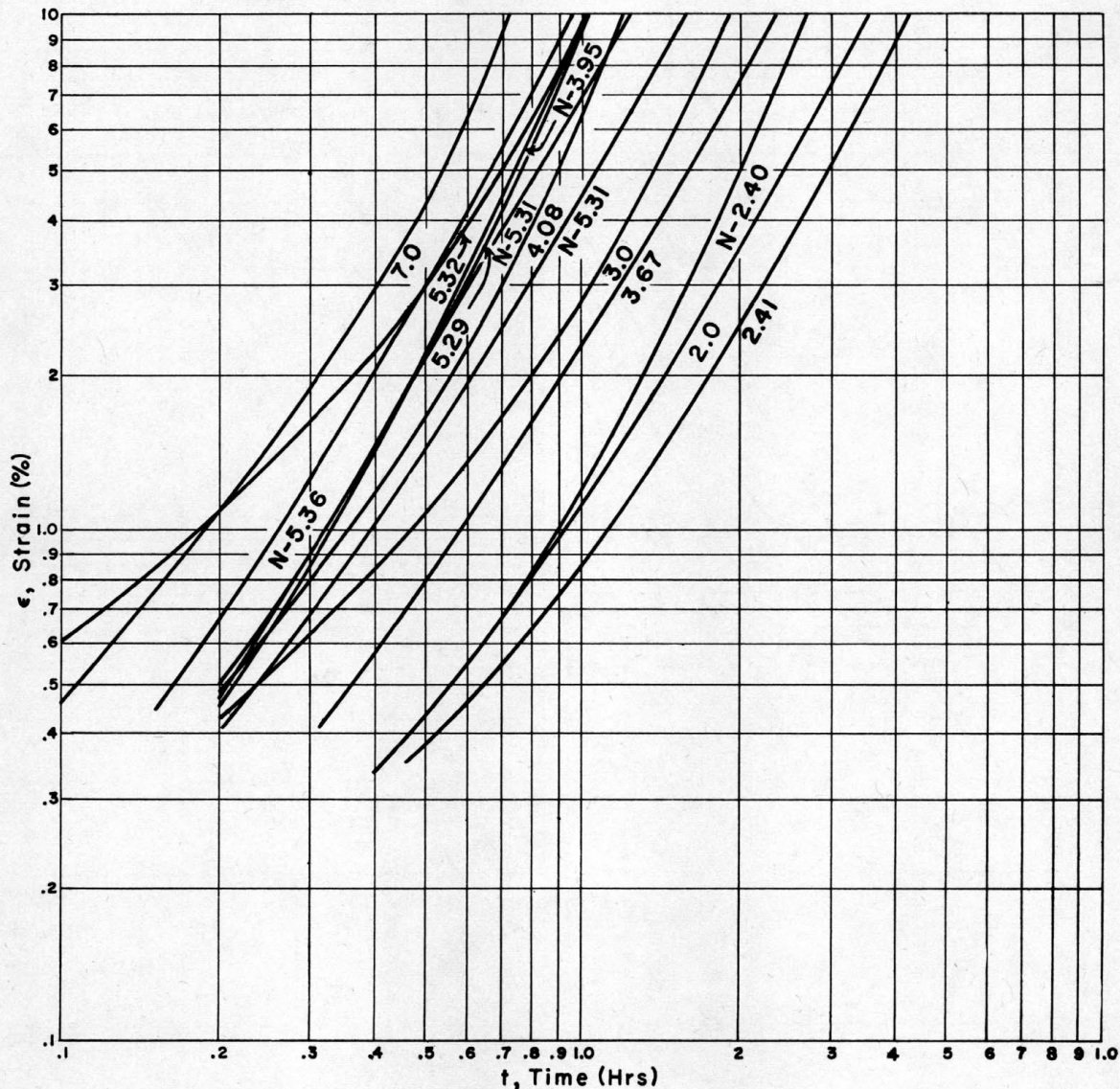


FIGURE 18. CREEP CURVES, AT  $-2.9^{\circ}\text{C}$   
 N indicates natural ice crystal.



