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# Pressures Developed during the Unidirectional Freezing of Water-Saturated Porous Materials

Experiment and Theory

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## Abstract

A new experimental apparatus is being used that permits the measurement of developing pressures during the freezing process in the environment of a small temperature gradient. Studies with pads of nearly uniform-size glass beads (in the micron range) indicate that after an ice lens has developed the ice-water interfacial energy, computed from the measured pressures and the pore geometry of glass bead pads, ranges between 30 and 35 ergs/cm<sup>2</sup>. The supporting theory assumes that such pressures arise because of the curved interfaces between the ice lens and water, ice and porous solid, and water and solid according to Everett's model.

Freezing experiments with pads of well-graded spheres (1-30  $\mu$ ) show the significance of the smaller pores in the porous system in determining the magnitude of pressures developed during ice lens growth. The study has been extended to soils and some common building materials.

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## I. Introduction

Frost heave experiments show that pressures developed at an ice-water boundary beneath an ice lens are related to the pore dimensions of the soil (Penner, 1958, 1963). In the absence of all confining pressures, the maximum suction (negative pressure) is developed in the water phase if the porous system does not have access to an external water supply. This suction is also related to the pore dimensions (Penner, 1957). It is possible also to halt ice lensing by a combination of suction and confining pressure (Penner, 1958).

The geometry of most porous materials is normally too complex for the investigation of theories that predict a precise relation between the heaving pressure induced by ice lensing and the geometry of the solid structure with its attendant pore system. The present paper describes freezing experiments with glass beads (which have only a small size variation and are in a close-pack array) to test more rigorously than has been done previously the prediction of heaving pressures by Everett and Haynes (1965). Based on the experimental technique devised and the agreement shown between theory and experiment, the studies have been extended to more complex porous systems, which contain a wide range in pore size distribution, in order to ascertain the significance of this size distribution on ultimate pressures. Such studies have been conducted in a preliminary way on a silty soil of known frost heave characteristics, on bricks, a cement mortar, and a sandstone.

## II. Theory

The temperature at which ice will propagate through a pore restriction of given size in a water-saturated porous medium is given by the following equation (Sill and Skapski, 1956), provided the suction in the water is zero :

$$T - T_m = \Delta T = \frac{-2T_m \sigma_{iw}}{Q_f \rho_i r_i}, \quad (1)$$

where

- $r_i$  is radius of curvature of the ice-water interface and also pore radius,
- $\rho_i$  density of the ice,
- $Q_f$  latent heat of fusion,
- $T_m$  temperature of melting at zero curvature of the solid-liquid interface at standard pressure,
- $T$  temperature at which freezing takes place with radius of curvature,  $r$ , in contact with water,
- $\sigma_{iw}$  ice-water interfacial energy.

Everett and Haynes (1965) have predicted heaving pressures in porous materials based on pore size, the parameter used previously by others (Penner, 1957, 1958, 1963) with the important addition that the geometry of the systems is defined precisely. The case of special interest in the present paper is the close-pack array of uniform-size spheres (Everett and Haynes, 1965). This porous body is overlain by a continuous ice lens, as depicted in the schematic drawing of Fig. 1 a and b.

The pressure difference across the ice-water interface is given by

$$p^i - p^w = \frac{2\sigma_{iw}}{r_i}, \quad (2)$$

where

- $r_i$  is radius of curvature of the ice-water interface,
- $p^i$  pressure on the ice,
- $p^w$  pressure in the water,
- and  $\sigma_{iw}$  interfacial energy term.

The pressure difference across the ice-solid interface at A is given by

$$p^i - p^s = \frac{-2\sigma_{is}}{r}, \quad (3)$$

where  $r$  is the radius of curvature of the solid.

Comparing eqs. (2) and (3) gives the pressure at point A as

$$p_A^s = p^w + \frac{2\sigma_{iw}}{r_i} + \frac{2\sigma_{is}}{r}. \quad (4)$$

In the region of the water-solid interface at B

$$p_B^s = p^w + \frac{2\sigma_{ws}}{r}. \quad (5)$$

The pressure difference across the region of the three-phase contact, if A and B are

brought close to C, is given by

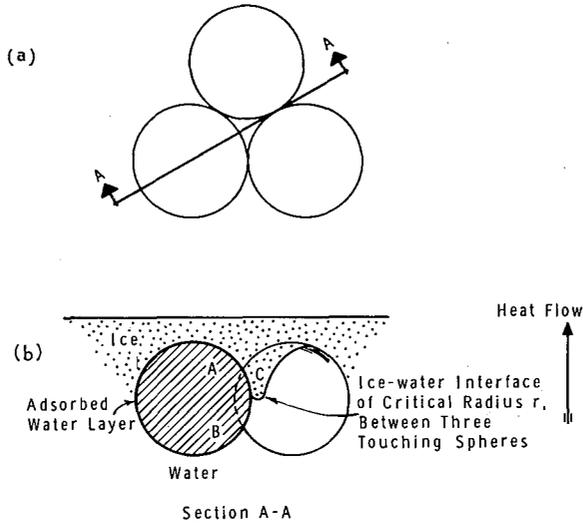
$$\Delta p = p_A^s - p_B^s = \frac{2\sigma_{iw}}{r_1} + \frac{2}{r}(\sigma_{is} - \sigma_{ws}). \tag{6}$$

