Influence of Matrix Quality and Environmental Conditions on Volume Change and Microcracking Behavior of Concrete

Raqeeb Abdulqader Hussein

Submitted to the
Institute of Graduate Studies and Research
in partial fulfillment of the requirements for the degree of

Master of Science
in
Civil Engineering

Eastern Mediterranean University
July 2015
Gazimağusa, North Cyprus

Approval of the Institute of Graduate Studies and Research

Prof. Dr. Serhan Çiftçioğlu
Acting Director

I certify that this thesis satisfies the requirements as a thesis for the degree of Master of Science in Civil Engineering.

Prof. Dr. Özgür Eren
Chair, Department of Civil Engineering

We certify that we have read this thesis and that in our opinion it is fully adequate in scope and quality as a thesis for the degree of Master of Science in Civil Engineering.

Asst. Prof. Dr. Tülin Akçaoğlu
Supervisor

Examining Committee

1. Prof. Dr. Özgür Eren
2. Assoc. Prof. Dr. Mustafa Ergil
3. Asst. Prof. Dr. Tülin Akçaoğlu

ABSTRACT

Concrete is a highly complex and heterogeneous engineering material. In its complex composite structure, it is not easy to understand its behavior either during hydration process or loading the material. Particularly the volume change during hydration results in initial defects and at the end these defects may influence its mechanical behavior under load. Individual properties of different phases like aggregate, matrix and the interfacial transition zone (ITZ) between the two, plays an important role on the microcracking behavior of the concrete.

In this study, various techniques were used in determining the effect of hydration shrinkage crack on the microcracking behavior of the concrete including w/c ratio, silica fume and environmental conditions. Direct measurement by means of optical processing through Scanning Electron Microscope (SEM) is a way. On the other hand, the indirect measurements dealt with an overall study of the material by means of the tensile and the compressive strength measurements, the length and the volume change measurements and the prediction of the critical crack load from stress-strain diagrams. Conclusions were drawn from the direct and the indirect methods. The ultimate purposes of all the performed tests were used to measure the initial defects either in the ITZ or the matrix and their effect on the whole microcracking behavior of concrete.

Keywords: SEM, ITZ, Cracks density, Compressive strength, Tensile strength, Volume change, Shrinkage.

ÖZ

Beton, oldukça karmaşık yapıya sahip heterojen bir yapı malzemesidir. Bu karmaşık yapısından dolayı; betonun hem hidratasyon sırasındaki hem de yük altındaki davranışının anlaşılması kolay değildir. Özellikle, hidratasyon sırasında oluşan hacim değişimi; ilk çatlakların oluşmasına yol açmakta bu çatlaklar da betonun yük altındaki davranışını etkilemektedir. Agrega, matriks ve de agrega ile matriks arasındaki arayüz bölgesi gibi betonu oluşturan fazların bireysel özellikleri betonun yük altındaki mikro-çatlak oluşumunu önemli derecede etkilemektedir.

Bu çalışmada, hidratasyon rötre çatlaklarının betonun yük altındaki mikro-çatlak davranışı üzerindeki etkisi farklı teknikler kullanılarak; s/ç oranı, silis dumanı ve çevre koşulları da dikkate alınarak incelenmiştir. Bu tekniklerden bir tanesi direct ölçüm yapılabilen electron mikroskop analizidir. Diğeri ise indirect yoldan çatlak oluşumunun incelenmesidir. Bunlar; çekme ve basınç dayanımlarının bulunması, boy ve hacim değişimlerinin ölçülmesi ve de gerilme-şekil değiştirme eğrilerinden kritik gerilmenin saptanması gibi ölçümleri içermektedir. Sonuçlar her iki metot çıktılarının incelenmesl neticesinde bulunmuştur. Yapılmış olan tüm deneylerden; yükleme öncesi var olan hasarın tespit edilmesi ve de bunların betonun yük altındaki mikro-çatlak davranışına etkilerinin bulunması amaçlanmıştır.

Anahtar kelimeler: Çatlak yoğunluğu, Arayüz, Mikroskop, Basınç dayanımı, Çekme dayanımı, Hacim değişimi, Rötre.

DEDICATION

To my dear mom, dad, wife, sisters and brothers who always supported and loved me.

ACKNOWLEDGMENT

I would like to express my gratitude to my supervisor Assistant Professor Dr.Tülin Akçaoğlu for her useful comments, remarks and engagement through the learning process of this master thesis. Furthermore, I would like to thank the Civil Engineering Department of the Eastern Mediterranean University.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

w/c:	Water to Cement Ratio.
σ_t:	Tensile Strength of Concrete (28-days).
σ_c:	Compressive Strength of Concrete (28-days).
σ_{cr}:	Critical Crack Load.
σ-ε:	Stress-Strain Diagram.

LIST OF ABBREVIATIONS

N:	Concrete without silica fume.
SF:	Concrete with silica fume.
Ne:	Normal environmental condition.
He:	Hot environmental condition.
NNe:	Concrete without silica fume under normal environmental condition.
NHe:	Concrete without silica fume under hot environmental condition.
SFNe:	Concrete with silica fume under normal environmental condition.
SFHe:	Concrete with silica fume under hot environmental condition.
NNeL:	Concrete without silica fume under normal environmental condition after loading.
NHeL:	Concrete without silica fume under hot environmental condition after loading.
SFNeL:	Concrete with silica fume under normal environmental condition after loading.
SFHeL:	Concrete with silica fume under hot environmental condition after loading.
0.35 NNe:	0.35 w/c concrete without silica fume under normal environmental condition.
0.35 NHe:	0.35 w/c concrete without silica fume under hot environmental condition.
0.35 SFNe:	0.35 w/c concrete with silica fume under normal environmental condition.
0.35 SFHe:	0.35 w/c concrete with silica fume under hot environmental condition.

0.50 NNe: 0.50 w/c concrete without silica fume under normal environmental condition.

0.50 NHe: 0.50 w/c concrete without silica fume under hot environmental condition.

0.50 SFNe: 0.50 w/c concrete with silica fume under normal environmental condition.

0.50 SFHe: 0.50 w/c concrete with silica fume under hot environmental condition.

0.70 NNe: 0.70 w/c concrete without silica fume under normal environmental condition.

0.70 NHe: 0.70 w/c concrete without silica fume under hot environmental condition.

0.70 SFNe: 0.70 w/c concrete with silica fume under normal environmental condition.

0.70 SFHe: 0.70 w/c concrete with silica fume under hot environmental condition.

Chapter 1

INTRODUCTION

1.1 General

Concrete is the most commonly used construction material all over the world. It is very well known that concrete provides distinguished mechanical performance, huge flexibility and economic efficiency in contrast with other construction materials. On the other hand, it must be noted that, concrete is discredited for its weakness, and strength to weight ratio. In services, structural concrete undergoes volumetric changes due to water loss by hydration, evaporation and carbonation. Drying and hydration shrinkage cracks have a great influence both on strength and durability of the concrete [1, 2]. Concrete shrinkage is a physical process that concrete displays due to many different parameters. These include w/c ratio, coarse and fine aggregate quantity, cement type, compressive strength, curing type, environmental temperature and humidity, percentage of exposed surface, and exposure time. Shrinkage can cause non-desired stress development, which results cracking and causes decrease in durability and strength of concrete. If it would be possible to prevent or minimize shrinkage occurrence it would be possible to minimize the decrease in strength and durability of the concrete. [3].

1.2 Objectives of the Thesis

In this experimental research, the microcracking behavior of three different w/c ratio concretes under uniaxial compression was studied. Conclusions are drawn from direct and indirect methods. The ultimate purposes of all the performed tests were to measure the initial defects either in the ITZ or within the matrix due to drying shrinkage, and their effects on the whole microcracking behavior of the concrete. Environmental temperature and humidity effects both on the formation of initial cracks and the whole microcracking behavior of concretes with and without silica fume were also discussed in this study.

1.3 Works Done

In this work, various techniques were used in studying the effect of hydration shrinkage crack on the microcracking behavior of concrete including w/c ratio, silica fume and environmental conditions. Among others, one way is direct measurement by means of optical processing on cut surfaces through SEM. Indirect measurements consider an overall study of the material by means of the tensile and the compressive strength measurements, the length and the volume change measurements and the prediction of the critical crack load from stress-strain diagrams.

The effect of w/c ratio, silica fume and two different environmental conditions on hydration shrinkage cracks and whole fracture behavior of concrete were analyzed by interpreting the following:

a) 28-days tensile strength (σ_t) and compressive strength (σ_c) for all mixes.

b) Length and volume changes during 28-days hydration for all mixes.

c) Relationship between stress-strain diagrams and volume changes for all mixes.

d) Direct SEM observation of ITZ and matrix either before or after loading the specimens for all mixes.

e) Correlation between tensile strength, volume change and SEM examination for all types of mixes and conditions.

1.4 Thesis Organization

The present chapter (Chapter 1) of this text includes the general information on concrete hydration shrinkage and microcracking behavior being a scope of this study. The second chapter reviews the state of knowledge on the effects of w/c ratio, silica fume, environmental conditions and hydration shrinkage on microcraking behavior of a concrete. The third chapter present experiment compounds and procedure. The fourth chapter contains the tests results and discussions obtained from the laboratory tests. Finally, the fifth chapter presents the conclusions of this study.

Chapter 2

LITERATURE REVIEW

2.1 Introduction

Concrete is a highly complex and heterogeneous construction material. It is a combination of different sized aggregates (gravel and sand), cementitious materials such as cement, mineral admixtures, and water. In its complex composite structure, it is not easy to understand its behavior either during hydration process or while loading the material. Particularly the volume change during hydration results in initial defects and then these defects may influence the mechanical behavior under load. Individual properties of different phases like aggregate, matrix and the interfacial transition zone (ITZ) between the two, plays an important role on the microcracking behavior of the concrete. Mainly the w/c ratio, mineral admixtures and the environmental conditions affect the mechanical properties of each individual phase in concrete. In previous studies, environmental exposure and temperature were found to be influential on shrinkage [4, 5]. The rest of this chapter present the findings related with the parameters influencing the hydration shrinkage and then the microcracking behavior of concrete by other researcher.

2.2 Effects of w/c Ratio on Volume Stability and Microcracking Behavior of Concrete

The water cement ratio (w/c) can be defined as the ratio of mass of water to mass of cement and binder used in concrete mix that has a great effect on the quality of the produced concrete. Lower w/c ratio leads to an increase in strength and hardness and decrease the volume changes and cracks in concrete, however it is difficult to mix and place low w/c ratio mixes. On the other hand, too much water in the mix results in segregation.

In addition, water that is not absorbed by the hydration process may evaporate as it hardens, resulting in microscopic pores that will reduce the ultimate strength of the concrete. Moreover, more shrinkage will occur due to water loss, resulting in internal cracks. Strict adherence to the water cement ratio limitation of ACI 318 for structural concrete based on the exposure conditions is of great importance, and the project specified design strength should be directly related to proven concrete performance at the maximum permitted water-cement-ratio. Results showed that, increase in the rate of water-cement-ratio in concrete mix with constant amount of micro silica 7% will reduce the compressive strength and the split tensile strength of the concrete at the age of 7 and 28- days. On the other hand, with rising w/c ratio from 0.35 to 0.60, the strain shrinkage and the ultimate shrinkage of concrete will increase nearly one and a half times at 30 and 60-days [6, 7].

From previous studies it is also found that, the plastic shrinkage cracking is significantly affected by the w/c ratio. With increasing w/c ratio from 0.30 to 0.50, the total cracking length will increase while the total width is kept approximately constant and it is moderated for any type of concrete mixture. Image analysis was found to be an effective instrument for the evaluation of plastic shrinkage cracking [8]. When normal concrete (N) is investigated, the interfacial transition zone microcracking was found to increase linearly with raising w/c ratio while this relationship became nonlinear for high strength concrete [9]. In high w/c ratio mixes, the interfacial transition zone is extra definite up to the beginning of crack spread, while it is significant at quick crack spread in low w/c mixtures [10].

Similarly, it has been reported that, compressive strength reduced as w/c of ordinary portland cement concrete increased from 0.26 to 0.35. On the other hand, the total shrinkage increased with increasing w/c ratio. This may be due to the large amount of water loss from concrete to environment. These results correlate well with the weight loss which increases with increasing w/c [11].

2.3 Effects of Silica Fume on Volume Stability and Microcracking Behavior of a Concrete

Silica fume, also recognized as micro silica, is a byproduct of the reduction of high-purity quartz with coal in electric oven in the making of silicon and ferrosilicon alloys. Due to its excessive fineness and high silica content, silica fume is a highly effective pozzolanic material and influences various concrete properties such as compressive strength, tensile strength, bond strength and water absorption capacity with reducing permeability [12].

Investigations show that, silica fume concrete (SF) with 15% and 5% ratio of cement has completely higher compressive strength than the normal concrete. There was a significant increase in compressive strength with increasing silica fume content for both 0.25 and 0.35 water cement ratio concretes. The rates of compressive strength progress were higher in silica fume enhanced concretes than that of normal control concrete for both w/c ratios. These pozzolans in general, were verified to be able to improve the strength of a concrete [11, 13, 14].

Another similar study, on four different ratios of silica fume showed that the best 7 and 28- days compressive strength and flexural strength have been achieved in the range of 10-15% silica fume replacement mixes. Raise in split tensile strength for mixes containing higher than 10% silica fume substitute was found nearly insignificant, while increase in flexural tensile strength have occurred even up to 15% substitutes. When compared to other mixtures, the loss in mass and compressive strength percentage was found to be decreasing by 2.23 and 7.69 when the cement was substituted by 10% of silica fume [15].

Similarly, addition of mineral admixture up to 20% as part substitute of cement improves the microstructure of the concrete in addition to rising the mechanical characteristics such as drying shrinkage for big and small samples, creep, compressive resistance, tensile resistance, flexural resistance, and modulus of elasticity at ages of 7 and 28- days [16,17,18,19,20].

In another research, it was found that, higher compressive strength can be achieved with silica fume inclusion. This is due to the decreased porosity in the ITZ. During the testing of cubes at 28 days, the failure plane passes through the aggregates but not along the ITZ, which led to the conclusion that the ITZ strength is enhanced with silica fume [21].

In contrast for all substitution rates, silica fume modified concretes showed less shrinkage strain in comparison to the plain/normal concretes and reduced the weight loss due to the hardening of the concrete. Silica fume was identified to have a delaying effect on crack initiation and spreading. The initial cracks occurred at 9 and 10- days for concretes containing 5% and 15% silica fume, respectively. The maximum crack width was calculated at control concrete as 0.69 mm, while the lowest crack density was measured as 0.33 mm at 15% silica fume concrete [14,22].

2.4 Effects of Environmental Condition on Volume Stability and Microcracking Behavior of Concrete

According to ACI 305 “Hot Weather Concreting” can be defined as any combination of high ambient temperature, low relative humidity, solar radiation and wind. Hot weather conditions can lead to difficulty in mixing, placing, finishing and curing hydraulic cement concrete that can adversely control the properties and serviceability of the concrete.

Investigations show that, in hot weathers, water addition would not reduce the compressive strength of concrete. The quantity of additional water can provide sufficient moisture for the hydration process . It would also help keeping workability and replacing the mixing water lost by evaporation. This situation can be considered at the mix design of the concrete. In contrast, low w/c ratio concretes result in reduced hardening time due to the increased rate of hydration. This might be the cause of decrease in resistance of these concretes under hot weather conditions. Furthermore, the rate of evaporation of water from the surface of concrete will exceed the rate of bleeding water on the freshly placed concrete which is assumed to be the most important cause of the plastic shrinkage cracks that appear on the surface [23, 24].

Based on some studies, although higher temperatures improved the early strength gain, the rate of strength gain at later ages (at 2 to 4 weeks) appears to be reduced [25]. However, it can be confirmed that higher temperatures usually cause a faster shrinkage process and self-induced stresses, which might accelerate the cracking hazard. At the same time, samples treated at lower temperatures would crack at later ages. It might be hypothetical that higher treated temperatures accelerate the cracking hazard, since the deformations expand at a higher rate [24, 25].

In another study, temperature effects on high-performance concrete (HPC) containing two different types of mineral admixtures such as fly ash and silica fume were studied. Results yielded that high temperature increased the rate of strength gain up to 28-days and mineral admixtures were found to be insignificant [26].

Generally, hot weather cured specimens attained a higher rate of strength development compared to moist cured specimens for all mixes [15].

2.5 Effects of Superplasticizer on Volume Stability and Microcracking Behavior of Concrete

The amount of concrete shrinkage varies depending on the components of the concrete mix, as well as the weather conditions. Those values change significantly, and are different from results quoted by other researchers. Researchers have investigated that, the rate of concrete with the plasticizer admixture shrinkage rate is about 30% greater, compared to the ordinary concrete without the plasticizer admixture, whereas Neville estimates that, the value of concrete with the plasticizer admixture shrinkage is only 10 to 20% greater than that of ordinary concrete [27].

Use of plasticizer or superplasticizer in concrete permitted a bigger dispersal to take place and improved liquidity of the concrete. Dispersal of cement, releasing free water, and low external humidity can increase cement particle hydration, which results in a rapid increase in shrinkage during the first hundred and twenty minutes of gypsum setting in concrete [28]. Hydration of ordinary concrete without superplasticizer and low w/c ratio is very slow and has no great effect on shrinkage stress. Decrease of grout water quantity and using superplasticizer, results in increased hydration, whereas keeping the similar w/c ratio and using the superplasticizer results in accelerated hydration and rise in shrinkage [29, 30].

Another study on the effect of superplasticizer on plastic shrinkage of plain and silica fume cement concrete investigated four types of superplasticizer (Sulfonated naphthalene polymer (SNP); Modified lignosulfate polymer (MLP); Polycarboxylic ether (PCE); and Sulfonated naphthalene formaldehyde (SNF)) with three types of silica fume under hot weather found that, the use of superplasticizer in silica fume increased its resistance to plastic cracks. Furthermore, concrete with silica fume illustrated a higher plastic shrinkage than concrete without silica fume [31].

Experiments and studies have investigated the crucial effects of surrounding humidity on the level of concrete shrinkage. It can be approved that concrete shrinkage in high-moisture environments is lower when compared to low-moisture levels. On the other hand, those principles are also known to be dependent on other factors. It is important to examine the shrinkage during rapid rate hardening which happens particularly within the first 6 -7 hours. The next stage of shrinkage takes place during hardening, and it is due to loss of water from concrete. Understanding the drying shrinkage phenomenon is rather difficult, since there are many factors involved, which are interconnected. It can be observed that, the compressive strength of concrete is reduced by adding superplasticizer to the concrete mix, which is the reverse of what takes place when flexural strength is concerned [28,32].

