Determination of the Maximum Placement and Curing Temperatures in Mass Concrete to Avoid Durability Problems and DEF

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16. Abstract

The Florida Department of Transportation specifies a maximum differential of 35°F, between the exterior and interior portions of the mass concrete elements during curing. However, the specification does not specify a maximum curing temperature or a maximum placing temperature. The FDOT mass concrete projects of the past reveal that the temperature of the core may reach up to 190°F to 200°F. The objective of this project was to determine the effect of concrete curing temperature on its strength, durability and other physical/chemical properties, and to determine the maximum internal concrete temperature above which the concrete properties will be affected. A survey of current US Highway Agency specifications relative to mass concrete showed that the majority of those responded to the survey (65%) believed that mass concrete pours should be controlled by a maximum differential temperature, which most agencies currently specify, and a maximum curing temperature, which most agencies do not currently specify. The reasons given to support their concerns for specifying a maximum curing temperature was to avoid durability problems, later age strength reduction, delayed ettringite formation (DEF), and cracking due to expansion of concrete. Experimental works revealed that when pure Portland cement concrete samples were introduced to a controlled ascending temperature rise simulating approximately conditions of mass concretes cured in the field, there was a moderate reduction in 28-day compressive strength and a significant increase in permeability compared to samples cured at room temperature. Results of compressive strength tests and RCP tests showed that addition of blended cement improves strength and durability of concrete. Microstructural analysis of mortar samples sieved from the concrete mixes using the Scanning Electron Microscope (SEM) showed that addition of pozzolanic materials reduces the possibility of formation of delayed ettringite. It also identified the formation of delayed ettringite in samples 28 days and older where curing temperature was 160F and 180F. Based of findings of this research project it is recommended that use of fly ash or slag as a cement replacement be required in mass concrete since these pozzolanic materials reduce the detrimental effect of high curing temperature on strength and durability of pure cement concrete. When pozzolanic materials are used as a cement replacement, based on ideal laboratory conditions and accurate batching proportions we found an 8 to 15% reduction in compressive strength due to elevated curing temperatures, however durability was not adversely affected. Formation of delayed ettringite in samples 28 days and older where temperature was 160F and 180F is a point of concern and more study is needed to look at the microstructural analysis of samples cured at temperatures more than 160F, specifically for detection of delayed ettringite formation.

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EXECUTIVE SUMMARY

The Florida Department of Transportation specifies a maximum differential of 35°F, between the exterior and interior portions of the mass concrete elements during curing. However, the specification does not specify a maximum curing temperature or a maximum placing temperature. The FDOT mass concrete projects of the past reveal that the temperature of the core may reach up to 170°F to 200°F. The objective of this project was to determine the effect of concrete curing temperature on its strength, durability and other physical/chemical properties, and to determine the maximum internal concrete temperature above which the concrete properties will be affected. The following is a summary of the work done in the execution of this research project:

A state-of-the-art review of work reported on heat generation in mass concrete was performed and measures taken to avoid cracks and premature deterioration of concrete were identified. The literature review revealed that higher curing temperatures increase the initial strength, but decrease the later-age strength. Additionally, in plain Portland cement concretes, elevated curing temperatures result in coarser pore structure and increase total porosity mostly in the volume of larger pores. This suggests that high curing temperatures could reduce durability of plain cement concrete since large pores have the greatest effect on permeability and reduce the concrete resistance to chloride ion penetration. Corrosion of reinforcing steel is a result of chloride ions, and cause premature deterioration of concrete structures. Another problem associated with high concrete curing temperature reported in the literature is early concrete distress due to

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delayed ettringite formation (DEF). Ettringite is a normal and apparently innocuous constituent of hydrated Portland cement. Its formation at the initial stages of hydration is seen as a positive effect because it enables the setting of the cement, however a damaging role is attributed to its formation in hardened concrete.

There was also a survey conducted of current US Highway Agency specification relative to mass concrete. It gave valuable information relative to the current opinions of highway agencies on the topic of specifications required for mass concrete. The majority of the US Highway Agencies that responded to the survey (65%) agreed that mass concrete pours should be controlled by a maximum differential temperature, which most agencies currently specify, and a maximum curing temperature, which most agencies do not currently specify. The reasons given to support their concerns for specifying a maximum curing temperature was to avoid durability problems, later age strength reduction, delayed ettringite formation, and cracking due to expansion of concrete. The opinions of the US Highway Agencies were similar to the concerns noted in the literature review. Both noted the affect of high temperature relative to the reduced durability of the concrete and delayed ettringite formation.

The effects of concrete curing temperature on the properties of hardened concrete were evaluated. The evaluation included the following tests: compressive strengths, rapid chloride permeability, time-to-corrosion, volume of permeable voids, and microstructure analysis using the Scanning Electron Microscope (SEM).

    a.   Class IV - Structural concrete mixes, consisting of 18 percent replacement by weight of cement with class F fly ash, were produced. Specimens

required for the above mentioned tests were cast at room temperature and stored immediately in water tanks where they were subjected to different curing temperatures (73 to 200°F).

b.   Other mixes were produced similar to Part "a" above except that 50 percent of cement was replaced by slag. Molds were cast and stored as explained in part "a".

The experiment performed first attempted to determine the historical development of the degree of hydration in cement pastes with 18% fly ash and 50% blast furnace slag at different adiabatic curing temperatures. The objective was to identify a point at which the concrete has reached a certain percentage of hydration and measure concrete properties at this point. This occurs when the development of degree of hydration reduces drastically, to a point where additional time does not provide a significant increase in the degree of hydration (approximately 70 percent). However, the results of the tests were not consistent and did not allow accurate determination of number of days required for each mix to reach a degree of hydration of approximately 70 percent at different adiabatic curing temperatures. It was decided instead to measure properties of concrete at 7, 28, and 90 days and measure the degree of hydration at these ages.

The results of these experiments revealed:

-    A substantial decrease in compressive strength of plain Portland cement concrete samples cast and stored immediately in water tanks under isothermal curing temperatures of 160 F and 200 F was recorded compared to samples cured at room temperature (73 F). This reduction was 34% and 62% for 28-

day compressive strength for samples cured at 160 F and 200 F, respectively. In addition, RCP test of these samples showed a significant increase in permeability of concrete cured at high temperature.

- When plain Portland cement concrete samples were introduced to a controlled ascending temperature rise simulating approximately conditions of mass concretes cured in the field (semi-adiabatic temperature rise), there was a moderate reduction in 28-day compressive strength of samples cured at elevated temperatures compared to samples cured at room temperature. The reduction was 15% and 18% for samples cured at temperatures of 160 F and 180 F, respectively. However, there was still a significant increase in permeability of concrete measured through RCP test.

- Semi-adiabatic curing of fly ash cement concrete samples (18% fly ash by weight) resulted in 8% reduction of 28-day compressive strength for samples cured at 160 F and 180 F compared to those cured at room temperature. However, permeability of concrete measured by RCP test improved noticeably at higher curing temperatures suggesting that at higher temperatures the fly ash becomes effective much earlier and reduces the RCP values. At normal curing temperature the RCP reducing effect of fly ash becomes effective after approximately two months. However, time-to-corrosion test results did not support this finding and showed reduction in time to corrosion for samples cured at higher temperatures compared to those cured at room temperature.

- When 50% (by weight) of Portland cement is replaced by blast furnace slag, the 28-day compressive strength of samples cured at elevated temperatures reduced by 7% and 15% for curing temperatures of 160 F and 180 F compared to those cured at room temperature. Durability of concrete in this case again showed conflicting results from RCP test and time-to-corrosion test, i.e., RCP test results indicated higher curing temperatures improve durability, whereas, time-to-corrosion test results showed a less durable concrete when it is cured at elevated temperature.

- Results of compressive strength tests and RCP tests revealed that addition of blended cement improves strength and durability of concrete.

- Microstructural analysis of mortar samples sieved from the concrete mixes using the Scanning Electron Microscope (SEM) showed that addition of pozolanic materials reduces the possibility of formation of delayed ettringite. It also identified the formation of delayed ettringite in samples 28 days and older where curing temperature was 160F and 180F. No DEF was found in concrete samples cured at room temperature.

Based of findings of this research project it is recommended that:

1. Use of fly ash or slag as a cement replacement should be required in mass concrete since these pozzolanic materials reduce the detrimental effect of high curing temperature on strength and durability of pure cement concrete. In addition, more ettringite was found in the pure cement concrete mix than in

blended cement at 28- and 91-day curing, and the microstructure of the blended cement mixes appeared denser than the pure cement mix.

2. When pozzolanic materials are used as a cement replacement, based on ideal laboratory conditions and accurate batching proportions we found an 8 to 15% reduction in compressive strength due to elevated curing temperatures. However, this loss could be inflated considerably if the concrete was produced at a batch plant with wider mixer proportions tolerances and the ever-present potential of unmetered water in the mix. Formation of delayed ettringite in samples 28 days and older where temperature was 160F and 180F is a point of concern and more study is needed to look at the microstructural analysis of samples cured at temperatures more than 160F, specifically for detection of delayed ettringite formation.

3. This study also showed that when the pure cement concrete specimens were placed in preheated curing tanks as soon as they were molded and cured under constant temperatures of 160 F and 200 F, their compressive strengths were significantly decreased (34% and 62% for 160 and 200 F, respectively) and their permeability were increased. This shows the importance of the age of precast concrete members when they are exposed to steam curing regimen to accelerate their strength gain. The Quality Control/Quality Assurance personnel at precast yards have to be alerted of this important issue.

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CHAPTER 1 INTRODUCTION

1.1 Background

Concrete structures like bridge piers, bridge foundation, and concrete abutments can be defined as "massive structures" by virtue of volume to surface area ratio. FDOT Structures Design Guidelines defines mass concrete as "Any large volume of cast-in-place or precast concrete with dimensions large enough to require that measures be taken to cope with the generation of heat and attendant volume change so as to minimize cracking." (FDOT, 2002) A definite size has not been determined, however, a concrete member, which is 2 ft. to 3 ft. thick is considered to be mass concrete.

Due to heat production in the hydration of Portland cement, a rise in temperature is inherent when concrete is placed. When mass concrete structures are poured, they cause higher temperatures to occur in the interior of the element. The Florida Department of Transportation concrete engineers have observed temperatures as high as 200ºF. The temperature at the core of the concrete pour is greater than at the surface because the surface is able to release its heat directly into the surrounding environment. This causes a significant temperature differential between the surface and the interior of the concrete. This temperature variation can result in tensile stresses that may cause cracking, loss of strength, excessive seepage and reduced durability of the structure.

There are several known potential causes of cracking in concrete. One is excessive stress due to applied loads and another is cracks due to drying shrinkage or temperature changes in restrained conditions. Mass concrete is often subject to both of these stresses, therefore, prevention of cracking is a vital consideration in the design of

these structures. However, during the construction process, the most pressing item is the control of drying shrinkage and differential temperature. The rise in temperature of mass concrete depends on the initial concrete temperature and volume to surface area ratio. Furthermore, the increase in temperature is affected predominantly by the chemical composition of cement, with C_3A (Tricalcium Aluminate) and C_3S (Tricalcium Silicate) being the compounds primarily responsible for elevated temperature development. The water-cement ratio, fineness of the cement, concrete mixture temperature and temperature of curing are also contributors to the development of heat. When the water-cement ratio, fineness, or curing temperature is increased, the heat of hydration is increased. The rate and amount of heat generated are important in any concrete construction requiring considerable mass. The heat accumulated must be rapidly dissipated in order to impede a significant rise in concrete temperature at the center of the structure. The excessive rise in concrete temperature is undesirable since the concrete will harden faster at an elevated temperature and any non-uniform cooling of the concrete structure may create stresses due to thermal contraction.

The current method of preventing cracking in mass concrete is to maintain a temperature differential (between the surface and the core) of no more than 35°F. Control of temperature gain is possible through the following:

1) low-heat-of-hydration portland or blended cement

2) reductions in the initial concrete temperature to approximately 50°F by cooling the concrete ingredients

3) cooling the concrete through the use of embedded cooling pipes water curing

4) low lifts—5 ft or less – during placement.

5) pozzolans – the heat of hydration of pozzolan is approximately 25% to 50% that of cement. (Kosmatka and Panarese, 1994)

When the massive concrete specified has high cement contents (500 to 1000 lb. per cu yard), many of the above mentioned placing methods cannot be used. For concretes that are often used in mat foundations and power plants good placing methods are the following:

6) place the entire concrete section in one continuous pour

7) avoid external restraint from adjacent concrete elements

8) control internal differential thermal strains by preventing the concrete from experiencing excessive temperature differential between the internal concrete and the surface. (Kosmatka and Panarese, 1994)

In order to control the internal temperature differential, the concrete is insulated to keep it warm (tenting, quilts, or sand on polyethylene sheeting). Studies have shown that the maximum temperature differential (MTD) between the interior and exterior concrete should not exceed 35°F to avoid surface cracking.

The Florida Department of Transportation specifies a maximum differential of 35°F, between the exterior and interior portions of the mass concrete elements during curing. However, the specification does not specify a maximum curing temperature or a maximum placing temperature for mass concrete (the maximum placing temperature for hot weather mix design is 100°F). The FDOT mass concrete projects of the past reveal

that the temperature of the core may reach up to 170°F to 200°F. The literature review

that follows reports that higher temperatures increase the initial strength, but decrease the

later-age strength. Additionally, the rate of diffusion of hydration products at increased

temperatures do not permit these elements to distribute evenly throughout the cement

paste matrix, resulting dense hydration products around the cement grains and an open,

porous structure between the grains. This contributes to an increase in total porosity and

void volume, which lends to greater penetration of chloride ions. Corrosion of

reinforcing steel is a result of chloride ions, and cause premature deterioration of concrete

structures.

Another problem associated with high concrete curing temperature is early

concrete distress due to delayed ettringite formation (DEF). Ettringite is a mineral

composed of hydrous basic calcium and aluminum sulfate. When cement hydrates, it

creates ettringite high form. As it looses water the ettringite goes to a low form. At high

curing temperatures (175 F) the low form returns to the high form and causes micro-

cracking.

The Florida Department of Transportation Standard Specifications Section 450

allows the maximum curing temperature of 158ºF to 176ºF for accelerated curing of pre-

stressed concrete elements. However, accelerated curing is conducted under suitable

enclosures with a controlled environment to avoid thermal shock and minimize moisture

loss. These conditions do not exist in mass concrete operations and such high

temperatures may have detrimental effects on concrete properties. It is therefore

necessary to revisit the current specification for mass concrete, and examine the need

for additional provisions. The provisions that need to be reconsidered are maximum internal concrete temperature and/or maximum concrete placing temperature.

1.2 Objectives and Scope

The objective of this project is to determine the effect of concrete curing temperature on its strength, durability and other physical/chemical properties, and to determine the maximum internal concrete temperature above which the concrete properties will be affected.   This will be done by performing the following tasks:

1. Perform a state-of-the-art review of work reported on heat generation in mass concrete and measures taken to avoid cracks and premature deterioration of concrete. This should include review of the current specifications used by other US highway agencies.

2. Evaluate the effects of concrete temperature on the properties of hardened concrete. The evaluation includes the following tests: compressive strengths, rapid chloride permeability, time-to-corrosion, volume of permeable voids, and microstructure analysis using the Scanning Electron Microscope (SEM).

    a. Class IV - Structural concrete mixes, consisting of 18 percent replacement by weight of cement with class F fly ash, will be produced. Specimens required for the above mentioned tests will be cast at room temperature and will be stored immediately in water tanks where they will be subjected to different curing temperatures (73 to 200°F).

    b. Other mixes will be produced similar to Part a except that 50 percent of

cement will be replaced by slag. Molds will be cast and stored as explained in

part a.

3.   Analyze the test results and determine the maximum internal concrete temperature

above which the concrete properties will be affected (later age strength reduction,

durability problems, and DEF).

4.   Re-examine the current FDOT mass concrete specifications and suggest, if

necessary, the requirement for maximum curing temperature or maximum

concrete placement temperature.

CHAPTER 2 LITERATURE REVIEW

2.1 Introduction

This chapter presents a state-of-the-art review of literature on how strength, durability and formation of delayed ettringite (DEF) in concrete are affected by high curing temperatures. In massive concrete structures, high curing temperatures result from a combination of heat produced by the hydration of concrete and the relatively poor heat dissipation of concrete. Although various measures are implemented to limit the maximum temperatures in mass concrete, a high core temperature of 200^oF has been recorded in Florida for a mass concrete structure cast during the summer. While such concrete meets the specification of maintaining a maximum differential temperature of 35^oF between the core and surface of the mass concrete structure, of major concern is what happens to the strength, durability and DEF in the concrete when subjected to such high curing temperatures.

This chapter reviews how heat is generated in concrete from the hydration of cement. Cracking of concrete due to the heat as well as the microstructure formed under such high temperature curing is examined. The influence of the microstructure formed under high curing temperatures on the strength and durability of concrete are presented. To improve the quality of concrete structures cured under high temperatures, other cementitious materials such as fly ash and blast furnace slag have gained increasing use in mass concrete structures. The effects on the microstructure of concrete due to the use of such materials and the influence on strength and durability of mass concrete structure are reviewed. A final review is presented on how high curing temperatures makes

hardened concrete structures susceptible to damage from the formation of delayed

ettringite (DEF).

2.2 Cement Hydration

The compounds of Portland cement (see Table 2.1) are nonequilibrium products

of high temperature reactions in a high-energy state. When cement is hydrated, the

compounds react with water to acquire stable low-energy states, and the process is

accompanied by the release of energy in the form of heat (Mehta and Monteiro, 1993).

Cement acquires its adhesive property from its reaction with water by forming products,

which possess setting, and hardening properties.

Table 2.1 Major Compounds of Portland Cement

Name of compound	Oxide composition	Abbreviation
Tricalcium Silicate	$3CaO.SiO_2$	C_3S
Dicalcium Silicate	$2CaO.SiO_2$	C_2S
Tricalcium Aluminate	$3CaO.Al_2O_3$	C_3A
Tetracalcium Aluminoferritte	$4CaO.Al_2O_3.Fe_2O_3$	C_4AF

The heat generated from the hydration of cement causes a rise in temperature of

concrete. If this rise occurred uniformly throughout a given concrete element without any

external restraint, the element would expand until the maximum temperature has been

reached. The concrete will then cool down with uniform contraction as it loses heat to the

ambient atmosphere. This uniform expansion and contraction will result in no thermal

stresses within the concrete element. According to Neville (1997), restraint exists in all

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but the smallest of concrete members. These thermal restraints result in external and

internal cracking of the concrete. Figure 2.1 shows an example of temperature change,

which causes external cracking of large concrete mass. The critical 20°C (35°F)

temperature difference occurs during cooling (FitzGibbon, 1976)
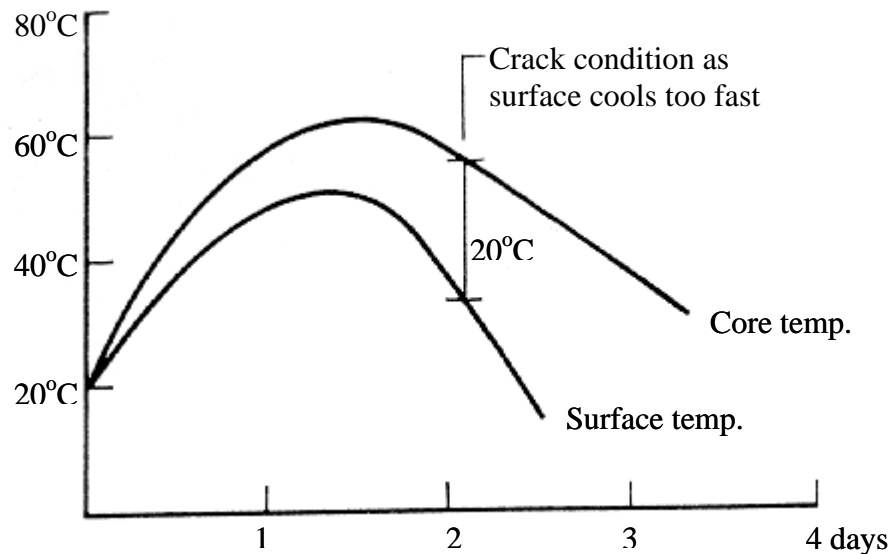
Figure 2.1 External thermal cracking

In massive concrete structures, internal restraint occurs from the inability of the

heat to dissipate quickly from the core of the member due to the low thermal diffusivity

of the concrete. A temperature differential is set up between the core of the concrete and

the surface due to the accumulation of the heat from the hydration process. The unequal

thermal expansion in the various parts of the concrete member results in stresses,

compressive in one part and tensile in the other. Cracking of the surface results when the

tensile stresses at the surface of the element due to the expansion of the core exceed

the tensile strength of the concrete. According to FitzGibbon (1976), the cracking strain of concrete is reached when an internal thermal differential of 20°C (36°F) is exceeded. Figure 2.2 shows a pattern of temperature change, which causes internal cracking of a large concrete mass. The critical 20°C (36°F) temperature is reached during heating but cracks open only when the interior has cooled through a greater temperature range than the exterior.
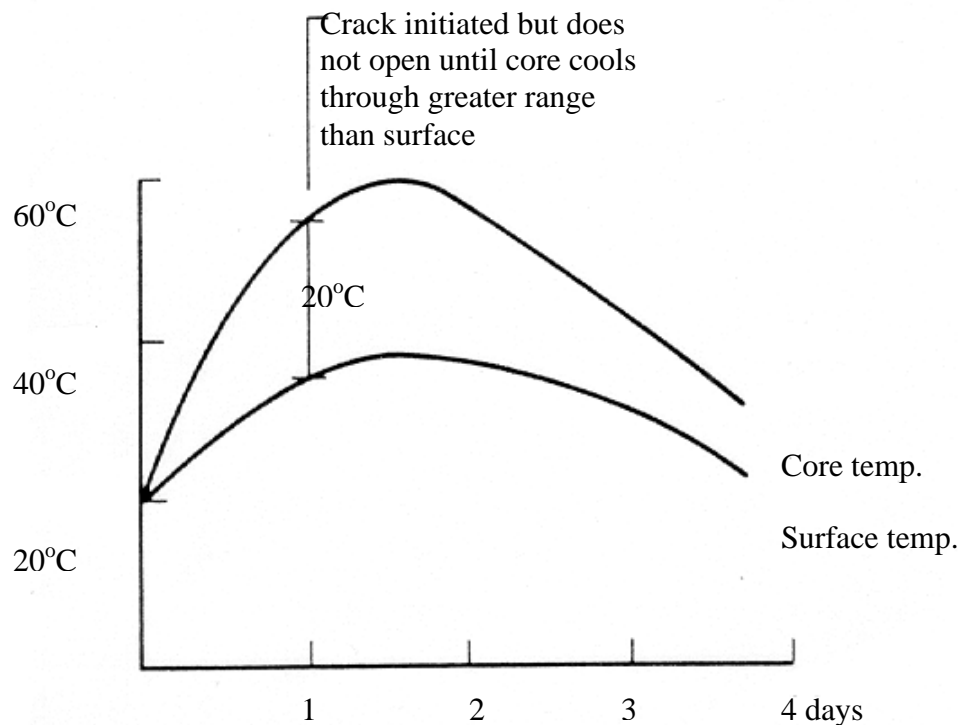
Figure 2.2 Internal thermal cracking

Cracking due to thermal behavior may cause loss of structural integrity and monolithic action or may cause extreme seepage and shorten the service life of the concrete structure. Various measure are undertaken to reduce the temperature rise in large concrete pours. Notable among these measures include:

- The prudent selection of a low-heat-generating cement system including pozzolans;

- The reduction of the cementitious content;

- The careful production control of aggregate gradations and the use of large-size aggregates in efficient mixes with low cement contents;

- The precooling of aggregates and mixing water (or the batching of ice in place of mixing water) to make possible a low concrete temperature as placed;

- The use of air-entraining admixtures and chemical admixtures to improve both the fresh and hardened properties of the concrete;

- Coordinating construction schedules with seasonal changes to establish lift heights and placing frequencies;

- The use of special mixing and placing equipment to quickly place cooled concrete with minimum absorption of ambient heat;

- Dissipating heat from the hardened concrete by circulating cold water through embedded piping;

- Insulating surfaces to minimize thermal differentials between the interior and the exterior of the concrete.

Despite the application of the above-mentioned measures to control temperature rise in concrete, maximum core temperatures of 200°F have been recorded in Florida. This high temperature have been reached while satisfying the specification for mass concrete of maintaining a maximum temperature differential of 35°F between the core and the

surface of the concrete structure. Of increasing concern is the effect on the properties of concrete when subjected to such high curing temperatures. An examination of the microstructure of concrete formed at high curing temperatures is reviewed next.

2.3 Effect of curing temperature on the microstructure of hydrated cement paste

Verbeck and Helmuth (1968) found the reactions between cement and water to be similar to any other chemical reaction, proceeding at a faster rate with increasing temperature. This rapid initial rate of hydration at higher temperatures they theorize retards subsequent hydration of the cement producing a non-uniform distribution of the products of hydration within the paste microstructure. At high temperatures, there is insufficient time available for the diffusion of the products of hydration away from the cement particles due to the low solubility and diffusivity of the products of hydration. This results in a non-uniform precipitation of the products of hydration within the hardened cement paste.

The results of a calorimetric study on the early hydration of cement as reported by Neville (1997), indicate that a heat evolution peak occurs at about 6 to 8 hours after the initialization of the hydration process at normal temperatures. This was revealed from early hydration reactions of cement, using the conduction calorimeter (Verbeck and Helmuth, 1968). During this period, the cement undergoes very rapid reactions with 20 percent of the cement hydrating over a 2 or 3-hour period. At an elevated temperature of 105°F, these reactions are accelerated with as much as 30 to 40 percent of the cement hydrating in a 2-hour period. At steam curing temperatures, 50 percent or more of the

cement hydrates in an hour or less.

Products of cement hydration have low solubility and diffusivity and at high curing temperatures, the rapid hydration does not allow for ample time for the products to diffuse within the voids. This results in a high concentration of hydration products in a zone immediately surrounding the grain. This forms a relatively impermeable rim around the cement grain, which subsequently retards any subsequent hydration (Verbeck and Helmuth, 1968). This situation does not occur in normal temperature curing where there is adequate time for the hydration products to diffuse and precipitate relatively uniformly throughout the interstitial space among the cement grains. The coarse pore structure in the interstitial space from the high temperature has a detrimental effect on the strength of concrete.

Further evidence from Goto and Roy (1981), confirms the observation by Verbeck and Helmuth (1968) of retardation of subsequent cement hydration at high temperatures. In an examination of the structure of the hydrated cement paste subjected to high temperatures in its early life, Goto and Roy (1981) found out that curing at 60°C (140°F) resulted in a much higher volume of pores larger than 150nm in diameter compared with curing at 27°C (81°F). These large pores make the concrete susceptible to deterioration from harmful substances, which are easily transported through the concrete structure.

A study by Kjelsen et al (1990) of the microstructure of cement pastes hydrated at temperatures ranging from 41°F – 122°F (5°C – 50°C) using backscattered imaging found that the low curing temperatures resulted in a uniform distribution of hydration

products and fine self-contained pores. Elevated temperatures on the other hand resulted

in a non-uniformly distributed hydration products and coarse, interconnected pores. The

microstructure of the hydrated cement paste formed at high curing temperatures affect the

strength and durability of the concrete. The large interconnected pores resulting from

high temperature curing does not make for durable concrete structures. Since strength

resides in the solid parts of a material, the presence of voids as a consequence of high

curing temperature are detrimental to the strength of the concrete.

2.4 Dense shell of hydration products

Kjellsen, Detwiler and Gjørv (1990) support the concept that a dense shell of

hydration products surrounding the cement grains is formed at higher curing

temperatures. Hydration products are more uniformly distributed at lower temperatures.

In addition, at higher temperatures of curing there are five phases as opposed to the

standard four phases at lower temperatures. The five phases are unhydrated cement,

calcium hydroxide, two densities of C-S-H and pores. The strength of the material is

greatly affected by the uniformity of the microstructure. At the elevated temperatures the

C-S-H close to the grains is much denser and stronger. However, the intervals between

the cement grains determine the strength of the concrete. Therefore curing at elevated

temperatures has a harmful effect on the later-age strength of concrete. Additionally

elevated curing temperatures according to Kjellsen, Detwiler and Gjørv (1990) result in

increased porosity. Kjellsen, Detwiler and Gjørv (1990) further noted that C_3S pastes that

were cured at higher temperatures (50ºC-100ºC) had a coarser structure, including an

increase of large pores, over those cured at 25ºC. Even steam curing (97ºC) resulted in

coarser pore structure. The difference in porosity is attributed mostly to the difference in volume of pores of radius 750-2300 A. For plain cement pastes of equal water-cement ratios cured to the same degree of hydration, the higher the curing temperature the greater the total porosity. The results indicate that large pores have the greatest effect on permeability. Permeability is a contributing component to most durability problems, therefore it is suggested that higher curing temperatures possibly reduce the durability of plain cement concretes.

2.5 Effect of curing temperature on concrete strength development

The strength of concrete is its ability to resist stress without failure. Strength of concrete is commonly considered its most valuable property. Strength usually gives an overall picture of the quality of concrete because it is directly related to the structure of the hydrated cement paste (Neville, 1997).

A rise in curing temperature according to Neville (1997) speeds up the hydration process so that the structure of the hydrated cement paste is established early. Although a higher temperature during placing and setting increases the very early strength, it may adversely affect the strength from 7 days (Neville, 1997). This is because the rapid initial hydration according to Verbeck and Helmuth (1968) appears to form products of a poorer physical structure, probably more porous, so that a proportion of the pores will always remain unfilled. Since the voids do not contribute to the strength of concrete, a low temperature with slow hydration will result in a uniform distribution of hydration products within the interstitial space and high strengths at latter ages.

A fast hydration of cement from high curing temperatures will result in a high early strength due to more hydration products being formed. At latter ages however, the retardation in hydration as a result of a dense shell around the hydrating cement grains will result in a more porous structure and reduced strengths as shown in Figure 2.3 (Verbeck and Helmuth, 1968).
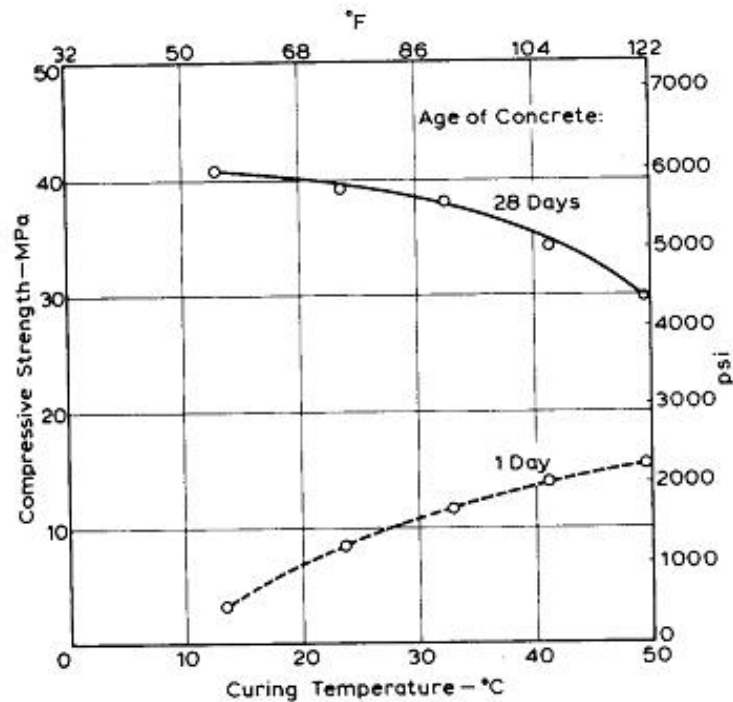
Figure 2.3 Effect of curing temperature on concrete strength development

2.6 Fresh Concrete Temperature vs. Ambient Temperature

According to Lachemi and Aïtcin (1997), often a broad spectrum of temperature variations exist in mass concrete based on the type and fineness of the cement, the effect of admixtures used, the shape of the concrete element and its aspect ratio (external surface vs. volume), the material used to make the forms, the initial temperature of the concrete, the ambient temperature, and other surrounding condition. They studied the

effects of on-site early-age temperature on high performance concrete by monitoring the

construction of a viaduct near Montreal Quebec, Canada.   They noted that some

designers specified a maximum temperature for the concrete. Various methods were

used to keep the curing temperature below the specified maximum including pouring the

concrete at night, which permits a relatively lower ambient temperature. Additionally

liquid nitrogen or crushed ice was included in the mix to produce a lower fresh concrete

temperature. However the most effective of the two methods of decreasing the maximum
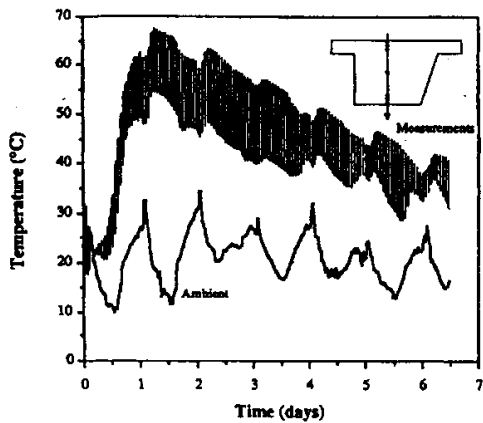
Fig. 2.4 Temperature range
through T-beam height
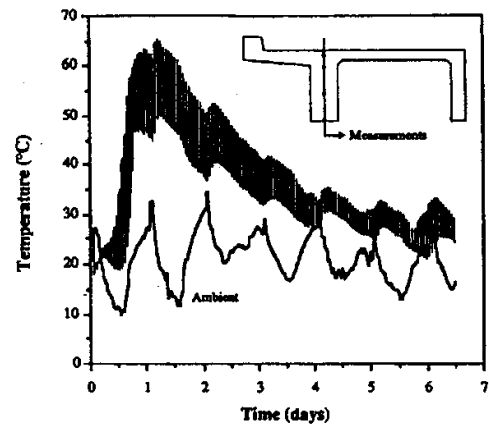
Fig. 2.5 - Temperature range
through girder height
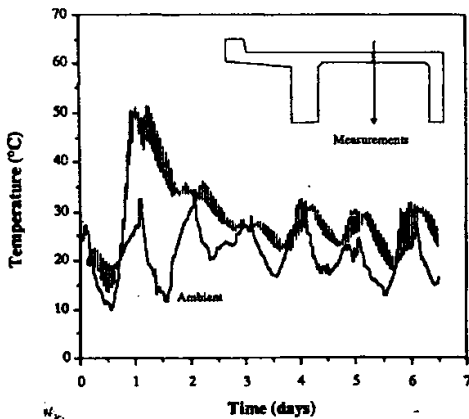
Fig. 2.6 Temperature range
through slab thickness
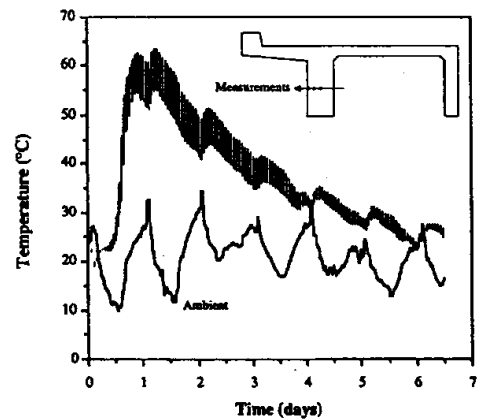
Fig. 2.7 Temperature range
through T-beam thickness

temperature of early-age high performance concrete has not been determined. The T-beams and massive girders monitored on the viaduct ranged in thickness from 24 inches to 83 inches. The air entrained high- performance concrete was placed when there was limited daylight. In addition a portion of the mixing water was substituted with crushed ice. Thirty-two thermocouples were strategically located before the placement of the concrete in order to track the thermal development of the concrete. The fresh concrete temperatures were measured hourly over 6.5 days. The ambient temperature was also monitored.

The on-site monitoring of the temperature gradient confirms that the dimensions of the concrete affect its ability to dissipate the heat generated and therefore contributing to the elevated temperature. Figures 2.4 through 2.7 record the temperature ranges for the T-beams and girders according to their respective dimensions. The temperatures recorded over the shorter dimensions (Figure 2.7) differ less than those recorded over a wider dimension (Figure 2.6). The maximum-recorded temperature was 154°F (67.5ºC), recorded for the girder height (Figure 2.5) and the maximum temperature differential was 29°F (16ºC).