Introducing the Young-Dupre equation

$$\sigma_{is} - \sigma_{ws} = \sigma_{iw} \cos \theta, \tag{7}$$

and defining  $B'$  as  $\frac{r}{r_1} \frac{1}{\cos \theta}$ , eq. (6) may be rewritten in the form

$$\Delta p = \frac{2\sigma_{iw} \cos \theta (1 + B')}{r}. \tag{8}$$



SCHEMATIC DRAWING OF ICE-WATER INTERFACE PRIOR TO PROPAGATING THROUGH PORE RESTRICTION BETWEEN THREE TOUCHING SPHERES

Fig. 1. (a) Spheres in close-pack array  
 (b) Schematic drawing of ice-water interface prior to propagating through pore restriction between three touching spheres

The critical radius of curvature at which a non-wetting phase displaces a completely wetting phase through the triangular shaped hole between three touching spheres has recently been investigated experimentally by Everett and Haynes (1965). It may be noted that geometrically the radius of the smallest sphere that can pass through such a hole is given by

$$\frac{r}{r_1} \simeq 6.41. \tag{9}$$

The reason for questioning the validity of the geometry in eq. (9) is that the interface between spheres is not a surface of revolution. Everett and Haynes determined the ratio of the sphere radius to critical radius of curvature by a series of experiments involving capillary rise between close-pack cylindrical rods, maximum bubble pressure between three spheres in mutual contact, and drainage of liquid from a pyramid of

steel balls in a cubic close-pack arrangement. It was concluded from these experiments that the critical radius of curvature was approximately

