Calorimeter-Strain Apparatus for Study of Freezing and Thawing Concrete

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Information and techniques available in the past have not been sufficient for a complete evaluation of the role of the various factors influencing the resistance of concrete to freezing and thawing.

It has long been recognized that the destruction of concrete exposed to freezing and thawing is due principally to the freezing of water within the concrete; concrete in a dry state will not be damaged by freezing. Water-saturated non-air-entrained concrete of normal water-cement ratio cannot resist the disruptive forces generated by the freezing of the water. Air entrainment provides a practical and fundamental solution to this freezing problem. Frost resistance is controlled by the freezable water content, air void characteristics, paste permeability, aggregate characteristics, and other factors, none of which are readily evaluated experimentally.

This paper describes the development and operation of an apparatus for determining (a) the amount of water actually freezing within a concrete specimen as a function of temperature and time, and (b) the physical response of the specimen in terms of length change as this water is frozen. Examples of the type of information that can be obtained are presented and discussed.

● WHEN WATER is cooled, its heat content as a function of temperature is almost linear, indicating almost constant specific heat, until the freezing point is reached. When water freezes, a definite amount of heat, proportional to the amount of water frozen, is liberated (latent heat of fusion of water = 80 calories per gram at 32 F). The apparatus described here utilizes this latent heat of fusion as a means of measuring the amount of water freezing within a concrete specimen at various temperatures.

As water is frozen within the concrete specimen, the amount of dilation observed for the concrete is evidence of the magnitude of the internal pressure developed. This dilation, measured in terms of length change, is indicated by deviations from the normal thermal contraction line at temperatures below the freezing point. Generally, for frost-resistant concretes, such as air-entrained concretes, the specimen actually contracts more during freezing than would be anticipated from thermal contraction alone. These length changes are measured by means of an external indicating system utilizing SR-4 electrical strain gages.

The apparatus makes it possible to measure the amount of ice produced and the changes in length of a test specimen caused by cooling the specimen into the range of freezing temperatures. This enables the study of the influence of such variables as air void characteristics, water-cement ratio, curing, and aggregates, on these aspects of freezing and thawing of concrete.

DESCRIPTION OF APPARATUS

The apparatus was designed for specimens used in regular freezing and thawing tests in the Portland Cement Association laboratory; that is, concrete prisms measuring 3 by 3 by $11\frac{1}{4}$ in. This size specimen permits the use of concrete, rather than paste or mortar, hence various coarse aggregates can be studied. In addition, specimens can be tested periodically during their exposure to the laboratory freezing and thawing test.

Figure 1 shows a cross-section of essential features of the calorimeter-strain apparatus. The concrete prism is housed in a copper jacket open at the top (Fig. 2), with about 1/8-in. clearance between the sides of the concrete prism and the inside surfaces of the copper jacket. The bottom of the jacket is reinforced with additional thicknesses of copper plate. A washer, centered at the bottom's inside surface, provides the centering necessary for the stainless steel gage plug in the bottom of the concrete prism. The top of the jacket is closed off with a housing containing the SR-4 strain indicator (Fig. 3).

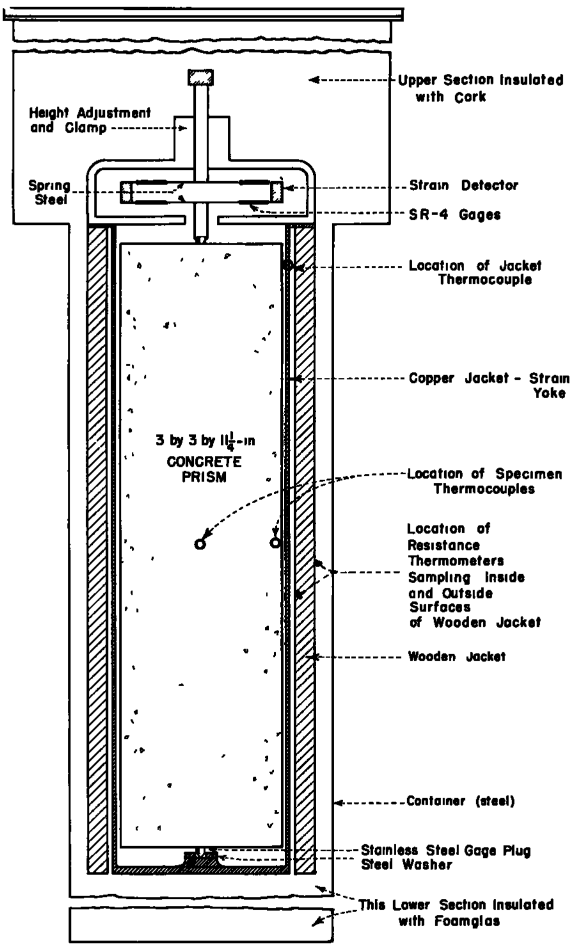

Figure 1. Cross-section of calorimeter-strain apparatus.

This jacket and housing provides the yoke within which length changes are measured. The stainless steel gage plug in the top of the specimen bears against the stem projecting through the base of the strain indicator housing. This stem is fastened to the center of one of two spring steel leaves, which are approximately $\frac{1}{4}$ in. apart and fastened rigidly together at the ends by means of separator blocks.

The stem fastened to the center of the upper spring steel leaf passes through a block at the top of the housing. This block is slotted and provides for vertical adjustment of the stem and spring leaf assembly. set screws enable rigid fastening of this upper stem in its final adjusted position. Affixed to the top and bottom of each spring leaf are SR-4 electrical strain gages comprising four arms of a Wheatstone bridge. As the specimen changes in length, the spring leaves flex. Changes in the electrical output of this gage circuit indicate the length change taking place.

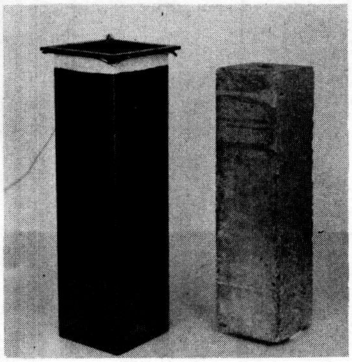

Figure 2. Copper jacket and concrete specimen.