Chapter 3

EXPRIEMENT MATERIALS AND THE PROCEDURE

3.1 Introduction

In order to investigate the microcraking behavior different types of concrete were produced and examined using direct and indirect methods. The main purpose was to measure the initial cracks due to drying shrinkage either in the ITZ or the matrix, and then to determine their effects on the whole microcracking behavior under load. For this purpose 3 different w/c concretes with and without silica fume were produced and each subjected to 2 different environmental conditions. The materials used and the methods utilized are discussed and explained in this chapter.

3.2 Materials

The materials used in various experiments are described below.

1) **Cement:** Ordinary Portland Cement, BEM 32.5 R confirming with ASTM type II. The physical properties such as fineness and initial and final setting times are given as 2,900 cm^2/gr, 145 minutes and 210 minutes, respectively.
2) **Silica Fume:** Dried silica fume was used in concrete mixes with the ratio of 7% of cement weight. The physical properties such as fineness and specific gravity are (150,000 – 170,000) cm^2/gr and 2.2 respectively [16].
3) **Superplasticizer:** The super-plasticizing admixture was used only in 0.35 w/c ratio concrete mixes to satisfy the required workability, where the slump is between 105 and 125 mm. Its density and pH value are given as 1.03 gr/cm^3 and 7, respectively.

4) **Aggregates:** The coarse and fine aggregate used in concrete mixes were crushed limestone from the mountains of North Cyprus with a maximum diameter size of 20 mm. The aggregates were clean, free from organic materials and they were washed with water to remove dust and other particles. The grading was within the specific range of ASTM C33 and the aggregates were at the saturated surface dry condition. The relative density for coarse and fine aggregates were 2.68 gr/cm^3 and 2.65 gr/cm^3, and the water absorption capacity as percentage of dry mass were 1.35% and 2.52%, respectively.
5) **Water:** Tap drinkable water.

3.3 Specimens

In this study, the specimens were 150 mm cubes and (100x200) mm cylinders, casted in steel molds. For each parameter or purpose, three specimens were prepared and they were kept in a curing room for 24 hours, where the humidity was 90% and the temperature was 24 ± 1 C°. Then, the molds were stripped and the specimens were prepared and cured under two different environmental conditions until testing, as will be described and discussed in the following section. All specimens were tested at the age of 28-days. The mix proportions related with three different w/c ratios (0.35, 0.50 and 0.70), with and without silica fume are presented in Table 3.1.

Table 3.1. Ingredients and mix proportions of concrete specimens

Ingredients (kg/m^3)	W/C Ratio					
	0.35		0.50		0.70	
	Normal Concrete	Silica Fume Concrete	Normal Concrete	Silica Fume Concrete	Normal Concrete	Silica Fume Concrete
Cement	600	560	420	390	300	280
Coarse Aggregates	925	925	980	980	975	975
Fine Aggregates	640	640	770	770	900	900
Water	210	210	210	210	210	210
Silica Fume	-	42	-	30	-	21
Super plasticizer	3.5	3.5	-	-	-	-

3.3.1 Mixing and Preparation of Concrete Specimens

Building Research Establishment (BRE) for designing normal concrete was used in the design of all concrete mixes. Concrete ingredients were weighed individually and mixed into a horizontal mixer. First, coarse and fine aggregates were put into the mixer and within the first 30 seconds of mixing, the cement and silica fume were added, followed by the superplasticizer. All materials were mixed for approximately three minutes, until a homogeneous concrete mixture was achieved. The slump of the fresh concrete was measured directly after the mixing, according to ASTM C 143, and was kept constant among all samples, in the range of 105 mm to 125 mm. After the mixing process, the fresh concrete was casted in (150x150x150) mm cubes and (100x200) mm cylinder steel molds.

Figure 3.1. Mixing and slump test of fresh concrete

3.3.2 Curing Under Different Environmental Conditions

Following the standard 24-hour curing upon casting, the specimens were cured under two different environmental conditions, representative of the two main weather conditions in the city of Dohuk, Iraq. They are defined as follows:

1) Normal (moderate) weather condition: This environmental condition was obtained in regular laboratory setting, with humidity ranging from 10 to 25% and the temperature between 15 and 25 C°.
2) Hot weather condition: To obtain the hot weather condition requirements, an isolated room was designed specifically inside the laboratory, where the relative humidity ranged from 5 to 15%, with temperature between 40 and 50 C°. This room was checked daily to ensure the accuracy and consistency of these conditions.

After the removal of the molds, shrinkage pins were fixed on the three different surfaces of each cube to allow recording of changes in length during hydration process. For 7-days, the concrete specimens were cured three times a day with spray water until all faces became saturated, as suggested in ASTM C157. After a week, the samples were kept in open air for 21- days, until testing.

(a) (b)

Figure 3.2. Specimen photographs under (a) hot and (b) normal weather conditions

3.4 Experimental Procedure

In this study, total of 96 150 mm cubic specimens and 36 (100x200) mm cylindrical specimens were produced from six different concrete mixes, with and without mineral admixture, under two different environmental conditions, adding up to a total of twelve testing parameters.

The three different w/c ratio (0.35, 0.50, and 0.70) concretes were mixed and were considered as normal concrete (denoted by N). When a mineral admixture silica fume was added to the same proportion of mixes, silica fume concretes were obtained (denoted by SF). The amount of silica fume used was 7% of the cement by weight. For all 0.35 w/c ratio concretes, superplasticizer was added to achieve the required workability. The environmental conditions were normal and hot weather, denoted by Ne and He, respectively.

For each purpose of analysis, eight cubic and three cylindrical specimens were produced, where the first three cubic specimens were used for determining compressive strength and mass loss, the next three were used both for volume change measurements and stress-strain diagrams, and the final two were used for SEM analyses either before or after loading the specimens. The cylindrical samples were only used in determining the split tensile strengths. SEM photographs were obtained for all types of concretes produced.

The effect of three different w/c ratios, silica fume and two different exposure conditions were observed both before and after loading of the specimens. Normal concrete specimens were loaded up to 80% and silica fume enhanced specimens were loaded up to 85% of their ultimate compressive strengths. After unloading of the specimens, they were subjected to SEM analyses for crack detection.

Figure 3.3. Cubic and cylindrical specimens used in various experiments

3.5 Tests Carried Out

The effect of w/c ratio, silica fume and two different environmental conditions on hydration shrinkage cracks and whole fracture behavior of concrete were analyzed by interpreting the following:

- 28-days tensile (σ_t) and compressive strength (σ_c) for all mixes.
- Length and volume changes during 28-days hydration.
- Relationship between stress-strain diagrams and volume changes.
- Direct SEM observation of ITZ and matrix either before or after loading of the specimens for 12 different conditions.
- Correlation between tensile strength, volume change and SEM examination for all types of mixes and conditions.

3.5.1 List of the Specimens Used in this Study

In this section, a chart representing the general names of the specimens depending on three different w/c ratio, silica fume admixture and two different environmental conditions is drawn.

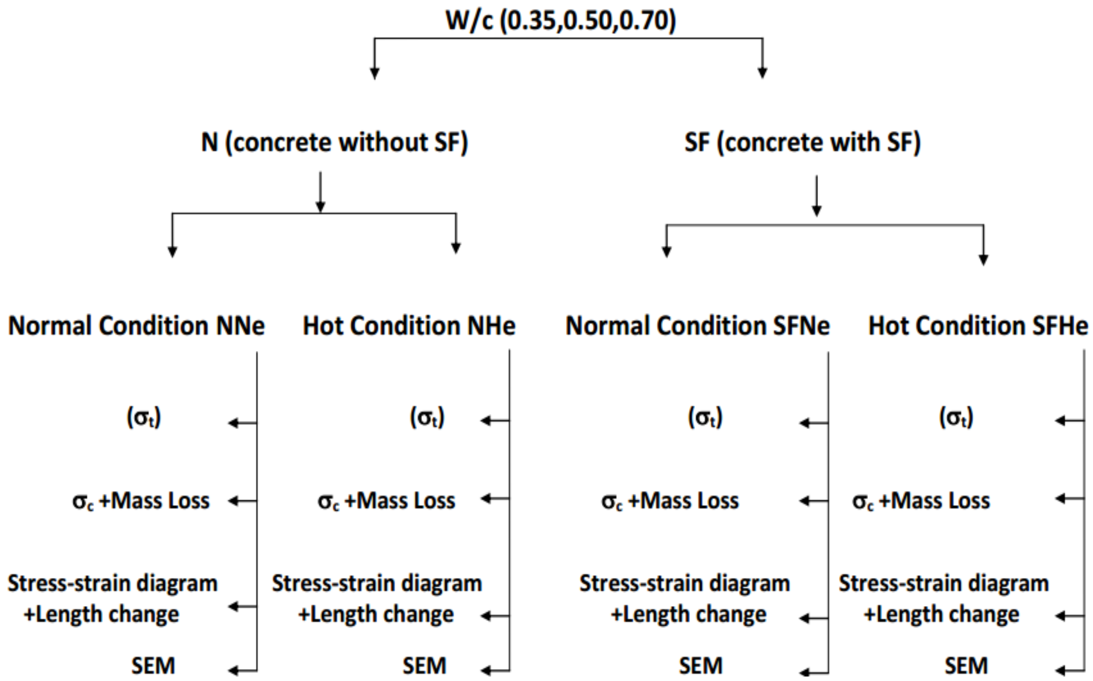

Figure 3.4. Names of the specimens depending on w/c ratio, silica fume and environmental conditions

According to the above given Figure 3.4, specimens can be named or abbreviated as listed on page xxii, under list of abbreviations.

N: Concrete without silica fume.

SF: Concrete with silica fume.

Ne: Normal environmental condition.

He: Hot environmental condition.

NNe: Concrete without silica fume under normal environmental condition.

NHe: Concrete without silica fume under hot environmental condition.

SFNe: Concrete with silica fume under normal environmental condition.

SFHe: Concrete with silica fume under hot environmental condition.

NNeL: Concrete without silica fume under normal environmental condition after loading.

NHeL: Concrete without silica fume under hot environmental condition after loading.

SFNeL: Concrete with silica fume under normal environmental condition after loading.

SFHeL: Concrete with silica fume under hot environmental condition after loading.

0.35 NNe: 0.35 w/c concrete without silica fume under normal environmental condition.

0.35 NHe: 0.35 w/c concrete without silica fume under hot environmental condition.

0.35 SFNe: 0.35 w/c concrete with silica fume under normal environmental condition.

0.35 SFHe: 0.35 w/c concrete with silica fume under hot environmental condition.

0.50 NNe: 0.50 w/c concrete without silica fume under normal environmental condition.

0.50 NHe: 0.50 w/c concrete without silica fume under hot environmental condition.

0.50 SFNe: 0.50 w/c concrete with silica fume under normal environmental condition.

0.50 SFHe: 0.50 w/c concrete with silica fume under hot environmental condition.

0.70 NNe: 0.70 w/c concrete without silica fume under normal environmental condition.

0.70 NHe: 0.70 w/c concrete without silica fume under hot environmental condition.

0.70 SFNe: 0.70 w/c concrete with silica fume under normal environmental condition.

0.70 SFHe: 0.70 w/c concrete with silica fume under hot environmental condition.

3.6 Techniques of Observation

In this work, various techniques were used in studying the effect of hydration shrinkage crack on the microcracking behavior of concrete including w/c ratio, silica fume and environmental conditions. One way is the direct measurement by means of optical processing on cut surfaces through SEM. On the other hand the indirect measurements consist of an overall study of the material by means of tensile and compressive strength measurements, the length and the volume change measurements and the prediction of critical crack load (σ_{cr}) from stress-strain diagrams.

3.6.1 Direct Observation of Microcracks

Recent developments on different microscopic engineering studies also gave chance to direct observation for determining the exact nature of cracks due to stress and strain (i.e. to what extent cracks may occur in the ITZ or the matrix even before loading the material). This gives precise information pertinent to the mechanism of cracking. Therefore, the study of microcrack network in concrete both before and after loading up to a certain stress level (i.e. up to σ_{cr}) be helpful, not only in understanding the fracture process but also in improving its behavior.

In order to characterize the microcrack network, two kinds of data are required. The first one is the morphological data (path of the microcracks related to the ITZ and the matrix) and the other one is the topographical data (length, density and orientation of the microcracks).

In this study, the location of the examined cross section including both the ITZ and the matrix under microscope is shown in Figures 3.5 to 3.7. To be able to map the crack using photography techniques, specimens had been sectioned transversely and longitudinally using a diamond saw until a small section of (30x30x10) mm, suitable for SEM analyses were obtained. For non-loaded specimens, this sample was taken perpendicular to the casting y-direction (Figure 3.5) whereas for loaded specimens (Figure 3.6), the examined surface was perpendicular to the loading x-direction. The microcrack density was measured by dividing the total microcrack area by the total matrix area.

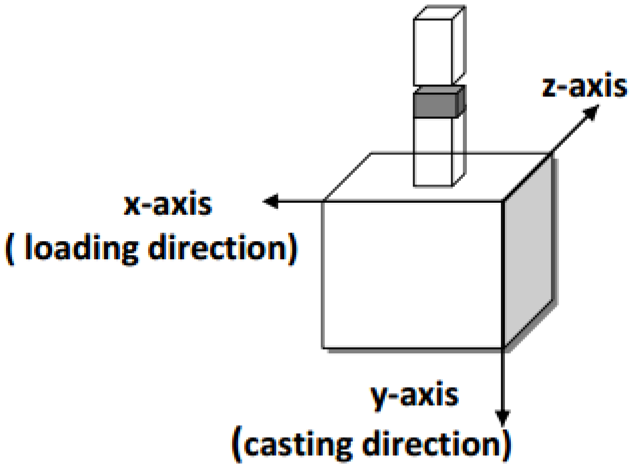

Figure 3.5. Schematic representation of the cubic specimen and the examined cross-section before loading the specimen

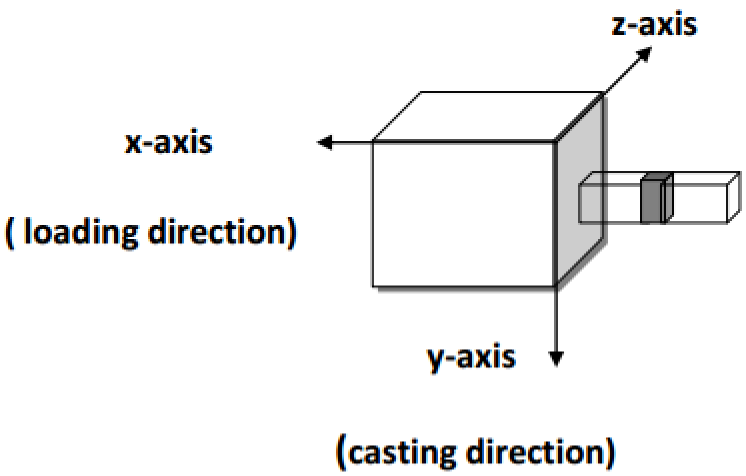

Figure 3.6. Schematic representation of the cubic specimen and the examined cross-section after loading the specimen

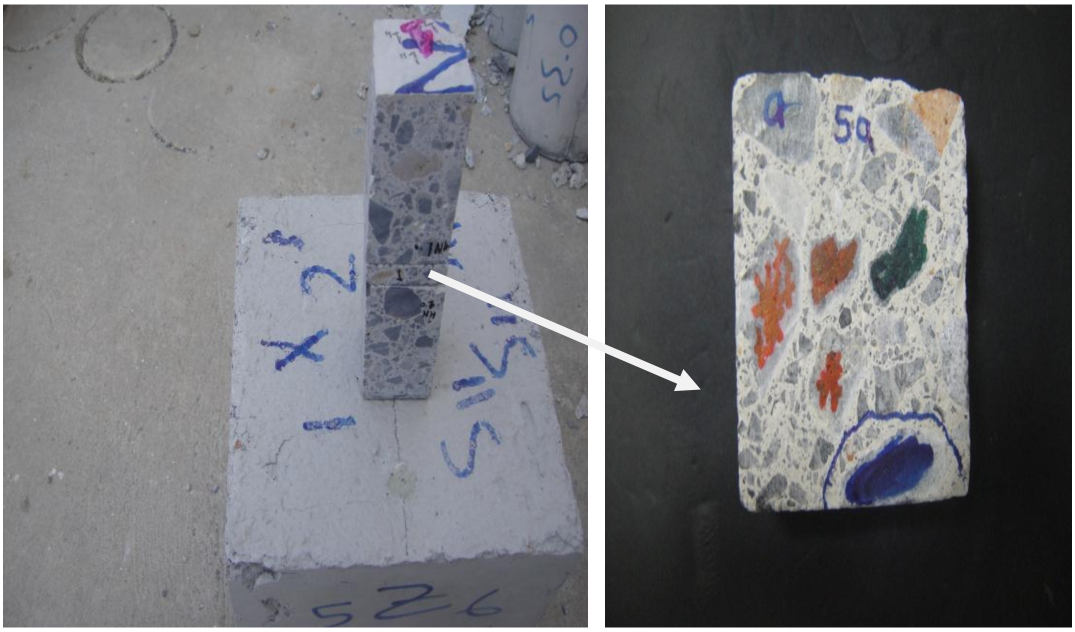
Figure 3.7. Photograph of the examined cross-section under SEM showing the colored aggregates with ink

For loaded specimens, the examined cross-section in SEM is the surface area of the section perpendicular to the loading direction obtained from the core of the sample. The average microcrack density was measured from the microcrack photographs obtained from the ITZ and the matrix. In each sample, circumferences of uniform size aggregates were selected. Aggregates were painted dark as shown in Figure 3.7 in order to clearly identify the aggregate, ITZ and matrix boundaries. In most of the cross-sections obtained from non-loaded specimens, the structural difference between ITZ and the matrix was very clear. For those samples, both the ITZ thickness and crack density had been measured easily, along with matrix crack density. This process was repeated for all types of concretes and conditions.