$$\frac{r}{r_1} = 5.60, \tag{10}$$

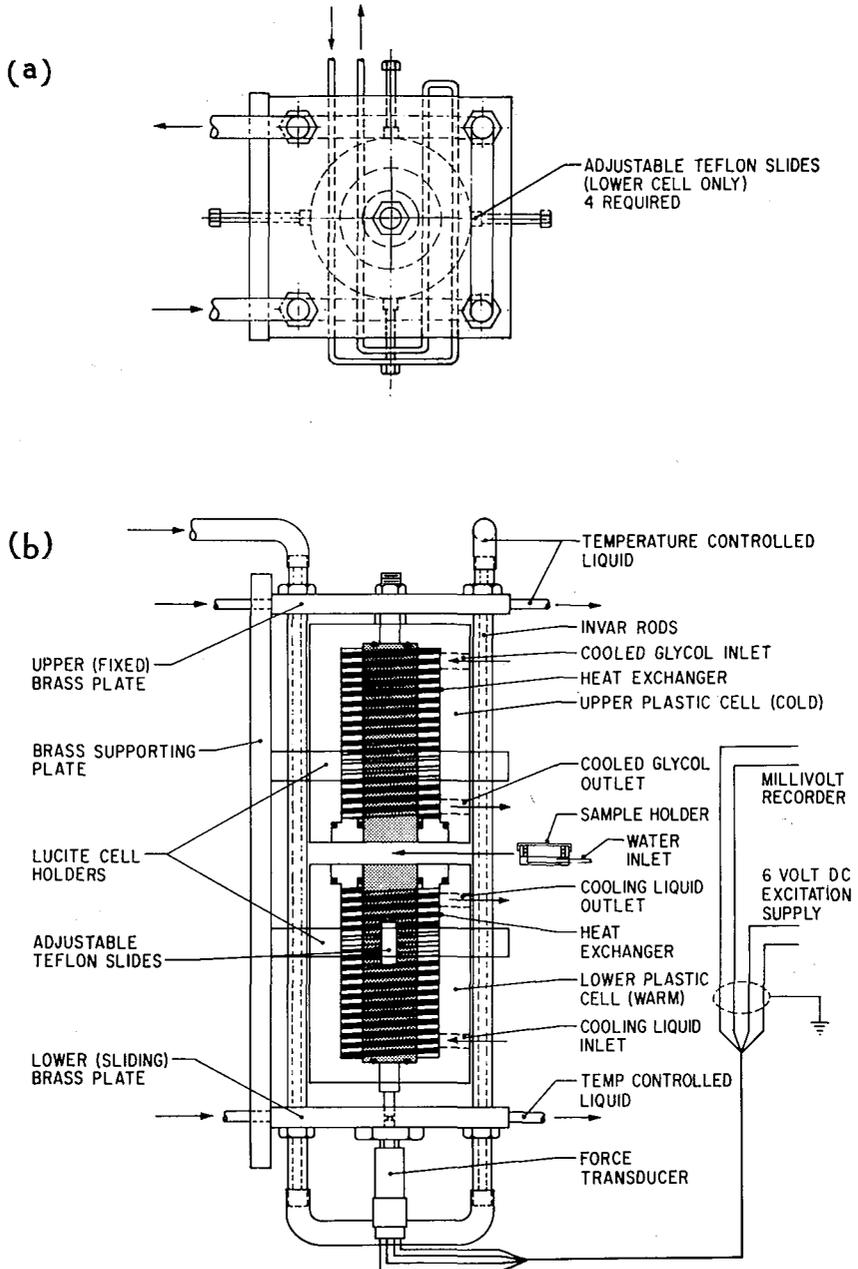


Fig. 2. (a) Heave pressure cell - top elevation  
 (b) Heave pressure cell - side elevation

where  $r$  is radius of spheres as before,  
and  $r_1$  critical radius of curvature at the narrowest constriction of  
three touching spheres.

The concept of Everett and Haynes recognizes two contributions to the heaving force: one, the increase in pressure on the ice that is required for the ice to pass through a narrow constriction; the other equalling  $2\pi r\sigma_{iw}$  which is taken into account in the development of eq. (8) by considering the difference in the interfacial tensions between water-solid and ice-solid.

### III. Experimental Investigation

The apparatus used to measure heaving forces is shown in Figs. 2 and 3. The upper cell is the cold side and is held in a fixed position; the lower cell (warm side) rests on a sensitive force transducer, which is fixed to the lower plate. The upper and lower plates at the extremities of the cells are held together with four Invar rods 1.27 cm in diameter. Fluctuations in room temperature, which would give rise to small movements are avoided by circulating a liquid at constant temperature through the Invar rods and end plates.

The specimen is placed in a sample holder between the two cells in the centre section so that it will be in direct contact with the finned metal heat exchangers. The temperature of the glycol solution circulating round the finned heat exchangers of the two cells imposes the temperature gradient across the specimen.

The sample holder has a small porous plate at the lower end for water transmission to the specimen and the external water level keeps the specimen water-saturated during the growth of the ice lens. Temperatures, which were monitored continuously at the ends of the specimen, showed fluctuations of less than  $0.01^\circ\text{C}$  over periods of several days; over shorter periods the variations were considerably less. Temperature variations at the freezing front were imperceptibly small as is indicated by the constancy of heaving pressures at equilibrium.

The experimental procedure with unconsolidated materials such as glass beads and soils was as follows. A water-saturated specimen inside a thin walled lucite sample holder was inserted between two cells and a thermal gradient established, with the upper side slightly below  $0^\circ\text{C}$  and the lower side slightly above the freezing temperature.

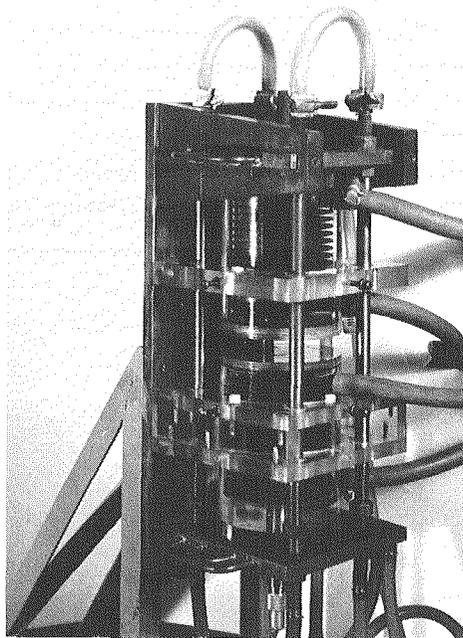


Fig. 3. Photograph of frost heave pressure apparatus

Freezing was initiated by seeding the cold side with a frosted wire. After a 24-hr period, while the pressure was increasing, the freezing plane was stationary within the sample. On stabilization the pressures were reduced to zero by adjustment of the nuts holding the upper and lower cells together. This was repeated several times until the ice lens was estimated to be about 2 mm thick. Maximum forces in the vertical direction were then allowed to develop fully and were monitored on a millivolt recorder from the output of the force transducer. The whole process was repeated, each run taking several days, depending somewhat on the kind of sample used and on the magnitude of the forces developed. As would be expected, equilibrium was approached slowly as the forces approached a maximum.