## CREEP OF SINGLE CRYSTALS OF ICE

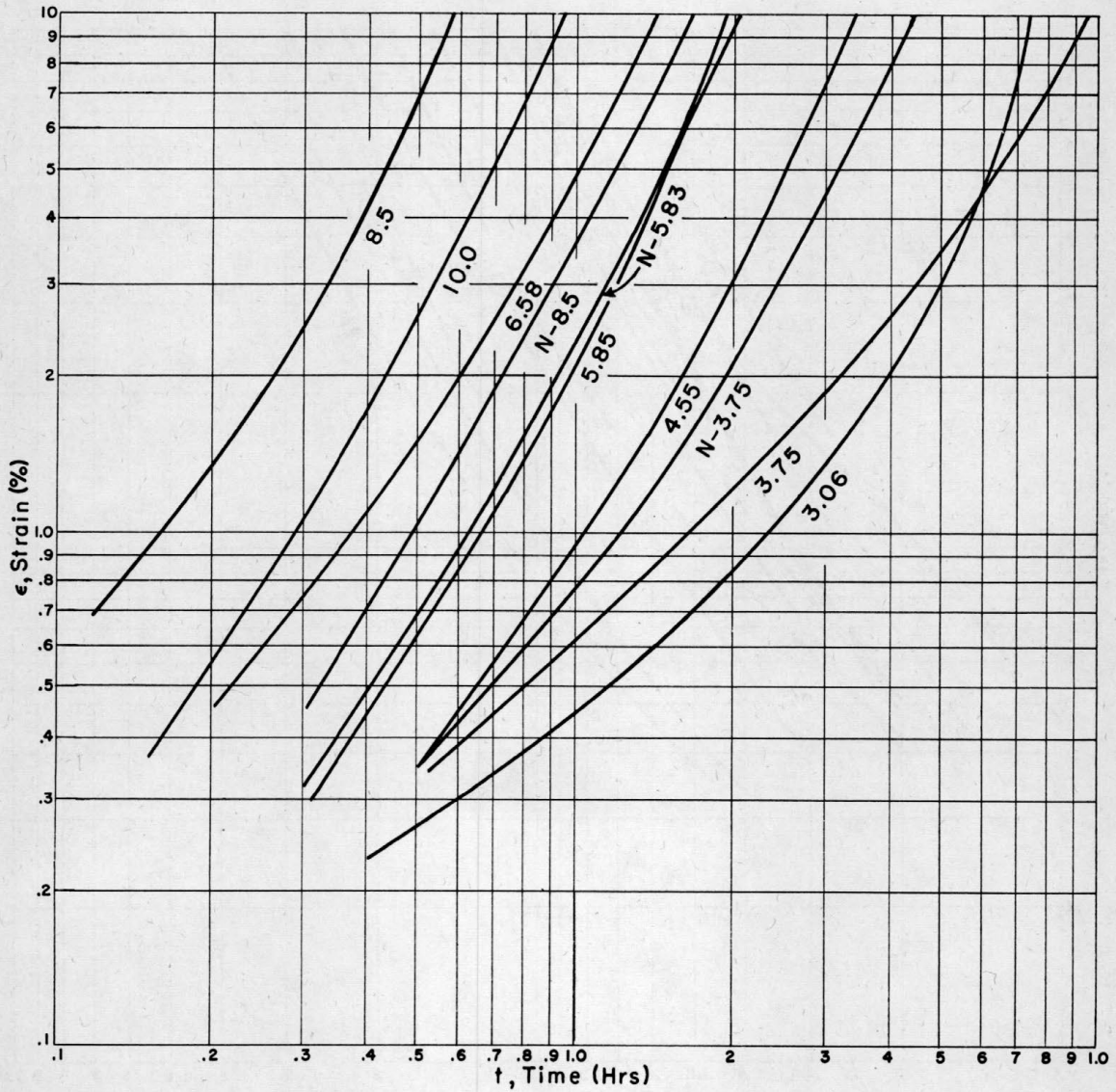


FIGURE 19. CREEP CURVES, AT  $-11^{\circ}\text{C}$   
 N indicates natural ice crystal.