The crack size and density information obtained from these tests are very valuable in understanding the influence of w/c ratio, environmental conditions and mechanical loading on the nature of crack growth both in the ITZ and the matrix. But the major disadvantage of sectioning is that, it does not allow the continuous monitoring of crack growth during loading. Furthermore, cracks may close when unloading, and new cracks may be introduced during sawing. Non-destructive techniques that have been used to monitor crack growth in concrete provide an average measure of crack density rather than information on individual cracks. Such information would only be sufficient for determining the whole damage occurred in concrete. Then the combination of data obtained either at the micro or the macro scale of concrete can lead to a better understanding of the behavior of concrete under load.

3.6.2 Indirect Detection of Microcracking

Besides the direct observation of cracks under microscope, several indirect methods are available and were used to understand the crack initiation and propagation in concrete.

3.6.2.1 Compressive and Tensile Strength Tests at 28-days

Compressive (σ_c) and tensile (σ_t) strength values have been obtained for all mixes and environmental conditions. 150 mm cubic specimens were used for compressive strength measurements and (100x200) mm cylindirical specimens were used for tensile strength measurements. The loading rate was 30 ± 2 MPa/minute. The average of three samples had been taken for each measurement if the sample values were not too far values from each other. The findings are tabulated in Table 4.1.

Figure 3.8. Compressive and tensile testing, respectively

3.6.2.2 Length and Volume Change Measurements

In this part of the experiment, the aim was to get an idea on initial cracks due to hydration shrinkage. Recording the length changes in all three directions led us to calculate the amount of volume change due to hydration. For this purpose, fixing pins were placed in three different directions of the specimens and length changes were recorded with the passing time, using dial gauges. Within the first 3-days length changes along x-y-z directions were measured three times a day due to higher rate of hydration within the first 3-days. The following three days, the recording was reduced to twice a day, and then dropped to only once a day.

Figure 3.9. Length change measurement with time by using shrinkage pins placed in three directions

3.6.2.3 Stress-Strain Measurements

The complete fracture characteristics of a specimen may be investigated in a stable, displacement-controlled experiment only. In this study, specimens were loaded so as to maintain a constant rate of increase of the measured elongation. For the stress-longitudinal strain measurements, specimens were tested at a constant deformation rate of 0.05 mm/minute. After obtaining the stress-strain diagrams, area under each curve had been calculated to enable fracture resistance calculation for each sample. For each specimen, the critical stress (σ_{cr}) was recorded by using the slopes of stress-strain diagrams. In each curve, the sharp return point towards horizontal direction is named as the critical point. The critical load for the normal concrete and the silica fume concrete was recorded as 80% and 85% of the σ_c, respectively.

Figure 3.10. Determination of stress-strain diagrams subjected to compressive stress

3.6.2.4 Workability and Mass Loss Calculations

In this study, the weight of each sample was taken once a week, and then the difference between the initial weight and weight of samples after each week was determined and plotted with time.

The slump of the fresh concrete was measured directly after the mixing of the concrete according to ASTM C 143-12. In this procedure, the cone was filled by three layers of concrete and compacted using a standard steel bar, and then the difference between the height of the cone and the height of the standing concrete mass was identified, hence giving the slump of the concrete.

Chapter 4

RESULTS AND DISCUSSION

4.1 Introduction

In this chapter; the outcomes, values and the results of all the previously described experiments will be plotted and be presented in charts, figures and tables, followed by their analysis and discussions. The experiments were conducted for total of twelve mixes with three different w/c ratios, with or without 7% silica fume under two different environmental conditions. These samples were tested for compressive strength, split tensile strength, stress-strain relationship, length change in x, y and z directions, volume change, mass loss with time and density, and finally by using SEM, crack densities of ITZ and matrix were calculated.

4.2 Effect of Matrix Quality and Environmental Conditions on Compressive and Tensile Strength

Effects of three different w/c ratios, silica fume inclusion and two different environmental conditions both on tensile strength (σ_t) and compressive strength (σ_c) were measured. The results are tabulated in Table 4.1. Each value represents the average of three specimens per trial.

Table 4.1. Effects of matrix quality and environmental conditions on 28-days compressive and tensile strength

		Hot Weather Conditions (He)				Normal Weather Conditions (Ne)			
		Split Tensile Strength-σ_t (MPa)		Compressive Strength-σ_c (MPa)		Split Tensile Strength-σ_t (MPa)		Compressive Strength-σ_c (MPa)	
W/C = 0.35	Normal Concrete (N)	3.62		57.40		3.69		57.20	
		3.95	3.84	56.80	57.70	3.76	3.79	59.30	58.33
		3.96		58.90		3.92		58.50	
	Silica Fume Concrete (SF)	4.41		59.40		4.16		61.40	
		4.09	4.23	58.90	60.53	4.13	4.05	71.40	65.90
		4.20		63.30		3.95		64.90	
W/C = 0.50	Normal Concrete (N)	3.16		36.20		2.90		35.00	
		3.05	3.11	35.90	36.30	3.07	3.03	34.70	34.93
		3.11		36.80		3.12		35.10	
	Silica Fume Concrete (SF)	4.32		37.00		3.31		35.50	
		3.32	3.64	37.80	37.80	3.60	3.62	35.20	35.67
		3.27		38.60		3.95		36.30	
W/C = 0.70	Normal Concrete (N)	2.75		33.60		2.08		23.80	
		2.79	2.80	33.40	33.67	2.30	2.25	24.80	24.23
		2.86		34.00		2.37		24.10	
	Silica Fume Concrete (SF)	3.13		34.80		2.60		25.50	
		3.11	3.04	35.60	35.47	3.15	2.77	25.70	25.40
		2.89		36.00		2.55		25.00	

It is important to know the tensile strength of materials because it represents a materials weaknesses. In other words, weak points in a material like the ITZ are known to affect the tensile strength and microcracking behavior of concrete in varying degrees. Also, as concluded by many researchers, although these weak points are influential on the tensile strength, they only have a little effect on the compressive strength.

When the compressive strength is concerned, the influence of three different w/c ratios, silica fume and two environmental conditions are represented in Figure 4.1. From this figure, the following conclusions can be made:

i) Under both environmental conditions, silica fume inclusion has no considerable effect on compressive strength at w/c ratios higher than 0.35, even then there is only a slight difference in σ_c within high w/c ratio mixes.

ii) Hot weather conditions led to a significantly higher compressive strength for both normal and silica fume concretes only at 0.70 w/c ratio, and less so for 0.50 w/c concrete. Therefore, it can be said that, the w/c ratio is the most influential parameter on compressive strength, where, as w/c ratio increase, compressive strength decrease.

iii) At low w/c ratios (0.35 and 0.50), humidity and temperature were found to have no considerable effect on compressive strength values.

The specimens where extreme points for compressive strength are observed are 0.35 SFNe for the highest and 0.70 NNe for the lowest.

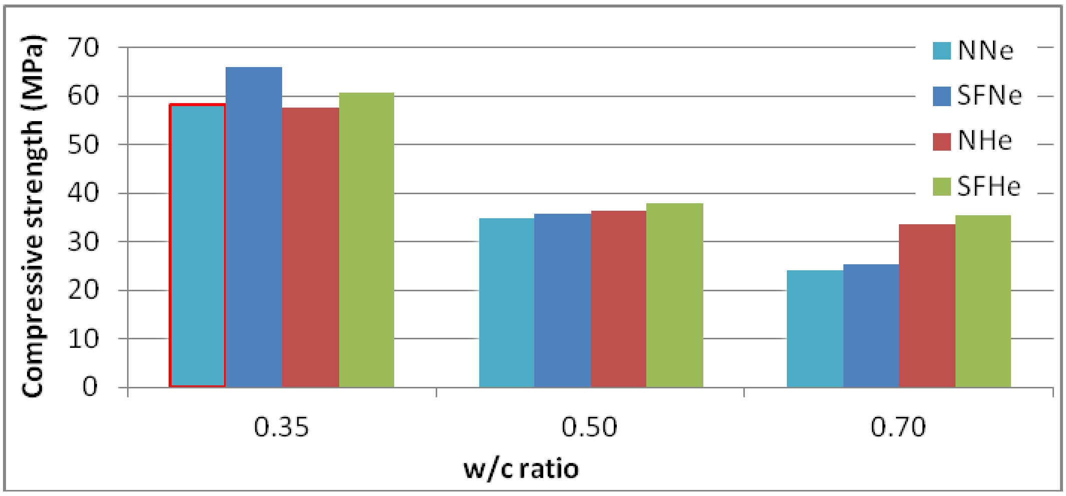

Figure 4.1. Effect of matrix quality and environmental conditions on 28-days compressive strength

Tensile strength values are represented in Figure 4.2 for all w/c ratios at two different environmental conditions, with or without silica fume and the conclusions to be drawn from this figure is as follows:

i) Different from its null effect on compressive strength, silica fume has found to increase the tensile strength at all w/c ratios under both environmental conditions.

ii) At low w/c ratios (0.35 and 0.50), environmental conditions are not influential, however at 0.70 w/c ratio, which is considered as high w/c ratio, tensile strength increased with increased humidity and temperature.

iii) When w/c ratio is considered, a linear increase is observed with decreasing ratio.

Among all specimens, 0.35 SFHe yielded the highest tensile strength and 0.70 NNe yielded the lowest, indicating that these two specimens are the critical specimens for both compressive and tensile strength tests.

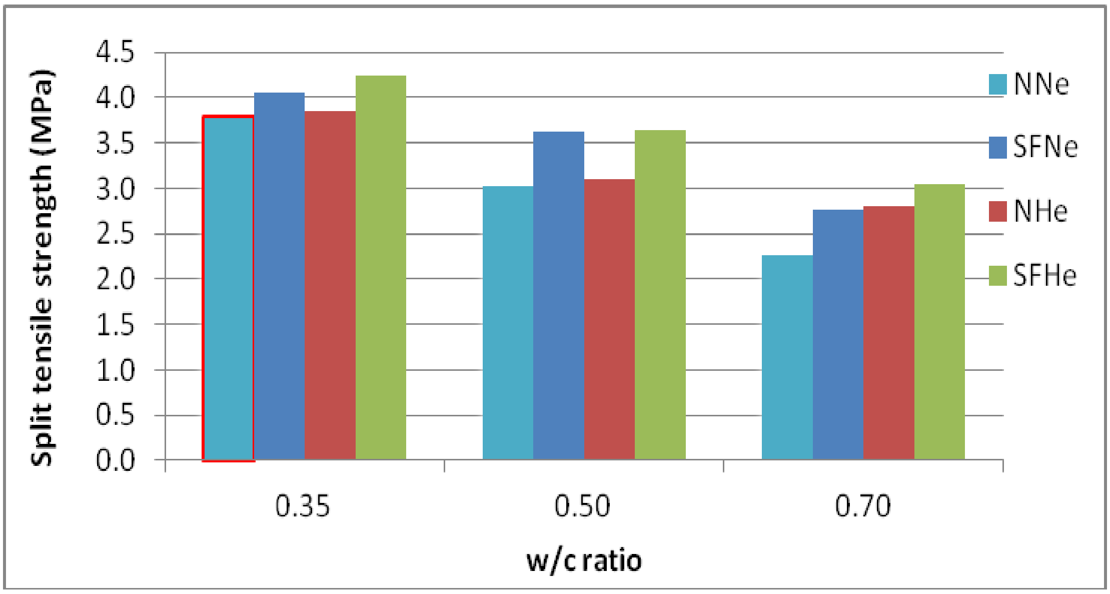

Figure 4.2. Effect of matrix quality and environmental conditions on 28-days split tensile strength

4.3 Effect of Matrix Quality and Environmental Conditions on Volume Change with Time

Volume change with respect to time for all samples is in decreasing manner and this is named as decrement. Under this criteria the decrement of the samples volume were compared accordingly for example after 5-days of curing the amount of decrement became less for a short time which is defined as swilling. Effect of w/c ratio, silica fume and different environments on volume change with time can be examined from Figures 4.3 to 4.8. In these figures, change (decrement) in volume with time or total change in volume for 3, 7 and 28- days are showed separately.

In Figure 4.3, the total change in volume for all types of specimens in 7- days can be observed. Based on this figure, it can be concluded that, decrease in volume is higher with silica fume inclusion. The difference is lowest in 0.50 and the highest in 0.70 w/c ratio mixes. An exception to this trend was seen at 0.35 SFHe, and with addition of silica fume, the volume change duuuuuuuecreased.

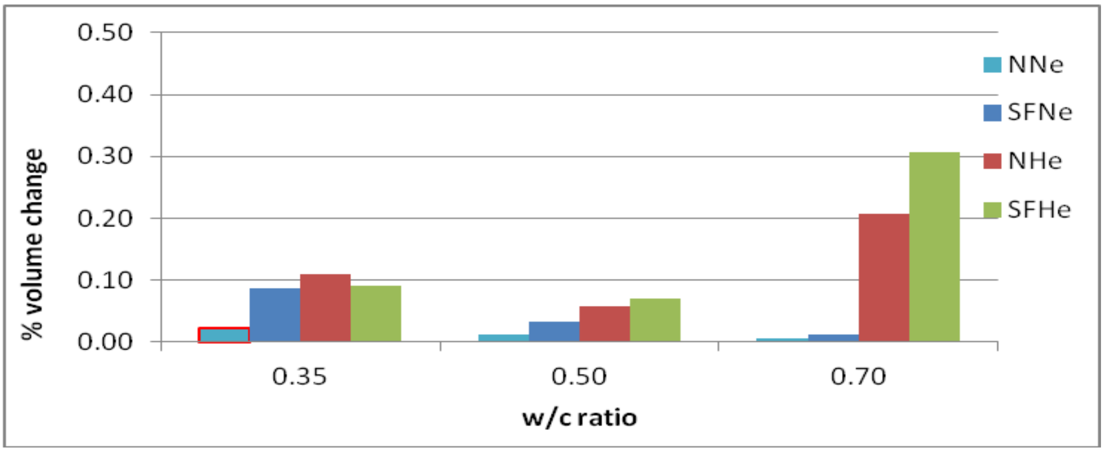

Figure 4.3. Effect of matrix quality and environmental conditions on 7-days volume change

According to the figure there is no uniform correlation between w/c ratio and volume change. However, higher volume change was observed for 0.70 w/c ratio under hot environmental condition (0.70 NHe and 0.70 SFHe) both in normal and silica fume concretes and the lowest value occurred in the same w/c ratio under normal environmental conditions (0.70 NNe and 0.70 SFNe).

28-days change in volume is represented in Figure 4.4. Similar to 7-days patterns, silica fume was found to increase volume change in all w/c ratios with the exception of 0.35 SFHe specimens. Additionally, no direct dependence on volume change due to w/c ratio was found, and in general, both the silica fume inclusion and hot environment increases the volume change (decrement). This is due to the high fineness of silica fume and rapid evaporation of water due to high environmental temperature. These factors are responsible for high volume decrement in concrete specimens. When the environmental effects are analyzed, under hot conditions, higher volume decrement occurs for all types of mixtures. As a summary, maximum volume decrement took place in 0.70 SFHe and the minimum change occurred in 0.70 NNe specimens. Volume decrement become critical at high w/c ratio mixes under hot environment, regardless of inclusion of silica fume.

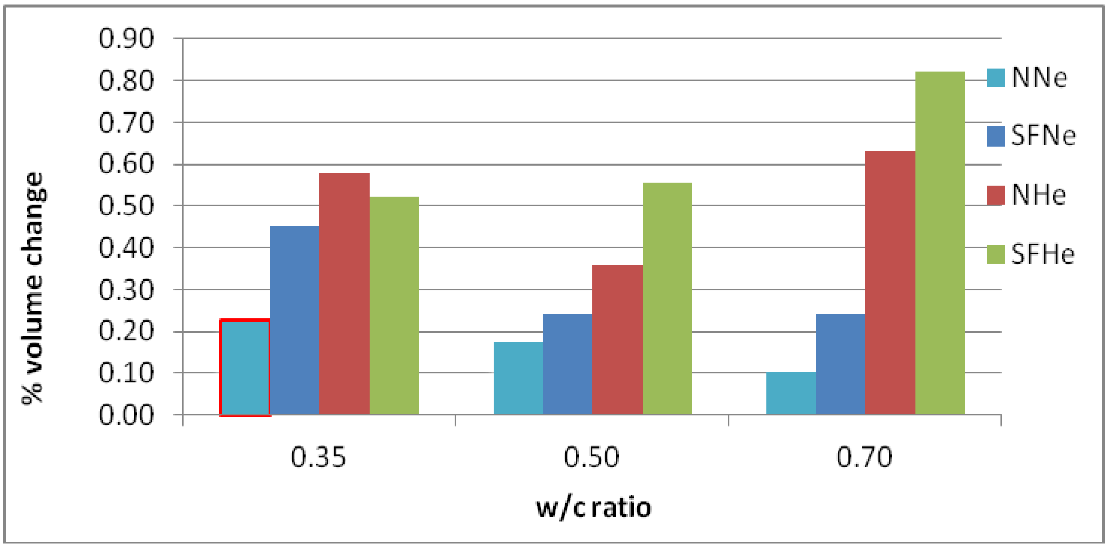

Figure 4.4. Effect of matrix quality and environmental conditions on 28-days volume change

As a conclusion, it can be inferred that, hot environments have a negative effect on volume change (decrement) and it is more pronounced at high w/c mixes (low matrix quality). In general, volume change (decrement) increases with silica fume inclusion except for 0.35 NHe and 0.35 SFHe specimens, where volume change decreased with silica fume inclusion. This can be defined as; negative effect of hot environment has been compensated by silica fume inclusion at low w/c ratio mixes. This also can be due to superplasticizer effect that was used only for 0.35 w/c ratios.

3-days volume changes (decrement) ratio are represented in Figures 4.7 and 4.8, and for 28-days, in Figures 4.5 and 4.6. It is seen that, the maximum volume change ratio occurred in 0.70 SFHe specimens in 72 hours. If we look at the total volume changes (decrement) ratio relative to the environmental temperatures and humidity, there is a huge difference in volume decrements between normal (Ne) and hot (He) environmental conditions. Under hot environment, volume decrement ratios are about four times more than the volume decrement ratios taking place under normal environmental conditions.

At early ages, rapid rate of hydration results in rapid rate of water loss and therefore volume decrement is significantly higher compared to later ages. When early and late ages are compared it can be seen that, the volume change (decrement) ratio at late ages caused by hot environment is not as effective as the early ages, although it still exists. Moreover, w/c ratio and silica fume effects also become less significant at later ages. However, one constant effect on volume decrement ratio is time, where with passing time, volume change (decrement) ratio increases. This rate of decrement increases further with inclusion of silica fume, and increasing temperature and humidity. Additionally, the rate of volume change decreases with decreasing w/c ratio.

