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Experimental Investigation of the Deformational Behavior of the Phases of Concrete during Freezing and Thawing Cycles

**凍結融解繰返し作用下のコンクリートの各相の変形挙動に
関する実験的研究**

By

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A thesis submitted in partial fulfilment of the requirements for the degree of Doctor of
Philosophy in Engineering

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ABSTRACT

This study presents the diverse experimental methods and results on the deformational behavior of the phases of concrete (mortar, aggregate and interfacial transition zone - ITZ) in meso-scale during freeze-thaw cycle (FTC). The FTC damage in concrete has long been a durability related problem in cold region like here in Hokkaido. This is why there is a large volume of research to clarify and understand the frost damage in concrete. However in spite of the large volume of research there are no consensus on the damage mechanism of concrete due primarily to concrete's heterogeneous characteristics and the complex behavior of moisture during FTC. With this regard, it is the purpose of this study to present the deformational behavior of the constituent part of concrete (mortar, aggregate and ITZ) which is believed to be distinct from each of the other to clarify each of their role during FTC and the overall deformation of concrete.

Unlike the common methodology in frost damage in concrete, specimens were sealed in the experiment so that moisture and temperature can be controlled. In addition, meso-scale size specimens are used in this study. The meso-scale size can be used to simulate the frost actions in a specific (localized) location of a bulk sample with conditions similar with the data used. Specimens were subjected to FTC under different moisture conditions. The increase in void ratio and decrease in elastic modulus associated with frost damage have been investigated and are related to the CTE change of frost damaged specimens. The study also investigates the deformational behavior of the ITZ which have not been presented in the microstructural investigation of concrete. The behavior of aggregate during FTC has been investigated as well.

Based on the experimental results, the findings indicate that the deformational behavior of the specimens under FTC is primarily dependent on the amount of its moisture. The larger the amount of moisture, the larger will be the resulting deformation in both expansion and contraction behavior. The expansion during FTC is understood and pointed to be caused by the hydraulic pressure caused by ice formation during FTC. The contraction phenomena is also observed prior to the expansion. Due to constant moisture content, a limitation in the increase in tensile strain is observed during FTC and this tensile strain decreases until contraction is observed. The contraction is attributed to the removal of gel pore water arising from negative pressures. With an increased pore volume (void ratio) due to microcracks arising from frost damage and more importantly absence of available water supply, the specimens becomes partially saturated and the displaced pore water cannot be refilled, thus the sample cannot re-expand and results in contraction at the end of the FTC.

After FTC tests, results show that the CTE of frost damaged mortar increases and its elastic modulus decreases which are primarily caused by microcracking when frost damage takes place. The reduction in elastic modulus has been related to the increase in void ratio which represents the formation of microcracks of frost damaged specimens. Microcracks act as broken bridges or links which can detach the aggregate from the hardened cement paste and in effect reduces (or removes) the thermal restraint that fine aggregate and cement paste exerts on the other. The hardened cement paste can then expand or contract more freely under temperature variation, and thus can significantly affect (increase) the CTE of the whole composite (mortar). The stress transfer in the material is prevented as well due to microcracking resulting in elastic modulus reduction.

On the one hand, the aggregates did not indicate considerable permanent deformation during the whole FTC though the specimens were conditioned to be fully saturated. This verifies the common acceptable knowledge that aggregate are unaffected by frost damage. The ITZ deformation is determined as distinct (being far higher) from the mortar and aggregate which is the crucial reason why the ITZ is treated as a separate phase in concrete. Besides its weaker strength, the much higher deformation of the ITZ during FTC where cracking and prominent damage are initiated and could extend to the matrix shows the important role of the ITZ during frost damage and other factors such as wet dry cycles (WDC). The obtained deformational behavior of the ITZ, mortar, and aggregate in this investigation reflects their commonly described behavior during FTC. The ITZ and mortar deformation agrees well with the observations on the occurrence of cracks for frost-damaged specimens and non-occurrence of cracks for dry specimens. These indicates the reliability of the presented method and suggests that the experimental method can be used as a basis to obtain the deformational behavior of mortar, aggregate and ITZ during FTC. Significantly, the results presented here are first and a complete data on the deformational behavior of mortar, aggregate, and ITZ have not yet been presented. The results and methodology presented in this study is therefore important in clarifying, understanding and simulation of frost damage in concrete.

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Chapter 1

INTRODUCTION

1.1 BACKGROUND

Concrete is the backbone of modern construction, it is used more than any other man made material in the world because of its desirable qualities which include strength, economy and durability. However, like other highly porous media it has the ability to absorb and retain moisture. This characteristic has an important consequence since unprotected concrete structures in contact with water are usually susceptible to frost damage. A typical example of concrete surrounded by ice (snow) is shown below, the surrounding ice will eventually melt when temperature rises above freezing temperature and will permeate into the concrete.



Fig. 1.1 Concrete with surrounding ice

Frost damage in concrete has long been a major deterioration problem in cold regions, like in Hokkaido. Frost damage deteriorates the structural performance of concrete structures such as safety and serviceability due to reduction in strength and stiffness. Frost damage not only degrades the aesthetics of concrete structures due to surface scaling but also decreases its durability and enhances its susceptibility against other deterioration factors such as chloride attack, carbonation, alkali silica reaction (ASR) and chemical attack.

In literatures it can be found that different theories were devised to explain the frost damage mechanism of concrete which led to the basis of the current understanding. The first and probably most well-known damage mechanism has been explained in the classic work of Powers (1955) - the hydraulic pressure theory, where a temporary hydraulic pressure is produced in the pores caused by the expansion of pore water when it freezes. The theory was then extended to the osmotic theory (Korhonen 2002), where pressures are created by differences in concentration of solutes left in the remaining unfrozen water after initial pore

fluids in capillary cavities froze and raised the solute concentration of the remaining frost fluid. Recently, the micro-ice lens (Setzer 2002) model and crystallization pressure (Schererer and Valenza 2005) of ice were developed to explain the frost damage mechanism. These are some of the recognizable frost damage mechanisms in literatures. Notably, most of these theories are usually developed from experimental observations subjecting concrete or mortar specimen under a continuous moisture supply or water submersion during freezing which is in accordance to ASTM C-666.

Frost damage in concrete has been one of the major topic of research in the past several decades in concrete due to its complex phenomenon. However, due to the multifaceted characteristics of concrete and complicated moisture behavior of pore water during freezing and thawing actions, no consensus has been widely accepted as mechanism of frost damage. In order to understand and formulate frost damage models of concrete during FTC, some basic information must at least be known for the common reader. One of which is that, when water turns into ice its volume expands by 9% which causes the expansive stresses in pores during freezing, ice formation also cause thermodynamic imbalance causing moisture movement. The primary factors that influence frost damage in concrete should also be noted are discussed briefly in the next section.

1.1.1 Primary Factor that Influences Frost Damage

Pore Structure of Concrete

The pore structure in a concrete system is perhaps the primary reason why deterioration of concrete takes place. The pore system in concrete are actually interconnected, thereby allowing the passage of moisture in and out of concrete. When moisture penetrates the concrete system, the associated deterioration take place, one of which is frost damage. The more pores present in concrete (porosity), the weaker it becomes and more it becomes susceptible to various deteriorations. Hence, the pore structure inside concrete is fundamental to understanding the freeze–thaw resistance of concrete (Korhonen 2002).

The size of the pore influences the temperature at which water will freeze. The volume, radius, and size distribution of pores decide the freezing point of pore solution and the amount of ice formed in pores. The Comité Euro-International du Béton (1989) design guide reports that due to the wide range of pore sizes in concrete only 2/3's of the pore water will be frozen at -60°C. In a concrete microstructure there three are kinds of pore sizes namely gel pores, capillary pores and entrained air which are defined below.

- Gel Pores - Consists of a system of very fine pores within the dense packing of cement hydration products. The radii of these pores are very small. Water present in this class of pores seldom freezes under usual freezing conditions of concrete use. However, water in gel pores exerts hydrostatic tension (and smaller capillaries with size of $< 0.05\mu\text{m}$) removal of pore water collapses the pore walls. (pore size $0.0005 - 0.01\mu\text{m}$) (Mehta and Monteiro 2006).
- Capillary Pores – These pores are the remnant of the original water filled spaces of fresh concrete mix. These pores are larger than the gel pores. Layers of cement hydration products with associated gel pores separate individual capillary pores from each other. The capillary pores are contiguous with the gel pores so that water can move in and out of the capillary pores with changes in the ambient conditions. Water present in the capillary pores freezes under usual freezing conditions which arises the phenomena for frost actions such

as expansion when water freezes and contraction driving out water from smaller pores and gel pores. These pores are perhaps the most important in frost damage in concrete. The smaller the capillary size the lower the freezing temperature (pore size 0.02 – 10 μm) (Mehta and Monteiro 2006).

- Entrained-air - These are mainly entrained air bubbles present in air-entrained paste or concrete. The sizes of these air bubbles are very much larger than the other two classes of pores. These air bubbles are supposed to be empty. Normally the capillary pores are separated from the air bubbles by layers of cement hydration products with associated gel pores. (recommended at 50 μm) (Mehta and Monteiro 2006).

Capillary pores can be considered as the most crucial pores in frost damage in concrete since water in them freezes quite easily. Capillary porosity is entirely dependent on the water-to-cement ratio because it defines the space that must be filled by the hydration products. Though all pores in cement paste are interconnected, capillary pores are mainly responsible for any appreciable movement of water through the concrete system and, as we will see, for its vulnerability to frost damage (Korhonen 2002). In this sense, the higher the porosity the more vulnerable it is to frost damage.

Degree of Saturation of Concrete

If the water within concrete's system of pores can freeze and generate disruptive pressures frost damage will eventually take place. According to Fagerlund (2002) there exist a moisture content that is fairly well defined, and that marks the border between frost resistance and frost damage. This moisture content seems to be almost uninfluenced by the number of freeze-thaw cycles, at least up to about 100 cycles. The critical moisture content is individual for each material. It is comparable with the fatigue limit in normal mechanical fatigue, since moisture contents are too low to be able to cause damage. In addition, Fagerlund (2002) also added that frost damage is directly proportional to the amount by which the critical moisture content is exceeded, indicating that the internal stresses increase in proportion to the amount of frozen water above critical. However, these stresses being pointed out are only associated with the hydraulic pressure generated when water forms into ice. On the one hand, Setzer (2002) explains that during freezing if there is a sufficient free space (partial saturation in pores), the frost shrinkage or the contraction of the gel predominantly controls the deformation which may also cause disruptive pressures. The amount of moisture in this sense is below the critical moisture content mentioned by Fagerlund (2002). In this regard, the amount of moisture is significant on the deformation of concrete during FTC as it could influence whether concrete would experience contraction or expansion.

1.2 STATEMENT OF PROBLEM AND OBJECTIVES OF THE STUDY

In the past recent decades, there have been plenty of experimental studies that deal 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 currently being undertaken by another researcher who considers the core findings of this study. Forgot

1.3 OUTLINE OF THE DISSERTATION

Chapter 1 presents the background of the study on the damage of concrete under FTC, the objectives, scope and delimitations of the study, and finally the outline of the study.

Chapter 2 describes the primary experimental methodology on freeze-thaw cycles (FTC) of meso-scale size specimens at varying temperature and constant moisture conditions. Divergent to most studies on frost damage in concrete where an increasing expansive deformation are observed, the results exhibits large expansions at the initial cycle which gradually decreases to contraction at the later stages of the FTC.

Chapter 3 describes that CTE of frost damaged mortar increases while its elastic modulus decreases, primarily owing to microcracking when frost damage sets in. Microcracks act as broken bridges that can detach the aggregate from the hardened cement paste and in effect reduces the thermal restraints that each part (fine aggregate and cement paste) exerts on the other. The hardened cement paste can then expand/contract more freely under temperature variation, and thus can significantly affect (increase) the CTE of the whole composite (mortar). Further, stress transfer in the material is prevented due to microcracking resulting in elastic modulus reduction.

Chapter 4 presents the results and experimental methodology in obtaining the real-time deformational behavior of the interfacial transition zone (ITZ) of concrete during freeze-thaw cycles (FTC). The investigation shows that the ITZ exhibits higher deformation than the matrix and aggregate due to its high porosity and weakness. The findings verify that aggregates are insignificantly affected by FTC while mortar show similar deformation tendency to ITZ. The results also show good correlation between obtained ITZ deformation and observed cracks after FTC. The findings suggests that the experimental methodology presented can be used to observe and approximate the deformational behavior of ITZ during FTC actions.

Chapter 5 summarizes the work done and significant findings in this study.

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Chapter 2

THE DEFORMATIONAL BEHAVIOR OF MORTAR DURING FREEZING AND THAWING CYCLES

2.1 BACKGROUND

Frost damage of concrete has long been a major deterioration problem in cold regions. This makes frost damage in concrete one of the major topic of research in concrete the past several decades. However, due to the multifaceted characteristics of concrete and complicated moisture behavior of pore water during freezing and thawing actions, no consensus has been widely accepted as mechanism of frost damage. Some of the most recognizable mechanism to explain the damage mechanism of concrete includes the hydraulic pressure theory (Powers 1945), osmotic pressure theory (Powers and Helmuth 1953) and recently the micro-ice lens model (Setzer 2002, 2001) and crystallization pressure (Scherer and Valenza II 2005). Notably, most of these theories are developed from experimental observations subjecting concrete or mortar specimen under a continuous moisture/water supply or by water submersion during freezing which is in accordance to ASTM C-666. Typically the observed behavior of concrete during FTC subjected to these investigations is an increasing expansive damage due to the stresses associated with the expansion of water. It is often taken for granted that in actual conditions concrete seldom reaches full saturation which is quite different from laboratory tests where specimens are constantly under continuous supply of moisture (water submerged) and this is why the relationship between laboratory tests and on site behavior of concrete shows discrepancy.

When concrete (specimens) are partially saturated as would be the case in actual conditions for vertical structures (Chatterji 1999), 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. Exploring the deformation behavior of mortar including the contraction during FTC could be instrumental in understanding the damage mechanism in concrete particularly on the on-site behavior of concrete. In this regard, this study attempts to discuss the associated mechanism during FTC and the observed contraction behavior during frost actions to further clarify and understand the damage mechanism in concrete.

2.2 EXPERIMENTAL PROGRAM

The experimental methods implemented in this study are divergent from previous experimental studies 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.

2.3 PREPARATION OF SPECIMENS

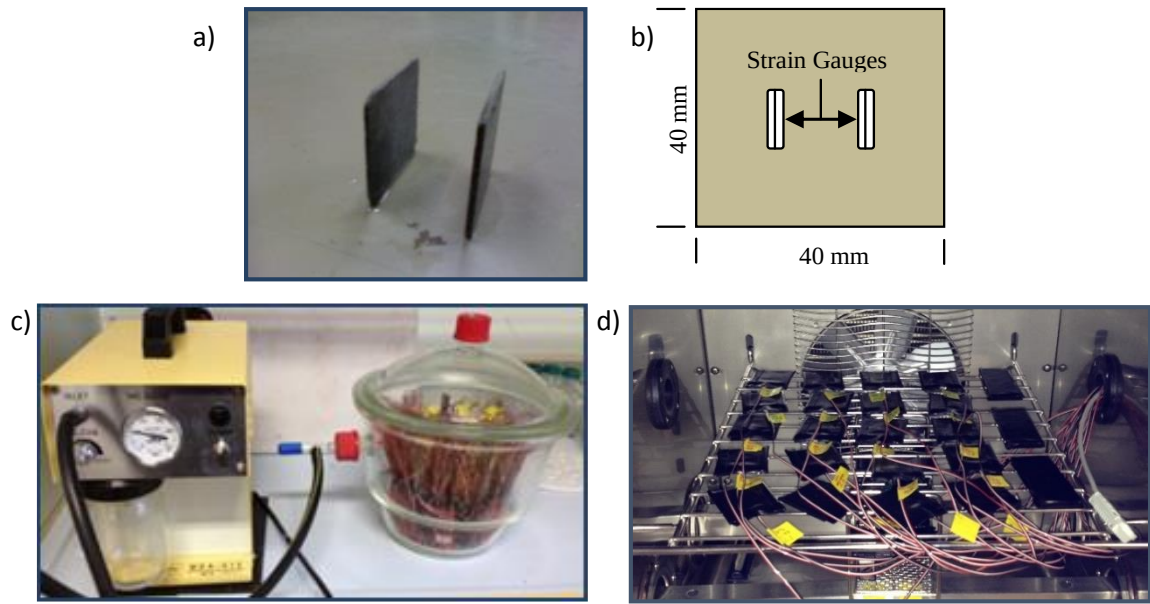
Mortar specimens were used in this experimental program. The materials used were

ordinary Portland cement with density of 3.14 g/cm^3 , fine aggregate which is 1.2 mm or less in size with density of 2.67 g/cm^3 and having water absorption rate of 1.2%. Mix proportions of mortar in Table 2.1 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 which can be checked 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. 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 120Ω , lead wires were 3-wire cable, and adhesive was made of polyurethane. All were designed for low temperature strain measurement.

Table 2.1 Mix Proportions of Mortar and Calculated CTE

Mixture	Water Cement Ratio (%)	Water (kg/m^3)	Cement (kg/m^3)	Fine aggregate (kg/m^3)	Average CTE ($\times 10^{-6}/^\circ\text{C}$)
A	70	207	296	1090	8.63
B	50	207	414	1090	8.83
C	50	207	414	990	9.04
D	50	207	414	755	9.37
E	30	207	690	755	9.73

For the attainment of different moisture content, specimens were exposed to different conditions. For fully (100%) saturated condition, specimens were submerged under water and subjected in a vacuum condition to effectively remove entrapped air inside the specimen. For partially saturated conditions, specimens were exposed to ambient 99% and 80% relative humidity (RH) for 24 hours or until their weight is constant to attain saturation condition of 92% and 68% respectively. The saturation degree is calculated in terms of the moisture content of the partially saturated specimens and 100% saturated specimens. When the saturation condition is constant, specimens 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. 2.1. 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.



a) 40 x 40 x 2 mm cut specimens; b) attachment of strain gauges; c) moisture adjustment (under vacuum); d) sealed specimens inside the environmental chamber

Fig. 2.1 Preparations of specimens.

2.4 FREEZE-THAW TESTS

The freeze-thaw cycle (FTC) test describe in this chapter is basically the similar FTC that will be applied in all FTC testing in the succeeding chapters.

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. 2.2 were put inside the chamber. The temperature cycle was set-up as shown in Fig. 2.3 wherein the maximum temperature is 10 °C and minimum temperature is -28 °C both maintained for 90 minutes. The temperature change to reach the maximum or minimum temperature is 0.25 °C per minute. This FTC cycle 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.

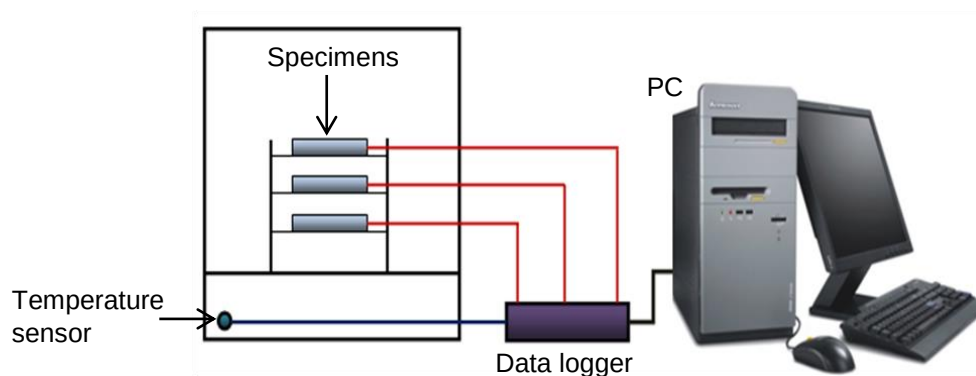


Fig. 2.2 Experimental Set-up

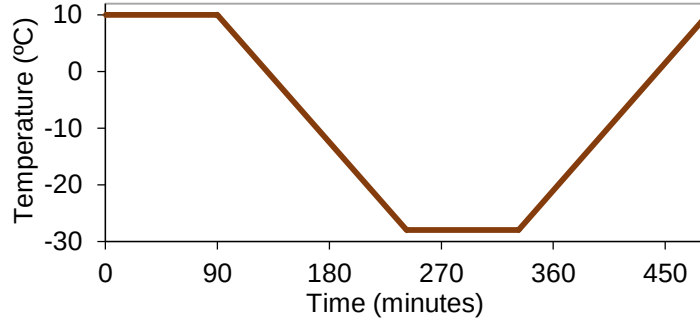


Fig. 2.3 Temperature cycle (1 FTC)

2.5 RESULTS AND DISCUSSION

2.5.1 Dry Specimen's Strain

Fig. 2.4 shows strains for all dry specimens, it can be observed that during the whole FTC the behavior of the strains remains almost constant as the number of cycle increases though there is a very slight increase in the residual strain at the end of each cycle as the FTC progresses. This strain behavior is similar for all dry specimens. The uniform strain behavior during the entire FTC is due to the absence of moisture in the specimens and the deformation is caused by temperature change alone. Using the relationship of strain and temperature variation in Eq. 2.1, the CTE of the materials is calculated from strains in Fig. 2.4 and summarized in Table 2.1. The calculated CTE is an average of all the cycles.

$$\varepsilon_t = \alpha_t \times \Delta T \quad (2.1)$$

Where, ε_t is the thermal strain, α_t is the coefficient of thermal expansion of mortar and ΔT is the temperature difference between the temperature reached at a certain time and the initial temperature which is set to 10 °C. According to many researches the linear expansion of mortar or concrete is $8 - 12 \times 10^{-6}/^\circ\text{C}$ (Al-Ostaz 2007; Federal Highway Administration) which makes the calculated values of mortar CTE shown in Table 2.1 within the typical range.

