Introduction

In the past recent decades, there have been plenty of experimental studies that deals with the frost damage in concrete. The theories and model on the damage mechanism of concrete are usually developed from experimental observations subjecting concrete or mortar specimen under a continuous moisture supply or water submersion during freezing, commonly in accordance to ASTM C-666 (2003). However, it is often taken for granted that in actual conditions concrete seldom reaches full saturation which is different from laboratory tests where specimens are constantly under continuous supply of moisture (water submerged). This is why the relationship between laboratory tests and on site behavior of concrete shows discrepancy which may cause uncertainty in the reliability of the results obtained. When concrete (specimens) are partially saturated as would be the case in actual conditions, deformation behavior may not always show expansion and may otherwise lead to contraction. The contraction during FTC is seldom discussed in literatures due to the lack of available data, understanding and knowledge. It may not be realized that the contraction during FTC plays an important role during FTC and water/moisture behavior may exert stresses on the matrix during freezing. For this reason, the study aims to show that the associated stresses during FTC damage in concrete is not only caused by the expansive stresses during ice formation. These stresses may also cause physical and mechanical property change when frost damage sets in. One important aspect of these changes could be the coefficient of thermal expansion (CTE) of frost damaged samples which is an important component of the total deformation of concrete during FTC. Therefore besides the expansion, contraction should also be considered as an aspect of the frost damage to predict the behavior of concrete during FTC, moreover the changes associated with such phenomena should be considered as well.

More importantly, concrete is a multiphase material and this remain one of the big challenge in understanding concrete’s deformation arising from frost action or other factors. The three phases of concrete are known as the matrix, aggregate and interfacial transition zone (ITZ). The treatment of the matrix can either be the mortar if concrete is considered as the composite material, the hardened cement paste if mortar is considered as the composite material otherwise. The microstructure, density, morphology, and other properties of the phases in concrete are different from each of the other. It is expected then that during frost actions their deformational characteristics is also distinct from each other. Most available studies on frost damage deals with a single composite, concrete, mortar or the hardened cement paste. However, none have presented the individual deformations of the constituent part of the concrete system due primarily to experimental difficulties specifically on obtaining the deformation of the ITZ in real-time. It is therefore the purpose of this study to present a simple yet ingenious experimental method to obtain the deformational behavior of concrete’s constituent parts of mortar, ITZ, and the aggregate phase. The obtained results will not only clarify each of the components role during FTC but during other factors as well. Moreover, the obtained results can be used to understand and simulate the over-all deformation of concrete deformation during FTC.

The results obtained in these investigations, have not been presented before and strongly indicate that they play an integral role in the total deformation of concrete under freeze-thaw cycles. The study is also a part of a series of studies which aims to predict the structural performance of concrete during FTC and with frost damage, the simulation of the damage evolution of concrete is excluded in this study. The development of model and simulation tool is being undertaken by another researcher who considers the core results of this study.

The Deformational Behavior of Mortar during Freezing and Thawing Cycles

Experimental Program

The experimental methods implemented in this study are divergent from previously presented which are commonly based from ASTM C-666. The objective of the experiment therefore is to explore the deformational behavior of mortar during FTC under temperature variation and constant moisture supply using meso-scale size specimens.

Mortar specimens were used in this experimental program. The materials used were ordinary Portland cement with density of 3.14 g/cm³, fine aggregate which is 1.2 mm or less in size with density of 2.67 g/cm³ and having water absorption rate of 1.2%. Mix proportions of mortar in Table 1 are based from ACI 211 (1991) and are without air entraining agent to promote frost damage. Specimens were first cast in a 40 mm x 40 mm x 160 mm form. Specimens were stripped after 24 hours and cured in water for 60 days. For experiment test, specimens were cut from the demolded specimens into a size of 40 mm x 40 mm x 2. This size is on the scale of meso-scale in order to have uniform moisture and temperature variation in the entirety of the specimen which results in a uniform frost damage. The meso-scale size can be used to simulate the frost actions in a specific (localized) location of a bulk sample with the similar conditions (i.e. uniform moisture and temperature variation throughout the sample).