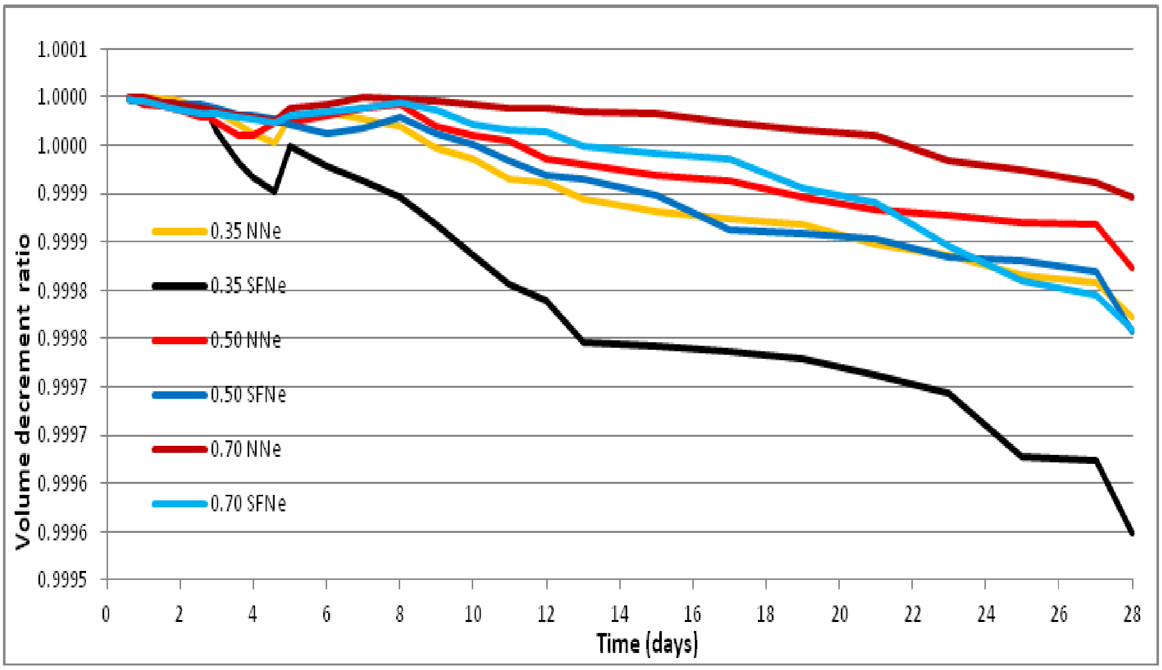

Figure 4.5. Effect of water cement ratio and silica fume on volume change under normal environmental conditions

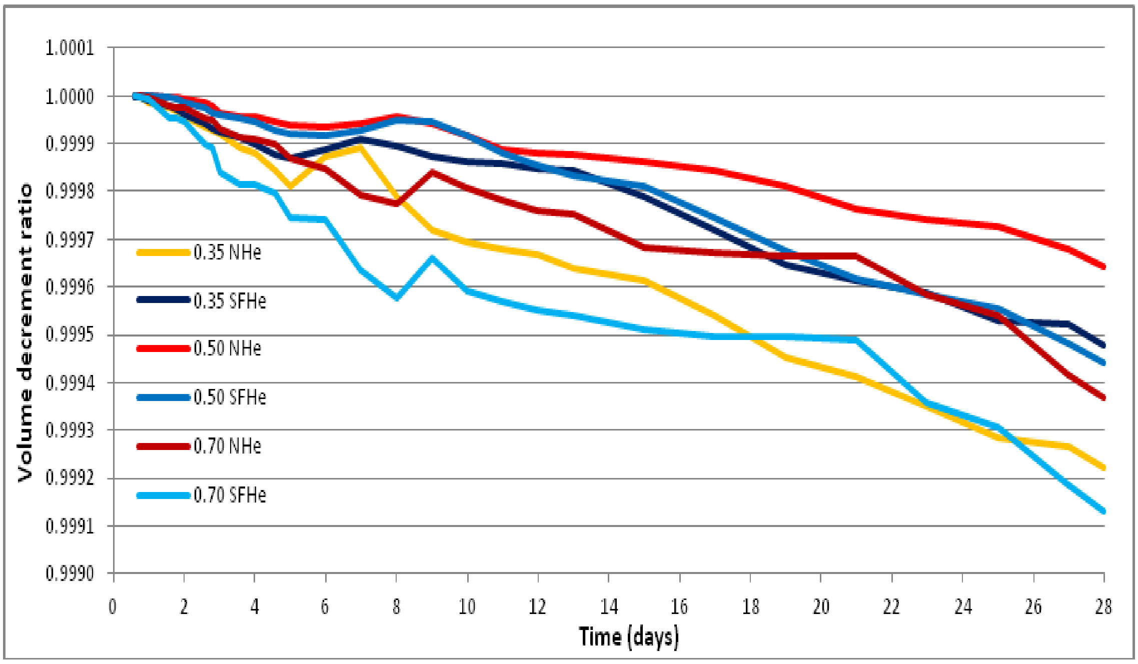

Figure 4.6. Effect of water cement ratio and silica fume on volume change under hot environmental conditions

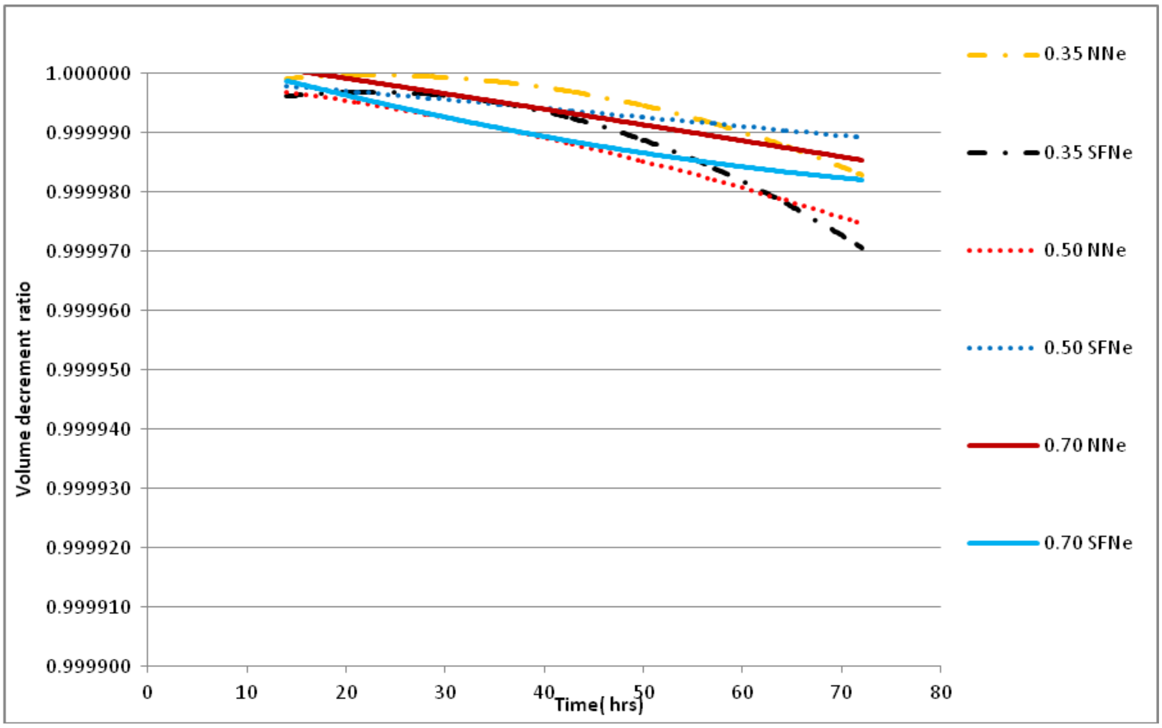

Figure 4.7. Effect of water cement ratio and silica fume on volume change under normal environmental conditions

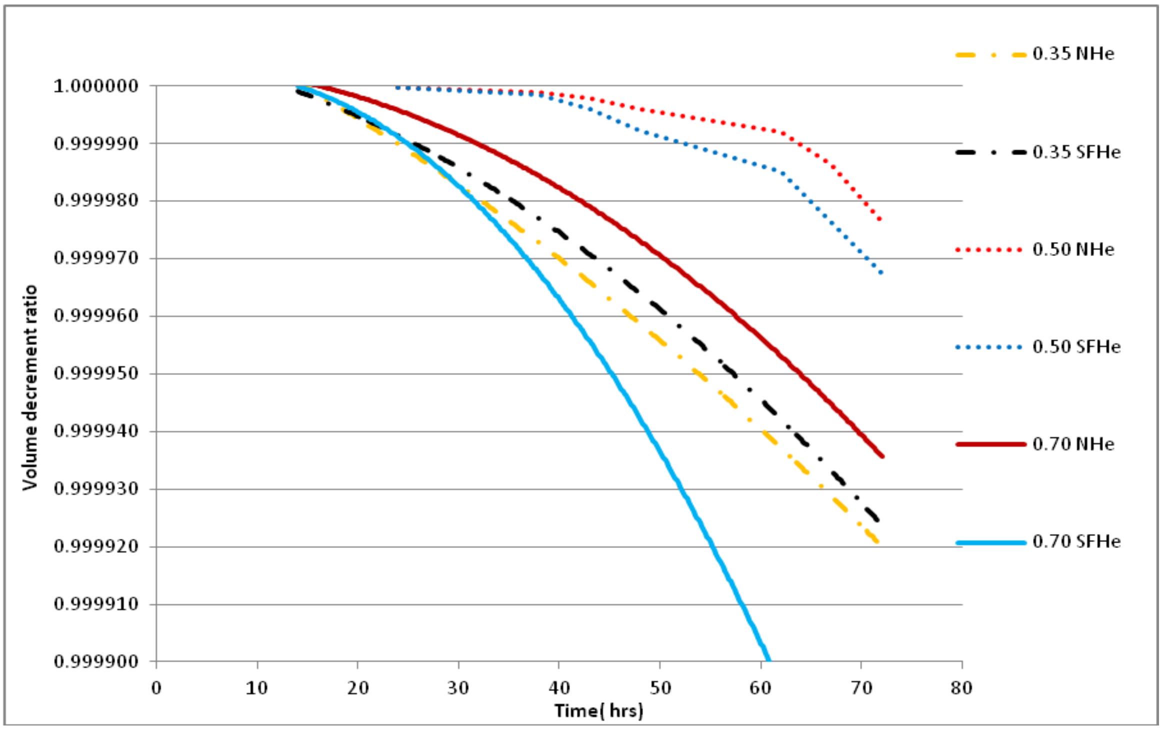

Figure 4.8. Effect of water cement ratio and silica fume on volume change under hot environmental conditions

4.4 Effect of Matrix Quality and Environmental Conditions on Length Change with Time

Figures 4.9, 4.10 and 4.11 represent the length changes along x, y and z directions for all specimens and two different environmental conditions over time. Although the volume changes discussed above are based on the changes in length, it might be useful to analyze the length changes separately to be able to examine the effect of casting direction on drying shrinkage. It is observed that, in all cases, silica fume inclusion increased length change (decrement), except for 0.70 SFHe in x and z directions. At medium w/c ratio (0.50), neither silica fume inclusion nor hot environment is effective on length change. A similar situation is observed between 0.70 NHe and 0.70 SFHe mixes. In general, there is no significant effect of w/c ratio on length changes in all types of mixes and environmental conditions.

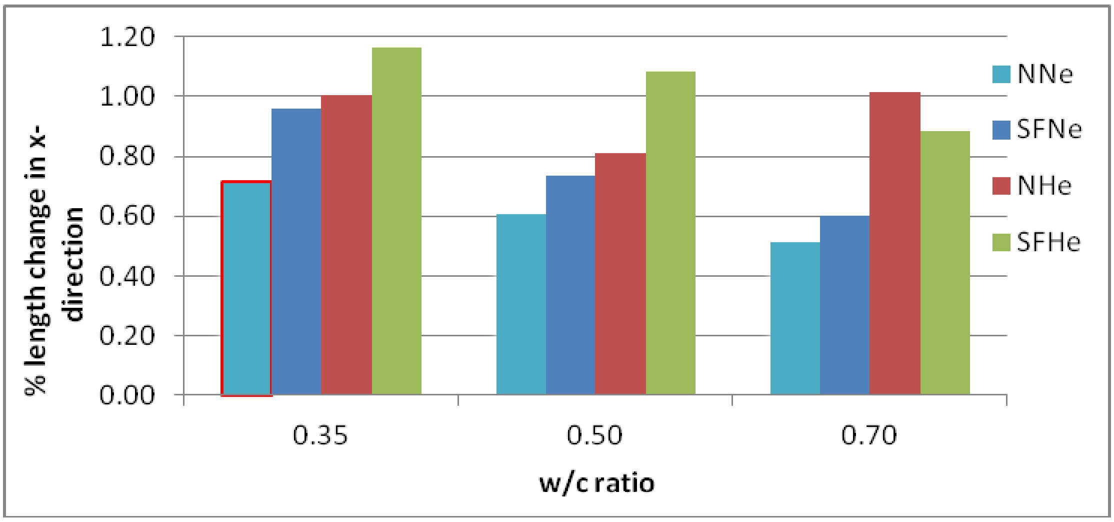

Figure 4.9. Effect of matrix quality and environmental conditions on 28-days length change in x-direction

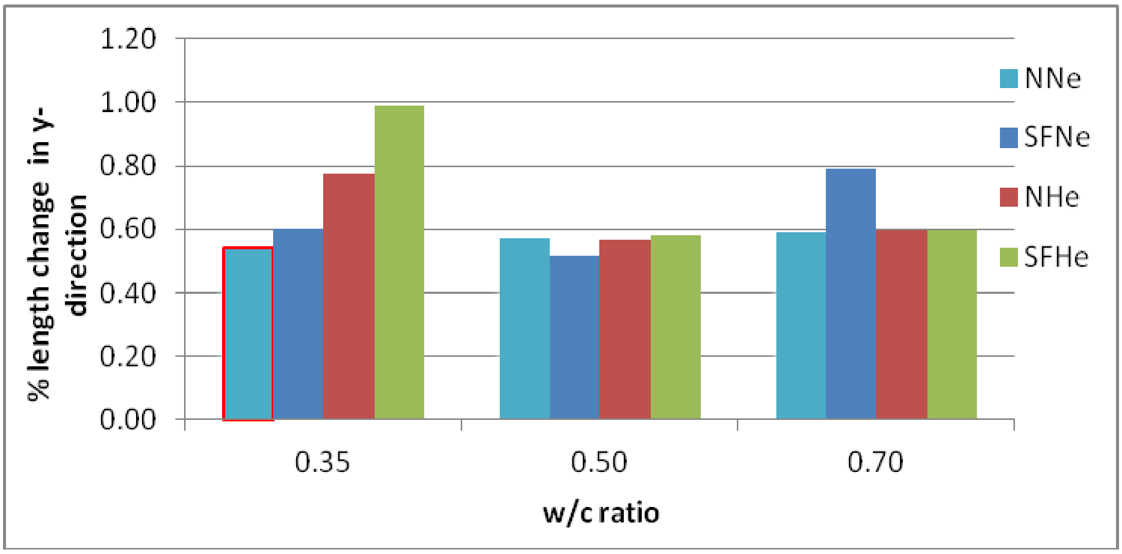

Figure 4.10. Effect of matrix quality and environmental conditions on 28-days length change in y-direction

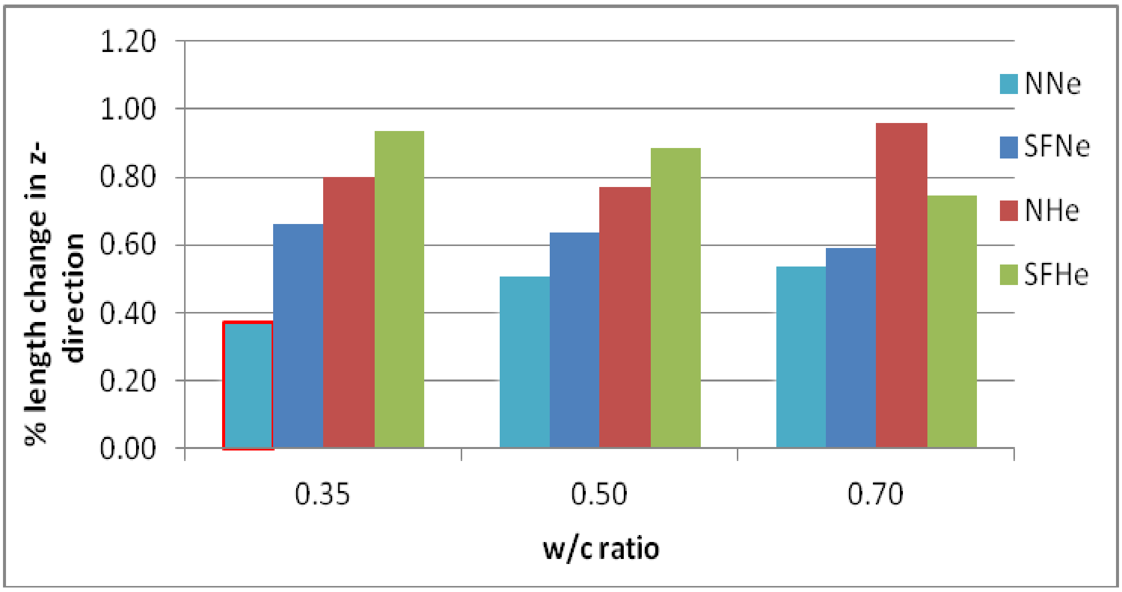

Figure 4.11. Effect of matrix quality and environmental conditions on 28-days length change in z-direction

4.5 Effect of Matrix Quality and Environmental Conditions on Mass Loss with Time

The decrease in the mass of concrete over time due to water consumption by hydration process or evaporation of water is known as mass loss. Figures 4.12 and 4.13 show this relationship, and from these curves it is seen that mass loss increases over time with increasing w/c ratio under normal environmental conditions and the rate of loss also increases with passing time. However this cannot be observed clearly for hot environments, especially at later ages. Moreover, for the first 7-days, mass loss under hot environmental conditions is higher than the normal one for all w/c ratio concretes. This pattern has continued for all ages for 0.35 mixes, but for other ratios this has changed after a week.