There is also a slight increase in strain or residual strain at the later stages of FTC for all of the specimens. This is caused by the repeated temperature cycles which is comparable to cyclic loading. Since mortar is a multiphase material, the difference in CTE of cement paste and fine aggregate may have caused this residual strain, however in comparison with the deformation caused by moisture behavior during FTC this change in strain can be insignificant.

2.5.2 Fully Saturated Specimens Strain

Specimens are pre-conditioned to be saturated by moisture where an obvious difference in strain behavior from dry specimens is noticeable. 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. 2.5. 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. 2.4, the strain induced by moisture behavior which causes damage during FTC is then obtained.

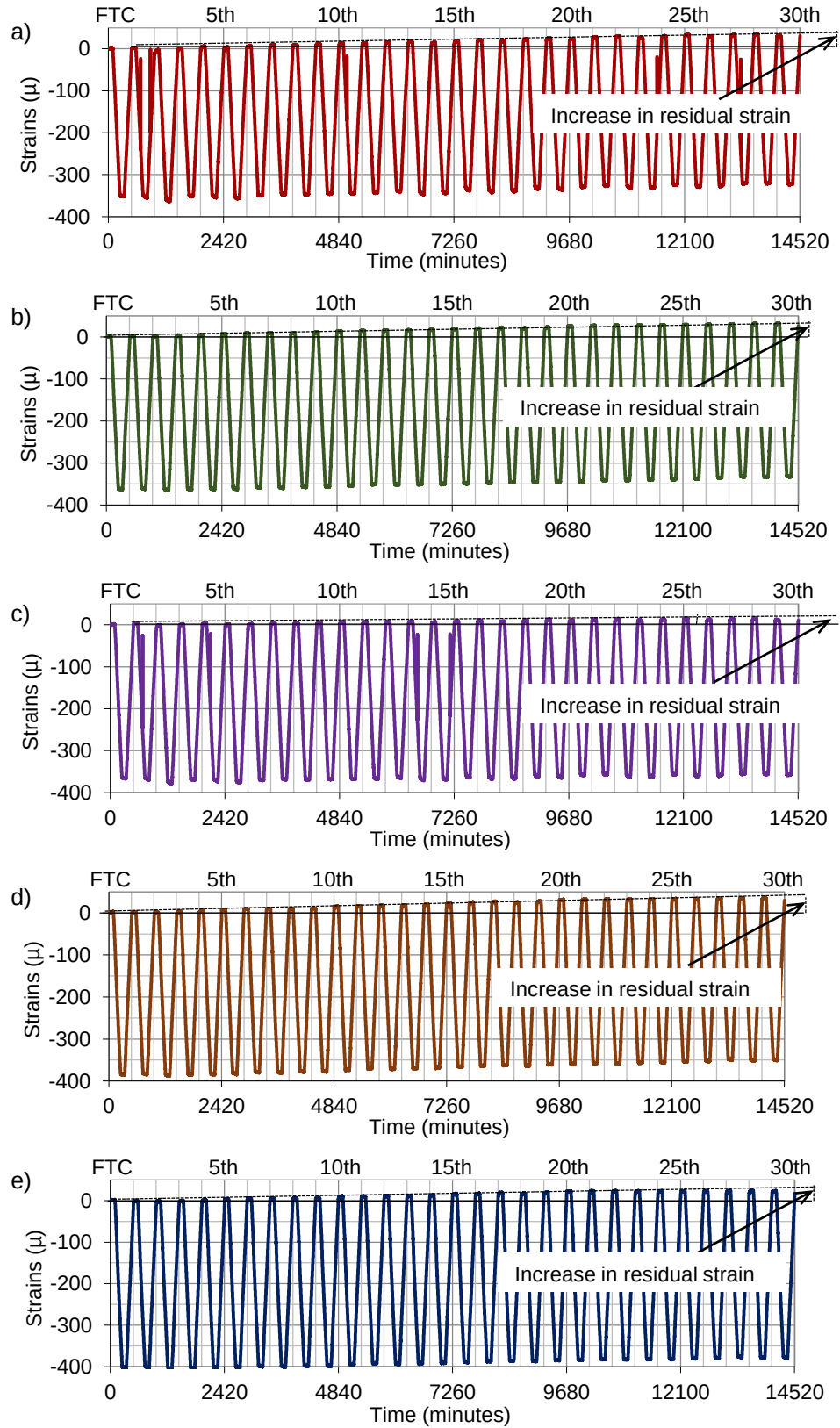


Fig. 2.4 Dry specimen's strains: (a) 70%-1090 FA, (b) 50%-1090 FA, (c) 50%-990 FA, (d) 50%-755 FA, (e) 30%-755 FA (% - water to cement ratio and FA - fine aggregate amount in kg/m^3)

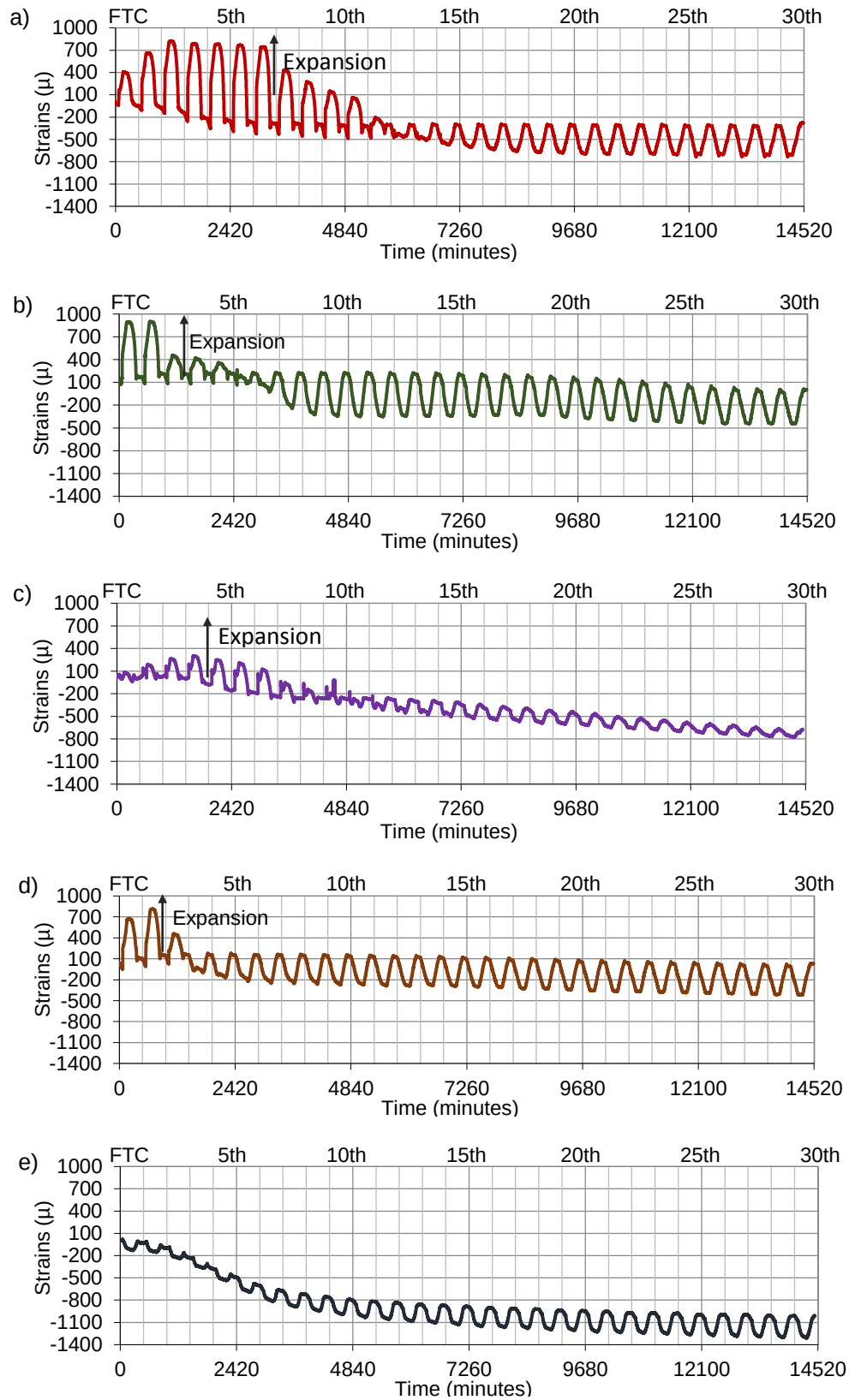
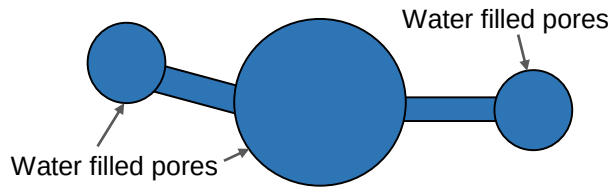


Fig. 2.5. Fully saturated specimen's strains: (a) 70%-1090 FA, (b) 50%-1090 FA, (c) 50%-990 FA, (d) 50%-755 FA, (e) 30%-755 FA

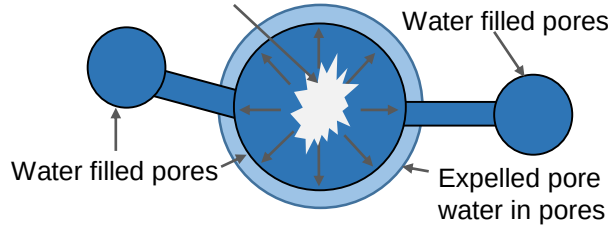
Once the thermal strains are removed as shown in Fig. 2.5, expansion is the dominant strain behavior in the initial FTC of fully (100%) saturated specimens (70%-1090 FA, 50%-1090 FA, 50%-990 FA and 50%-755 FA) except for 30%-755 FA (% is water to cement ratio and FA denotes amount of fine aggregates in kg/m³) 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 as illustrated in Fig. 2.6a. Based on the hydraulic pressure theory, a temporary hydraulic pressure is produced causing the abrupt expansion when water suddenly freezes (Gruebl 1980; Korhonen 2002; Setzer and Liebrecht 2002). 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 (Fig. 2.6b). 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. It can be further observed the positive maximum strain reached its peak around the 2nd to 4th cycle for most of the specimens except for 30%-755 FA. This shows that the limited amount of moisture has associated effect of the maximum deformation or damage that it can deliver to the frost subjected specimens. Upon reaching the maximum deformation the maximum strain slowly decreases.

As the FTC progresses during the initial FTC, pores becomes partially saturated either due to the increase in pore caused by microcracking or hydrodynamic relaxation or a combination of the two. This happens during freezing when water expansion takes place and causes temporary hydraulic pressure which leads to microcracking. Additionally, since the sealing material (*Saran*) is not completely attached to the surface of the specimens, the expanding water could be expelled out of the pores and will flow on the surface of the specimens as seen in Fig. 2.6b. Ciardullo *et al.* (2005) explains that in theory, hydrodynamic relaxation occurs by flow of pore water to the surface of the body, rather to the internal air pockets. The space between the sealing material and mortar can act as a space for the escaping water and remains there. Assuming the theory is correct, most likely then the expanding pore water flows out to available spaces in the surface which is exacerbated by the increased permeability of the specimens due to microcracking. Since the specimens are sealed, water uptake could not take place to refill and re-expand the pore system, as a result the pore structure becomes partially saturated. The partial saturation of the pore system serves as the pre-requisite for the contraction of the system. As the partial saturation of pores continues the expansive strains becomes less efficient until such point where the amount of moisture may no longer be effective to cause expansion. In a partially saturated pore the expanding water is relieved by the available spaces as illustrated in Fig. 2.6c. When ice forms in a partially saturated pore, the ice causes thermodynamic imbalance in the pore solution where the contraction behavior initiates. Kaufman (2002) explains that the contraction is due to chemical potential difference. Ice that is formed in larger pores has lower chemical potential than unfrozen liquid in smaller pores (freezing of pore water is dependent on size, the smaller the size the lower will be the freezing point (Korhonen 2002). To bring the pore solution into thermodynamic balance unfrozen pore solution in smaller pores are drawn toward the existing ice in larger pore. While Setzer (2002, 2001) explains the contraction phenomena by the micro-ice lens model. When ice is formed in larger pores, pressure differences between the ice and unfrozen water in small pore arises wherein the unfrozen water is under increasing negative pressure with decreasing temperature. Thus, the unfrozen water is squeezed out of the gel matrix and is trapped in existing ice crystals in capillaries which is termed as frost shrinkage and comparable to drying shrinkage (Setzer 2002, 2001). These are just some of the recognizable mechanism that explains the contraction phenomena. Either due to chemical potential differences (Kaufman 2002) or pressure differences (Setzer 2002, 2001) between the unfrozen water and ice in larger pores both are in



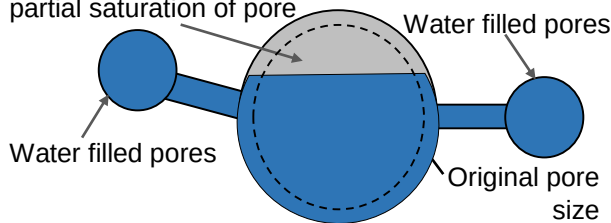
a. For a fully saturated specimens all pores are assumed to be fully saturated. At this stage prior to freezing no expansion of water takes place.

Ice formation which causes expansion and tensile stresses



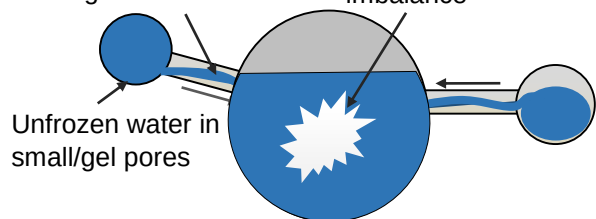
b. During freezing, water in larger pores freezes first. This causes the expansion of water by 9% which can exert tensile stresses on the surrounding matrix. If the stresses cannot be relieved this leads to microcracking. As a consequence of the expansion of water during freezing, **some** will be expelled out of the pores. According to the hydrodynamic relaxation, the expelled water may flow to the surface of specimen rather than empty air pockets.

Increased space, resulting in partial saturation of pore



c. When completely thawed at 10 °C, the expelled pore water from pores cannot refill the original water filled pore and as a result the pore becomes partially saturated. In addition since specimens are sealed, water supply is not available to refill the pores. Microcracking may add available spaces increasing the permeability.

Removal of unfrozen water moving toward ice front causing the contraction



d. At this stage during freezing when ice forms, the stresses associated with water expansion are relieved by the available spaces therefore the expansion continues to decrease or becomes less efficient as increasing volume of pores becomes partially saturated until the deformational behavior results in the more evident contraction. The contraction of the system arises due to ice formation in partially filled pores which causes thermodynamic imbalance of the pore system. Chemical potential differences (Kaufman 2002) or pressure differences (Setzer 2002, 2001) arises between the unfrozen water and ice in larger pores resulting in negative pressures driving out the water in smaller ($< 0.05\mu\text{m}$) or gel pores (Mehta P.K. and Monteiro P.J.M 2006) as seen in the above figure. Water in smaller capillaries or gel pores exerts hydrostatic tension, and removal of pore water collapses the pore walls. This results in the permanent contraction at the end of the FTC since no available outside water is available to re-saturate (or refill) the pores.

When redistribution of pore water stabilizes, the deformation of the specimens becomes reproducible.

Fig. 2.6 Illustration of the moisture behavior during FTC

agreement that the resulting negative pressures drives out the water in smaller ($< 0.05\mu\text{m}$) or gel pores (Mehta P.K. and Monteiro P.J.M 2006) as shown in Fig. 2.6d which causes the contraction of the system. Water in smaller capillaries or gel pores exerts hydrostatic tension, and the removal of pore water collapses the pore walls this results in the contraction of the specimens. As increased pore water are drawn out from smaller pores, increased in contraction is noticeable as the FTC progresses. Further, as can be observed at the end of each of FTC the contraction cannot be recovered because of the absence of available water supply from outside to re-fill and re-expand the drawn water from smaller pores. When redistribution of pore water stabilizes from smaller pores, the deformation of the specimens becomes reproducible as observed at the later stages of the FTC.

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; Goto and Roy 1981). 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 as is represented in Figs. 2.6c and 2.6d.

2.5.3 Partially Saturated Specimens Strain

Observations for partially saturated specimens subjected to RH 99% from Fig. 2.7 indicate similar behavior to fully saturated specimens. The continued contraction is also observed for the 30%-755 FA. However, in comparison with the strains for fully saturated specimens all of the strains' magnitudes are lesser for both expansion at the initial stage of the FTC and the resulting contraction at the later stages of the FTC. This is because specimens in this group specimens contain less moisture content as compared with those of the 100% saturated specimens.

For partially saturated specimens subjected to RH 80% shown in Fig. 2.8 during the entire FTC a slightly reproducible contraction is observed at the lowest temperature for all specimens. This differs from 100% saturated specimens and partially saturated specimens conditioned at RH 99%. Moisture present in pores is not be enough to cause significant expansion in the system. Moreover, lesser pore water may be drawn out from smaller pores which causes the slight increase in the contraction of the system as the FTC progresses. The contraction is obviously much lesser in comparison with fully saturated case and RH 99% conditioned specimens. Thus, based on the observed strain behavior of specimens it is evident that the deformation of specimens is dependent on its moisture content regardless of number of FTC, the higher the amount of moisture the larger is the deformation in either expansion or contraction while low moisture content results in lower deformation otherwise. The relationship of moisture and deformation is illustrated in Table 2.2 where the amount of moisture for each specimen corresponds to the obtained deformation during expansion and contraction.

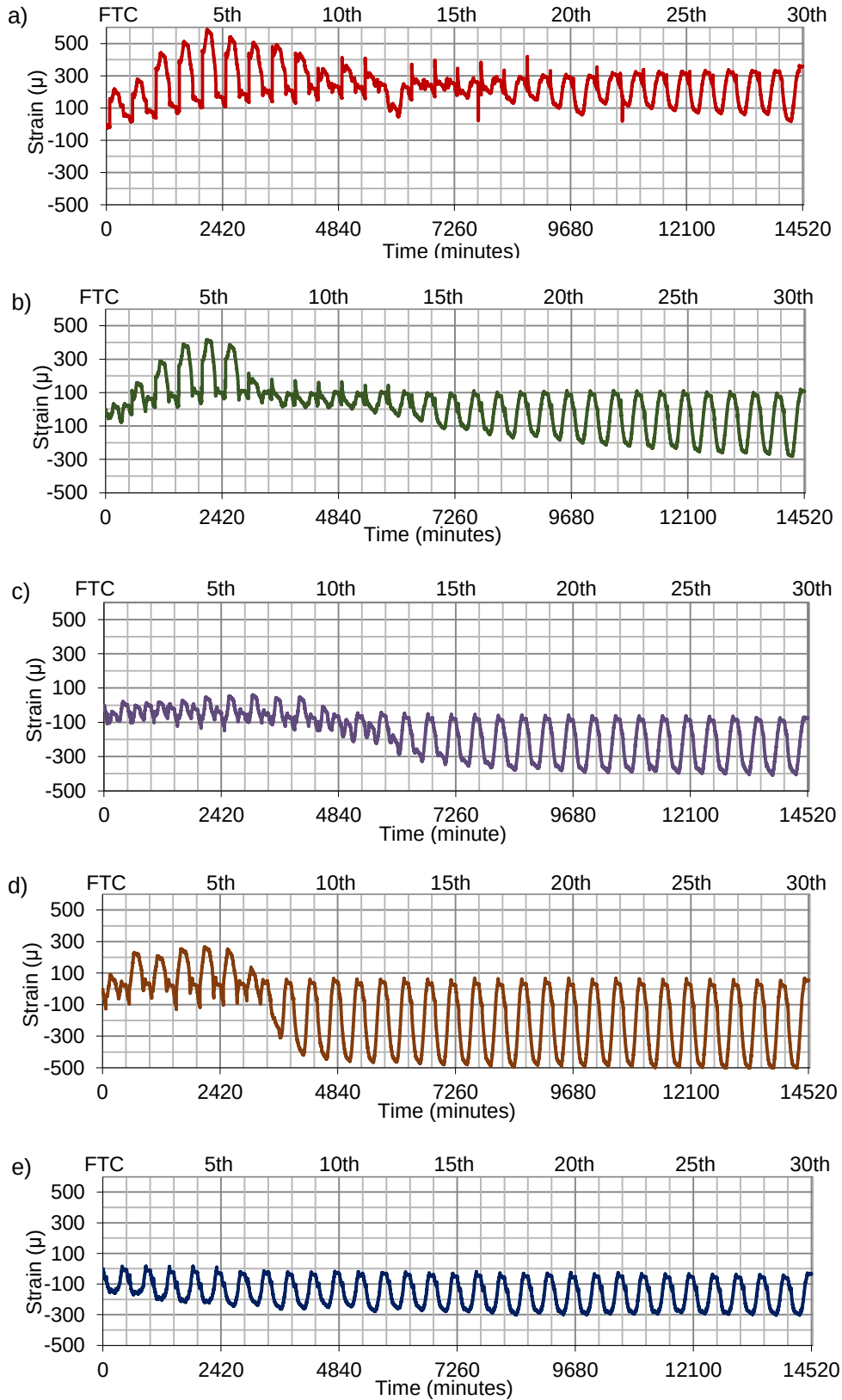


Fig. 2.7. Partially saturated specimen's strains (RH 99%): (a) 70%-1090 FA, (b) 50%-1090 FA, (c) 50%-990 FA, (d) 50%-755 FA, (e) 30%-755 FA