During pressure build-up the temperature at each end of the specimen was not permitted to vary, so that the forces to be measured would rise only at the ice-water interface below the ice lens and not as a result of expansion of water freezing at reduced temperatures behind the ice lens.

#### IV. Results

*Heaving pressure measurements in pads of glass beads.* Glass beads were size fractionated by sedimentation in water to obtain size uniformity. Figure 4, curve A, gives the size distribution of the original stock from which the fractionations were made. The degree of variation was established by preparing smears on glass slides and photographing them at random. Some 800 to 900 beads were measured and counted in each size range to establish this variation. The results are plotted in Fig. 4.

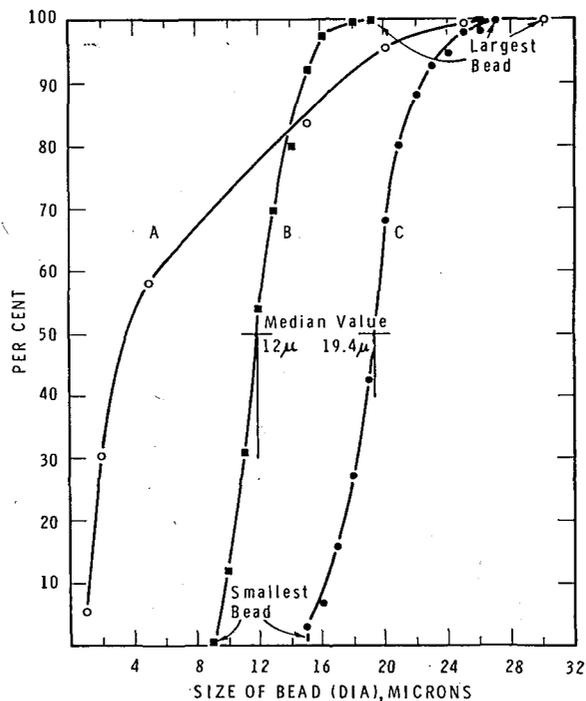


Fig. 4. Particle size distribution of unsorted and sorted glass beads

In curves B and C the median was used for calculations where the size of the particles was involved. It may be seen that the median diameter of the smaller fraction was  $12.0 \mu$ ; the median diameter of the larger size fraction was  $19.4 \mu$ . The heaving force was also measured on pads of the original stock of beads.

The size varied from 9 to  $19 \mu$  (diam.) in the batch of smaller beads. From Fig. 4 it may be seen that some 10% were between 9 and  $10 \mu$ , that another 10% were from 15 to  $19 \mu$ , and that 80% were between 10 and  $15 \mu$ . The larger beads ranged in size from 15 to  $27 \mu$ , but 80% of them were between 17 and  $22 \mu$ . Five percent of the unsorted beads were smaller than  $1 \mu$ , and the largest were  $30 \mu$ .

The Everett and Haynes relation (eq. (8)) relating bead radius to heaving pressure is plotted in Fig. 5 for the two  $\sigma_{iw}$  values of 30 and 40. The experimental relations between bead radius and heaving pressure are indicated on the same figure. The value for the  $12 \mu$  diameter beads is an average of 10 different heaving force measurements; for the  $19.4 \mu$  size it consists of two different measurements.

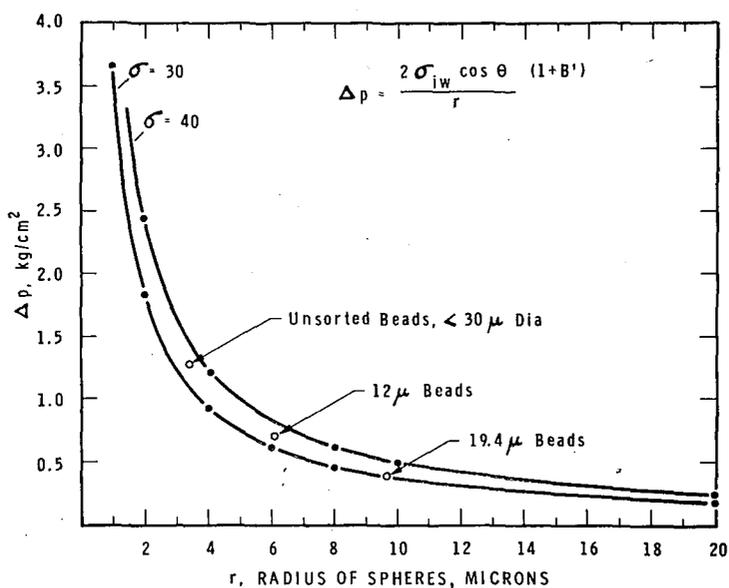


Fig. 5. Everett and Haynes equation relating heaving pressure to sphere radius

The experimental results give values for  $\sigma_{iw}$  of 30 and 35. At this point in the studies it was assumed that 35 is the more reliable value because the experiments were repeated a greater number of times for the  $12 \mu$  beads. Another assumption in the preparation of the bead pads was that a close-pack assemblage had been achieved, since it was for this geometry that the equation held. The pads were prepared by both jarring and tamping the sediment. Because the ice front has some capacity for rearranging the material at the freezing front, it is believed that something very close to a close-pack array was achieved at the interface between the particles and the ice lens.