Furthermore concrete with silica fume under normal conditions exhibit less weight loss than normal concrete for high and medium matrix quality due to improving of concrete microstructure by its higher surface area which leads to reducing voids which hold extra water. Moreover, there is a rapid increase in the rate of mass loss after 7-days of curing until 14-days due to an increase in the rate of water loss in concrete once curing is stopped.

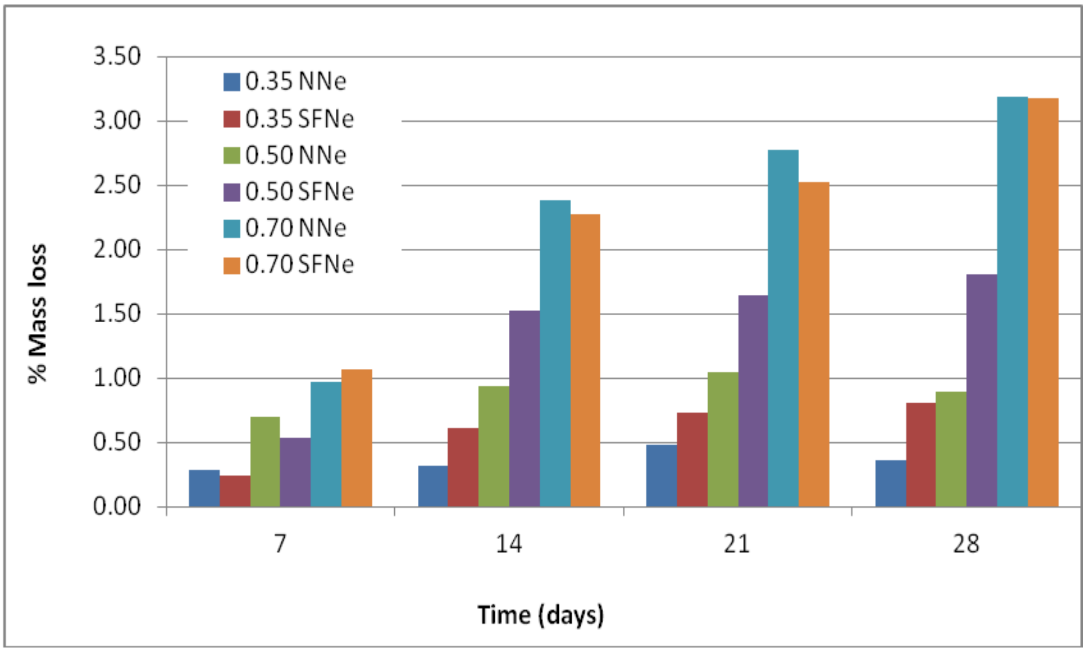

Figure 4.12. Effect of water cement ratio and silica fume on mass loss under normal environmental conditions

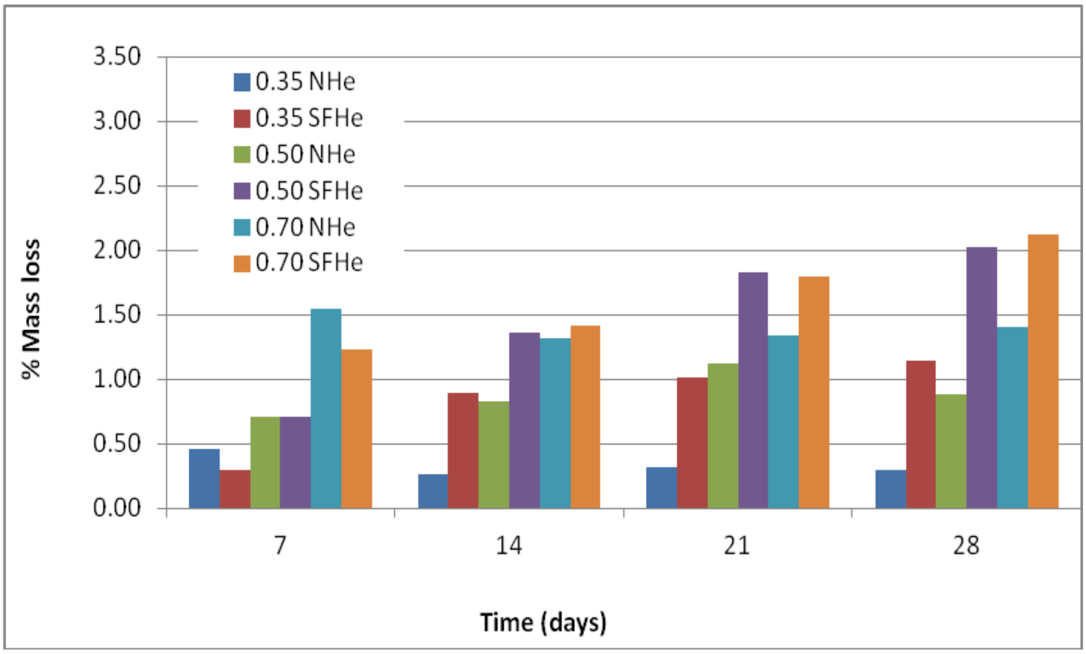

Figure 4.13. Effect of water cement ratio and silica fume on mass loss under hot environmental conditions

4.6 Effect of Matrix Quality and Environmental Conditions on Stress-Strain Diagrams

The microcracking behavior of concrete under uniaxial compressive loading can be sectioned and examined from the stress-strain diagrams obtained experimentally. Crack initiation, growth and propagation happen at different stress levels of loading, and results in different slopes and sections on these diagrams. So, following and sectioning these diagrams, it is possible to indirectly detect the critical stress levels depending on varying parameters (w/c ratio, environmental conditions and silica fume inclusion).

The stages that can be followed while examining the stress strain diagrams can be summarized as follows which has been thoroughly experimented and concluded by many researchers:

1) Up to 30% of σ_c loading: microcracks are stable and it represents the beginning of localized cracking.
2) Between 30% and 50% of σ_c loading: interfacial (bond) cracks begin to expand due to stress concentration at the crack tips. Matrix cracks are non-existent or insignificant. Crack propagation is steady for the reason that internal energy is balanced by the crack release energy. Bond cracks start to expand due to stress concentration at the crack tips.
3) Between 50% and 75% of σ_c loading: Accessible interfacial cracks extend in the shape of matrix cracks and new interfacial cracks go on to form.
4) For more than 75% of σ_c loading: the largest cracks reach their significant lengths. At this stage the available internal energy is bigger than the necessary crack release energy. The rate of crack spread increases and the

system is unbalanced. Hence, 75% of σ_c loading is called the beginning of unbalanced fracture propagation or critical stress.

In this study, the effect of silica fume, water-cement ratios and two environmental conditions on stress-strain diagrams are shown in Figures 4.14 and 4.15. According to these figures, the point where linearity ends is considered as the end of the second stage. In general, under hot environments, linearity ends earlier for all specimens compared to the normal environment. That means, under high temperature and humidity, crack initiation and growth take place earlier.

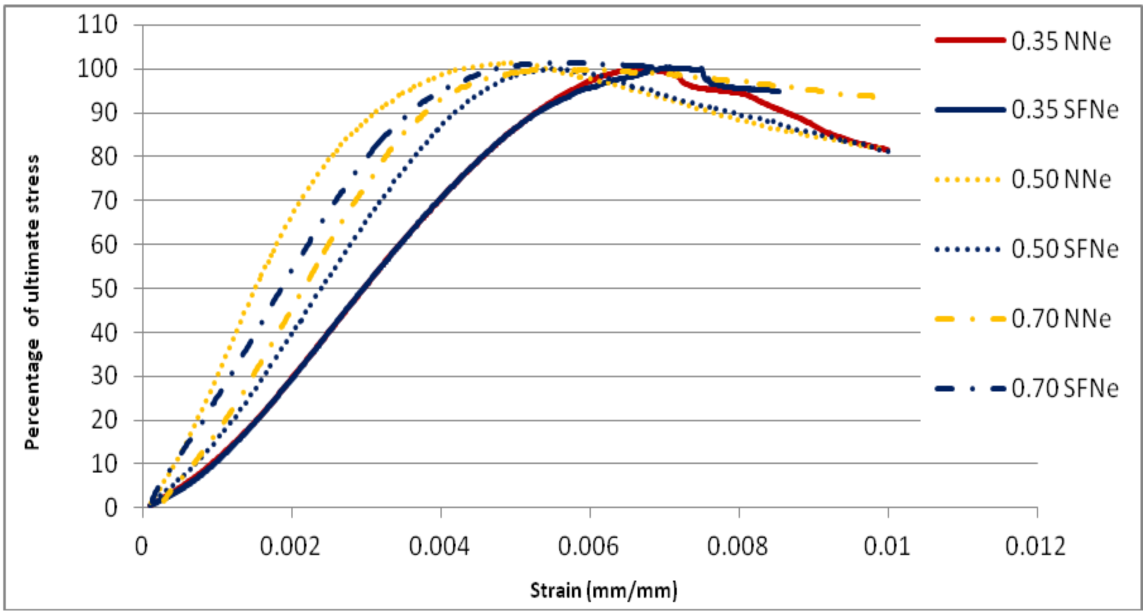

Figure 4.14. Effect of water cement ratio and silica fume on stress-strain relationship under normal environmental conditions

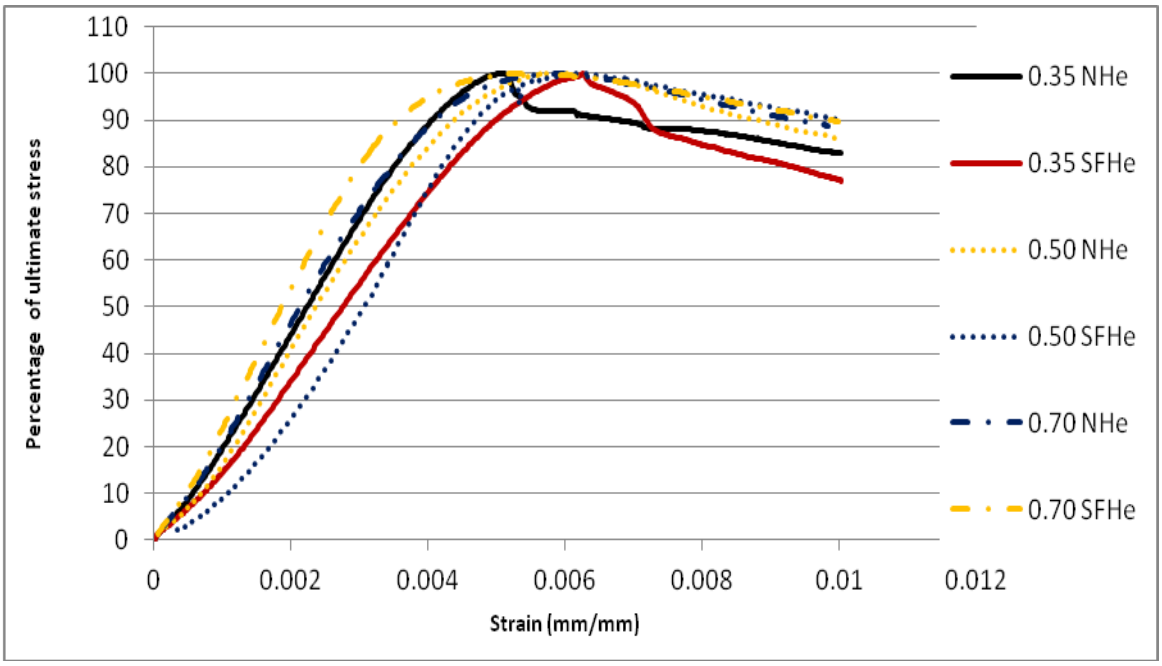

Figure 4.15. Effect of water cement ratio and silica fume on stress-strain relationship under hot environmental conditions

4.7 Direct Detection of Microcracks

In this experimental study, direct detection of microcracks either within the ITZ or the matrix before and after loading for different w/c ratio concretes had been done by means of Scanning Electron Microscope (SEM) analyses. Effects of silica fume and two different environmental conditions were analyzed . SEM analyses can be summarized as follows. Small size samples (30x30x10) mm prepared for SEM were cleaned in water using fine grained sand paper to get a good appearance for SEM photo. The selected aggregates for analyzing were located in the middle face of samples to minimize the effect of cutting on cracks evaluations and types in range (10-12) mm size and they were colored by soft pencil with keeping a distance to edges of aggregate. The magnification of photographs is different starting from 300 to indicate the matrix, aggregate and ITZ thickness and 500 for details of matrix and ITZ. The results of 12 different mixes are shown in Table 4.2.

Table 4.2. Effect of matrix quality and environmental conditions on ITZ thickness and crack density

	Specimen name	ITZ thickness (μm)	ITZ crack density (%)	Matrix crack density (%)		Total crack density in matrix (%)	Total (ITZ+Matrix) crack density (%)
				1.5 mm	1 mm		
w/c=0.35	NNe	52.00	0.62	1.28	0.20	1.48	2.10
	NHe	54.00	0.91	0.82	1.00	1.82	2.73
	SFNe	45.00	0.78	0.67	0.35	1.02	1.80
	SFHe	57.00	1.04	0.86	0.41	1.27	2.31
	NNeL	39.00	1.53	1.31	1.08	2.39	3.92
	NHeL	77.00	1.61	1.24	0.76	2.00	3.61
	SFNeL	58.00	1.79	1.55	1.24	2.79	4.58
	SFHeL	57.00	1.00	1.70	0.54	2.24	3.24
w/c=0.50	NNe	53.00	0.81	1.29	0.35	1.64	2.45
	NHe	61.00	0.77	1.06	0.60	1.66	2.43
	SFNe	47.00	0.36	0.78	0.28	1.05	1.41
	SFHe	67.00	1.47	0.67	0.72	1.39	2.86
	NNeL	79.00	0.98	1.48	0.92	2.40	3.38
	NHeL	92.00	2.30	1.23	1.20	2.43	4.73
	SFNeL	108.00	1.70	1.80	1.06	2.86	4.56
	SFHeL	93.00	0.93	1.77	0.50	2.27	3.20
w/c=0.70	NNe	120.00	1.20	1.48	1.11	2.59	3.79
	NHe	113.00	1.04	1.06	0.50	1.56	2.60
	SFNe	60.00	0.72	1.28	0.51	1.79	2.51
	SFHe	77.00	0.70	1.00	0.60	1.60	2.30
	NNeL	178.00	2.03	2.92	1.80	4.72	6.75
	NHeL	124.00	1.00	1.44	1.11	2.55	3.55
	SFNeL	122.00	1.58	3.08	2.45	5.53	7.11
	SFHeL	124.00	1.13	1.94	1.12	3.06	4.19

4.7.1 Effect of Matrix Quality and Environmental Conditions on ITZ Thickness

The effect of w/c ratio and the environmental conditions with and without silica fume prior and after loading is represented in Figures 4.16 and 4.17. From Figure 4.16, it can be observed that, at low w/c ratios (0.35 and 0.50), there is only a slight difference (increment) in ITZ thickness when the silica fume free samples are (NHe) subjected to hot environment. The main factor affecting the ITZ thickness is seen as the w/c ratio, where at 0.70 ratios, the thickness is almost twice as high when compared to other w/c ratios in normal concrete. Similar analysis applies for silica fume concrete samples without loading. However, the difference between w/c ratios on both environmental conditions is relatively low when compared to silica fume free samples, and there is a uniform change in ITZ thickness with increasing w/c ratio. When the two environmental conditions are compared, the thickness is found to increase in higher temperature and humidity conditions, different from the N samples. In SF samples, under normal conditions, the ITZ thickness fluctuates between 45-60 micrometer and under hot conditions these values increase to 58-78 micrometer.

An overall look at samples before loading materials indicate that, the addition of silica fume into concrete mixes creates approximately 35 micrometers decrease in ITZ thickness at 0.70 w/c ratios, but there was no such significant difference at other w/c ratios. At normal environments, at low w/c ratios, the normal and silica fume concrete yielded almost exact ITZ thicknesses whereas in hot environments there was a slight increase. When the quality of the matrix is low and the surrounding environmental conditions are considered as bad, the thickness of ITZ increases. It should be emphasized that, the observations up to this point had been done considering the specimens prior to loading.

When the loaded samples are investigated, with respect to the non-loaded specimens, based on the w/c ratios, with increasing w/c cement ratio, the ITZ thickness also increases. The biggest difference between w/c ratios observed under normal environmental conditions for normal concrete, where there ITZ thickness jumps from 80 micrometers at 0.50 w/c ratio to approximately 180 micrometers at 0.70 w/c ratio, revealing more than two times difference. Normal concrete under normal environmental condition at 0.70 w/c ratio. While for all other 10 samples, with high temperature and humidity levels the ITZ gets thicker, for this particular specimen the thickness decreases by 50 micrometers. There is also an insignificant decrease in thickness for 0.50 w/c ratio silica fume concrete from normal to hot environmental conditions. When specimens with and without silica fume are compared, under hot environment, addition of silica fume has almost no effect on the ITZ thickness, but causes a mild decrease for low w/c ratio specimens under normal environmental conditions and a sharp increase for 0.70 w/c ratio.

Moreover, for the same w/c ratio and environmental conditions, the ITZ is found to get thicker after loading, with the exception of 0.35 w/c ratio normal concrete, where a decrease of about 10 micrometers is observed.