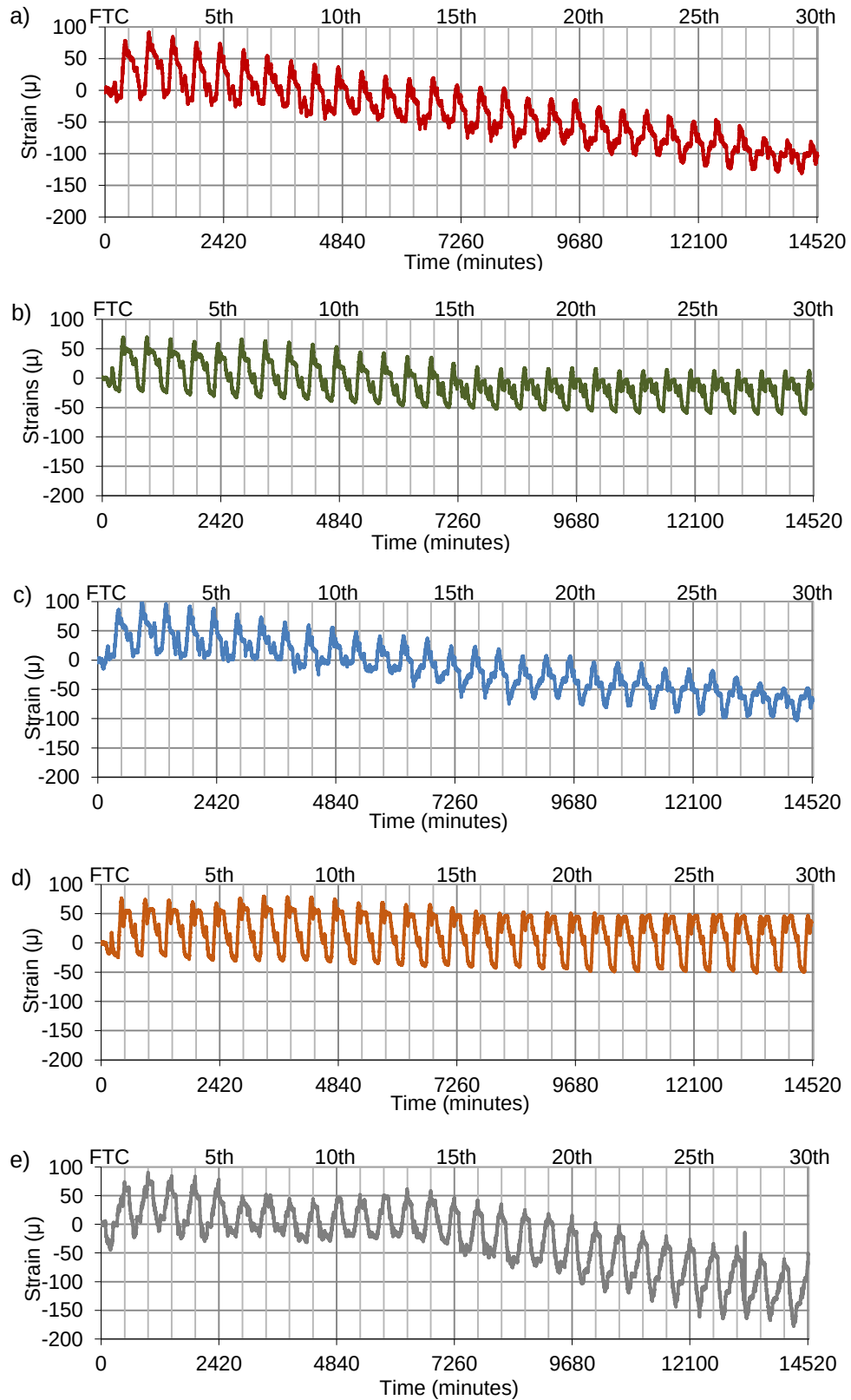


Fig. 2.8. Partially saturated specimen's strains (RH 99%): (a) 70%-1090 FA, (b) 50%-1090 FA, (c) 50%-990 FA, (d) 50%-755 FA, (e) 30%-755 FA

Table 2.2 Maximum expansion/contraction during FTC associated with the amount of moisture

Mixture		Maximum strain (μ)		
		Fully saturated	Part. Saturated RH 99%	Part. Saturated RH 80%
70%-1090 FA	Expansion	805.74	586.06	84.28
	Contraction	-696.22	-26.16	-163.3
50%-1090 FA	Expansion	893.98	417.91	69.83
	Contraction	-411.45	-279.73	-61.33
50%-990 FA	Expansion	305.964	142.06	118.82
	Contraction	-827.55	-400.44	-51.08
50%-755 FA	Expansion	801.63	266.66	79.67
	Contraction	-380.87	-509.02	-53.00
30%-755 FA	Expansion	20.56	18.25	71.33
	Contraction	-1297.06	-304.44	-133.79

2.5.4 Difference in behavior between a constantly saturated sample and sealed sample during FTC

To further understand the contraction behavior of the specimens during FTC. The results of the present study were compared with a previous study. The previous study uses concrete as specimens: cylinders of 100 mm diameter and height of 200 mm and prisms with the size of 100 x 100 x 400 mm. A bigger prism was also used, however this will not be compared with the present results. Specimens were subjected to a much higher number of FTC, for cylinders it was 300 cycles and for prisms it was 200 cycles. Specimens were not pre-saturated prior to testing. Frost cycles test was started when the age of the specimens of 50 days in a climate chamber, in which the maximum input temperature was 20 °C and the minimum is 20 °C. During thawing when the temperature reaches 19 °C, water is sprayed for 15 minutes to keep the specimens saturated. Details of the experiment procedures can be found from Hassan *et al.* (2003).

The average strains obtained from cylinders and prisms is shown in Fig. 2.9 and 2.10. Cycle number is notated as FCx. The strain-temperature relationship between cylinders and prisms was almost the same. The strain induced shown in Fig. 2.9 and 2.10 includes the thermal strains. In Fig. 2.11 the thermal strains were removed. For comparison purposes strains for 70%-1090 FA and 30%-755 FA saturated specimens were presented. Figs. 2.12 and 2.13 shows strains including the thermal strains and Figs. 2.14 and 2.15 show the strains excluding the thermal strain to illustrate the moisture behavior during FTC.

For cylinders in Fig. 2.11 (Fig. 2.9 and 2.10 as well) it can be observed in the first FTC that contraction is observed, this is because of the negative pressure that is produced during freezing due to the limited amount of moisture in pores. The amount of moisture is not enough to cause expansion. On the one hand, for the present study for 70%-1090FA in Fig. 2.14 and similarly for the rest of the specimens, expansion is observed primarily because the specimens are saturated at the beginning of the cycle. The hydraulic pressure is the dominant strain behavior in this case. This is also observed when the specimen's strain includes thermal effect in Fig. 2.12 where the strain begins to increase when temperature reaches -6 °C indicating the effect of high hydraulic pressure. While for the previous study, this increase in strain is not observed. 30%-755FA specimens is an exception which showed contraction at the first cycle and all succeeding cycles in Fig. 2.13 and 2.15 with and without the thermal strain. In the previous section, it was mentioned that the specimens is probably partially saturated even

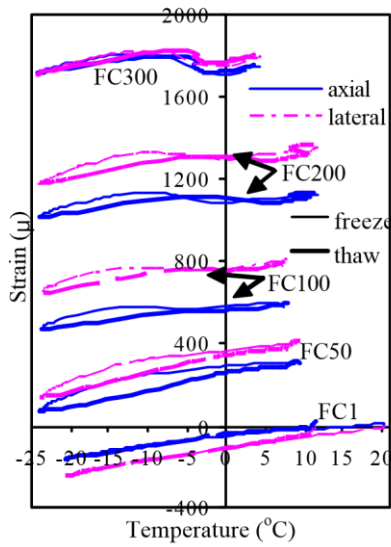


Fig. 2.9 Strains induced in cylinder specimens

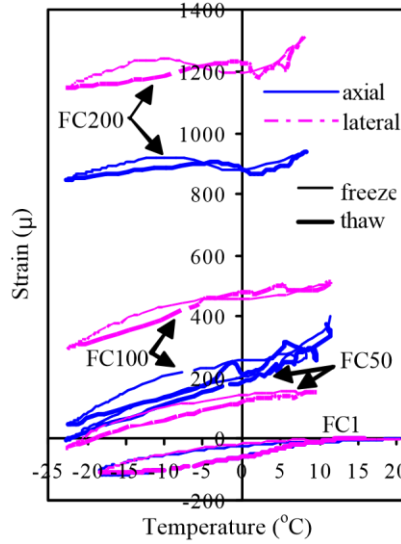


Fig. 2.10 Strains induced in prism specimens

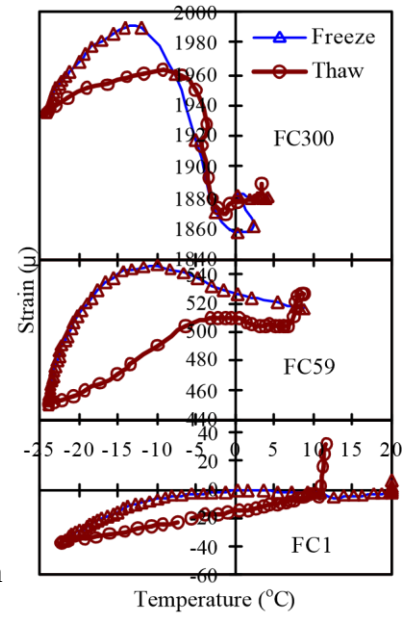


Fig. 2.11 Strains induced in cylinder specimens (thermal strains excluded)

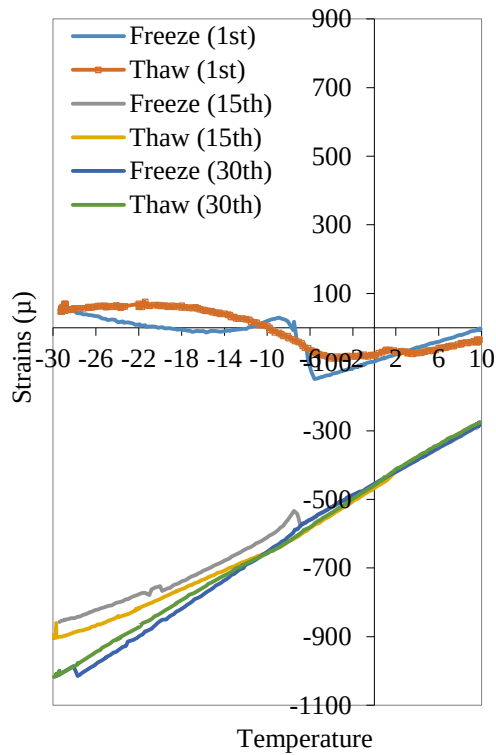


Fig. 2.12 Strains for 70%-1090 FA specimens with thermal strains (current study)

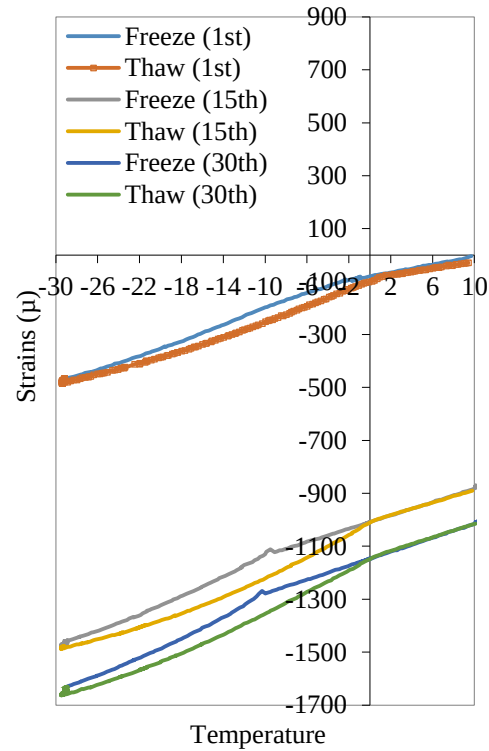


Fig. 2.13 Strains for 30%-755 FA specimens with thermal strains (current study)

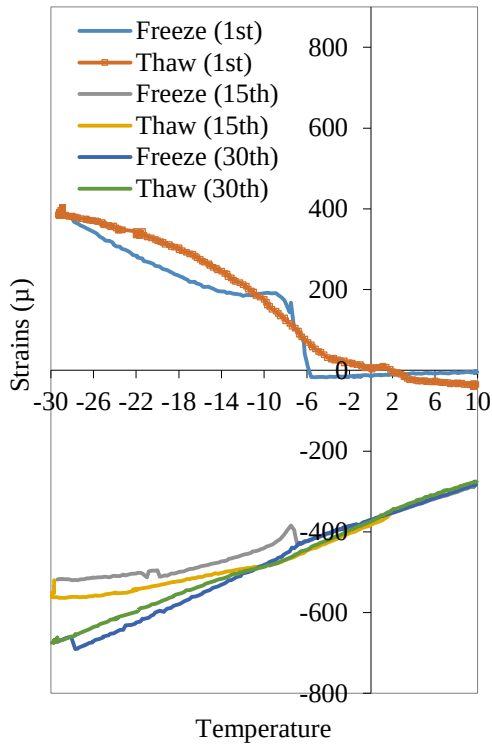


Fig. 2.14 Strains for 70%-1090 FA specimens without thermal strains (current study)

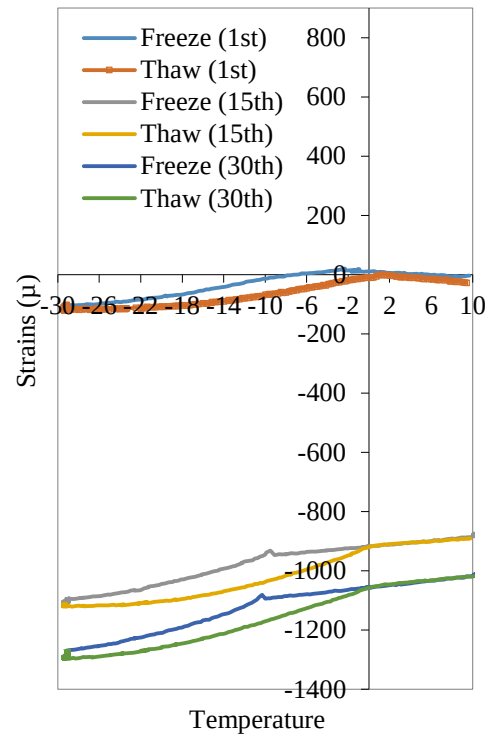


Fig. 2.15 Strains for 30%-755 FA specimens without thermal strains (current study)

though it was preconditioned to be fully saturated because of its presence of very fine pores.

When the FTC cycle progresses in Fig. 2.11, the degree of pore saturation increases for the cylinder and prism specimens since water supply is available from the outside (sprayed during thawing). The expansive stresses cause micro cracking in the specimens. In this sense, since available moisture is present from outside, excess pore water that is expelled out of pores during expansion are refilled and the moisture content increases due to increased pore volume in pores as a result of the microcracking. Thus the increased pore water volume present in pores results in a higher expansion of concrete as the cycle increases which can be seen in Fig. 2.11 in the 59th cycle and finally in the 300th cycle. Contrary in the present case, since the specimens (except for 30%-755 FA) are preconditioned to be fully saturated when the FTC initiates large expansion are observed initially. However, as the FTC progresses contraction is observed since the pore volume of the specimens becomes partially saturated which again contrary to the previous study where increased in pore volume results in higher expansion as the FTC progresses.

What can be observed in this comparison is that there a striking contrast between the two strain behavior of the specimens. This can be obviously attributed to the pore condition of the specimens, the pore saturation in the previous case increases while for the present case the pore saturation decreases.

2.6 CONCLUSIONS

The deformational behavior of mortar was presented where in specimens were subjected under FTC under varying temperature and constant moisture conditions. The experimental investigation suggests that the behavior of a sample under FTC is primarily dependent on the amount of its moisture. The larger the amount of moisture, the larger will be the resulting deformation in both expansion and contraction behavior.

The mechanism of the deformation of mortar is also introduced diverse to the presented mechanism in literatures as in the case of Hasan *et al.* (2003) where expansion of water during FTC is attributed as the primary cause of the deformation during FTC. This expansion is understood and pointed to be caused by the hydraulic pressure. In the present study, the contraction phenomena is observed prior to the expansion. When the pore system becomes partially saturated, the ice formation causes the thermodynamic imbalance (where either chemical potential or pressure difference between ice in large pores and water in smaller pores arises) this causes negative pressures which draw out water in smaller pores which causes the contraction.

Moreover, in the comparison between the two experimental investigations there is an obvious difference in strain behavior. For specimens with continuous moisture supply as the FTC progresses, increasing expansive strains are observed however for specimens with limited moisture content even though specimens are pre-saturated, the strain at the later stages of the FTC shows (increasing) contraction otherwise.

In actual conditions, a constant moisture supply may not always be available to concrete under FTC except for structures near water splashes such sea docks etc. This is why the consideration of the contraction presented in this study is very significant in understanding the damage mechanism of concrete under FTC. Therefore it is suggested that the contraction during freezing should also be considered in the modeling and simulation of frost damage in concrete.

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Chapter 3

THE CHANGE IN THE COEFFICIENT OF THERMAL EXPANSION AND ELASTIC MODULUS OF MORTAR DURING FREEZING AND THAWING CYCLES

3.1 BACKGROUND

It is common knowledge that when materials are heated or cooled, they typically expand or contract depending on the change in temperature. For homogeneous materials, the common knowledge is that the coefficient of thermal expansion (CTE) is not affected by microstructural change. However this could be different for heterogeneous materials such as concrete.

In civil engineering, the CTE is a significant property of concrete structures such as pavements, bridges and other continuous structures (Uygunog̃lu and Topcu 2009). The CTE is determined or estimated for the structures' design, proper management and maintenance (Ndon and Bergeson 1995; Mallela *et al.* 2005; Al-Ostaz 2007). However, typically the CTE used in these designs is based on undamaged concrete without considering the possibility that the CTE might change owing to inevitable deterioration from various factors (Sicat *et al.* 2012). Such is the case in cold regions, in which concrete's most persistent problem is deterioration caused by freezing and thawing cycles (FTC). Frost damage deteriorates the structural performance of concrete structures such as safety and serviceability due to reduction in strength and stiffness (Hasan *et al.* 2003). Numerous studies regarding the mechanism of freezing and thawing action in concrete have been presented, yet no unified mechanism has been widely accepted (Chatterji 1999). In a previous study (Sicat and Ueda 2011), the mechanism of FTC was presented based on the observed deformation of mortar specimens obtained from experimental findings. From that study, the total strain (ε) during FTC is proposed to be composed of three strain components, as shown in the equation below.

$$\varepsilon = \varepsilon_i + \varepsilon_s + \varepsilon_t \quad (3.1)$$

The components of the total strain ε are the freezing expansion strain ε_i , which is a product of the volume expansion of water when it turns into ice, the shrinkage strain ε_s , which is a product of transport of unfrozen water to empty or partially filled pores caused by thermodynamic imbalance when ice forms, and the thermal strain ε_t , which is the response of the material itself to temperature change. These strains are observed to happen simultaneously, and either expansion or contraction behavior may be dominant depending on the moisture content of the specimen.

The deterioration of concrete due to FTC has adverse effects, which could include the CTE. Currently, it may not be realized that such deterioration (or deterioration from other factors) could unfavorably alter the CTE of concrete and its durability, safety and serviceability (Sicat *et al.* 2012). The thermal strain ε_t as a product of temperature variation and the CTE of the material will significantly be affected when the CTE changes, and when that happens, the total strain during FTC, which includes the thermal strain ε_t as one of the components, will also be greatly influenced. The mechanism behind the change in CTE of concrete/mortar may not be clear as of the moment, however since deterioration due to FTC causes microcracks and eventually microstructural change, this could be the primary factor for such change. In the case of homogeneous materials, these microstructural change are said not to affect the CTE. Yet, concrete is a heterogeneous and multiphase material and its response to microstructural change

is different, e.g. reduction in elastic modulus is due to microcracking. On these accounts mentioned, this study aims to show that the damage brought by FTC significantly alters the CTE of concrete.

3.2 EXPERIMENTAL PROGRAM

The aim of the experiment is the collection of data in order to present the drastic changes of the CTE and elastic modulus of mortar during FTC. Based on the findings the significant development of microcracks in the material may initiate these changes.

3.2.1 Specimens

The preparation the specimens used in the experimental study is similar as previously discussed in Chapter 2 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) (see Fig. 3.1). Similarly, sealing of specimens were also done prior to the final sealing using mastic tape for all specimens under different testing.

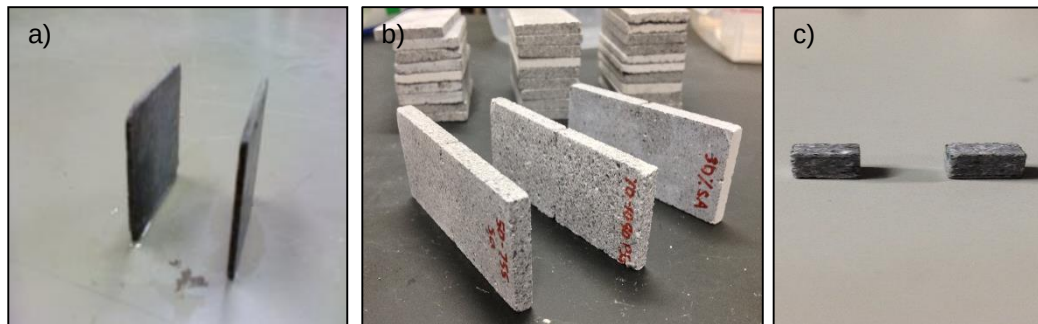


Fig. 3.1 Specimens used for experiment testing: (a) 40x40x2 mm for FTC tests, (b) 70x30x5 mm for elastic modulus tests, (c) 10x10x3 mm for X-ray CT observations

3.2.2 Freeze-thaw Tests

The FTC tests involve in this chapter was done similarly as discussed in Chapter 2. The difference for the FTC tests is that for of fully saturated specimens were respectively subjected to 1, 3, 5 and 30 FTC. The results of dry specimens and fully saturated specimens obtained in Chapter 2 will be utilized here.

