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HARDNESS OF SINGLE ICE CRYSTALS

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Abstract

Brinell and scratch hardness tests were made on single ice crystals. The results of these measurements show that the hardness of single ice crystals increases with decreasing temperature; the Brinell hardness numbers range from about 4 at −5° C. to 17 at −50° C. The greatest increase in the hardness values occurs at the higher temperatures. The temperature dependence of the scratch hardness was similar to that of Brinell hardness. An anisotropism of hardness is evident; the single ice crystal is harder parallel to the *c*-axis than in the direction normal to the *c*-axis. An apparent difference in surface structure with respect to orientation was noticed during the scratch hardness tests. A consistent wavy scratch was produced normal to the *c*-axis, while the scratch parallel to the *c*-axis was always straight.

Introduction

Two methods for determining the hardness of single ice crystals were used in the measurements discussed in this paper. One series of tests employed the Brinell hardness tester, the other the Microcharacter. The Brinell principle involves forcing a hardened steel ball into the specimen under a definite pressure. The dimensions of the impression thus obtained form the basis for calculating the hardness. With the Microcharacter instrument, the hardness of the material tested is a function of the width of the microcut made on the test surface as it is moved along under a diamond edge which bears down on the surface with a constant pressure.

No detailed work on the Brinell hardness and the microhardness of ice can be found in the literature except for some rather crude experiments by Andrews described in Barnes (1928). Moor (1940) and Blackwelder (1940) conducted experiments on scratch hardness of crystal aggregates using the Moh's scale.

The work described in this paper was undertaken to determine how the hardness of ice varies with temperature, and also to determine whether there is appreciable anisotropism with respect to hardness of single ice crystals. The single ice crystals used in these tests were originally quite large, weighing 2–3 pounds, and were obtained from the Mendenhall Glacier near Juneau, Alaska.

Experimental Procedure

For measuring Brinell hardness of single ice crystals, an Olsen Baby Brinell Hardness Tester was used. This apparatus as delivered by the manufacturer was developed primarily for testing soft metal specimens, which could not be satisfactorily tested by applying the standard Brinell loads under standard Brinell conditions. It operates on the Brinell principle but with light loads and a small ball penetrator. The machine comes equipped with a 1/16-inch ball to be used with a load of 12.61 kg. The load is applied directly from dead weights to the ball stem. Preliminary tests with ice samples showed that this load caused excessive cracking of the ice, so that the machine had to be modified thus: (1) the 12.61 kg. load was replaced by a platform on which different weights could be placed, and (2) the $\frac{1}{16}$-inch ball was replaced by a hardened $\frac{1}{8}$-inch ball. The weight of the ball stem plus platform was 570 g., which was added to the weights placed on the platform to give the total load applied.

The speed of loading, that is, the speed with which the indentor and the specimen approach each other immediately prior to contact, is quite important in order to get satisfactory and consistent readings. Bergsman (1948) states that the loading speed should be kept as constant as possible, and should be given with the hardness number. Therefore, the hardness tester was equipped with a 1800 rpm synchronous motor with suitable gear reductions to provide a loading speed of about 9 mm. per minute.

The test blocks of ice were cut from large single ice crystals into rectangular plates of approximately $7 \times 4 \times 1$ cm, oriented so that the *c*-axis lay parallel to the large face of one plate and normal to the large face of the other. The ice plates were machined plane parallel, polished to a mirror finish on a piece of fine silk stretched across a hard surface, and mounted on a piece of plate glass by heating the glass and allowing the sample to melt slightly and then refreeze onto the glass.

After a series of indentations were made, one of the most obvious physical processes was the piling up of the material around the edge of the imprint. Since this would make the diameter of the penetration appear larger than it actually was, this ridge was polished off on silk before the diameter was measured.

All measurements of diameter were made with a filar micrometer mounted on a microscope with a 32 mm. objective. Temperature measurements were made with a copper-constantan thermocouple placed on the surface of the ice within a few millimeters of the ball penetrator. Temperature varied up to 0.25° C. in any one series of measurements.

Williams (1942) in his book on hardness measurement states that comparative Brinell hardness numbers should be used only as determined by a special table of numbers furnished with the machine. However, since the machine was modified for the purposes already stated, Brinell hardness numbers were computed from the standard Brinell formula:

$$H = \frac{K}{\pi D/2(D - \sqrt{D^2 - d^2})}$$

where

H is the Brinell hardness number
K is the applied load in kilograms
D is the diameter of steel ball in mm.
d is the diameter of the impression in mm.