The values for  $\sigma_{iw}$  obtained in this experiment compare favourably with the most reliable results reported in the literature from other methods of obtaining values for  $\sigma_{iw}$ ; they give credence, therefore, to the validity of the Everett and Haynes equation.

It is believed that the experimental technique used is sufficiently refined for use in exploring ice lens pressures for materials of more complicated pore geometry. An attempt has been made to assess which pore sizes are predominant in establishing the maximum force attainable when a large pore size variation exists.

The important role of the smaller beads in the unsorted batch ranging up to  $30 \mu$  can be seen from the heaving pressures developed. The beads in Fig. 4, curve A, gave a heaving pressure of  $1.28 \text{ kg/cm}^2$ , which corresponds to bead size radius of  $3.5 \mu$  using a value of 35 for  $\sigma_{iw}$ .

*Preliminary ice lensing pressure measurements on the surface of saturated porous solids.*

*Bricks.* Frost action deterioration of building materials in structures has not been associated with the ice lensing mechanism until recently (Garden, 1965). It is, however, of real value to be able to understand the mechanism and so to be able to predict the possible range of stresses to which structural components can be subjected, due solely to freezing. In the context of this paper the practical significance is simply referred to, since the main objective is to relate the magnitude of the heaving stresses due to lens growth with the pore geometry.

Four bricks, as received from the manufacturer, have been studied. Two were prepared by the extrusion method, one was a "pressed" brick, and the last was a soft mud variety. Samples were cut to fit the sample holder of the heaving pressure apparatus described previously. The specimens were short cylinders 1.905 cm diameter and 0.635 cm long. The samples were taken at least 1.27 cm from any outside surface in order to avoid skin effects.

Sample preparation simply involved water saturation under vacuum. At the completion of the ice lens pressure measurements the same specimens were oven-dried and again saturated under vacuum in order to obtain total porosity. Finally, they were oven-dried and their pore size distribution determined down to  $0.1 \mu$  (diam.) with a mercury porosimeter.

The freezing technique was much the same as that described earlier, except that the ice lens was grown at the surface. The brick samples were not, in fact, frozen, but were merely in contact with a growing ice lens. Again, the crystallization of the water was induced with a frosted wire. The pressure was kept sufficiently low for a lens from 2 to 4 mm to develop rapidly, after which maximum forces were allowed to develop.

For the purpose of calculating the critical size of the pores involved a pore geometry has been assumed consisting of cylindrical capillary openings to the surface. The equation

$$p = \frac{2\sigma_{iw}}{r_1}, \quad (11)$$

is assumed to apply, where  $r_1$  is the radius of the pore constriction of the brick in direct contact with the ice lens.

Based on the area exposed to the ice and the heaving force measured experimentally, the heaving pressure calculated was  $0.95 \text{ kg/cm}^2$  for the soft mud brick;  $1.75 \text{ kg/cm}^2$  for the pressed brick;  $2.12 \text{ kg/cm}^2$  for one extruded type; and  $1.84 \text{ kg/cm}^2$  for the other.

The pore size distribution for the bricks is shown in Fig. 6; other pertinent information is given in Table 1. It may be noted that the porosimeter that was used is capable of filling pores down to  $0.1 \mu$  diameter and gives almost complete saturation for the bricks studied.