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Table 2.2 Calculated peak temperature (deg C) at the center of massive girder

Fresh concrete temperature, deg C	Ambient temperature, deg C		
	10	20	28
10	58	61	63
18	67	69	70
25	75	76	77

Table 2.3 Differential values* of temperature (deg C) at the center of massive girder

Fresh concrete temperature, deg C	Ambient temperature, deg C		
	10	20	28
10	-11	-8	-6
18	-2	0	+1
25	+6	+7	+8
*(x,y) - (18,20)			

The slab was greatly influenced by ambient temperature. This could be due to the fact that the slab had a high surface to volume ratio. Therefore presenting more contact area for the ambient temperature to affect. Table 2.2 documents the effects of ambient temperature relative to fresh concrete temperature as derived from the numerical study. The TEXO module was implemented for the numerical study to reveal the true influence of fresh concrete and ambient temperatures. The tables show that the fresh concrete temperature is more influential on the increase in temperature of the concrete than the ambient temperature. Lachemi and Aïtcin from this experiment and thermal investigation on fresh water temperature versus ambient temperature concluded that

1) the rise in temperature during hydration increases as the smallest elemental dimension increases and the rate of cooling is proportional to this elemental dimension.

2) the temperature of fresh concrete has a greater influence than the ambient

   temperature or the maximum temperature of the concrete within the

   viaduct.

3) that for constant fresh concrete temperature the greater the ambient temperature

   the lesser the thermal gradient. Thus, the risk of cracking due to thermal gradients

   is less during summer that during winter.

2.7 Effect of temperature on the durability of concrete

According to ACI Committee 201, durability of Portland cement concrete is

defined as its ability to resist weathering action, chemical attack, abrasion, or any other

process of deterioration. Durable concrete will retain its original form, quality, and

serviceability when exposed to its environment. Although designers of concrete

structures have been mostly interested in the strength characteristics of concrete,

durability issues in concrete technology have been brought to the forefront in recent times

as a result of the premature failure of nondurable concrete structures.

The pore structure of the concrete determines the ease with which deleterious

harmful substances such as chloride ions are transported into the concrete. Harmful

substance such as chloride ions in concrete attack and corrode the steel resulting in

premature failure of the structure. High curing temperatures in concrete result in porous

concrete. This is because the low diffusivity of the hydration products does not allow for

uniform distribution at high curing temperatures due to the faster reaction rate. These

hydration products precipitate in the vicinity of the cement grains resulting in a more

porous concrete. At low curing temperatures, the hydration products are uniformly

distributed within the interstitial spaces making it difficult for deleterious harmful substances to be transported into the concrete

Kjellsen et al. (1990) performed an investigation of the pore structure of plain cement pastes hydrated at 41°F, 68°F and 122°F (5°C, 20°C and 50°C respectively). The specimens were tested when they reached 70% hydration, a time marking adequate development of the microstructure. Two techniques used to measure porosity in this study were mercury intrusion and backscattered electron images. They theorized that during hydration at elevated temperatures cement hydration proceeds more rapidly. Subsequently since the cement has low solubility and low diffusibility, cement hydration products are not able to disperse at a significant distance from the cement grain in the limited time provided at high temperature curing. This causes areas of dense hydration products that act as a barrier, preventing further hydration. When there is a development of dense hydration product there is also a development of greater volume of large pores and a coarser pore structure. The large pores correspond to a reduction in the modulus of elasticity of the concrete indicating increased cracking as it is exposed to structural stresses.

The curing temperature clearly affected the pore structure of hydrated cement paste as shown in Table 2.4. The higher curing temperature resulted in a greater quantity of larger pores as well as an increase in the total porosity. These results are in agreement with the observation by Goto and Roy (1981) that curing at 60°C (140°F) resulted in a much higher volume of pores larger than 150nm in diameter compared to curing at 27°C (81°F). These larger pores make the concrete more susceptible to attack by harmful

substances since they provide an easier pathway through the concrete. Permeability is a

contributing factor to various kinds of durability problems, therefore suggesting that high

curing temperatures could reduce the durability of plain cement concretes. The increased

permeability also leads to increased water intrusion to the reinforcing steel and promoting

an increase to the rate of corrosion of the members.

Table 2.4 Measured Porosity

Curing temperature	Porosity (MIP + HP)	Porosity (BSEI)	Standard deviation
41ºF	33.2%	4.27%	.818%
68ºF	34.2%	10.93%	1.086%
122ºF	35.7%	15.11%	1.881%

Campbell and Detwiler (1993) explain that the durability of concrete is a primary

contributor to its satisfactory performance.   Agencies typically control the durability of

concrete by restricting the water-cement ratio to 0.45 or less. However, the curing

process is often overlooked, though it also affects the durability of the concrete. A basic

principle noted is that the Portland cement concretes resistance to penetration by chloride

ions is reduced due to coarsening of the cement paste pore structure. Specifying a low

water-cement ratio provides limited effectiveness in bettering the performance of the

concrete.

2.8 Fly ash and Slag in concrete

Class F fly ash is an artificial pozzolanic material, which possesses no

cementitious value, but in finely divided form, in the presence of moisture, chemically

reacts with calcium hydroxide from the Portland cement reaction to form compounds possessing cementitious properties. The fly ash reaction products closely resemble the calcium silicate hydrate produced by hydration of Portland cement (Neville, 1997). The fly ash reaction does not start until sometime after mixing. According to Fraay et al (1989), the glass material in fly ash is broken down only when the pH value of the pore water is at least about 13.2. The increase in alkalinity required for the fly ash reaction is achieved through the reaction of the Portland cement. At high temperatures, the fly ash reaction takes place sooner due to the increased hydration rate of the cement. Prior to the reaction of the fly ash particles, they act as nuclei for the precipitation of the cement hydration. When the pH of the pore water becomes high enough, the products of reaction of fly ash are formed on the fly ash particles and in their vicinity. With the passage of time, further products diffuse away and precipitate within the capillary pore system, this result in a reduction of the capillary porosity and consequently a finer pore structure (Fraay et al, 1989). Figure 2.8 shows the changes in pore size distribution determined by mercury porosimetry, in cement paste containing 30 percent of Class F fly ash by means of total cementitious material (Fraay et al, 1989). The cement paste becomes increasingly denser after the initiation of the pozzolanic reaction of fly ash.
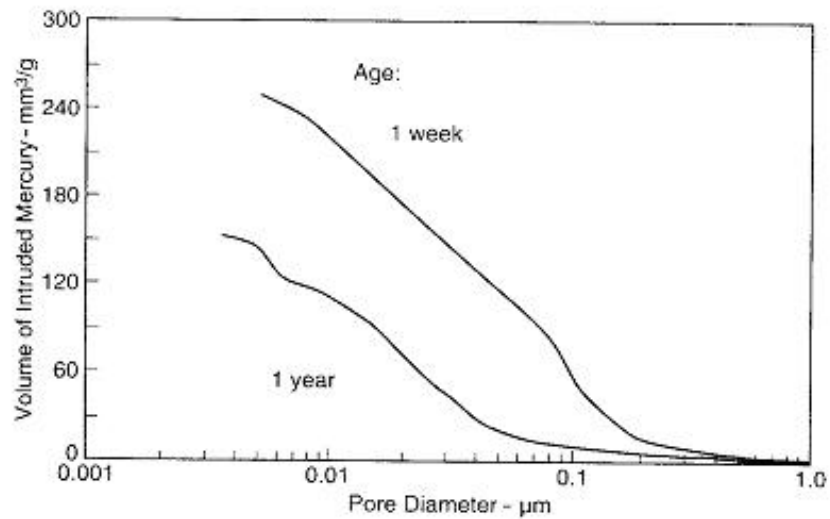
Figure 2.8 Pore size distribution with age for 30% Fly ash mix

Slag is a waste product in the manufacture of pig iron. Chemically, slag is a mixture of lime, silica and alumina, the same oxides that make up Portland cement (Neville, 1997). Compared to the fly ash, finely ground granulated blast-furnace slag is self-cementing. It does not require calcium hydroxide to form cementitious product such as calcium silicate hydrates. When used on its own, the amounts of hydration products formed by the blast-furnace slag is insufficient for application of the material to structural purposes. Used in combination with Portland cement, the hydration of the slag is accelerated in the presence of calcium hydroxide and gypsum (Mehta and Monteiro, 1993). The beneficial effects of slag arise form the denser microstructure of the hydrated cement paste, more of the pore space is filled with the hydration products than in cement only mixes.

Supplementary cementing materials are suggested to increase the performance of the concrete. Campbell and Detwiler investigated the optimum mix design for

satisfactory strength and durability of steam-cured concrete with 0 .45 water-cement ratio

and various compositions of Canadian Type 10 cement (ASTM Type I) with slag and

silica fume. The compressive strength of the cylinders after 18 hours of steam curing and

one day of moist curing were compared. The results as shown in Table 2.5 reveal that

slag is effective in reducing the rate of chloride ion diffusion and therefore increasing the

durability of the concrete. However the mixes with silica fume and slag, or silica fume

alone were more durable.

Table 2.5 - Results of compressive strength and AASHTO T-277 test

Mix no.	Description	Compressive strength MPa	Total Charge passes, coulombs, average of three slices	Rating
M1	Control:100% PC	27.3	11130*	
M2	30% Slag	25.3	7800	High
M3	40% Slag	27.9	7690	
M4	50% Slag	28.9	4500	
M5	5.0% SF	32.6	1780	Low
M6	7.5% SF	33.3	910	Very Low
M7	10.0% SF	36.4	290	
M8	30% Slag; 7.5% SF	28.5	350	
M9	40% Slag; 7.5% SF	31.3	200	
M10	30% Slag; 10% SF	34.5	150	
*Extrapolated value.				

Detwiler, et al. (1994) investigated the chloride penetration of 0.4 and 0.5 water-cement ratio concretes containing either 5 percent silica fume or 30 percent slag (substitution by mass) cured at elevated temperatures. They found that higher curing temperatures resulted in greater penetration of chloride ions. In addition, at any given temperature, both the silica fume and slag concretes performed better than the Portland cement concrete. Their studies showed that the use of pozzolanic materials is more effective than lowering the water-cement ratio from 0.5 to 0.4 in improving the resistance to chloride ions (Tables 2.6 and 2.7).

Table 2.6 AASHTO T-277 tests for charge passed

Mix	w/c	73ºF	122ºF	158ºF
Portland	.40	5700	12,000†	18,000†
Cement	.50	9800	13,000†	16,000†
5 % Silica	.40	1500	3000	4100
Fume	.50	1800	3400	13,000
30% Slag	.40	1300	1500	4300
	.50	1700	2200	5400

*Charge(coulombs) passed in 6 hr for concretes cured at constant temperatures indicated to degree of hydration of approximately 70 percent.
†Extrapolated values. These tests were terminated before the full 6 hr had elapsed due to excessive temperature increases.

Table 2.7 Rate of chloride diffusion ppm/day (average of three replicates, Norwegian test)

Concrete	w/c	73ºF	122ºF	158ºF
Plain Cement	.40	10	12	34
	.50	13	15	38
5 % Silica Fume	.40	4	7	12
	.50	3	5	22
30% Slag	.40	3	4	13
	.50	6	7	18

2.9 Delayed Ettringite Formation (DEF) in concrete

Recent research has identified the formation of delayed ettringite in hardened concrete cured at temperatures above 158°F to be a cause of premature deterioration of concrete structures. Core temperatures of 200°F have been recorded during curing of mass concrete structure in Florida increasing the likelihood of damage due to the delayed formation of ettringite. There is the need to investigate the phenomenon delayed ettringite formation in mass concrete in order to develop measures to avoid its damaging effects.

Ettringite (Calcium Aluminate Trisulphate Hydrate) is a normal and apparently innocuous constituent of hydrated Portland cement. Its formation at the initial stages of hydration is seen as a positive effect because it enables the setting of the cement, however a damaging role is attributed to its formation in hardened concrete. Delayed Ettringite Formation (DEF) is the term given to the formation of ettringite in a cementitious material by a process that begins after hardening is substantially complete and in which none of the sulphate comes from outside the cement paste. DEF takes place in

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concretes subjected to high temperatures above 158°F during curing. Conditions leading to temperatures above 158°F in concrete include heat treatment, concrete placement under elevated temperatures such as during summer weather and heat liberated during hydration in massive concrete elements. In the course of heat curing at temperatures above 158°F, calcium aluminate monosulphate hydrate is formed as the sole product of hydration of tricalcium aluminate. After the heat curing has been concluded and the temperature drops, the monosulphate becomes metastable so that, if there is sufficient water available reprecipitation of ettringite occurs in voids and crack surfaces.

Possible damage mechanisms on ettringite formation in hardened concrete according to Stark and Bollmann, are as follows:

- The primary ettringite formed during the initial hydration does not lead to damage, because this ettringite formation occurs in the plastic matrix and thus no stresses will be produced.

- If the ettringite formed primarily or delayed inside the microstructure is microcrystalline, then in hardened concrete, it may develop an expansion pressure due to adsorption of water, which can cause damages if the tensile strength of the microstructure is exceeded.

- DEF formed in hardened concrete subjected to higher temperatures during initial curing causes damages to the structure. These damages are due to the stresses from the crystal growth or increases in volume, which exceed the tensile strength of the structure. The transformation of monosulphate into ettringite causes a 2.3 times increase in volume.

- The recrystallization of ettringite in the hardened structure, due to moisture changes and accumulation of reactants, may lead to structure damages because of the crystallization pressure and the increase in volume.

The sudden emergence of DEF as a distress mechanism in concrete cured at high temperatures can be attributed to three possible reasons:

1. It has been misidentified in the past as alkali-silica reaction (ASR)

2. Only recently have clinker sulphate concentrations reached several percent; and

3. Only recently have total sulphate concentrations exceeded 4%.

CHAPTER 3 SURVEY OF US TRANSPORTATION AGENCIES

3.1 Introduction

The Florida Department of Transportation currently has a specification that includes mass concrete. The measures taken by the FDOT to produce a mass concrete product with the required qualities include controlling the differential temperature of the concrete during the curing process. The contractor is required to maintain a temperature differential of no more than 35°F between the surface of the concrete and the core of the concrete in the shortest direction. The pending question is "should a maximum temperature for mass concrete curing be specified?" This specification would require the contractor to limit the heat of the concrete at all times to a maximum temperature. A survey was developed to acquire information about parameters outlined in various mass concrete specifications in order to produce a concrete with the required strength and durability. The agencies were asked to provide their current specifications and their opinion on this matter.

3.2 Survey Methodology

The survey was sent to fifty-one highway agencies in the United States (including Puerto Rico). Forty-three highway agencies responded to the survey, representing a response rate of eighty-four percent. It was requested that the agencies provide a copy of their current specification on mass concrete. Each agency was also asked to give the specified differential temperature for mass concrete (if any) and the specified maximum curing temperature (if any). However, some agencies reported placement temperatures or ambient temperature as opposed to differential temperature or maximum allowable

temperature during the entire curing process. Of the agencies that responded, twenty-one percent had a specification, which directly addressed mass concrete pours.

The states that responded to the survey regarding mass concrete specification are shown on Table 3.1 and Figure 3.1. Sixty percent of the agencies that responded did not have a specification concerning mass concrete, and twenty percent provided controls for the mass concrete pour by special provisions on a project-to-project basis. The data gathered is analyzed according to three survey categories: agencies with mass concrete specifications, agencies without mass concrete specifications, and agencies with special provisions. Most agencies without specifications for mass concrete or some of those that used special provisions did not have much experience with mass concrete. The concrete work typically performed by these agencies did not include mass concrete pours. In addition, states with predominately cold climates had greater concern for low concrete temperatures as opposed to high concrete temperatures. For these reasons, the results were evaluated according to the aforementioned categories. The agencies that which currently have mass concrete specifications, also have experience with mass concrete pours.   The information provided by the different groups will be analyzed considering the mass concrete experience of the agency.

Table 3-1 U.S. Highway Mass Concrete Specification Survey Response

	With specification (Yes)	Without specification (No)	Special Provisions	
	CA	AK	AR	
	FL	AZ	CT	
	ID	DC	GA	
	IL	DE	IA	
	KY	HI	MN	
	NC	IN	MO	
	SC	KS	ND	
	TX	LA	NE	
	VA	MD		
		ME		
		MI		
		MS		
		MT		
		NH		
		NJ		
		NM		
		NV		
		NY		
		OK		
		OR		
		PA		
		PR		
		SD		
		UT		
		WA		
		WI		
Responses	9	26	8	43
%	21%	60%	19%	100%
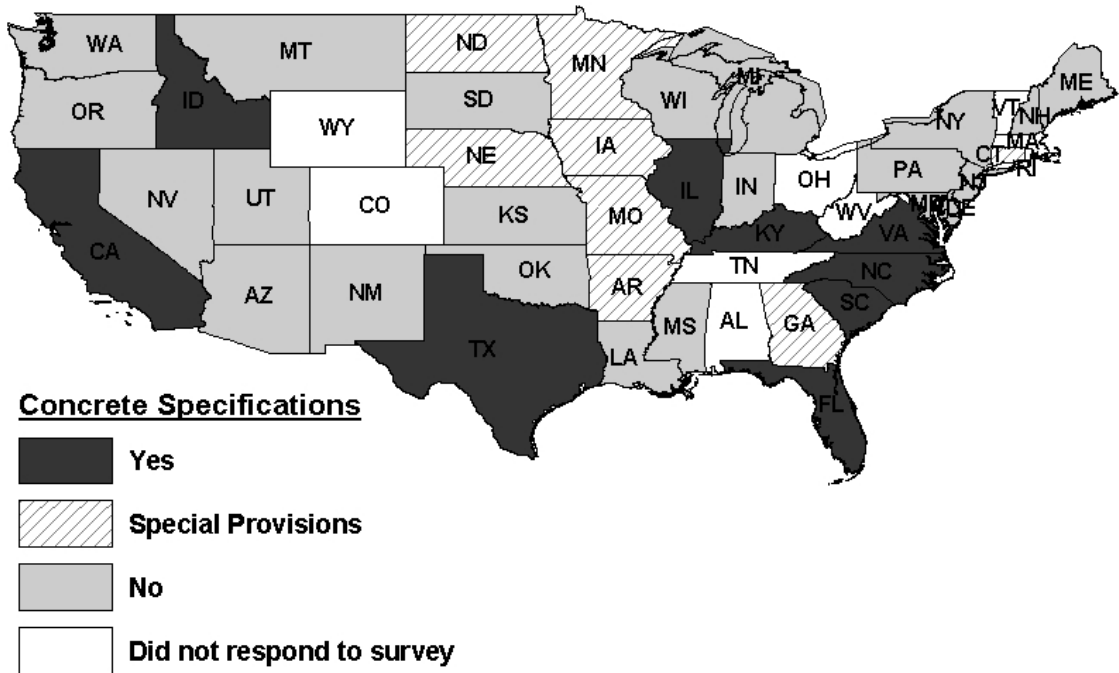
Figure 3.1 U.S. Highway Mass Concrete Specification Survey Response.

3.3 Agencies with Mass Concrete Specifications

As previously mentioned, nine agencies that responded to the survey have a mass concrete specification. The survey revealed that of these nine agencies seven agencies specify a maximum differential temperature and two specify a maximum curing temperature for mass concrete. The differential temperature is most commonly 35ºF and the maximum curing temperature is 160ºF. Three US Highway Agencies have a maximum concrete placement temperature of 75°F or 80°F. Eight agencies have

identified a need to specify a maximum curing temperature for mass concrete. The need

for a maximum curing temperature is explained by the later age strength reduction,

durability problems and early concrete distress due to delayed ettringite formation (DEF).

Table 3.2 indicates the states that have a specified maximum differential temperature, a

specified maximum curing temperature and the opinion of the agency regarding the need

to specify a maximum temperature.

Table 3.2 U.S. Highway Agencies with Mass Concrete Specification Survey Response.

State	Max. Diff. Temp (^{o}F)	Max. Temp. (^{o}F)		Should Max. Temp. for mass concrete be specified	
		Placing	Curing	Opinion	Reason
CA	-	-	-	Yes	
FL	35	-	-	Yes	
ID	35	-	-	Yes	Strength and durability problems
IL	35	-	160	Yes	Later age strength reduction and microstructure
KY	35	-	160	Yes	
NC	36	75	-	Unsure	
SC	35	80	-	Yes	Reduction in later-age strength
TX	35	75	-	Yes	Early concrete distress due to DEF
VA	-	-	-	Yes	

3.4 Agencies without Mass Concrete Specifications

Approximately fifty-eight percent of the agencies surveyed do not currently have a mass concrete specification. Since there are no current specifications from these agencies, neither maximum temperature nor differential temperature for mass concrete is specified.   However, of this group, forty-six percent believe that a maximum temperature for mass concrete pours should be specified. The recommended temperatures were 130ºF and 160ºF. These maximums were considered necessary to avoid later strength reduction, durability problems and delayed ettringite formation (DEF). In contrast, twenty-two percent of the agencies did not perceive a need for a maximum specified temperature. These agencies preferred to specify the end result of the concrete instead of the means used to mix, place, pour or cure the concrete. The specifications noted the quality of concrete required instead of controlling the process of the pour. In addition some were assured that the differential temperature was enough to provide the strength and durability required from the concrete.

3.5 Agencies with Special Provisions for Mass Concrete

Eight agencies noted that mass concrete pours under their jurisdiction were controlled through special provisions. An outside consulting engineer on a project-by-project basis often prepared these special provisions. The Nebraska Department of Roads implements their specifications for Precast/Prestressed Concrete Structural Units for the curing of mass concrete structures. The survey results from these agencies are documented in Table 3.3. In the special provision provided for mass concrete six agencies included a differential temperature ranging from 35ºF to 50ºF. Three

included a maximum temperature ranging from 160º F - 176ºF. In addition five agencies

believed that a maximum specified temperature was necessary for long-term strength and

durability. The three agencies that did not see a need for maximum temperature,

mentioned that a small percentage of mass concrete work was performed within their

jurisdiction or that the differential temperature was a sufficient measure.

3.6 Summary of State Highway Agency Specifications

Of the nine highway agencies that stated that their office currently had highway

specification for mass concrete, seven specifications were reviewed. The agencies that

submitted a copy of the state specifications were the Kentucky Transportation Cabinet,

North Carolina Department of Transportation, South Carolina Department of

Transportation, Idaho Transportation Department, State of California-Department of

Transportation, Texas Department of Transportation, Florida Department of

Transportation and Illinois Department of Transportation. The specifications ranged in

length from two paragraphs to four pages. The Florida Department of Transportation

specification was readily available in its entirety and was also familiar to the reviewing

body. The survey requested that the participants submit specifications specific to mass

concrete. It is important to note that the specification submitted must not be considered as

the complete body that defines the quality of mass concrete. Other divisions may possibly

give parameters that better define the requirements for mass concrete within the

respective state. Of the specifications submitted, the State of Kentucky's specifications

were most specific. The review that follows is formatted according to the Kentucky

specifications. This format was referenced because it provided a clear and concise

method of stating the requirements and controls. The state of Kentucky mass concrete

specifications identify the requirements for cement, aggregate, temperature sensing

equipment, construction methods, thermal control plan, temperature differential

restrictions, temperature sensing and recording, trial mixtures, acceptance testing, and

payment adjustments for mass concrete that did not comply with the specification. All

other agencies included select items of the above noted categories.

Table 3.3 U.S. Highway Agencies with Special Provisions for Mass Concrete

State	Max. Diff. Temp (^{o}F)	Max. Temp. (^{o}F)		Should Max. Temp. for mass concrete be specified	
		Placing	Curing	Opinion	Reason
AR	36	75	-	No	Maximum differential temp will govern
CT	No answer	-	-	No opinion	
GA	50	-	-	No	Mass concrete small percentage of work
IA	35	65	-	Yes	Long term strength and durability (DEF)
MN	35	-	160	Yes	ASR at high core temperatures
MO	No answer	-	-	Yes	
ND	50	-	160	Yes	Long term strength and durability
NE	27	-	176	Yes	Damaging stress in concrete

Table 3.4 Opinions on Maximum Curing Temperature.

Should there be a specified maximum temperature for mass concrete?			
State	Opinion	%	Reasons
AZ	Yes		
DE	Yes		
KS	Yes		130 max to avoid durability problems
LA	Yes		To avoid DEF (Delayed Ettringite Formation)
ME	Yes		To avoid later strength reduction
MI	Yes		
MS	Yes		To reduce cracking due to expansion of concrete
NJ	Yes		Article provided on the Effect of Elevated temp.
NV	Yes		
NY	Yes		
OK	Yes		
OR	Yes		To minimize swelling and shrinkage cracking
UT	Yes		160 max to avoid DEF
WI	Yes		160 max to avoid DEF
KY	Yes		
IL	Yes		Later age strength reduction and microstructure
SC	Yes		Reduction in later-age strength
TX	Yes		Early concrete distress due to DEF
NH	Yes		
VA	Yes		
CA	Yes		
ND	Yes		Long term strength and permeability
MN	Yes		ASR at high core temperatures
NE	Yes		Damaging stress in concrete
MO	Yes		
ID	Yes		Strength and durability problems
FL	Yes		
IA	Yes	65%	Long term strength and durability (DEF)
GA	No		Mass concrete small percentage of work
AR	No		Maximum temp diff. will govern
IN	No		
MD	No		
NM	No		A maximum temp Diff. Would be appropriate
WA	No	14%	
NC	Unsure		
AK	Unsure		
MT	Unsure	7%	
CT	No Opinion		
DC	No Opinion		
HI	No Opinion		
PA	No Opinion		
PR	No Opinion		
SD	No Opinion	14%	

3.7 Review of Available Mass Concrete specifications

3.7.1 California Department of Transportation

Specification Reference: XE "51MASS_R07-01-99"

Definition

Mass structural concrete is defined as all concrete used in the portions of

structures where the concrete being placed has a minimum dimension that exceeds 2 m.

Aggregate

Aggregate for mass concrete shall conform to the 37.5mm maximum combined

aggregate grading.

Cement

Cement for mass concrete shall be Type 2 Modified and in addition the sum of the

Tricalcium Silicate and Tricalcium Aluminates shall not exceed fifty-eight percent in

accordance with ASTM Designation: C150. Mass concrete shall contain 375kg of

cement per cubic meter. The amount of free water used in mass concrete shall not exceed

184kg per cubic meter. Either Type A admixture or a type D admixture conforming to

the requirements of ASTM Designation: C494 and Section 90-4 shall be used.

Thermal Control

The temperature of the concrete at the time of discharge from the mixer shall not

exceed 64.4ºF except when ice is substituted for one hundred percent of the mixing

water, which in this case the temperature of the concrete shall not exceed 69.8ºF. The

minimum temperature requirements of Section 90-6.02, " Machine Mixing," or the

Standard Specifications will not apply. When ice is used, all the ice shall be melted before discharging the concrete from the mixer.

Construction

After mass concrete pours has been topped out and finished it shall be re-vibrated and refinished. Re-vibration shall extend below the top mat of reinforcement and shall be done as late as the concrete will again respond to vibration. For concrete pours without top reinforcement, re-vibration shall extend to a depth of 150 mm.

3.7.2 Idaho Transportation Department

Specification Reference: 502.03

Definition

Footing thicker than 3.93 ft. will be considered as massive placement.

Thermal Control

The maximum temperature difference shall be 35°F between all points across the top surface of a footing, during placing and throughout the full seven-day curing period of the concrete. The Contractor shall propose for approval, construction methods that will achieve this uniformity of temperature and if any methods prove inadequate, the Contractor shall adopt different and/or additional measures as necessary to achieve the uniformity.

Construction

Special measures shall be taken to minimize the possibility of drying shrinkage cracks developing in massive footings during placing and curing of the concrete.

3.7.3 Illinois Department of Transportation

Definition

Large concrete shall be defined as any concrete pour which has a least dimension of 4 feet or greater.

Thermal Control

The Contractor will develop a procedure that, during the period of heat dissipation following concrete placement, the temperature differential between the interior of the section and the outside surface of the section does not exceed 35ºF. The maximum temperature will be 160ºF. The contractor shall provide and install temperature sensing devices of type approved by and at location as designated by the Engineer. Procedures open to the contractor include, but are not limited to, the following:

Use of Type IV cement.

Cooling component materials prior to addition to the mix

Adding ice to mix water.

Controlling rate of concrete placement.

Other acceptable methods, which may be developed by the Contractor.

3.7.4 Kentucky Transportation Cabinet

Definition

The department considers mass concrete to be any concrete placement with its least dimension being 5 feet or greater.

Aggregate

Use coarse aggregate conforming to the freeze-thaw expansion requirements of Subsection 805.04.01 for use in all classes of structural mass concrete.

Cement, Pazzolans, additives

Use of Type II or IV Portland cement, conforming to the chemical requirements in ASTM C 150, Table 2.

Thermal Control

The temperature differential between the geometric center of each placement and the geometric surface does not exceed 35°F at any time. Maintain thermal control of each placement until the temperature at the center is within 35°F of the average outside air temperature. Determine the average outside air temperature by averaging the daily high and low temperatures over the preceding 7 calendar days. The department will allow the inclusion of the following items in the Thermal Control Plan.

1) Sprinkle mixer trucks for cooling

2) Arrange with supplier to avoid delivery of hot cement.

3) Cooling of aggregate stockpiles

4) Use of a nitrogen gas cooling system to cool the concrete mass before placement.

5) Use of shaved, flaked, or chipped ice as part of the mixing water.

6) Embedment in the structural mass concrete of a cooling system, approved by the engineer, consisting of non-corrosive piping and circulating fresh

water. Filing of the pipe with concrete or grout after its usefulness has
ended is required.

7) Placing concrete during the coolest part of the day, or during cooler
weather.

8) Use of special cements or additives that will reduce heat of hydration
without affecting strength or durability.

Construction

The following requirements are mandatory for all structural mass concrete placements
on the project:

1) Submit and follow a Thermal Control Plan

2) Produce trial batches for each class of concrete

3) Include at least the minimum cement content specified in each design.

4) Substitute Class F fly ash for cement at the rate of twenty-five to thirty
percent of the minimum cement content for all structural mass concrete.
Apply a substitution rate of 1.0 to 1.25 lbs of fly ash added for each 1.0 lbs
of cement removed.

5) When placing the mixture, do not allow its temperature to exceed 70°F.

6) Insulate the concrete until the thermal cure is finished

7) Do not allow the concrete to exceed the maximum temperature of 160°F at
any time during the curing period.

3.7.5 North Carolina Department of Transportation

Definition

   Mass concrete elements are those as designated in the contract plans.

Cement, pozzolans, additives

   Class AA, vibrated, air-entrained, and shall contain an approved set-retarding, water-reducing admixture, and thirty percent fly ash and five percent microsilica by weight of the total cementitious material. The total cementitious material shall not exceed 410 kg per cubic meter of concrete. The maximum water-cementitious material ratio shall be 0.366 for rounded aggregate and 0.410 for angular aggregate. Cement shall be Type II meeting requirements of Subarticle 1024-1(B) of the standard Specifications.

Thermal Control

   The contractor shall provide an analysis of the anticipated thermal developments in the mass concrete elements using his proposed mix design, casting procedures, and materials. Additionally, the Contractor shall describe the measures and procedures he intends to use to limit the temperature differential to 36°F or less between the interior and exterior of the designated mass concrete elements during curing. The temperature of mass concrete at the time of placement shall not be less than 39.2°F nor more than 75.2°F. Select concrete ingredients such that it minimizes the heat generated by hydration of the cement. Maintenance of the specified thermal differential may be accomplished through a combination of the following:

1) Cooling component materials to reduce the temperature of the concrete while it is in its plastic state.

2) Controlling the rate of placing the concrete.

3) Insulating the surface of the concrete to prevent heat loss.

4) Providing supplemental heat at the surface of the concrete to prevent heat loss.

5) Other acceptable methods developed by the Contractor.

Construction

The placement of the mass concrete shall be continuous. The contractor shall provide and install a minimum of six temperature-sensing devices in each mass concrete pour to monitor temperature differentials between interior and exterior of the pour. The monitoring devices shall be read and reading recorded at one-hour intervals, beginning when casting is complete and continuing until the maximum temperature is reached and two consecutive readings indicate a temperature differential decrease between the interior and exterior of the element.

3.7.6 South Carolina Department of Transportation

Specification Reference: 702.16

Definition

Mass concrete is defined as any pour in which the concrete being cast has dimensions of 5 feet or greater in three different directions or any pour with a circular cross-section of 6 feet or greater and a length of 5 feet or greater.

Cement

If the contractor is proposing a special concrete mix design as part of the temperature control plan, this mix design should also be submitted for review.

Thermal Control

For all mass concrete pours, the mix temperature shall not exceed 80°F as measured at discharge into the form. Further, the Contractor shall be required to maintain a temperature differential of 35°F or less between the interior and exterior of all mass pour elements during curing. The contractor shall provide temperature-monitoring devices to record temperature development between the interior and exterior of the pour. Before placement the contractor is required to submit a Mass Concrete Placement Plan containing an analysis of the anticipated thermal development, the specific measures to be taken to control the temperature differential and details of the proposed monitoring system.

Construction

Mix temperature shall not exceed 80°F as measured at discharge into the forms. The contractor shall be required to maintain a temperature differential of 35°F or less during curing. The contractor shall provide temperature monitoring devices to record temperature development between the interior and exterior of the element at points approved by the Engineer and shall monitor the mass pours to measure temperature differential. Temperature monitoring shall continue until the interior temperature is within 35°F of the lowest ambient temperature or a maximum of two weeks.

3.7.7 Texas Department of Transportation

Specification Reference: Pg. 588

Definition

Mass concrete is defined as monolithic mass placements having a least dimension greater than 5 feet.

Cement/Aggregate

Concrete ingredients should be selected to minimize heat of hydration. Fly ash may be used in the mix design.

Thermal Control

The temperature differential between the central core of the placement and the exposed concrete surface shall not exceed 35ºF. A detailed plan, along with an analysis of associated heat generation and dissipation shall be submitted to the Engineer for approval. The detailed plan may incorporate the use of ice or cooling concrete ingredients, controlling the rate of concrete placement, using supplemental heat to control heat loss and using insulation to control heat loss.

3.7.8 Florida Department of Transportation

Definition

Mass concrete is designated in the FDOT Structures Design Guidelines.

Cement, Pozzolans, additives

Slag may be used as a substitute for cement at 50 to 70 percent. Fly ash when used as a substitute for cement by weight must be a minimum of eighteen percent and

a maximum of fifty percent. The mass concrete mix design shall be submitted to the State Materials Engineer for approval.

Thermal Control

A maximum temperature differential of 35°F or less shall be maintained between the interior and exterior portions of the designated mass concrete elements during curing. The anticipated thermal development of the proposed mix shall be provided to the State Materials Engineer for approval. The proposed plan to control and monitor the temperature differential shall also be submitted concurrently to the State Materials Engineer for approval.

Construction

The contractor shall provide temperature monitoring devices, which shall be read by the Contractor and reading recorded at not greater than six-hour intervals, as approved by the Engineer, beginning when casting is complete and continuing until the maximum temperature differential is reached and begins to drop.

CHAPTER 4 RESEARCH METHODOLOGY

4.1 Introduction

This chapter presents the materials, mixtures, and test methods used to evaluate the effects of elevated curing temperatures on the strength, durability and formation of Delayed Ettringite (DEF) in mass concrete. The work was divided into three phases as follows:

a. <u>Phase I.</u> In phase 1 of this study, three mixes of pastes comprising plain cement, cement with 18% fly ash and cement with 50% fly ash were cured at temperatures of 73^oF, 160^oF and 200^oF for various durations to determine the age at which a maturity of 70% degree of hydration of the cement was attained. Once this age was determined for the various mixes and curing temperatures, mass concrete with binders in the same proportions as in the paste would be made and tested when they reached 70% degree of hydration. This would ensure that all the mass concrete properties would be determined at the same maturity and make for easy comparison. Difficulty in establishing and exact time to reach 70% degree of hydration as well as inability to reach this maturity in the cement/fly ash mixes resulted in using the curing durations of 7, 28 and 91 days as the bases of comparing the mass concrete properties.

b. <u>Phase II.</u> In phase II, four FDOT Class IV mass concrete mixtures were made and cured at temperatures of 73^oF, 160^oF and 180^oF for durations of 7, 28 and 91 days. The concrete samples were tested to determine the following properties:

    i. Compressive strength – ASTM C 39 (ASTM 1996)

        ii.   Resistance to chloride penetration – ASTM C 1202 (ASTM 1994)

       iii.   Time to Corrosion – FM 5-522

       iv.   Density and percentage of voids – ASTM C 642 (ASTM 1997)

c. <u>Phase III.</u> This phase involved microstructure analysis of the mass concrete by the aid of a scanning electron microscope. Mortar samples sieved from the concrete mixes were subjected to the same curing regime. At each test age, the mortar samples were removed and placed in methanol to stop further hydration of the cement. After a minimum of 7 days in the methanol, ¼ inches thick wafers were cut from the samples. These wafers were fractured and examined to determine the presence or lack of ettringite crystals.