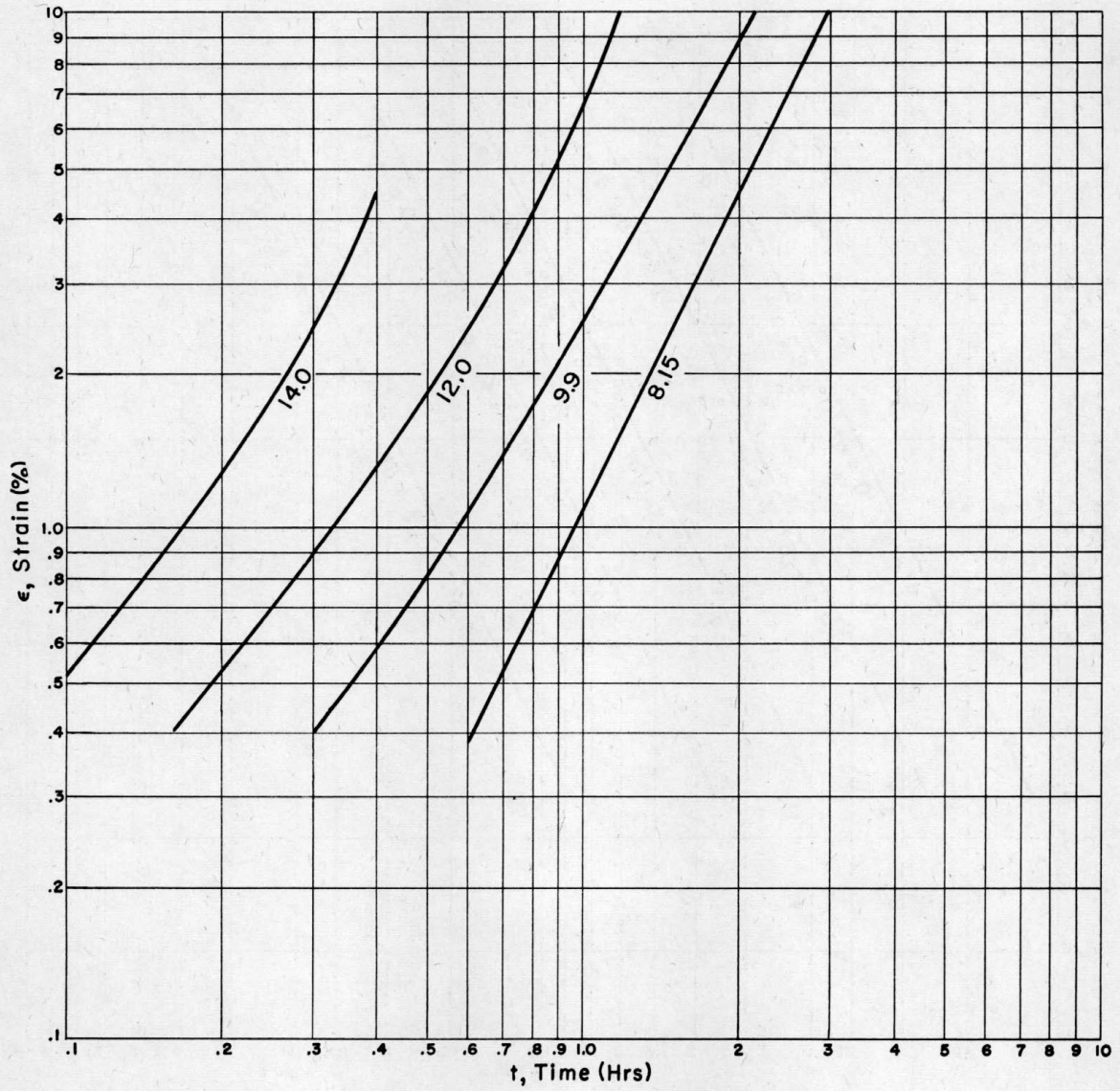


FIGURE 20. CREEP CURVES, AT  $-18^{\circ}\text{C}$



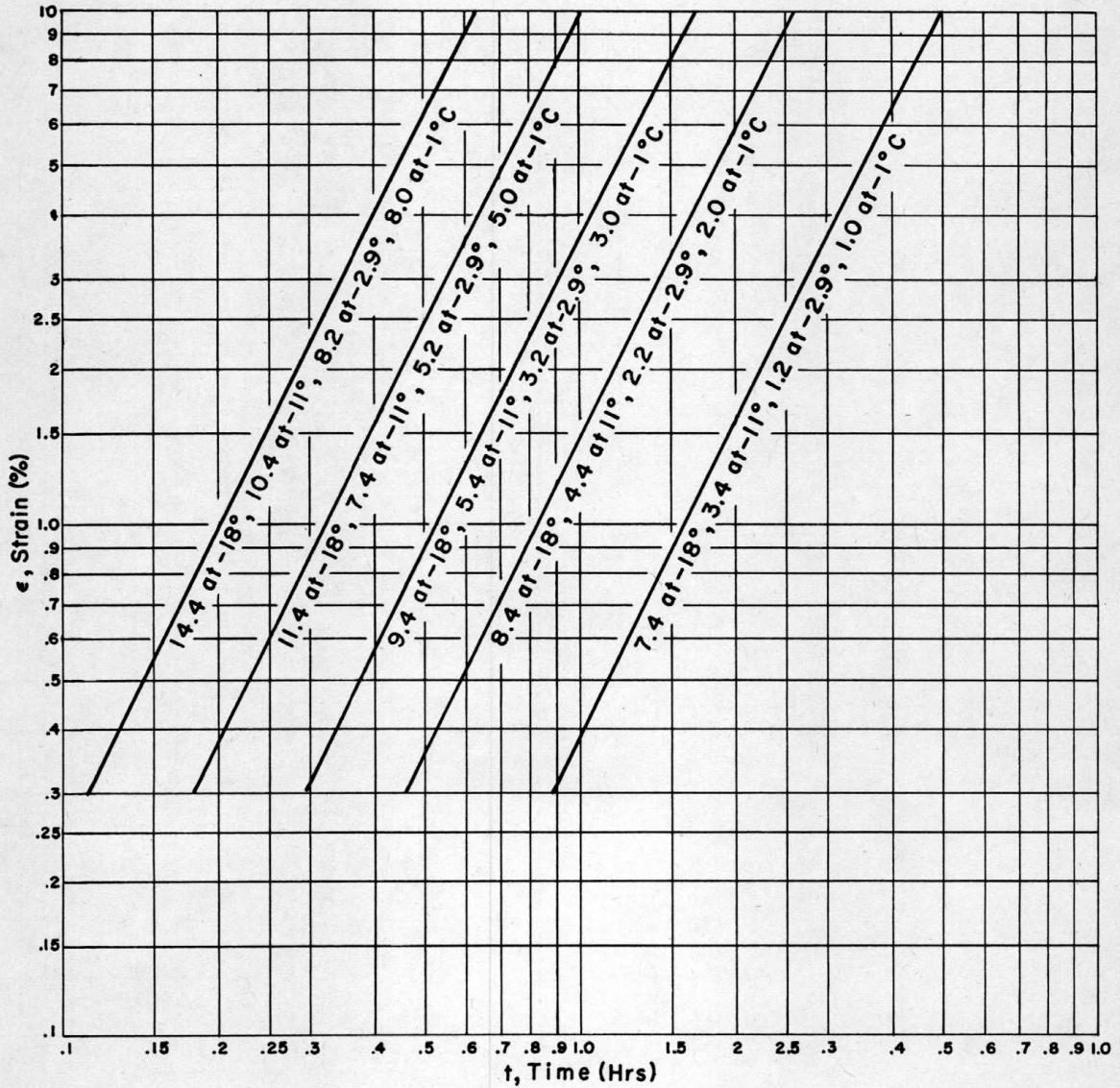


FIGURE 21. CREEP CURVES CALCULATED FROM EQUATION 2

The mechanism of deformation of these single crystals in creep seems to have been translation on the basal plane. The evidence for this is two-fold: (1) When the load is applied normal to the *c* axis, so that the resolved shear stress on the basal plane is nearly zero, the crystals creep much slower than when the load is applied at 45° to the *c* axis, so that the maximum resolved shear-stress coefficient is a maximum. (2) The deformation usually produces surface markings parallel to the basal plane, and sometimes produces offsets which leave no doubt that these surface marks are traces of the translation plane.

The appearance of the specimens after deformation is of some interest. Figures 22-31 give the best illustration of the various effects observed. In experiment 21 (Fig. 22), the specimen was apparently mounted eccentrically so that the deformation did not follow its usual course. This experiment was discarded. Figures 23 and 24 show respectively 28 and 21% deformation. Besides the large local offset on the translation planes, they show the effects of sublimation which has rounded the offset corners. Figures 25-28 show typical specimens with localized translation offsets, sublimation at a minimum, even in Figure 25 which was done at -1°C. Figures 29-31 show distributed translation. The deformation in these specimens is of the same magnitude as that in Figures 25-28. There would appear to be a possibility that, in the case of Figures 29-31, there was some pressure melting at the platen contacts. We can not rigorously exclude this, but the ice observed on the platen is mainly from the water which was used to freeze the specimen to the platen.