All the tests were conducted in cold rooms in which any temperature down to −55° C. could be obtained. The actual temperatures were chosen arbitrarily, and enough points were obtained to be able to plot the curves shown in the data section.

The Microcharacter, manufactured by the Spencer Lens Company, was used to determine the microhardness of single ice crystals. A 9 g. weight was used during this series of tests. The formula developed for the instrument is:

$$k = \frac{4 \times 10^4}{\lambda^2} \text{ with the 9 g. weight}$$

where

k is the microhardness number
λ is the width of the cut in microns
4×10^4 is an arbitrary number.

The width of the microcut is given in microns and is measured with a filar micrometer eye-piece of the microscope.

Because the microcuts were made at temperatures as low as −50° and it was necessary to measure the width of the line at a higher temperature (−5° C.), there was considerable fogging due to condenstation on the ice specimens. This condensed vapor quickly obliterated the lines, making it impossible to measure them. Consequently, the following method was developed. The specimen was coated with liquid polystyrene, which was allowed to harden and then peeled off. The exact replicas of the microcuts were thus impressed on the coating. These replicas were then measured for the results obtained.

Data

Because of the necessity of modifying the Baby Brinell Hardness Tester as described in the previous section, it was necessary to verify that the hardness values obtained, as calculated from the standard Brinell formula, are independent of ball diameter and load. The hardness values, within the limits of accuracy of the apparatus, are nearly identical for the $\frac{1}{16}$-inch ball and the $\frac{1}{8}$-inch ball. A slight general difference in hardness is probably due to the fact that the average temperature was 0.5° C. higher during the time the measurements were made with the $\frac{1}{8}$-inch ball.

Different loads were tried to determine how small a load was necessary to prevent cracking. It was found that a 2 kg. load placed on the platform could be used through the temperature range for the penetrations parallel to the *c*-axis, while it was necessary to reduce the load to 1 kg. for penetration normal to the *c*-axis at temperatures above −20° C. Tests were run to check the independence of calculated Brinell hardness values in

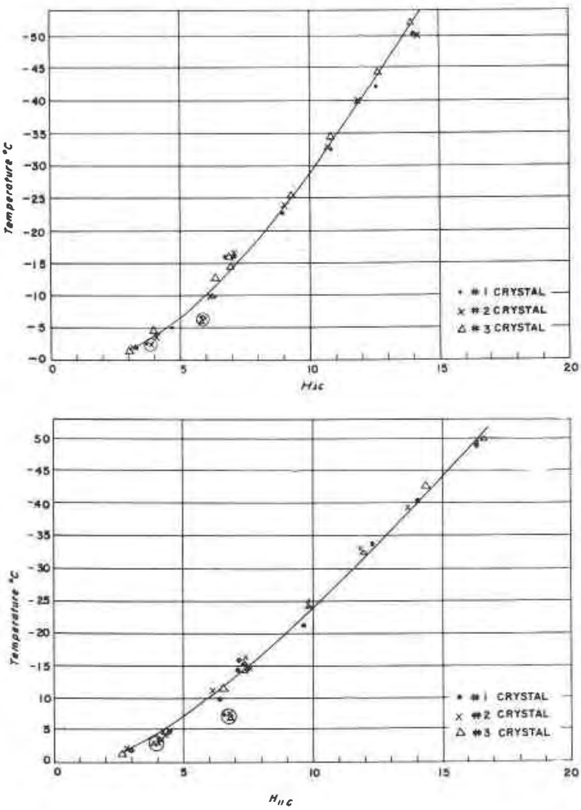

FIG. 1. Brinell hardness of single ice crystals vs. temperature. Twelve second application of load. (Above) Normal to *c*-axis. (Below) Parallel to *c*-axis.

relation to the load. This showed that the hardness is independent of the load within the limit of accuracy of the apparatus used.

After these preliminary tests were completed experiments were carried out over a temperature range of −5° to −50° C., with 10 impressions at each of six temperatures; and for times of full load applications of 1, 5, 10, 15, and 30 seconds. The load was applied manually. Analysis of the data obtained showed that the hardness is anisotropic in the two directions, parallel and normal to the *c*-axis. The spread of hardness values was greatest at one-second application of load, as could be expected since this is where the error in measurement is greatest.