4.2 Degree of Hydration

<u>4.2.1 Introduction</u>

A well-hydrated Portland cement paste consists mainly of calcium silicate hydrates, calcium sulphoaluminate hydrates and calcium hydroxide (Metha and Monteiro, 1993). When the cement paste is ignited to a temperature of 1832°F (1000°C), the nonevaporable water chemically combine in the hydration products is released. The degree of hydration is a measure of the nonevaporable water content of the paste expressed as a percentage of the nonevaporable water content of fully-hydrated cement paste. The nonevaporable water content of fully-hydrated cement paste is 0.23 grams of water per gram of cement (Basma et al, 1999).

For this study a degree of hydration of 70% was decided as the maturity level at which the mass concrete properties would be determined. The choice of 70% degree of

hydration was based on a study by Kjellsen et al (1990) who found that the time required to attain this level of maturity is not so long as to be impractical to replicate in the laboratory. Additionally, by this point, the rate of hydration has slowed enough that small variations in curing time will not result in significant error making for easy comparison of the various samples.

4.2.2 Methodology

Tables 4.1 and 4.2 show the chemical composition and physical properties of the cement, fly ash and blast furnace slag used in the study. The Portland cement used was AASHTO Type II. Described here are the methods applied to determine the time to attain 70% degree of hydration for three paste mixes isothermally cured at temperatures of 73ºF, 160ºF and 200ºF. The three paste mix designs tested are as follows

1. Plain cement paste mix
2. Cement and 18% Fly ash paste mix
3. Cement and 50% Fly ash paste mix

Table 4.1 Properties of Cement and Fly ash

Chemical Composition	Portland Cement	Fly Ash
% Silicon Dioxide (SiO_2)	20.6	86.9
% Aluminum Oxide (Al_2O_3)	5.1	
% Ferric Oxide (Fe_2O_3)	4.7	
% Magnesium Oxide (MgO)	0.7	-
% Sulfur Trioxide (SO_3):	3.2	0.2
% Tricalcium Silicate (C_3S)	50.0	-
% Tricalcium Aluminate (C_3A)	5.6	-
% Total Alkalis as Na_2O	0.52	-
% Insoluble Residue	0.12	-
Loss of Ignition (%)	1.5	3.2
Physical Properties		
Fineness:	Blaine (m^2/kg) - 341	#325 Sieve - 34%
Time of Setting (Gilmore): Initial (Minutes) Final (Minutes)	145 235	-
Compressive Strength (PSI): 3 Days 7 Days 28 Days	3200 4070 -	- - 71%

Table 4.2 Properties of Blast furnace slag

Chemical Analysis	Slag
% Silicon Trioxide (SiO_3)	2.3
% Sulfide Sulfur	0.9
Slag Activity Index	
7 Days	96%
28 Days	132%
Physical Properties	
Fineness: #325 Sieve (45um)	2%
Compressive Strength (PSI):	
7 Days STD Average	4750
7 Days Slag Average	4380
28 Days STD Average	5900
28 Days Slag Average	7810
Blast furnace slag produced by Lafarge in Tampa	

Table 4.3 Mix proportions of paste mixes

Mix design	Cement (lbs)	Fly Ash (lbs)	Water (lbs)	w/b ratio	Mixing Water ºF
18% fly ash at 73ºF	3.540	.777	1.77	.41	73
18% fly ash at 160ºF and 200ºF	5.057	1.110	2.53	.41	136
50% fly ash at 73ºF	2.467	2.467	2.02	.41	73
50% fly ash at 160ºF and 200ºF	3.083	3.083	2.53	.41	136

The proportions of materials used in the paste mixes are shown in Table 4.3. The

pastes were made in accordance to ASTM C 305-99. The procedures followed to

determine the degree of hydration were as follows:

a. Three samples each was made of each paste mix to be tested at each curing period and temperature. Samples at 73ºF were tested at ages of 1, 3, 7, 10, 14, 28 and 56 days. Samples at 160ºF and 200ºF were tested at ages of 1, 3, 7, 10 and 14 days.

b. The water used for samples cured at 160ºF and 200ºF was preheated to 136ºF, to produce a cement paste with temperature of approximately 98ºF. This was done to reduce the time for samples cured at 160ºF and 200ºF to be in equilibrium in the curing environment.

c. Samples were cast in 1-ounce polypropylene screw cap jars (1.78 cubic inches) as shown in Figure 4.1. The polypropylene jars offer high temperature resistance up to 275ºF for short periods and 212ºF continuously. Each jar was capped and placed in watertight bags, which were submerged in a bucket of water. The watertight bags were used to ensure that during the first 24 hours of curing no additional water was permitted to affect the designated water cement ratio. The water in the buckets for samples cured at 160ºF and 200ºF was preheated to approximately 100ºF to ensure a short time lag to attain the elevated temperatures in the ovens as shown in Figure 4.2.

Figure. 4.1 Paste samples cast in one-ounce polypropylene screw cap jars.

d. The samples cured at 73ºF were placed in watertight bags immersed in water and cured in a moisture room kept at 100% humidity and 73ºF, water.
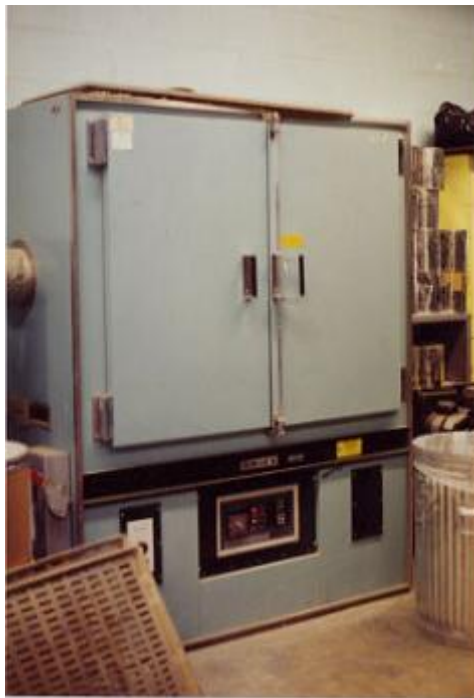
Figure 4.2 Oven used to cure samples at 200ºF

e. After 24 hours, the samples were demolded, placed in four-ounce polypropylene jars as shown in Figure 4.3 and placed in their curing

environment to continue the isothermal curing for the remaining curing

duration.

Figure 4.3 Samples cured in four-ounce polypropylene jars after demolding

f. At the end of curing duration three samples for each mix and temperature

were removed and placed in methanol. Samples cured at 160ºF and 200ºF

were cooled to room temperature before placing in the methanol. This was

done to avoid igniting the methanol. The samples were placed in the

methanol to stop further hydration of the cement.

g. After at least 7 days in the methanol, the samples were removed and wiped

clean. The samples were then crushed in a mechanical crusher (see Figure

4.4). The crushed sample was then pulverized.

Figure 4.4 samples crushed in mechanical crusher

h. Approximately 3 grams of the pulverized sample was then weighed as shown in Figure 4.5. the scale used was accurate to 1/10,000 of a gram. The samples were dried for 24 hours in an oven maintained at 221 ± 5ºF (105ºC) to remove the evaporable water from the sample. After removal from the oven the samples were cooled to room temperature and the weight was recorded as w_1.

i. The samples were then ignited for 45 minutes at 1832ºF (1000ºC) to remove the nonevaporable water chemically combined in the hydration products. The samples were cooled to room temperature and the weight recorded as w_2. Figure 4.6 shows samples removed from the oven after ignition.

Figure 4.5 Approximately 3 grams of samples weighed.

Figure 4.6 Samples removed after ignition at 1832ºF.

4.2.3 Calculations to determine the Degree of Hydration

The calculation of the degree of hydration was based on the formula given by Zhang et al (2000).
The nonevaporable water content, w_n was calculated according to the following equation:

$$w_n = \frac{(w_1 - w_2)}{w_2} - \frac{r_{fc}}{(1 - r_{fc})}$$

$$r_{fc} = p_f\, r_f + p_c\, r_c$$

The degree of hydration was determined as a ratio of
$$w_n / w_{nu}$$

w_{nu} - nonevaporable water content per gram of fully hydrated cement 0.23

w_1 - weight of the sample after drying

w_2 - weight of the sample after ignition

p_f - weight percent of fly ash in the mix, 18% and 50%

p_c - weight percent of cement in the mix

r_f - loss of ignition of fly ash 4.7%

r_c - loss of ignition of cement 2.1%

4.2.4 Problems encountered in the experimental process

Various problems were encountered during the experimental process to determine the degree of hydration of the paste samples. These problems and how they were resolved is presented below.

1. The oven used to cure samples at 160ºF failed five days into the curing process requiring a new oven to be used.

2. Some of the samples kept cured in the ovens at 160ºF and 200ºF lost the water in which they were immersed during the course of the curing duration. Cracking of the jar covers and evaporation of the water caused this.

3. To resolve the above problems, curing tanks as shown in Figure 4.7 were used in place of the ovens for the elevated temperature curing. These tanks were filled with water maintained at 160ºF and 200ºF.

4. The degree of hydration tests were repeated based on curing for elevated temperatures in the curing tanks. The results of the degree of hydration are shown in Figure 4.8.
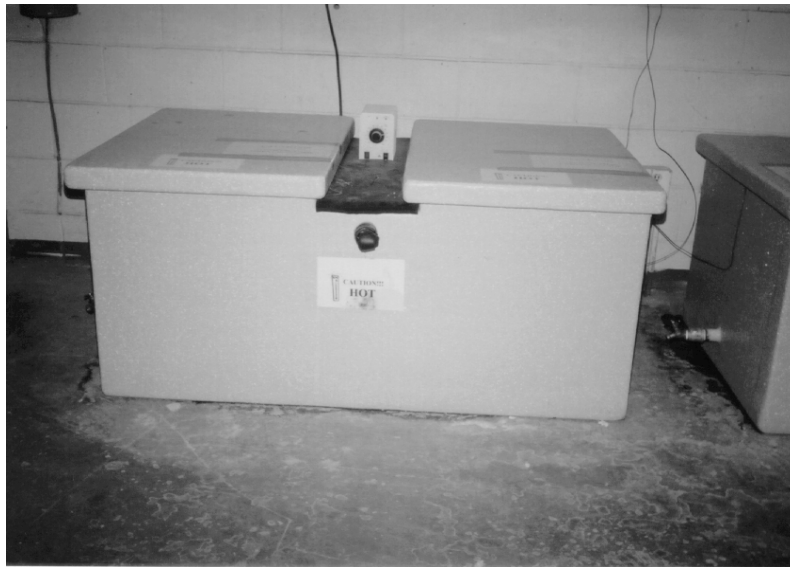
Figure 4.7 Curing tanks used for samples at elevated temperatures.
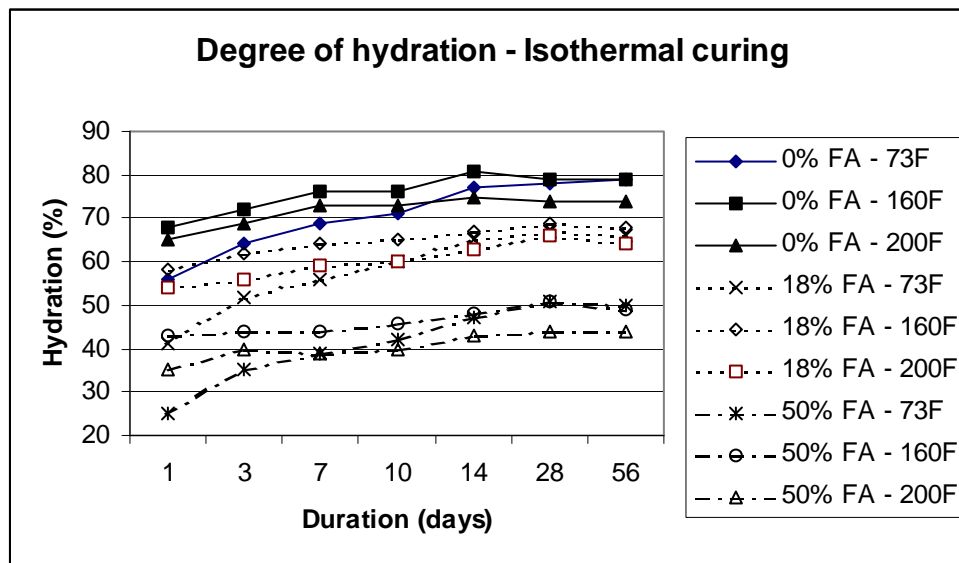
Figure 4.8 Degree of hydration ($w_{nu} = 0.23$)

Based on the results of the degree of hydration shown in Figure 4.8, samples

made from the cement fly ash paste mixes did not attain a 70% degree of hydration for

the temperatures and curing durations used in this test. The times to reach 70% degree of

hydration in the plain cement mix was established as shown in Table 4.4.

Table 4.4 Time to 70% hydration in plain cement mix

Curing Temperature (^{o}F)	Duration (approximate)
73	7
160	3
200	3

Based on the durations in Table 4.4, samples of FDOT Class IV mass concrete

(Mix 1 – appendix) based on the paste mix were made and cured isothermally following

the curing conditions used for the paste samples. Three samples were tested for each

temperature to determine the compressive strength in accordance with ASTM C 39 – 96.

Compressive strength results are presented in Table 4.5 and Figure 4.9.

Table 4.5 Concrete Mix 1 – 0% Fly Ash (Isothermal Curing)

Temp (^{o}F)	Compressive strength (psi)				RCP (coulombs)	
	70% DH	28 Days	90 Days		70% DH	28 Days
73	6,839	7,472	8252		5,845	4,720
160	4,621	4,963	5616		8,763	7,110
200	2,910	2,872	2636		9,756	11,070

Concrete 0% FA (ISO) - Compressive strength
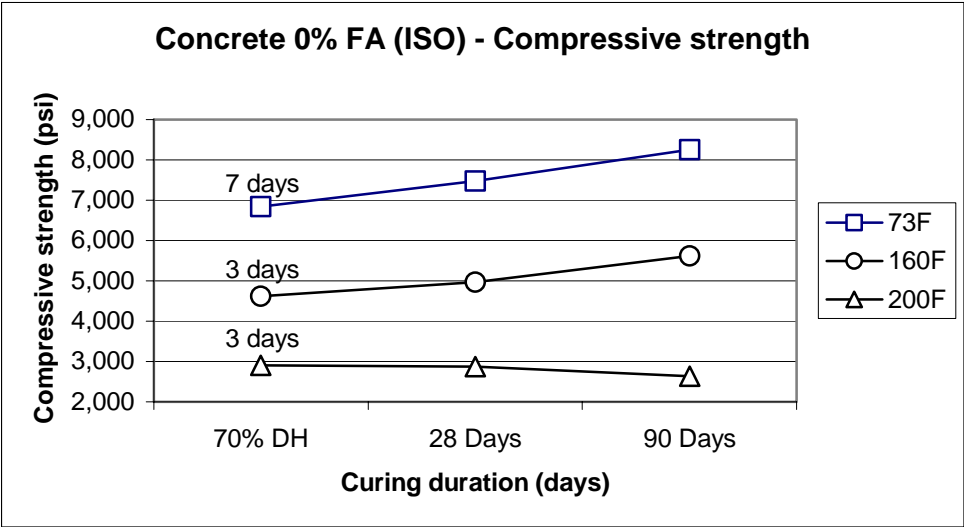
Figure 4.9 Compressive strength results

Figure 4.9 shows a substantial decrease in compressive strength of samples cured isothermally at 160°F and 200°F compared to samples cured at room temperature. This is specially pronounced for 200°F curing temperature where compressive strength of a 90 days old sample is less than a 3 days old sample. The reduction in compressive strength of samples cured at elevated temperatures might be due to high temperature changes (100°F to 200°F) as the freshly mixed concrete is introduced into the high temperature curing environment and thermal shock to concrete.
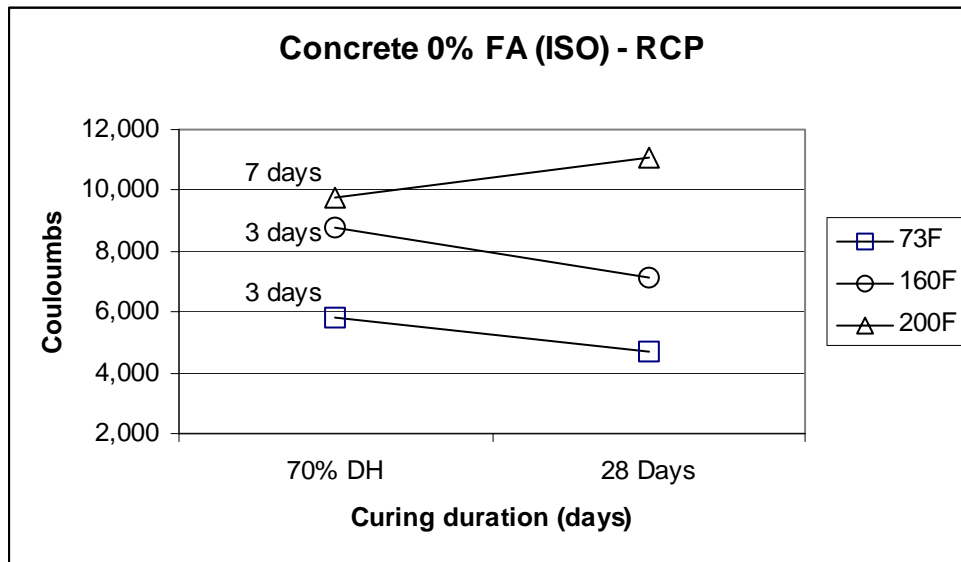
Figure 4.10 Rapid Chloride Permeability (RCP) results

Figure 4.10 shows the RCP results of the concrete samples cured isothermally at 160$^\mathrm{o}$F and 200$^\mathrm{o}$F compared to samples cured at room temperature (73$^\mathrm{o}$F). Samples cured at the high temperatures recorded high charges passing through the samples indicating reduced durability for the concrete. The samples cured at 200$^\mathrm{o}$F showed reduced durability from 7 days to 28 days as the charged passed increased over the curing duration. At 28 days, the samples cured at 73$^\mathrm{o}$F had the least current passing indicating better durability than the samples cured at 200$^\mathrm{o}$F, which recorded the highest charges passing at 28 days.

With the drastic reduction in compressive strength of the mass concrete samples cured at the elevated temperatures, the experimental set-up was changed as follows:

1. The curing was done adiabatically to simulate conditions as in mass concrete cured in the field. All the samples were introduced to the curing environment

approximately 6 hours after the start of the mixing process. Samples at 160ºF and 180ºF were introduced into the curing tanks at 80ºF after which the heat was turned on. The curing temperature of 160ºF was attained within 2 days after which the heat was turned off. The 180ºF tank attained the maximum temperature after about 2½ days after which the heat was turned off. The lids of the curing tanks were kept on whilst the temperature cooled to 73ºF within approximately two weeks of turning the heat off in both tanks.

2. The maximum temperature was reduced from 200ºF to 180ºF.

3. A review of mass concrete mixes used by the FDOT, was undertaken to examine the proportions of binders commonly used by the department. The results as shown in Table 4.6, indicated that eighty (80) per cent of the cement/fly ash mixes had 0.18 and 0.20 of the cement replaced by fly ash. Two (2) per cent of the cement /fly ash mixes had a fly ash replacement of 0.40. Based on this review, the proportion of fly ash replacement for the mass concrete tests was limited to 18%.

4. The samples were then transferred to the moisture room into curing baths, as were the 73 samples. Lime was then added to the water at this point in the curing cycle.

5. Following the change in the curing conditions, new tests were conducted to determine the time to achieve 70% degree of hydration for the mix. The results from this test are shown in Figure 4.11.

6. As can be observed in Figure 4.11, the curves were very erratic making it difficult to establish the duration to attain maturity of 70% degree of hydration in the

various mixes. This led to the use of curing durations of 7, 28 and 91 days as the bases to compare the mass concrete properties in this study.

7. At specified curing durations, mass concrete samples described in the next section were tested and the degree of hydration of the cement was determined for that age and curing temperature.

Table 4.6 Binders used in mass concrete mixes by the FDOT

BINDERS USED IN MASS CONCRETE DESIGNS IN FLORIDA			
A. Cement mixes			
Proportion of Cement	Number of mixes	% Cement Mixes	% Total mixes
1.00	**10**	**100**	**11**
B. Cement / Fly Ash mixes			
Proportion of fly ash	Number of mixes	% Cement/ Fly ash Mixes	% Total mixes
0.18	18	32	21
0.19	6	11	7
0.20	21	37	24
0.21	1	2	1
0.22	5	9	6
0.30	1	2	1
0.35	2	4	2
0.39	2	4	2
0.40	1	2	1
Total	**57**	**100**	**66**
C. Cement / Blast Furnace Slag mixes			
Proportion of Slag	Number of mixes	% Cement/Slag Mixes	% Total mixes
0.50	11	55	13
0.60	2	10	2
0.70	7	35	8
Total	**20**	**100**	**23**

SUMMARY		
Binder	**Number of Mixes**	**% of Mixes**
Cement	10	11
Cement/ Fly Ash	57	66
Cement/Slag	20	23
Total	**87**	**100**
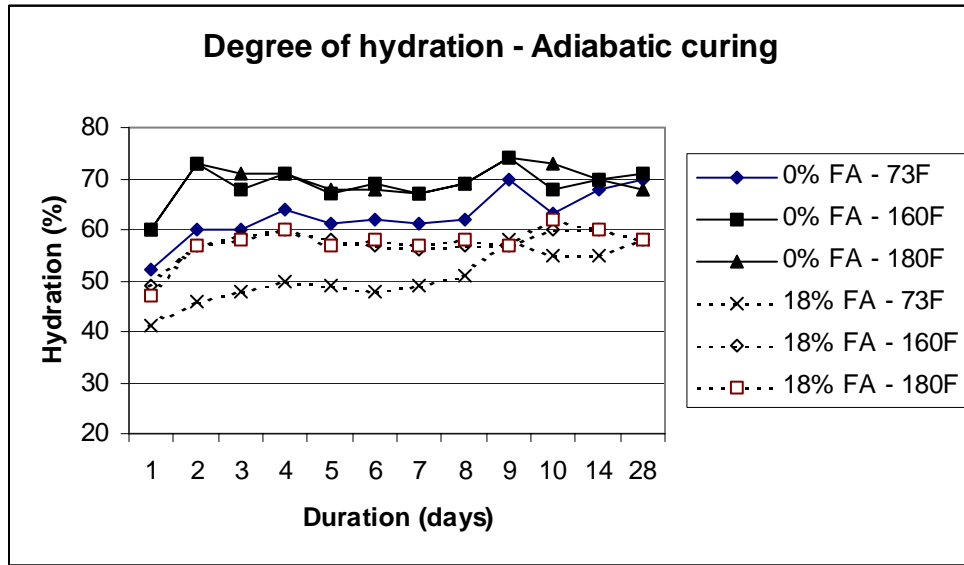
Figure 4.11 Degree of hydration based on adiabatic curing ($w_{nu} = 0.23$)

4.3 Mass Concrete Experiments

Two typical FDOT Class IV concrete with fly ash or slag as the supplementary

cementitious material to cement were used in the mass concrete tests (See appendix A).

The goal of the tests was to determine the effects of elevated curing temperate on the

strength and durability of concrete properties. Four mixes with different proportion of

cement, fly ash and blast furnace slag as shown in Table 4.7, were made and tested. The

tests were repeated. The mixes tested were as follows:

- ▪ 0% Fly ash mixes (Mix 1 and Mix 3)
- ▪ 18% Fly ash mixes (Mix 2 and Mix 4)
- ▪ 0% Blast furnace slag mixes (Mix 5 and Mix 7)
- ▪ 50% Blast furnace slag mixes (Mix 6 and Mix 8)

Table 4.7 Mixture Proportions for FDOT Class IV mass concrete

| Mixture | Saturated Surface-Dry Weights, lb/cu yd | | | | | | | | |
	Cement	Fly ash	Slag	Fine aggregate	Coarse aggregate	Air Entrainer (Darex)	Admixture (WRDA)	Water	w/b ratio
Mix 1 & 3	744	-	-	936	1746	4 oz	24.4 oz	305	0.41
Mix 2 & 4	610	134	-	918	1729	4 oz	24.4 oz	305	0.41
Mix 5 & 7	660	-	-	1076	1794	5 oz	33.0 oz	267	0.40
Mix 6 & 8	330	-	330	1066	1785	5 oz	33.0 oz	267	0.40

Samples made from the various mixes were cured adiabaticlly as before described. All the samples were mechanically vibrated during their preparation. After casting in their molds, the samples were kept in watertight bags for 24 hours after which they were demolded and placed directly in the curing water. Samples cured at 73ºF were kept in the moisture room. The heat in the 160ºF and 180ºF tanks was turned off after the maximum temperature was attained. Cooling of the tanks to a temperature of approximately 73ºF occurred over a 14-day period. The samples in the curing tanks were transferred into the moisture room and placed in limewater. Samples cured at 73ºF were also places in limewater at this time.

Various tests were performed on the mass concrete samples after 7, 28 and 91 days of curing. The tests performed were:

    I. Determination of degree of hydration

    II. Compressive strength – ASTM C 39 (ASTM 1996)

    III. Resistance to chloride penetration – ASTM C 1202 (ASTM 1994)

IV. Time to Corrosion – FM 5-522

V. Density and percentage of voids – ASTM C 642 (ASTM 1997)

VI. Microstructure analysis

4.3.1 Compressive strength

The compressive strengths of the samples were determined at curing durations of 7, 28 and 91 days. The compressive strengths were determined according to ASTM C 39-93a, *Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens,* by the FDOT physical laboratory. Twenty-seven 4" diameter x 8" cylinders were molded for each mix. Nine samples for each mix were tested at 7-, 14-, and 28-days age three for each curing temperature.

4.3.2 Resistance to chloride penetration

Each mix cured at the 73ºF, 160ºF and 180ºF was tested at 28-, and 91-days age to determine its ability to resist the chloride-ion penetration. The rapid chloride permeability for each sample was estimated following ASTM C 1202-94, *Standard Test Method for Electrical Indication of Concrete's Ability to Resist Chloride-Ion Penetration.* In this test, the chloride-ion penetrability of each sample was determined by measuring the number of coulombs that can pass through a sample in 6 hours. This provided an accelerated indication of concrete's resistance to the penetration of chloride-ions, which may corrode steel reinforcement or prestressed strands. Six 4" diameter x 8" cylinders of each mix, two for each curing temperature were tested at 28-, and 91-days age. A 2" thick disc was sawed from the top of each cylinder and used as the test specimen. It has been

determined that the total charge passed is related to the resistance of the specimen to chloride-ion penetration. The surface resistivity of each sample to the penetration of chloride ions was measured at the curing durations from the remaining portions of the samples used in the rapid chloride permeability tests.

4.3.3 Time to Corrosion

The time to corrosion test determines the duration of time for reinforcement within a sample to corrode. The time to corrosion was determined according to *Florida Method of Test for An Accelerated Laboratory Method for Corrosion Testing of Reinforced Concrete Using Impressed Current.* The samples used in this test were cylinders of 4" diameter x 6" long. Nine samples were made for each mix three for each curing temperature. Each sample contained a #4 reinforcing bar, 12" long. The bottom of the reinforcing bar was elevated by 0.75" from the bottom of the mold. Fresh concrete was placed in each mold and each mold was overfilled. The apparatus that had the reinforcing bars attached to it was placed over the cylinders. The apparatus was then placed on an external vibrator that caused the reinforcing bars to submerged into the overfilled fresh concrete when the vibrator was turned on. After vibration, a trowel was used to slope the overfilled top of the mold at a 15-degree angle from the outer rim of the sample to the center of the sample. After 28 days of curing, the samples were further cured in a solution of 3% NaCl after which they were tested. The tests were performed at the Florida Department of Transportation Corrosion Laboratory.

4.3.4 Density and percentage voids in hardened concrete

The density and percentage of voids for each mix and curing temperature were determined at curing durations of 7, 28 and 91 days. The tests were done according to ASTM C 642 - 97, *Standard Test Method for Density, Absorption, and Voids in Hardened Concrete,* by the FDOT physical laboratory. Approximately 800 grams of each sample was tested.

4.3.5 Microstructure analysis - Scanning Electron Microscope (SEM)

The scanning electron microscope used in this research was the SEM JSM 35CF. All analyses were conducted at the Major Analytical and Instrumentation Center at the University of Florida. The SEM is one of the most versatile instruments available for the examination and analysis of the microstructural characteristics of solid objects. This instrument aids in the analysis and correct explanation of complex phenomena occurring at the micrometer or submicrometer scale. The primary reason for the SEM' usefulness is the high resolution that can be obtained when bulk objects are examined, values to the order of 2 to 5 nm. The SEM provides a three dimensional appearance of the secondary and backscatter electron contrast. The greater depth of field of SEM provides much more information about the specimen.

The SEM analysis was used to determine the presence and location of delayed ettringite formation (DEF) in mortar samples sieved from the concrete mixes. The signals of interest used in this study when the electron beam impinges on the specimen were secondary electrons and characteristic x-rays. The secondary electrons enabled high

topographical spatial resolution and were useful in determining the topography of the specimen and the various shapes of the different phases. The secondary electron images were three-dimensional making it easier to identify the various phases present in the sample. Measuring the energy and intensity distribution of the characteristic x-rays enabled a chemical analysis of the samples.

The samples used for the microstructural analysis were made from mortar sieved from the concrete mixes. The samples were cured adiabatically as the mass concrete samples. After 24 hours in watertight bags, the samples were demolded from the two-ounce jar and placed directly in the curing environment. At the end of the curing period, the sample is removed from the curing tank and placed in methanol to stop further hydration. After at least 7 days in the methanol, the sample is removed and finely cut into ¼" thick wafers. The sample is then placed in an oven maintained at a temperature of 230°F for 24 hours to remove all the evaporable water, not used in the hydration process. The specimen is then stored in a desiccator after removal from the oven.

To obtain very good images using the SEM and avoid the introduction of cracks or removal of any ettringite nests, cutting of the surface of the sample was avoided, instead pieces were fractured and the fractured surfaces were analyzed. The fractured pieces were cut and coated with carbon. Since the samples were non-conducting, coating was necessary to eliminate or reduce the electric charge that builds up rapidly in a non-conducting specimen when it is scanned by a beam of high-energy electrons. Figure 4.12 shows samples mounted and ready for examination in the SEM. Secondary electron images were taken to show the presence and location of ettringite. Energy Dispersive

Analysis of the X-rays (EDAX) was used to confirm or deny the presence of the

ettringite. According to Hime et al. (2000), EDAX of a ettringite mass has a "step"

pattern of the aluminum, sulfur and calcium peaks as shown in Figure 4.13. For

monosulphate, the sulfur peak is the lowest of the three.

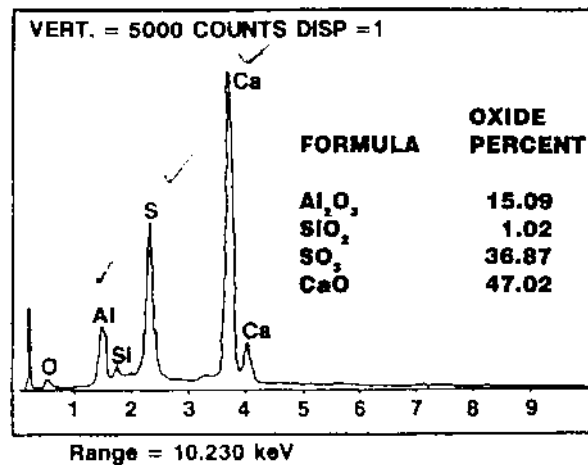Figure 4.12 Mortar samples mounted on stubs for SEM examination

Figure 4.13 EDAX analysis of "gel" showing calcium, sulfur, and aluminum peaks
typical for ettringite (Hime at al.)

CHAPTER 5 TEST RESULTS AND DISCUSSION

5.1 Introduction

This chapter presents results of the experimental work conducted to determine the maximum curing temperature to avoid durability problems and the formation of delayed ettringite in mass concrete. The study was conducted in three phases:

I. Phase 1 involved tests to determine the time to achieve a maturity of 70% degree of hydration of cement in mass concrete mixes with cement, fly ash and blast furnace slag as the cementitious materials.

II. Phase 2 involved tests of two FDOT Class IV mass concretes at temperatures of 73ºF, 160ºF and 180ºF for 7, 28 and 91 days. Various tests were performed on the concrete to evaluate the effect of the curing conditions on the strength and durability of the concrete.

III. In phase 3, mortar samples prepared from the sieved mass concrete mixes were subjected to the same curing conditions. The samples were then examined under a scanning electron microscope to determine the formation or otherwise of delayed ettringite.

5.2 Phase 1 – Determination of Degree of Hydration

The results for the degree of the hydration for the different mixes tested are shown in Tables 5.2 through Table 5.5. Initially, all calculations for the degree of hydration were based on the fact that the nonevaporable water content for 1 gram of fully hydrated cement was 0.23 grams of water. This value was found to be inapplicable to cement blends with fly ash or slag.

Scanning Electron Microscope (SEM) observations by Maltais & Marchand (1997) for

pastes some incorporating fly ash as a 10, 20 and 30 per cent replacement of cement and

cured at 68ºF and 104ºF showed that the fly ash did not react before at least 28 days.

Although the fly ash did not react during the first days of curing, their test results

indicated that it could not be considered as a totally inert material. Despite the very little

pozzolanic activity, Maltais and Marchand (1997) found out that the presence of fly ash

appeared to increase the mortar nonevaporable water content at early days. This increase

was attributed to an acceleration of the early cement hydration in the presence of fly ash.

Two reasons for the acceleration of cement hydration in the presence of fly ash are

physical and chemical effects.

- Physical
    - The addition of fly ash tends to increase the number of fine particles in the system. The presence of these fine particles contributes to increase the density of the matrix making for better hydration of the cement.
    - The replacement of cement particles by fly ash is also believed to increase the available space in the floc structure created by the cement grains.
    - The fine particles provide additional nucleation sites for cement hydration products.
- Chemical
    - According to Takemoto and Uchikawa (quoted in Maltais & Marchand, 1997), the acceleration of cement hydration in the presence of fly ash is mainly related to the preferential adsorption of calcium ions on the fly

ash particles. The phenomenon contributes to decrease the calcium ion concentration in the liquid phase, which subsequently favors the dissolution of calcium phases from the cement grains.

Fly ash reacts with the calcium hydroxide formed from the hydration of the cement. The reduction in calcium hydroxide content form the pozzolanic reaction will not enable a value of 0.23 to be a good estimate of the degree of hydration. The pozzolanic reaction will reduce the amount of calcium hydroxide and replace it with hydrates formed by the pozzolanic reaction. The amount of water released from a mole weight of the hydrates is less than the amount of water released form a mole weight of calcium hydroxide due to its large molecular weight. A more reliable estimate was determined for the cement fly ash mixes after hydrating paste at 73ºF in the moisture room for 1 year and calculating the nonevaporable water content. The new values for the nonevaporable water content for the cement and fly ash mixes were as follows:

- 0.19 for cement and 18% fly ash mix with w/b ratio of 0.41
- 0.15 for cement and 50% fly ash mix with w/b ratio of 0.41.

These values for the nonevaporable water content are comparable to that determined for various mixes of fly ash by Lam et al. (2000) and shown in Table 5.1 below.

Table 5.1 Nonevaporabe water content for various Fly ash mixes Lam et al. (2000)

w/b	Fly ash replacement (%)	*Wn_u at 90 days
0.3	25	0.19
0.3	55	0.15
0.5	25	0.18
0.5	55	0.15
* Calculated from ratio of Wn / Degree of hydration		

Legend: Degree of Hydration results

IOP –Isothermal curing of paste samples in oven

- (Row # - 1, 6, 14, 16, 17, 24, 25, 36, 37, 40, 41, 44, 45, 48, 49)

ITP –Isothermal curing of paste samples in tanks

- (Row # - 2, 7, 15, 18, 19, 26, 27, 38, 39, 42, 43, 46, 47, 50, 51)

ATM – Adiabatic curing of mortar samples in tank

- (Row # - 3, 8, 11, 20, 21, 28, 29, 32, 33)

ATC – Adiabatic curing of mortar sieved from concrete in tank

- (Row # - 4, 5, 9, 10, 12, 13, 22, 23, 30, 31, 34, 35, 52, 53, 54)

C & F - Calculation of degree of hydration based on total cement and fly ash content and the nonevaporable water content at full hydration after curing for 1 year at 73°F. the nonevaporable content for 1 gram of 18%FA mix was determined to be 0.19 and that for the 50%FA was determined to be 0.15.