It should be noted that the highly deformed shape of these specimens is a fairly late feature of the test. In no case were surface markings visible at less than 3% strain, and usually they were not visible until about 10% strain. The late stages of the deformation in some cases (e.g. Figs. 25-28) seem to have been localized on thin translation zones, while the early stages of deformation were not. Hence, although it is clear that our creep curves have no particular meaning at 20% strain, the method should give good results at low strains.

All of the experiments are listed in chronological order in Table II. Experiments 1-N to 8-N used glacier crystals.

TABLE II. EXPERIMENTS

Experiment Number	$\sigma$ kg/cm <sup>2</sup>	T °C	Orientation	$\epsilon$ max %	t max hrs
1	.4	-3.2°	1c	.06	.9
2	1.9	-2.0	1c	0	2.2
3	2.8	-3.2	1c	2.9	22.5
4	2.8	-3.2	1c	5.0	6.1
5	3.2	-2.5	1c	.03	35.0
6	6.2	-4.0	1c	2.7	5.2
7	6.5	-10.0	1c	.7	55.4
8	10.2	-9.5	1c	2.9	3.2
9	8.5	-11.	41° ^ c	11.7	.62
10	3.1	-10.3	41°	9.6	7.4
11	3.7	-10.5	41°	11.0	10.0
12	5.8	-10.5	37°	17.9	2.4
13	2.4	-2.9	46°	14.9	5.8
14	5.3	-2.8	51°	23.5	1.5
15	5.3	-3.0	40°	21.0	1.5
16	3.7	-2.6	46°	27.8	3.4
17	4.1	-3.0	45°	24.4	1.7
18	7.0	-2.9	45°	21.0	1.0
19	3.0	-2.9	45°	20.1	2.7
20	2.0	-2.9	45°	18.7	4.8
*21	6.5	-10.8	45°	5.8	.8
22	6.6	-11.0	45°	21.0	2.0
23	4.5	-10.9	45°	20.2	4.6
24	10.0	-11.0	45°	21.6	1.3
25	3.0	-1.0	45°	21.5	3.2
26	3.0	-1.0	45°	20.7	2.6
27	3.0	-1.0	45°	21.0	1.8
28	2.0	-1.0	45°	20.0	5.2
29	1.4	-1.0	45°	21.4	6.1
30	9.9	-18	45°	20.1	3.2
31	14.0	-18	45°	4.5	.4
32	8.2	-18	45°	20.1	4.1
33	12.0	-18	45°	20.4	1.5
1-N	3.9	-2.9°	45°	20.5	1.5
2-N	5.3	-2.9°	45°	20.9	1.5
3-N	2.4	-2.9°	45°	20.0	4.0
4-N	5.3	-2.9°	45°	20.4	2.2
5-N	5.3	-2.9°	45°	17.7	2.3
6-N	8.5	-11.0°	45°	20.2	2.8
7-N	5.8	-11.0°	45°	21.0	3.0
8-N	3.8	-11.0°	45°	16.1	6.0

\*This experiment was discarded.

CONCLUSIONS

Our experiments show a new type of creep curve for ice single crystals tested in compression at constant stress. The rate of strain continuously increases with increasing time, yielding a parabolic curve. The data are all consistent with the following expression

$$\epsilon = a[(\sigma - bT^2)t]^2.$$

The dominant mechanism of deformation is translation on the basal plane. There is no evidence of any other mechanism of deformation.

The reproducibility of the data is poor—about 25 percent average deviation in time. The cause of this lack of reproducibility is not known completely, but it is clear that any further work in this field should include a complete knowledge of the crystal orientation rather than just the orientation of the *c* axis, as was the case in our experiments. This requires x-ray determination or the measurement of crystal faces, which can be produced



by etching. Also, greater precautions should be taken with alignment of the specimens.

The single crystals grown by the modified Bridgman method seemed to be nearly perfect by our optical tests and behaved in creep similarly to the crystals of glacier ice supplied to use by SIPRE. Future work should include more stringent tests of the crystals.

We found it possible to do effective work on ice single crystals in a commercial deep-freeze unit. There seems to be no necessary operation that cannot be performed in such a unit. Use of the deep-freeze, however, is sufficiently awkward and fraught with difficulty that we would heartily recommend a well equipped coldroom such as those at SIPRE whenever possible.