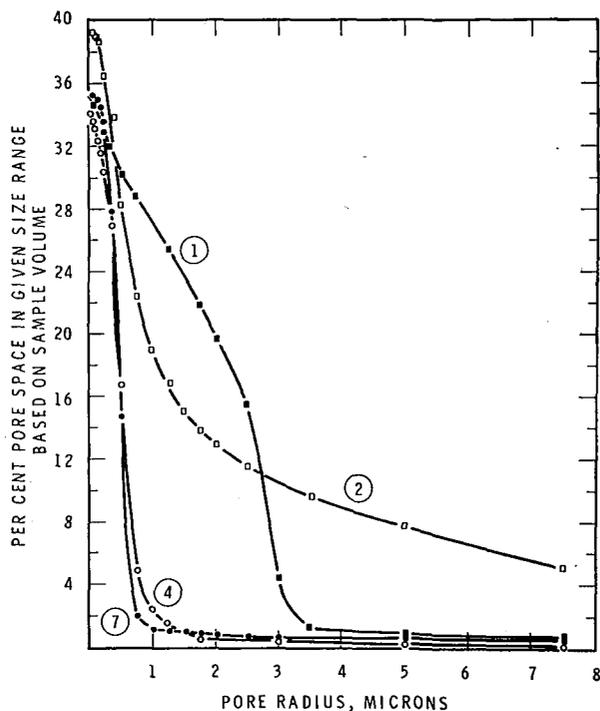


Fig. 6. Pore size distribution curves for bricks

Table 1. Percentage of pore volume and pore size properties of bricks

Brick identity	Total pore vol. of sample vol. (%)	Pore vol. greater than 0.05 size* (%)	Limiting pore size* active in lensing by eq. (11) ( $\mu$ )	Pore size smaller than active in lensing (%)
1 soft mud	37.73	34.66	0.75	$\approx 9$
2 pressed	42.68	39.13	0.41	$\approx 11$
4 extruded	34.11	33.92	0.34	$\approx 7$
7 extruded	37.42	35.08	0.39	$\approx 11$

\* radius of pores

The limiting radii calculated from heaving pressures did not vary greatly from brick to brick. Bricks Nos. 1 and 2, which were the soft mud and pressed varieties, had a larger percentage of pores in the large size range and these gave lower heaving pressures, particularly brick No. 1. Almost all of the pores in the two extruded bricks were smaller than  $1 \mu$  radius.

The last column in Table 1 shows the approximate percentage volume, consisting of pores smaller than the pore size calculated from the maximum heaving pressure.

This shows the importance of the smaller pores in assuming an active role in the ice lensing mechanism. The maximum pressure measured did not in any of the cases studied correspond to the smallest pores of the bricks.

*Other porous materials.* Ice lens heaving pressures were measured also in a sandstone and in a cement mortar. The pore size distribution down to  $0.05 \mu$  radius is given in Fig. 7. Equation (11) gave the limiting pore size radius active in heaving as  $0.20 \mu$  for the cement mortar. Comparing total pore volume with the volume of pores greater than  $0.05 \mu$  shows that half the total volume of pores in the cement mortar is less than  $0.05 \mu$ . In sandstone, limiting heaving radius  $0.20$ , only 4% of the total pore volume was contained in pores smaller than  $0.05 \mu$ . The heaving pressures measured in the cement mortar were  $3.51 \text{ kg/cm}^2$  and in the sandstone,  $3.57 \text{ kg/cm}^2$ .

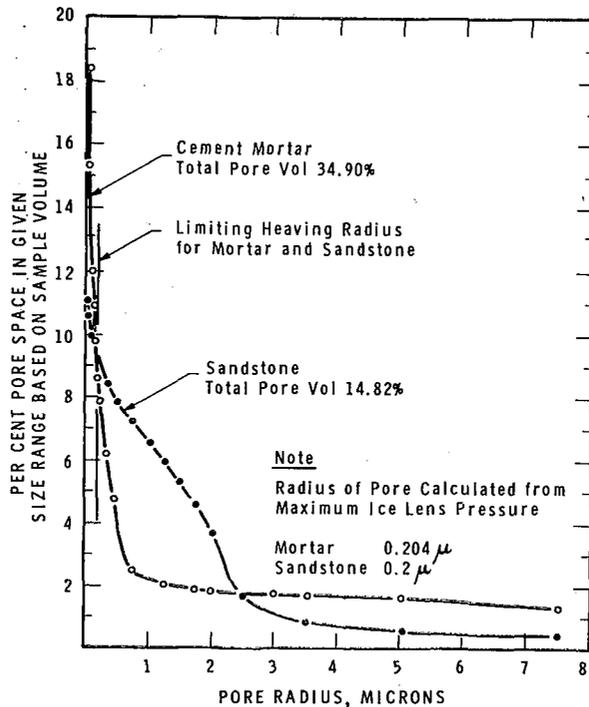


Fig. 7. Mercury porosimeter pore distribution curves for sandstone and cement mortar

*Ice lensing pressures in silty soil.* This particular soil, described elsewhere (Penner, 1960), has been shown to be highly frost-susceptible. It contains about 9% clay size, 43% silt, and 48% sand. Samples were prepared at two different densities to compare behaviour at radically different pore size ranges. The less dense sample ( $1.87 \text{ g/cm}^3$ ) was prepared by compaction in a normal compaction apparatus. The highly dense sample was compressed at  $9000 \text{ kg/cm}^2$  for 10 min in the air-dry state. The samples were then oven dried and trimmed to the correct size for the heaving measurements. They were allowed to saturate from one direction prior to the start of freezing.