After strains for fully saturated specimens were obtained, the said specimens were dried again in an oven at 105°C for 24 hours in order to remove the influence of moisture then sealed once again and finally subjected under 5 FTC. Thermal strains were then obtained and CTE is calculated and compared with specimens not damaged by FTC.

3.2.3 Elastic Modulus Test

Widely used by most experimental studies, the elastic modulus is measured as a reference for the damage obtained during FTC (Fagerlund 2002; Hasan *et al.* 2003). The decrease in elastic modulus is also used in this study as reference for the damage accumulated during testing. The specimens used for the elastic modulus test are the meso-scale size of 70 mm x 30 mm x 5 mm. The specimens were pre-conditioned in the same way as specimens used for strain measurement test. After pre-conditioning the specimens, which involved drying, saturation and sealing as explained in Section 2.1, the specimens were subjected to 1, 3, 5 and 30 FTC. Then, a three point bending test was performed using a universal testing machine designed to test small scale size specimen such as shown in Fig. 3.2 to determine the load-displacement curve of the specimens. The elastic modulus of each specimen was then calculated.

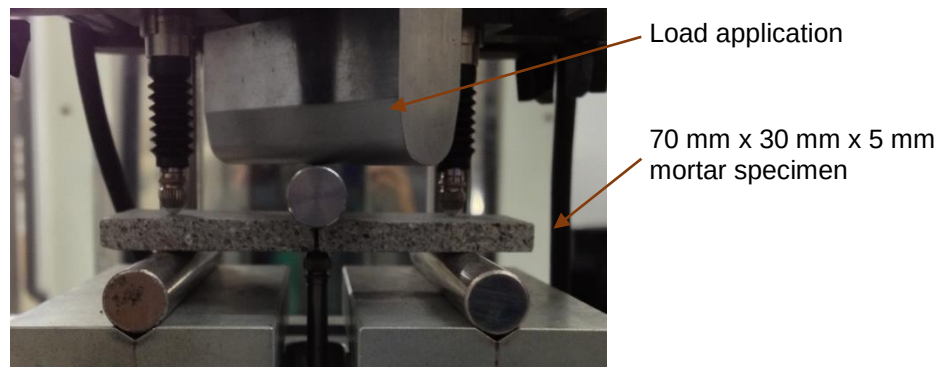


Fig. 3.2 Three point bending test set-up of meso-scale size specimen

3.2.4 X-ray Computed Tomography (CT) Test

X-ray computed tomography is a non-invasive and non-destructive imaging technique to investigate the microstructure of cement based materials (Promentilla and Sugiyama 2010). Unlike other methods, X-ray CT eliminates the tedious sectioning of specimens needed in image observation with the use of microscope or similar techniques (Promentilla and Sugiyama 2010). Promentilla and Sugiyama (2010) discuss the detailed methodology of the use of and the potential of X-ray CT to investigate the microstructure of mortars exposed to FTC. Similar methods and techniques were applied in this study.

The X-ray CT scanner (Fig. 3.3) is composed of a micro-focus X-ray source, a specimen manipulator, an image intensifier detector coupled to a CCD camera, and an image processing unit. Five μm is the smallest scale that the scanner can detect. The specimens used for scanning were 10 mm x 10 mm x 3mm specimens (see Fig. 3.3), a size that was chosen to fit in the rotation table and to obtain the highest possible resolution image. Scanning was done after every cycle (the experimental conditions were the same as those for specimens used for the strain measurement tests, except for the attachment of a strain gauge) during the first five FTC and after every five cycles starting from the 10th until the 30th cycle.

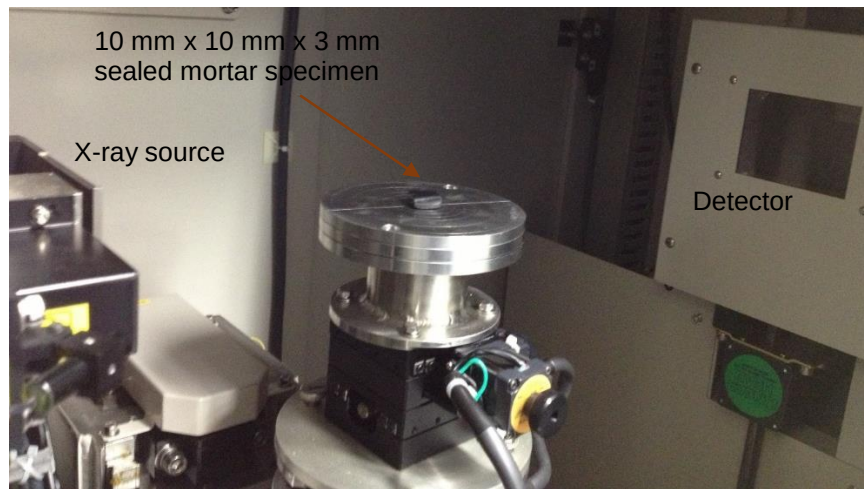


Fig. 3.3 Experimental set-up of the micro focus X-ray CT

3.3 CLARRIFICATION OF THE OVEN DRYING

Oven drying was performed in order to remove the moisture content of the specimens for the purpose of obtaining their respective CTE. It is suggested that oven drying has an effect on the microstructure of cement based materials but may or may not have an effect on the CTE of such materials. In this regard, prior to the FTC tests, pretesting to clarify the effect of oven drying on CTE was first performed. The testing involved two sets of conditions, oven dried samples (subjected to 105 °C for 24 hours), and samples without drying. The specimens used for both tests used 50% W/C ratio with 990 kg/m³ of fine aggregate mixture and were cut into 40 mm x 40 mm x 2 mm size. Since the CTE of cement based materials is known to be affected by moisture content (Sellevold and Bjøntegaard 2006; Al-Ostaz 2007), the specimens were fully saturated by a vacuum process similar to that described in Section 2.1 to achieve a similar effect of moisture in both cases. The specimens were then sealed and underwent three times the heat-cool temperature cycle shown in Fig. 3.4. A heat-cool cycle was chosen instead of FTC to cancel the effect of ice formation, which causes frost damage.

The average strain results of the three specimens are shown in Fig. 3.5 for the three heat-cool cycles (HCC). Theoretically, complex behavior of the specimens is plausible owing to their being greatly influenced by the presence of moisture. If the samples are dry, then the results will be stable and reproducible, as have been discussed on Section 2.5.1 in Chapter 2 and succeeding section on dried specimens subjected to FTC.

At the beginning of the HCC, there is an observed slight expansion before the temperature rises, which suggests that the specimens are still equilibrating. However, this does not affect the purpose of the test, which is to clarify the effect of oven drying on the CTE of specimens, which is shown in the similar strain behavior of the samples at least until the 2nd cycle. It can be observed that both samples contract more at the end of each cycle and the peak strain at the highest temperature decreases as the HCC progresses. The behavior of the specimens could be influenced by the dominant redistribution of pore water from gel pores to capillary pores, which arises due to the higher chemical potential of gel pore water than capillary water during the increase of temperature (Sellevold, E.J. and Bjøntegaard 2006). Expulsion of the gel water collapses the pore walls due to hydrostatic tension, which leads to shrinkage of the matrix (Mehta P.K. and Monteiro P.J.M. 2006, Bordallo *et al.* 2006). During heating, pore water also expand more than the mortar matrix (Ai *et al.* 2001; Ciardullo *et al.* 2005), however this behavior is less efficient and is dominated by the contraction since the expanding pore water

simply flows out to the surface or available pore spaces due to the high permeability of the mortar and slow rate of heating (Ciardullo *et al.* 2005). This relieves the build-up of pore pressure. Take note also of the fact that samples are sealed and water supply is not available. This means that the displaced pore water will not be resupplied, and thus re-expansion of the matrix is not observed and the samples remains contracted at the end of the HCC. As gel-capillary water redistribution increases, the contraction becomes more prevalent in the succeeding cycles. Eventually when both positive and negative pressures equilibrate, the strain plots become reproducible.

During the third HCC however, the oven-dried sample contracted more than the undried sample. It is possible that oven-drying as reported (Galle 2001) has some effect on the microstructure of concrete, however not to the extent that the CTE is affected. This might have increased the pore water that could be redistributed and could have caused the difference in the third cycle. However, the exact reason is not understood as of this writing and further studies should be conducted to fully understand this phenomena.

Since this study is not focused on the behavior of mortar during HCC and involves data limitations, an in-depth discussion and explanation are outside the scope of this paper. The purpose of this experiment is to clarify the effect of oven-drying on mortar's CTE prior to FTC. Based on the results presented, we can point out the striking similarity of both the oven-dried and undried samples' strain behavior at least up until the 2nd cycle. Therefore, it is clear enough to say that oven-drying, though it causes slight microstructural change on the body, does not have much effect on the deformation of mortar and thus on its CTE (as a product of the strain behavior).

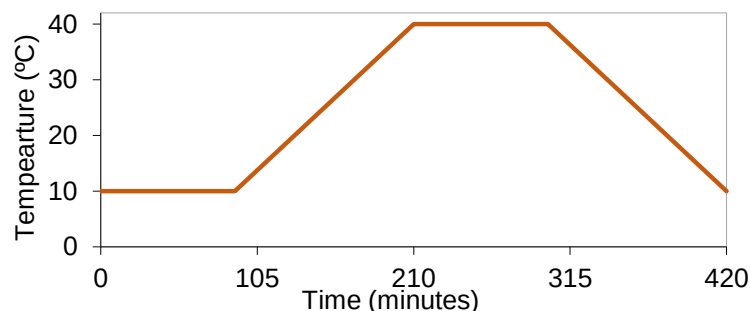


Fig. 3.4 One heat-cool cycle

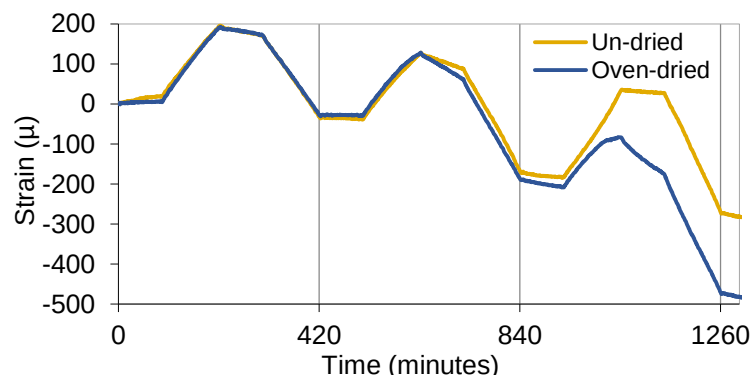


Fig. 3.5 Strain behaviors of oven-dried and undried saturated specimens

3.4 RESULTS AND DISCUSSIONS

3.4.1 The CTE of Dry Mortar

The thermal strains and the calculated CTE of dry mortar has been presented in Table 2.1 of Section 2.5.1 in Chapter 2. The calculated CTE of dry mortar are within the commonly obtained CTE of $8 - 12 \times 10^{-6}/^{\circ}\text{C}$. Further, it can also be observed that slight differences in thermal strain can be observed from each specimen. 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 (Mallela *et al.* 2005; Al-Ostaz 2007). The CTE of aggregate depend on a number of factors, particularly its nature, location and mineralogical composition. It is reported (Mukhopadhyay *et. al* 2007) that the CTE for different types of aggregates are: siliceous aggregates such as chert, quartzite and sandstone is between 2.5 and $3.6 \times 10^{-6}/^{\circ}\text{C}$, for pure limestone, basalt, granite, and gneiss it may vary between 0.7 and $2.5 \times 10^{-6}/^{\circ}\text{C}$ and single-mineral crystals for example, feldspar has values of 5.4 , 0.3 , and $0.6 \times 10^{-6}/^{\circ}\text{C}$ along three different axes. In comparison with hardened cement paste, the CTE is given as 11 to $20 \times 10^{-6}/^{\circ}\text{C}$ by Uygunog̃lu and Topcu (2003) and between 14 to $26 \times 10^{-6}/^{\circ}\text{C}$ by Sabrii and Illston (1982) depending on the moisture content. With the wide availability of reports on the higher CTE of hardened cement paste than any type of aggregates, thus (fine) aggregates have much lower CTE in comparison with hardened cement paste. The same is applicable then in the current experiment. Therefore, the larger the amount of fine aggregates (referring to Table 2.1 in Chapter 2) the lower the CTE as in the case for the thermal strains of specimen having 70% W/C and while the amount of hardened cement increases (and amount of fine aggregates decreases) the CTE will slightly increase as with specimen having 30% W/C.

3.4.2 Specimen's Strain at Different FTC Number

Figs. 3.6a show strain behavior obtained from saturated specimens at different cycles. All specimens are pre-saturated in the same condition. Take note that different batch of specimens is used for each specific cycle conditions of 1, 3, 5, and 30 FTC. After being subjected to FTC, the change in CTE will be determined for each cycle conditions. For instance, samples used for 1 FTC will not be used for 3 FTC, 5 FTC, and 30 FTC. The same applies for the other conditions.

To observe the effect of moisture behavior during FTC the thermal strains is removed for all specimens and results are shown in Fig. 3.6b. Expansion is observed from almost all specimens and at different FTC in Figs. 3.6b. In the first few FTC, as observed from 1, 3, and 5 FTC and even at the initial stages of 30 FTC, expansion is the dominant strain behavior of the specimens for 70%-1090 FA, 50%-1090 FA, 50%-990 FA and 50%-755 FA. As previously discussed in detail in Section 2.5.2 in Chapter 2, these expansions are product of volume expansion of water when it turns into ice and the contraction that is observed in the later stages of the FTC specifically for 30 FTC is due to the thermodynamic imbalance when ice forms initiating water in smaller pores to be drawn out. Furthermore it can be observed that regardless of the FTC particularly for 3, 5 and 30 FTC, the positive maximum strain is reached around the second to fourth FTC which is again due to the limited amount of moisture present in specimens.

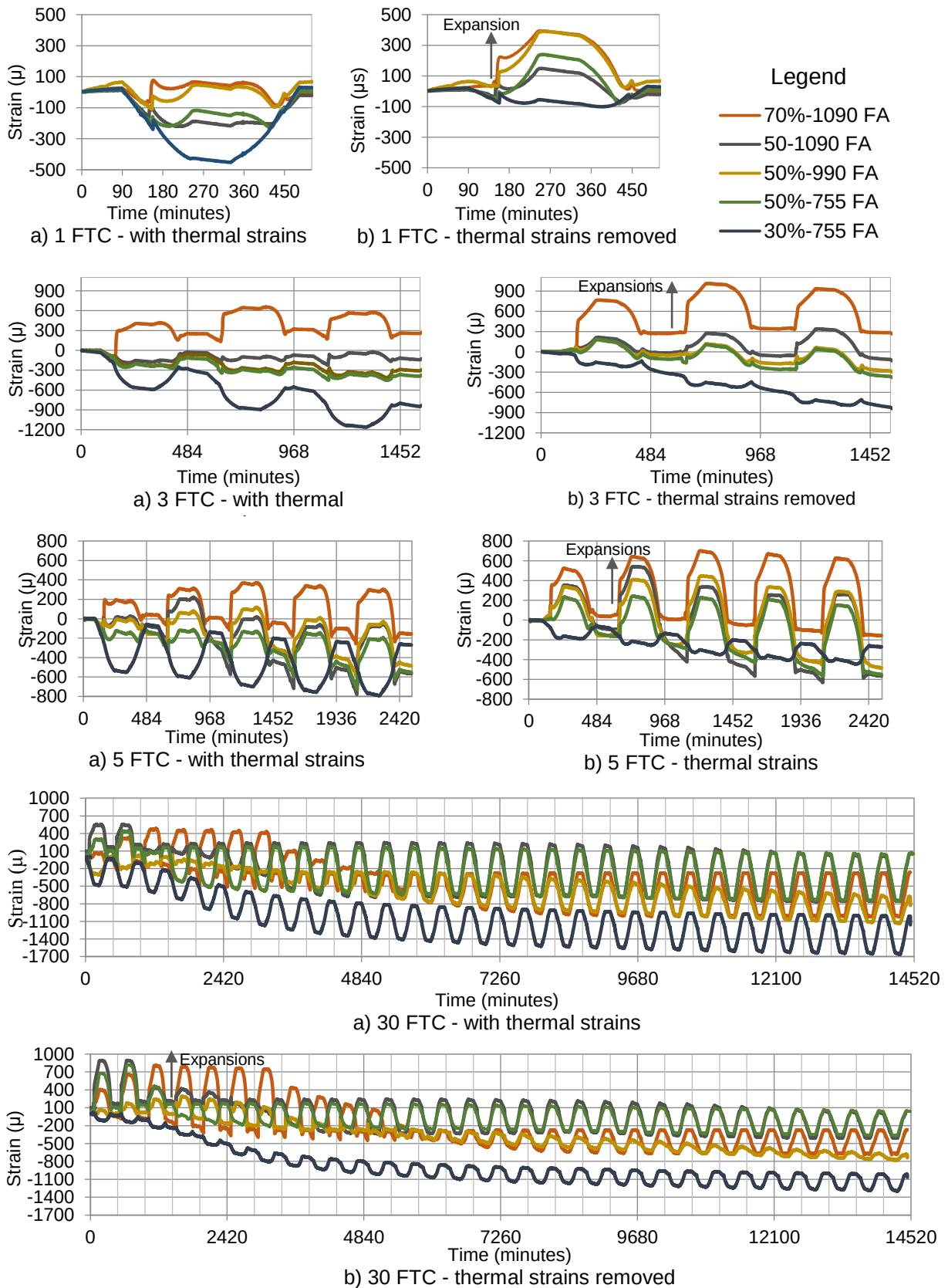


Fig. 3.6 Strain behavior of specimens a) with thermal strains b) without thermal strains

3.4.3 Elastic Modulus of FTC Damaged Mortar

The elastic modulus (E) of specimens subjected to FTC were obtained using Eq. 3.2 used for specimens tested in a 3-point bending test.

$$E = \frac{F}{\delta} \frac{L^3}{4bd^3} \quad (3.2)$$

where F is the force applied, L is the length of the specimen, b is the width, d is the height and δ is the total vertical displacement. The elastic modulus of the material was obtained by using the slope of the load-displacement (F - δ) curve until the point where it is linear. The summary of the evolution of the elastic modulus of specimens after FTC is shown in Fig. 3.7. From the figure, 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. With the limitation in moisture content, there were no further decreases in the elastic modulus in the succeeding cycles (3rd and 5th). This implies that frost damage is associated with the amount of moisture inside the specimen. The internal stresses developed during freezing are dependent on the amount of moisture. 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. An increase in FTC could also affect the strength of the specimens. The dependency of frost damage on the amount of moisture and its increase due to additional FTC were also presented by Fagerlund (2002).

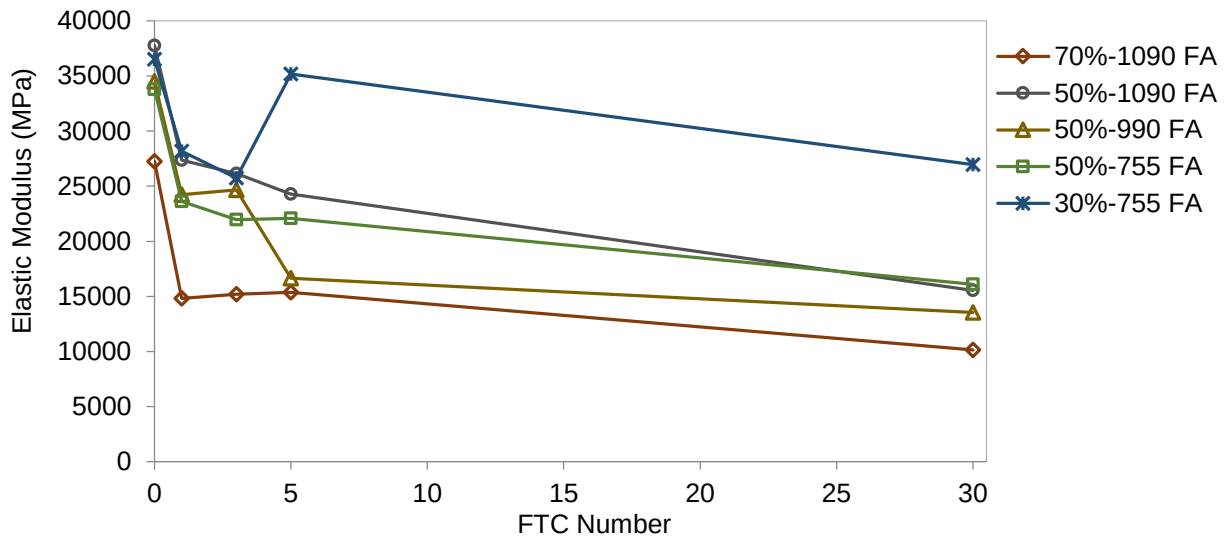


Fig. 3.7 Change in Elastic Modulus

The decrease in elastic modulus has been used as a reference for frost damage in experimental studies (Fagerlund, 2002, Hasan *et al.* 2003). It is an experimental fact that the cause of degradation of the elastic modulus of concrete/mortar is due to the development of microcracks when frost damage sets in. When microcracking occurs, the stiffness of the material is reduced because of the broken bridges, which do not permit stress transfer in the

material, thus resulting in the reduction of the mechanical properties (Hasan *et al.* 2003) i.e. the elastic modulus.