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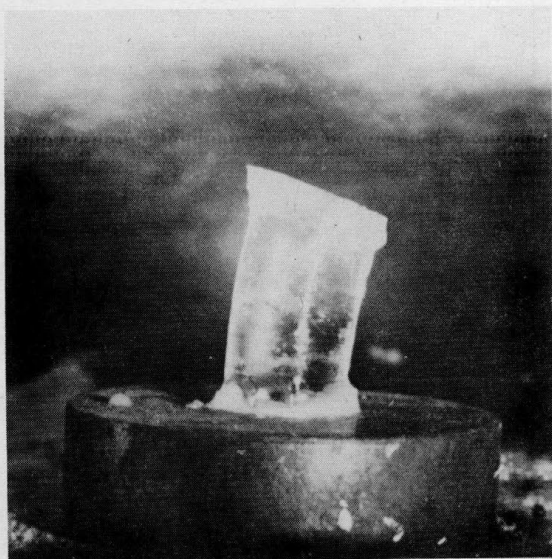


FIGURE 22. EXPERIMENT 21  
Eccentric loading.

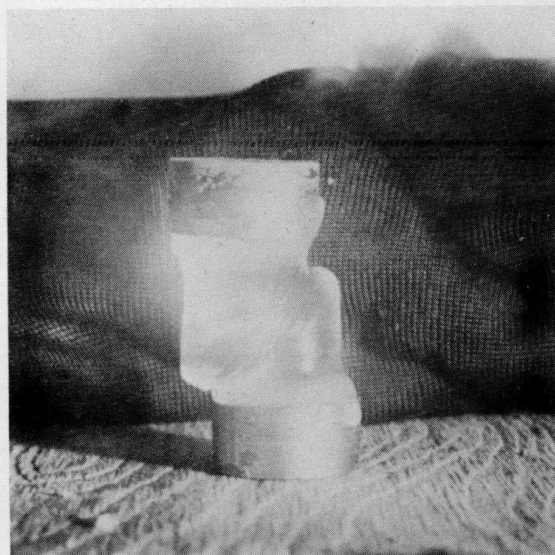


FIGURE 23. EXPERIMENT 16  
28% shortening at  $-2.6^{\circ}\text{C}$ ;  $\sigma = 3.7 \text{ kg/cm}^2$ .

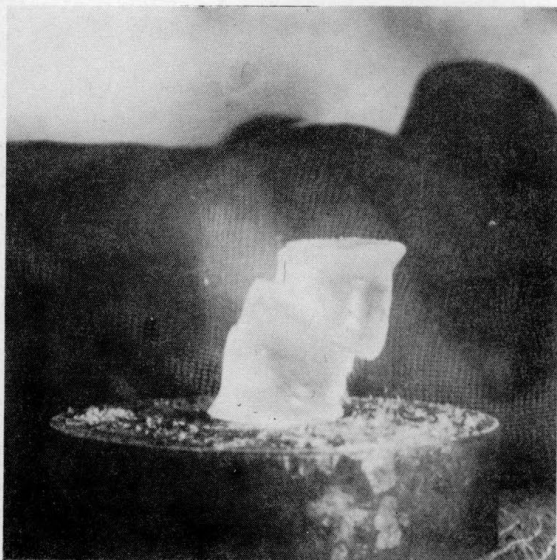


FIGURE 24. EXPERIMENT 15  
21% shortening at  $-3.0^{\circ}$ ;  $\sigma = 4.3 \text{ kg/cm}^2$ .