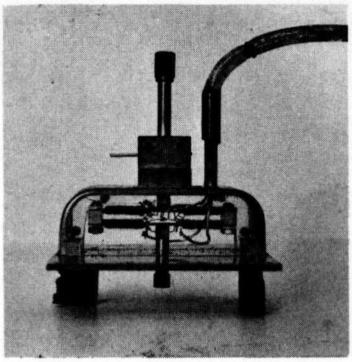

Figure 3. Strain indicator and housing.

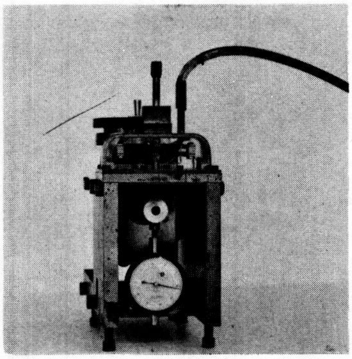

Figure 4. Calibration apparatus for strain indicator.

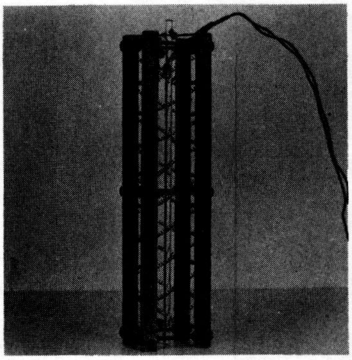

Figure 5. Resistance coil for heat heat calibration.

Figure 4 shows the device used for calibrating the electrical output of the gage circuit in terms of vertical length changes as measured by a dial indicator having readability of 0.0001 in. The output of the gage circuit is recorded on a 1-millivolt full-scale 12-point recorder. For this strain device, 1-mv output equals 0.0077-in. length change; 0.001 mv (smallest chart division 0.005 mv) represents slightly less than 0.0000008 in. per in. strain (10-in. gage length).

This measuring device was designed to be temperature compensating. The output of the gage vs the length change was demonstrated to be linear, as indicated by various check calibrations made at -20, 0, and 83 F. The slopes of these calibration lines were the same at these temperatures.

The output of the strain indicator depends on the resultant of two simultaneous length changes: (a) that of the yoke (copper jacket and strain indicator housing), and (b) that of the concrete specimen. Knowing the thermal coefficient of the yoke, the length change of the concrete specimen can be calculated. The thermal coefficient of the yoke is determined by using a rod of Pyrex glass of known thermal coefficient in place of a specimen. The Pyrex rod can be seen at the center of the heater shown in Figure 5.

Surrounding the four sides and covering the full length of the copper jacket is a 3/8-in. thick wooden jacket. This wooden jacket was dried and then sealed to prevent ingress of moisture. Zig-zag patterns of No. 40 copper wire fastened to the inside and outside surfaces, serve as resistance thermometers. As heat is withdrawn from a specimen through this wooden jacket, the difference in resistance between these two resistance thermometers changes. The amount of heat per unit time represented by a unit change in resistance is determined directly by placing an electrical resistance heater within the jacket and determining the resistance changes for known quantities of heat per unit time supplied by this heater. The heater used for this calibration is shown in Figure 5. By suitable bridge circuitry, these resistance changes are converted to potential changes (bridge unbalance). The millivolt recorder used for the strain indicator is used to record these potential changes as heat is withdrawn from the specimen in the copper jacket.

Figure 6 shows the strain indicator and the copper jacket containing a concrete prism assembled and ready for test. The thin wire to the left

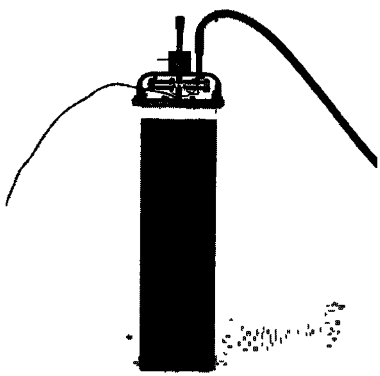

Figure 6. Copper jacket and strain indicator assembly.

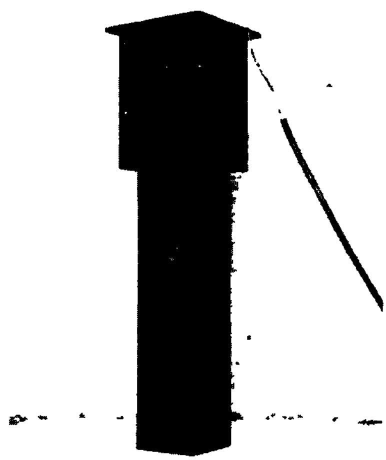

Figure 7. Container for copper jacket and strain indicator assembly.

is a thermocouple used to measure specimen temperature. The plastic tube at the right of the strain indicator housing contains the lead wires to the SR-4 electrical strain gages. This assembly is placed in the container shown in Figure 7, in which the wooden jacket outfitted with the resistance thermomemters is already in place. Figure 8 is a top view of the container, in which some of the inside surfaces of this wooden jacket can be seen. The lead wires extending up to the top of this container connect the two resistance thermometers to the recorder circuit. The copper jacket fits into this wooden jacket in the area of the container immediately below the widened top portion. The remainder or bottom of this narrow portion is filled with about 6 in. of foam glass. The section above the copper jacket, that surrounding the strain indicator housing, is insulated with cork blocks. The amount of insulation between the container and the outer surfaces of the wooden jacket regulates the rate of freezing. The thick insulation at the top and bottom insures that practically all the heat withdrawn from a specimen will pass through the wooden jacket containing the resistance thermometers.

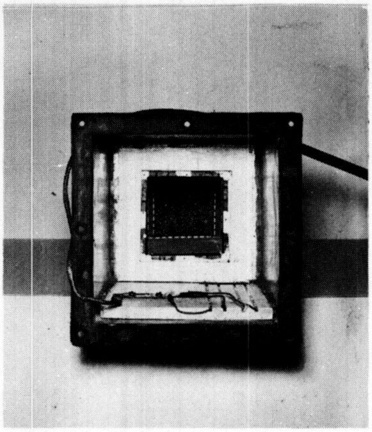

Figure 8. Top view of container, showing wooden jacket.