3.4.4 Increase in Void Ratio during FTC

The data set obtained from X-ray CT scanning of the central portion of the specimen (10x10x3 mm) consists of 100 contiguous slices of reconstructed CT images with thickness of 19 micron each. Stacking up these slices creates a 3D image of the scanned section of the specimen. Each slice is a grayscale image having a matrix size of 1024x1024 pixels with an in-plane resolution (x-y plane) of 23 $\mu\text{m}/\text{pixel}$. Accordingly, each voxel (volume element or volumetric pixel) in the raw CT data has anisotropic dimensions of 23x23x19 μm . Figure 3.8 shows example of images of a particular slice acquired from X-ray CT scanning of specimens having different mix proportions. Fine aggregates are imaged as patches of various shades of gray and appear brighter relative to the darker shades of gray of the surrounding cement paste. On the other hand, the air voids and/or pores are seen as black or very dark pixels in the images. As can be observed, air voids are visibly increasing on the sample images as the W/C ratio increases for different mixtures. To visualize and quantify the air voids, a number of image analysis routines were applied to the images using ImageJ software (Rasband 2007). The techniques and principles will not be discussed in this study; such have been presented by Promentilla and Sugiyama (2010), and have also been adopted in this study.

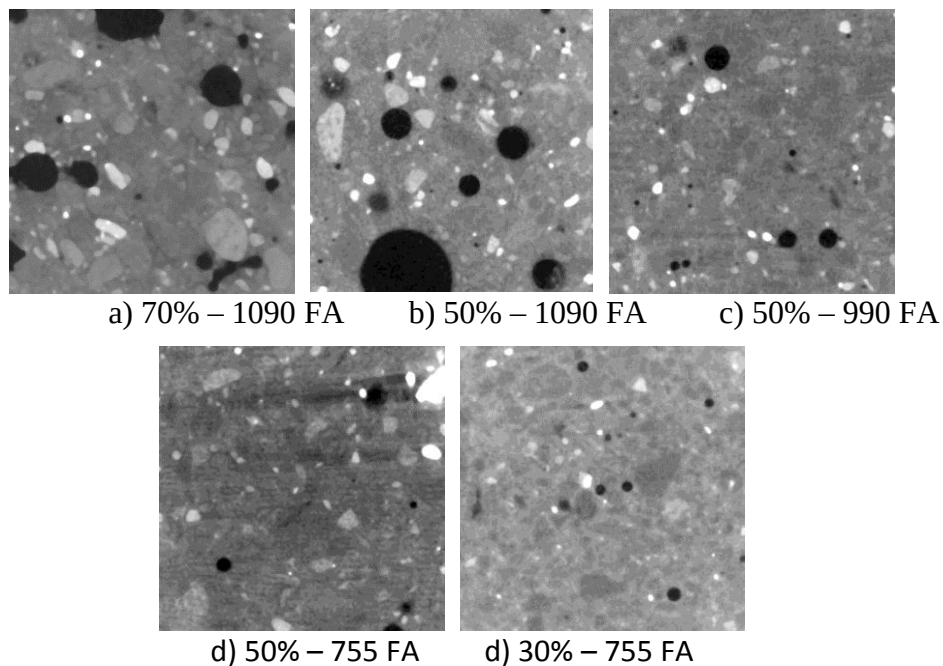


Fig. 3.8 Sample images from X-ray CT scanning with different mix proportions – sample scale 6.2x6.2 mm (No FTC)

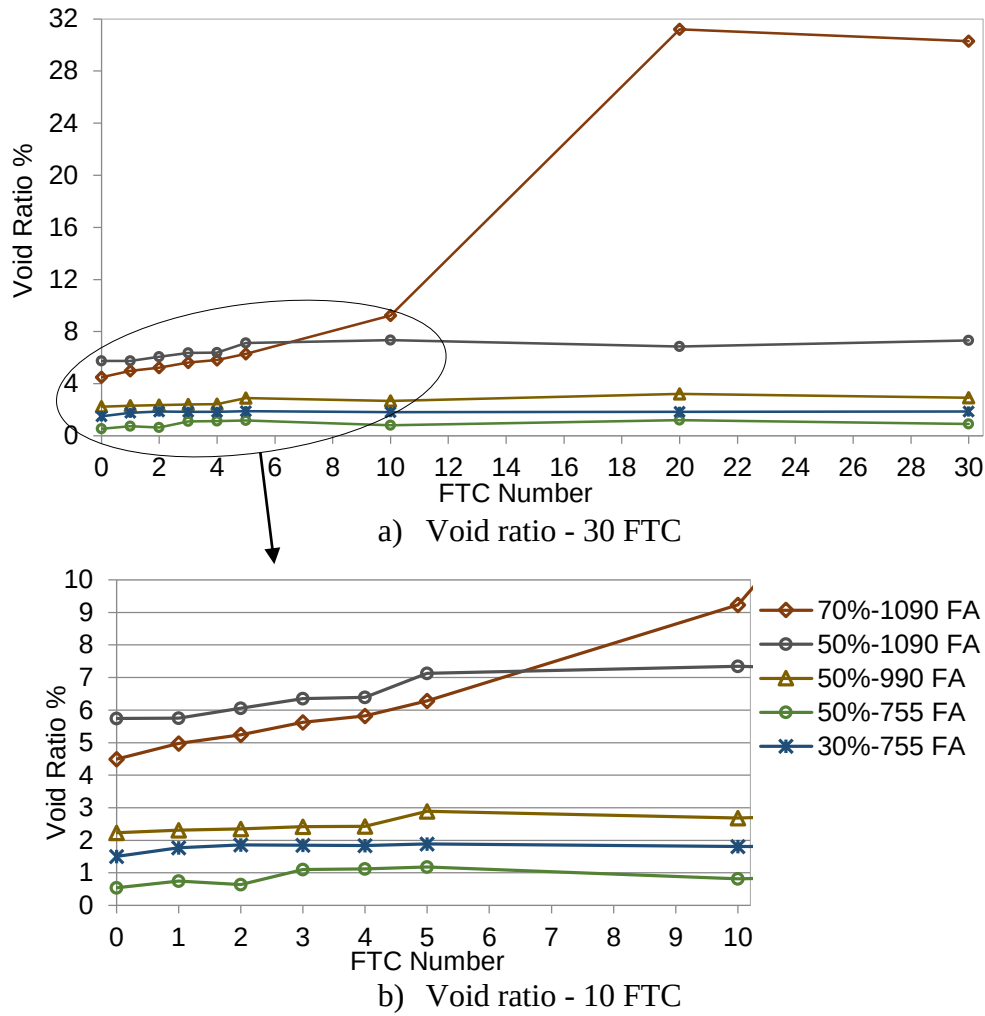


Fig. 3.9 Increase in void ratio for each FTC

The void ratio at every FTC (during the first five cycles) and every five FTC after the 5th FTC are shown in Fig. 3.9. Handling damage occurred during the 20th to 30th cycle for 70%-1090 FA specimens displaying very large and unusual increase in void ratio due to fragileness caused by FTC damage (Fig. 3.9a), the results from 0 to 10 FTC will be discussed instead as shown in Fig. 3.9b. Figure 3.9b indicates 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, display also a 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. The increase in void ratio for the 50%-990 FA, 50%-755 FA and 30%-755 FA specimens is approximately 0.5%. For the 70%-1090 FA and 50%-1090 FA specimens, the increase in void ratio is approximately 2%. 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 scale of the observation is limited to 23 μm . For smaller pores ($< 23 \mu\text{m}$), the formation of microcracks on this scale is not observable at this time. However, during FTC, microcracks develop and can increase pore size, and some percentage of this size increase is detectable by the X-ray CT and adds to the total void ratio of the specimens as reflected in the experimental results (Fig. 3.9). This increase in total void ratio can then be used to validate the development of microcracks reflecting FTC damage. This means that as the void ratio increases, microcracks further develop, suggesting increased damage. Therefore, as the results suggests, X-ray micro computed tomography is capable enough to estimate increase in pore volume and validate microcracking that causes damage during FTC.

3.5 CHANGE IN CTE DUE TO DETERIORATION BY FTC

Figure 3.10 shows results of thermal strains of specimens both for FTC damaged (1, 3, 5 and 30 FTC) and dry (undamaged) specimens. Thermal strains in Fig. 3.10a are obtained from dry specimens strain presented in Fig. 2.4 in Chapter 2 displaying only until the 5th cycle for comparison purposes. Fig. 3.10b to 3.10e are thermal strains of frost damaged specimens (1, 3, 5, and 30 FTC from Fig. 3.6) from different cycles described in Section 2.3. Table 3.1 shows the series of testing and conditions of specimens used for FTC strain tests and subsequently their thermal strain tests (CTE). Observation from Fig. 3.10b to 3.10e suggests that thermal strain of specimens after frost damage increased in negative strain almost twice as much as dry (undamaged) specimens. Using the relationship of strain and temperature variation, the CTE of FTC damaged specimens were obtained and summarized in Fig. 3.11. This shows that the CTE of the said specimens have changed drastically.

Table 3.1 Testing Conditions from FTC Strain tests to Thermal Strain tests

		FTC strain tests		Thermal strain test (for CTE)	
	Specimens	Saturation condition	Number of cycles	Saturation condition (oven dry)	Number of cycles
Dry (undamaged)	*all	-	-	dry	30
1 FTC	all	100%	1	dry	5
3 FTC	all	100%	3	dry	5
5 FTC	all	100%	5	dry	5
30 FTC	all	100%	30	dry	5

*all designates that all mixtures (e.g. 30%-755FA, 50%-755FA, 50%-990FA, 50%-1090FA and 70%-10190 FA) were subjected in the same series of tests

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 such as shown in Section 3.4 and mentioned by Hasan *et al.* (2003). Relating CTE and elastic modulus, when the decrease in elastic modulus occurs after the 1st FTC (see Fig. 3.7), the CTE significantly increases (Fig. 3.11). 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. 3.11. 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.

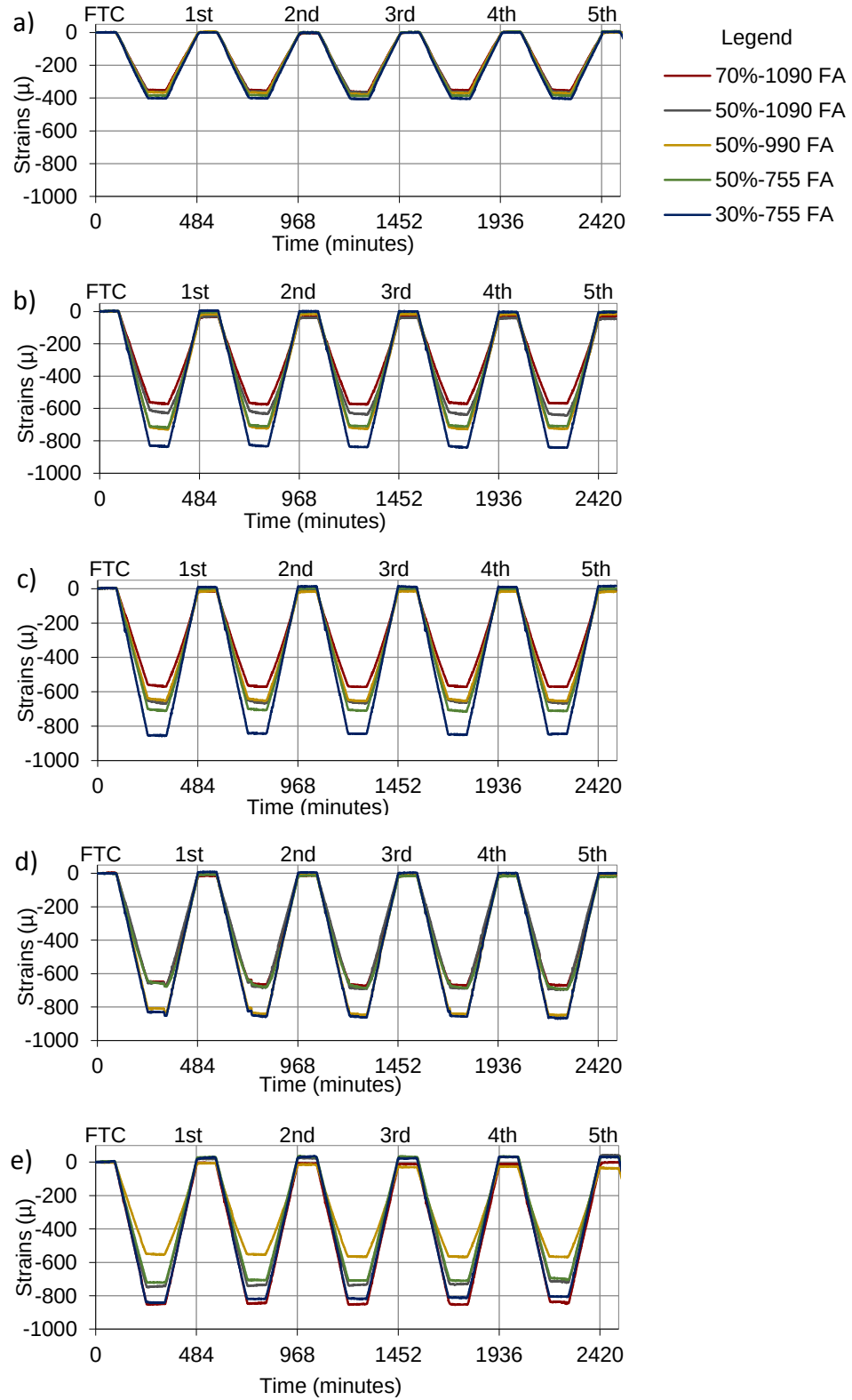


Fig. 3.10 Thermal Strains of FTC Subjected (damaged) Specimens a) dry (undamaged) b) 1 FTC, c) 3 FTC, d) 5 FTC, e) 30 FT

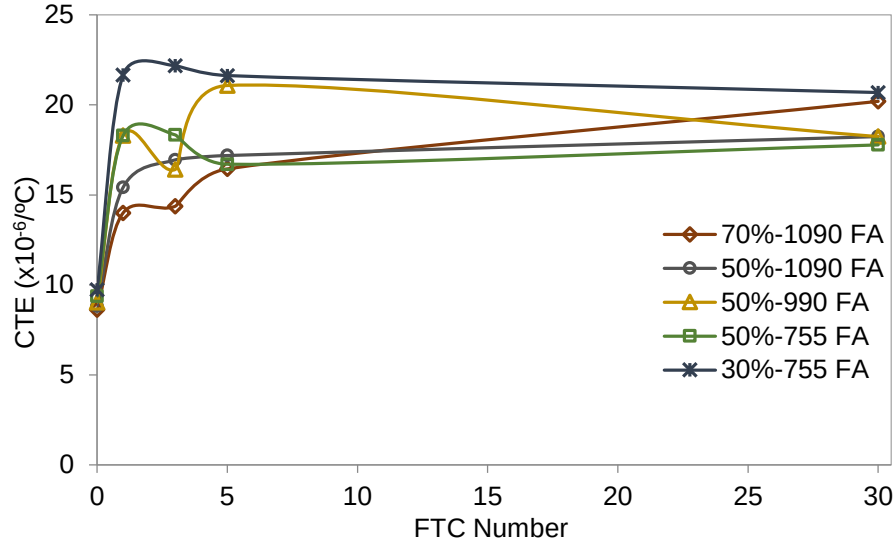


Fig. 3.11 Evolution of the change in CTE

The remaining elastic modulus (REM) is plotted against the increase in CTE in Fig. 3.12. The REM after FTC damage is calculated by the ratio of the change in elastic modulus after FTC (E_N) to the initial undamaged elastic modulus (E_O), while the increase in CTE is obtained from the ratio of the change in CTE after FTC (CTE_N) to the initial CTE (CTE_O). Considering the tendency of the specimens first without taking into account the initial conditions ($\frac{E_N}{E_O} = 1, \frac{CTE_N}{CTE_O} = 1$), it can be observed that upon reaching around 75% REM for 30%-755 FA and 70% REM for 50%-755 FA, there is no further increase in CTE, and any succeeding decrease in REM seems not to influence the CTE. For 50%-990 FA, the same appears to apply, that is, even though there is an increase in CTE upon reaching 48% REM, the CTE returns to the same level when the REM is 70%. For the 70%-1090 FA and 50%-1090 FA specimens, however, as the REM decreases, the CTE correspondingly increases. However, the incremental increase in CTE for 50%-1090 FA is decreasing as the REM decreases, which indicates that if the REM decreases further, eventually the CTE will be unaffected. It therefore appears that the CTE will become uninfluenced by a certain threshold decrease in REM. This threshold is lesser for specimens with high stiffness than specimens with low stiffness. In summary, nonetheless, considering the initial conditions of REM and increase in CTE, Fig. 3.12 shows a clear tendency for CTE to increase when REM decrease.

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 (Shui *et al.* 2010; Uygungöçlü and Topcu 2003; Mukhopadhyay *et al.* 2007; 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, 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 (Monteiro *et al.* 1985) also demonstrates that the detachment between aggregate and cement paste can take place due to microcracking. It is verified from SEM observations (Jacobsen *et al.* 1995, 1996; Yang *et al.* 2006 cited by Wang and Ueda 2009) that after FTC exposure, cracks are mostly seen in the interface between cement paste and aggregates.

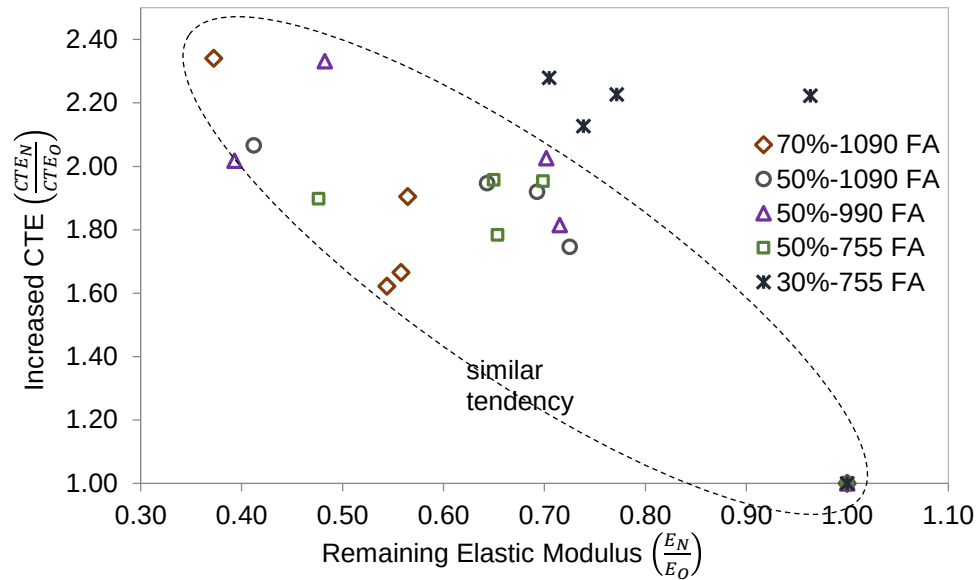


Fig. 3.12 Increased in CTE and Remaining Elastic Modulus

The concept being discussed can be further explored from Table 3.2 (Cruz and Gillen 1980), wherein the thermal expansion of hardened cement paste, mortar and concrete is measured from temperature ranging from 27 to 871°C. It can be observed from Table 3.2 that even though Elgin and Ottawa mortar have identical mixture as hardened cement paste, both CTE values of the composites is lower than that of hardened cement paste. For both concretes, which have almost the same W/C but some differences in the mix proportions, the CTE is also lower. This validates the thermal restraint that exists between the constituent parts resulting in lower CTE of composite when aggregate is incorporated. If the restraint is removed (e.g. by microcracking), the composite can then expand/contract more due to the higher CTE of the original hardened cement paste, but can only be lesser or equal to the original CTE of hardened cement paste. It can be true then from these observations that the CTE of mortar or concrete changes.

Table 3.2 Mix Proportions and CTE of Hardened Cement Paste, Mortar, and Concrete

Specimen Type	Mix proportions by weight (C:S:A)*	W/C by weight	Cement (kg/m ³)	Air (%)	Temperature range	CTE (x10 ⁻⁶ /°C)
Cement Paste	-	0.4	1411	-	27-149	14.8
Elgin Mortar	1:1.75	0.4	561	-	27-871	7.4
Ottawa Mortar	1:1.75	0.4	561	-	27-871	9.9
Elgin/Dolomite Concrete	1:2.70:3.05	0.42	337	4.5	27-871	12.6
Elgin Concrete	1:2.70:2.98	0.42	337	4.5	27-871	11.2

*C-cement, S-sand, A-aggregate

In addition, Schapery (1968) proposed methods to calculate the CTE of composite materials derived from the principles of thermoelasticity which relates the material's CTE and bulk modulus. For a two-phase material, a simple solution is presented in Eq. 3.3:

$$\frac{\alpha_v - 3\alpha_2}{3(\alpha_1 - \alpha_2)} = \frac{K_1 (K_2 - K)}{K (K_2 - K_1)} \quad (3.3)$$

If we apply Schapery's theory in mortar's case, then α_v is the composite's (mortar) volumetric expansion and K is the bulk modulus, α_1 and K_1 are hardened cement paste's CTE and bulk modulus respectively, and α_2 and K_2 are CTE and bulk modulus, respectively, of fine aggregates. The elastic modulus can be calculated using its established relationship from the bulk modulus. Assuming microcracks take place due to frost damage, the CTE of hardened cement paste increases, its elastic modulus decreases, fine aggregates' CTE and elastic modulus remains unchanged since it is not affected by frost damage, and the composite's (mortar) elastic modulus decreases, as experimental results show. It can be obtained from Eq. 3.3 then that the resulting composite's volumetric thermal expansion α_v increases. From this demonstration, it can be seen that the concept and experimental results agree with the existing theory; that is, when the elastic modulus decreases, the CTE increases. In this regard, it can finally be said that due to frost damage, development of microcracks takes place, which causes the increase in CTE and decrease in elastic modulus.