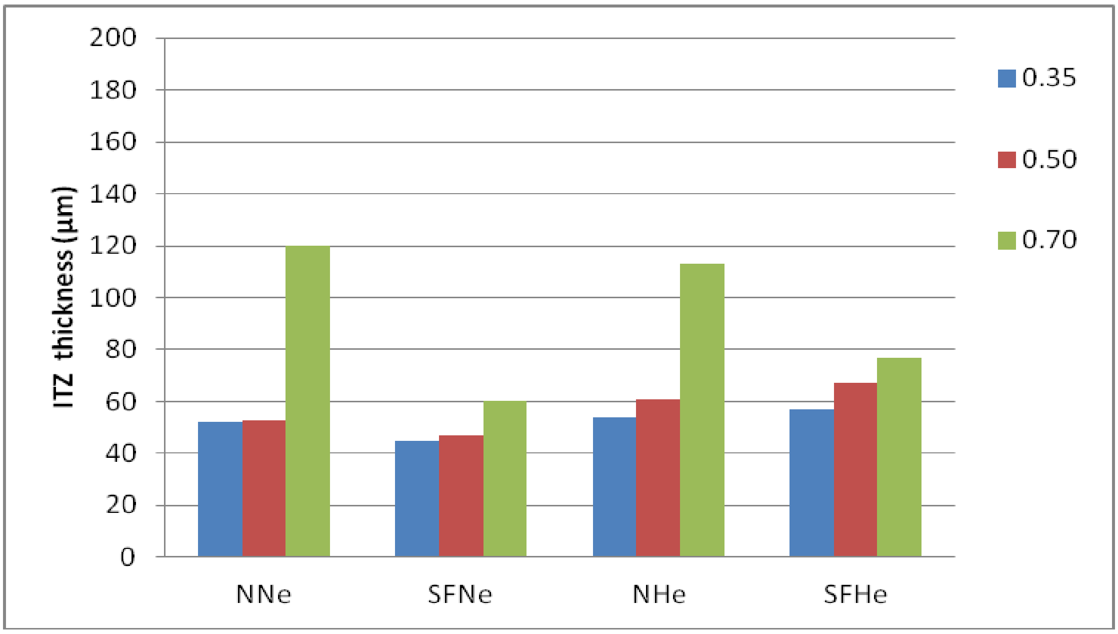

Figure 4.16. Effect of matrix quality and environmental conditions on ITZ thickness before loading

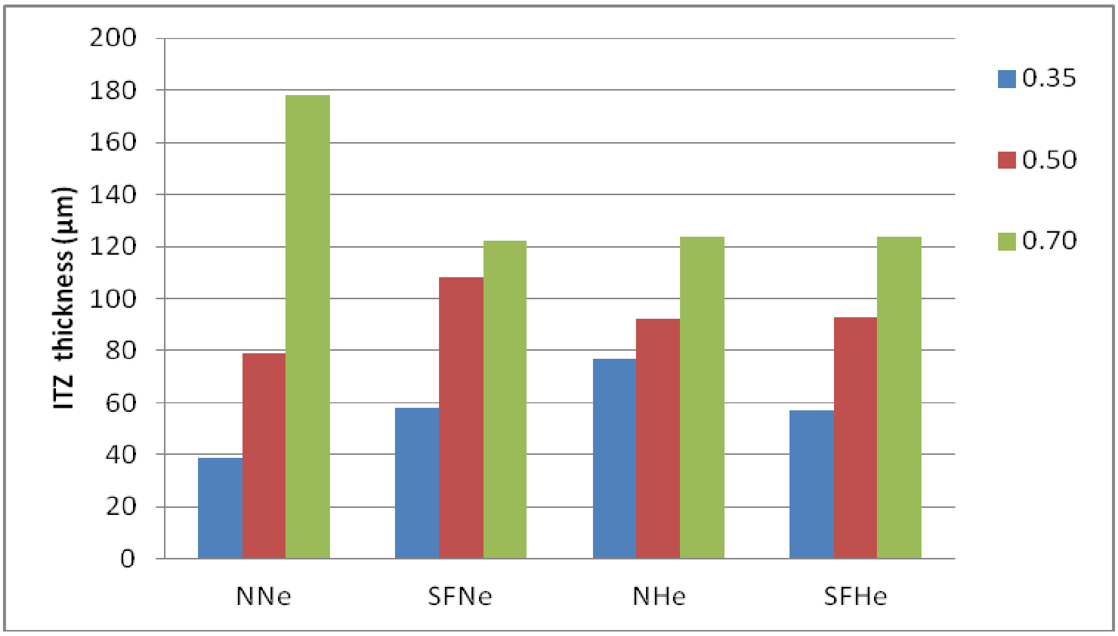

Figure 4.17. Effect of matrix quality and environmental conditions on ITZ thickness after loading

4.7.2 Effect of Matrix Quality and Environmental Conditions on ITZ and Matrix Crack Densities

In this section, crack densities are measured either in the ITZ or within the matrix. This has been achieved by measuring the crack lengths with a ruler and calculating the crack area taking into account the crack length and width. 1 mm and 1.5 mm thick cracks are considered as microcracks in this study. At the end, crack density was examined (mm^2/mm^2), calculated by dividing the crack area to the cross-sectional area. The results are shown in Table 4.2 and Figures 4.18 to 4.23. This study has been done to be able to understand the effects of matrix quality and environmental conditions on crack initiation and then their effect on the whole microcracking behavior after loading.

4.7.2.1 ITZ Crack Density Before Loading

Effect of matrix quality and environmental conditions on ITZ crack density before loading the concrete specimen is represented in Figure 4.18. ITZ itself was analyzed for the crack formation and density, in N specimens before loading, ITZ crack density for 0.50 w/c ratio there is no change in between two environmental conditions. On the other hand, while 0.70 w/c ratios there is a decrement in crack density by approximately 15% and for 0.35 w/c ratio, there is a sharp increase in crack density within 46% from normal to hot environment.

When silica fume is added to the concrete mixes, crack densities increase both for mixes with 0.35 and 0.50 w/c ratio after transition from normal to hot climate conditions. For 0.70 w/c ratio, the densities are almost equal under both environmental conditions. Generally, when the normal environment mixes are compared, with addition of silica fume, the crack density is decreased in all w/c ratio mixes in hot climate conditions, with the exception of 0.70 w/c ratio, silica fume enhanced mixes decreased the ITZ crack density.

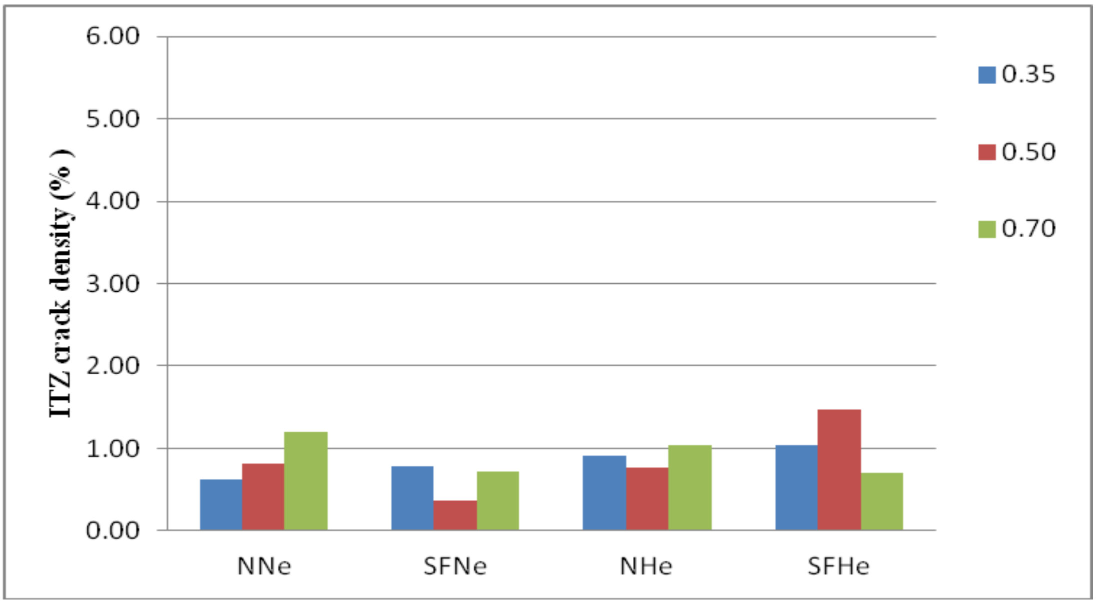

Figure 4.18. Effect of matrix quality and environmental conditions on ITZ crack density before loading

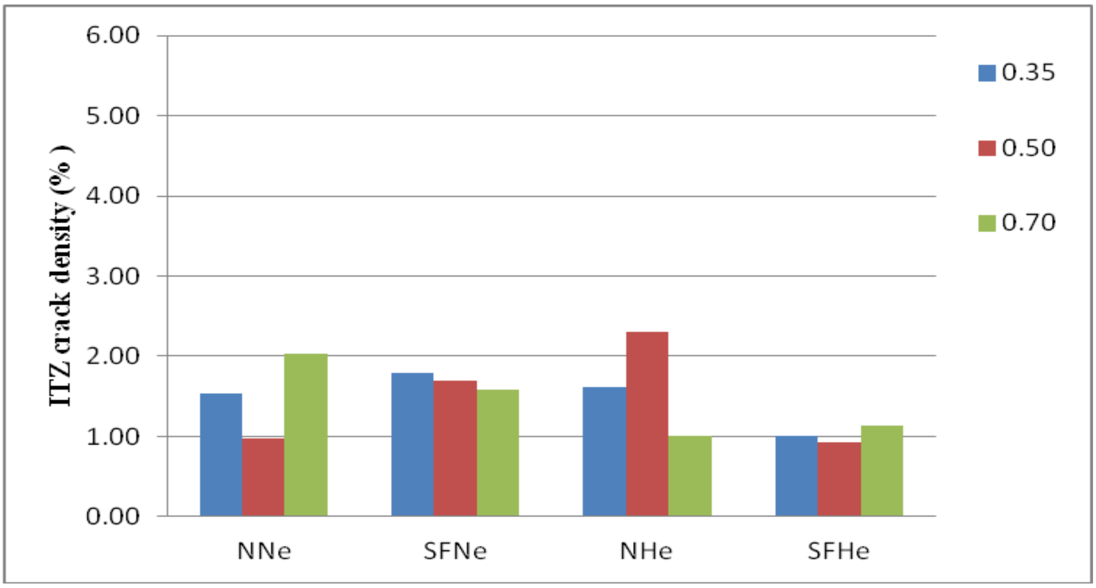

Figure 4.19. Effect of matrix quality and environmental conditions on ITZ crack density after loading

4.7.2.2 Matrix Crack Density Before Loading

Effect of matrix quality and environmental conditions on matrix crack density before loading the concrete specimen is represented in Figure 4.20. Matrix crack densities are observed at the highest point under normal environmental conditions of 0.70 w/c ratio. Under normal environments, silica fume inclusion does not make a difference, and at low w/c ratios it is lower than that of high w/c ratio of 0.70.

Under high humidity and temperature, the crack density for all w/c ratios are lower for SF, compared to N, indicating a positive effect of silica fume on crack density. When the hot environment conditions are investigated, N samples show a gradual decrease with increasing w/c ratio, whereas an inverse pattern is observed for SF, with increasing density with increasing w/c ratio. In general, silica fume inclusion has a positive effect on matrix crack density and it is more effective at low w/c ratio matrices.

4.7.2.3 ITZ and Matrix Crack Densities After Loading

Effect of matrix quality and environmental conditions on ITZ crack and matrix crack densities after loading, the concrete specimen is represented in Figures 4.19 to 4.21.When the crack densities were analyzed, after loading the concrete specimens up to 80-85% of their ultimate strengths, the following can be commented:

- A separate look at individual w/c ratios yield that, the lowest crack propagation occurs for loaded, silica fume concrete under hot climatic conditions in ITZ region.
- When matrix crack density under normal environment conditions is examined, normal specimens crack density at low level w/c ratios is equal to each other, similar to all other conditions. For 0.70 w/c ratio, this value is much higher compared to the other two (about twice).

- With silica fume, the crack densities also increase in all cases in the matrix area since the volume change also was higher.
- Where there is higher temperature and humidity, the effect of w/c ratio on matrix crack density decrease compared to the normal environments.

Before loading the materials, the highest crack initiation took place in 0.70 N specimens and the lowest crack formation occurred in 0.50 SF mixtures, both under normal environmental conditions. For 0.35 w/c ratio, NNe specimens was observed to have the same crack density as SFHe. In this case, it can be concluded that the negative effect of the hot environmental conditions is compensated with the silica fume inclusion.

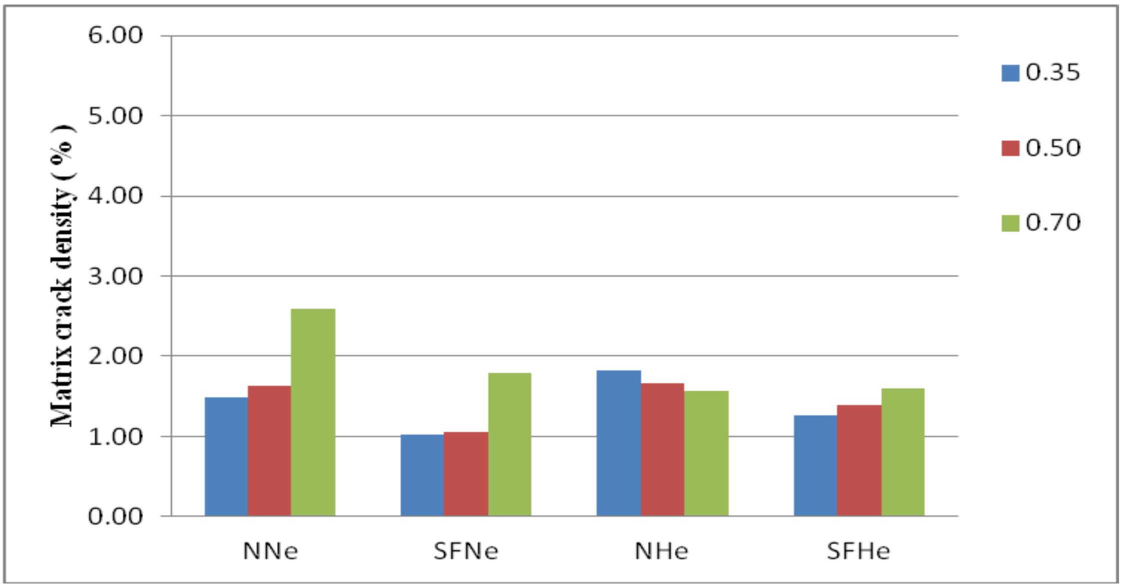

Figure 4.20. Effect of matrix quality and environmental conditions on matrix crack density before loading

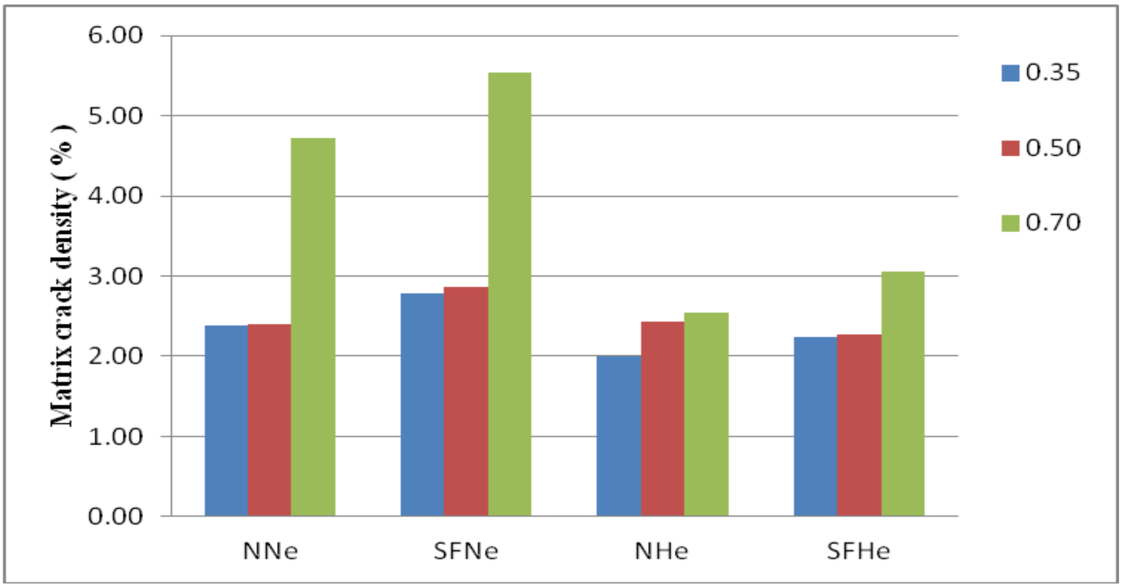

Figure 4.21. Effect of matrix quality and environmental conditions on matrix crack density after loading

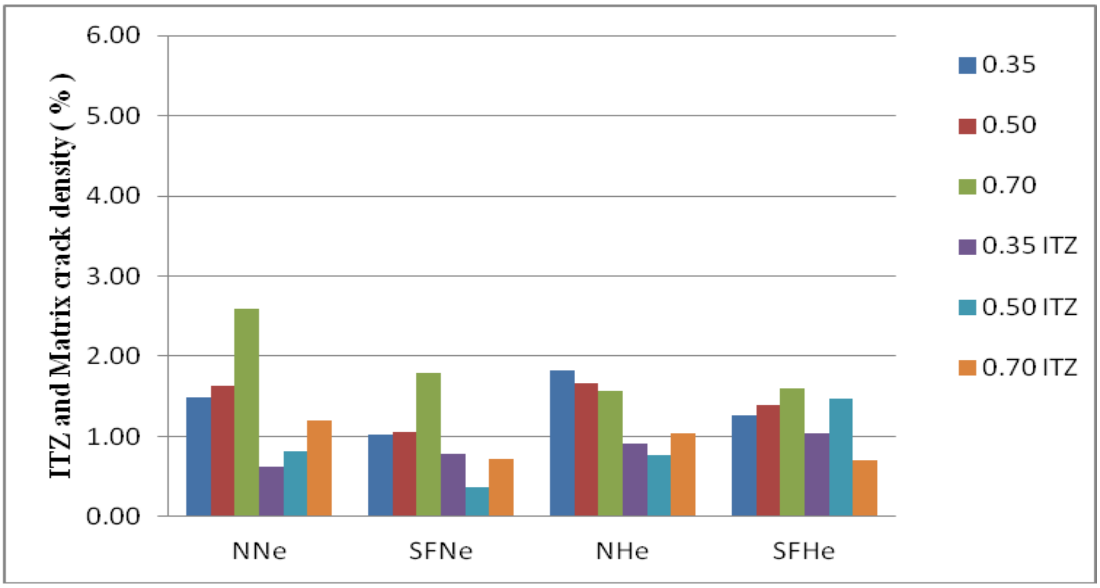

Figure 4.22. Effect of matrix quality and environmental conditions on total crack density before loading