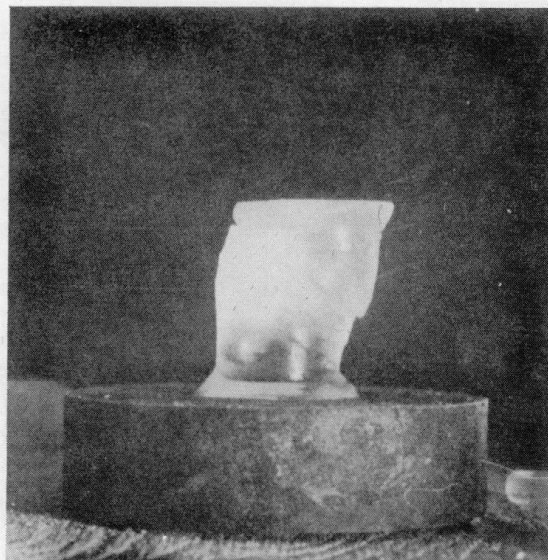


FIGURE 25. EXPERIMENT 25

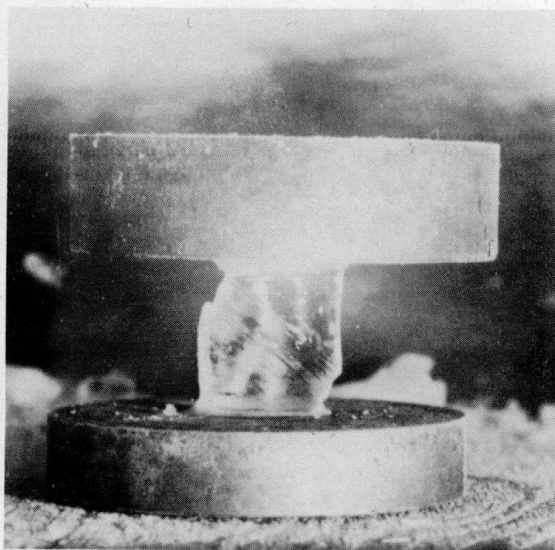


FIGURE 26. EXPERIMENT 22

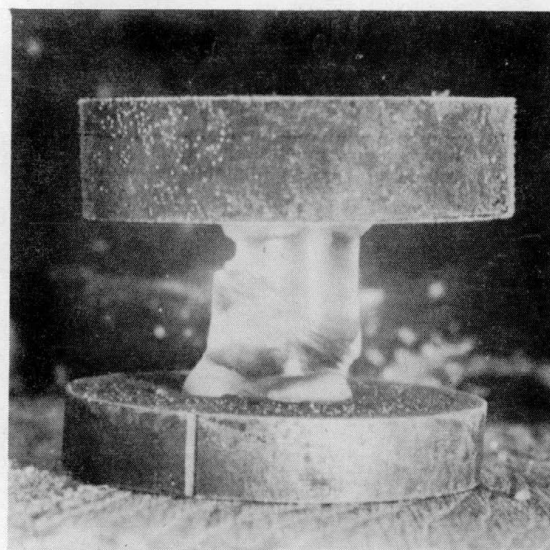


FIGURE 27. EXPERIMENT 20



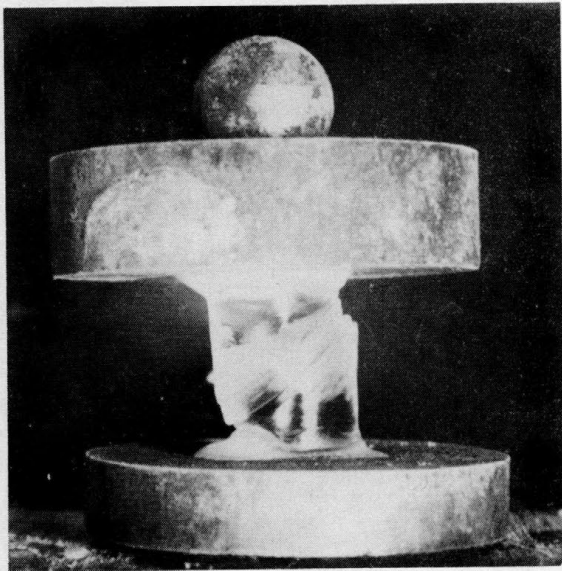


FIGURE 28. EXPERIMENT 19



FIGURE 29. EXPERIMENT 27



FIGURE 30. EXPERIMENT 30

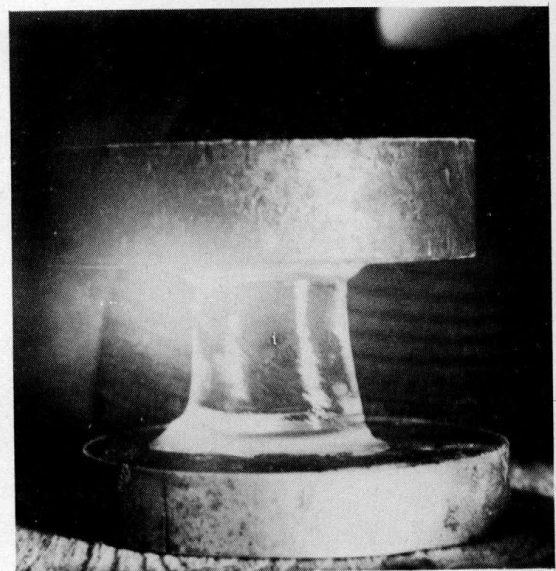


FIGURE 31. EXPERIMENT 14