3.6 APPLICATION OF THE CHANGE IN CTE

The change in CTE implies that the strain behavior of specimens shown in Fig. 3.6b may not be the actual strain history under moisture influence as the experimental results imply. Through the course of the 1st FTC the change in CTE variably changes in relation to the microcracking until a point where the maximum change in CTE happened either before the 1st cycle ends or when the FTC ended. However for simple presentation, it is assumed that the maximum change in CTE happened after the 1st FTC. To obtain the actual strain history, the average change in CTE (after the first FTC until the 30th FTC) is obtained and this was used to remove the thermal strain after the first FTC. The actual strain history of specimens is shown in Fig. 3.13 and an example comparison of the strain behavior of specimens is presented in Fig. 3.14 using undamaged CTE and FTC damaged CTE.

Comparing Fig. 3.6b and 3.13, it can be observed that the strains have the same tendency wherein large expansion which are the product of the volume expansion of water when it turns into ice can be observed at the initial stages of the FTC. This expansion is the dominant strain behavior of the specimens for 70%-1090 FA, 50%-1090 FA, 50%-990 FA and 50%-755 FA. This reverses to negative strain as can be observed particularly for 30 FTC.

As the specimens exhibited the same tendency in strain behavior, if observed more closely, it is apparent that the strains induced have changed. Noticeably the expansion at the initial stages of FTC have increased up to 300 micro strains for all the specimen and every cycle in 3, 5 and 30 FTC in Fig. 3.13 from Fig. 3.6b as a result of the CTE change. More importantly, the large negative strain in Fig. 3.6b observed at the later stages of the FTC decreased significantly in Fig. 3.12 which makes the contraction to a rather credible value where in it could be said a product of moisture behavior. The change in strain (decrease in contraction) is due to the change in CTE. This observation on the difference in strain behavior of specimens can be clearly observed in Fig. 3.14. In this strain comparison, the thermal strains are removed using

FTC damaged and undamaged CTE. This illustration suggests that using the undamaged CTE in obtaining the strains induced by moisture may not be the actual strains. Applying the change in CTE will enable researchers to obtain the actual strain behavior and the associated stresses caused by moisture behavior during FTC. Obtaining the actual deformation and stresses in concrete during FTC in concrete are crucial in understanding the frost damage and prediction of life cycle of concrete.

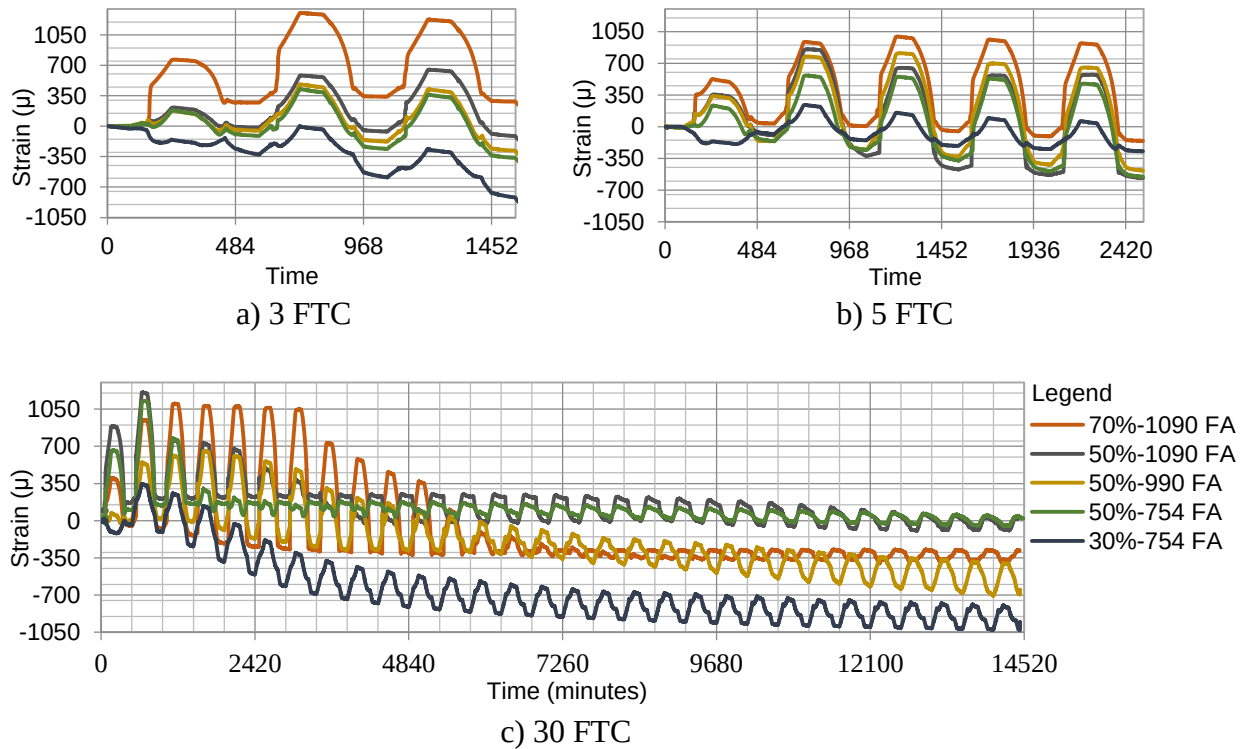


Fig. 3.13 Strain behavior of saturated specimens applying the FTC damaged CTE (change in CTE).

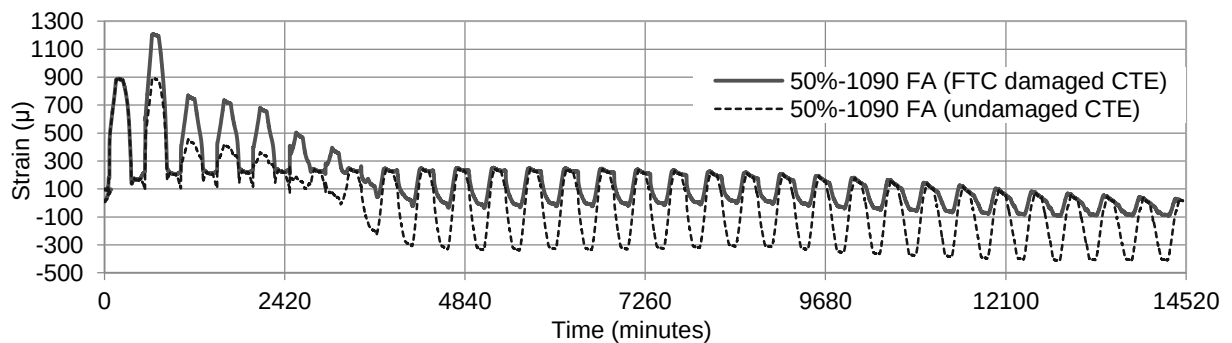


Fig. 3.14 Strain behavior of 50%-1090 FA saturated specimens applying FTC damaged CTE and undamaged CTE in removing thermal strains

3.7 CONCLUSIONS

The study demonstrates that the damage brought by FTC significantly alters the CTE of mortar. Prior to FTC tests, an HCC test was performed between an oven-dried and undried

mortar. Results suggests that oven drying (up to 105 °C) does not much affect the CTE of mortar which ensures that the results obtained are reliable. Both the elastic modulus and CTE of mortar were then determined after FTC tests and the results shows that the CTE of frost damaged mortar increases while its elastic modulus decreases which verifies the damage from frost actions. The increase in CTE and decrease in elastic modulus are explained as the result of micro cracking which can be initiated or be prominent in the weak ITZ when frost damage sets in. The presence of microcracks are represented by the increase in void ratio of the specimens which are observed to increase until the 5th FTC. Microcracks act as broken bridges or links which can detach the aggregate from the hardened cement paste and in effect reduces (or removes) the thermal restraints that each part (fine aggregate and cement paste) exerts on the other. The hardened cement paste can then expand or contract more freely under temperature variation, and thus can significantly affect (increase) the CTE of the whole composite (mortar). Furthermore, microcracks acts as broken bridges which prevents the stress transfer in the material resulting in decrease of the elastic modulus.

There also appears a maximum frost damage as reflected in the reduction of elastic modulus associated with the limited moisture content of the samples, where in the change in CTE will be maximum and remains uninfluenced in the succeeding FTC. The reduction in elastic modulus and change in CTE are also in agreement with the increase in void ratio which represents the microcracking of the specimens. All these physical changes in concrete - the decrease in elastic modulus, change in CTE, and increase in void ratio all took place during the initial FTC which relates to the frost damage experience during FTC.

The change in CTE of mortar has been taken for granted in both design and maintenance of concrete structure with the common understanding that it is not affected by frost actions or other deteriorations. However, in the current study the findings indicate otherwise which shows the increase in CTE when damage sets in. The study illustrates that using the undamaged CTE in obtaining the strains induced by moisture is not the actual strains induced by moisture behavior. By applying the change in CTE (FTC damaged), the actual strain behavior and the associated stresses caused by moisture behavior during FTC will be obtained, in which these are crucial in understanding and simulation of frost damage in concrete. The results of the study could be of importance in the life cycle prediction of concrete structures which can potentially open a new perspective in research, design and maintenance of concrete (structures).

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Chapter 4

THE DEFORMATIONAL BEHAVIOR OF THE INTERFACIAL TRANSITION ZONE AND AGGREGATE PHASE DURING FREEZING AND THAWING CYCLES

4.1 BACKGROUND

Generally, concrete is a two-phase material – the aggregate phase and the matrix which is the mortar phase (when mortar is treated as the composite material then the hardened cement paste is classified as matrix). In reality, a third phase exists in concrete – the interfacial transition zone (ITZ). The ITZ may be composed of the same materials as the bulk matrix, however its microstructure, morphology, density and other properties are different from that of the matrix (Akçaoğlu *et. al* 2004; Mehta and Monteiro 2006). This is why the ITZ is treated as a separate phase of the concrete microstructure.

The ITZ is commonly identified as the weakest link and has relatively the smallest size among the other phase of concrete, yet it has the most profound effect on the strength, durability and mechanical behavior of concrete. The higher porosity of the ITZ plays a key role in this weakness by enhancing transport processes within concrete and facilitating ingress of moisture and aggressive agents into concrete (Cwirzen and Pentalla 2005).

Despite the important role of the ITZ, information and studies about the ITZ particularly during freezing and thawing cycles are scarce due to experimental difficulties. Among the available studies (Hussin and Poole 2010, Cwirzen and Pentalla 2005, Basheer *et. al* 2005, Akçaoğlu *et. al* 2004, Monteiro *et. al* 1985) the thickness of the ITZ is often described between 10 – 50 μm . However, few studies focus on the deformational behavior of the ITZ which is very important in understanding the over-all behavior of concrete. Since the ITZ is separate phase in concrete, it is expected that its deformational characteristics is different from mortar/cement paste (matrix) and aggregate. To understand or simulate the over-all deformation of concrete then it is significant that each of the phase's (aggregate, matrix, and ITZ) deformational behavior must be known.

In addition, there is no universally accepted test method to determine the deformational behavior of the ITZ. It is therefore the purpose of this study to present an experimental method which can obtain the deformational behavior of the ITZ from the matrix and the aggregate phase during freezing and thawing cycles (FTC). The results of the investigation are first in this study and have not been obtained in the microstructural investigation of the ITZ. Most importantly, the results aims to better understand the behavior of the ITZ not only during FTC but also its over-all role in combination with that of matrix and aggregate during concrete deformation.

4.2 EXPERIMENT METHODOLOGY

The purpose of the experiment is to obtain the deformational behavior of the ITZ during freezing and thawing actions using strain gages and observations using a digital microscope. 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.

4.2.1 Specimen Preparation

The materials used in this experimental program were ordinary Portland cement with density of 3.14 g/cm^3 , fine aggregate which is 1.2 mm or less in size with density of 2.67 g/cm^3 and with water absorption rate of 1.2% , and coarse aggregate obtained from a single parent rock which is a type of diorite igneous rock. No air entraining agent is incorporated in order to promote frost damage.

Most 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 (Hussin and Poole 2011, Monteiro *et al.* 1985). 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. Preparation of specimens is shown in Fig. 4.1.

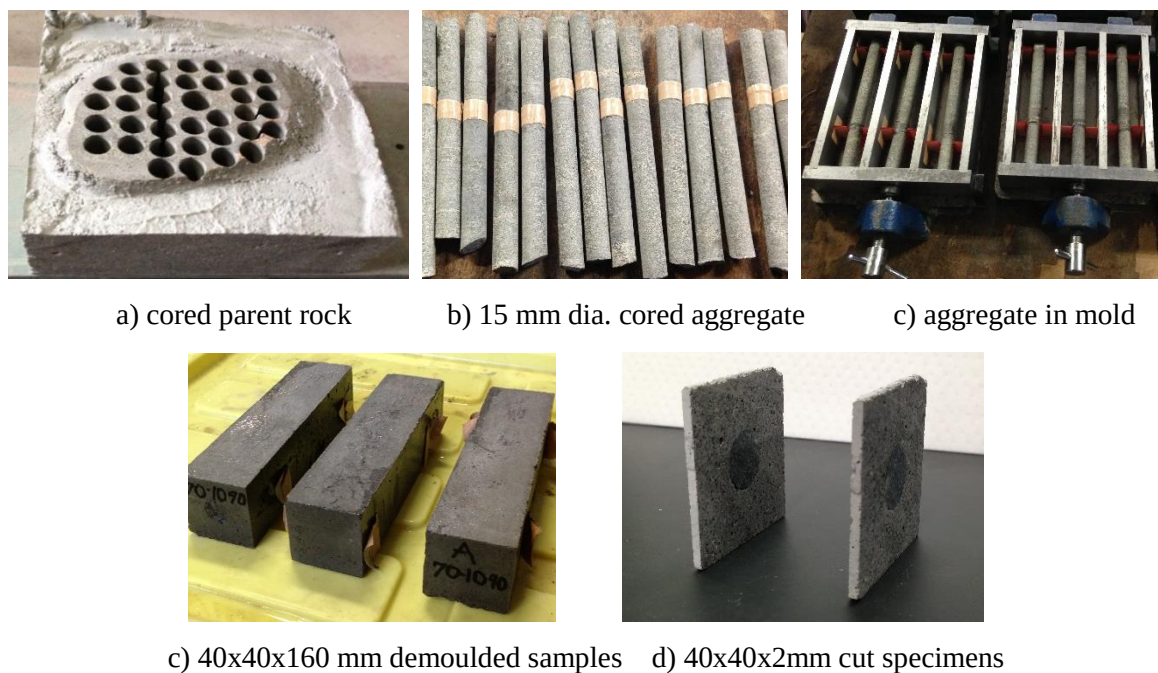


Fig. 4.1 Preparation of specimens

For the concrete proportions, the mortar mixture was also based from ACI 211.1 with the same mix proportions however in this case only three mix proportions are used as seen in Table 4.1. 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 $40 \times 40 \times 160 \text{ mm}$ 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 $40 \times 40 \times 2 \text{ mm}$ the typical specimens used in this study. The meso-scale was chosen so that moisture content and temperature change will be uniform in each of the specimen's constituent parts (mortar, aggregate and ITZ) resulting in uniform frost damage in each of the respective part. For this purpose the meso-scale size can be used to simulate the frost actions in a specific (localized) location of a bulk sample with conditions

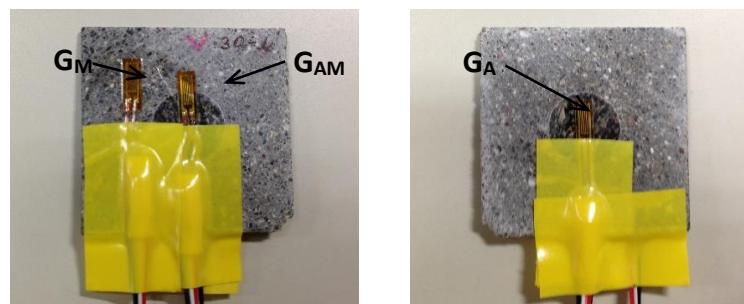
similar with the reference data (i.e. uniform moisture and temperature variation as with the current samples).

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. 4.2: G_M represents the attached strain gauge on mortar, G_{AM} is the attached strain gauge on the aggregate-mortar boundary, and G_A is strain gauge attached on the aggregate. By this method, strains can be obtained from the aggregate phase, mortar phase, and mortar-aggregate boundary. The strain gauge used are self-temperature compensation gage having grid width of 2.5 mm, length of 5 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. Strain gages were ensured to be tightly bonded in the material by curing the adhesive for 24 hours. 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 which is discussed in Section 4.3.

Table 4.1 Mix Proportions of matrix and elastic modulus, approximate ITZ width

Water Cement Ratio (%)	Water (kg/m ³)	Cement (kg/m ³)	Fine aggregate (kg/m ³)	Average ITZ width (μ m)	Elastic Modulus (GPa)
70	207	296	1090	29.38	27.24
50	207	414	990	24.37	34.50
30	207	690	755	16.45	36.51

Dry and saturated conditions were prepared for each specimen's mixture. For dry condition, oven drying of specimens for 24 hours was also undertaken. While for fully saturated condition, specimens were put in water under vacuum until their mass is constant. Similarly, after moisture condition for each of the specimens is attained, sealing quickly follows using Polyvinylidene chloride plastic commonly known as Saran as the first layer of sealing. Then on top of it the vinyl/mastic tape was used to seal the specimens used specifically for moisture sealing.



(a) Front side: attached strain gauges on mortar and aggregate-mortar boundary;
(b) Backside: attached strain gauge on aggregate

Fig. 4.2 Attachment on strain gauges on both sides of mortar

4.2.2 Freeze-thaw Tests

Tests for FTC were set-up similarly with the previous chapters as illustrated in Fig. 2.2 in Chapter 2. FTC have also the same temperature history however in this case it was repeated 20 times for all the specimens.

4.3 OBTAINING EXPERIMENTAL 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 shown in Fig. 4.3. 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). The measured (approximate) width of the ITZ from eight samples are also compiled in Table 4.1. There is no existing standard or study which describes the method to determine the exact width of the ITZ, however the obtained values are in agreement with the given descriptions from previous investigations where the ITZ width is 10-50 μm (Hussin and Poole 2010, Cwirzen and Pentalla 2005, Basheer *et al.* 2005, Akçaoğlu *et al.* 2004, Monteiro *et al.* 1985). 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%).