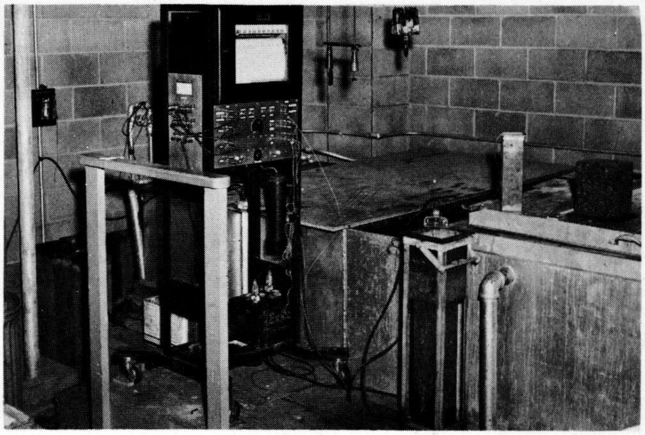

Figure 9. Assembly of calorimeter-strain apparatus. Freezing cabinet to right of container; thaw tank immediately behind container; recording equipment at left provides a continuous record of heat flow from specimen, length change, and temperature of specimen during test.

Figure 9 is an over-all view showing the assembled calorimeter-strain unit. To the left is the recorder which provides a continuous record of length change, heat flow, and temperature. The tank at the right contains -30 F brine. Freezing is accomplished by lowering the assembled container into the brine tank with the brine level approximately 2 in. above the top of the housing of the strain indicator (about halfway up the enlarged top portion of the container). Once the unit is assembled and lowered into the brine tank, no further manipulations other than minor recorder adjustments are required. The tank to the left contains water at room temperature for thawing after the freeze portion of the cycle.

EXPERIMENTAL RESULTS

Relative heat content and change in length of specimen as a function of temperature are readily calculated from heat calibration and length change calibration data. The deviations from the extrapolation of the normal thermal contraction line (length change as a function of temperature above the freezing point) below the freezing point represent the expansion or shrinkage of the concrete specimen due to freezing. The deviation from the extrapolation of the relative heat content line (relative heat content as a function of temperature above the freezing point) below the freezing point represents the cumulative heat given off by all of the water which has frozen down to that particular temperature.

Initially, two thermocouples were used to measure specimen temperature, one at the surface and one at the center of the specimen. It was observed that the difference in these two temperatures ranged from 1 to 2 F during the early stages of freezing; during the latter stages of freezing the two temperatures were nearly equal. Because the average concrete temperature would more nearly approach the surface temperature than the temperature at the center, and because the difference was relatively small, tests were generally conducted using only surface temperature measurements.

In these tests the cooling was relatively slow (approximately 20 F per hour, depending on the water content of the specimen).

Typical data are shown in Figure 10. The specimen was a non-air-entrained concrete prism cured continuously moist for eight days. The