Investigation of the possible anisotropism of single ice crystals with respect to parallelism or normality to the secondary crystallographic axis (*a*-axes) was made. No difference in hardness was evident.

At this point during the tests, it was decided to mechanize the hardness tester so that a constant loading speed could be maintained, thereby reducing the spread of the hardness values obtained. Samples were cut

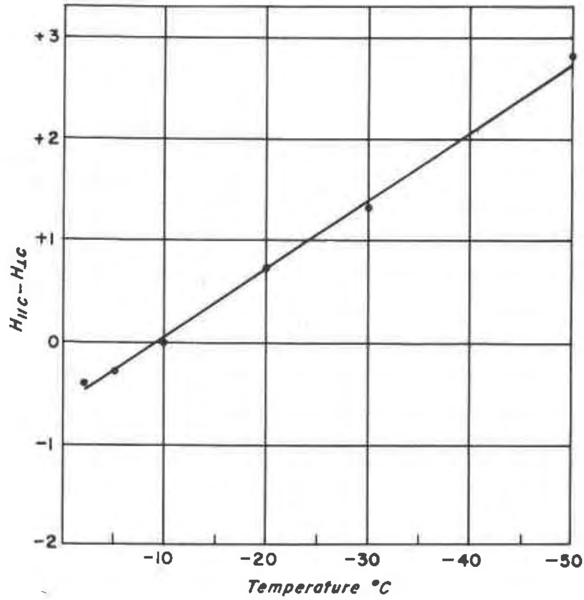

FIG. 2. Difference in Brinell hardness of single ice crystals parallel and normal to *c*-axis.

normal and parallel to the *c*-axis from three different single ice crystals, in order to be able to compare their hardness. It was found most convenient to use a 12 second application of load. Figure 1 shows the results of these trials, and Fig. 2 shows a definite anisotropism of hardness. Upon examination of Fig. 1 it can be seen that there are two series of hardness values for temperatures above $-20°$. The first series (circled) yielded values of hardness higher than expected. These points were rechecked to rule out a possibility of error in measurement. It was found that some unexplainable error appeared in the first series of measurements. These points were included in the graphs to show the magnitude of the errors.

Figure 2 shows that a crossover of the two curves for hardness normal and parallel to the *c*-axis probably exists at about −10° C. This was also evident in the tests where the load was applied manually. This crossover may indicate that the crystal is harder normal to the main axis below this range and harder parallel to it above this range of temperatures.

Experiments were conducted to check the possible polarity of single ice crystals and the possibility of twinning. No polarity of the main axis was detected in the crystal used. The tests were performed in numerical order, as is shown in Fig. 3. After a series of measurements were made

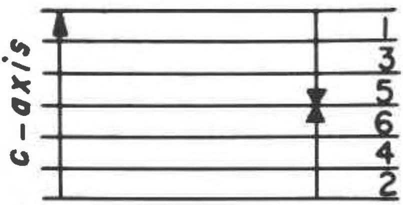

Fig. 3. Order of tests to check possible polarity and twinning.

on surfaces 1 and 2, the crystal was planed off about 2 mm. on both sides to new surfaces 3 and 4, on which the next series of measurements were made, and so on.

Scratch Hardness

The microhardness data were taken on two rectangular plates of approximately 2.5×2.5×1 cm. One of these plates had the *c*-axis oriented normal to its surface so that a scratch in any direction on this surface was normal to the axis. The other plate had the *c*-axis oriented parallel to the surface of the plate so that scratches at different angles to the *c*-axis could be made. Temperatures were chosen arbitrarily but included enough points to draw the curves on Figs. 4 and 5.

A peculiar phenomenon was noticed while measuring the scratches on the plate with the *c*-axis parallel to its surface. A photomicrograph (Fig. 6) shows that the scratches normal to the *c*-axis are wavy, while those parallel to the *c*-axis are quite straight. This phenomenon was particularly evident below −10° C. No apparent mechanical reason for this could be detected, for exactly the same thing happened on several different trials.