C – Calculation of degree of hydration based on cement solely responsible for the hydration products formed assuming no reaction of fly ash. NA designation is applied to durations and temperatures for which fly ash reaction is assumed to have started, invalidating an extension of this calculation.

C & S - Calculation of degree of hydration based on total cement and blast furnace slag content and the nonevaporable water content at full hydration of 0.23 as for plain cement mixes.

Table 5.2 Degree of hydration results for plain cement mixes

Mix	Temp	Row	Mix design	Curing Duration (days)													
				1	2	3	4	5	6	7	8	9	10	14	28	56	91
0%	73	1	IOP – 0% .41w/c	58	-	67	-	-	-	70	-	-	71	71	73	74	-
		2	ITP – 0% .41 w/c	56	-	64	-	-	-	69	-	-	71	77	78	79	-
		3	ATM – 0% .41 w/c	52	60	60	64	61	62	61	62	70	63	68	70	-	-
		4	ATC – 0% .41 w/c	-	-	-	-	-	-	59	-	-	-	-	64	-	69
		5	ATC – 0% .40w/c	-	-	-	-	-	-	54	-	-	-	-	64	-	62
	160	6	IOP – 0% .41w/c	68	-	72	-	-	-	75	-	-	73	72	69	69	-
		7	ITP – 0% .41 w/c	68	-	72	-	-	-	76	-	-	76	81	79	79	-
		8	ATM – 0% .41 w/c	60	73	68	71	67	69	67	69	74	68	70	71	-	-
		9	ATC – 0% .41 w/c	-	-	-	-	-	-	61	-	-	-	-	61	-	66
		10	ATC – 0% .40w/c	-	-	-	-	-	-	62	-	-	-	-	59	-	61
	180	11	ATM – 0% .41 w/c	60	73	71	71	68	68	67	69	74	73	70	68	-	-
		12	ATC – 0% .41 w/c	-	-	-	-	-	-	59	-	-	-	-	63	-	69
		13	ATC – 0% .40w/c	-	-	-	-	-	-	60	-	-	-	-	59	-	60
	200	14	IOP – 0% .41w/c	73	-	75	-	-	-	80	-	-	78	75	75	76	-
		15	ITP – 0% .41 w/c	65	-	69	-	-	-	73	-	-	73	75	74	74	-

Table 5.3 Degree of hydration for cement and 18% fly ash mixes

Mix	Temp	Row	Mix design		1	2	3	4	5	6	7	8	9	10	14	28	56	91
					Curing Duration (days)													
18%	73	16	IOP – 18% FA	C	63	-	65	-	-	-	75	-	-	78	76	79	NA	-
		17		C&F	60		63				73			75	74	76	79	-
		18	ITP – 18% FA	C	51	-	65	-	-	-	70	-	-	75	81	84	NA	-
		19		C&F	50	-	63	-	-	-	68	-	-	72	78	82	80	-
		20	ATM – 18% FA	C	51	58	60	63	61	60	61	64	73	69	69	73	-	-
		21		C&F	49	55	59	61	60	58	59	62	70	66	67	70	-	-
		22	ATC – 18% FA	C	-	-	-	-	-	-	55	-	-	-	-	64	-	NA
		23		C&F	-	-	-	-	-	-	54					62		76
	160	24	IOP – 18% FA	C	78	-	81	-	-	-	84	-	-	NA	NA	NA	NA	-
		25		C&F	75	-	78	-	-	-	81			83	82	78	76	
		26	ITP – 18% FA	C	73	-	78	-	-	-	80	-	-	NA	NA	NA	NA	-
		27		C&F	70	-	75	-	-	-	77			79	81	83	82	
		28	ATM – 18% FA	C	61	71	74	75	73	71	70	NA	NA	NA	NA	NA	-	-
		29		C&F	59	69	72	73	71	69	68	70	69	72	72	71	-	-
		30	ATC – 18% FA	C	-	-	-	-	-	-	65	-	-	-	-	NA	-	NA
		31		C&F	-	-	-	-	-	-	63					64	-	73
	180	32	ATM – 18% FA	C	59	71	73	75	71	73	71	NA	NA	NA	NA	NA	-	-
		33		C&F	56	69	72	73	69	70	69	70	69	75	73	71	-	-
		34	ATC – 18% FA	C	-	-	-	-	-	-	63	-	-	-	-	NA	-	NA
		35		C&F	-	-	-	-	-	-	61	-	-	-	-	68	-	70
	200	36	IOP – 18% FA	C	80	-	84	-	-	-	84	-	-	NA	NA	NA	NA	-
		37		C&F	78	-	81	-	-	-	81	-	-	83	82	81	84	-
		38	ITP – 18% FA	C	68	-	70	-	-	-	74	-	-	NA	NA	NA	NA	-
		39		C&F	66	-	68	-	-	-	71	-	-	73	75	79	77	-

Table 5.4 Degree of hydration for cement and 50% fly ash mixes

Mix	Temp	Row	Mix design		Curing Duration (days)													
					1	2	3	4	5	6	7	8	9	10	14	28	56	91
50%	73	40	IOP – 50% FA	C	58	-	68	-	-	-	80	-	-	82	84	86	NA	-
		41		C&F	44		53				61			63	65	66	64	
		42	ITP – 50% FA	C	50	-	70	-	-	-	78	-	-	84	94	102	NA	-
		43		C&F	38		53				60			65	71	78	76	
	160	44	IOP – 50% FA	C	80	-	80	-	-	-	82	-	-	NA	NA	NA	NA	-
		45		C&F	61		62				63			64	63	66	68	
		46	ITP – 50% FA	C	86	-	88	-	-	-	88	-	-	NA	NA	NA	NA	-
		47		C&F	65		67				67			71	74	78	75	
	200	48	IOP – 50% FA	C	86	-	88	-	-	-	86	-	-	NA	NA	NA	NA	-
		49		C&F	66		67				66			64	65	68	61	
		50	ITP – 50% FA	C	70	-	80	-	-	-	78	-	-	NA	NA	NA	NA	-
		51		C&F	53		61				59			60	66	67	68	

Table 5.5 Degree of hydration for cement and blast furnace slag mixes

Mix	Temp	Row	Mix design		Curing Duration (days)													
					1	2	3	4	5	6	7	8	9	10	14	28	56	91
50%	73	52	ATC – 50% SL	C & S	-	-	-	-	-	-	69	-	-	-	-	78	-	81
	160	53	ATC – 50% SL	C & S	-	-	-	-	-	-	84	-	-	-	-	86	-	82
	180	54	ATC – 50% SL	C & S	-	-	-	-	-	-	82	-	-	-	-	85	-	84

5.3 Phase 2 – Tests of mass concrete

Table 5.6 provides a summary of the plastic properties of the mass concrete mixes

tested. Tests for each mix included determination of the degree of hydration, compressive

strength, time to corrosion, rapid chloride permeability, density and the percentage of

voids at curing durations of 7, 28 and 91 days and curing temperatures of 73 ^{o}F, 160 ^{o}F

and 180°F. The test for each mix was repeated and the average results for each mix is

provided in Tables 5.7 and 5.8.

Table 5.6 Summary of Plastic Properties of Fresh Concrete.

Test #	Mixture	Concrete Temperature (^{o}F)	Air Temperature (^{o}F)	Slump in	Air Content %	Workability	w/b ratio
1	Mix 1	75	72	6.50	4.0	Good	0.41
	Mix 2	75	72	5.00	2.0	OK	0.41
2	Mix 3	69	70	6.50	4.5	Good	0.41
	Mix 4	70	70	7.50	2.5	Good	0.41
3	Mix 5	74	72	5.25	6.0	Good	0.40
	Mix 6	74	72	3.25	3.9	Sticky	0.40
4	Mix 7	75	71	4.00	5.50	Good	0.40
	Mix 8	73	70	2.75	3.00	Stiff	0.40

Table 5.7 Results of concrete mixes M1 and M2

Test #	Mix ID	Temp (°F)	Duration (days)	Hydration (%)	Comp (psi)	Density (lbs/ft³)	Voids (%)	RCP (coulombs)	Resistivity (kohms/cm)	Corrosion (days)
		73	7	56	6048	-	-	-	-	-
			28	66	6860	155.70	16.00	5507	8.63	12
			91	70	7457	155.70	16.30	4456	11.07	-
	M1 (0% Fly Ash) w/c = 0.41 slump = 6.5"	160	7	56	5992	-	-	-	-	-
			28	60	6051	157.10	16.70	8701	7.16	8
			91	63	6280	156.50	16.00	6693	8.28	-
		180	7	56	5761	-	-	-	-	-
			28	67	5790	159.05	15.95	9317	8.46	5
1			91	66	6037	156.50	15.80	7695	8.27	-
		73	7	49	6610	-	-	-	-	-
			28	58	7770	161.20	16.75	5014	9.04	14
			91	58	8517	159.90	15.90	2285	17.84	-
	M2 (18% Fly Ash) w/c = 0.41 slump = 5.0"	160	7	62	6942	-	-	-	-	-
			28	61	7149	162.15	16.60	2470	21.33	9
			91	60	7810	160.30	16.70	1701	29.80	-
		180	7	57	6660	-	-	-	-	-
			28	62	7069	162.15	16.90	2646	19.42	9
			91	61	7437	160.70	16.20	1822	23.06	-

Table 5.8 Results of concrete mixes M3 and M4

Test #	Mix ID	Temp (°F)	Duration (days)	Hydration (%)	Comp (psi)	Density (lbs/ft³)	Voids (%)	RCP (coulombs)	Resistivity (kohms/cm)	Corrosion (days)
2 (repeat of test 1)	M3 - same as M1 (0% Fly Ash) w/c = 0.41 slump = 6.5"	73	7	61	5800	154.20	16.30	-	-	
			28	61	6387	156.30	15.30	5616	11.22	15
			91	69	7043	152.20	14.50	4531	12.18	
		160	7	66	5567	155.30	15.90	-	-	
			28	62	5613	155.10	16.20	8565	8.42	5
			91	70	5957	155.20	15.80	6851	9.78	
		180	7	61	5323	155.80	16.00	-	-	
			28	59	5353	155.20	15.80	10459	8.85	7
			91	72	5343	154.10	15.50	7370	9.12	
	M4 - same as M2 (18% Fly Ash) w/c = 0.41 slump = 7.5"	73	7	58	6320	159.70	16.60	-	-	
			28	66	7173	159.40	16.00	5331	10.83	27
			91	93	8220	158.10	15.70	2272	18.95	
		160	7	64	6647	161.40	16.80	-	-	
			28	66	6570	160.40	17.40	2626	21.33	15
			91	86	7180	158.90	15.80	1872	26.09	
		180	7	64	6247	159.00	17.40	-	-	
			28	74	6643	158.70	17.10	2360	18.67	15
			91	90	7187	158.90	15.80	1923	22.13	

Table 5.9 Results of concrete mixes M5 and M6

Test #	Mix ID	Temp (°F)	Duration (days)	Hydration (%)	Comp (psi)	Density (lbs/ft^3)	Voids (%)	RCP (coulombs)	Resistivity (kohms/cm)	Corrosion (days)
3	M5 (0% Slag) w/c = 0.40 slump = 5.25"	73	7	50	4780	156.2	14.7	-	-	
			28	63	5673	150.0	14.0	5850	9.84	18
			91	63	6310	152.4	14.2	4105	12.1	
		160	7	54	4783	155.6	15.8	-	-	
			28	58	4673	152.9	15.1	10108	7.93	11
			91	62	4903	153.7	16.6	7770	8.45	
		180	7	55	4830	155.1	15.7	-	-	
			28	61	4673	151.9	14.9	8996	11.45	14
			91	65	4707	155.9	15.8	9233	9.65	
	M6 (50% Slag) w/c = 0.40 slump = 3.25"	73	7	69	5293	161.5	16.0	-	-	
			28	80	7165	158.5	14.7	2845	17.88	42
			91	83	8003	157.5	14.1	2114	24.50	
		160	7	80	6790	161.4	16.0	-	-	
			28	84	6877	159.3	15.1	1919	21.68	16
			91	84	7617	158.5	14.3	1662	25.90	
		180	7	80	5853	161.4	15.5	-	-	
			28	84	6273	158.7	14.7	2689	18.49	11
			91	87	7180	159.5	15.0	1753	25.00	

Table 5.10 Results of concrete mixes M7 and M8

Test #	Mix ID	Temp (°F)	Duration (days)	Hydration (%)	Comp (psi)	Density (lbs/ft³)	Voids (%)	RCP (coulombs)	Resistivity (kohms/cm)	Corrosion (days)
4 (repeat of test 3)	M7 - same as M5 (0% Slag) w/c = 0.40 slump = 4.0"	73	7	57	5760	152.3	12.3	-	-	
			28	65	6733	155.7	13.6	4430	11.34	21
			91	61	7307	152.4	12.3	3960	12.5	
		160	7	69	5947	151.4	11.9	-	-	
			28	60	5653	156.3	14.3	8135	7.93	11
			91	60	5890	154.4	14.0	7119	8.45	
		180	7	64	5450	152.5	11.8	-	-	
			28	57	5430	154.8	14.7	6175	10.97	7
			91	54	5373	153.7	14.0	8347	9.05	
	M8 - same as M6 (50% Slag) w/c = 0.40 slump = 2.75"	73	7	69	5770	155.5	12.8	-	-	
			28	75	8303	159.8	15.3	2540	19.45	85
			91	79	9003	155.7	12.6	1890	22.54	
		160	7	87	7307	156.3	12.0	-	-	
			28	87	7470	160.5	14.4	1943	23.67	22
			91	80	8230	156.1	12.9	1481	27.00	
		180	7	83	6567	155.4	11.8	-	-	
			28	85	6883	160.8	14.9	2105	20.55	44
			91	80	7370	157.3	12.1	1802	24.76	

Table 5.11 Summary of Results of concrete mixes M1 , M2, M3 and M4

Mix ID	Temp (°F)	Duration (days)	Hydration (%)	Comp (psi)	Density (lbs/ft^3)	Voids (%)	RCP (coulombs)	Resistivity (kohms/cm)	Corrosion (days)
M1 & M3 (0% Fly Ash) w/c = 0.41	73	7	59	5924	154.20	16.30	-	-	-
		28	64	6624	156.00	15.65	5562	9.93	14
		91	69	7250	153.95	15.40	4494	11.63	-
	160	7	61	5780	155.30	15.90	-	-	-
		28	61	5832	156.10	16.45	8633	7.79	7
		91	67	6119	155.85	15.90	6772	9.03	-
	180	7	59	5542	155.80	16.00	-	-	-
		28	63	5572	157.10	15.88	9888	8.66	6
		91	69	5690	155.30	15.65	7533	8.70	-
M2 & M4 (18% Fly Ash) w/c = 0.41	73	7	54	6465	159.70	16.60	-	-	-
		28	62	7472	160.30	16.38	5173	9.94	20
		91	76	8369	159.00	15.80	2279	18.40	-
	160	7	63	6795	161.40	16.80	-	-	-
		28	64	6860	161.30	17.00	2548	21.33	12
		91	73	7495	159.60	16.25	1787	27.95	-
	180	7	61	6454	159.00	17.40	-	-	-
		28	68	6856	162.15	17.00	2503	19.05	12
		91	70	7312	159.80	16.00	1873	22.60	-

Table 5.12 Summary of Results of concrete mixes M5, M6, M7 and M8

Mix ID	Temp (^{o}F)	Duration (days)	Hydration (%)	Compressive (psi)	Density (lbs/ft^3)	Voids (%)	RCP (coulombs)	Resistivity (kohms/cm)	Corrosion (days)
M5 & M7 (0% Slag) w/c = 0.40	73	7	54	5270	154.3	13.5	-	-	
		28	64	6203	152.9	14.2	5140	10.59	20
		91	62	6808	152.4	13.3	4033	12.3	
	160	7	62	5363	153.5	13.9	-	-	
		28	59	5163	154.6	14.7	9122	7.93	11
		91	61	5397	154.1	15.3	7445	8.45	
	180	7	60	5140	153.8	13.8	-	-	
		28	59	5052	153.4	14.8	7586	11.21	11
		91	60	5040	154.8	14.9	8794	9.35	
M6 & M8 (50% Slag) w/c = 0.40	73	7	69	5532	158.5	14.4	-	-	
		28	78	7734	159.2	15.0	2693	18.67	*64
		91	81	8503	156.6	13.4	2002	23.52	
	160	7	84	7049	158.9	14.0	-	-	
		28	86	7174	159.9	14.8	1919	21.68	19
		91	82	7924	157.3	13.6	1572	26.45	
	180	7	82	6210	158.4	13.7	-	-	
		28	85	6578	159.8	14.8	2689	18.49	28
		91	84	7275	158.4	13.6	1778	24.88	

Legend: * indicates results from Test 3; Test 4 is still ongoing.

The degree of hydration was determined from mortar samples sieved from the concrete mixes. The samples were cured adiabatically for the same durations as the mass concrete samples. At the end of the curing durations, the hydration process was stopped by immersion in methanol and then tested to determine the degree of hydration. Figures 5.1 and 5. 2 show the results of the hydration tests.
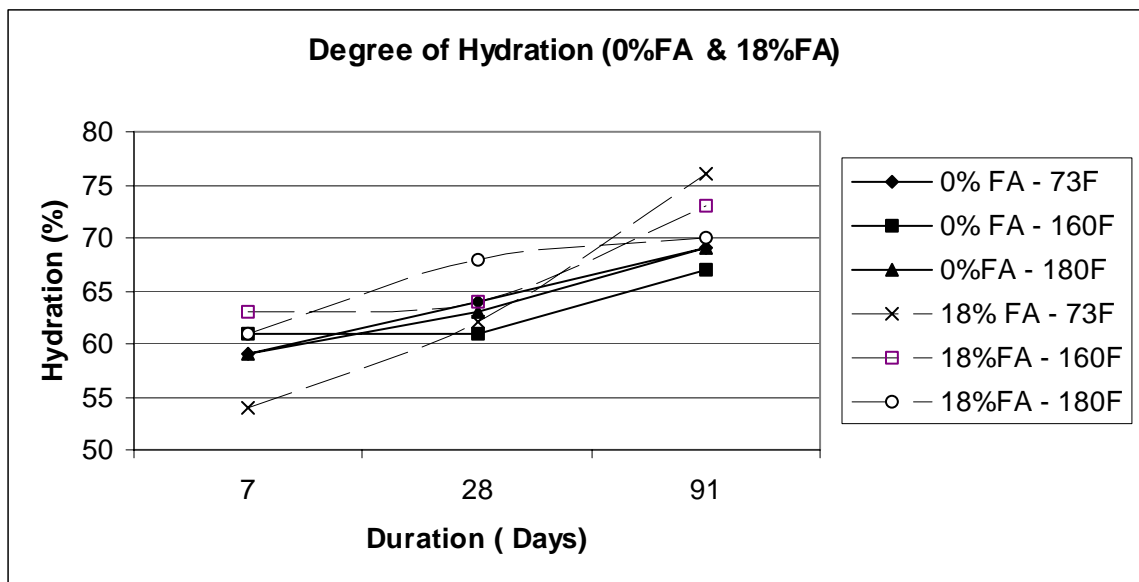
Figure 5.1 Degree of hydration for 0%FA and 18%FA mixes

Addition of Fly ash increases the degree of hydration at early ages for the samples cured at the higher temperatures. Higher curing temperatures have not been effective however in increasing the degree of hydration at early age for the mix without fly ash. At later ages, the degree of hydration for the fly ash mix is higher than that without fly ash at all curing temperatures. The degree of hydration for the fly ash mix at later ages is

highest in the samples cured at 73°F and decreases with increasing curing temperature.
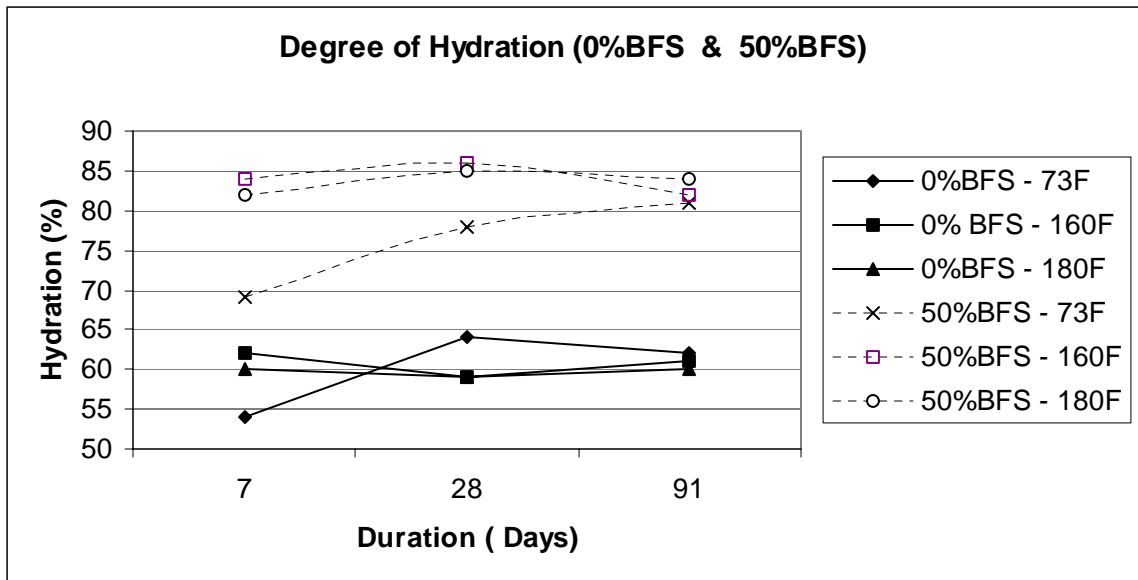
Figure 5.2 Degree of hydration for 0%BFS and 50%BFS mixes

Addition of slag as seen in Figure 5.2, resulted in a drastic increase in the degree of hydration over the mix without slag at both early and later ages for all curing temperatures. The slag mixes cured at higher temperatures showed a much higher degree of hydration at early ages, however at later ages all curing temperatures had reach to about the same degree of hydration.

5.3.2 Compressive strength results

The compressive tests were performed on twenty-seven different cylinders for each mix. Nine samples of each mix were tested at curing durations of 7, 28 and 91 days from the mixing date. Three samples each were tested for three different temperatures of 73°F, 160°F and 180°F. The compressive strengths were determined in accordance

with ASTM C39-93a. Figures 5.3 through 5.7 show the results of the compressive

strength for the different mixes and curing conditions.
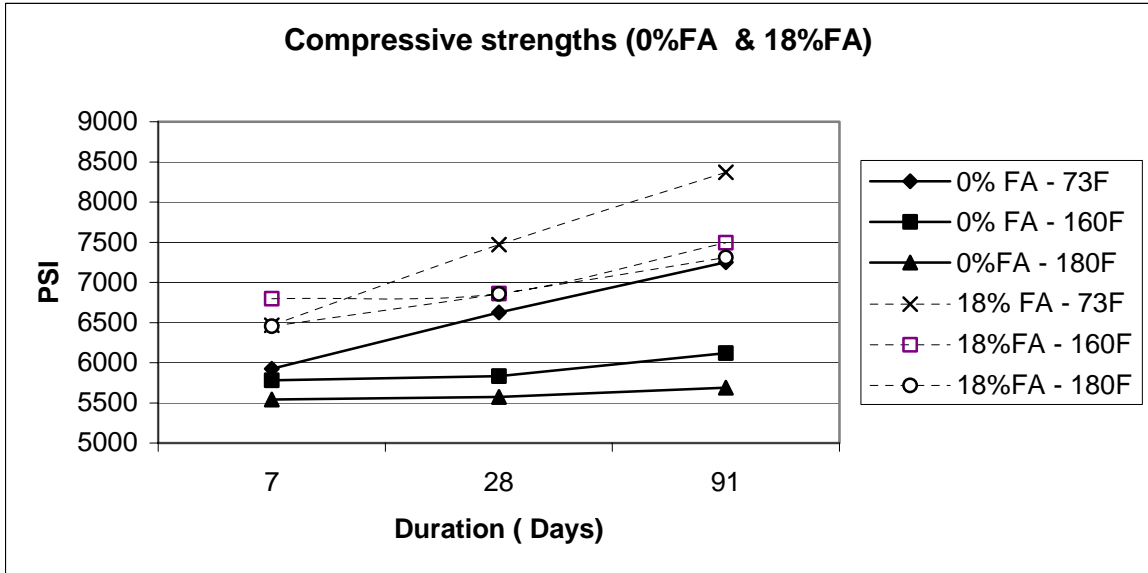
Figure 5.3 Compressive strengths for 0%FA and 18%FA mixes

Table 5.13 Compressive strength as a ratio of 28-day samples cured at 73°F

Mix	Duration (days)	Temperature (°F)		
		73	160	180
0% FA	7	0.89	0.87	0.84
	28	1.00	0.88	0.84
	91	1.09	0.92	0.86
18% FA	7	0.87	0.91	0.86
	28	1.00	0.92	o.92
	91	1.12	1.00	0.99

Higher curing temperatures resulted in lower strength for all ages and mixtures

except fly ash mix at 7 days age, which had a higher strength at the 160F as shown in

Figure 5.3 and Table 5.13. Addition of fly ash increased the strength at all ages and

curing temperatures when compared to the mix without fly ash, mirroring the higher

degree of hydration of the fly ash mixes over the plain cement mixes. The highest

strength at later age was recorded for the fly ash mix cured at 73^oF, which also had the

highest degree of hydration at this age.
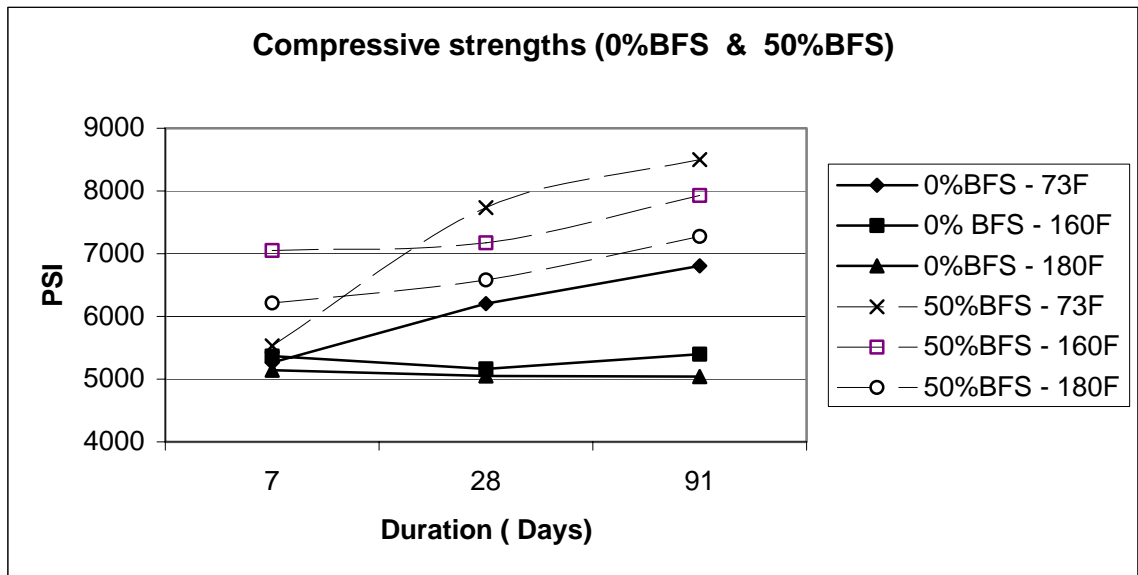
Figure 5.4 Compressive strengths for 0%BFS and 50%BFS mixes

Table 5.14 Compressive strength as a ratio of the 28-day samples cured at 73^oF

Mix	Duration (days)	Temperature (^{o}F)		
		73	160	180
	7	0.85	0.86	0.83
0% Slag	28	1.00	0.83	0.81
	91	1.10	0.87	0.81
	7	0.71	0.91	0.80
50% Slag	28	1.00	0.93	0.85
	91	1.10	1.02	0.94

Higher curing temperatures resulted in increased early age strength in the slag mix but reduced later age strength as seen in Figure 5.4 and Table 5.14. Higher curing temperatures in the mix without slag generally resulted in a decrease in both early and later age strength. The mix with slag had higher strengths at all curing durations and temperatures compared to the mix without the slag. This observation agrees with the higher degree of hydration in the slag mix over the mix without slag.

5.3.3 Resistance to chloride ion penetration

Six samples of each mix were tested at 28 and 91 days, two for each curing temperature. The tests were done in accordance with ASTM C 1202-94. Results of this test are shown for the various mixes in Figures 5.5 and 5.6. The blended cement mixes were observed to have a higher resistance to chloride ion penetration than the plain cement mixes as shown by the lower charges passing through during the test.
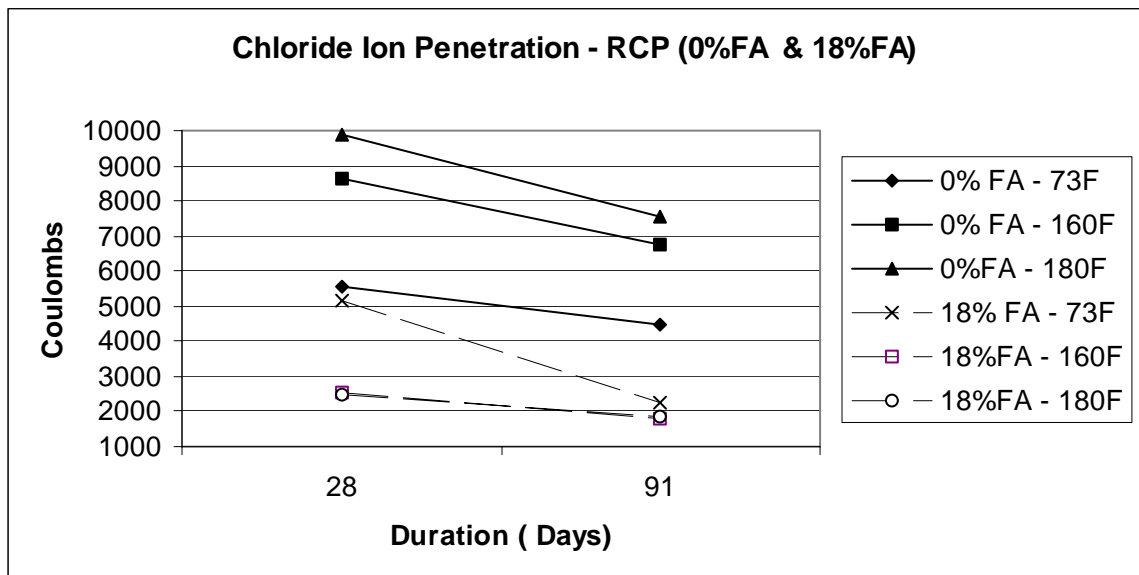
Figure 5.5 Chloride Ion Penetration results for 0%FA and 18%FA mixes

As shown in Figure 5.5 above, at higher curing temperatures, the mixes without fly ash, had higher chloride penetration at both 28 and 91 days. For the fly ash mixes however higher curing temperatures resulted at much reduced chloride ion penetration at 28 days although their influence on chloride penetration at 91 days was about the same as curing at 73^oF. Overall, the fly ash mixes had lower chloride ion penetration at all curing durations and temperatures when compared to the mixes without fly ash.
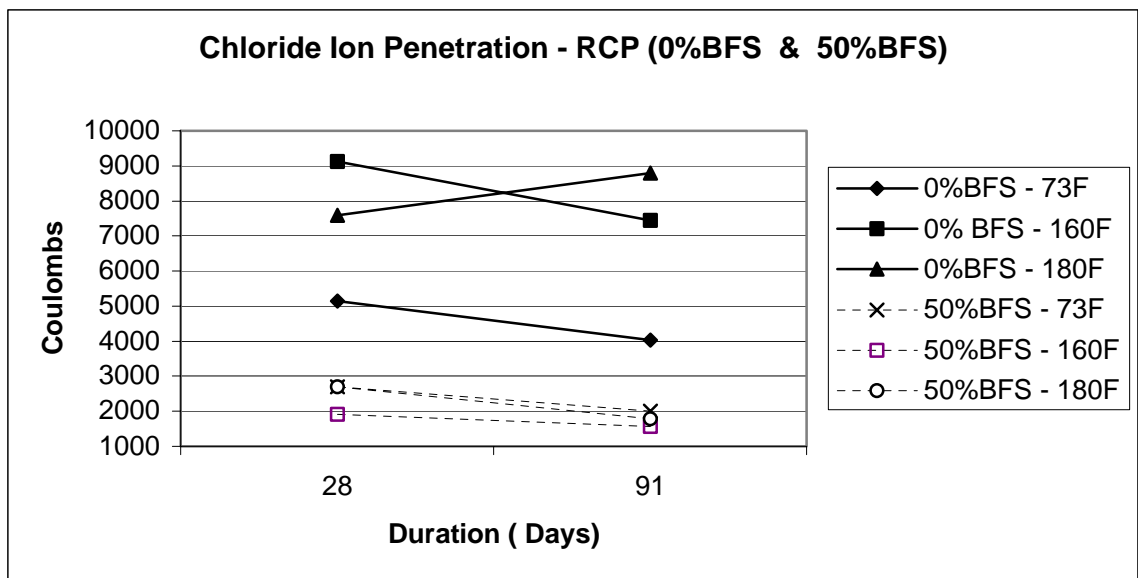
Figure 5.6 Chloride Ion Penetration results for 0%BFS and 50%BFS mixes

The mix without slag as seen in Figure 5.6, showed increased RCP values at higher temperatures. The RCP values for the slag mixes were not much affected by the curing temperatures. Overall, RCP values for the slag mixes were considerably reduced when compared to the mixes without slag at all curing temperatures and durations.