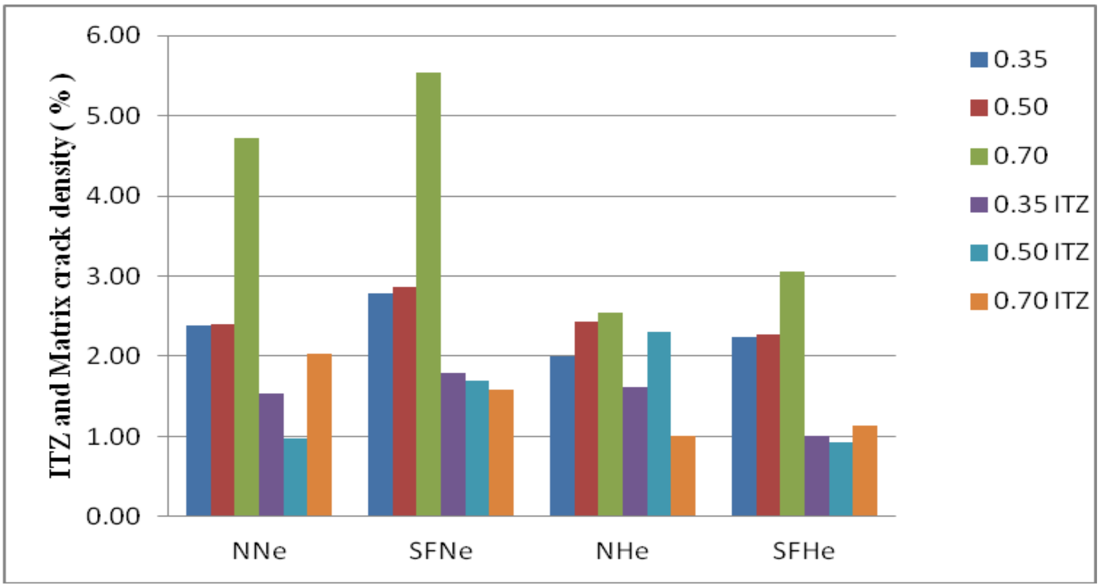

Figure 4.23. Effect of matrix quality and environmental conditions on total crack density after loading