In an attempt to observe this phenomenon, a rigidly mounted steel mending needle was substituted for the diamond. The ice specimen with the *c*-axis lying in the plane of the surface was drawn past the needle,

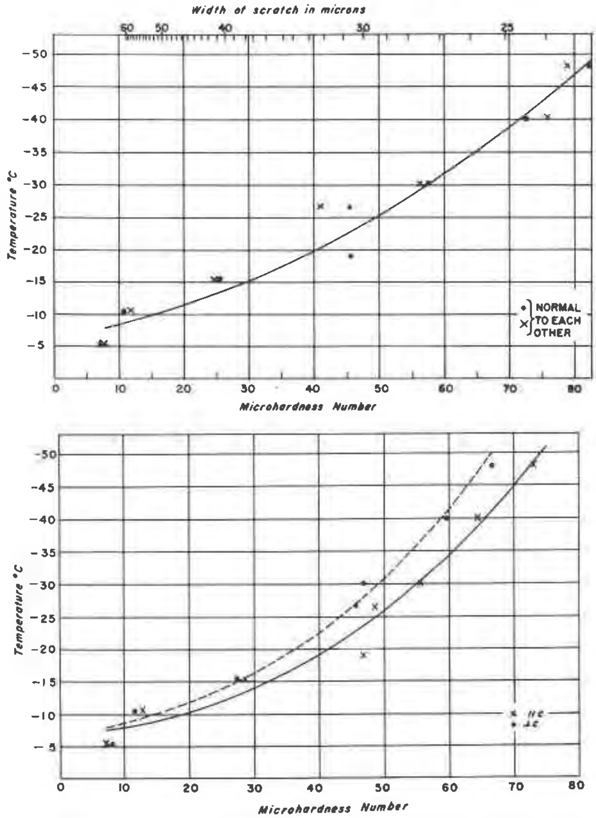

FIG. 4. Microhardness of single ice crystals. (Above) c-axis normal to surface. (Below) c-axis parallel to surface.

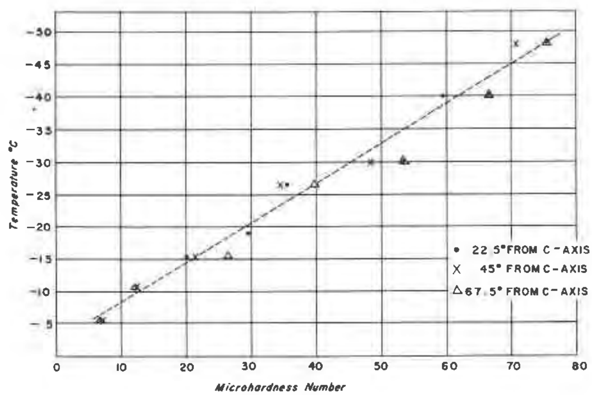

FIG. 5. Microhardness of single ice crystals at various angles to c-axis.

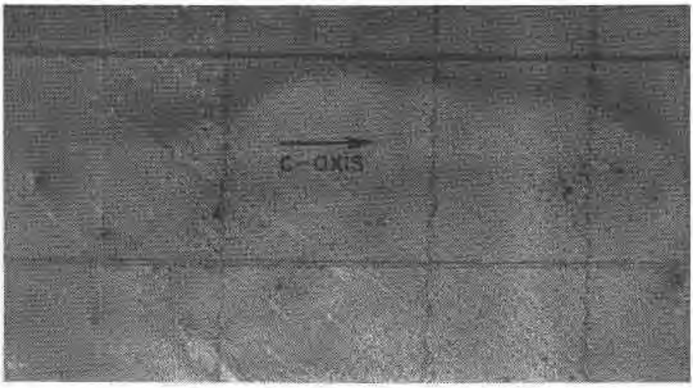

FIG. 6. Scratches made by microcharacter instrument on ice surface parallel and normal to *c*-axis.

first in the direction of the *c*-axis, then normal to this axis, leaving scratches on its surface. When these scratches were observed under the microscope, there was no evidence of the wavy line in either direction. However, all the scratches in these tests were quite irregular, leading to the conclusion that this was not a valid test.