5.3.4 Density and percentage of voids

      Two samples of each mix for each curing temperature weighing approximately 800g were tested at 7, 28 and 91 days to determine the density and percentage of voids. Figure 5.7 shows the density for the plain cement and fly ash mixes. Figure 5.8 shows the density for the blast furnace slag mix.
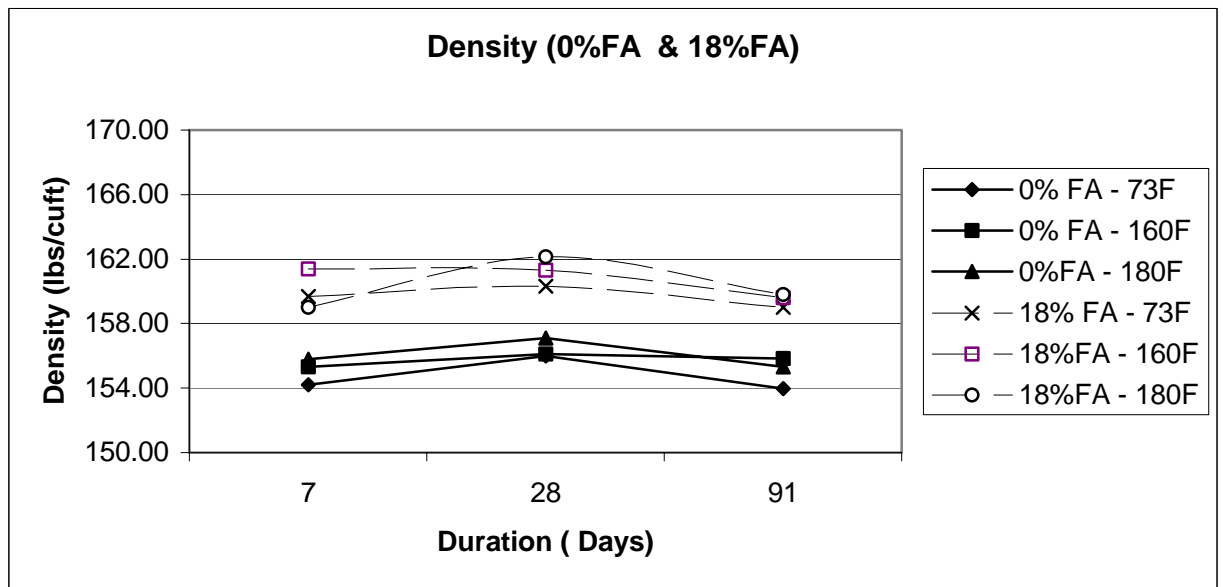
Figure 5.7 Density for 0%FA and 18%FA mixes

      The mix with fly ash showed a higher density at all curing temperatures and curing durations, than the mix without it as seen in Figure 5.7. The curing temparature of the concrete had a minimal influence on the resulting density.
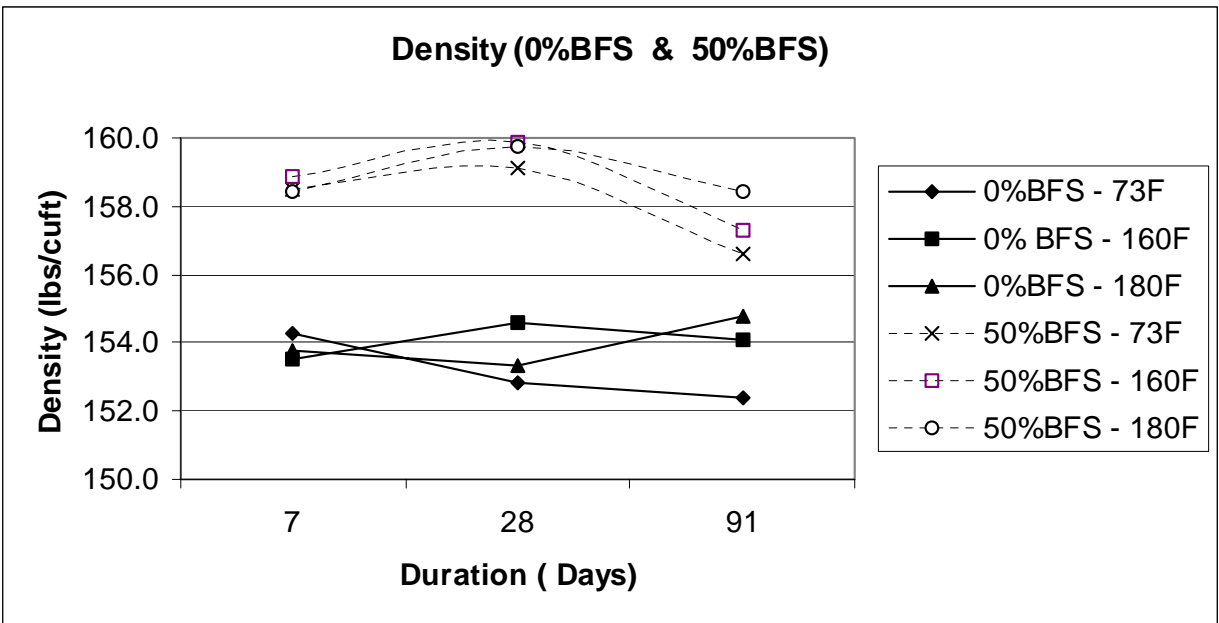
Figure 5.8 Density for 0%BFS and 50%BFS mixes

Addition of slag has increased density for all curing temperatures and ages.

Higher curing temperatures have slightly increased density at 91 days for mixes with and

without slag as seen in Figure 5.8.
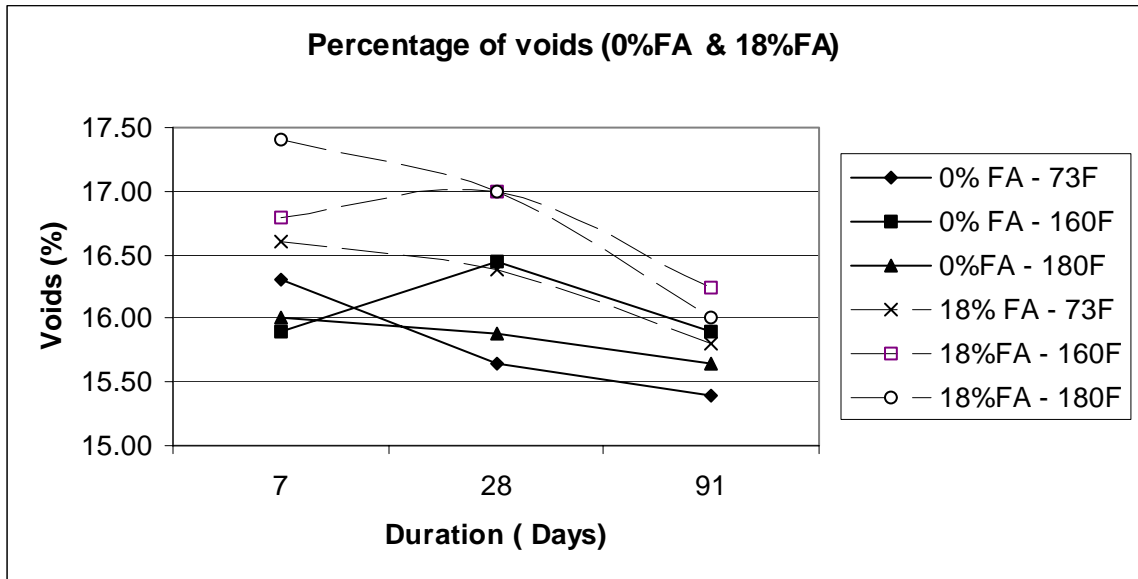
Figure 5.9 Percentage of voids for 0%FA and 18%FA mixes

At 7 days, the fly ash mixes had a higher percentage of voids at all curing temperatures when compared to the mix without fly ash as shown in Figure 5.9. The percentage voids in the fly ash mixes is higher in the samples cured at elevated temperatures. For both mixes with and without fly ash, the percentage of voids at 91 days was least in the samples cured at 73°F.
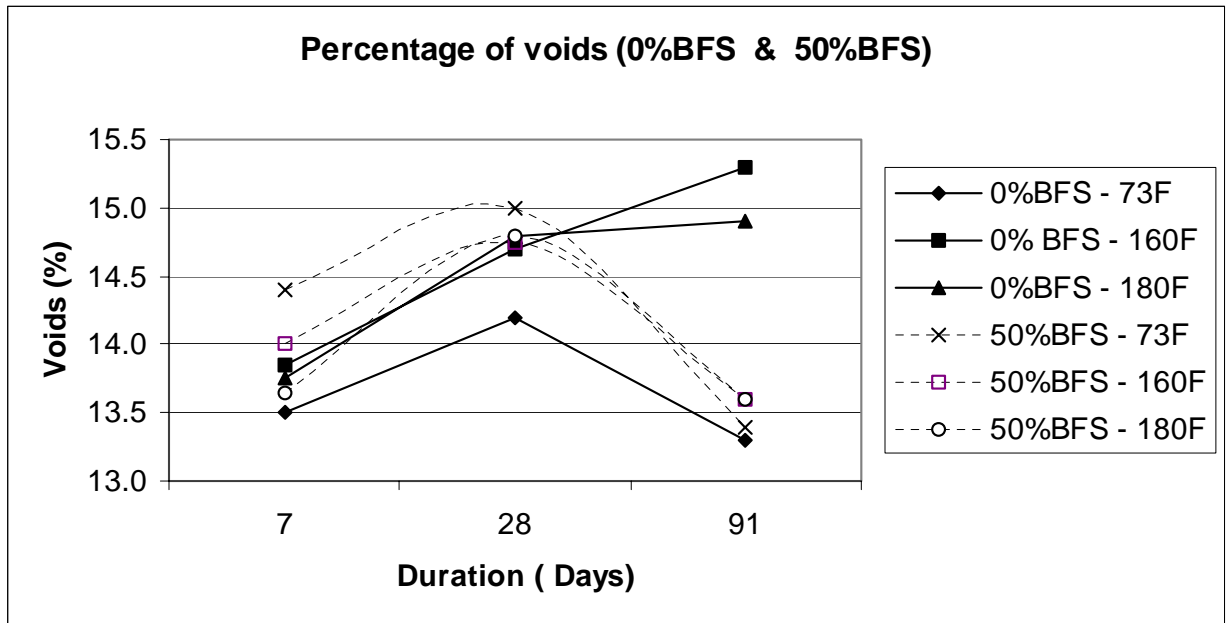
Figure 5.10 Percentage of voids for 0%BFS and 50%BFS mixes

At 7 days, the mixes with slag showed a lower percentage of voids in the samples

cured at the elevated temperatures, however, this situation was reversed at 91 days in

which the percentage of voids was lower in the samples cured at 73°F. At all curing

temperatures and durations, the mix without slag cured at 73°F had the least percentage

of voids as seen in Figure 5.10.

5.3.5 Time to Corrosion results

The corrosion results for each mix and curing temperature is presented

individually as an average of three samples. The results as shown in Figure 5.11 indicate

the increase in the concrete durability by the use of slag. The use of fly ash also increased

the time to corrosion when compared to the plain cement mixes, but to a smaller extent.

Increasing the curing temperature for all the mixes resulted in reduction of time to

corrosion.

Time to Corrosion (TTC) - Impressed current
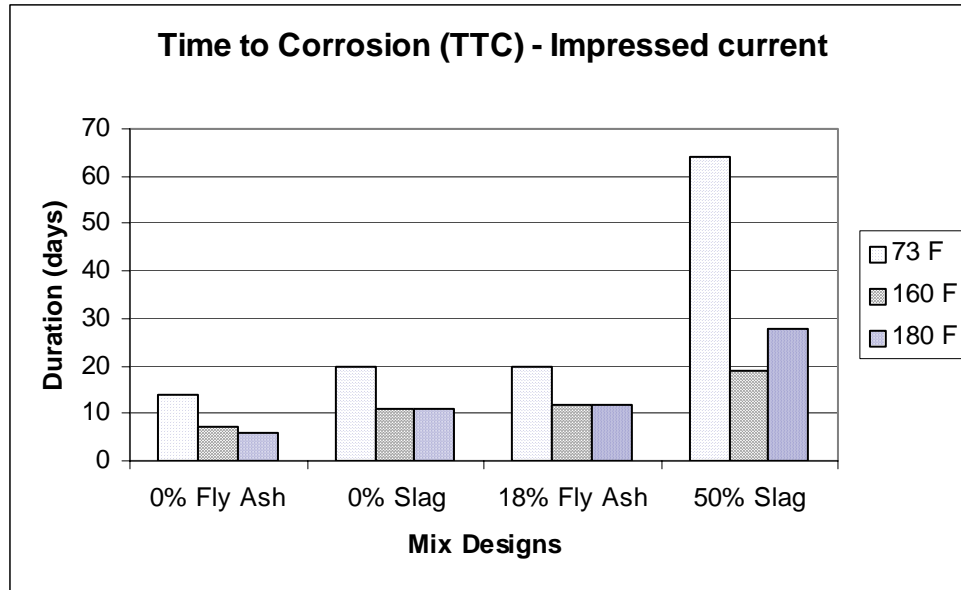
Figure 5.11 Time to Corrosion results for all mixes

RCP (91 days) - Expressed in terms of TTC unit
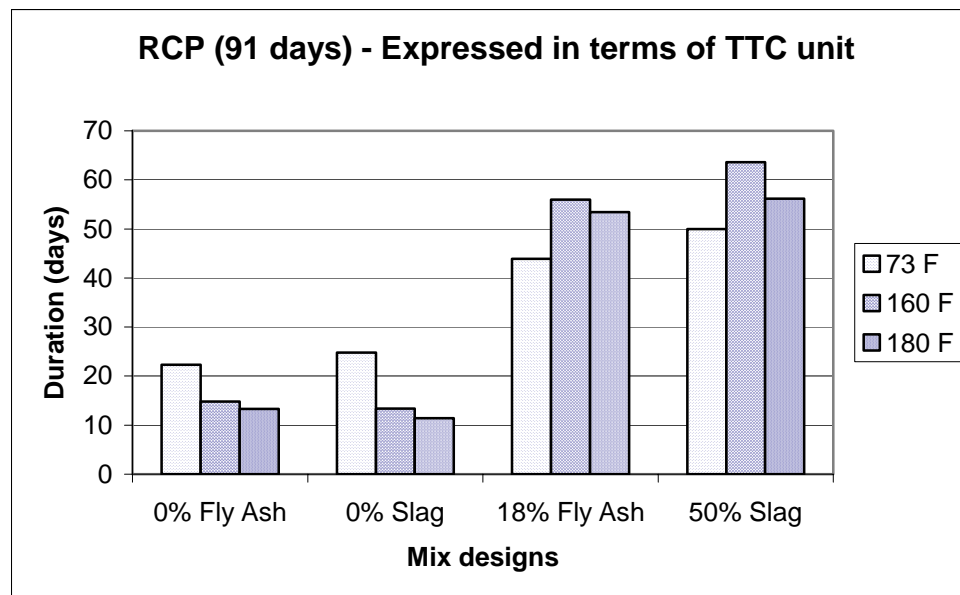
Figure 5.12 RCP at 91days expressed in terms of Time to Corrosion unit

Three parameters were used in this research to study the durability of the concrete:

1. Percentage of voids

2. Rapid chloride permeability (RCP) and

3. Time to corrosion (TTC) – Impressed current

The following observations were made in relating these parameters:

- The percentage of voids determined for the various samples did not have much variation in the values and could not be used to establish differences in the durability of the mixes.

- The RCP and TTC tests exhibited similar results for the plain cement mixes (0% FA and 0% BFS), that is both tests showed reduction in durability when curing temperature increased (see Figures 5.11 and 5.12).

- The mixes with the Fly ash and slag showed conflicting durability results from the RCP and TTC tests as seen in Figures 5.11 and 5.12. The TTC test indicated better durability for the samples cured at 73°F (Figure 5.11), whereas the RCP test showed that the Fly ash and slag mixes had better durability at the higher curing temperatures.

5.4 Phase 3 – Microstructural Analysis

5.4.1 Introduction

Initial examinations of samples from mixes with Portland cement as the sole binder (0%FA) cured at the different temperatures showed no formation of ettringite

for the first 7 days of curing. Later examinations were therefore limited to samples cured

for 28 days and 91 days. Below is a summary of the observations made. All images

shown are from secondary electron micrographs.

5.4.2 Mix 1 – Portland cement only mix (0%FA)

1. Curing temperature - 73F

    a. 28days - Voids contain well-defined hexagonal plates of monosulphate as
        shown in Figure 5.13. There were no ettringite crystals found. Lots of
        voids were found in the sample. Figure 5.14 shows an EDAX image with
        sulfur peak being the lowest when compared to the aluminum and calcium
        peaks indicating the presence of monosulphate in the sample tested.

    b. 91days - There was still no sighting of ettringite in the voids. Voids
        contain well-defined hexagonal plates of monosulphate as was seen at 28
        days.

2. Curing temperature – 160F

    a. 28days - Samples contain fewer voids than that cured at 73F. There is no
        sighting of monosulphate crystals. Voids contain clusters of fibrous
        crystals of ettringite shown in Figure 5.15. EDAX for ettringite shown in
        Figure 5.16

    b. 91days - Voids can be seen as shown in Figure 5.17 containing hexagonal
        plates of monosulphate with transformation to fibrous crystals of ettringite
        in progress.

3. Curing temperature – 180F

    a. 28days - Samples contain more voids than that cured at 160F. Some of the voids appear completely full of long crystalline fibrous ettringite (Figure 5.18)

    b. 91days - There are many voids in this samples and every one of them contains monosulphate and ettringite crystals as in Figure 5.19. In some of the voids such as Figure 5.20, all the crystals of monosulphate has transformed into ettringite crystals.
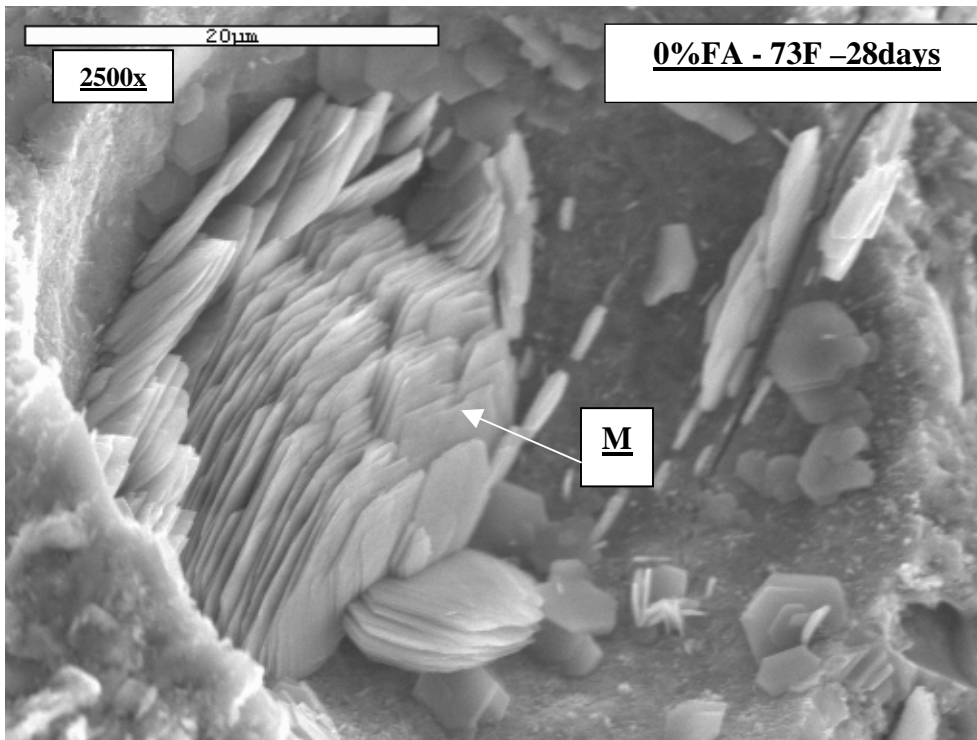
Figure 5.13 Well-defined Monosulphate (M) crystals in void
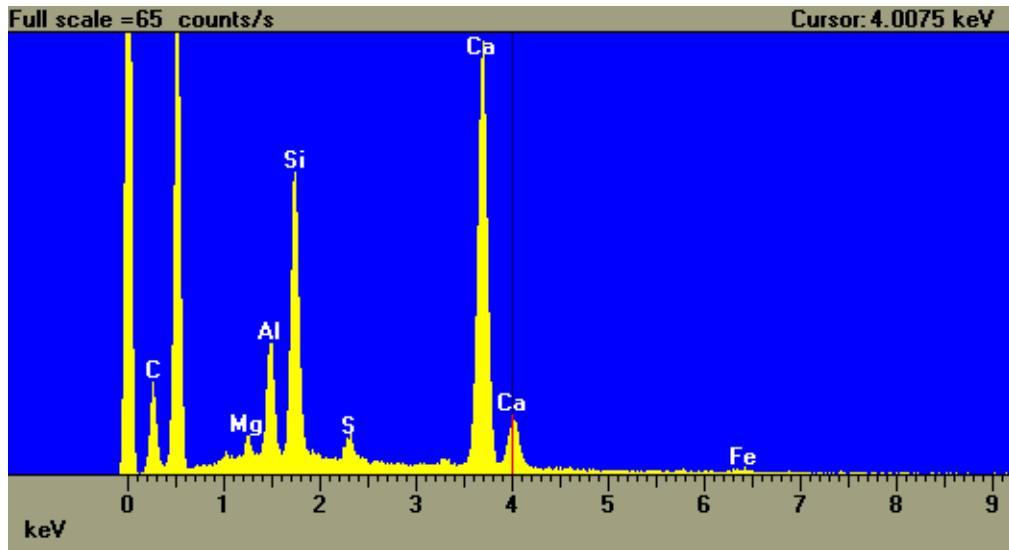
Figure 5.14 EDX showing typical Aluminum (Al), Sulphur (S) and Calcium (Ca), peaks
for monosulphate.
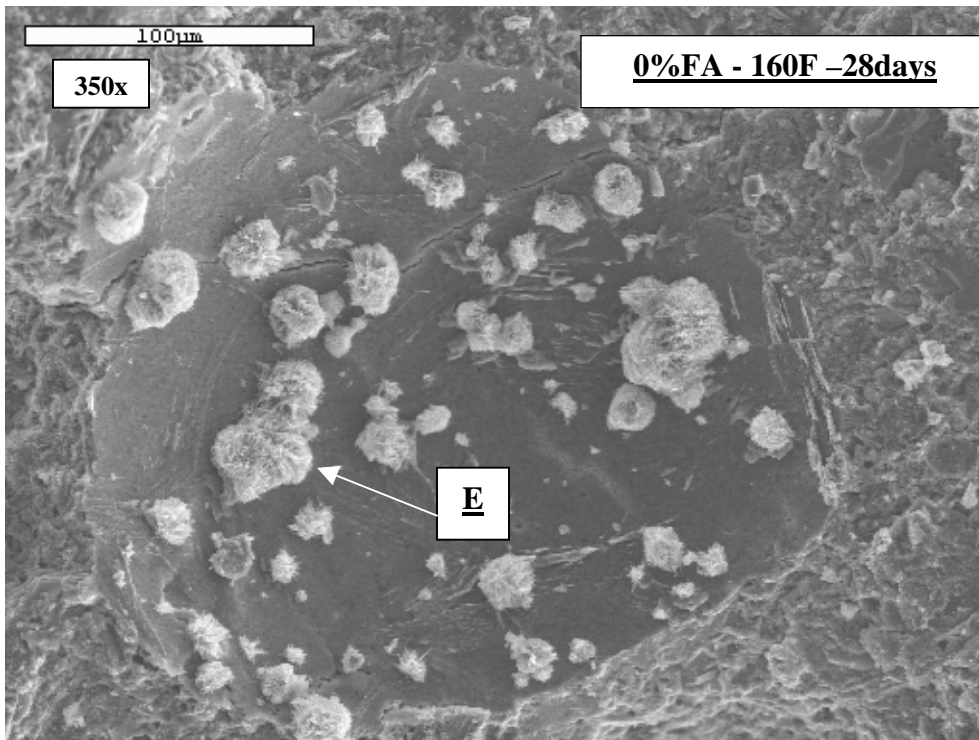
Figure 5.15 Void with clusters of Ettringite (E) crystals
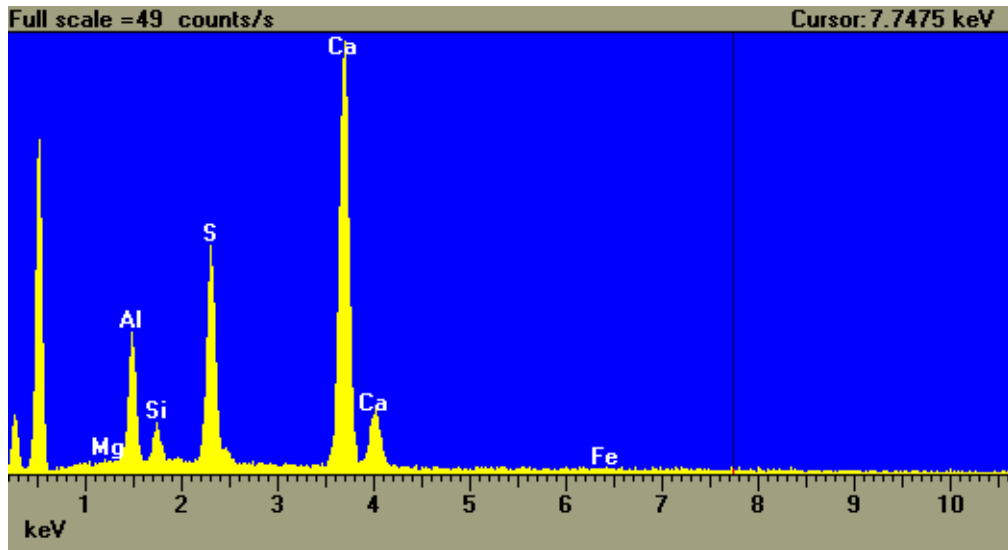
Figure 5.16 EDAX spectrum showing typical Aluminum (Al), Sulfur (S) and Calcium (Ca), peaks for ettringite
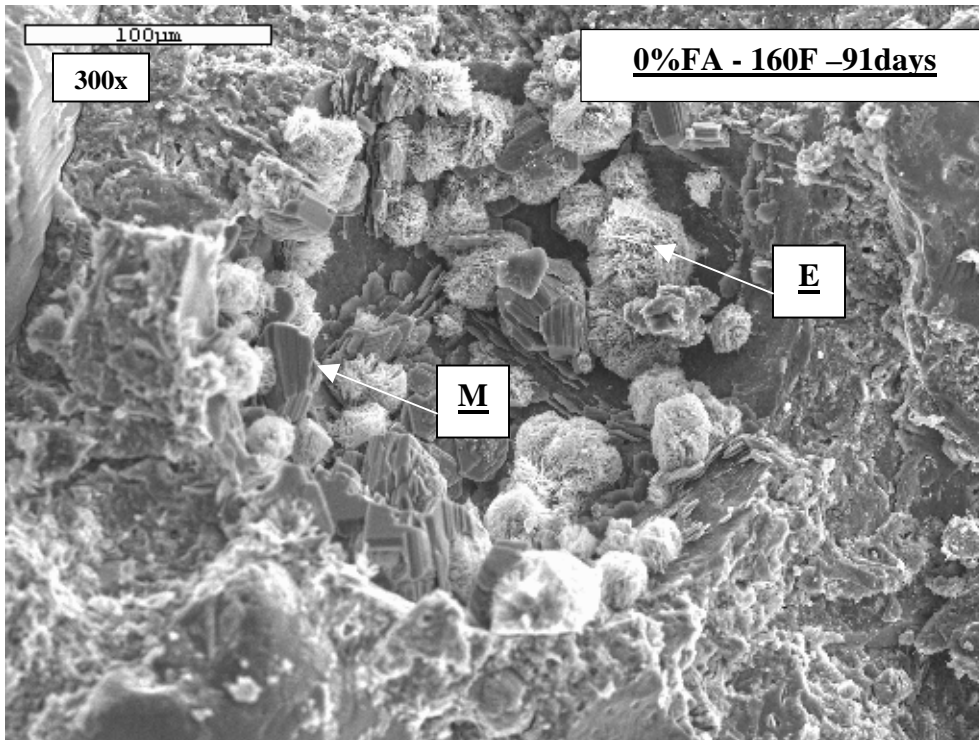
Figure 5.17 Void containing both Monosulphate (M) and Ettringite (E) crystals
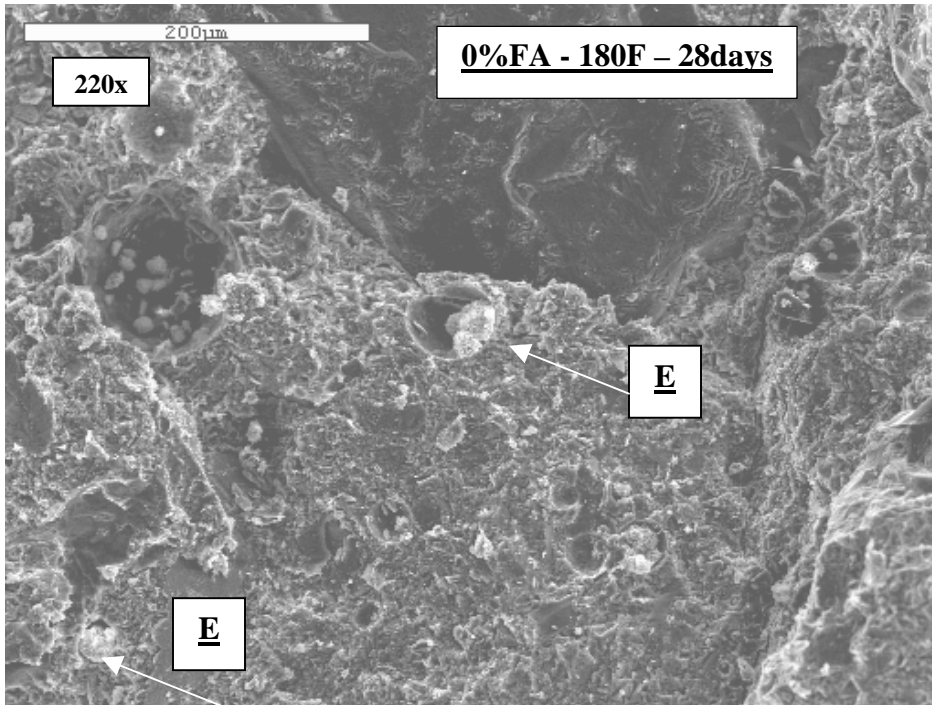
Figure 5.18 Voids containing Ettringite (E) some appear almost full of it.
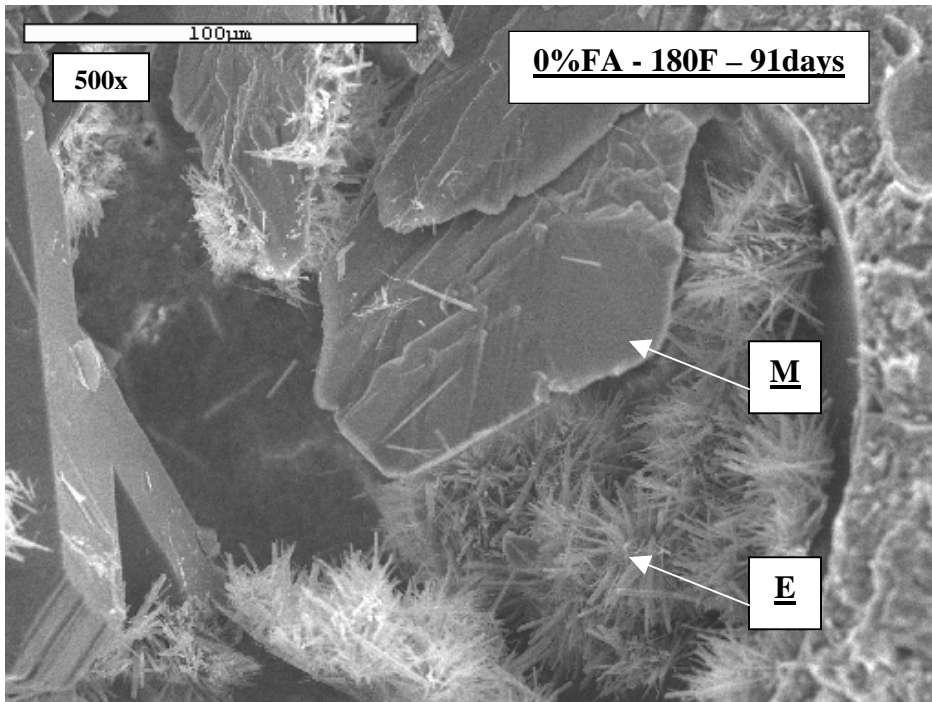
Figure 5.19 Void containing both Monosulphate (M) and Ettringite (E)
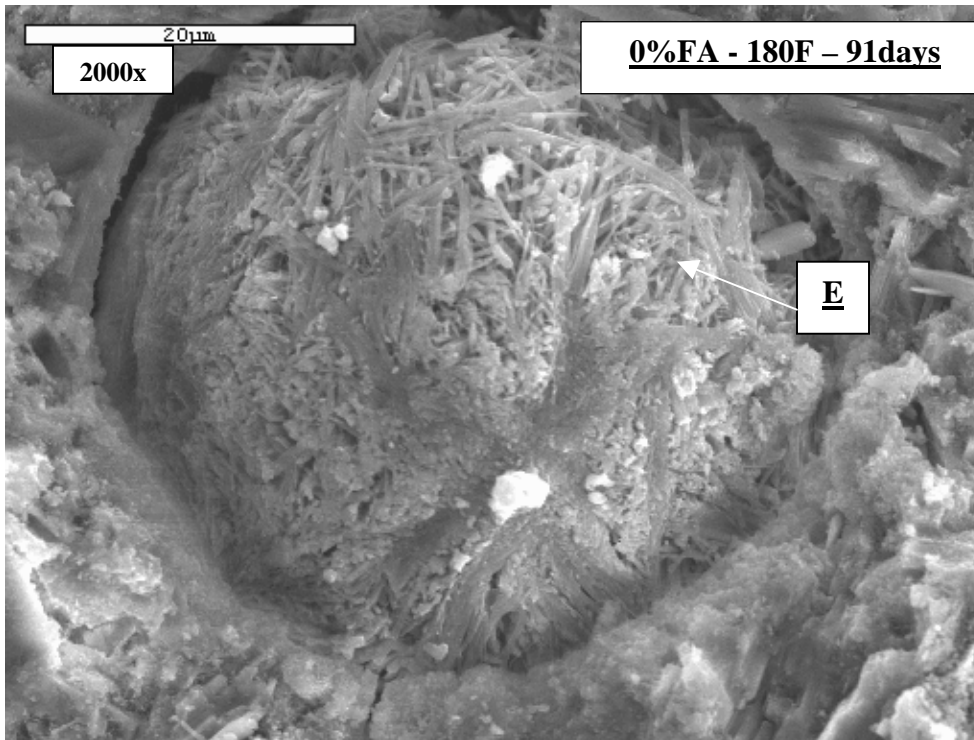
Figure 5.20 Void completely filled with fibrous Ettringite (E)

5.4.3 Mix 2: Portland cement / fly ash mix (18%FA)

1. Curing temperature - 73F

    a. 28days - There are fewer voids in this sample when compared to the plain
       Portland cement sample cured under similar curing conditions. There were
       no ettringite crystals found.

    b. 91days - There is no monosulphate or ettringite found in the voids. Figure
       5.21 shows a reacting particle of fly ash.

2. Curing temperature – 160F

a. 28days - Sample has fewer air voids compared to the corresponding plain

   Portland cement sample. Some of the voids contain hexagonal plates of

   monosulphate crystals as shown in Figure 5.22. Figure 5.23 shows an

   EDAX image of a fly ash particle with high peaks for silica and calcium

   typical of fly ash.

b. 91days - Voids contain clusters of fibrous crystals of ettringite as shown in

   Figure 5.24.

3. Curing temperature – 180F

   a. 28days - Sample has fewer air voids compared to the corresponding plain

      Portland cement sample. Voids contain both hexagonal plates of

      monosulphate with transformation into fibrous crystals of ettringite

      (Figure 5.25).

   b. 91days - Clusters of fibrous ettringite can be seen in voids (Figure 5.26).