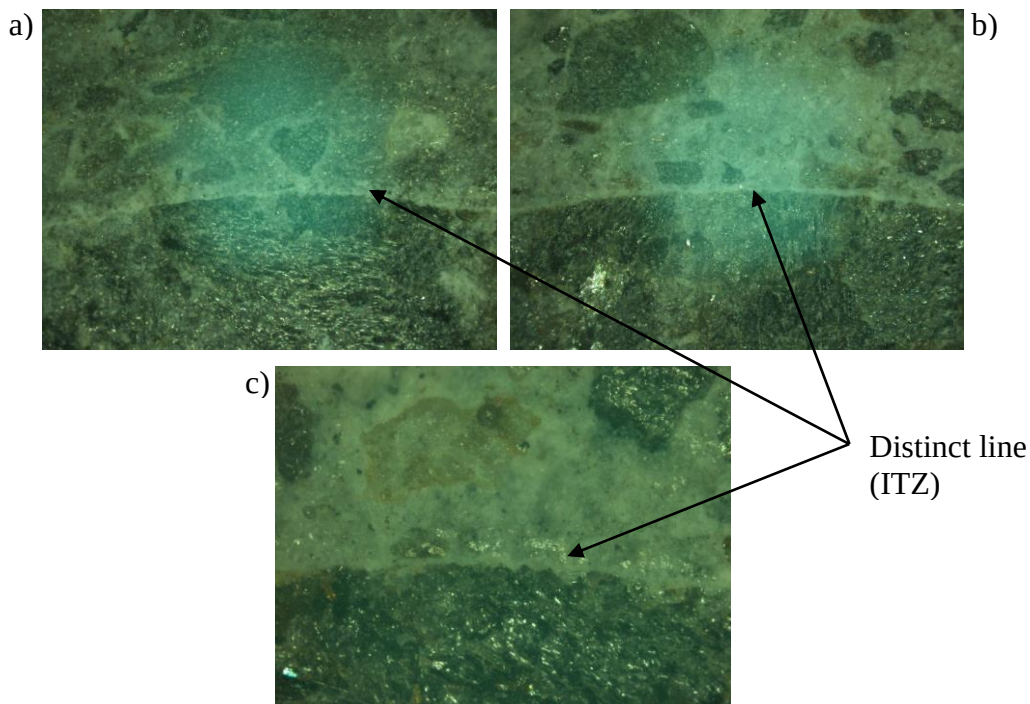


Fig. 4.3 Distinct ITZ width (a) 30% w/c, (b) 50% w/c, (c) 70% w/c

The strain gage attached on the mortar-aggregate boundary (G_{AM}) shown in Fig. 4.2 was particularly observed using an electron microscope. The illustration in Fig. 4.4 shows the magnified lengths of the portions attached on each of the phases, on mortar – L_M , aggregate – L_A , and on the L_{ITZ} were measured. Gages are ensured to be tightly bonded on the material so that any minute deformation by the constituent parts (mortar on L_M , aggregate on L_A , and ITZ on L_{ITZ}) will be picked up by the strain gage. In this sense considering gage G_{AM} , individual

deformation of the portions will not affect the deformation of the other portions where the strains gage is attached. For instance, if mortar and ITZ expands then the gage portion in L_M and L_{ITZ} expands respectively however if the aggregate portion did not experience any deformation then this portion on L_A will not deform, while the output of strain gage implies the total behavior of each individual portions. This account has been verified by an engineer from the gage manufacturer. Thus Eq. 4.1 then represents the total deformation on G_{AM} which is composed of the deformations on the attached portions - ΔL_A for the aggregate deformation, ΔL_M for mortar and ΔL_{ITZ} for the ITZ.

$$\Delta L_{AM} = \Delta L_A + \Delta L_M + \Delta L_{ITZ} \quad (4.1)$$

From the strain relationship, the total deformation (ΔL) 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. Then, strain obtained from length L_M (attached on mortar) is the same as the strain from G_M , the same applies for the strain in G_{AM} and G_A . Using these strains, the total deformation of each of the constituent parts - mortar on gauge G_M and aggregate on gauge G_A can be obtained. With all the requirements obtained and based on Eq. 4.1, using Eq. 4.2 the total deformation (ΔL_{ITZ}) of the ITZ can then be calculated where ε_{AM} is the strain on the aggregate–mortar boundary (G_{AM}), where ε_A is strain obtained aggregate phase (G_A) and ε_M is strain obtained mortar phase (G_M).

$$\Delta L_{ITZ} = \varepsilon_{AM} \cdot L_{AM} - \varepsilon_A \cdot L_A - \varepsilon_M \cdot L_M \quad (4.2)$$

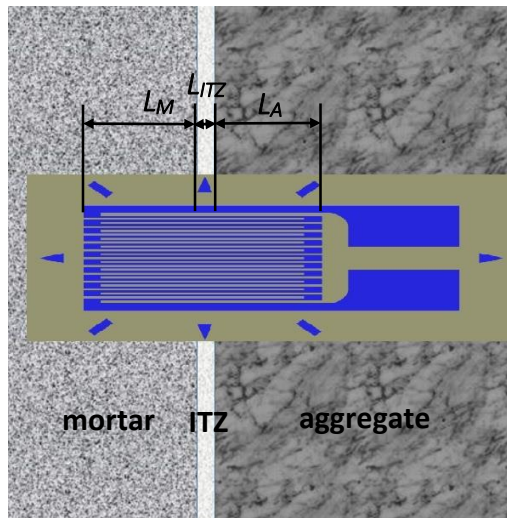
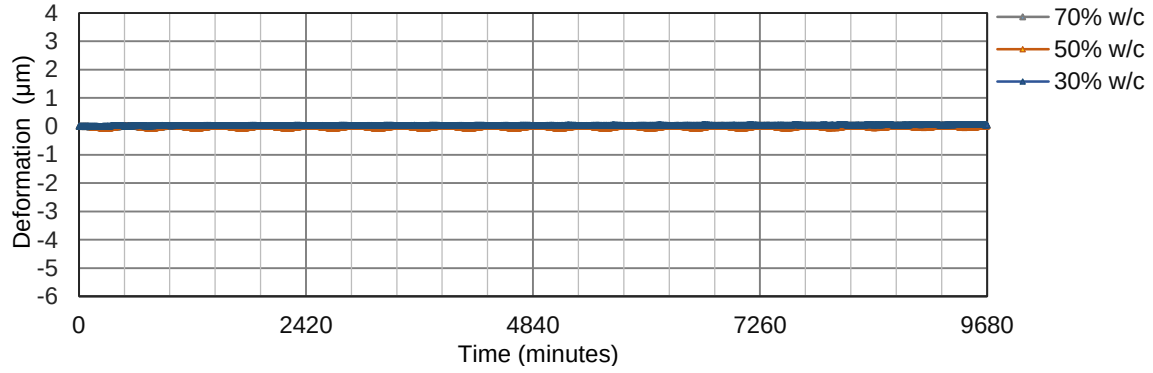


Fig. 4.4 Illustration of attached strain gauge on aggregate-mortar boundary

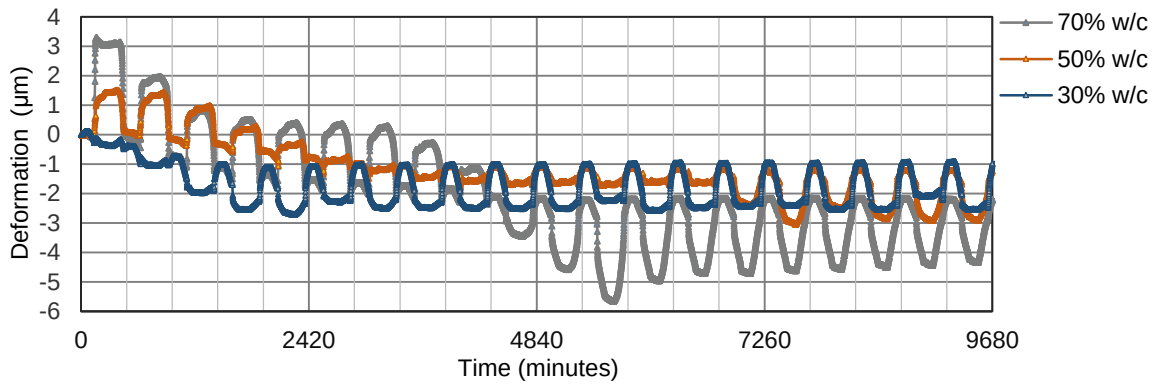
4.4 RESULTS AND DISCUSSIONS

4.1 Deformation of the ITZ during FTC

The average deformations during FTC obtained from different moisture conditions are shown in Figs. 4.5a, and 4.5b. The behavior of the ITZ is obtained based on the average of three specimens.



(a) ITZ deformation for dry specimens

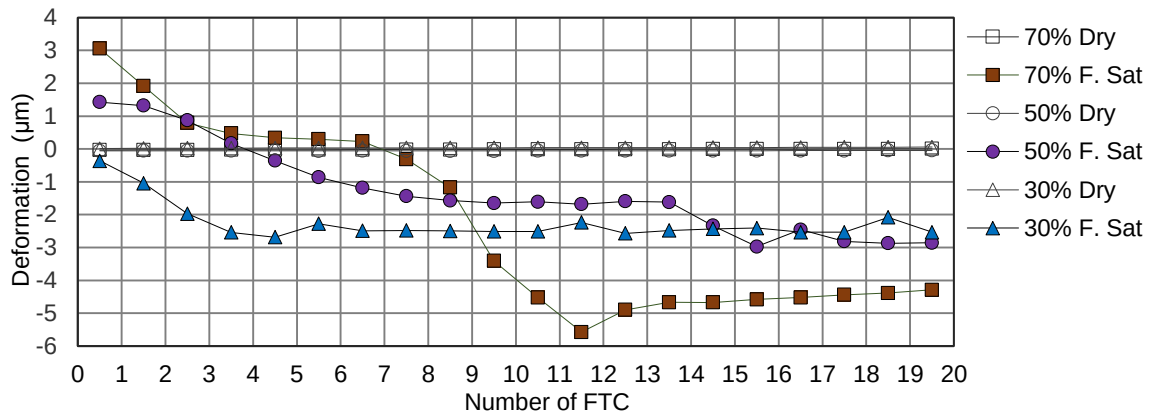


(b) ITZ deformation for fully saturated specimens

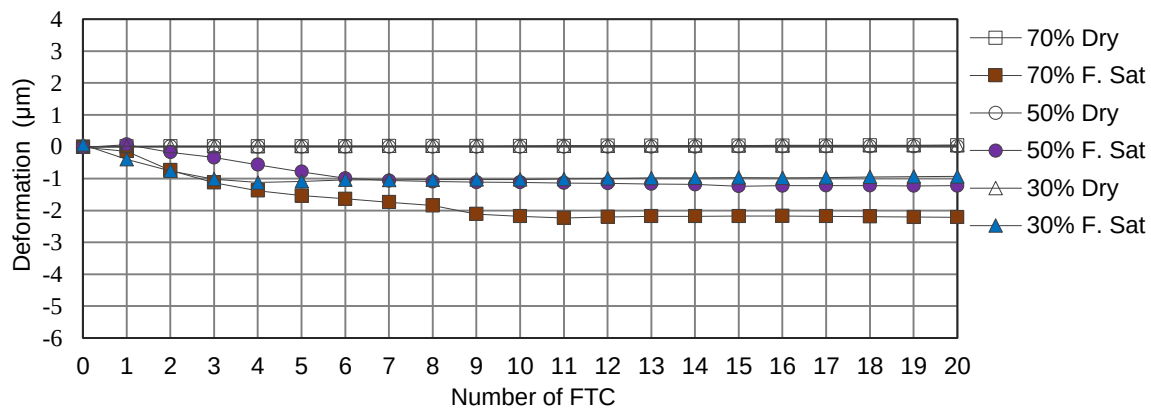
Fig. 4.5 Calculated deformation of the ITZ

For dried condition in Fig. 4.5a, 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. 4.5b. As have been pointed out in the previous chapters, 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. The large deformation observed for fully saturated specimens can be clearly observed in Fig. 4.6a where the maximum relative deformation (expansion and contraction) at each cycle is extracted while Fig. 4.6b shows the remaining deformation after each FTC. 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 (Akçaoğlu et. al 2004; Cwirzen and Pentalla

2005, Ayhan *et al.* 2010). 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 (Fig. 4.5b) is similarly attributed to the sudden volume expansion of water when it turns into ice. The expansion of frozen water may cause tensile stresses in the surrounding matrix and if cannot be relieved microcracks initiate (Hale *et al.* 2009) particularly in the ITZ.



a) Maximum relative deformation at each FTC.



b) Remaining deformation after each FTC.

Fig. 4.6 Extracted strains.

Besides the high expansion observed for 70% and 50% saturated specimens, it can also be observed that the large pore water volume results in higher contraction marked in the later stages of the FTC in Fig. 4.5b or 4.6a. Notice that after the first FTC the maximum expansion slowly decreases for both saturated case and reverses to contraction as the FTC progresses. The same patterns have been observed for mortar's deformation in Chapter 2. However, since the ITZ is higher in porosity then higher moisture presence will cause higher deformation in both expansion and contraction than mortar. The mechanisms that governs these behavior can then be attributed for the same reason where the expansion is caused by hydraulic pressure when ice forms, the partial saturation of pores caused by flow of the expanding water to available spaces and microcracks formation leading to the decrease of the maximum expansion and eventually reversing to contraction, the permanent contraction of the system is due to removal

of pore water in small capillaries and gel pores and absence of moisture supply to re-saturate the pore system. The same is also applicable for 30% w/c saturated case where the contraction of the specimens is due to the partial saturation of its pore system because of low w/c ratio of the specimen which result in the low porosity with high percentage of small/fine pores (Okpala 1989; Goto and Roy 1981) in the ITZ (Akçaoğlu et. al 2004, Ayhan *et al.* 2010). The detailed discussion can be referred in Section 2.5.2 in Chapter 2.

Significantly, the higher the maximum deformation experienced the resulting permanent deformation as shown in Fig. 4.8b will also be large. In Fig. 4.6b it can be seen that the residual deformation for all saturated specimens specifically 70% w/c has relatively the highest remaining deformation followed by 50% w/c then 30% w/c which are more obvious in the later stages of the FTC. However, for dry specimens there is almost no deformation observed, particularly both the maximum deformation (Fig. 4.6a) and remaining deformation (Fig. 4.6b) remains unchanged through FTC. Based on these observations, it is obvious to say that the higher the moisture content the larger will be the maximum deformation and the resulting remaining deformation of the ITZ.

4.4.2 ITZ Observation after FTC

After FTC tests, ITZ observations was conducted. Prior to the observations, the sealing is removed. No visible cracking was observed for dry specimens as observed in Fig. 4.7a indicating the non-occurrence of frost damage and evident in the non-changing remaining deformation in Figs. 4.5a and 4.6a. However, though the Saran cancels the affinity of the specimen's surface to the sealing material, brakeage occurs upon removing the seal for 70% w/c and 50% w/c saturated specimens shown in Fig. 4.7b. This has not been observed to happen in mortar in Chapter 3, wherein after FTC test specimens were oven dried and retested for FTC. This indicates that frost actions have caused considerable damage on the ITZ of specimens making them fragile thus removing the seal aids in the breakage of the specimens. 70% w/c fully saturated specimens are almost completely shattered upon removal of the seal indicating greater damage in comparison with 50% w/c specimens which shows visible cracking. This confirms that higher moisture content in pores increases damage during FTC. This is also reflected in the large deformation (Figs. 4.5b and 4.6a) and remaining deformation (Fig. 4.6b) obtained where 70% w/c shows larger deformation and followed by 50% w/c. On the one hand, no visible cracking occurs for 30% w/c saturated specimen. However microcracks in the ITZ itself may have occurred though not visible in the observations used which are suggested in the remaining deformation (Fig. 4.8b) of the specimen and a similar investigation conducted (Sicat *et al.* 2013). We have to take note that concrete is very weak in tension which is why the high expansion caused considerable damage and cracking in 70% and 50% saturated specimens, however concrete's compressive strength is magnitude higher than in tension which indicates that high compressive stress are needed to cause the similar cracking and brakeage. This suggests why no cracks are visible for 30% saturated case in Figs. 4.7b which exhibited purely contraction during the FTC. A closer look of the unbroken sections of the ITZ of all specimens by electron microscope in Fig. 4.8 confirms these observations on crack occurrence. There were no visible cracking in the ITZ of dry specimens (Fig. 4.8a) and for 30 % w/c saturated specimens (Fig. 8b). For 70% and 50% w/c saturated specimen in Fig. 4.8b, however, prominent cracking along the ITZ and mortar are visible. Overall as shown, the observation in Figs. 4.7 and 4.8 agrees well with the deformations obtained in Fig. 4.5 and 4.6 which indicates the reliability of the presented method.

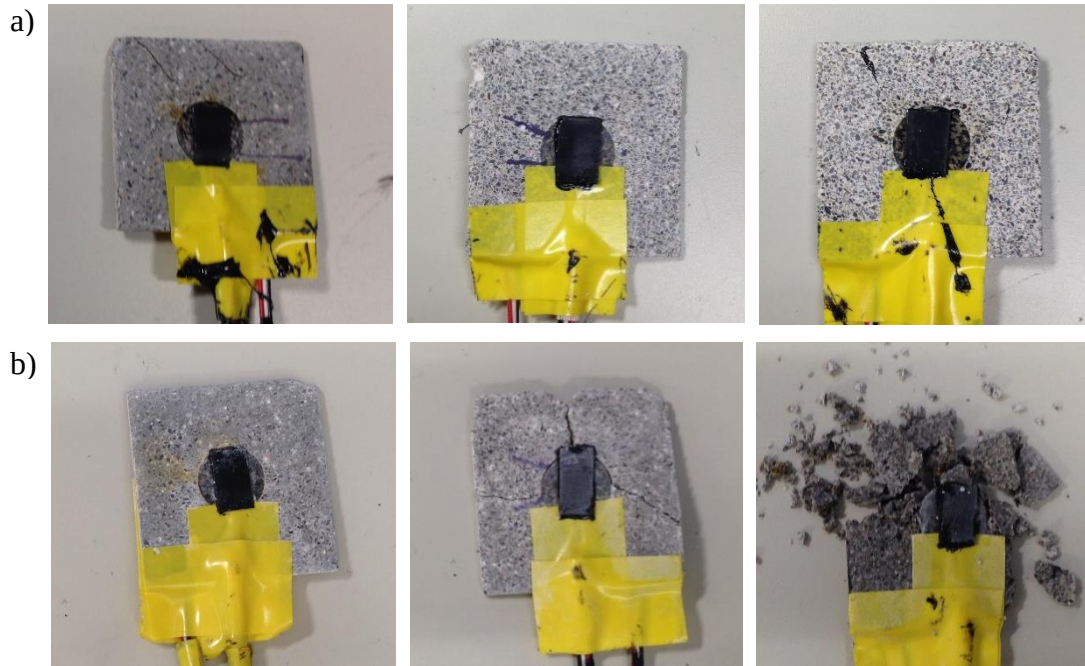


Fig. 4.7 Specimens after FTC test (from left to right 30%, 50%, and 70% w/c): (a) dry conditions, (c) saturated conditions.

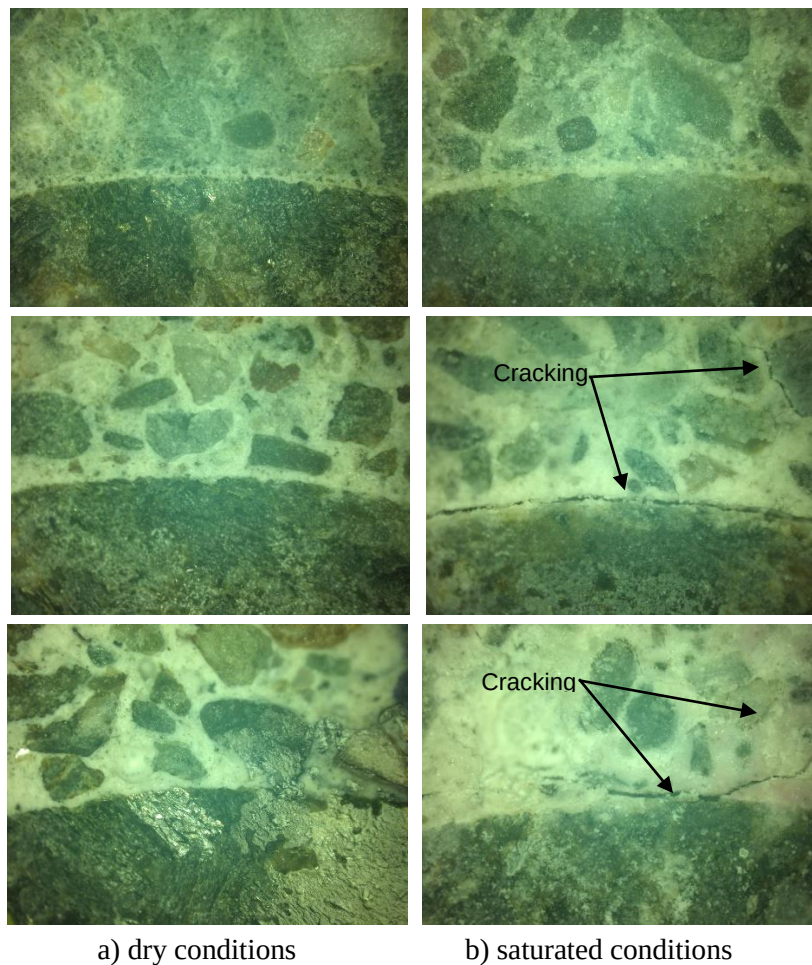


Fig. 4.8 ITZ after FTC (from left to right 30%, 50%, and 70% w/c);

Further, the cracking along the ITZ may have been exacerbated by the removal of seal, nonetheless cracking along the ITZ are still inevitable to occur due to the high deformation experienced during FTC. The high deformation experienced (Figs. 4.5b) and the visible cracks along the ITZ in Fig. 4.8b for 50% and 70% w/c saturated specimens show evidence of crack and prominent damage initiation in the ITZ which suggests its important role in frost damage of concrete and validate that the ITZ is indeed the weakest link in the concrete system.

4.4.3 Strains during FTC

Strains for all saturated specimens' ITZ, mortar and aggregate were compiled in Fig. 4.9 to explore their difference. Strains for ITZ (ε_{ITZ}) were obtained using the simple relationship of strains and deformation shown in Eq. 4.3.

$$\varepsilon_{ITZ} = \frac{\Delta L_{ITZ}}{L_{ITZ}} \quad (4.3)$$

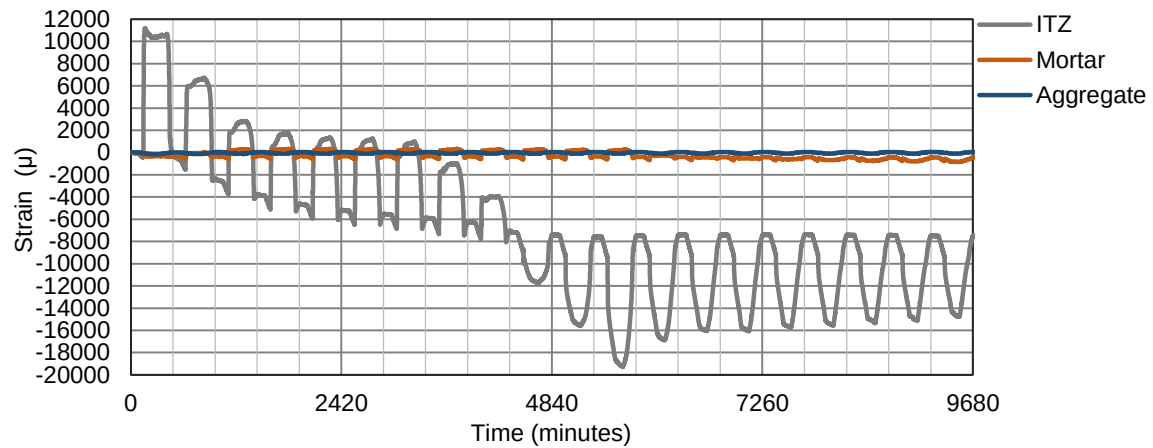
Where ΔL_{ITZ} is obtained from ITZ deformation in Fig. 4.5 and L_{ITZ} is the averaged measured ITZ width from observations shown in Table 4.1. While strains for mortar and aggregate were obtained during testing from the attached strain gages (G_M and G_A). Take note however, that the obtained strains for the ITZ is an estimate since the width L_{ITZ} is based from the approximate measurement of the ITZ obtained from microscope observations. Adjustment of the ITZ width used in the calculation will give a variation in the obtained strain. The displayed strains are for comparison purposes only between the three phases, nonetheless the obtained strain in mortar reflects the usual range basing from previous results (Sicat *et al.* 2012).