Moisture content of the specimens was removed by oven drying to 105 °C for 24 hours or until all evaporable water is
removed; that is, when the specimen’s weight remains unchanged. The purpose of drying was to prepare the specimens for strain gauge attachment and to be used to calculate the thermal strains. For strain measurements of dry specimens, attachment of strain gauge quickly follows as well as sealing the specimens after oven drying to prevent considerable moisture uptake from the atmosphere. While fully (100%) saturated specimens were submerged under water and subjected in a vacuum condition to effectively remove entrapped air inside the specimen. After saturation were first sealed with polyvinylidene chloride plastic or Saran. Mastic/vinyl tape is then used to seal the specimens to prevent moisture uptake and loss. The preparation of specimens is displayed in Fig. 1. To check the effectiveness of the sealing method, the mass of the specimens were checked before and after FTC using a similar sample with the same sealing technique however without the attachment of strain gauges to avoid added water that could be attached on the gauge’s lead wires which could affect the total mass of the specimens. The specimens exhibits the same mass before and after testing, thus verifies that the specimens moisture content remains constant throughout the tests. The strain gauge used are self-temperature compensation gauges having base size of 4 x 2.7 mm, length of 1 mm and resistance of 120 Ω, lead wires were 3-wire cable, and adhesive was made of polyurethane. All were designed for low temperature strain measurement.

Freeze-thaw cycle tests was accomplished by using an environmental chamber capable of controlling the temperature and the ambient relative humidity (RH). Sealed specimens as seen in Fig. 2 were put inside the chamber. The temperature cycle was set-up as shown in Fig. 3 which was repeated 30 times for all specimens under different moisture conditions. Sensors records and measures the temperature while strain gauges measured the induced strain during FTC. While the environmental chamber can control the RH, its effect on the specimens is insignificant since specimens are completely sealed.

![Fig. 2 Experimental set-up](image)

**Results and Discussions**

Figure 4 shows strains for absolutely dry specimens, from all specimens’ results it can be observed that during the whole FTC the behavior of the strains remains the same though the number of cycle increases. This behavior is due to the absence of water in the specimens and only deformation caused by the effect of CTE of the material is observed which depends on the temperature change. Obviously, with the absence of moisture there will be no frost damage.

![Fig. 4 Dry specimens’ strain (undamaged)](image)

Slight differences in thermal strain can be observed from each specimen (Sicat et. al 2013). This difference is attributed to the fact that the CTE of mortar is affected primarily by the amount of its constituent parts – fine aggregate and hardened cement paste. Fine aggregates have much lower CTE (0.3 to 5.4 x 10⁻⁶/°C) in comparison with hardened cement paste (11

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**Table 1 Mix proportions of mortar**

<table>
<thead>
<tr>
<th>Mixture</th>
<th>W/C (%)</th>
<th>Water (kg/m³)</th>
<th>Cement (kg/m³)</th>
<th>F. Aggregate (kg/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>70</td>
<td>207</td>
<td>296</td>
<td>1090</td>
</tr>
<tr>
<td>B</td>
<td>50</td>
<td>207</td>
<td>414</td>
<td>1090</td>
</tr>
<tr>
<td>C</td>
<td>50</td>
<td>207</td>
<td>414</td>
<td>990</td>
</tr>
<tr>
<td>D</td>
<td>50</td>
<td>207</td>
<td>414</td>
<td>755</td>
</tr>
<tr>
<td>E</td>
<td>30</td>
<td>207</td>
<td>690</td>
<td>755</td>
</tr>
</tbody>
</table>
the amount of hardened cement increases (and fine aggregates decreases) the higher will be the CTE as with specimen having 30% W/C referring to the thermal strains.

While dry specimen’s strain has uniform strain behavior during the entire FTC, saturated specimen’s total strain behavior on the other hand is more complicated as shown in Fig. 5. The primary cause of this complicated strain behavior is due to the fact of the presence of moisture. To understand the effect of FTC, the thermal strains is removed using CTE obtained from dry specimens in Fig. 4, the strain induced by moisture behavior which causes damage during FTC is then obtained.