Figure 8 shows mercury porosimeter values for both densities in the air-dry state, and shows the radically different porosities and pore size distributions for the two

28. The total pore volume determined by water saturation was 32.0% of the sample volume for the compacted sample and 15.7% for the compressed sample. Heaving pressures of 1.43 kg/cm<sup>2</sup> for the high density sample and 1.23 kg/cm<sup>2</sup> for the low density sample were measured, giving a critical pore size value, using eq. (11), and 0.6  $\mu$ , respectively. There is, therefore, some influence of density, as was shown previously (Penner, 1958), but because of the similarity in heaving pressures the effect appears to be derived from the finest particles.

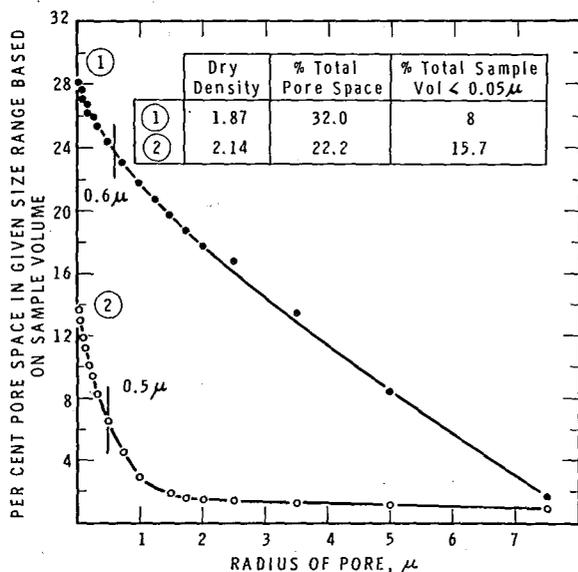


Fig. 8. Pore size distribution in silt at two densities in the oven dry state

It was obvious that on wetting the material before freezing some swelling occurred so that the curves shown in Fig. 8 would not apply exactly; but in many ways this preliminary method of pore size evaluation had merit. Regardless of how the sample was prepared, the freezing front modifies the distribution of particles to some extent, as has been shown by Corte (1962), and some doubt remains as to how applicable bulk properties are to the situation at the contact between the ice lens and the particles at the freezing front.

## V. Summary

1. Pressures resulting from ice lens growth have been measured on pads of glass beads, which are believed to be sufficiently uniform to test the equation of Everett and Jones (1965). The pressures predicted from the equation correspond to experimental heaving pressures and give values for  $\sigma_{iw}$  between 30 and 35. The most reliable value is considered to be 35.

2. Heaving pressure measurements with unsorted glass beads show the significance of the smaller beads in producing the ultimate ice lens pressures.

3. (a) Preliminary measurement of the maximum ice lens pressures on solid porous

materials such as bricks, a cement mortar, and a sandstone show that pore size distribution is a significant factor. Brick No. 1, which had a large percentage of large pores, gave considerably lower pressures. From 7 to 11% of the pore volume, based on the total volume of the samples, had a smaller radius than that corresponding to the maximum heaving pressure, using eq. (11).

(b) Of the porous solids studied cement mortar and sandstone gave the highest pressures corresponding to critical operative radii of about  $0.2 \mu$ .

(c) Pressures in a silty soil measured at two densities indicate the influence of the finest particles in establishing heaving pressures at the ice lens front in silty soils.

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## AUTHOR INDEX

The boldface figures indicate the theme of the group where the author's paper belongs and the following lightface figures indicate the first page of the paper.

The correspondence between the boldface figure and the theme.

- 1: Physical Properties of Ice (Part 1),
- 2: Physical Properties of Sea Ice (Part 1),
- 3: Physical Properties of Deposited Snow (Part 2),
- 4: Mechanism of Avalanche (Part 2),
- 5: Physical Properties of Frost Heaving (Part 2),