From the compilation of strains in Fig. 4.9, though with the least relative size, the ITZ has the largest strains reached in comparison with mortar and aggregate. The strain reached for the ITZ is almost 30 times than mortar which indicates that the relatively small sized ITZ has deformed distinctly and critically.

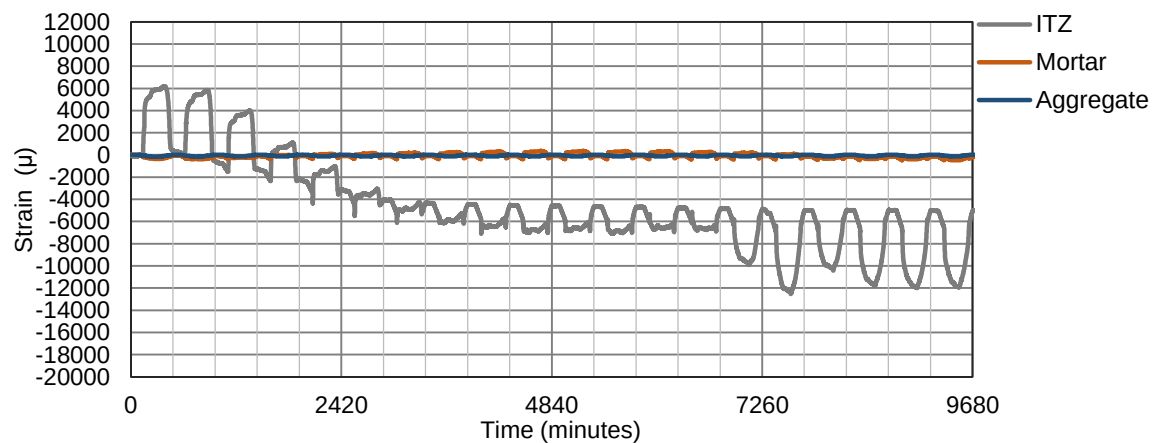
It is also worth mentioning that the observations on the ITZ behavior (seen in Fig. 4.9) are almost similar with the mortar behavior (Fig. 4.10a) and in Chapters 2 and 3 wherein large increase in expansion and its gradual decrease to contraction for fully saturated case are observed. One difference could be, the gradual decrease in expansion in mortar begins after several FTC whereas the expansion in ITZ decreases right after the first FTC. The partial saturation in pores took place faster in the ITZ than the mortar due to the higher permeability (as a result of the high porosity), therefore pore pressure due to water expansion becomes less efficient in the succeeding cycle. The partial saturation in pores takes additional or several cycle for mortar. In this sense, it can be said that the FTC mechanism in mortar is almost similar to that of ITZ as discussed in Section 4.4.1 however to a far lesser extent.

Taking into consideration of the aggregate phase, a reproducible strain behavior during FTC without the complexity displayed in both mortar and ITZ is evident in Fig. 4.10b even though the samples are preconditioned for full saturation. In addition, all three specimens under different w/c reflects the same reproducible behavior which is obviously because the same origin of the aggregate is used. This behavior indicates that aggregate is insignificantly affected by FTC actions. Moisture may not be able to penetrate the dense structure of the aggregate and the strain behavior is generally due to thermal response of the material during FTC. This can be further understood in Fig. 4.11 where the residual strain after FTC for the aggregate, mortar and ITZ are summarized. In Fig. 4.11 the residual strain of aggregates are the least and are

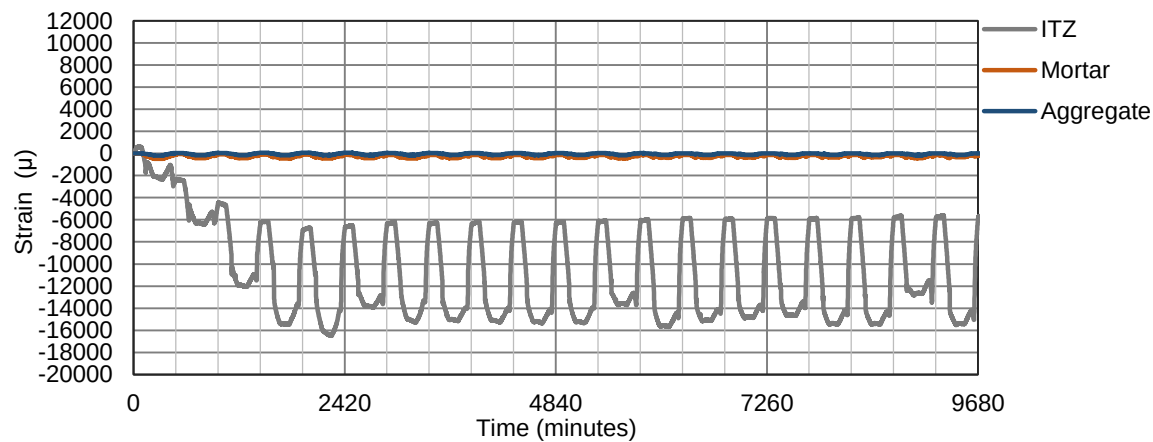
insignificant in comparison with the much larger permanent deformation of ITZ and mortar. The permanent deformation is also noticeably unchanged all through the FTC unlike mortar and ITZ which varies and increases. This verifies the common understanding that aggregate are less unaffected by frost damage however this may still be dependent on the type, properties, and pore structure of the aggregate used (Hale *et al.* 2009).



(a) 70% w/c strains

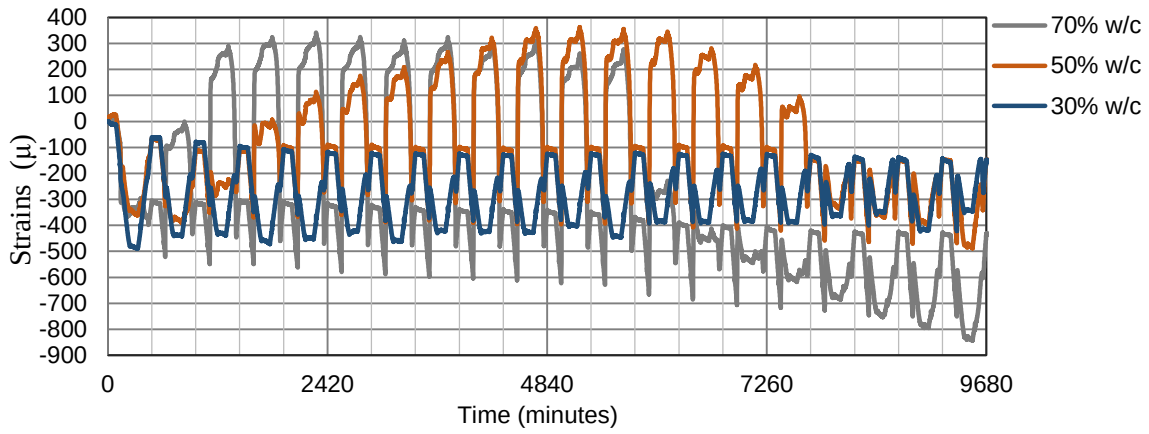


(b) 50% w/c strains

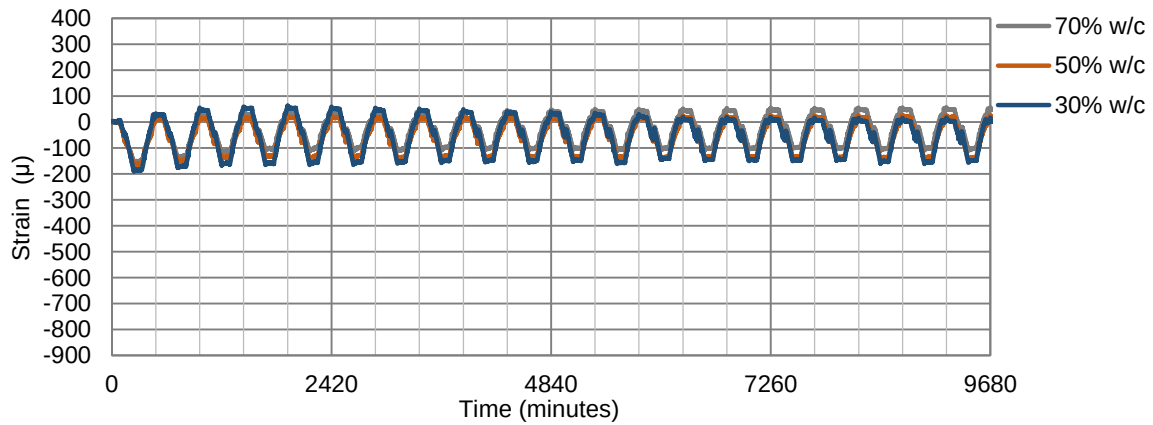


(c) 30% w/c strains

Fig. 4.9 Strain comparisons for ITZ, mortar and aggregate



(a) Mortar strains



(b) Aggregate strains

Fig. 4.10 Compilation of strains

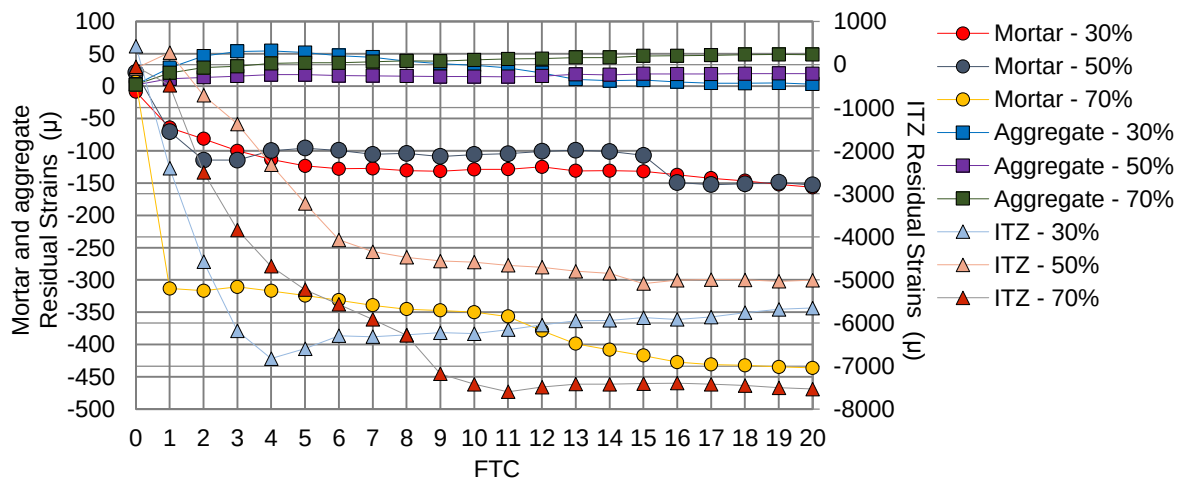


Fig. 4.11 Remaining Strains for ITZ, mortar and aggregate

The experimental methods illustrating the real-time deformation of the specimens particularly the ITZ during FTC is the first in this study and may not be obtained in image analysis techniques. When specimens breaks (Fig. 4.6b) due to fragileness caused by frost damage image analysis may not be applicable. The behavior of mortar and aggregate in this investigation reflects similar findings in studies of frost damage in mortar (Sicat *et al.* 2012)

and the high deformation of the ITZ obtained in this investigation reflects its expected behavior due to its high porosity and weakness. In addition as mentioned in Section 4.4.2, the deformation of the ITZ obtained using the methods discussed here agrees very well with the observations on the occurrence and non-occurrence of cracks during FTC. Therefore it can be said that the simple experimental methods presented in this investigation may be used as a basis to obtain the deformational behavior of ITZ during FTC.

4.4.4 Effect of ITZ on Concrete Deformation and Damage

ITZ is introduced in the concrete system with aggregate. Due to its low strength and higher porosity than the bulk matrix ITZ is expected to have lower strength and stiffness than the other two phases. The higher the w/c the lower will be the strength and stiffness (see elastic modulus in Table 4.1). The 70% w/c saturated specimen showed the highest ITZ deformation (Fig.4.7) and breakage at the ITZ after FTC. This also indicates that with wider ITZ (70% w/c) specimens will have lower tensile strength. The presence of weak and less stiff ITZ in the concrete system prevents the full restraint between aggregate and matrix. With the absence of full restraint, under repeated thermal changes differential deformation between the two phases takes place. This results in residual strains as presented recently (Sicat *et al.* 2012, 2013), such strains are very small and may be insignificant in comparison with other deformation such as FTC deformation (excluding the thermal effect) but could produce minimal microcracking and increased permeability.

The high expansion observed at the initial stages of FTC may cause extensive damage resulting in cracking in the ITZ and extends in the surrounding mortar as seen in Fig. 4.10b in 70% and 50% w/c since the ITZ tends to expand more than the matrix and aggregate (Fig. 4.11). While contraction in the ITZ can also cause (micro) cracking in the ITZ itself since its greater contraction is restricted by the low contraction of the surrounding phases. The microcracking characterized by the increase in void ratio due to FTC damage was presented in the previous study (Sicat *et al.* 2013). Both cracking in this case are considered as frost damage.

Aggregate has lower coefficient of thermal expansion (CTE) than hardened cement paste (Mukhopadhyay *et al.* 2007; Al-Ostaz 2007). When incorporated into concrete it restrains the thermal movement of the hardened cement paste. Frost damage in mortar has been observed to cause drastic (increase) changes in the material's coefficient of thermal expansion (CTE) (Sicat *et al.* 2013). The relationship of the increase in CTE and reduction in elastic modulus which represents the frost damage was shown in Fig. 3.12. The ITZ serve as the link between the aggregate phase and the matrix (mortar phase). However with the occurrence of cracking (frost damage) in the ITZ, the link between aggregate and matrix weakens. Thus, the hardened cement paste (matrix in the previous study) can expand or contract more freely than the aggregate (having lower CTE) resulting in significant increase in the CTE of the whole composite.

On the one hand, during wet-dry cycles (WDC) with the involvement of moisture during the phenomena, expansion and contraction (shrinkage) can be observed similarly during FTC. Having higher porosity resulting in increased moisture presence, it is expected that the ITZ may contract and expand more than mortar and aggregate which may induce cracking in the ITZ itself and mortar. Moisture expansion during drying (temperature elevation) have been observed to increase permeability due to microcracking (Ai *et al.* 2001) while the shrinkage (contraction) during moisture loss has been discussed to induce cracking by various studies (Gao *et al.* 2013, Vandewalle 2000). With this regard, both FTC and WDC may induce cracking

in concrete. Cracking in ITZ is due not only to the weaker strength of the ITZ but also to the greater expansion and contraction of ITZ than mortar and aggregate.

4.5 CONCLUSIONS

This chapter presents a simple yet ingenious method to obtain the real-time deformation of the ITZ particularly during FTC using strain gage, which is first in this study. The obtained deformational behavior of the ITZ, mortar, and aggregate in this investigation reflects their commonly described behavior during FTC. The ITZ deformation agrees well with the observations on the occurrence of cracks for frost-damaged specimens and non-occurrence of cracks for dry specimens. These suggest that the experimental method presented in this study can be used as a basis to obtain the deformational behavior of ITZ during FTC.

Though the specimens were conditioned to be fully saturated, the aggregates did not indicate considerable permanent deformation during the whole FTC. This verifies the common acceptable knowledge that aggregate are unaffected by frost damage. While mortar shows a complex behavior which is similar to the ITZ however to a lesser extent since in reality the mortar behavior is a combination of (fine) aggregates, ITZ around them, and hardened cement paste (matrix). Moreover, the ITZ has higher porosity resulting in larger moisture presence which is the primary driver for deformation during FTC.

The ITZ deformation is determined as distinct (being far higher) from the matrix and aggregate which is the crucial reason why the ITZ is treated as a separate phase in concrete. Besides its weaker strength, the much higher deformation of the ITZ during FTC where cracking and prominent damage are initiated and could extend to the matrix shows the important role of the ITZ during frost damage and other factors such as wet dry cycles (WDC). Thus, arises the need for the development of such methodology as presented. In combination with the deformational behavior of mortar and aggregate, the ITZ deformation obtained in this investigation can be used as basis to understand and simulate the deformational behavior of concrete not only during FTC but also by various other reasons.

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Chapter 5

CONCLUSIONS AND SUMMARY

The core findings of the experimental investigation are summarized below in order to provide significant information in clarifying and understanding the deformational behavior of concrete. The obtained results are suggested to be applied

1. The experimental investigation suggests that the behavior of mortar under FTC is primarily dependent on the amount of its moisture. The larger the amount of moisture, the larger will be the resulting deformation in both expansion and contraction behavior.
2. Divergent to typical FTC experimental investigation wherein expansion is attributed as the primary cause of frost damage. In the present study, contraction is also observed prior to the expansion. The contraction is caused by the thermodynamic imbalance in the pore system when ice is formed in a partially saturated pore. Negative pressure arises between ice in larger pores and unfrozen water in small capillaries either due to chemical potential or pressure differences which draw out water in small capillaries/pores resulting in the contraction.
3. In comparing experimental results between a continuously saturated sample and sealed sample during FTC, obvious difference in strain behavior is evident between the two experimental methods. In the former's test, increasing expansive strains are observed as the FTC progresses on the one hand for the latter's result, the strain at the later stages of the FTC shows (increasing) contraction otherwise even if the specimens are preconditioned to be saturated.
4. After FTC tests, the findings demonstrates that the damage brought by FTC significantly alters the CTE of mortar. Results shows that the CTE of frost damaged mortar increases while its elastic modulus decreases. The increase in CTE and decrease in elastic modulus are explained as the result of micro cracking which can be initiated or be prominent in the weak ITZ when frost damage sets in. The presence of microcracks are represented by the increase in void ratio of specimens. The occurrence of microcracks act as broken bridges or links which can detach the aggregate from the hardened cement paste and in effect reduces (or removes) the thermal restraints that each part (fine aggregate and cement paste) exerts on the other. The hardened cement paste can then expand or contract more freely under temperature variation, and thus significantly affect (increase) the CTE of the whole composite (mortar). Furthermore, microcracks acts as broken bridges which prevents the stress transfer in the material resulting in decrease of the elastic modulus which verifies the damage from FTC.
5. There appears a maximum frost damage as reflected in the reduction of elastic modulus associated with the limited moisture content of the samples. In this state, change in CTE will be maximum and remains uninfluenced in the succeeding FTC. The reduction in elastic modulus and change in CTE are also in agreement with the increase in void ratio

which represents the microcracking of the specimens. All these physical changes in concrete - the decrease in elastic modulus, change in CTE, and increase in void ratio all took place during the initial FTC which relates to the frost damage experience during FTC.

6. The study illustrates that using the undamaged CTE in obtaining the strains induced by moisture is not the actual strain induced by moisture behavior. By applying the change in CTE (FTC damaged), the actual strain behavior and the associated stresses caused by moisture behavior during FTC will be obtained.
7. The ITZ deformation is determined as distinct (being far higher) from the matrix and aggregate which is the crucial reason why the ITZ is treated as a separate phase in concrete. Besides its weaker strength, the much higher deformation of the ITZ during FTC where cracking and prominent damage are initiated and could extend to the matrix shows the important role of the ITZ during frost damage and other factors such as wet dry cycles (WDC).
8. Though the specimens were conditioned to be fully saturated, the aggregates did not indicate considerable permanent deformation during the whole FTC. This verifies the common acceptable knowledge that aggregate are unaffected by frost damage. While mortar shows a complex behavior which is similar to the ITZ however to a lesser extent since in reality the mortar behavior is a combination of (fine) aggregates, ITZ around them, and hardened cement paste (matrix). Moreover, the ITZ has higher porosity resulting in larger moisture presence which is the primary driver for deformation during FTC.
9. As a part of a series of studies to predict the structural performance of member with frost damage, the next task is to enhance the previously developed meso-scale model of mortar under FTC by applying the findings on the CTE change and damage caused by the contraction behavior. The model of mortar-aggregate interface (ITZ) in meso-scale can be developed based on the data and findings obtained in the developed methodology in Chapter 4. The study confirms that aggregate are not affected by FTC, then the combination of the meso-scale model in mortar and ITZ can simulate the frost damage in concrete.

The deformational behavior of the ITZ, mortar, and aggregate obtained in this investigation reflects their commonly described behavior during FTC. Moreover, the deformation of the ITZ and mortar obtained using the methods discussed here agrees very well with the observations on the occurrence of cracks for frost damaged specimens and non-occurrence of cracks for dry/non frost damaged specimens. These indicate the reliability of the presented method and suggests that the experimental method can be used as a basis to obtain the deformational behavior of mortar, aggregate and ITZ during FTC. Most significantly, the combination of the obtained real-time deformation of the phases of concrete, mortar, aggregate and particularly the ITZ have not been attempted before. Significantly, the results presented here are first and a complete data on the deformational behavior of mortar, aggregate, and ITZ have not yet been presented. The observed contraction and change in CTE of concrete during FTC have also not been observed previously and shows great influence in the total deformation of concrete. Thus, the results and methodology presented in this study are therefore important in clarifying, understanding and application in the simulation of frost damage in concrete.

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