      A reacting particle of fly ash is seen in Figure 5.27.
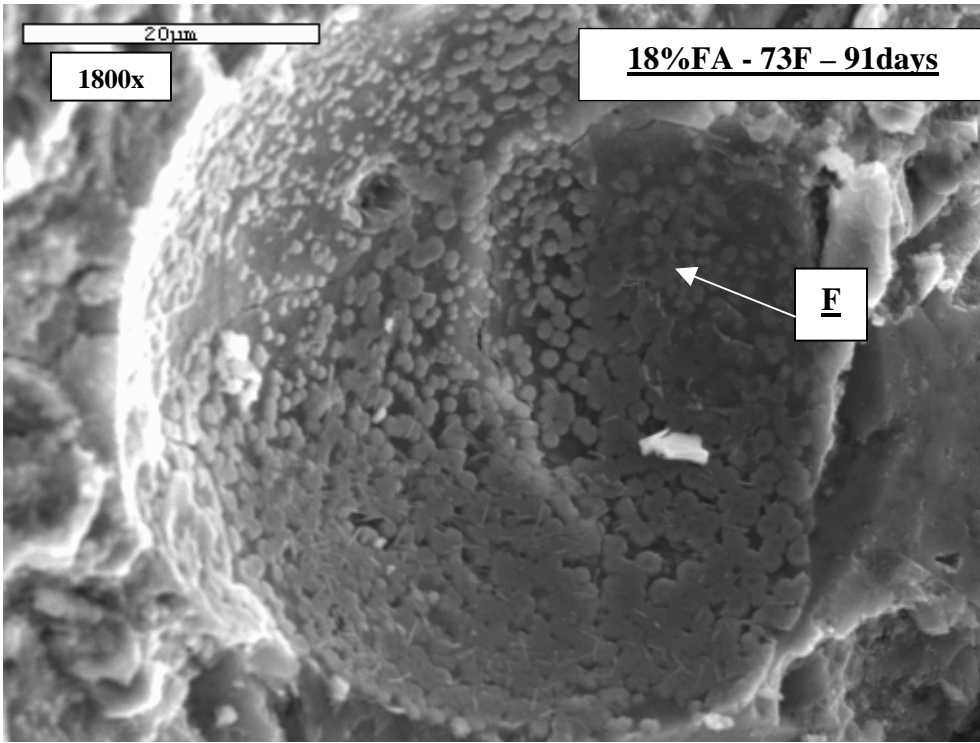
Figure 5.21 A reacting Fly ash (F) particle
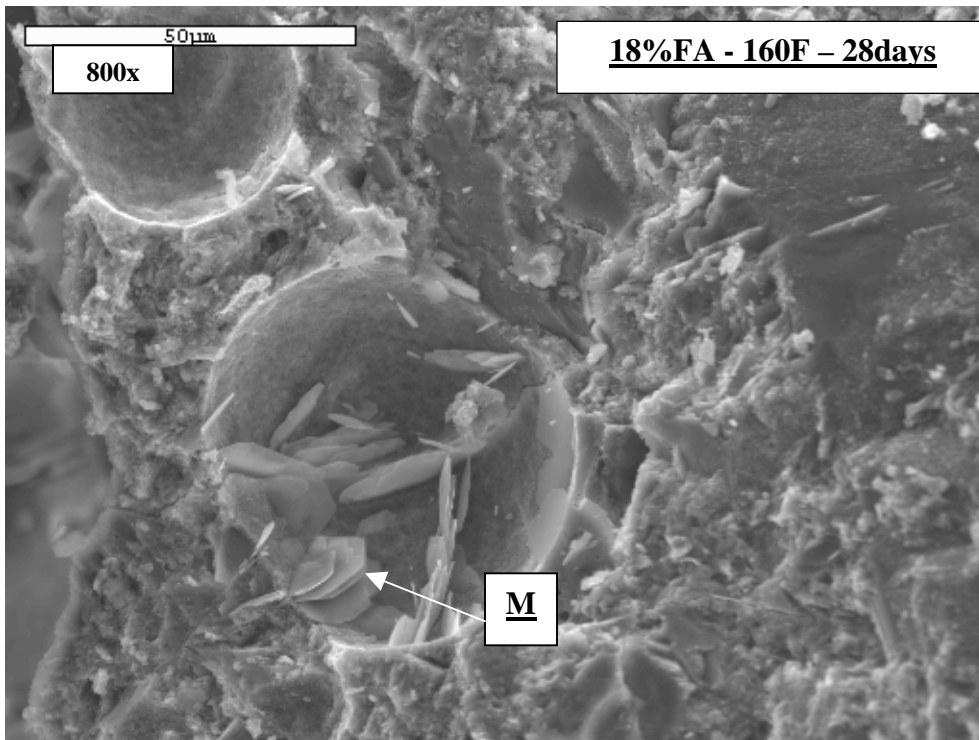
Figure 5.22 Void containing hexagonal plates of Monosulphate (M).
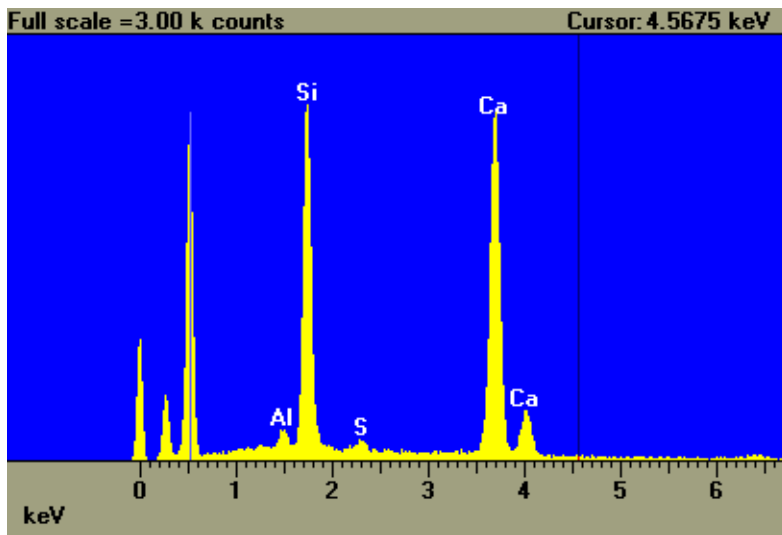
Figure 5.23 EDAX of fly ash particle
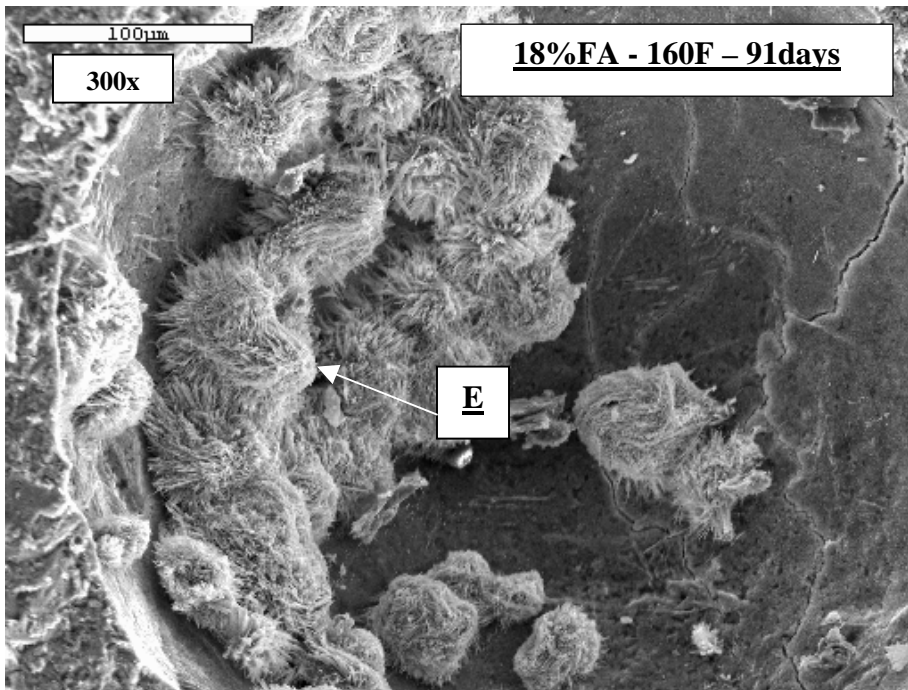
Figure 5.24 Void containing fibrous crystals of Ettringite (E)
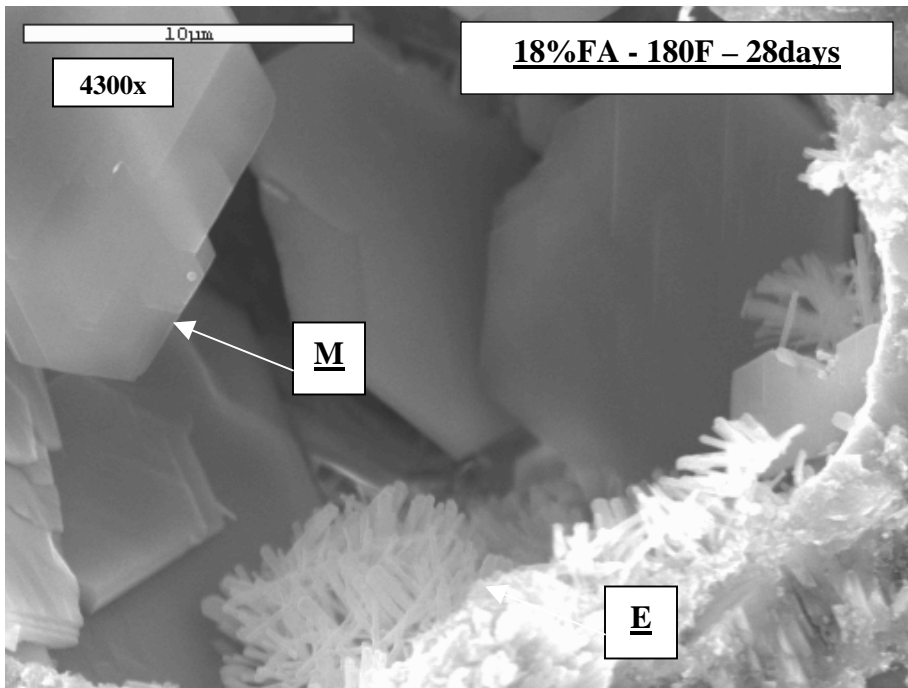
Figure 5.25 Void showing Monosulphate (M) transformed into Ettringite (E)
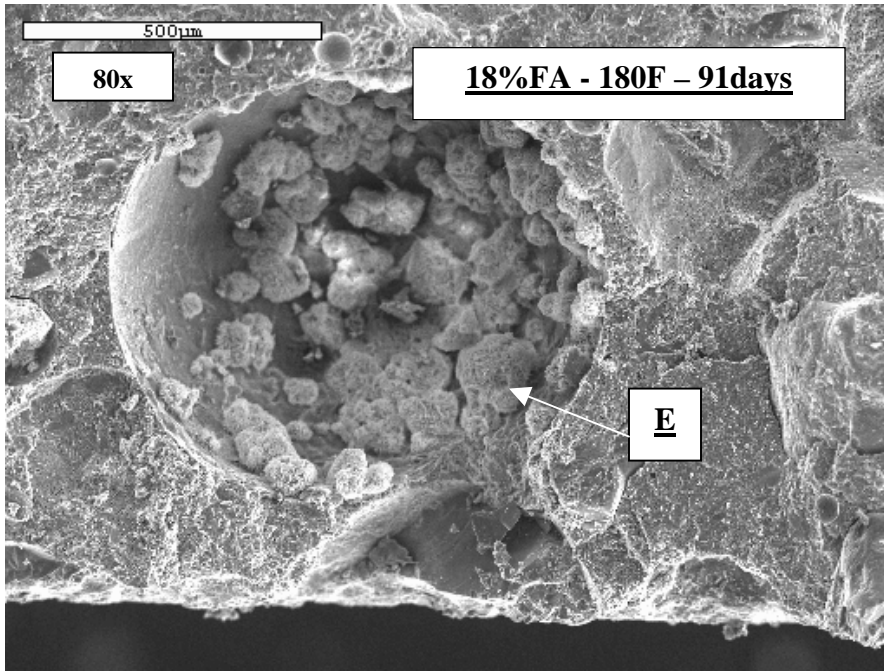
Figure 5.26 Clusters of Fibrous Ettringite (E) in void
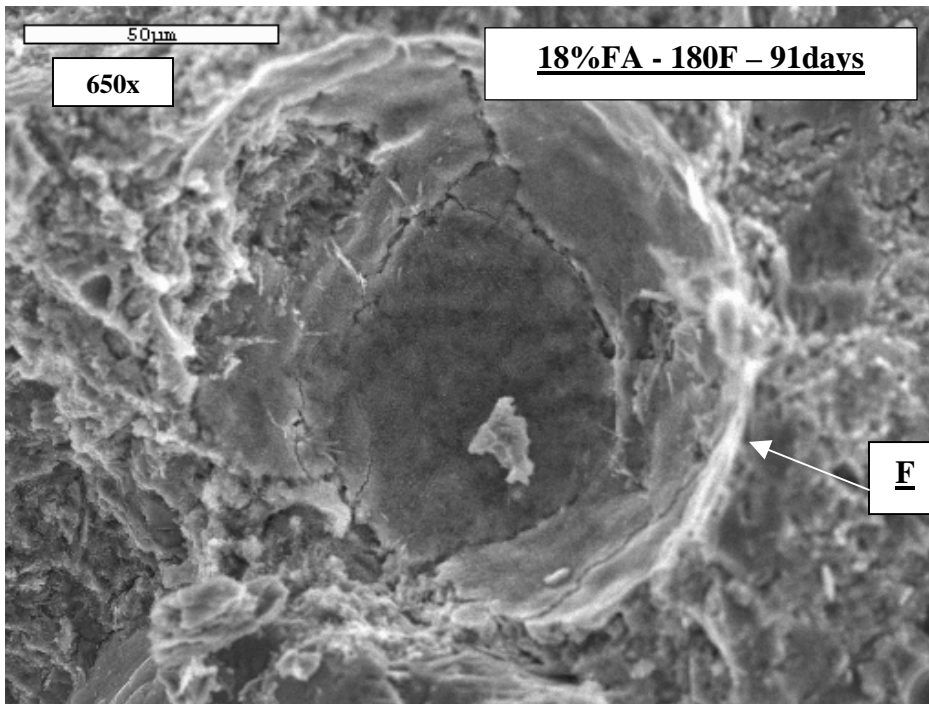
Figure 5.27 A particle of Fly ash (F) in chemical reaction

5.4.4 Mix 3 – Portland cement / Blast furnace slag mix (50%BFS)

1. Curing temperature - 73F

   a. 28days - Compared with other samples cured under similar conditions, the Portland/blast furnace samples have the fewest voids and have a uniform and dense appearance. Voids contain hexagonal plates of monosulphate (Figure 5.28). There were no ettringite crystals found. There are very few voids in this sample.

   b. 91days - There is still no sighting of ettringite in the voids. Figure 5.29 shows the break-up of a reacting slag particle. The EDAX of the slag particle is shown in Figure 5.30.

2. Curing temperature – 160F

   a. 28days - Compared with other samples cured under similar conditions, the Portland/blast furnace samples have the fewest voids and have a uniform and very dense appearance. These voids are empty with no sighting of monosulphate or ettringite crystals as shown in Figure 5.31.

   b. 91days - The very few voids found in the sample are empty of monosulphate or ettringite as shown in Figure 5.32. The microstructure of the sample appears very dense and uniform. Figure 5.33 shows a close-up view of a reacting slag particle. At very high magnification (Figure 5.34), the slag particle shows reaction products of hexagonal plates of

monosulphate as well as the early transformation into fibrous ettringite
crystals.

3.   Curing temperature – 180F

   a.   28 days - Compared with other samples cured under similar conditions,
        the Portland/blast furnace samples have the fewest voids and have a
        uniform and very dense appearance. These voids as shown in Figure 5.35
        are empty with no sighting of monosulphate or ettringite crystals.

   b.   91days – There were very few voids found in the sample these voids were
        sporadic and empty of monosulphate or ettringite. However, some
        ettringite crystals were found located in the vicinity of the reacting slag
        particles (Figure 5.36). Figure 5.37 shows a reacting slag particle with the
        surface covered with ettringite crystals.
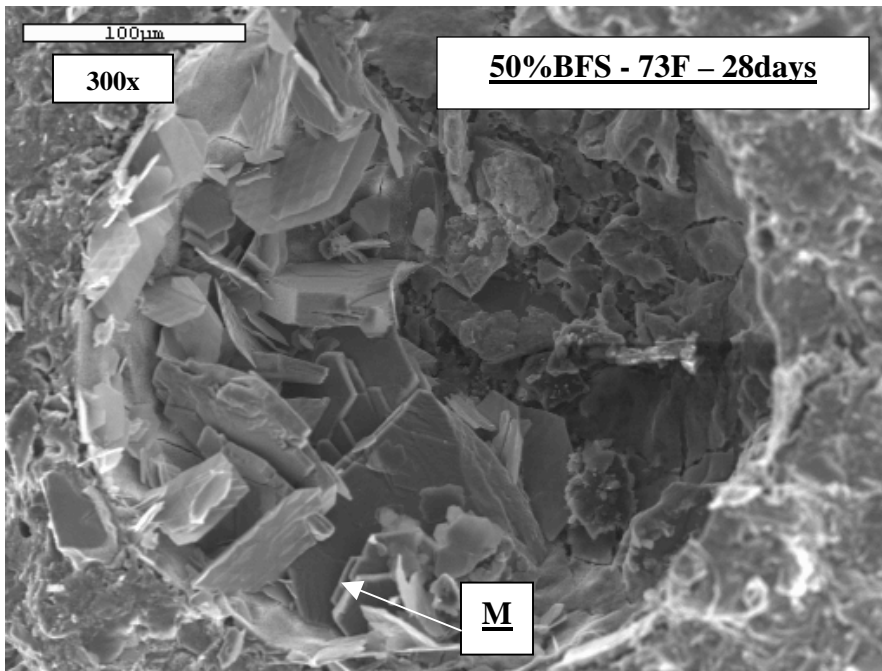
Figure 5.28 Void containing Monosulphate (M) crystals
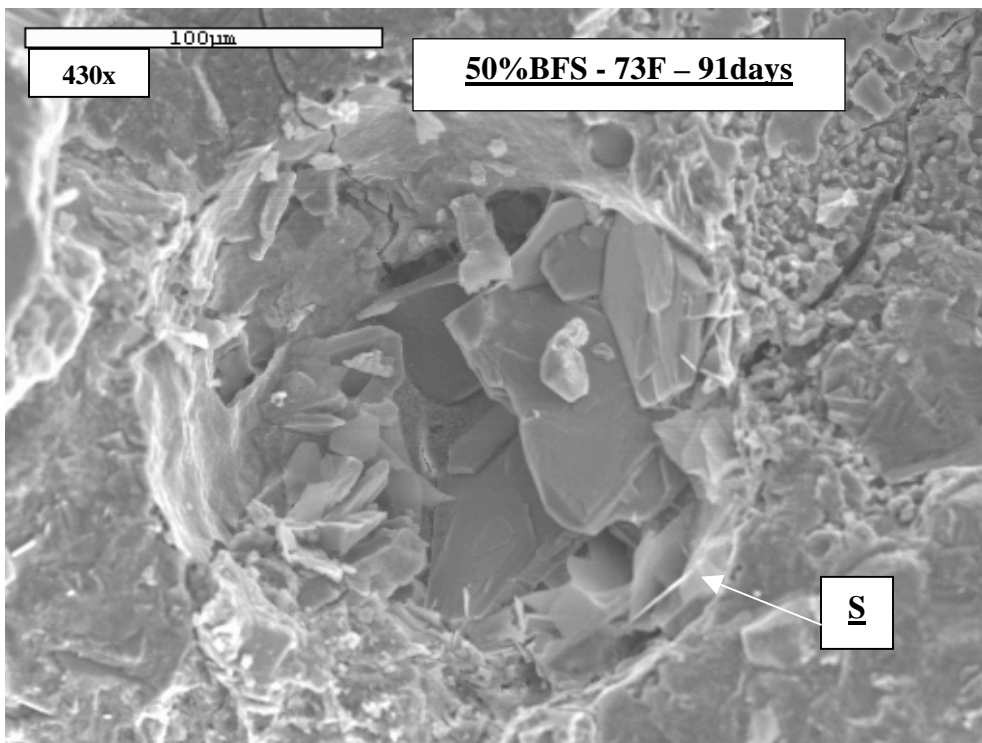
Figure 5.29 Reacting Slag (S) particle
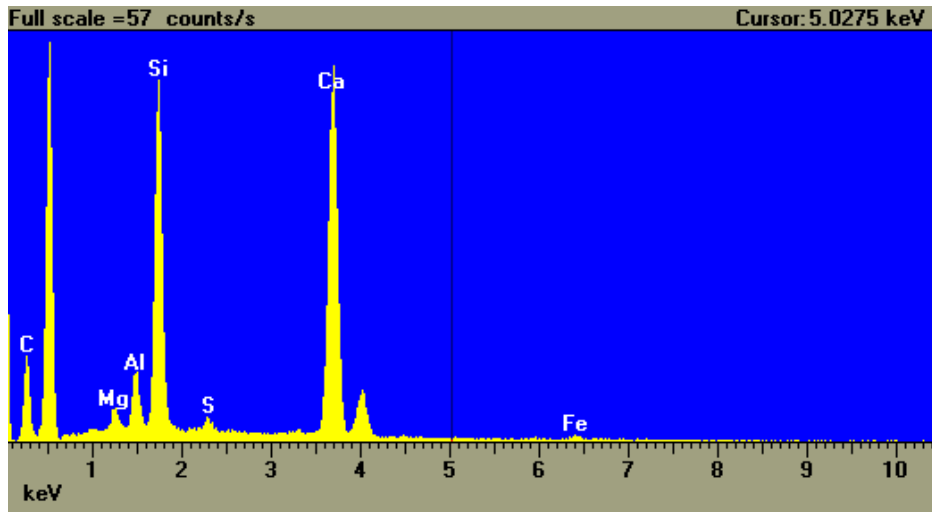
Figure 5.30 EDAX of slag particle
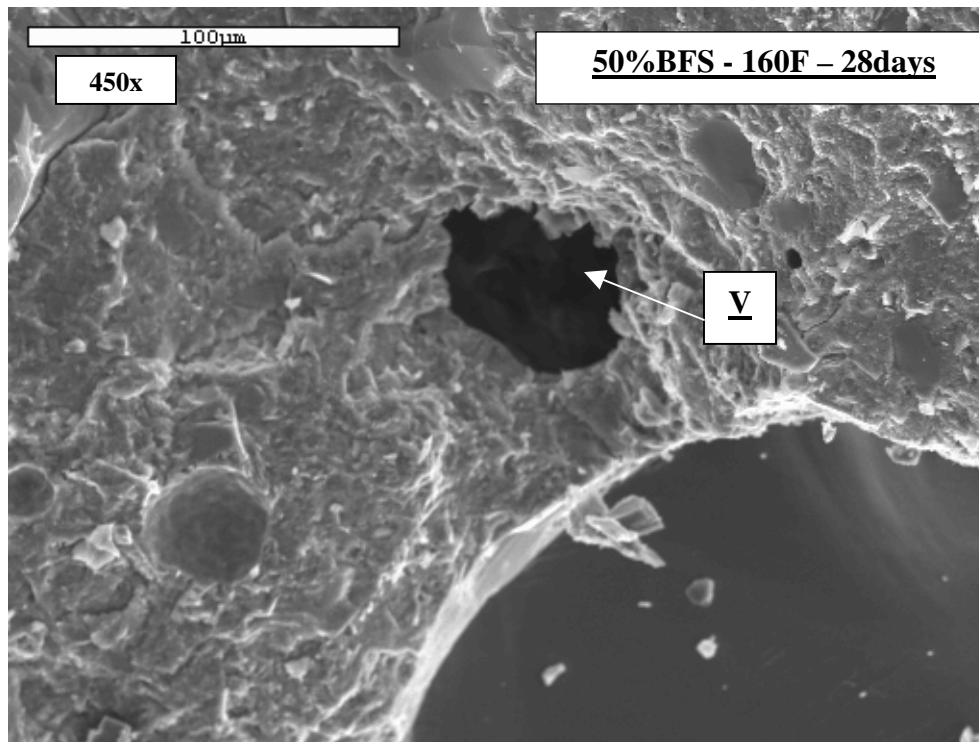
Figure 5.31 Sample with empty air Voids (V)
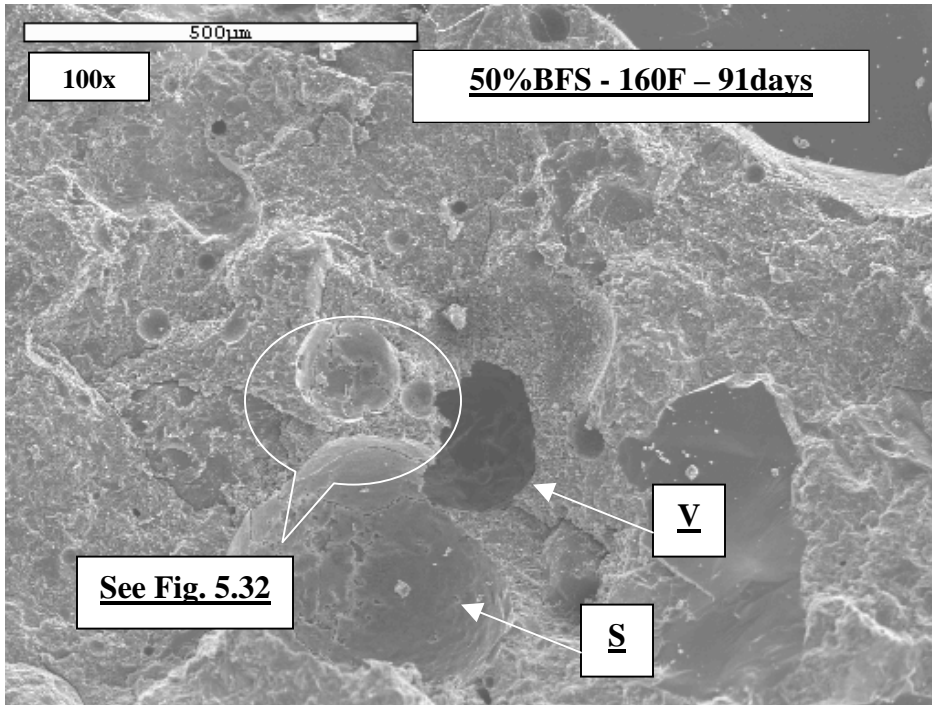
Figure 5.32 Sample with dense and uniform microstructure and empty air voids
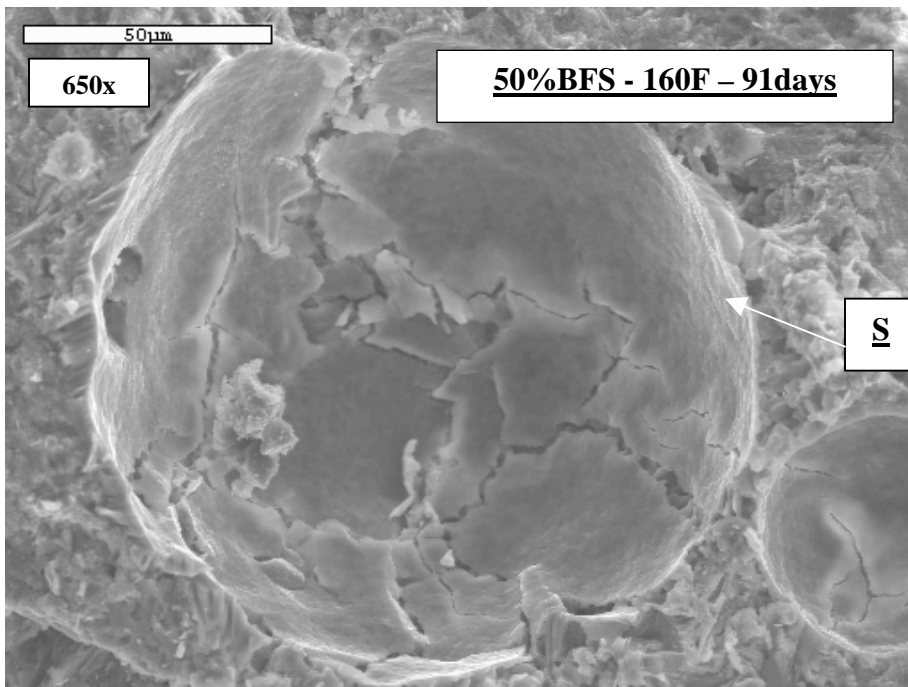
Figure 5.33 Reacting slag (S) particle at higher magnification
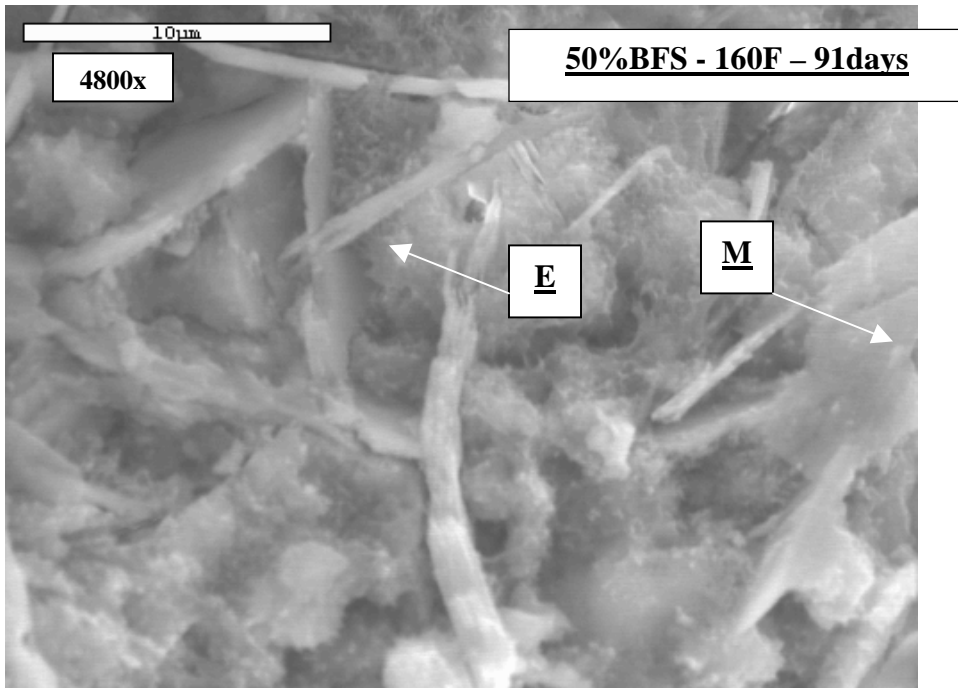
Figure 5.34 Higher magnification of Figure 5.32
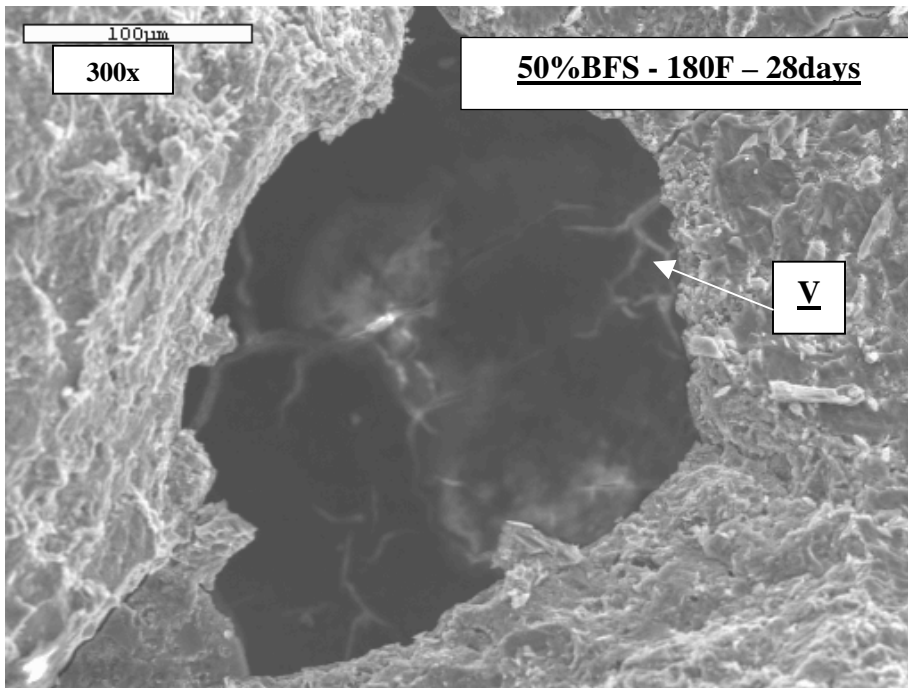
Figure 5.35 Sample with empty air Void (V)
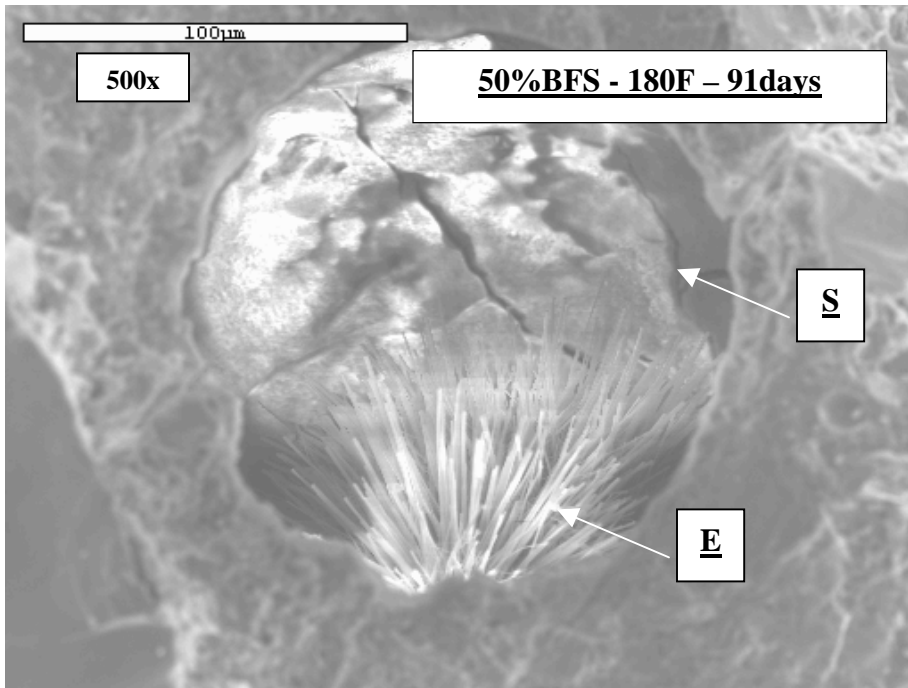
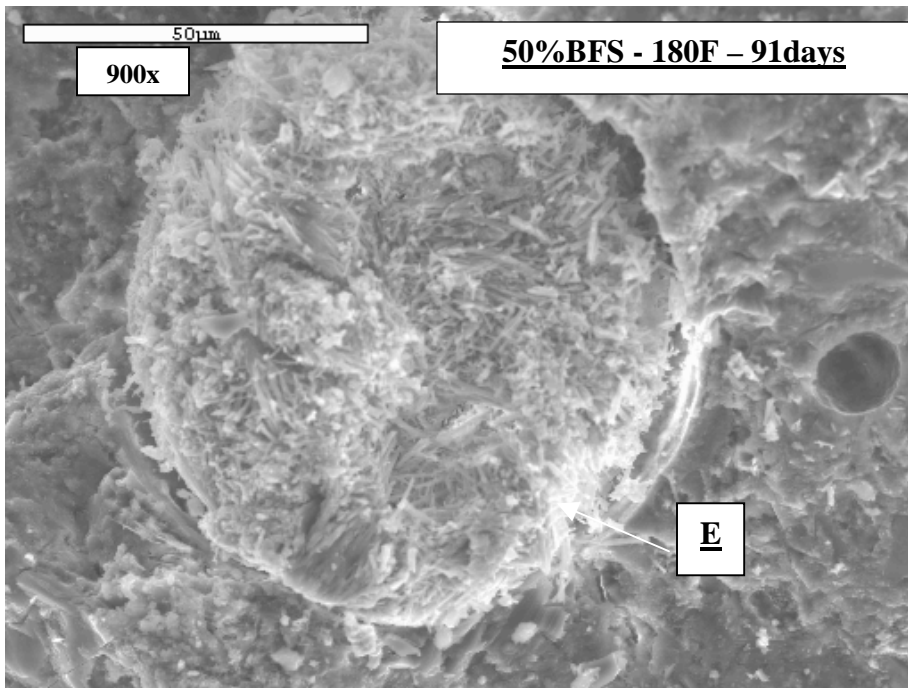Figure 5.36 Reacting Slag (S) particle with Ettringite (E) formed

Figure 5.37 Slag particle completely covered with Ettringite (E)

Table 5.15 summarizes the observations made of formation of ettringite in samples from mixes with plain and blended cement cured for 28 and 91 days at different curing temperatures.

Table 5.15 Summary of ettringite observations

Duration (days)	Temperature (^{o}F)	Mixes		
		Plain cement	Cement/Fly ash	Cement/Slag
28	73	None	None	None
	160	Ettringite	None	None
	180	Ettringite	Ettringite	None
91	73	None	None	None
	160	Ettringite	Ettringite	Ettringite (low)
	180	Ettringite (high)	Ettringite	Ettringite (low)

5.4.5 Findings

1. There was no ettringite found at 7 days for both the plain and blended cements at all curing temperatures.

2. There was no ettringite found in both the plain and blended cements at a curing temperature of 73°F for all curing durations (7, 28 and 91 days).

3. At the elevated curing temperatures of 160°F and 180°F, ettringite was found in the plain cement at 28 days.

4. The use of pozzolanic materials had a delaying effect on the formation of ettringite. The fly ash blend showed ettringite formed at 28 days only at a curing temperature of 180°F. Whilst the slag blend showed no ettringite formed at 28 days at all curing temperatures. At 91days, the blended as well as the plain

cements showed the formation of ettringite at the elevated curing temperatures.

5. At corresponding curing temperatures of 160°F and 180°F, more ettringite was observed in the plain cement mix than in the blended cements at 28 and 91 days of curing.

6. At the elevated curing temperatures of 160°F and 180°F, more ettringite was found at 91 days than at 28 days of curing for all mixes.

7. The presence of ettringite was confirmed through energy dispersive analysis of the x-rays (EDAX).

8. More voids were observed in the plain cement mix compared to the blended cement mixes. The void ratio increased with increased curing temperature for the plain cement mixes. Although this observation is in conflict with the results of the percentage of voids, it agrees with the increased RCP results which were higher in the plain cement mixes and lower in the blended mixes.

9. At 28 days and 91 days for all curing temperatures, the microstructure of the blended cement mixes appeared denser than the plain cement mix.