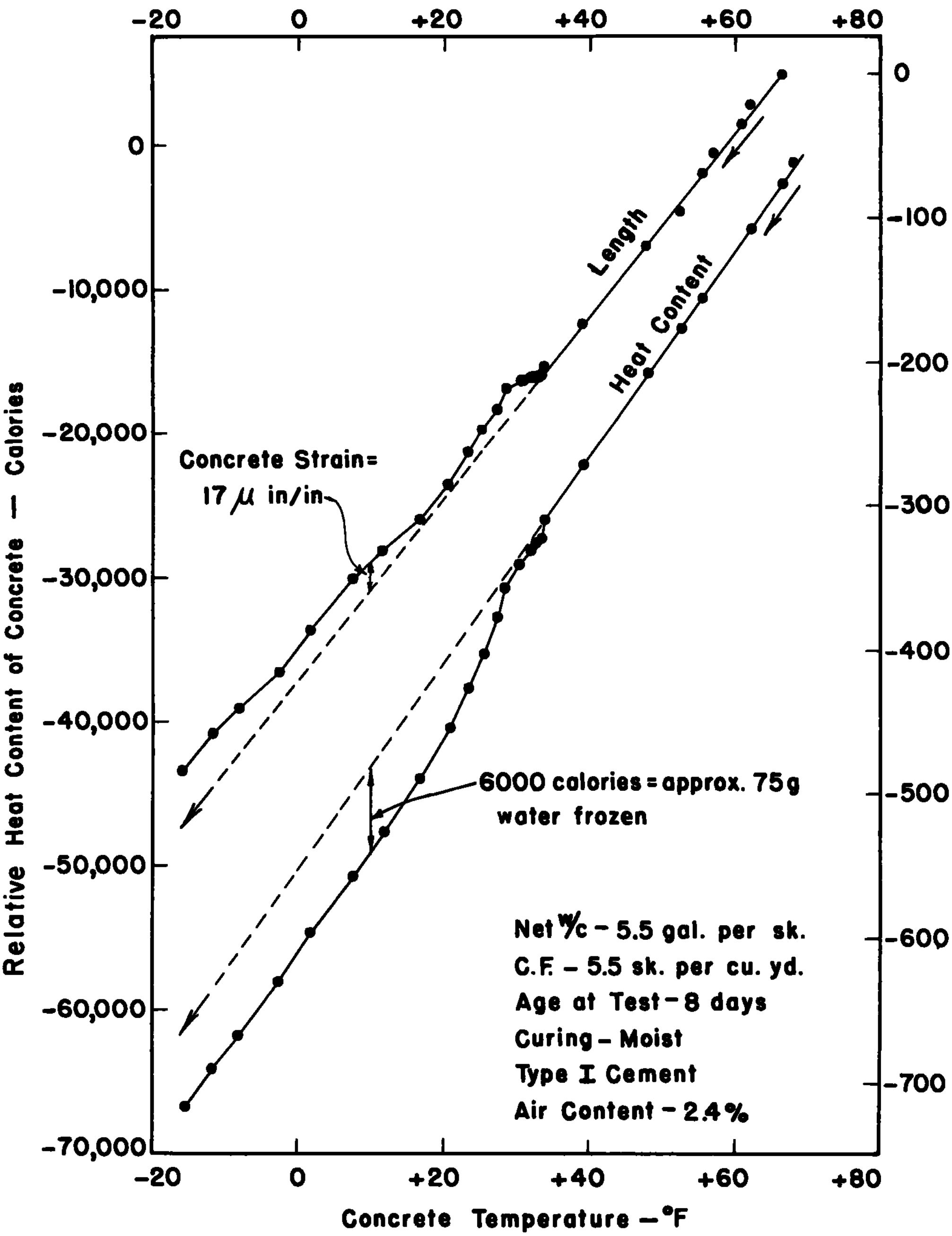

Figure 10. Typical heat content and length change data obtained by means of calorimeter-strain apparatus during freezing of concrete specimen.

specimen temperature at the start of this test was 68 F. The bottom line shows the relative heat content (relative to the heat content at 68 F) in calories as a function of temperature. As the specimen cooled, the relative heat content-temperature line remained linear until freezing began. Below this point, greater increments of heat were extracted as the temperature continued to decrease, indicating that water was freezing within the specimen. Freezing was progressive as the temperature dropped below 32 F and reached a maximum at about +10 F. At this point, 6,000 calories had been withdrawn in addition to the amount necessary to bring the temperature of the specimen to this level in the absence of freezing. For a latent heat of fusion of 80 cal. per gram of ice formed, this represents approximately 75 grams of ice. Actually, in calculating the amount of ice formed, corrections are made for the change in latent heat of fusion with temperature (decrease with decrease in temperature) and the change in the slope of the extrapolated relative heat content line, because the ice already formed affects the heat capacity of the concrete.

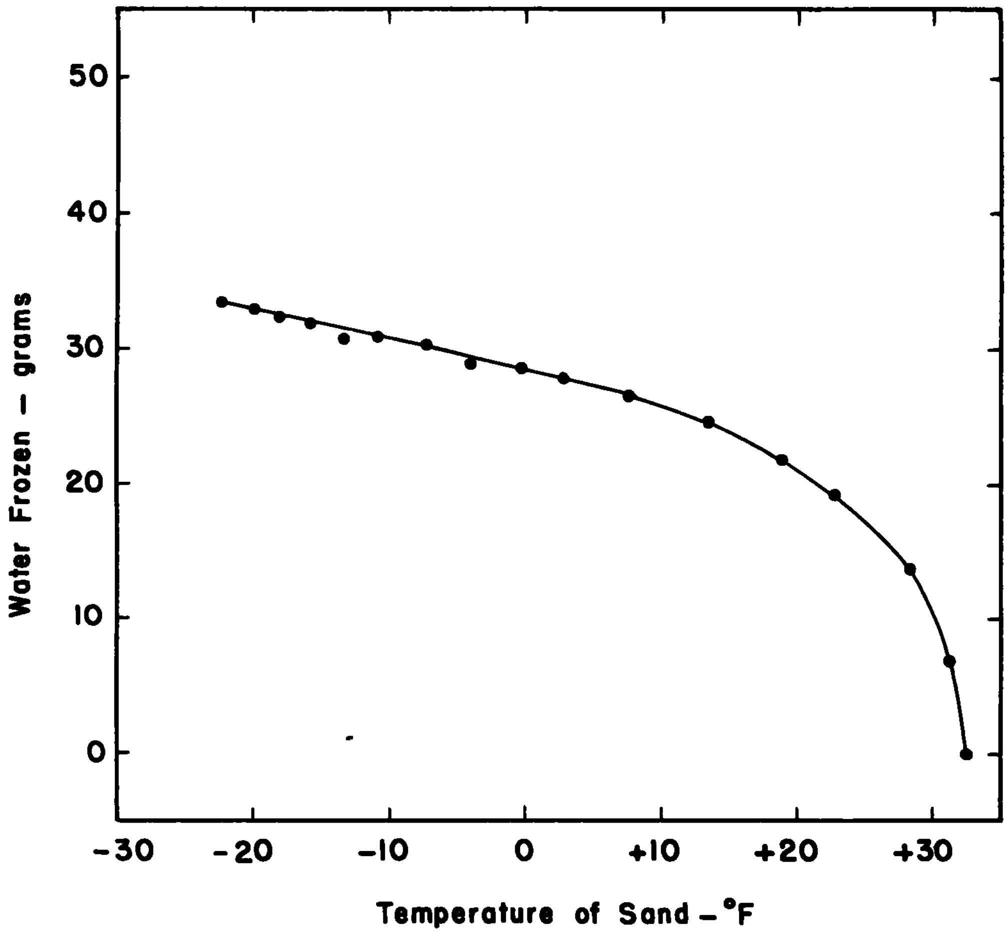

Figure 11. Freezable water in fine aggregate as a function of temperature; Elgin, Ill., sand; saturated surface-dry sample containing 34 grams of absorbed water.

The top line shows the length as a function of specimen temperature. Above the freezing point the line is linear, the slope representing the thermal coefficient of the concrete. Below the freezing point the deviations from this line represent the length changes due to the freezing of water within the specimen. At +10 F, the point at which the maximum amount of ice was formed, the specimen strain was about 0.000017 in. per in. An average internal hydraulic pressure of about 200 psi would produce a strain of this magnitude.

Freezing of Known Amount of Water

To demonstrate the accuracy of the calorimeter-strain apparatus in determining the amount of freezable water, a sample of saturated surface-dry sand was used in place of the test specimen. This sand sample contained 34 grams of absorbed water. Figure 11 shows the plot of the amount of water frozen as a function of temperature. Freezing was progressive, and at the final temperature the amount of water frozen was indicated to be slightly over 34 grams. The observed depression may be due to materials dissolved by the water in the aggregate, or due to the water being contained in pores of various sizes within the aggregate, or both.