Addison, J. R.	2-649	Haefeli, R.	3-983 4-1199
Ager, B.	3-1029	Hanagud, S.	3-807
Akifyeva, K. V.	4-1295	Herrmann, M. R.	3-797
Akitaya, E.	3-713 4-1177	Higashi, A.	1-277 1-409
Andersland, O. B.	1-313	Higuchi, K.	1-79
Aota, M.	2-539	Huzioka, T.	4-1177
Arai, H.	3-1075	Ishida, T.	2-551 3-1099
Assur, A.	2-557	Ishihara, K.	3-1037
Avsiuk, G. A.	1-389	Itagaki, K.	1-233
Beaumont, R. T.	3-1007	Jaccard, C.	1-173
Bender, J. A.	3-973	Jackson, K.	5-1361
Benson, C. S.	3-1039	Jones, S. J.	1-267
Bilello, M. A.	3-1015	Judson, A.	4-1151
Bowles, D.	4-1243	Jumikis, A. R.	5-1387
Bradley, C. C.	4-1243	Kinosita, S.	3-911 5-1345
Briukhanov, A. V.	4-1223	Kobayashi, D.	3-1099
Budd, W.	1-431 1-447	Kobayashi, S.	3-1099
Bull, C.	1-395	Kobayashi, T.	1-95
Camp, P. R.	1-181	Kojima, K.	3-929
Chae, Y. S.	3-827	Kondakova, N. L.	4-1295
Corte, A. E.	5-1333	Konstantinova, G. S.	4-1295
De Micheli, S. M.	1-259	Kotlyakov, V. M.	1-389
Dillon, H. B.	1-313	Koyama, M.	5-1323
Doumani, G. A.	3-1119	Krasser, L. M. A.	4-1261
Dunbar, M.	1-479 2-687	Kravtsova, V. I.	4-1295
Dyunin, A. K.	3-1065	Kuroda, M.	4-1277
Dykins, J. E.	2-523	Kuroiwa, D.	3-751 3-953
Eglit, M. E.	4-1223	Kusunoki, K.	2-705
Ermakov, V. F.	1-339	La Chapelle, E. R.	4-1169
Fujino, K.	2-539 2-633	Levi, L.	1-43 1-159
Furukawa, I.	4-1291	Lewis, E. L.	2-611
Gavanovitch, S.	1-43	Licenblat, A. R.	1-259
Glen, J. W.	1-267	Lofgen, G.	2-579
Gold, L. W.	1-359	Lossev, K. S.	1-385
Gow, A.	1-469	Luyet, B. J.	1-51 1-71
Grave, N. A.	5-1339	Lyon, W.	2-707
Grigorian, S. S.	4-1223	Mackey, J. R.	4-1185

- |                  |               |                    |             |
|------------------|---------------|--------------------|-------------|
| Maeno, N.        | 1-207         | Shumskly, P. A.    | 1-371       |
| Magono, C.       | 1-137         | Shurova, I. Ye.    | 4-1223      |
| Marangunic, C.   | 1-395         | Smith, I. L.       | 1-447       |
| Mathews, W. H.   | 4-1185        | Smith, J. H.       | 3-843       |
| Mellor, M.       | 3-843         | Spears, D. L.      | 1-181       |
| Miagkov, S. M.   | 4-1223        | Stehle, N. S.      | 1-219 3-797 |
| Michel, B.       | 1-119 1-129   | Stephenson, P. J.  | 3-725       |
| Mizuno, Y.       | 3-751         | Suzuki, Y.         | 1-21 2-661  |
| Moser, E. H.     | 3-993         | Tabata, T.         | 2-481 2-539 |
| Moskalev, Yu. D. | 4-1215        | Takahashi, T.      | 1-151       |
| Nakamura, T.     | 1-247         | Takeuchi, M.       | 3-751       |
| Nakaya, U.       | 3-953         | Troshkina, E. S.   | 4-1295      |
| Narita, H.       | 4-1177        | Tushinsky, G. K.   | 4-1295      |
| Ohtake T.        | 1-105         | Uhlmann, D. R.     | 5-1361      |
| Ono, N.          | 2-599         | Untersteiner, N.   | 2-569       |
| Onuma, T.        | 3-785         | Vyalov, S. S.      | 1-339 1-349 |
| Ôura, H.         | 3-1085 3-1099 | Volodicheva, N. A. | 4-1295      |
| Penner, E.       | 5-1311 5-1401 | Voitkovsky, K. F.  | 1-329       |
| Plam, M. Ya.     | 4-1223        | Wakahama, G.       | 1-291 1-357 |
| Pounder, E. R.   | 2-649         |                    | 3-909       |
| Rakita, S. A.    | 4-1295        | Watanabe, K.       | 2-667       |
| Ross, B.         | 2-499         | Watanabe, Z.       | 3-741       |
| Salm, B.         | 3-857         | Weeks, W. F.       | 2-579       |
| Sasaki, H.       | 5-1323        | Wishart, E.        | 1-447       |
| Schaerer, P. A.  | 4-1255        | Yakimov, Yu. L.    | 4-1223      |
| Sherwood, G. E.  | 3-993         | Yamada, T.         | 3-1099      |
| Shimizu, H.      | 4-1177 4-1269 | Yong, R. N.        | 5-1375      |
| Shinojima, K.    | 3-875         | Yosida, T.         | 1-79        |
| Shio, H.         | 1-137         | Yosida, Z.         | 1-1 3-773   |
| Shiotani, M.     | 3-1075        | Zotikov, I. A.     | 1-469       |
| Shoda, M.        | 4-1137        |                    |             |