CHAPTER 6 CONCLUSIONS AND RECOMMENDATIONS

This research project was performed to investigate the durability of mass concrete at elevated curing temperatures. It provides a literature review, which document current known industry challenges relative to mass concrete. The literature review of the past studies revealed that higher curing temperatures increase the initial strength, but decrease the later-age strength. Additionally, in plain Portland cement concretes, elevated curing temperatures result in coarser pore structure and increase total porosity mostly in the volume of larger pores. This suggests that high curing temperatures could reduce durability of plain cement concrete since large pores have the greatest effect on permeability and reduce the concrete resistance to chloride ion penetration. Corrosion of reinforcing steel is a result of chloride ions, and cause premature deterioration of concrete structures. Another problem associated with high concrete curing temperature reported in the literature is early concrete distress due to delayed ettringite formation (DEF). Ettringite is a mineral composed of hydrous basic calcium and aluminum sulfate. When cement hydrates, it creates ettringite high form. As it looses water the ettringite goes to a low form. At high curing temperatures (175 F) the low form returns to the high form and causes micro cracking.

There was also a survey conducted of current US Highway Agency specification relative to mass concrete. It gave valuable information relative to the current opinions of highway agencies on the topic of specifications required for mass concrete. The majority of the US Highway Agencies that responded to the survey (65%) agreed that mass

concrete pours should be controlled by a maximum differential temperature, which most agencies currently specify, and a maximum curing temperature, which most agencies do not currently specify. The reasons given to support their concerns for specifying a maximum curing temperature was to avoid durability problems, later age strength reduction, delayed ettringite formation, and cracking due to expansion of concrete. The opinions of the US Highway Agencies were similar to the concerns noted in the literature review. Both noted the affect of high temperature relative to the reduced durability of the concrete and delayed ettringite formation.

The experiment performed first attempted to determine the historical development of the degree of hydration in cement pastes with 18% fly ash and 50% blast furnace slag at different adiabatic curing temperatures. The objective was to identify a point at which the concrete has reached a certain percentage of hydration and measure concrete properties at this point. This occurs when the development of degree of hydration reduces drastically, to a point where additional time does not provide a significant increase in the degree of hydration (approximately 70 percent). However, the results of the tests were not consistent and did not allow accurate determination of number of days required for each mix to reach a degree of hydration of approximately 70 percent at different adiabatic curing temperatures. It was decided instead to measure properties of concrete at 7, 28, and 90 days and measure the degree of hydration at these ages.

The compressive strength test results showed that:

- Higher curing temperatures resulted in higher early age strength and lower later age strength for mixes with and without fly ash.

- Addition of fly ash increased the strength at all ages and curing temperatures when compared to the mix without fly ash, mirroring the higher degree of hydration of the fly ash mixes over the plain cement mixes.

- Higher curing temperatures resulted in increased early age strength in slag mixes but much reduced later age strength.

- Higher curing temperatures did not have any pronounced effect on the early age strength in the mixes without slag, however at later ages, the higher temperatures resulted in a decrease in the strength compared to those samples cured at lower temperatures.

- The mix with slag had higher strengths at all curing durations and temperatures compared to the mix without the slag, mimicking the higher degree of hydration in the slag mixes over the mixes without slag.

The results of the resistance to chloride ion penetration tests revealed that:

- At higher curing temperatures, the mixes without fly ash had higher chloride penetration at both 28 and 91 days. For the fly ash mixes however higher curing temperatures resulted at much reduced chloride ion penetration at 28 days. The influence of curing temperature on chloride penetration at 91 days was minimal. Overall, the fly ash mixes had lower chloride ion penetration at all curing

durations and temperatures when compared to the mixes without fly ash.

- The mix without slag showed increased RCP values at higher temperatures. The RCP values for the slag mixes were considerably reduced compared to mixes without slag, and reduced slightly when curing temperature increased.

The results of density test exhibited that:

- The mix with fly ash exhibited a higher density at all curing temperatures and curing durations, than the mix without it.

- The curing temparature of the concrete had a minimal influence on the resulting density.

- Addition of slag has increased density for all curing temperatures and ages.

- Higher curing temperatures have slightly increased density at 91 days for mixes with and without slag.

The time-to-corrosion test suggested that:

- Increasing the curing temperature reduces the time for the rebar embedded in the sample to corrode.

- The use of fly ash or slag in the mix generally increased the time for the rebar to corrode when compared to corresponding plain cement samples cured under similar conditions.

Microstructural analysis of mortar samples sieved from the concrete mixes using the Scanning Electron Microscope (SEM) revealed that:

-        Delayed ettringite formation was not observed at 7 days at all curing temperatures. No ettringite was found at a curing temperature of 73 F for all curing durations. At the elevated curing temperature of 160 F, ettringite was found at 28 days of curing in the plain cement mix, and at 91 days of curing in plain cement and fly ash blend mixes. At the elevated curing temperature of 180 F, ettringite was found at 28 days of curing in the plain cement and fly ash blend mixes; and at 91 days of curing in plain cement, fly ash blend, and slag blend mixes. More ettringite was found in the plain cement mix than in the blended cement mixes at all curing temperatures.

-        More voids were observed in the plain cement mix compared to the blended cement mixes. The void ratio increased with increased curing temperature for the plain cement mixes. Although this observation is in conflict with the results of the percentage of voids, it agrees with the increased RCP results which were higher in the plain cement mixes and lower in the blended mixes.

-        At 28 days and 91 days for all curing temperatures, the microstructure of the blended cement mixes appeared denser than the plain cement mix.

In summary, the results of this research revealed a substantial decrease in compressive strength of plain Portland cement concrete samples cast and stored immediately in water tanks under isothermal curing temperatures of 160 F and 200 F compared to samples cured at room temperature (73 F). This reduction was 34% and 62% for 28-day compressive strength for samples cured at 160 F and 200 F, respectively. In addition, RCP test of these samples showed a significant increase in permeability of concrete cured at high temperature.

When plain Portland cement concrete samples were introduced to a controlled ascending temperature rise simulating approximately conditions of mass concretes cured in the field (semi-adiabatic temperature rise), there was a moderate reduction in 28-day compressive strength of samples cured at elevated temperatures compared to samples cured at room temperature. The reduction was approximately 15% and 18% for samples cured at temperatures of 160 F and 180 F, respectively. However, there was still a significant increase in permeability of concrete measured through RCP test.

Semi-adiabatic curing of fly ash cement concrete samples (18% fly ash by weight) resulted in 8% reduction of 28-day compressive strength for samples cured at 160 F and 180 F compared to those cured at room temperature. However, permeability of concrete measured by RCP test improved noticeably at higher curing temperatures suggesting that at higher temperatures the fly ash becomes effective much earlier and reduces the RCP values. At normal curing temperature the RCP reducing effect of fly ash becomes effective after approximately two months. However, time-to-corrosion test results did

not support this finding and showed reduction in time to corrosion for samples cured at higher temperatures compared to those cured at room temperature.

When 50% (by weight) of Portland cement is replaced by blast furnace slag, the 28-day compressive strength of samples cured adiabatically at elevated temperatures reduced by 7% and 15% for curing temperatures of 160 F and 180 F compared to those cured at room temperature. Durability of concrete in this case again showed conflicting results from RCP test and time-to-corrosion test, i.e., RCP test results indicated higher curing temperatures improve durability, whereas, time-to-corrosion test results showed a less durable concrete when it is cured at elevated temperature.

Results of compressive strength tests and RCP tests revealed that addition of blended cement improves strength and durability of concrete.

Microstructural analysis of mortar samples sieved from the concrete mixes using the Scanning Electron Microscope (SEM) showed that addition of pozolanic materials reduces the possibility of formation of delayed ettringite. It also identified the formation of delayed ettringite in samples 28 days and older where curing temperature was 160F and 180F. No DEF was found in concrete samples cured at room temperature.

Recommendations

The objective of this research was to investigate the performance of Portland cement concrete cured at elevated temperatures and determine if reported high curing temperatures (170°F to 200°F) in the Florida Department of Transportation mass concrete projects have detrimental effects on strength, durability and other physical/chemical

properties of concrete. Based on findings of this research project it is recommended that:

1. Use of fly ash or slag as a cement replacement should be required in mass concrete since these pozzolanic materials reduce the detrimental effect of high curing temperature on strength and durability of pure cement concrete. In addition, more ettringite was found in the pure cement concrete mix than in blended cement at 28- and 91-day curing, and the microstructure of the blended cement mixes appeared denser than the pure cement mix.

2. When pozzolanic materials are used as a cement replacement, based on ideal laboratory conditions and accurate batching proportions we found an 8 to 15% reduction in compressive strength due to elevated curing temperatures. However, this loss could be inflated considerably if the concrete was produced at a batch plant with wider mixer proportions tolerances and the ever-present potential of unmetered water in the mix. Formation of delayed ettringite in samples 28 days and older where temperature was 160F and 180F is a point of concern and more study is needed to look at the microstructural analysis of samples cured at temperatures more than 160F, specifically for detection of delayed ettringite formation.

3. This study also showed that when the pure cement concrete specimens were placed in preheated curing tanks as soon as they were molded and cured under constant temperatures of 160 F and 200 F, their

compressive strengths were significantly decreased (34% and 62% for 160 and 200 F, respectively) and their permeability were increased. This shows the importance of the age of precast concrete members when they are exposed to steam curing regimen to accelerate their strength gain. The Quality Control/Quality Assurance personnel at precast yards have to be alerted of this important issue.

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APPENDIX

Appendix A – Concrete Mix Designs

TRAIL BATCH-- DATA AND CALCULATIONS
(Saturated, Surface-dry Aggregates)

0% Fly Ash

Date: *Nov. 19, 2001*

Aggregate Properties: Grade *#57*
Specific gravity: Fine *2.64* Coarse *2.42*
  Moisture Calculations:
    FA= 0.00-0.50=0.50
    CA= *5.65 - 2.60 = 3.05*

Specifications:
  Cement content *744* lb. Cement Type *I/II*

Project: *Degree of Hydration - Mix No.1*
Weights By: *Lucy, Matt. and Richard*
Mixing By: *R. DeLorenzo*
Air Content *1 to 6* %
Slump range *3"* in. ± *1.5"*

Batch *6.0* Cu.Ft. CF= *0.222*

MATERIAL	SOURCE	WT. PER CU.YD.(LB)	VOL. PER CU.YD.(CF)	WT. PER BATCH(LB)	ADJ. WT. PER BATCH(LB)	REMARKS
Cement	Southdown	744	3.79	165.2	165.2	
FLYASH						
WATER	G'ville	305	4.89	67.8	57.0	- 10.8
FINE AGG.	76-137	936	5.68	208.0	207	- 1.0
COARSE AGG.	08-005	1746	11.56	388.0	399.8	+ 11.8
AIR ENTRAINER	Darex AEA	4 oz	1.06	26.3 ml	26.3 ml	
ADMIXTURE	WRDA 64	24.4 oz		160.2	160.3	
TOTAL			27.0			

SLUMP(IN) *6½"* AIR(%) *4.0* MIX TEMP *75°F* WORKABILITY *Good* AIR TEMP *72°F*
SLUMP BY: *Fitzgerald* AIR BY: *DeLorenzo* CYLS. BY: *Joe Terry Richard / Lucy Matt Lucy* START TIME= *8:24*
W/C RATIO *.41* H20 HELD OUT *✓* ADDED *✓* END TIME= *8:32*

CYLINDERS-COMPRESSIVE STRENGTH

(/ /)
3 DAYS-
(/)
7 DAYS-
(/ /)
28 DAYS-

TIME OF SET

INITIAL SET: _____ (/)
FINAL SET: _____
REMARKS: _____

DESIGNED BY: _____
WITNESSED BY: _____

TRAIL BATCH-- DATA AND CALCULATIONS
(Saturated, Surface-dry Aggregates)

18% FlyAsh

Date: _Nov. 19, 2001_

Project: _Degree of Hydration - Mix No. 2_

Aggregate Properties: Grade _#57_
Specific gravity: Fine _2.64_ Coarse _2.42_
  Moisture Calculations:
    FA= 0.00-0.50=0.50
    CA= 5.65-2.60 = 3.05

Weights By: _Lucy, Matt and Richard_

Mixing By: _R. Delorenzo_

Air Content _1 to 6%_

Specifications:
  Cement content _744_ lb. Cement Type _I/II_

Slump range _3"_ in. ± 1.5"

Batch _60_ Cu.Ft. CF= _0.2222_

MATERIAL	SOURCE	WT. PER CU.YD.(LB)	VOL. PER CU.YD.(CF)	WT. PER BATCH(LB)	ADJ. WT. PER BATCH(LB)	REMARKS
Cement	Southdown	610	3.10	135.5	135.5	
FLYASH	Boral	134	0.91	29.8	29.8	
WATER	G'ville	205	4.89	67.8	57.1	-10.7
FINE AGG.	76-127	918	5.57	204.0	203.0	-1.0
COARSE AGG.	08-005	1729	11.45	384.2	395.9	+11.7
AIR ENTRAINER	Larex AEA	4 oz	1.08	26.3 ml	26.3 ml	
ADMIXTURE	ADVA 64	24.4 oz		160.3 ml	160.3 ml	
TOTAL			27.0			

SLUMP(IN) _5_ AIR(%) _2.0_ MIX TEMP _75°F_ WORKABILITY _OK_ AIR TEMP _72_

SLUMP BY: _Thomas Reynold_ AIR BY: _Delorenzo_ CYLS. BY: _Lucy, Matt, Green_ START TIME= _?:?0_

W/C RATIO _.41_ H20 HELD OUT ___ ADDED ___ END TIME= _9:28_

CYLINDERS-COMPRESSIVE STRENGTH

(/ /)
3 DAYS- _____

(/ /)
7 DAYS- _____

(/ /)
28 DAYS- _____

TIME OF SET

INITIAL SET: _____ (/ /)

FINAL SET: _____

REMARKS: _____

_____

_____

_____

DESIGNED BY: _____

WITNESSED BY: _____

TRAIL BATCH-- DATA AND CALCULATIONS
(Saturated, Surface-dry Aggregates)   (0% FlyAsh)

Date: Feb 5, 2002   Project: Degree of Hydration - Mix 3

Aggregate Properties: Grade __57__   Weights By: Lucy, Richard & Matt
  ecific gravity: Fine _2.64_ Coarse _2.42_
  Moisture Calculations: Mixing By: R. DeLorenzo
    FA= 0.00-0.50=0.50
    CA= 4.90-2.60 =2.30   Air Content _1.6 %_

Specifications:
  Cement content __744__ lb. Cement Type _I/II_   Slump range __3"__ in. ± 1.5"

Batch __6.0__ Cu.Ft. CF= __0.2222__

MATERIAL	SOURCE	WT. PER CU.YD.(LB)	VOL. PER CU.YD.(CF)	WT. PER BATCH(LB)	ADJ. WT. PER BATCH(LB)	REMARKS
Cement	Southdown	744	3.79	165.3	165.3	
FLYASH	—					
WATER	G'ville	305	4.89	67.8	58.7	- 9.1
FINE AGG.	76-137	936	5.66	208.0	207	- 1.0
COARSE AGG.	08-005	1746	11.56	388.0	398.1	+ 10.1
AIR ENTRAINER	Darex AEA	4 oz	1.08	26.3 mL	26.3 mL	
ADMIXTURE	WRDA C4	24.4 oz		160.3 mL	160.3 mL	
TOTAL			27.0			

,UMP(IN) _6½"_ AIR(%) _4.5_ MIX TEMP _69°F_ WORKABILITY _Good_ AIR TEMP _70°F_
SLUMP BY: _J Fitzgerald_ AIR BY: _T. Thomas_ CYLS. BY: _Lucy/Richard, Matt Robert/Fitzgerald_ START TIME= _9:07ᴬ_
W/C RATIO _0.41_ H2O HELD OUT ___—___ ADDED ___—___ END TIME= _9:15ᴬ_

CYLINDERS-COMPRESSIVE STRENGTH   TIME OF SET

(/ /)
3 DAYS-_____   INITIAL SET:_____ (/)
(/)
7 DAYS-_____   FINAL SET:_____
(/ /)
28 DAYS-_____   REMARKS:_____
_____   _____
_____   _____
_____   _____

DESIGNED BY:_____

WITNESSED BY:_____

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TRAIL BATCH-- DATA AND CALCULATIONS
(Saturated, Surface-dry Aggregates)

(18% Fly Ash)

Date: _Feb 5 2002_

Project: _Degree of Hydration - Mix 4_

Aggregate Properties: Grade ___57___
  ecific gravity: Fine _2.64_ Coarse _2.42_
  Moisture Calculations:
  FA= 0.00-0.50=0.50
  CA= 1.90 - 2.60 = 2.30

Weights By: _Lucy, Richard, & Matt_

Mixing By: _R. Delorenzo_

Air Content _1-6 %_

Specifications:
  Cement content ___744___ lb. Cement Type _I/II_

Slump range ___3'___ in. _± 1.5"_

Batch _6.0_ Cu.Ft. CF= _0.2222_

MATERIAL	SOURCE	WT. PER CU.YD.(LB)	VOL. PER CU.YD.(CF)	WT. PER BATCH(LB)	ADJ. WT. PER BATCH(LB)	REMARKS
Cement	Southdown	610	3.10	135.5	135.5	
FLYASH	Recal	134	0.91	29.8	29.8	
WATER	G.ville	305	4.89	67.8	58.8	-9.0
FINE AGG.	76-127	918	5.57	204.0	203.0	-1.0
COARSE AGG.	DF-005	1729	11.45	384.2	394.2	+10.0
AIR ENTRAINER	Darex AEA	4 oz	1.08	26.3 ml	26.3 ml	
ADMIXTURE	v.ADF 64	24.4 oz		160.3 ml	160.3 ml	
TOTAL			27.0			

JMP(IN) _7.5_ AIR(%) _2 ½_ MIX TEMP _70.0_ WORKABILITY _Good_ AIR TEMP _20°F_

Lucy, Matt Richard

SLUMP BY: _Fitzgerld_ AIR BY: _Delorenzo_ CYLS. BY: _Roberts, Fitzgerld_ START TIME= _10:04ª_

W/C RATIO _0.41_ H20 HELD OUT ___—___ ADDED ___—___ END TIME= _10:12ª_

CYLINDERS-COMPRESSIVE STRENGTH

TIME OF SET

(/ /)
3 DAYS-_____

INITIAL SET:_____ (/)

(/)
7 DAYS-_____

FINAL SET:_____

(/ /)
28 DAYS-_____

REMARKS:_____

DESIGNED BY:_____

WITNESSED BY:_____

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TRAIL BATCH-- DATA AND CALCULATIONS
(Saturated, Surface-dry Aggregates)

0% Slag

Date: 3-13-02

Project: Degree of Hydration-Mix 5

jgregate Properties: Grade #57
Specific gravity: Fine 2.64 Coarse 2.42
   Moisture Calculations:
     FA= 0.00-0.50=0.50
     CA= 4.59-2.60=1.99

Weights By: Richad & Lucy

Mixing By: Delorenzo / Roberts

Air Content _____ 1-6 %

Specifications:
   Cement content 660 lb. Cement Type I/II

Slump range 3" ± 1.5"

Batch 6.0 Cu.Ft. CF= 0.2222

MATERIAL	SOURCE	WT. PER CU.YD.(LB)	VOL. PER CU.YD.(CF)	WT. PER BATCH(LB)	ADJ. WT. PER BATCH(LB)	REMARKS
CEMENT	Cemex-(Southdown)	660	3.36	146.7	146.7	
~~FLYASH/SLAG~~	—					
WATER	G'ville	267	4.28	59.3	52.6	- 6.7
FINE AGG.	76-137	1076	6.53	239.1	237.9	- 1.2
COARSE AGG.	08-005	1794	11.88	398.6	406.5	+ 7.9
AIR ENTRAINER	Darex AEA	5.0 oz	0.95	32.9 mL	32.9 mL	.
ADMIXTURE	WRDA 64	33.0 oz	—	216.9 mL	216.9 mL	
ADMIXTURE	—					
ADMIXTURE	—					
TOTAL			27.00			

SLUMP(IN) 5.25   AIR(%) 6.0   MIX TEMP 74°F   WORKABILITY Good   AIR TEMP 72°F

SLUMP BY: Roberts   AIR BY: Roberts   CYLS. BY: Richd/Lucy   START TIME= 0900

W/C RATIO 0.40   H2O HELD OUT _____ ADDED _____   END TIME= 0918

CYLINDERS-COMPRESSIVE STRENGTH

REMARKS: _____

_____

ESIGNED BY: _____
WITNESSED BY: _____

TRAIL BATCH-- DATA AND CALCULATIONS
(Saturated, Surface-dry Aggregates)

50% Slag

Date: 3-13-02

Aggregate Properties: Grade #57
specific gravity: Fine 2.64   Coarse 2.42
   Moisture Calculations:
   FA= 0.00-0.50=0.50
   CA= 4.59 -2.60 = 1.99

Specifications:
   Cement content 660 lb. Cement Type I/II

Project: Degree of Hydration-Mix 6
Weights By: Richard & Lucy
Mixing By: DeLorenzo / Roberts
Air Content   1-6 %

Slump range   3" ± 1.5"

Batch 6.0 Cu.Ft. CF= 0.2222

MATERIAL	SOURCE	WT. PER CU.YD.(LB)	VOL. PER CU.YD.(CF)	WT. PER BATCH(LB)	ADJ. WT. PER BATCH(LB)	REMARKS
CEMENT	Cemex-(Southdown)	330	1.68	73.3	73.3	
FLYASH/SLAG	Lafarge Slag Cem	320	1.80	73.3	73.3	
WATER	G'ville	267	4.28	59.3	52.6	- 6.7
FINE AGG.	76-137	1066	6.47	236.9	235.7	-1.2
COARSE AGG.	08-005	1785	11.82	396.6	404.5	+7.9
AIR ENTRAINER	Daricx AEA	5.0 oz	0.95	32.9 mL	32.9 mL	
ADMIXTURE	WRLA 64	33.0 oz	—	216.9 mL	32.9 mL	
DMIXTURE						
ADMIXTURE						
TOTAL			27.00			

SLUMP(IN) 3.25 AIR(%) 3.9 MIX TEMP 74°F WORKABILITY sticky AIR TEMP 72°F

SLUMP BY: Roberts   AIR BY: Roberts   CYLS. BY: Richard/Lucy START TIME= 8955

W/C RATIO 0.40   H20 HELD OUT — ADDED —   END TIME= 10:03

CYLINDERS-COMPRESSIVE STRENGTH

REMARKS:

SIGNED BY:

WITNESSED BY:

S:\Physical Lab\Research\CONCRETE MIXES\BLANK MIX FORMS\MIX DES QPL.doc

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TRAIL BATCH-- DATA AND CALCULATIONS
(Saturated, Surface-dry Aggregates)

0% Slag

Date: 5-8-02

Project: Degree of Hydration-Mix 7

aggregate Properties: Grade #57
Specific gravity: Fine 2.64 Coarse 2.42
Moisture Calculations:
FA= 0.00-0.50=0.50
CA=   -   "

Weights By: DeLorenzo

Mixing By: DeLorenzo

Air Content   1-6 %

Specifications:
Cement content 660 lb. Cement Type I/II

Slump range   3" ± 1.5"

Batch   Cu.Ft.   CF=

MATERIAL	SOURCE	WT. PER CU.YD.(LB)	VOL. PER CU.YD.(CF)	WT. PER BATCH(LB)	ADJ. WT. PER BATCH(LB)	REMARKS
CEMENT	Cemex-(Southdown)	660	3.36	146.7	146.7	5.7 1 bag
~~FLYASH/SLAG~~	—					
WATER	G'ville	267	4.28	59.3	52.6	-6.7
FINE AGG.	76-137	1076	6.53	239.1	237.7	-1.2
COARSE AGG.	08-005	1794	11.88	398.6	406.5	+7.9
AIR ENTRAINER	Darex AEA	5.0 oz	0.95	32.9 mL	32.9 mL	.
ADMIXTURE	WRDA 64	33.0 oz	—	216.9 mL	216.9 mL	
ADMIXTURE	—					
ADMIXTURE	—					
TOTAL			27.00			

SLUMP(IN) 4.0   AIR(%) 5½   MIX TEMP 75   WORKABILITY Good   AIR TEMP 71°F

SLUMP BY:_____   AIR BY:_____   CYLS. BY:_____   START TIME=_____

W/C RATIO_____   H2O HELD OUT_____   ADDED_____   END TIME=_____

CYLINDERS-COMPRESSIVE STRENGTH

_____   REMARKS:_____
_____   _____
_____   _____
_____   _____
_____   _____
_____   _____
_____   _____
_____   _____

DESIGNED BY:_____   _____
WITNESSED BY:_____   _____

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Appendix B – Additional SEM Images

Part 1: Mix 1 – Portland cement only mix (0%FA)
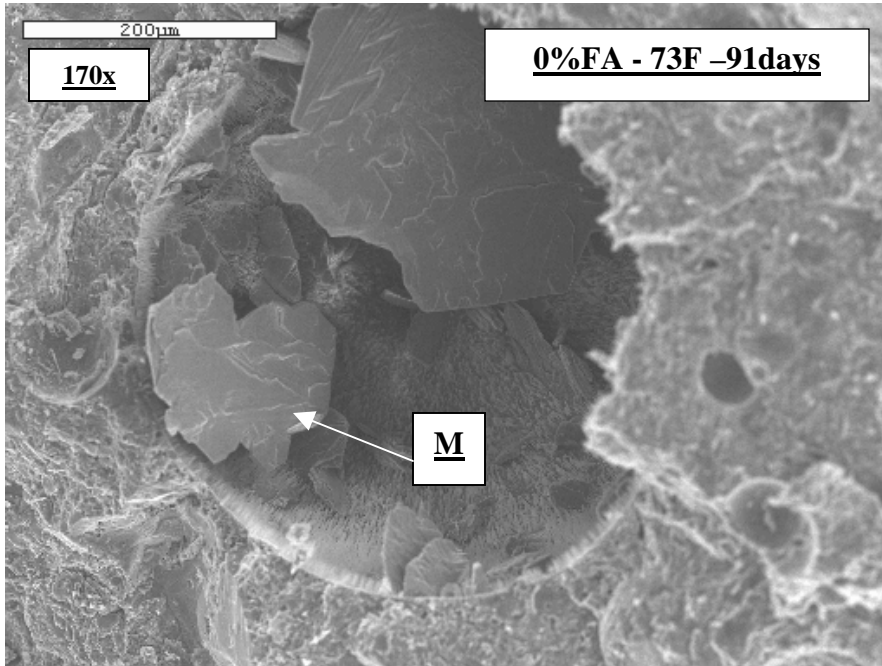
Figure B.1 Void with monosulphate (M), no ettringite found
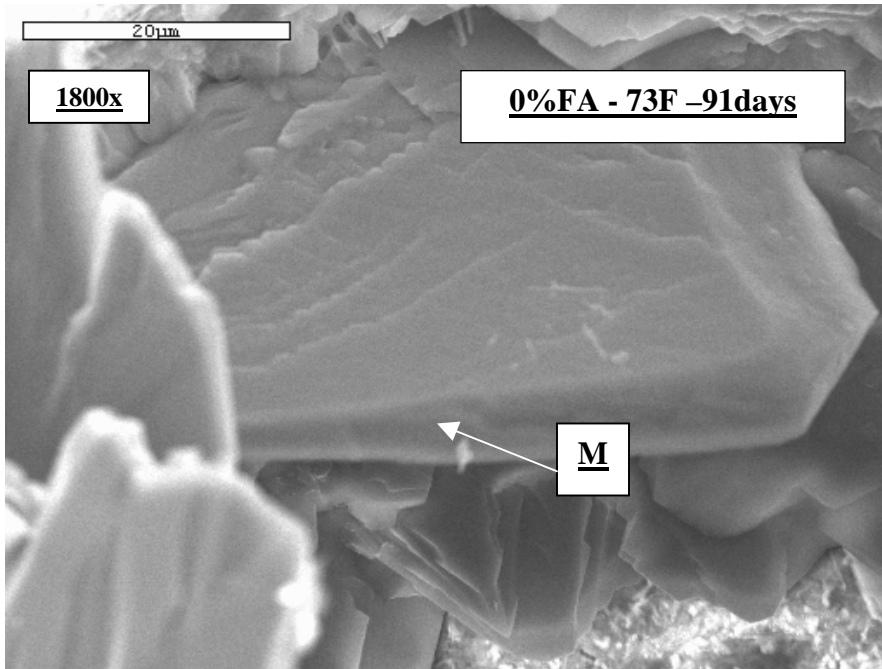
Figure B.2 Close-up view of Fig B.1
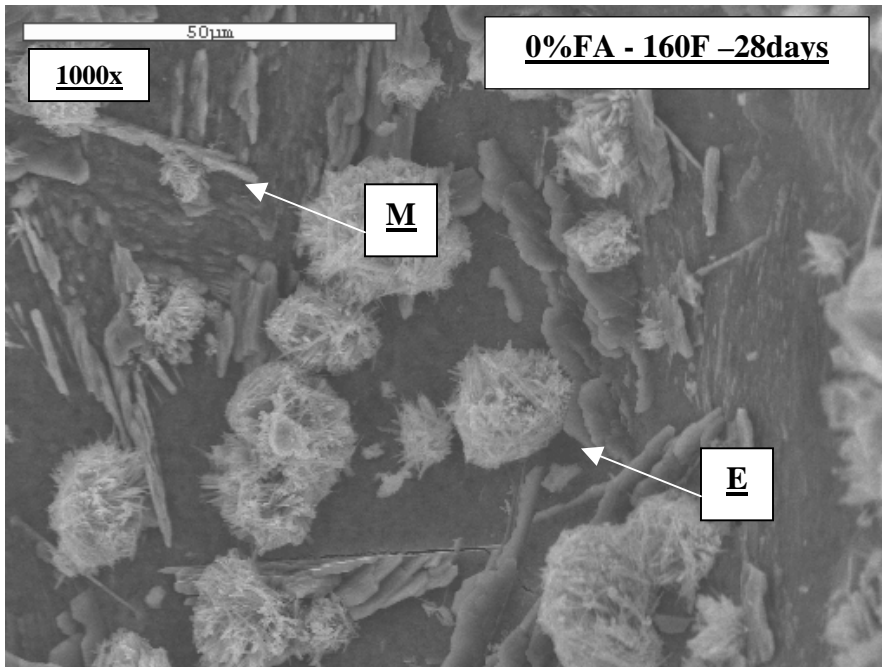
Figure B.3 Void with ettringite (E) and monosulphate (M)
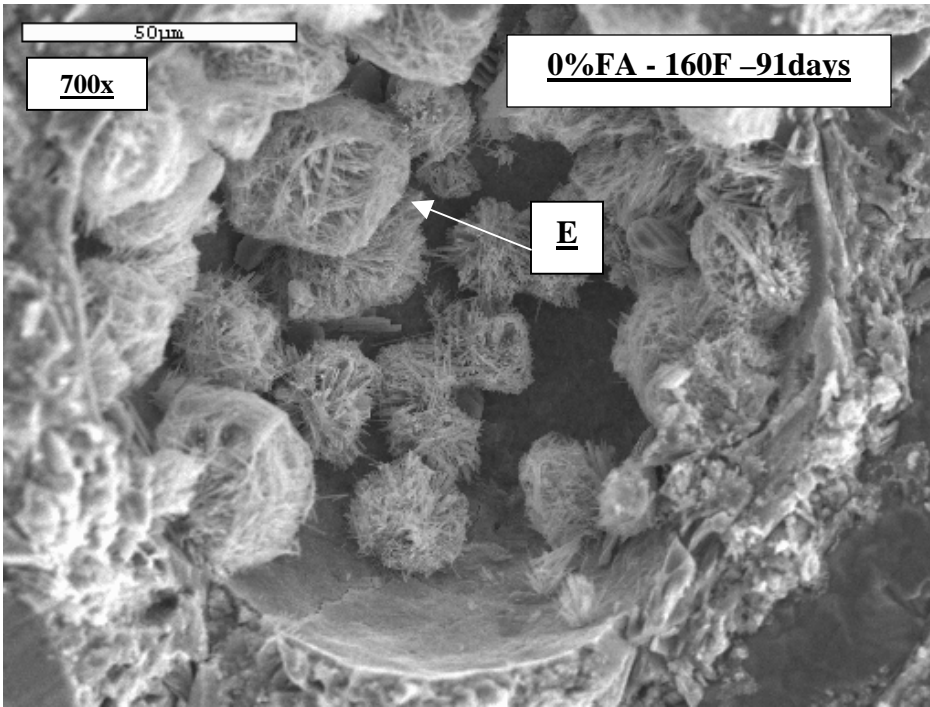
Figure B.4 Void with ettringite (E) crystals
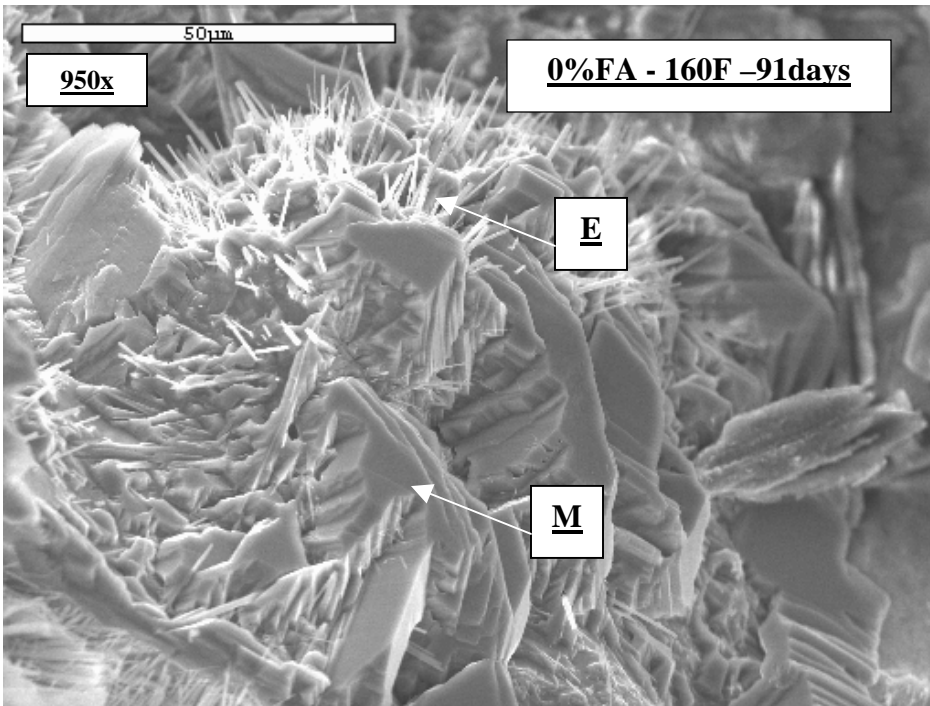
Figure B.5 Void showing monosulphate and the early formation of ettringite (E) crystals
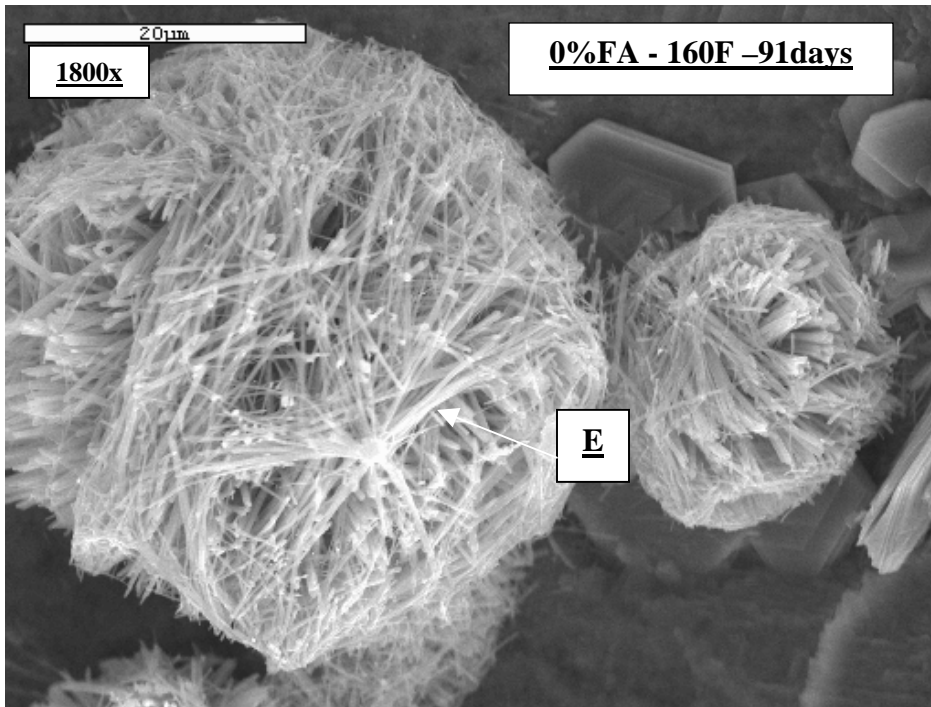
Figure B.6 Void showing ball of ettringite (E) crystals
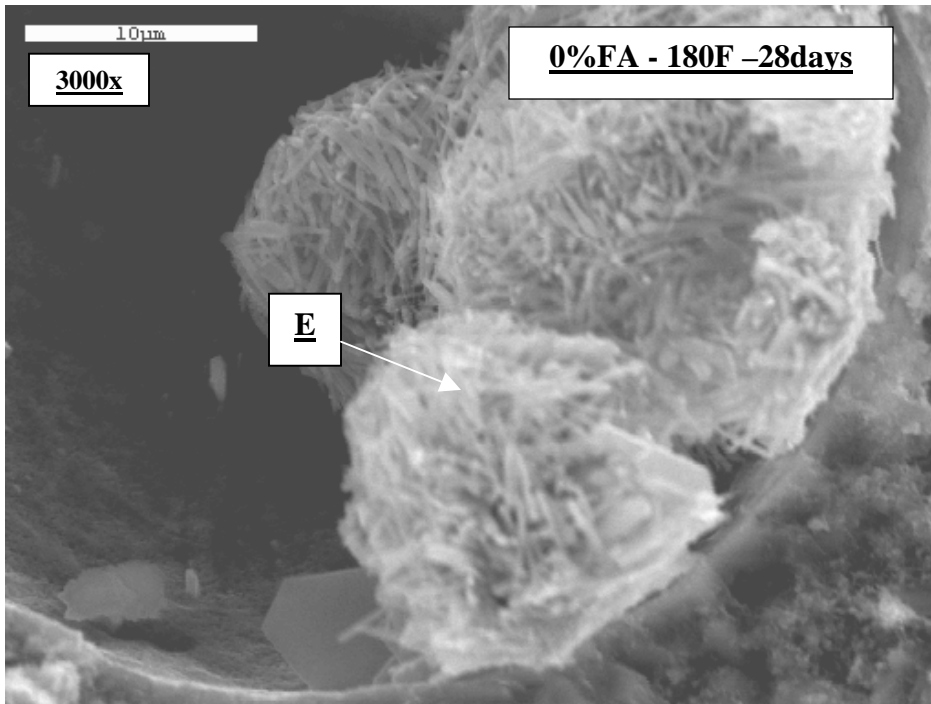
Figure B.7 Void showing balls of ettringite (E) crystals
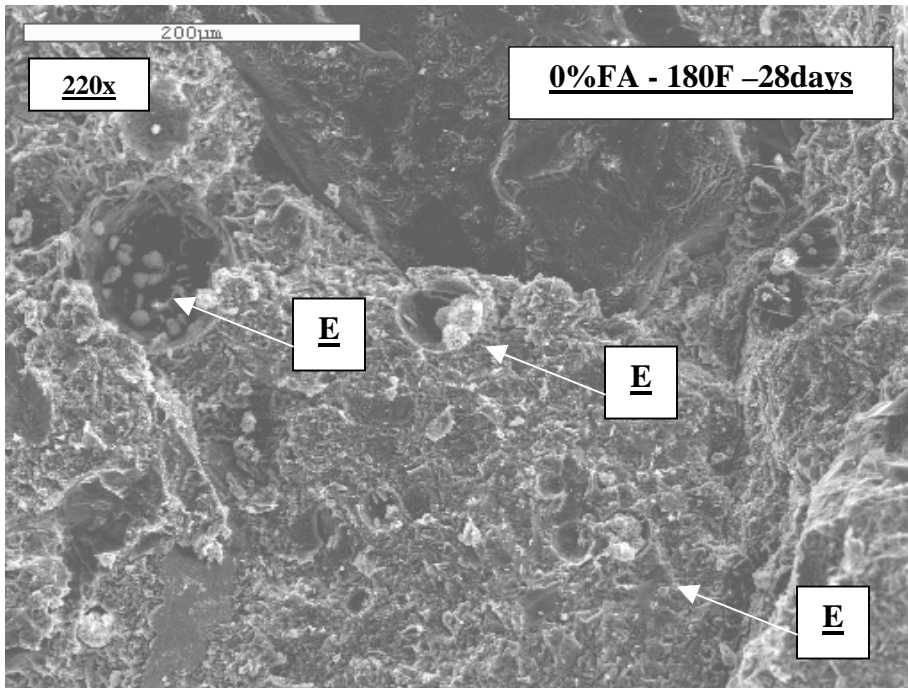
Figure B.8 Voids showing ettringite (E) crystals some almost full
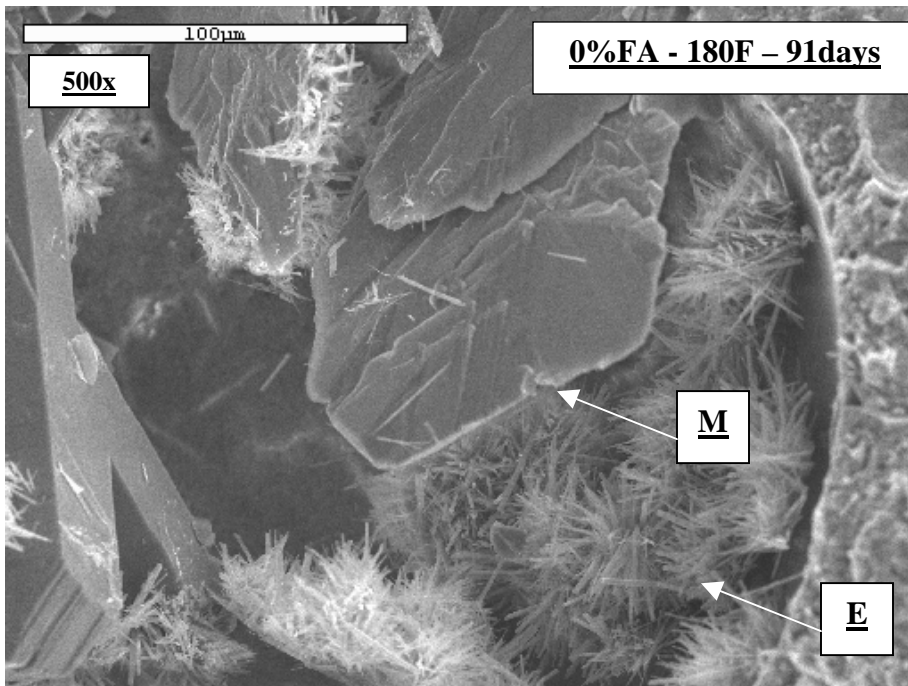
Figure B.9 Voids showing ettringite (E) and monosulphate crystals