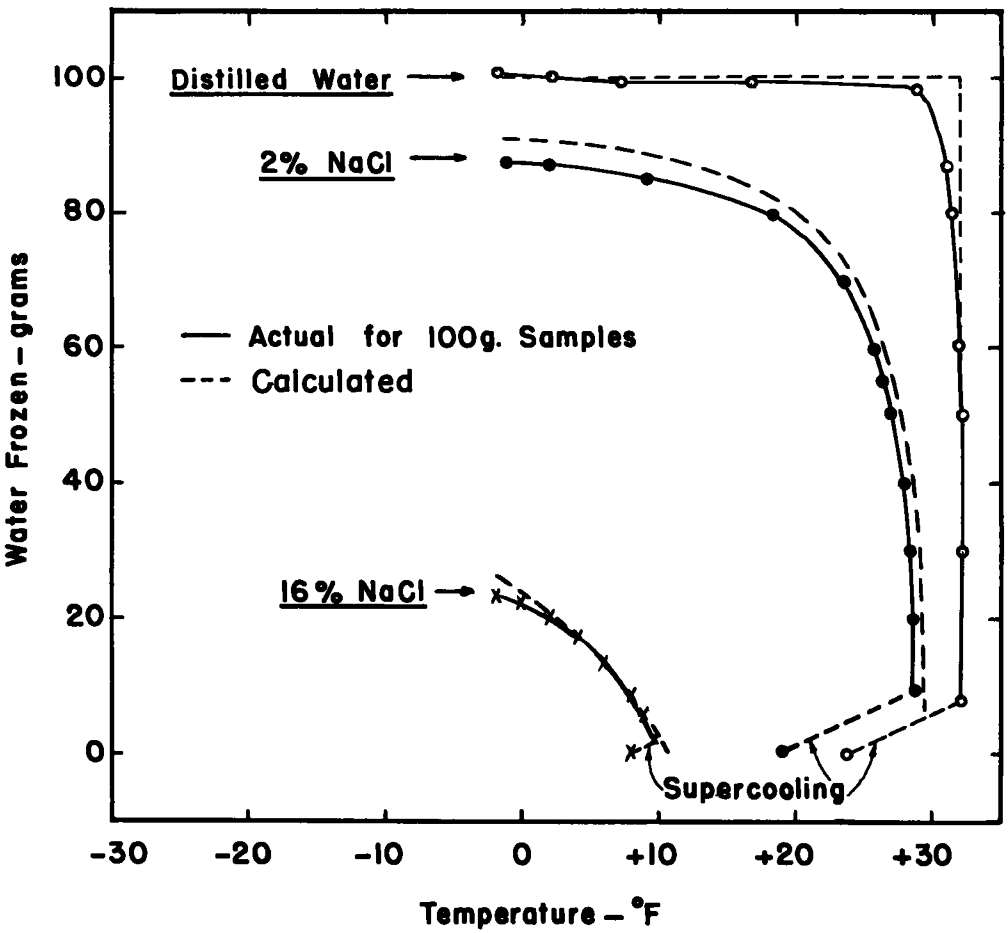

Figure 12. Amount of water frozen, as a function of temperature.

More positive checks were afforded by the freezing of 100 grams of distilled water, a 20 percent NaCl solution, and a 16 percent NaCl solution. The results of these determinations, together with the amounts which should have frozen as a function of temperature, are shown in Figure 12. The actual determinations and the calculated amounts were nearly the same at the various temperatures.

