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Principal Type of Water Responsible for Damage of Concrete Repeated Freeze-Thaw Cycles

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***Abstract*—**The first and basic cause of the failure of concrete is repeated freezing (thawing) of moisture contained in the pores, microcracks, and cavities of the concrete. On transition to ice, water existing in the free state in cracks increases in volume, expanding the recess in which freezing occurs. A reduction in strength below the initial value is to be expected and further cycle of freezing and thawing have a further marked effect. By using some experimental parameters like nuclear magnetic resonance variation (NMR), enthalpy-temperature (or heat capacity) variation, we can resolve between the various water states and their effect on concrete properties during cooling through the freezing transition temperature range. The main objective of this paper is to describe the principal type of water responsible for the reduction in strength and structural damage (frost damage) of concrete following repeated freeze –thaw cycles. Some experimental work was carried out at the institute of cryogenics to determine what happens to water in concrete during the freezing transition.

***Keywords*—**Concrete, frost proof, strength, water diffusion.

I. Introduction

MANY researches [2], [5]-[7], [9] believed that the degree of saturation is a key parameter for the frost resistance of concrete. Due to its porous nature, it may hold considerable amounts of water that may cause frost damage. Cycles of freezing and thawing might result in progressive disruption of the cement paste due to the expansion of the absorbed water on freezing.

The water in concrete can be in three forms [3], [7], [8], [11]:

a) chemically bound water,
b) physically adsorbed water,
c) rewetting water.

By using some experimental parameters [11]-[14] like nuclear magnetic resonance variation (NMR), we can resolve between the various water states, and their effect on concrete properties during cooling through the freezing transition temperature range.

II. Nuclear Magnetic Resonance NMR Studies

Since its discovery in 1946 Nuclear Magnetic Resonance (NMR) [1], [16] has evolved from a scientific curiosity into one of the most powerful spectroscopic techniques. Today it is routinely used as an analytical tool in chemical and bio-chemical research but also in physics, material science and even in geochemistry. It provides information about the physical state of matter. By studying Hydrogen Nuclear Magnetic Resonance (^{1}H-NMR) signals of water in porous materials, it is possible to determine their effect on concrete properties during cooling through the freezing transition temperature range [11]-[14]. NMR studies can provide detailed information about the porous microstructure (porosity, pore-size distribution) of these materials [4], [10].

The proton nuclear magnetic resonance absorption signal measured at various temperatures for small samples of concrete sealed in glass tube, have identified two type of water in concrete [11], [12], Alpha water which is tightly bound by chemical hydration, and Beta water which is weakly bound by physical adsorption and capillary action. These two types are clearly revealed by NMR spectrum of two peaks, the Alpha and Beta peak respectively.

III. NMR Study in a Saturated Mist Cured Concrete Sample with W/C Ratio of 0.37 [11]-[14]

A. Effect of Reducing Temperature

By reducing the temperature from 12°C to -73°C, the observed spectra for a saturated moist cured concrete sample with a water cement ratio of 0.37 is shown in Fig. 1, a change in the relative size of the alpha and beta peak is apparent. The beta peak diminishes in magnitude and totally disappears at temperature of -53°C as the beta water freeze to ice.

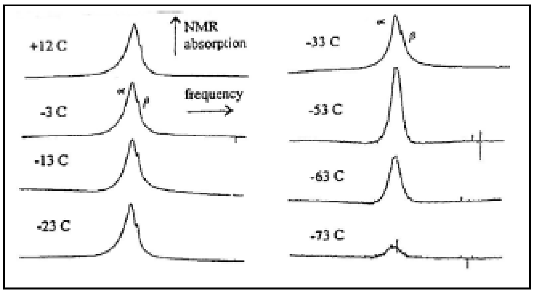

Fig. 1 Proton NMR in a saturated moist – cured concrete sample w/c = 0.37 Effect of reducing temperature [12], [13]

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IV. NMR Study in a Dried and Rewetted Concrete Sample

A. Effect of Cooling

The saturated moist cured concrete sample now is dried to a constant weight and then rewetted in the glass tube and sealed. On cooling to -53°C, the beta peak in Fig. 2 diminish with decreasing temperature, again as the beta water freeze to ice.

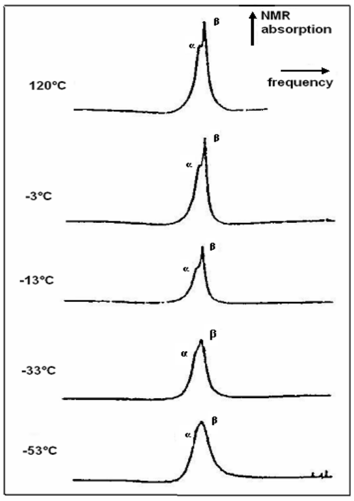

Fig. 2 NMR in dried and rewetted concrete sample. Effect of cooling to -53°C [12], [13]

B. Effect of Heating

On heating to 120°C, the beta peak first grows in relation to the alpha peak, and then diminishes with time. Fig. 3 indicates the relative magnitude of the alpha and beta line after ½ hour, 1 hour, and 10 hours respectively.