Once the thermal strains are removed as shown in Fig. 5, expansion is the dominant strain behavior in the initial FTC of fully (100%) saturated specimens (70%-1090 FA, 50%-1090 FA, 50%-990 FA and 50%-755 FA) except for 30%-755 FA (% is water to cement ratio and FA denotes amount of fine aggregates in kg/m$^3$) which displays purely contraction. The expansive stresses are said to be the product of the volume expansion of water when it turns into ice assuming that the pores are water filled. Based on the hydraulic pressure theory, a temporary hydraulic pressure is produced causing the abrupt expansion when water suddenly freezes (Sicat et al. 2013). The expansion of frozen water may cause tensile stresses in the surrounding matrix and if the stresses exceeds the tensile strength of the matrix, microcracks develop which increase the pore volume of the mortar specimens. It should be noted that the specimens are sealed and supply of moisture is absent which prevents the continued increase in strain as the FTC progresses. Upon reaching the maximum deformation the maximum strain slowly decreases as amount of pore moisture decreases. This moisture reduction is contributed by the sealing method employed which allows the expanding water to flow to the surface or the hydrodynamic relaxation effect (Ciardullo et al. 2005) in addition to the pore size increase due to microcracking. The partial saturation of the pore system serves as the pre-requisite for the contraction of the concrete system. When ice forms in a partially saturated pore, the ice causes thermodynamic imbalance which causes either chemical potential difference and pressure difference between smaller pores and ice in larger (and partially saturated) pores where the contraction behavior initiates. The resulting negative pressures drives out the water in smaller (<0.05µm) or gel pores which causes the contraction of the system as seen in the later stages of FTC in Figs. 5a-d.

For 30%-755 FA on the one hand, contraction is observed during the entire FTC which suggests that specimens could already be partially saturated even before the FTC begins. The low W/C ratio of the specimen may result in the low pore volume with high percentage of small/fine pores (Okpala 1989). During saturation process it may be difficult for moisture to penetrate or saturate the pores resulting in partial saturation. Setzer (2002) explains that during freezing if there is a sufficient free space, the frost shrinkage or the contraction of the gel predominantly controls the deformation as what is observed in the increasing contraction of the specimen.

Fig. 5 Fully saturated specimens’ strain to $20 \times 10^{-6} ^\circ\text{C}$ (Sicat et al. 2013). Therefore the larger the amount of fine aggregates (referring to Table 1) the lower the CTE as in the case for the thermal strains of 70% w/c, while
The Change in Coefficient of Thermal Expansion and Elastic Modulus of Mortar during Freezing and Thawing Cycles

The aim of the experiment in this section is the collection of data in order to present the drastic changes of the CTE and elastic modulus of mortar during FTC.

The preparation the specimens used in the experimental study is similar as previously discussed where specimens are cut into meso-scale size, dried and conditioned to be fully saturated. No partially saturated specimens were prepared in this experiment. Besides the freeze-thaw cycle tests, elastic modulus tests and X-ray Computed Tomography observations were also done to relate with the change in coefficient of thermal expansion (CTE) of frost damaged specimens. Specimen sizes were 40 mm x 40 mm x 2 mm used for FTC strain tests, 70 mm x 30 mm x 5 mm used for determination of the elastic modulus and 10 mm x 10 mm x 3 mm for X-ray micro computed tomography (CT). Similarly, sealing of specimens were also done prior to the final sealing using mastic tape for all specimens under different testing.

Results and Discussions

The summary of the evolution of the elastic modulus of specimens after FTC is shown in Fig. 6. It can be seen that just after the first FTC, there is already a large decrease in the elastic modulus of all the specimens. The average decrease in elastic modulus at this point is 31%. In the following 3rd and 5th FTC, the average decrease in elastic modulus from its initial value is 34%, indicating that there were no or very little variation in the decrease of elastic modulus from the 1st cycle. This implies that frost damage is associated with the amount of moisture inside the specimen. The results are also in agreement with the strain behavior of the specimens wherein the maximum positive strains were reached during the 2nd to 4th FTC, also suggesting the association of the maximum strain with the moisture content of the specimens. On the 30th cycle, the average decrease of the elastic modulus is 52%, which could be accumulated by the repeated cycles.

The void ratio at every FTC (during the first five cycles) until the 10th FTC are shown in Fig. 7. Handling damage occurred during the 20th to 30th cycle for 70%-1090 FA specimens due to fragility caused by FTC damage, the results from 0 to 10 FTC are shown instead. It’s indicated that as the w/c ratio of specimens decreases, so does their void ratio. In addition, the specimens with a large amount of fine aggregates, 70%-1090 and 50%-1090 FA, also display greater void ratio. For specimens with low w/c and less fine aggregates, 50%-990 FA, 50%-755 FA and 30%-755 FA specimens, a low void ratio is observed. More importantly, during the first five FTC, there is a gradual increase in void ratio for all specimens. During the 10th FTC, no additional increase in void ratio was observed for most of the specimens. However, for the 70%-1090 FA specimen, an increase in void ratio is still visible, indicating the continuous formation of microcracks. The increase in void ratio seems to be in agreement with the strain results of specimens, where the maximum positive strain happens during the initial stage of the FTC, causing FTC damage to the specimens. The same is true for the elastic modulus test, where the decrease happens at the initial stage of the FTC, particularly in the 1st to 5th FTC.