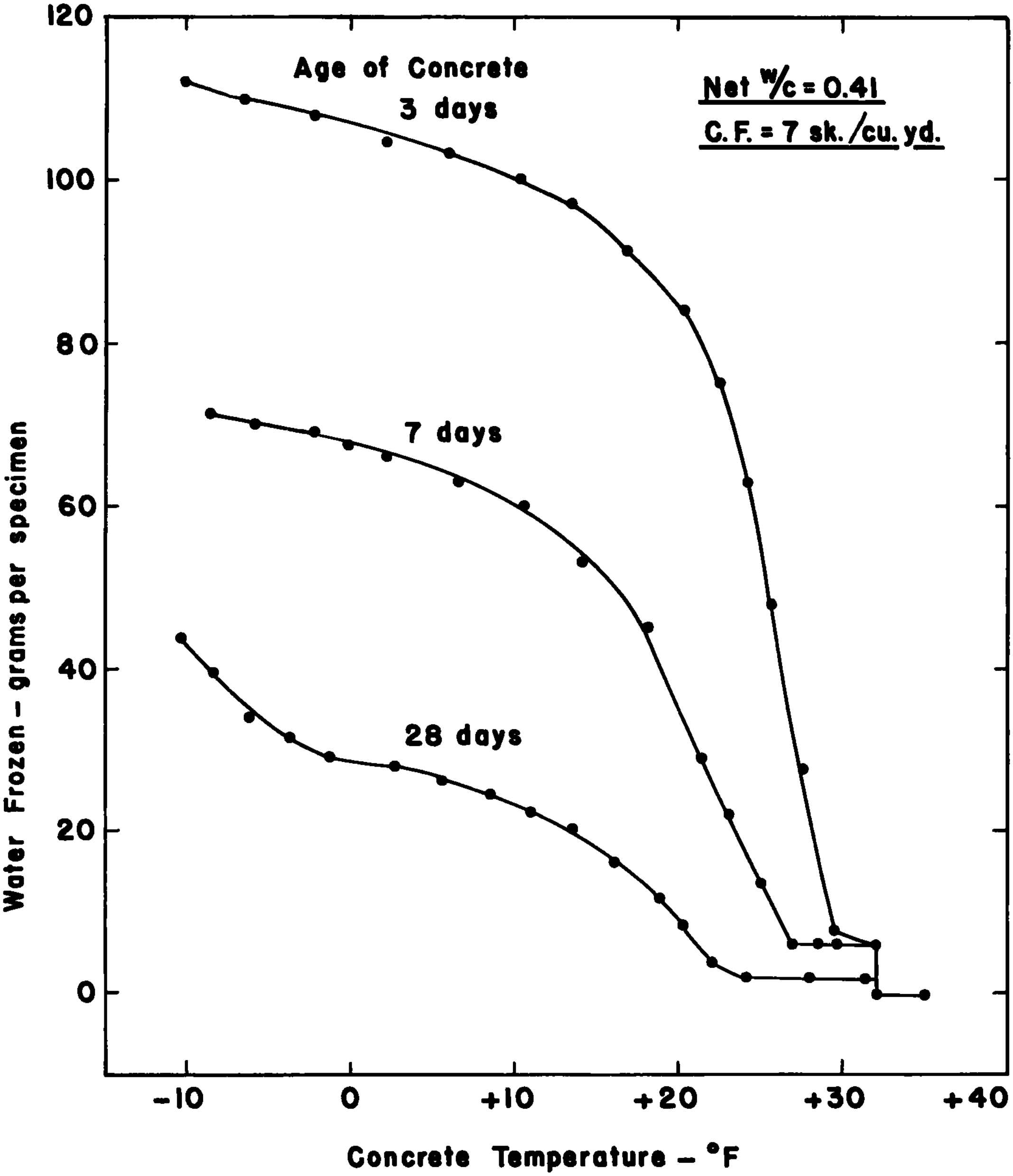

Figure 13. Effect of age of concrete on amount of water freezing as a function of temperature; concretes cured continuously moist at 73 F, Type I cement, no entrained air, Elgin, Ill., sand and Eau Claire, Wis., gravel.

Effect of Concrete Age on Amount of Freezable Water

Figure 13 shows freezable water data obtained for a 7-sack per cu yd non-air-entrained concrete for three different periods of continuous moist curing. Continued hydration reduces the amount of water that will freeze at any particular temperature. At 3 days the amount of water frozen at -10 F was 112 grams, at 7 days 71 grams, and at 28 days 44 grams. In addition, as hydration proceeds the alkalies in solution in the remaining paste water become more concentrated and result in a further depression of the freezing point. The few grams of water that froze at 32 F for each of the concretes shown in Figure 13 possibly represent free surface water on the specimen. The actual freezing point of the water within the concrete is believed to be indicated if this small amount of water initially freezing is disregarded. On this basis, the actual freezing points of the water within the concrete decreased with continued curing, being 30 F at 3 days, 27 F at 7 days, and about 23 F at 28 days.

Effect of Water-Cement Ratio on Freezable Water

The effect of water-cement ratio on the amount of water freezing as a function of temperature is shown in Figure 14. Non-air-entrained concrete specimens were prepared at water-cement ratios of 0.41, 0.49, and

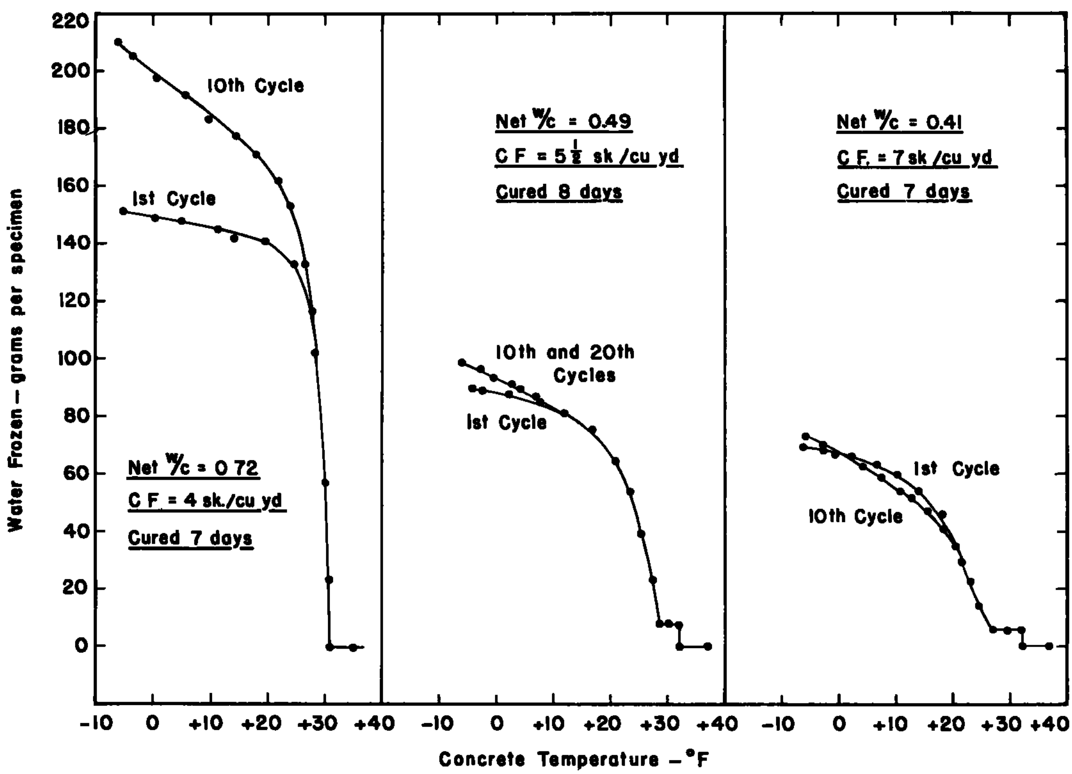

Figure 14. Effect of water-cement ratio and cycles of freezing and thawing on amount of water freezing as a function of temperature; concretes cured continuously moist at 73 F prior to test.

0.72 by weight. Concretes were cured moist for 7 days (8 days for the 0.49 water-cement ratio). For each concrete the first and tenth cycles of freezing were performed in the calorimeter-strain apparatus, and in one case the 20th cycle. The intervening cycles were performed in the laboratory freezing and thawing apparatus.

For the first cycle, the amount of water frozen at -5 F was 69 grams for 0.41 water-cement ratio, 90 grams for 0.49 water-cement ratio, and 151 grams for 0.72 water-cement ratio. For 0.41 water-cement ratio, the 10th cycle produced about the same freezing curve as the first; for 0.49 water-cement ratio the 10th and 20th cycles showed slightly more freezable water than the first; whereas for 0.72 water-cement ratio the 10th cycle showed a considerable increase in the freezable water.