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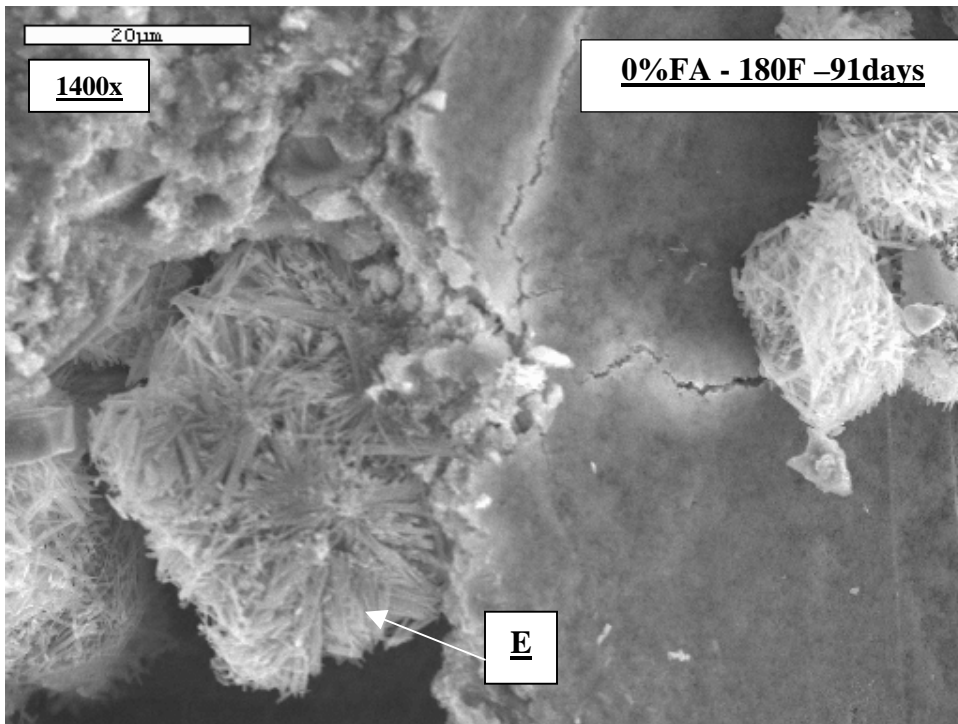
Figure B.10 Ettringite (E) crystals in and around vicinity of void
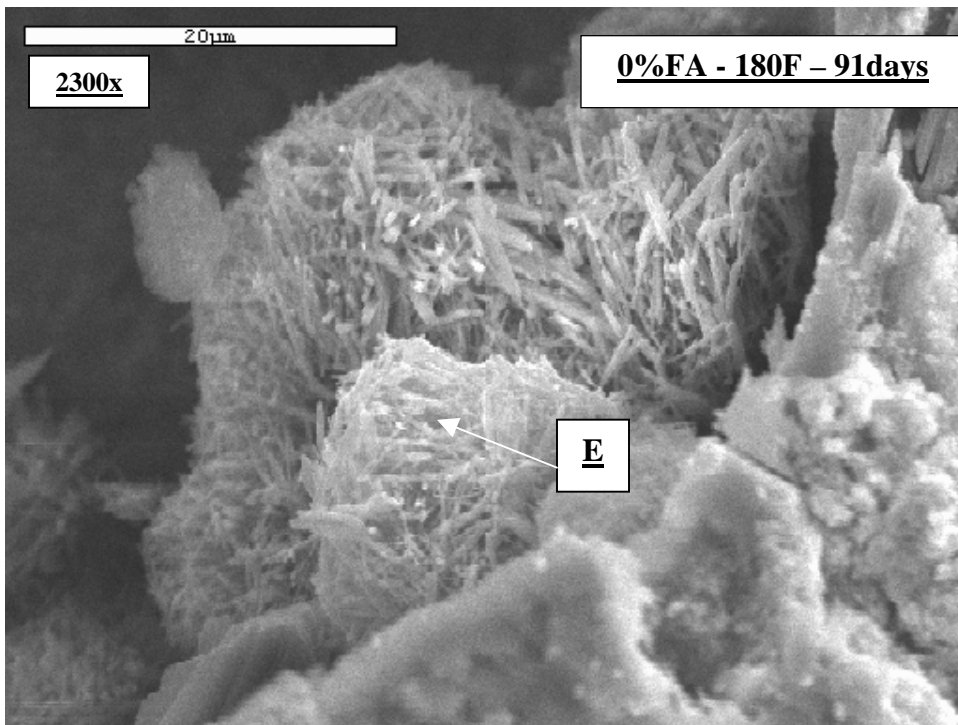
Figure B.11 Ettringite (E) crystals in void
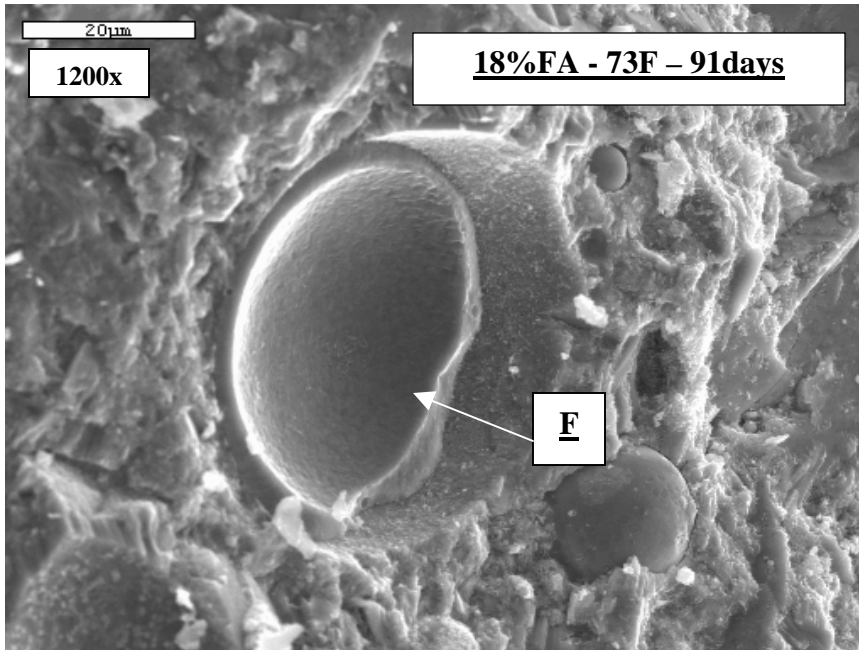
Figure B.12 Fly ash particle with reaction around rim
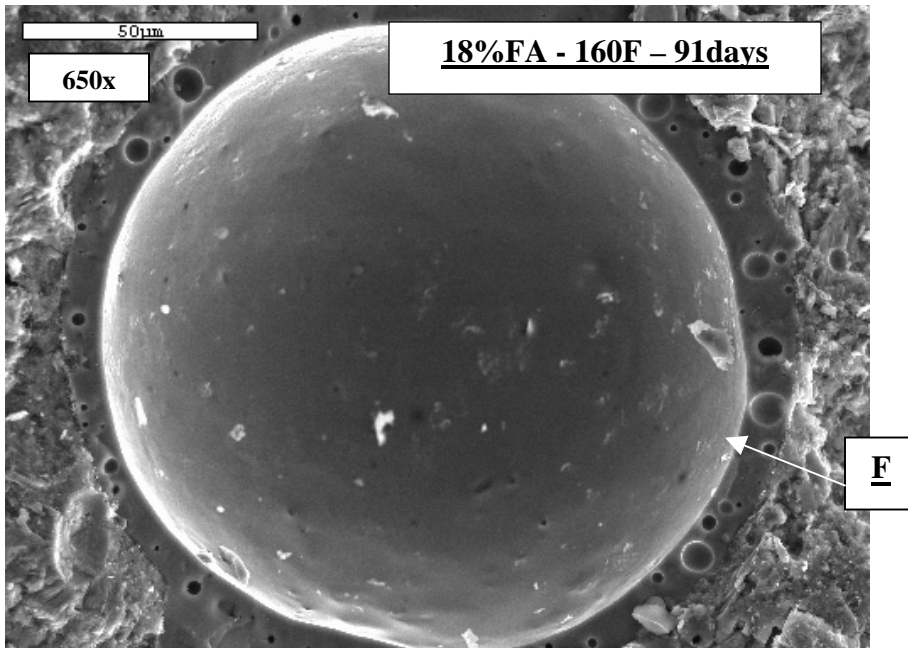
Figure B.13 Fly ash particle with reaction around rim
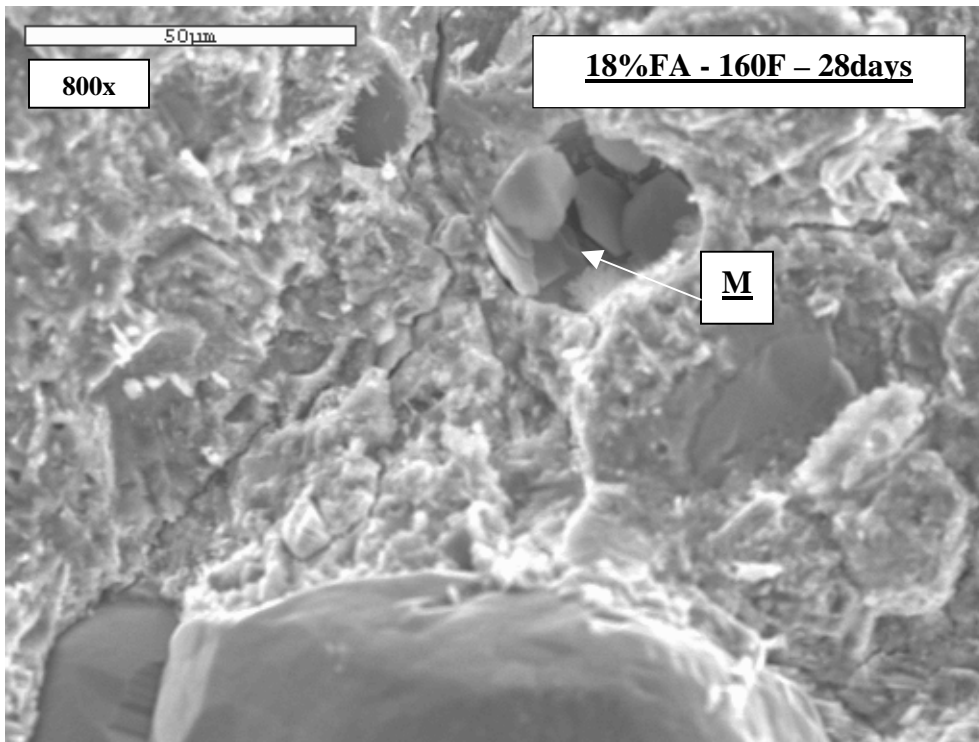
Figure B.14 Void containing monosulphate
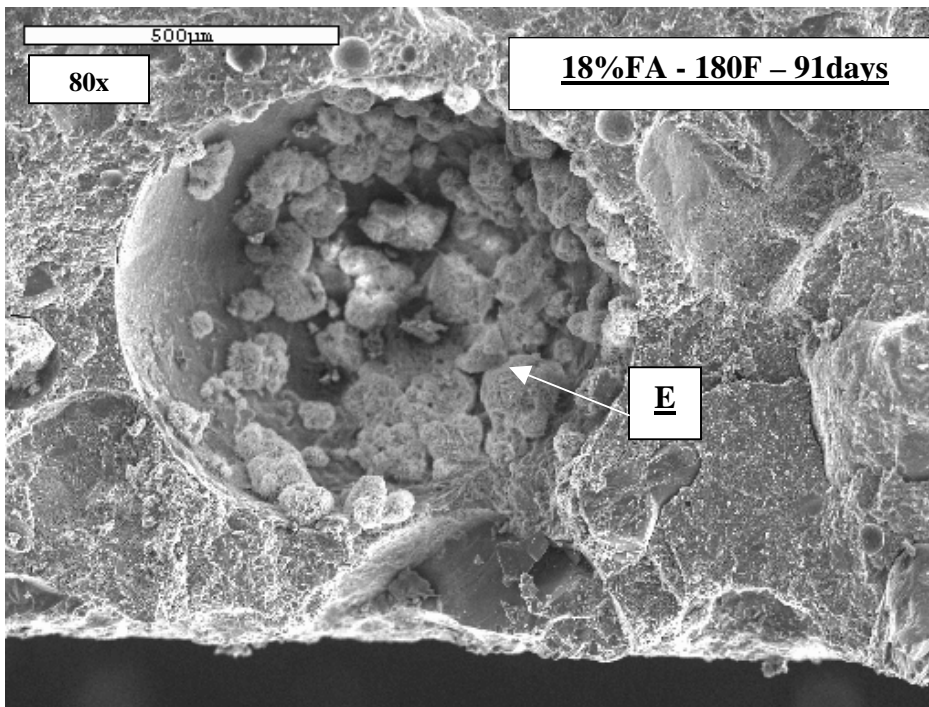
Figure B.15 Void containing ettringite crystals
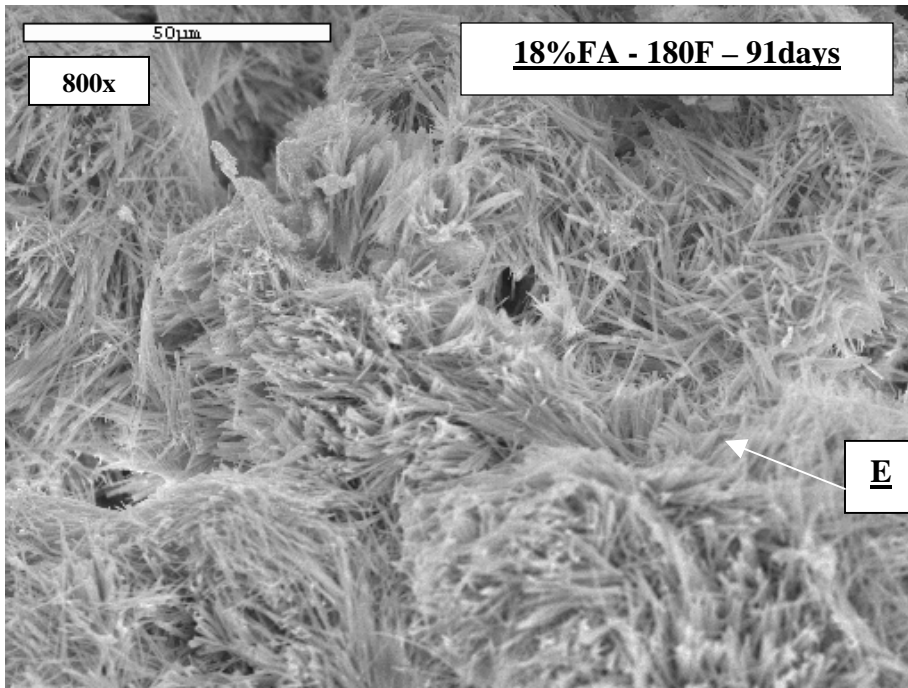
Figure B.16 Close up view of ettringite crystals in Fig B.15
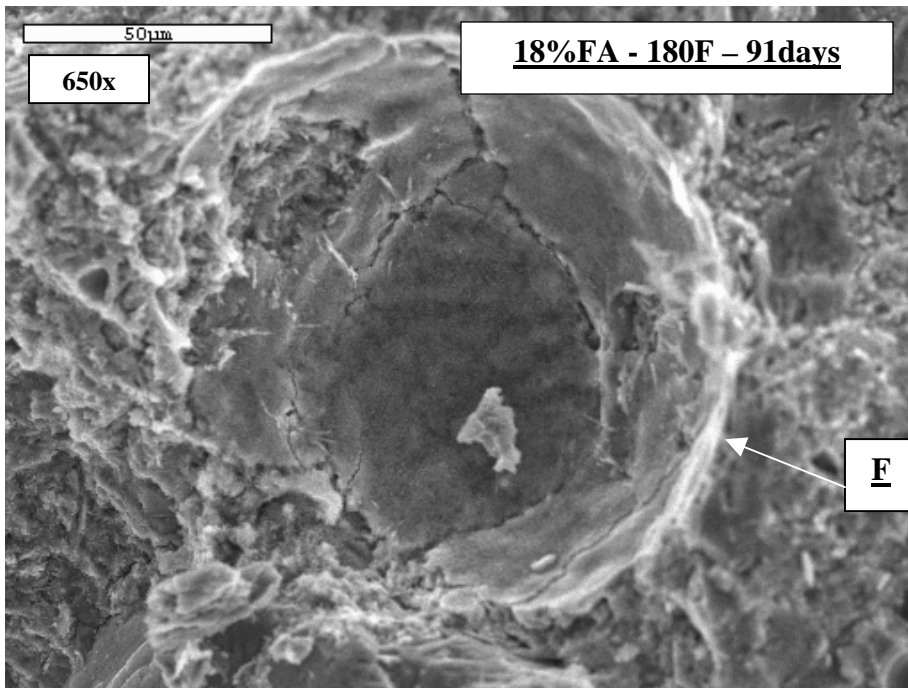
Figure B.17 Reacting fly ash particle
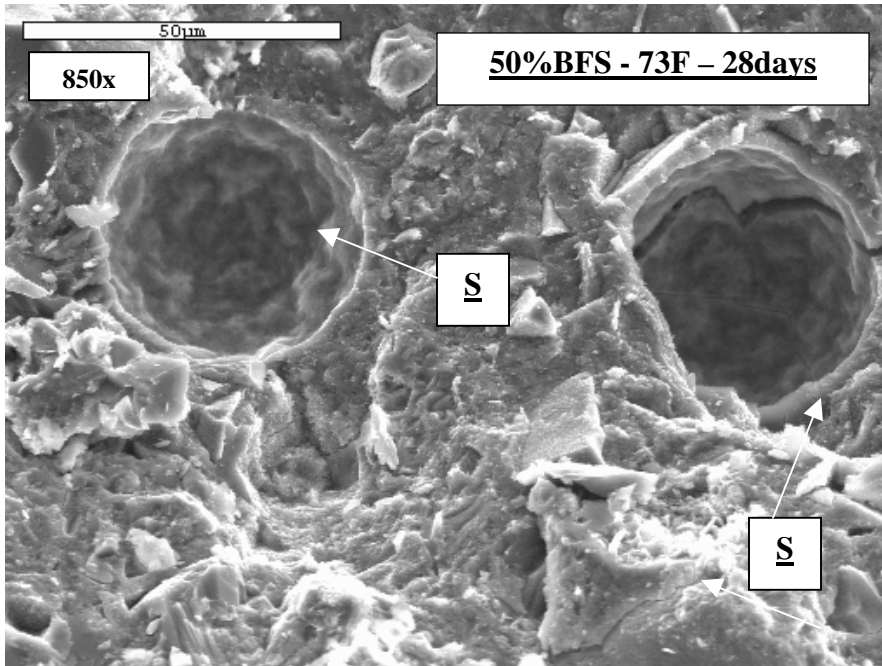
Figure B.18 Slag particles showing some early reaction
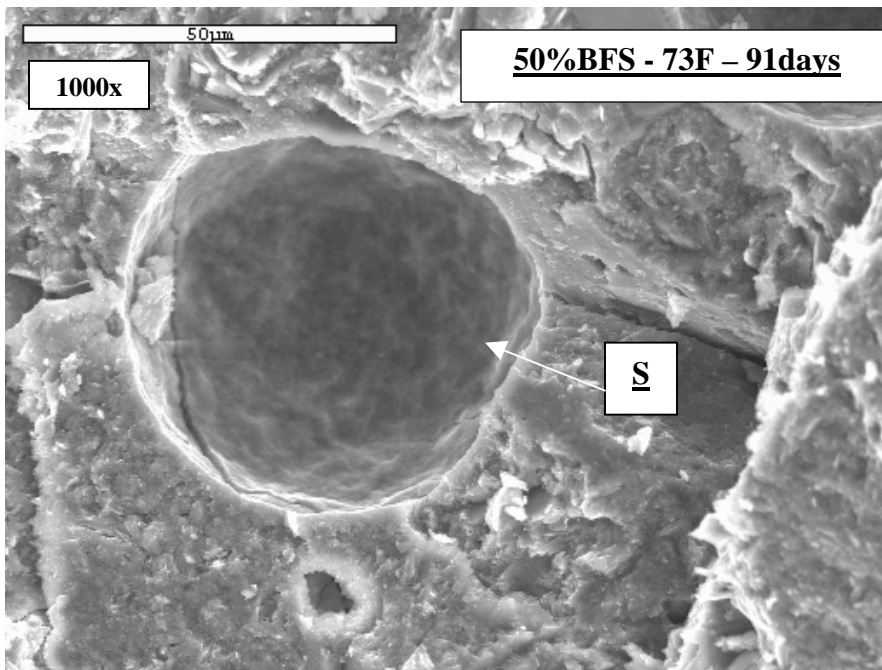
Figure B.19 Slag particles showing reaction on surface
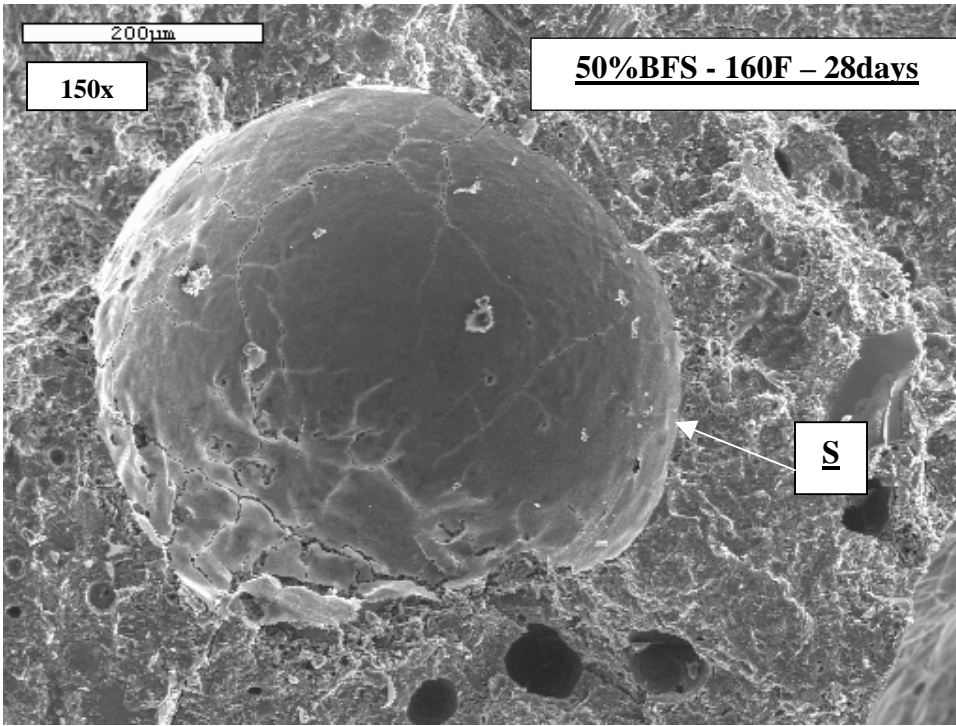
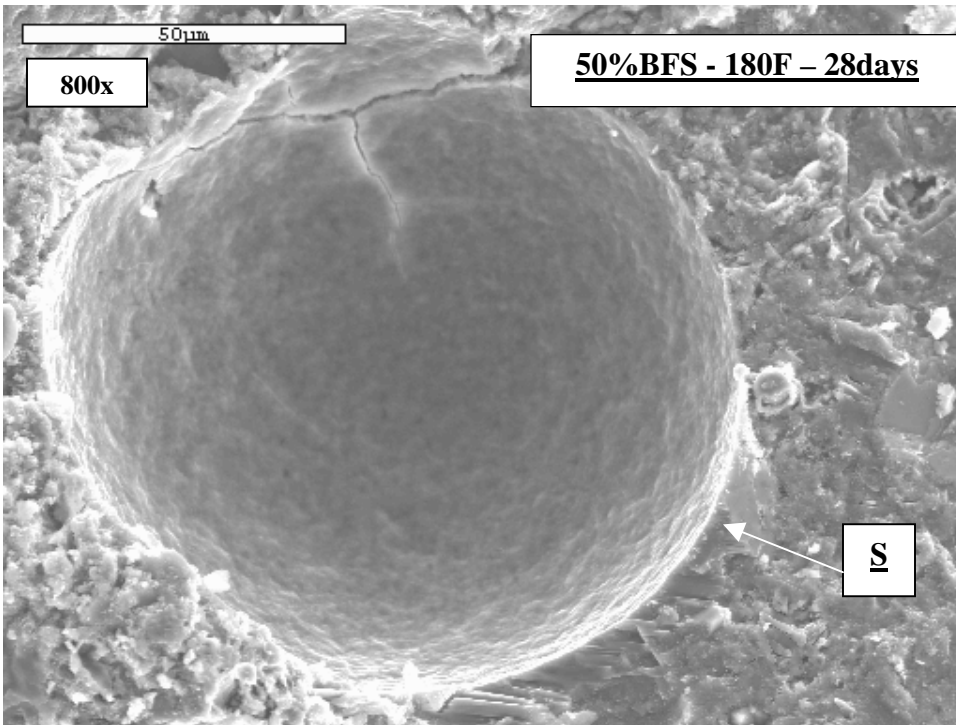Figure B.20 Slag particle showing reaction on surface

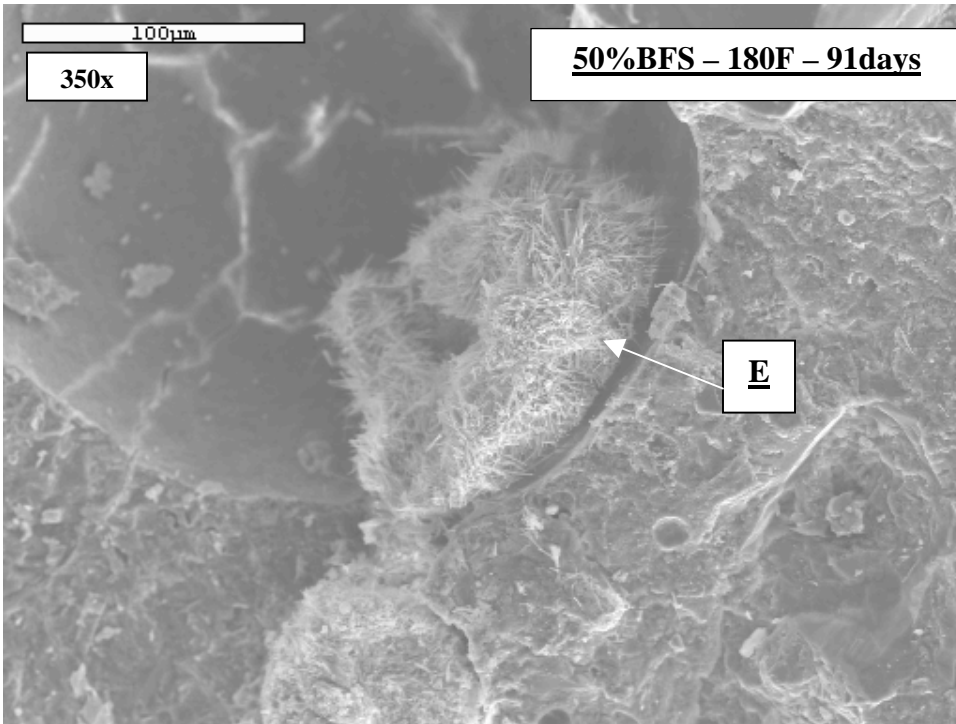Figure B.21 Slag particle showing reaction on surface
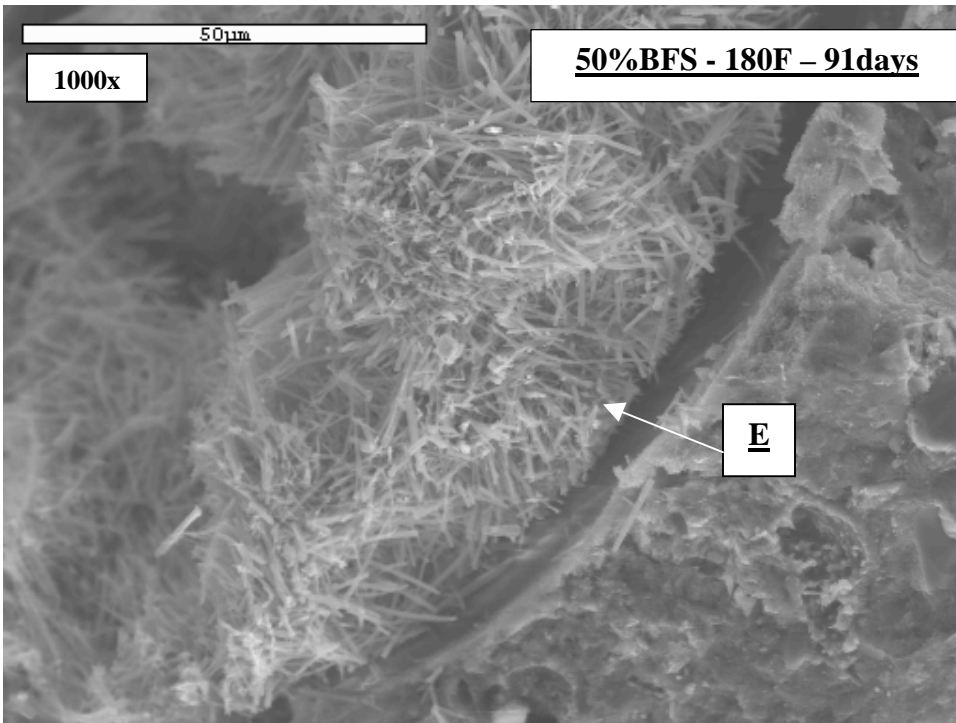
Figure B.22 Ettringite formed around surface of reacting slag particle

Figure B.23 Close-up view of Fig. B.22