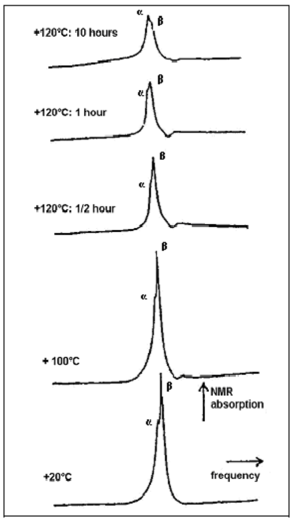

Fig. 3 NMR in dried and rewetted concrete sample. Effect of heating to 120°C [12], [13]

C. Concluding Remarks

It is concluded that beta-water, corresponding to the beta peak is at least part of the evaporable water which is more rapidly removed by heating to 120°C.

It should also be noted that on re-wetting the dried sample, the beta peak reappears hence the beta water.

These examples indicate how NMR spectra provide a qualitative means for observing the behavior of different type of water in the concrete.

V. Enthalpy-Temperature Measurements

The samples used to produce enthalpy-temperature plot down to -80°C were circular cylinders 96mm diameter 200mm high [8], [12]-[14]. The heat capacity measurements were carried out in a large calorimeter. All samples were moist cured.

The enthalpy versus temperature (H-T) curve for dry (sample D), saturated (samples A,B, and C) and re-wetted (sample E) concrete samples are shown in Figs. 4 and 5, the respective differential enthalpy (heat capacity) plots are shown in Figs. 6 and 7.

We note that the H-T plot for the dry sample (D) is a straight line and that the heat capacity is a constant. It is concluded that there is no change of phase tacks place in the water remaining in a dried sample.

The normalized H-T curves for dry, saturated and re-wetted concrete samples and water ice are shown in Fig. 8.

It can be seen that the normalized enthalpy plots are closely similar for samples A, B, and C with different water / cement ratio.

The dried and re-wetted sample E shows a totally different behavior. The excess heat capacity is confined to a narrower temperature range from 0 to -10°C. (90 % of the freezing is confined to a narrower temperature range from 0 to -10°C). The remaining 10% of the freezing appears to take place between -35°C and 45°C.

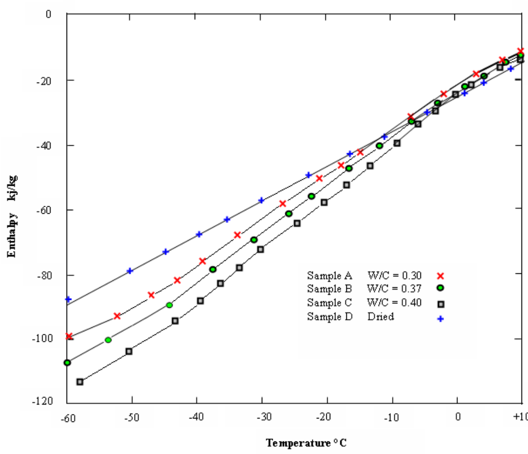

Fig. 4 Enthalpy of concrete samples A, B, C, and D [13]-[15]

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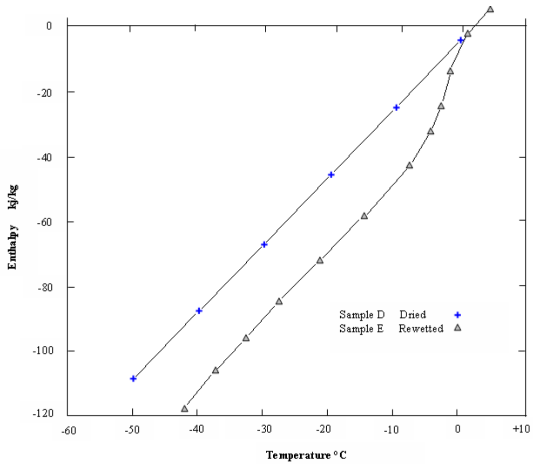

Fig. 5 Enthalpy of concrete samples D, and E [13]-[15]

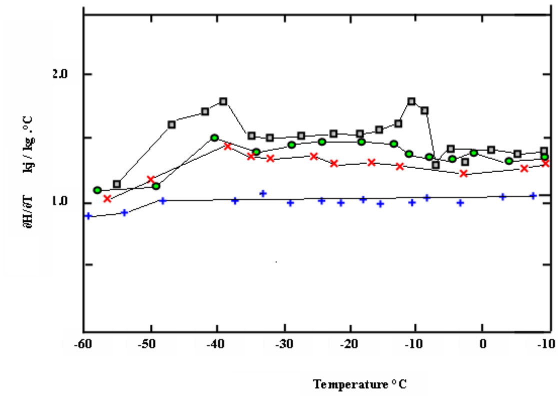

Fig. 6 Specific heat of concrete samples [13]-[15]

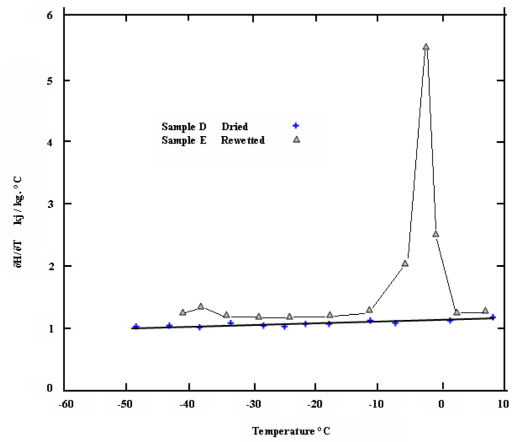

Fig. 7 Specific heat of rewetted concrete sample [13]-[15]

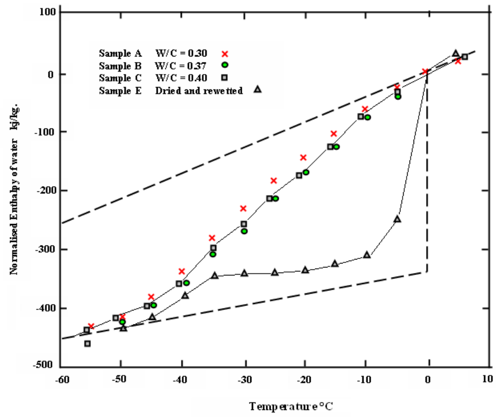

Fig. 8 Normalised enthalpy of water in concrete samples [13]-[15]

A. Concluding Remarks

Some of the evaporable water is strongly physically bound and does not exhibit a latent heat of freezing.

For saturate moist cured concrete samples A, B, and C, the freezing of the non-chemically bound water takes place in a similar manner over the whole temperature range between 0 and -60°C.

For a dried and re-wetted concrete sample E, 90% of the freezing is confined to a narrower temperature range from 0 to -10°C. The remaining 10% of the freezing appears to take place between -35 and -45°C.

VII. Strain Temperature Measurements

Fig. 9 shows the thermal strain behavior of a saturated moist cured concrete (sample C) with a water cement ratio of 0.4. There is dilation on freezing for a saturated sample, however after drying for three weeks in a drying chamber, there is no dilation on freezing. Fig. 10 shows the thermal strain of the re-wetted concrete sample. The dilation and hysteretic behavior has returned.

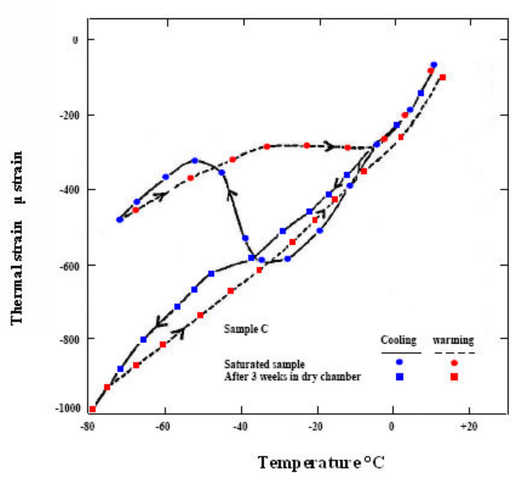

Fig. 9 Thermal strain of moist-cured concrete samples W/C = 0.40 [13]-[15]

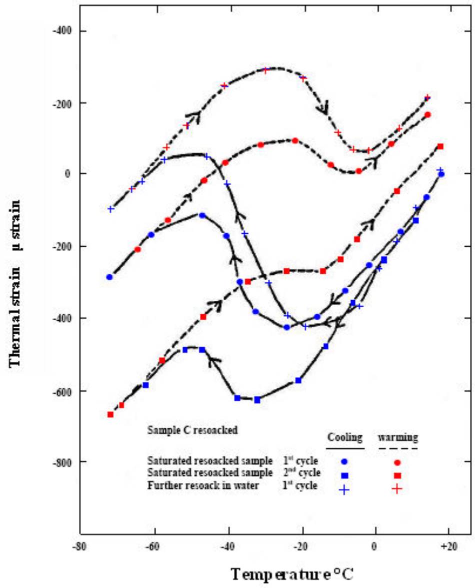

Fig. 10 Thermal strain of rewetted concrete samples W/C = 0.40 after repeated cycles [13]-[15]

VIII. General Conclusion

By using proton (hydrogen) nuclear magnetic resonance (NMR), heat capacity and thermal strain measurement on concrete samples, the following remarks can be made;

- Only some of the physically bound water (Beta water) change phase from liquid to solid with a latent heat of freezing.
- The chemically bound water (Alpha water) does not change state.
- There is a sharp change of state for a rewetting water (Beta water) just below °C

The Beta water is present in a saturated moist-cured concrete or in a dried and rewetted concrete as a mixture of free water and capillary water which constitute a major portion of evaporable water.

These studies have shown that Beta water is almost exclusively responsible for the thermal strain hysterisis behavior and for cracking ect..

When Beta water is absent [13] the cryogenic concrete has repeatable properties of strength, permeability, resistance to cracking etc.

The solution to manufacturing reliable cryogenic concrete is therefore to remove beta water (by appropriate drying) after curing, and to stop beta water re-entering subsequently. The air entrainment is not necessary.

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