This increase in freezable water for 0.72 water-cement ratio concrete after 10 cycles may be attributed to the early disintegration of this non-air-entrained concrete of relatively high water-cement ratio. The expansions and gains in weight per specimen during the cycles of freezing and thawing were as follows:

Net W/C	Expansion, %		Weight Gain, g		Water Frozen*, g	
(by wt)	10 cycles	20 cycles	10 cycles	20 cycles	10 cycles	20 cycles
0.41	0.009	—	7	—	72	—
0.49	0.014	0.050	7	5	98	98
0.72	0.273	—	26	—	208	—

*See Figure 14.

These data indicate that concretes remaining relatively undamaged by the freezing and thawing exposure exhibit little differences when retested for freezable water. When expansion is excessive, water enters the concrete during the period of thawing in water, as indicated by the 26-g weight gain for the 0.72 water-cement ratio concrete. The measured increase in freezable water was actually about 60 g. The weight gain observed does not reflect the total gain in water of the specimen, because this deterioration simultaneously caused the loss of solid material.

Effect of Brine Solution Storage on Freezable Water

In current studies of "salt" scaling, it was of interest to determine the influence of different concentrations of de-icer solution on the amount of freezable water. Companion concrete specimens were cured continuously moist for 28 days and then stored for 7 days in a calcium chloride solution of either 4, 8, or 16 percent concentration by weight. Figure 15 shows the influence of these storage conditions on the amount of water frozen as a function of temperature. The top line indicates the data from the 28-day continuously moist-cured specimens. A test of a companion specimen cured 90 days moist indicates that an additional 7 days after 28-day curing would only have reduced the freezable water at -10 F about 3 to 4 g below that shown for the 28-day specimen.

The reduction in freezable water with increased de-icer concentration is attributable to two factors: (a) water moves from the concrete specimen into the relatively more concentrated surrounding solution by osmosis, and (b) diffusion of the de-icer into the water within the concrete raises the concentration of electrolyte and depresses the freezing point and amount of freezable water. This depression of the freezing point can be seen in

Figure 15. The weight changes of the specimens during the 7-day storage in the de-icer solutions were as follows:

Solution Conc., % (by wt)	Change in Specimen Weight, g
4	-2
8	-6
16	-14

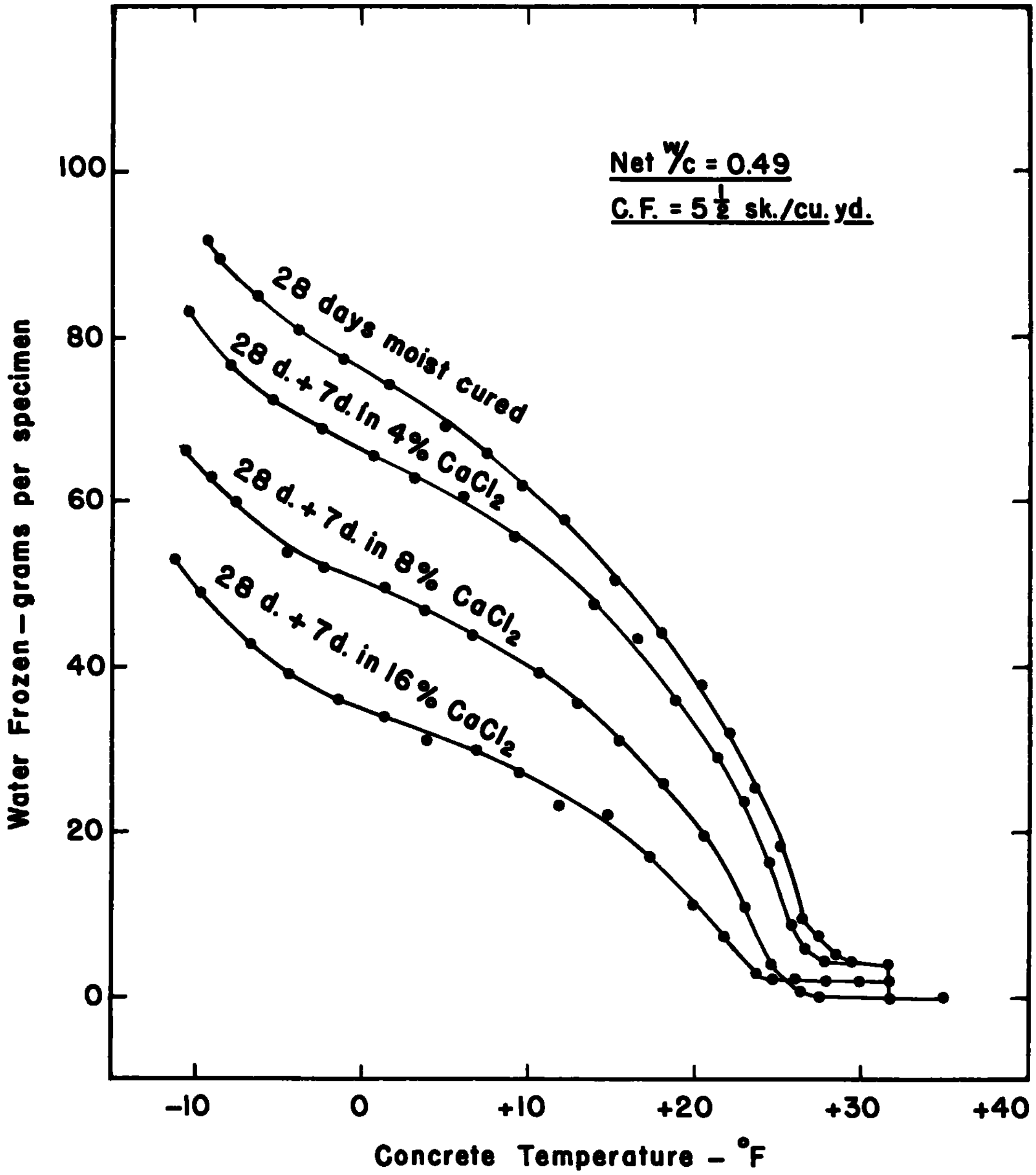

Figure 15. Effect of storage in $CaCl_2$ solutions on amount of water freezing as a function of temperature; concretes cured continuously moist for first 28 days, Type I cement, no entrained air, Elgin, Ill., sand and Eau Claire, Wis., gravel.