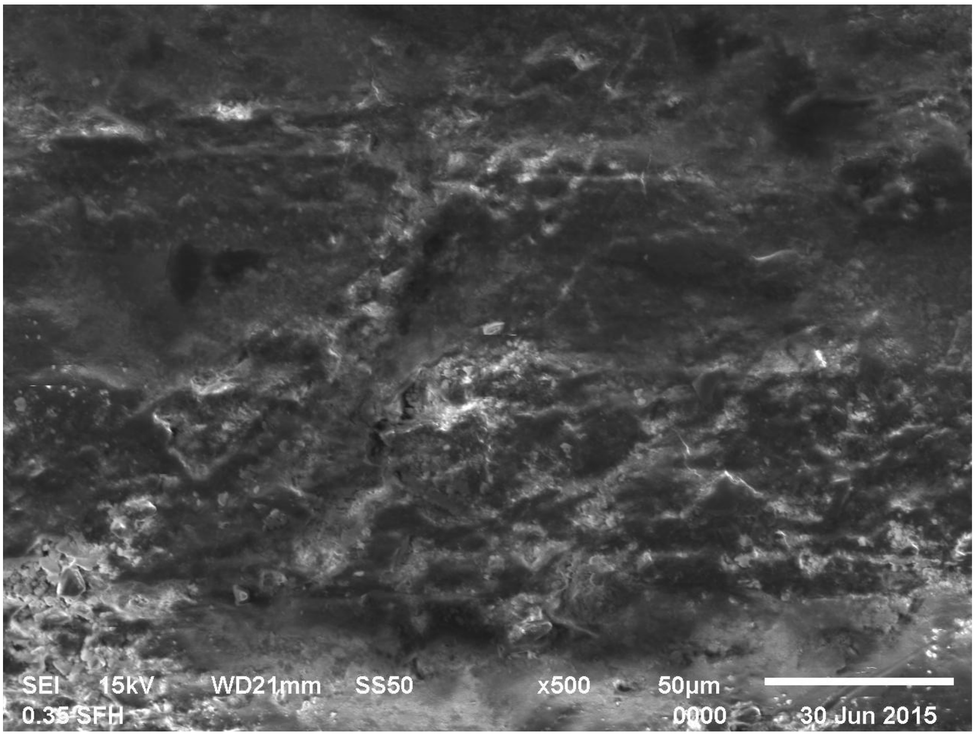

Figure 4.24. Matrix cracks for 0.35 SFHe concrete before loading

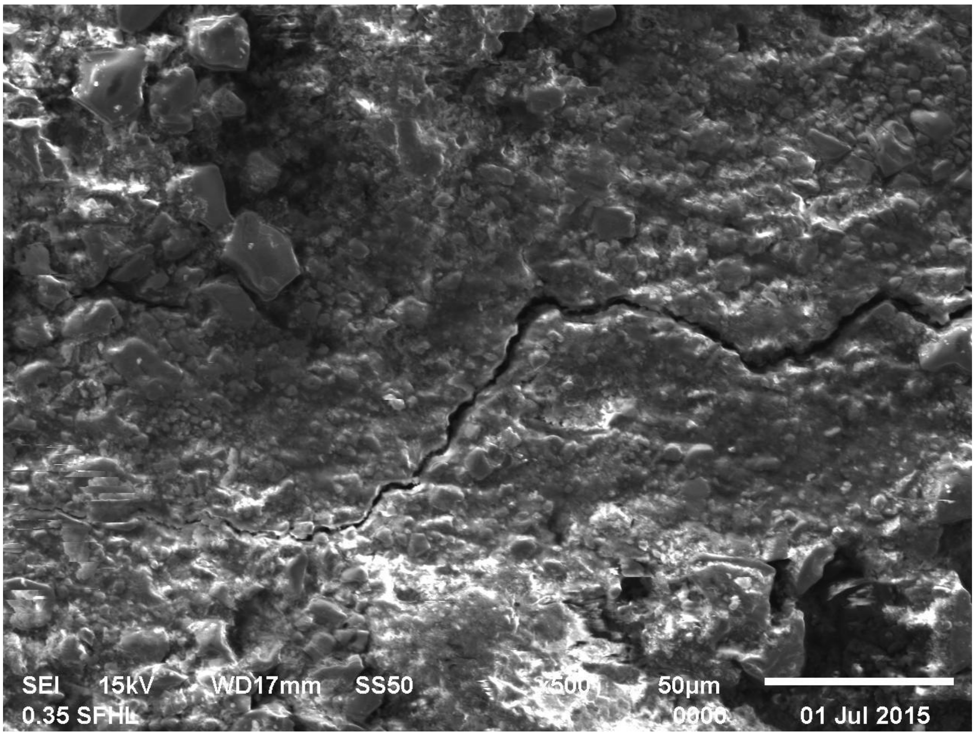

Figure 4.25. Matrix cracks for 0.35 SFHe concrete after loading

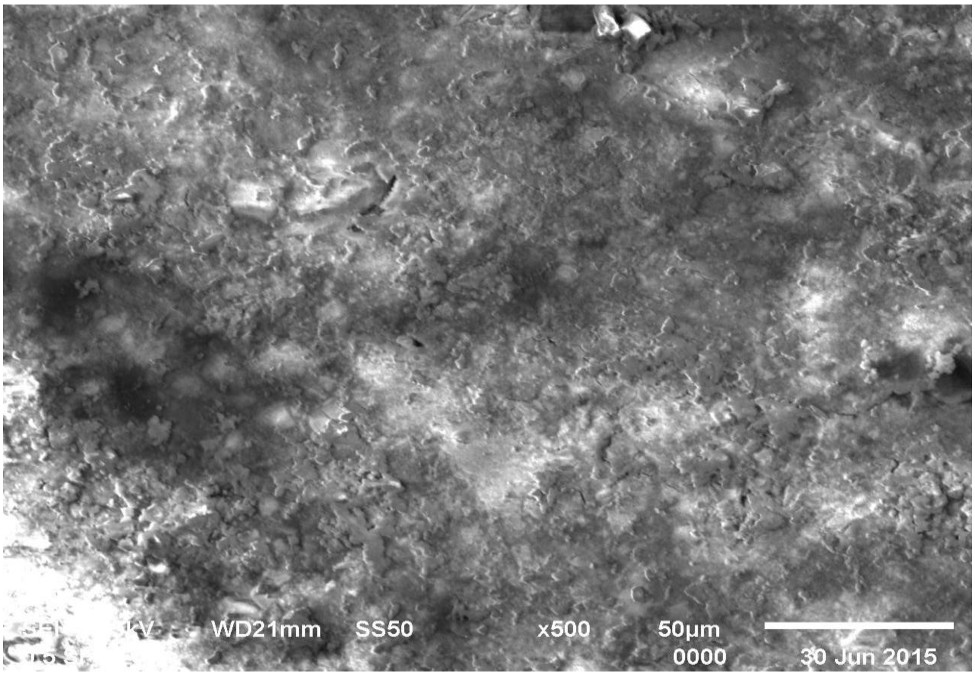

Figure 4.26. Matrix cracks for 0.50 SFHe concrete before loading

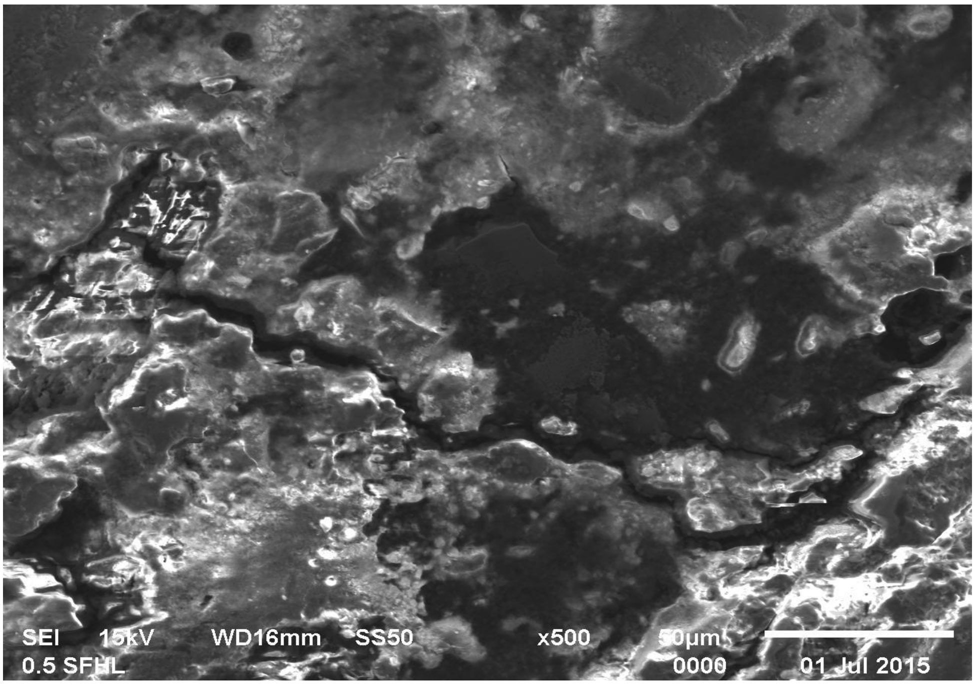

Figure 4.27. Matrix cracks for 0.50 SFHe concrete after loading

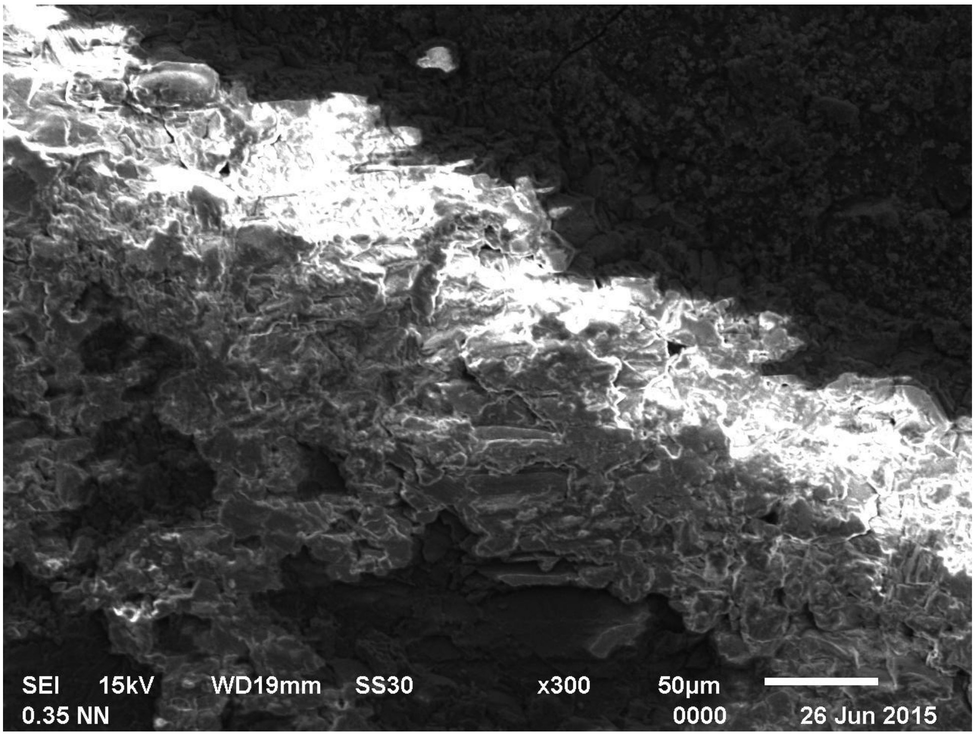

Figure 4.28. A typical ITZ region for 0.35 NNe concrete before loading

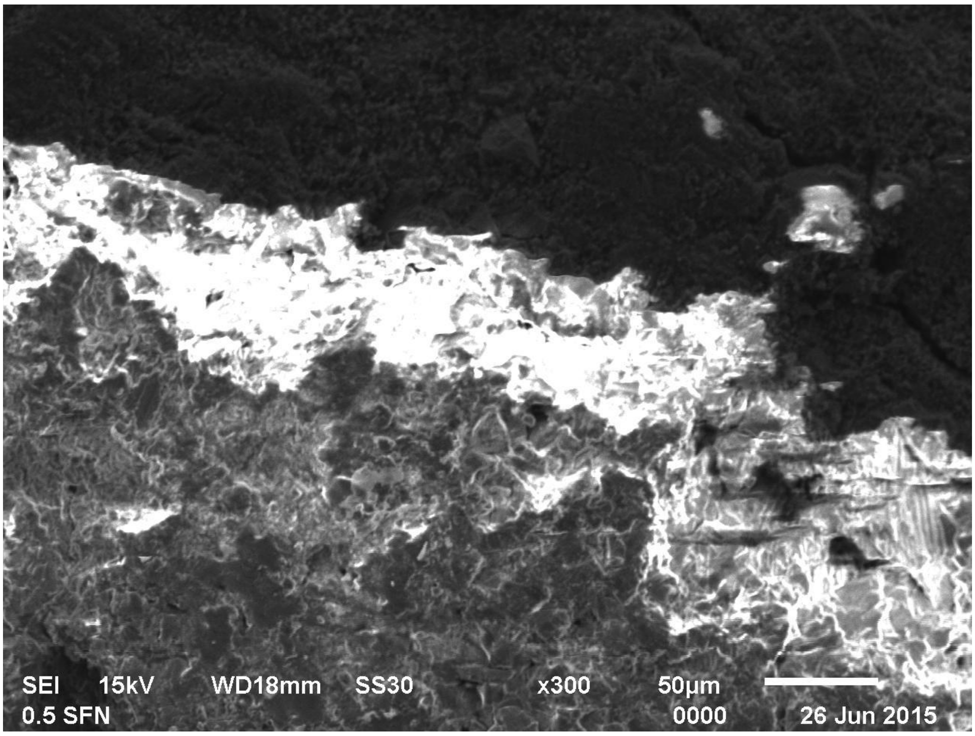

Figure 4.29. A typical ITZ region for 0.50 SFNe concrete before loading

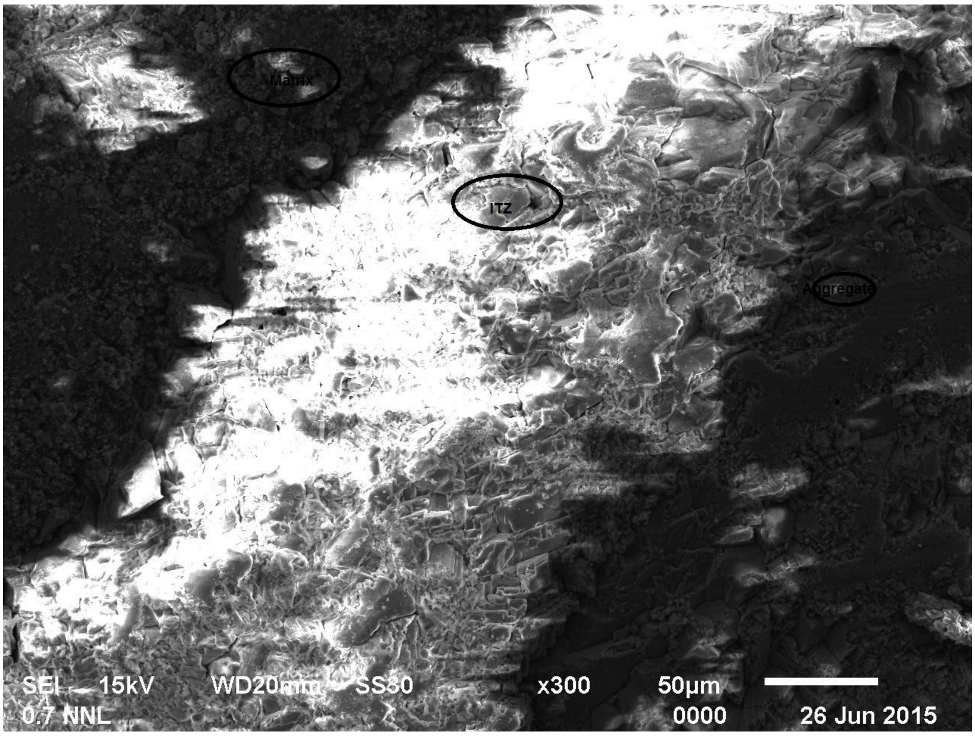

Figure 4.30. A typical ITZ region for 0.70 NNe concrete after loading

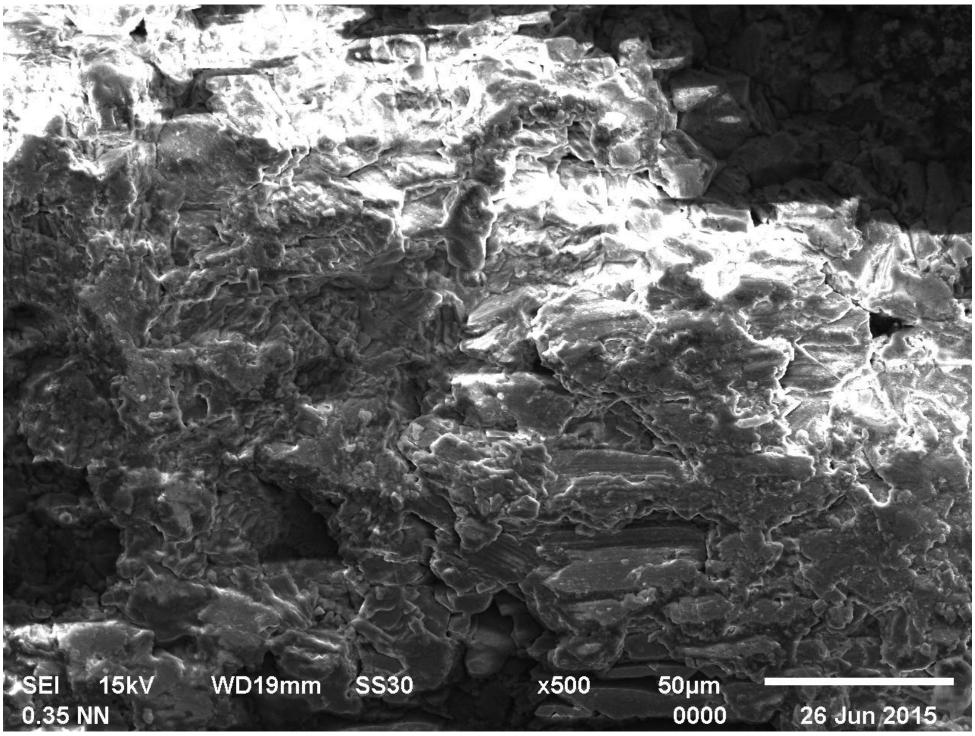

Figure 4.31. ITZ cracks for 0.35 NNe concrete specimens before loading

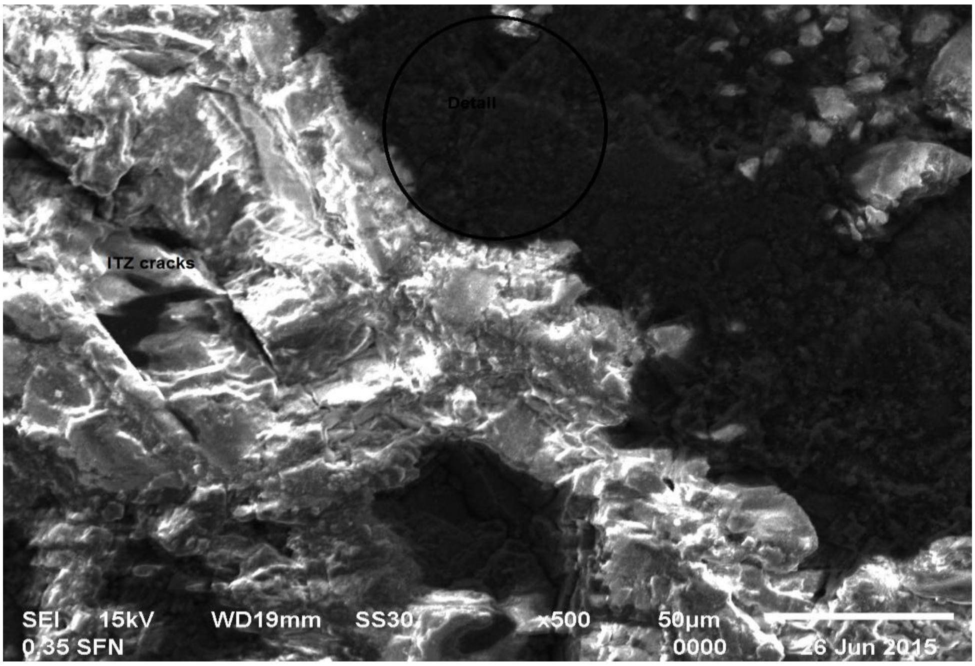

Figure 4.32. ITZ and matrix cracks for 0.35 SFNe concrete specimens before loading

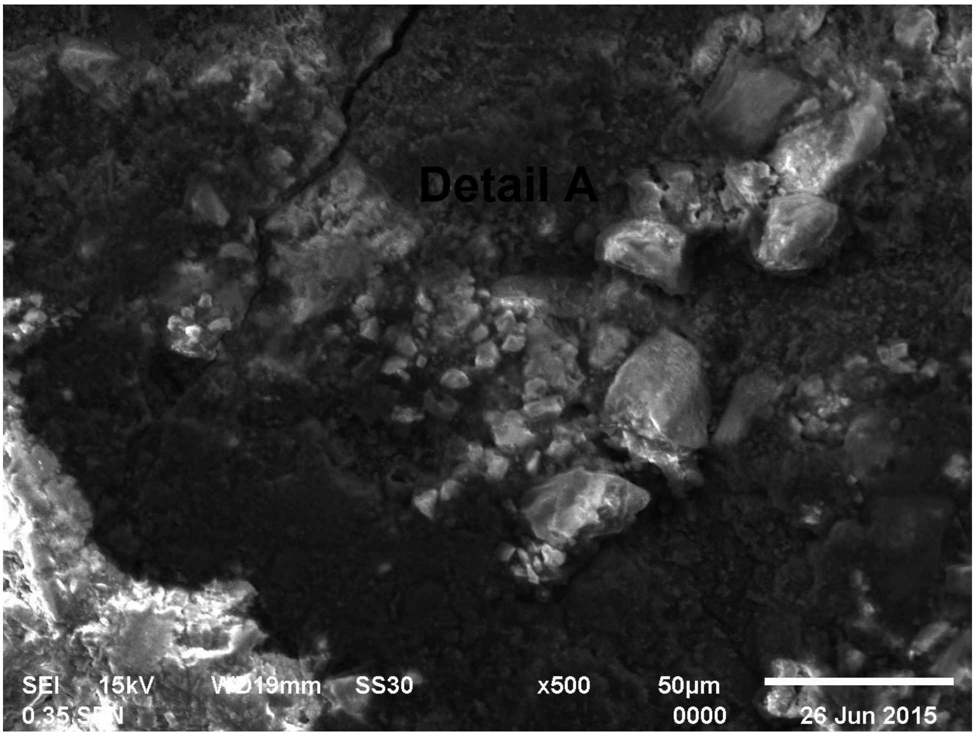

Figure 4.33. Matrix cracks for 0.35 SFNe concrete specimen before loading (Detail A)

Chapter 5

CONCLUSION

Investigating and determining the effect of w/c ratio, silica fume and environmental conditions on the properties of hardened concrete such as volume change and microcracking behavior before and after loading were the main objectives of this study. Upon making different concrete mixes with three different w/c ratios, with and without silica fume under two environmental conditions, these properties were tested and investigated, and finally SEM analysis was conducted on these specimens. In short, the following conclusions can be drawn from this study:

1) The water cement ratio is the most influential parameter on compressive strength, where, as the w/c ratio increase, compressive strength decreases. At low w/c ratios, humidity and temperate were found to have no considerable effect on compressive strength values. However at 0.70 w/c ratios under hot weather conditions higher compressive strength for both normal and silica fume concretes were obtained.
2) Different from its null effect on compressive strength, silica fume has found to increase the tensile strength at all w/c ratios under both environmental conditions. At low w/c ratios (0.35 and 0.50), environmental conditions are not influential however, at 0.70 w/c ratio, which is considered as high w/c ratio, tensile strength increased with increased humidity and temperature. When w/c ratio is considered, a linear increase is observed with a decreasing ratio.

3) Hot environments have a negative effect on volume change (decrement) and it is more pronounced at high w/c mixes (low matrix quality). In general, volume change (decrement) increases with silica fume inclusion except for between 0.35 NHe and 0.35 SFHe specimens, where volume change (decrement) decreased with silica fume inclusion. This can be defined as; negative effect of hot environment has been compensated by silica fume inclusion at low w/c ratio mixes. This also can be due to superplasticizer effect that was used only for 0.35 w/c ratios.
4) Silica fume inclusion increased length change (decrement), except for 0.70 SFHe in x and z directions. Furthermore, there is no significant effect of w/c ratio on length changes in all types of mixes and environmental conditions.
5) Weight loss with time increased for all mixes with increasing w/c ratio. Mixes with silica fume show less mass loss than normal concrete. Furthermore, there is a rapid increase observed in mass loss between 7 and 14-days due to ending of curing period.
6) Under hot environments, linearity ends up earlier (from σ-ε diagrams) for all specimens compared to the normal environment. That means, under high temperature and humidity, crack initiation and growth take place earlier.
7) ITZ thickness increase with increasing of w/c ratio. Increment rate is less between w/c 0.35 and 0.50 compared to 0.70 for normal concrete samples.
8) Under normal environmental conditions, with addition of silica fume, the crack density is decreased in all w/c ratio mixes. Iin hot climate conditions, with the exception of 0.70 w/c ratio, silica fume enhanced mixes decreased the ITZ crack density. Moreover when the hot environmental conditions are investigated, normal concrete samples show a gradual decrease with

increasing w/c ratio, whereas an inverse pattern is observed for silica fume concrete, where there is an increase in density with increasing w/c ratio.

9) Silica fume inclusion has a positive effect on matrix crack density and it is more effective at low w/c ratio matrixes. After loading it can be concluded that the negative effect of the hot environmental conditions is compensated with the silica fume inclusion.

10) ITZ crack densities photo analises illustrate that, the effect of w/c and silica fume are not clear. In contrast the effect of loading on all samples is significant on crack density and the rate of increase is approximately 100% than unloaded samples.

REFERENCES

[1] Alhozaimy, A. M., & Shannag, M. J., (2009). Performance of Concretes Reinforced with Recycled Plastic Fibres, *Magazine of Concrete Research*. 61(4), 293–298.

[2] Park, J. J., Yoo D., Kim S., & Yoon, Y., (2013). Drying Shrinkage Cracking Characteristics of Ultra-high-performance Fibre Reinforced Concrete with Expansive and Shrinkage Reducing Agents, *Magazine of Concrete Research*. 65, 248–256.

[3] Clarke, C., (2009). Concrete Shrinkage Prediction Using Maturity and Activation Energy, University of Maryland, College Park, *Master of Science*. 1, 1-3.

[4] Barr, B .I. G., & El-Baden, A. S. A., (2003). Shrinkage Properties of Normal and High Strength Fibre Reinforced Concrete, Proceedings of the Institution of Civil Engineers: *Structures and Buildings*. 156(1), 15–25.

[5] Bissonnette, B., Pierre, P.,& Pigeon, M., (1999). Influence of Key Parameters on Drying Shrinkage of Cementitious Materials, *Cement and Concrete Research*. 29, 1655–1662.

[6] Reddy, V. M., (2013). Effect of W/C Ratio on Workability and Mechanical Properties of High Strength Self Compacting Concrete (M70 Grade), *International Journal of Engineering Research and Development*. 7(1), 6-13.

[7] Kwan, K. H., McKinley, M. & Chen, J. J., (2013). Adding Limestone Fines as Cement Paste Replacement to Reduce Shrinkage of Concrete, *Magazine of Concrete Research*. 65(15), 942–995.

[8] Sivakumar, A., (2013). Studies on Influence of Water-Cement Ratio on the Early Age Shrinkage Cracking of Concrete Systems, *Journal of Civil Engineering and Construction Technology*. 4(1), 1-5.

[9] Akçaoğlu, T., Tokyay, M., & Çelik, T., (2005). Assessing the ITZ Microcracking via Scanning Electron Microscope and its Effect on the Failure Behaviour of Concrete, *Cement and Concrete Research*. 35, 358-363.

[10] Akçaoğlu, T., Tokyay, M., & Çelik, T., (2004). Effect of Coarse Aggregate Size and Matrix Quality on ITZ and Failure Behavior of Concrete Under Uniaxial Compression, *Cement and Concrete Composites*. 26(6), 633-638.

[11] Zhang, M. H., Tam, C. T., & Leow, M. P., (2003). Effect of Water-to-Cementitious Materials Ratio and Silica Fume on the Autogenous Shrinkage of Concrete, *Cement and Concrete Research* . 33, 1687–1694.

[12] Pawade, P. Y, Nagarnaik, P. B., & Pande, A. M., (2011). Influence of Silica Fume in Enhancement of Compressive Strength, Flexural Strength of Steel Fibers

Concrete and Their Relationship, *International Journal of Civil and Structural Engineering*. 2(1), 43-55.

[13] Regunathan, P., Wong, L. S.,& Nordin, N., (2015). A Review on the Compressive Strength of Concrete Improved with Various Types of Pozzolan, *The third National Graduate Conference*. *Universiti Tenaga Nasional, Putrajaya Campus*. (64), 240-244.

[14] Güneyisi, E., Gesoglu, M., & Karaoglu, S., (2012). Strength, Permeability and Shrinkage Cracking of Silica Fume and Metakaolin Concretes, *Construction and Building Materials*. 34, 120–130.

[15] Naik, T. R. , & Kraus, R. N., (2002). Temperature Effects on High-Performance Concrete, *Sixth International Symposium on Utilization of High Strength/High Performance Concrete, Leipzig, Germany*. (7),1-18.

[16] Biswal, K. C., & Sadangi, S. Ch., (2011). Effect of Superplasticizer and Silica Fume on Properties of Concrete, *ACEE International Journal on Civil and Environmental Engineering*. (1), 41-43.

[17] Al-Amoudi, O. S. B., Maslehuddin, M., & Shameem, M., (2007). Shrinkage of Plain and Silica Fume Cement Concrete Under Hot Weather, *Cement & Concrete Composites*. 29, 690–699.

[18] Al-Amoudi, O. S. B., Maslehuddin, M., & Abiola, T. O., (2004). Effect of Type and Dosage of Silica Fume on Plastic Shrinkage in Concrete Exposed to Hot Weather,*Construction and Building Materials*. 18, 737–743.

[19] Ayub, T., Khan, S. U., & Memon, F. A., (2014). Mechanical Characteristics of Hardened Concrete with Different Mineral Admixtures, *Scientific World Journal*. 2014, 1-15.

[20] Sandvik, M., & Gjorv, O. E., (1992). Prediction of Strength Development for Silica Fume Concrete, *Proceedings of fourth International Conference on the Use of Fly Ash, Silica Fume, Slag and Natural Pozzolans in Concrete, Istanbul, Turkey. ACI Special Publication.* 132, 987–996.

[21] Pradhan D., & Dutta, D., (2013). Influence of Silica Fume on Normal Concrete, *Journal of Engineering Research and Applications*. 3(5), 79-82.

[22] Yazıcı, H., (2008). The Effect of Silica Fume and High-Volume Class C Flyash on Mechanical Properties, Chloride Penetration and Freeze–Thaw Resistance of Self-Compacting Concrete, *Construction Building Material*. 22(4), 456–462.

[23] Yang, K., & Mun, J., (2015). Effect of Curing Temperature Histories on the Compressive Strength Development of High-Strength Concrete, *Advances in Materials Science and Engineering*. 2015, 1-12.

[24] Ait-Aider, H., Hannachi, N. E., & Mouret, M., (2007). Importance of W/C ratio on Compressive Strength of Concrete in Hot Climate Conditions, *Building and Environment*. 42, 2461–2465.

[25] Lura, P., Breugel, K., & Maruyama, I., (2001). Effect of Curing Temperature and Type of Cement on Early-age Shrinkage of High-Performance Concrete, *Cement and Concrete Research.* 31, 1867–1872.

[26] Hadjab, H., Thimus, J. F., & Chabaat, M., (2010). Comparative Study of Acoustic Emission and Scanning Electron Microscope to Evaluate Fracture Process Zone in Concrete Beams, *Journal of Materials in Civil Engineering*. 22, 1156-1163.

[27] Neville, A. M, (2002). *Properties of Concrete*, British Library, Fourth Edition.

[28] Jasiczak, J., & Szymanski, P., (2004). The Influence of Melamine Based Superplasticizer on Early Concrete Shrinkage, *Foundations of Civil and Environmental Engineering*. 1, 1642-9303.

[29] Colak, A., (2005). Properties of Plain and Latex Modified Portland Cement Pastes and Concretes with and without Superplasticizer, *Cement and Concrete Research*. 3, 1510–1521.

[30] Alsayed, S. H., (1998). Influence of Superplasticizer, Plasticizer and Silica Fume on the Drying Shrinkage of High Strength Concrete Subjected to Hot-Dry Field Conditions, *Cement and Concrete Research*. 28(10), 1405–1415.

[31] Al-Amoudi, B. S., Abiola, T. O., & Maslehuddin, M., (2006). Effect of Superplasticizer on Plastic Shrinkage of Plain and Silica Fume Cement Concretes, *Construction and Building Materials*. 20, 642–647.

[32] Aruntas, H. Y., Cemalgil, S., & Simsek O., (2008). Effects of Superplasticizer and Curing Conditions on Properties of Concrete with and without Fiber, *Materials Letters*. 62, 3441-3443.

[33] ASTM C 33/C- 33M, (2011), Standard Specification for Concrete Aggregates.

[34] ASTM C 143/C-143 M, (2012), Standard Test Method for Slump of Hydraulic-Cement Concrete.

[35] ASTMC 157/C 157M, (2014), Standard Test Method for Length Change of Hardened Hydraulic-Cement Mortar and Concrete.

[36] ASTM, C 192/C 192M, (2015), Standard Practices for Making and Curing Concrete Test Specimens in the Laboratory.

[37] ACI, 305 -1, (2014), Specification for Hot Weather Concreting.

[38] ACI, 318, (2014), Building Code Requirements for Structural Concrete and Commentary.