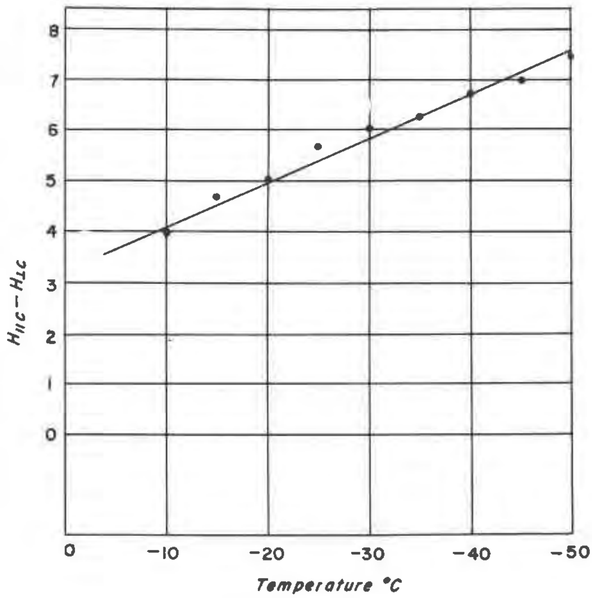

FIG. 7. Difference in microhardness of single ice crystal parallel and normal to *c*-axis.

Discussion of Results

Upon analysis of the data on Brinell and Microcharacter hardness of single ice crystals, the following conclusions were made:

(1) The hardness of ice increases with decreasing temperature. The Brinell hardness test is more reproducible and accurate than the scratch

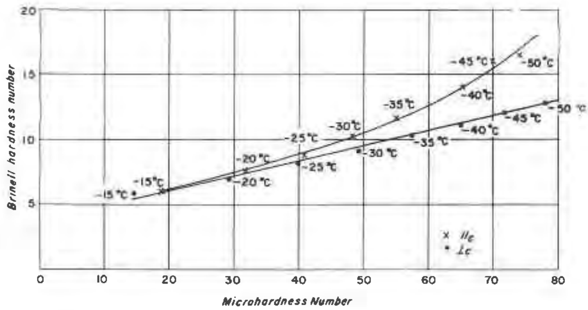

Fig. 8. Empirical relationship between Brinell and micro-hardness numbers, parallel and normal to c-axis.

test for measuring the difference in hardness with temperature. The micro-hardness data become less accurate at higher hardness (lower temperature) because a fraction of a micron difference in width of the scratch becomes quite significant. A scale at the top of Fig. 4 gives the difference in hardness values for decreasing width of cut.

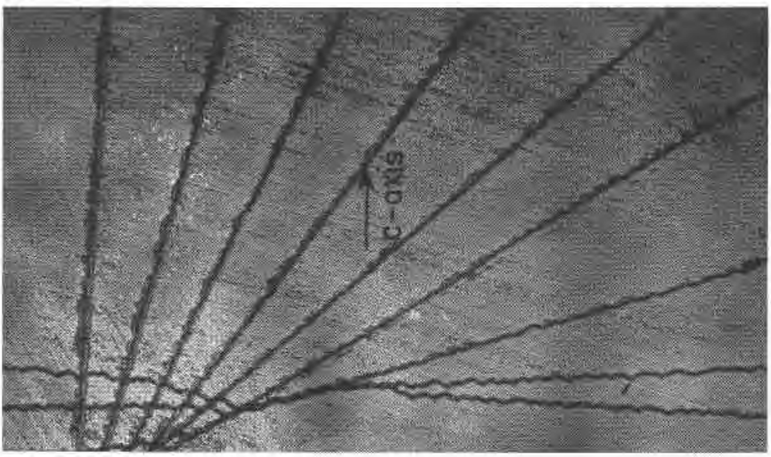

Fig. 9. Scratches made by microcharacter instrument on ice surface. Temperature −30° C. Magnification15.5×.

(2) There is an anisotropism of hardness with respect to the orientation of the *c*-axis. This is more evident from the Brinell hardness data than from the microhardness data. Brinell hardness $H_{\|_c} - H_{\perp_c}$ changes linearly with temperature. The value of $H_{\|_c} - H_{\perp_c}$ is negative at high temperatures, zero at approximately $-10°$ C., and positive at lower temperatures (Fig. 2). Anisotropism of hardness is also evident in the microhardness data (Fig. 7), though anisotropy is maximum where the accuracy of determination is least.

(3) A linear empirical relationship exists between the Brinell hardness numbers and the microhardness numbers normal to the *c*-axis, but the relationship between the Brinell and microhardness numbers parallel to the *c*-axis is not linear (Fig. 8).

(4) Apparently, some difference in the structure of the surface with respect to orientation of the single ice crystal consistently causes a wavy scratch normal to the *c*-axis but a straight scratch parallel to it. Figure 9 shows that waviness appears only in the scratches almost normal to *c*. No mechanical or other reason could be found for this phenomenon.

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