The change of CTE of concrete could be elucidated based on its similarity with the modulus of elasticity in terms of its relation with the change in microstructure of concrete. Such change reduces the elastic modulus of concrete. Relating CTE and elastic modulus, when the decrease in elastic modulus occurs after the 1st FTC (see Fig. 6), the CTE significantly increases (Fig. 8). In the succeeding 3rd and 5th cycles, the elastic modulus remains almost the same for the majority of the specimens, and the same is true for the CTE. However, when the elastic modulus reduced in the 30th cycle, the CTE of the specimens was in the same range as the previous cycle with the exception of 70%-1090 FA, where the change in CTE continued until the last cycle. In this case, damage evolution probably continued to affect the CTE until the 30th FTC. These findings suggest that there seems to exist a level of damage reflected in the reduction of elastic modulus where the change in CTE is maximum and remains uninfluenced in the succeeding FTC as seen in Fig. 8. This finding also agrees with the increase in void ratio that takes place mostly at the initial FTC from the 1st to 5th FTC, indicating microcracks development.
The mechanism behind the response of concrete to CTE remains unclear, moreover the physical mechanism how its CTE changes. However, since the only physical change during frost damage is microstructural change due to microcracking, it is practical to say that this is what causes the change in CTE. This can be explained further in terms of the effect of microcracking on thermal restraint between mortar’s constituent parts in the succeeding discussion.

Aggregate has lower CTE than hardened cement paste and greatly affects the CTE of concrete depending on its volume and properties (Sicat et al. 2013; Al-Oztaz 2007). When incorporated into concrete, it restrains the thermal movement of hardened cement paste. Due to frost damage, microcracks occur as presented in the current experimental results represented by the increase in void ratio, and can act as broken bridges or links detaching the aggregate from the hardened cement paste. This reduces the thermal restraint that each constituent part exerts on the other. The hardened cement paste can then expand or contract freely, thus significantly altering the CTE of the whole composite. The existence of the interfacial transition zone (ITZ) due to its high porosity and low strength also demonstrates that the detachment between aggregate and cement paste can take place due to microcracking. It is verified from SEM observations that after FTC exposure, cracks are mostly seen in the interface between cement paste and aggregates.

The Deformational Behavior of the Interfacial Transition Zone and Aggregate Phase during Freezing and Thawing Cycles

Studies regarding the ITZ suggests that the size and properties of the ITZ is mainly affected by the type, shape and mineralogical properties of the aggregate used (Sicat et al. 2013). To obtain uniform effects of the aggregate used on the ITZ width, 15 mm diameter cylindrical aggregates were cored from a single parent rock. The major factor that affects the ITZ property in this case is the water/cement ratio (w/c) used in each mixture. The treatment of the ITZ in this study is the ITZ around the coarse aggregate, the mortar in this case includes the hardened cement paste, fine aggregates, and ITZ around them. Mortar is thus treated as a single material (the matrix) since meso-scale approach is used in this study.

For the concrete proportions, the mortar mixture was also based on ACI 211.1 with the same mix proportions however in this case only three mix proportions are used (70%-1090 FA, 50%-990 FA and 30%-755 FA). Prior to casting the mixture, the cored aggregate was first washed to remove any dust and dried at room temperature for 24 hours. The 15 mm diameter cored aggregate were then positioned in the middle of 40x40x160 mm form supported by a thin plastic brace to maintain their position during casting. The space around the aggregate was filled with mortar while vibrating to obtain a good compaction during casting. Samples were stripped after 24 hours and cured under water for sixty days. Once cured, specimens were prepared by cutting the samples into meso-scale size of 40x40x2 mm the typical specimens used in this study.