Influence of Air Content on Length Change During Freezing

To illustrate the influence of entrained air on the length change during freezing, concrete specimens were prepared using 3/4-in. maximum size aggregate and having air contents ranging from 3 percent (non-air-entraining cement) to 9.1 percent (air-entraining cement plus additional air-entraining agent). After moist curing for 28 days, followed by 3 days in water, the concretes were tested in the calorimeter-strain apparatus.

Figure 16 shows the concrete strain as a function of the amount of water frozen within the various specimens. Somewhat similar relationships are obtained if these strains are plotted against concrete temperature. All of the concretes showed an initial dilation, presumably due to the generation of hydraulic pressures (the forced movement of water during freezing), this being followed by a relief of this pressure. As freezing progresses the non-air-entrained concrete shows further expansion with increase in the amount of water frozen. The air-entrained concretes show

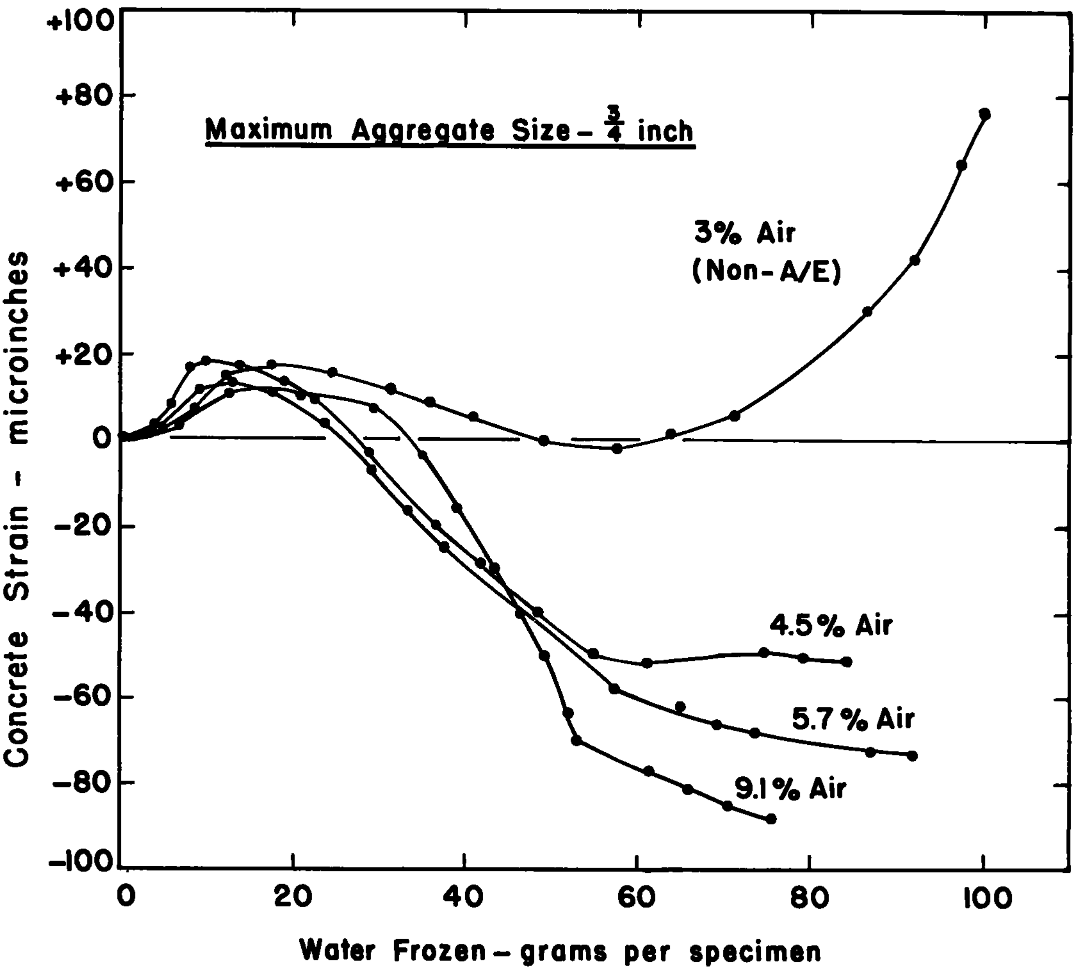

Figure 16. Effect of amount of water frozen on concrete strain for concretes of different air contents; cement content - $5\frac{1}{2}$ sacks per cu yd, slump - 2 to 3 in., curing - 28 days moist followed by 3 days in water, Elgin, Ill., sand and Eau Claire, Wis. gravel.

instead a net shrinkage as freezing continues, the magnitude of this shrinkage increasing with increase in air content. A detailed explanation of phenomena of this type is available in the studies of freezing of pastes conducted by Powers and Helmuth (1). These concretes containing intentionally entrained air showed excellent resistance in the laboratory freezing and thawing test, as shown in the following:

Air Content, %	No. of Cycles for 0.10% Expansion
3.0	92
4.5	850
5.7	1,150
9.1	1,150+*

*Discontinued at 1,150 cycles due to lack of freezer space.

SUMMARY

The calorimeter-strain apparatus described in this report appears to be an excellent means for studying the mechanism of frost action and the influence of various factors on the frost resistance of the concrete. The apparatus enables simultaneous determination of the amount of water actually freezing in concrete and the length changes resulting from this freezing. All measurements are automatically recorded.

The apparatus can be constructed to accommodate any standard laboratory concrete freezing and thawing specimen, thus enabling correlation with actual freezing and thawing tests and "spot" testing of concrete specimens at any time during the standard laboratory freezing and thawing test or after different periods of water saturation.

REFERENCES

1. Powers, T. C., and Helmuth, R. A., "Theory in Volume Change in Hardened Portland Cement Paste During Freezing." HRB Proc., 32:285 (1953).