When specimens are dry enough after about 24 hours, strain gauges were attached on all phases of the specimen as can be seen in Fig. 9: \( G_M \) is strain gage on mortar, \( G_{AM} \) is strain gage on the aggregate-mortar boundary, and \( G_A \) is strain gage on the aggregate. By this method, strains can be obtained from the aggregate phase, mortar phase, and mortar-aggregate boundary. The gage attached on mortar-aggregate boundary (\( G_{AM} \)) is observed under an electron microscope in order to measure the length of the gage’s portion attached on the mortar and aggregate phases. The measurements will be used to obtain the deformation history of the ITZ during FTC. Dry and fully saturated specimens were tested in this experiment. Sealing was also employed after moisture conditioning, then subjected to 20 FTC.

To obtain data, an approximate width of the ITZ was measured using an electron microscope wherein a distinct line was observed at the mortar and aggregate boundary. The basis for this distinct line which is recognizable from the observations is a thin shell of CH film and elongated CSH particles depleted on the aggregates and has been described previously by Basheer et al. (2005) and Cwirzen and Pentalla (2005). Depending on the w/c ratio, the ITZ width will be wider for high w/c and narrower for lower w/c. This is true for the current study where the lowest w/c (30%) has narrow ITZ width and widest for the highest w/c (70%). The obtained values are in agreement with the given descriptions from previous investigations where the ITZ width is 10-50 \( \mu \)m (Hussin and Poole 2010, Cwirzen and Pentalla 2005, Basheer et. al. 2005).

The strain gage attached on the mortar-aggregate boundary (\( G_{AM} \)) shown in Fig. 9 was particularly observed using an electron microscope. The illustration in Fig. 10 shows the magnified lengths of the portions attached on each of the phases, on mortar – \( L_M \), aggregate – \( L_A \), and on the ITZ \( L_{ITZ} \) were measured. The total deformation \( \Delta L_M \) on \( G_{AM} \), then it is composed of the deformations on the attached portions - \( \Delta L_A \) for the aggregate deformation, \( \Delta L_M \) for mortar and \( \Delta L_{ITZ} \) for the ITZ. From the strain relationship, the total deformation (\( \Delta L \)) of the constituent parts (aggregate, mortar, and ITZ) can be obtained. Since the samples are very thin and on the meso scale, moisture and temperature variation are uniform in every location. It can be assumed that the strains obtained from any location of the constituent parts are uniform as well. Using Eq. 1 the total deformation (\( \Delta L_{ITZ} \)) of the ITZ can then be calculated where \( \varepsilon_{AM} \) is the strain on the aggregate-mortar boundary (\( G_{AM} \)), where \( \varepsilon_A \) is strain obtained aggregate phase (\( G_A \)) and \( \varepsilon_M \) is strain obtained mortar phase (\( G_M \)).

\[
\Delta L_{ITZ} = \varepsilon_{AM} \cdot L_M - \varepsilon_A \cdot L_A - \varepsilon_M \cdot L_M \tag{1}
\]
Results and Discussions

The average deformations during FTC obtained from different moisture conditions are shown in Fig. 11. The behavior of the ITZ is obtained based on the average of three specimens.

For dried condition in Fig. 11a, for the three sets of specimen (70%, 50% and 30% w/c) uniform behavior is observed, indicating that there is almost no deformation during the FTC. Comparing this with saturated specimens, the deformation may be insignificant considering the very high deformation of the saturated case in Fig. 11b. As have been pointed out in the previous discussions, moisture is the primary cause of frost damage in concrete and with moisture absence, dry concrete is simply not affected by FTC. This fact also applies for the ITZ deformation. Specimens for dry condition shows non-existence of damage in the ITZ during the FTC evident on the non-changing deformation during the cycle. However, for fully saturated case large deformation and variation is observed between the specimens. The maximum ITZ deformation (positive deformation) in the first cycle and negative deformation during the later stages of FTC for 70% specimen is greater than 50% and 30% w/c specimens. Increasing the w/c increases the porosity of the matrix and of the ITZ (Cwirzen and Pentalla 2005). In effect, the higher the porosity, the higher will be the presence of total pore water volume in the transition zone. This suggests that the higher water volume present in pores would yield in greater volume of water that could expand resulting in higher deformation. The large increase in ITZ deformation for 70% and 50% fully saturated specimens observed at the initial FTC is similarly attributed to the moisture behavior during freezing. The same patterns is observed for mortar’s deformation however since the ITZ is higher in porosity then higher moisture presence will cause higher deformation in both expansion and contraction than mortar. The deformation obtained in this study agrees very well with the occurrence and non-occurrence of cracks in the ITZ observed in the study which suggests that the methods employed here are reliable (Sicat et al. 2013).

References:


