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THE EFFECTS OF PREMATURE FREEZING ON PLAIN AND FLY ASH CONCRETE STRENGTH DEVELOPMENT

by

ERIKA WACKERLE

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ABSTRACT

The effects of premature freezing (freezing at low maturity) on strength development were studied in plain and fly ash concretes.

Both non air-entrained and air-entrained concretes were used. Mix design criteria included an 80 mm slump, a 35 MPa 28-day strength and, when applicable, fly ash replacement of cement at 25% by weight. At 8, 17 and 24 hours from mixing, specimens were subjected to a 2-day period at -20°C. Flexural and compressive strengths were measured at 3, 7, 28 and 90 days, and were compared with the strengths of normally-cured specimens. Non air-entrained fly ash concrete frozen at 8 hours was the only concrete to suffer permanent strength loss. The rate of strength development was hampered, particularly in the fly ash concretes. Soaking specimens prior to freezing did not affect the results.

Supplementary studies included measurement of fresh concrete properties and setting times, internal temperature monitoring, air void analysis, statistical analysis of strength results and fracture mode analysis.

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NOTATION

CON-A	=	air-entrained plain (control) mix
CON-N	=	non air-entrained plain (control) mix
D	=	dry (no soaking in curing regime)
FA-A	=	air-entrained fly ash mix
FA-N	=	non air-entrained fly ash mix
F#	=	age at freezing
F8*	=	frozen at -40°C, rather than standard freezing regime
S	=	soaked in curing regime
SSD	=	saturated-surface dry
w/c	=	water-cement ratio
w/c´	=	water-(cement + fly ash) ratio

CHAPTER 1: INTRODUCTION

Concreting during cold weather poses a number of unique problems. The three main areas of concern covered in concreting standards are:

1. premature freezing damage,
2. low curing temperatures resulting in slow strength gain and inadequate strength at the time of loading, and
3. thermal shock or gradients which can induce significant stresses.

This thesis concentrates on the first concern — premature freezing. In particular, it deals with the effects premature freezing has on the strength development of fly ash concrete.

"Premature freezing" is the term indicating freezing of the concrete in the first few days after casting. Unexpected cold temperatures, inadequate protection or faulty heating equipment can all contribute to a potential freezing problem. There are documented cases of structures with damage ranging from structural collapse *[Feld, 1964]* to poor quality concrete at the surface *[CPCA, 1984; Matti, 1986]* because of premature freezing.

Fly ash (the finely divided residue that results from the combustion of pulverized coal) is gaining popularity in Canada as a supplementary cementing material. The economic advantages and basic properties of fly ash concretes are well documented. However, the behaviour of fly ash concretes under cold-weather concreting conditions is not so well researched. In order to develop standards that are conservative enough to ensure adequate concrete quality and yet realistic enough to prevent unnecessary economic hardship on contractors, a better understanding of premature freezing, particularly with respect to fly ash concretes, is required.

The main objective of the research presented in this thesis is to compare the strength development of plain and fly ash concretes which are exposed to premature freezing. The factors studied in this comparison include air-entrainment, degree of saturation, age at freezing, type of strength testing and age at testing. Monitoring of concrete internal temperatures and analysis of the mode of fracture are used to further understand the processes which occur during premature freezing.

Stage I of the research involved development of the mix designs and experimental procedures and was carried out in the summer of 1990. The actual testing program, Stage II, took place between September 1990 and June 1991. Following the literature review in Chapter 2, Chapter 3 outlines the objectives and rationale behind the research program. The details of the experimental procedures are discussed in Chapter 4. Chapter 5 summarizes and discusses the various testing results and Chapter 6 contains the final conclusions and recommendations.

CHAPTER 2: LITERATURE REVIEW

2.1 Introduction

Premature freezing of concrete is a major concern in countries where cold weather construction is common practice. Most of the papers reviewed for this chapter originated in the United States, Canada, the Soviet Union and Northern Europe — that is, in countries where economics dictate that cold weather concreting cannot be avoided. The earliest report published by the American Concrete Institute (ACI) was "Winter Concreting Methods", in a 1930 ACI Journal *[Johnson, 1930]*. In 1948, ACI published its first standard titled "Recommended Practice for Winter Concreting Methods" (ACI 604-48) *[ACI Committee 604, 1948]*. Research efforts have concentrated on the mechanical effects of premature freezing such as strength loss and, to a lesser degree, durability loss. As time progressed, there was also an increasing amount of interest in the damage mechanism and microstructural aspects of premature freezing. Much of this research has been carried out to explain the freeze-thaw damage mechanism which affects durability in freeze-thaw environments. A wide range of experimental procedures and concretes were (and continue to be) used in these research projects and although this has created an extensive base of knowledge, it also makes it difficult to compare results and arrive at some general conclusions about premature freezing.

This chapter reviews some of the literature that has resulted from previous research. It begins with a look at the effects of premature freezing and continues with the freezing damage mechanisms that have been proposed to explain these effects. This is followed with a discussion of the criteria that have been suggested to measure freezing resistance and examples of how these criteria are applied in some current concreting standards and recommendations. This leads to various topics including preventing premature freezing, improving freezing resistance and detecting freezing damage. The chapter concludes with the role of fly ash in cold weather concreting.

2.2 The Effects of Premature Freezing

The first question tackled by researchers was "What are the effects of exposing concrete to premature freezing?". To answer this question many research projects have been carried out. The concrete mix designs have included a wide range of cement types and proportions, water-cement ratios, aggregate sizes and types, and other variables. Thus, the research has yielded varying results. In addition to this, concrete materials, particularly cement composition and fineness, have also changed greatly over the years. This means that tests carried out with a mix design from 50 years ago may yield different results today.

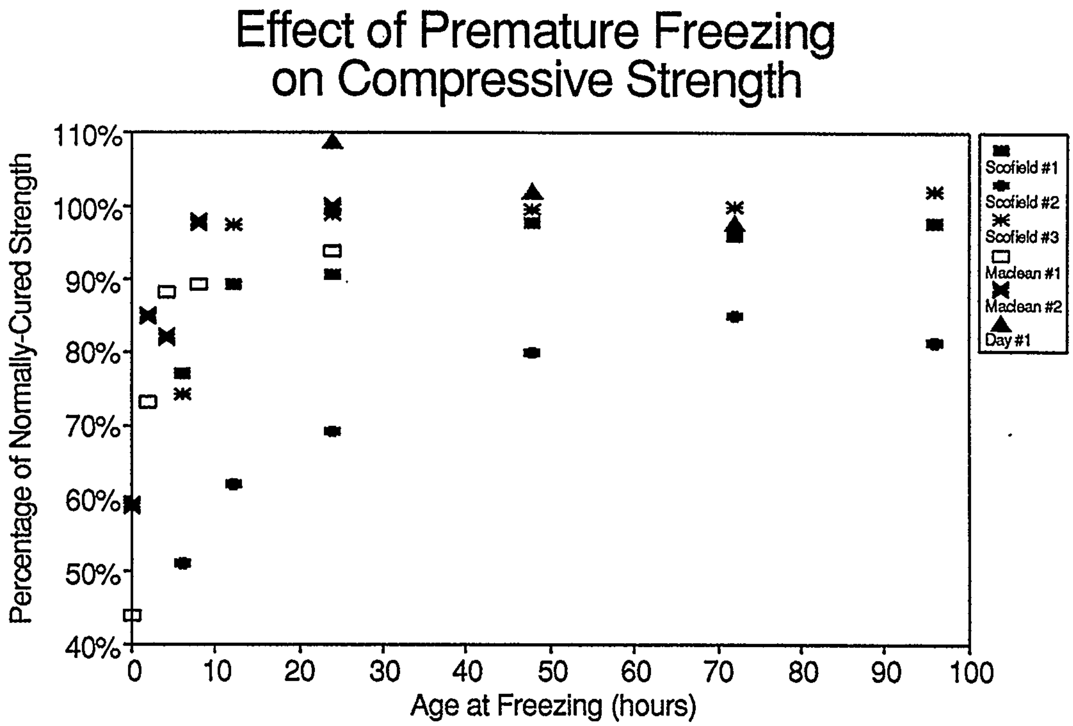
Along with a diversity of concrete mixes, a wide variety of experimental procedures has been used. The freezing concrete's temperature profile with respect to time and location in a specimen has the potential to affect the freezing effects and is dependant on a number of factors. These factors include the size and shape of the specimen and the nature of the freezing regime (i.e. pre-freezing and freezing temperatures; instant exposure to the freezing temperature or gradual temperature decline; and presence or absence of moisture in the curing environment). Although there has been no consistent manner to simulate the premature freezing of concrete in the laboratory, it is still important to look at previous research and get a general sense of the effects of premature freezing.

One possible effect of freezing is a loss of compressive strength in comparison to concrete which is not frozen. The magnitude of this loss depends on the concrete mix design, the pre- and post-freezing curing, and the freezing regime. The results from some typical studies are summarized in Figure 2.1 *[Scofield, 1937; Maclean et al, 1981; Day, 1991]*. The strength loss values used in this figure were based on measurements made at 28 days. Two common trends are apparent from this figure:

1. concrete with high water-cement ratios suffers greater damage, and
2. the susceptibility to damage decreases with an increase in the length of prehardening before exposure to freezing conditions.

Moist curing normally increases the damage as the Scofield results show. In the Maclean results, a higher curing temperature which accelerates strength gain offsets the negative effect of moist curing. It should be noted that the Scofield results are 55 years old. Cements of that time period were much coarser and had a lower tricalcium silicate content than today's cements. Therefore, they would have a slower rate of strength gain and be more susceptible to premature freezing damage.

Typically, the magnitude of the compressive strength loss is much greater in the short-term than in the long-term. Concrete subjected to freezing temperatures will develop strength more slowly than comparable unfrozen concrete because cement hydration occurs at a slower rate at low temperatures. This has always been a major concern in reporting test results: how much of the apparent freezing damage is actually due to a slower rate of strength gain? In the short term there may appear to be some freezing damage, but in the long term the frozen concrete is as strong as, or stronger, than the unfrozen concrete. If a specimen is frozen, but doesn't suffer any



Notes:

1. Results are 28-day strengths expressed as a percentage of 28-day normally-cured strength.
2. Ordinary portland cement and no additives were used in the mix designs.
3. Other Experimental Details:
   Scofield #1 - 9 in. (230 mm) slump, cured in dry air, frozen 2 days at 5 F (-15 C)
   Scofield #2 - as #1 but cured in moist air
   Scofield #3 - as #1 but 2 in. (50 mm slump)
   Maclean #1 - low w/c mix, cured at +20 C, frozen 5 days at -20 C
   Maclean #2 - as #1 but steam cured at +50 C
   Day #1 - 80 mm slump, cured at +20 C, frozen 2 days at -20 C
4. Data Sources: Scofield, 1937; Maclean, 1981; Day, 1991.

FIGURE 2.1: Typical Results from Studies of the Effect of Premature Freezing on Compressive Strength

permanent damage, the subsequent curing procedures are very important in determining the concrete's ultimate strength.

Mechanical properties other than compressive strength have also been studied. Tensile strength losses have been measured in terms of splitting tensile *[Fagerlund, 1985]* and modulus of rupture tests *[Möller, 1956; Blondel, 1956; Houde, 1990]*. The dynamic modulus of elasticity also shows a reduction when the concrete is subjected to freezing *[Möller, 1956; Fagerlund, 1985]*. Kayyali et al *[1979]* reported an irreversible deterioration in the fracture toughness of plain cement-pastes, but an increase with air-entrained pastes.

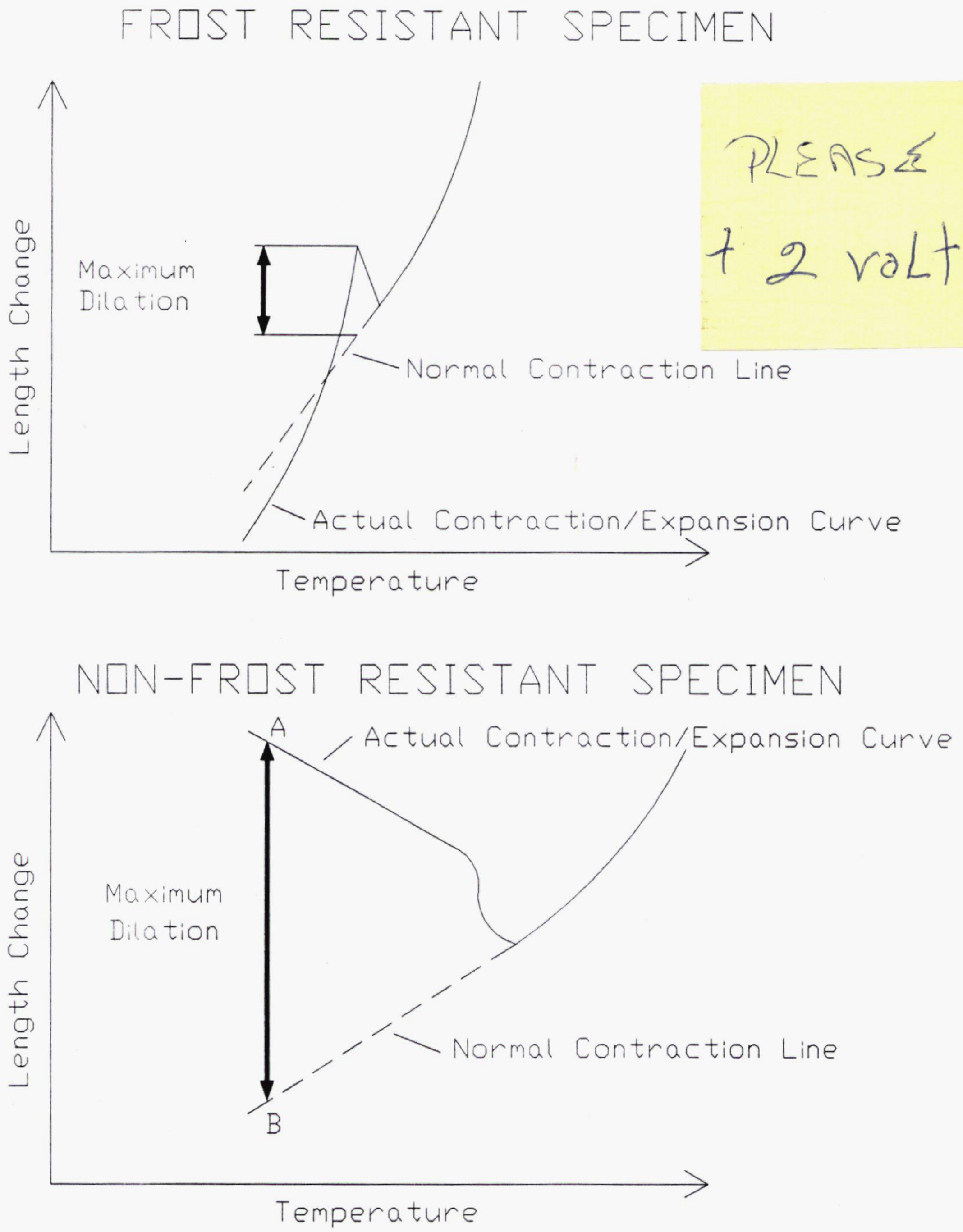
With frozen, plain, cement-paste specimens, the mode of compressive failure is more explosive than with normally-cured specimens even when the strength is the same *[Kayyali et al, 1979]*. This was not evident with air-entrained pastes. It has been noted that with frozen concrete specimens there is frequently more paste-aggregate bond failure *[Maclean et al, 1981; Krylov, 1985; Matti, 1986]*. In some instances, there is essentially no bonding between paste and aggregate — the coarse aggregate particles can be easily removed from smooth aggregate sockets.

Durability should be a major concern with all concreting. Concrete damaged by freezing will subsequently have a low freeze-thaw resistance and a high permeability making it susceptible to attack by aggressive agents. It has been stated that frozen concrete which has not suffered strength damage will still be less weather resistant and less watertight than unfrozen concrete *[CPCA, 1984]*. Roshore's extensive experiments *[1967]* revealed that durability (measured by a freeze-thaw durability factor) was reduced by freezing even when the compressive strength would not have been.

The increase in porosity (and hence permeability) in frozen concrete is an important cause of reduced durability *[Pink, 1967]*. Houde *[1990]* examined the cracking pattern and crack density of frozen specimens. Cracks due to freezing will result in an increased porosity in the concrete which, according to Bergstrom *[1976]*, will increase the permeability tenfold. (Houde also related the crack density to loss in compressive strength.) Note that in Houde's tests the concrete was frozen while it was still in the plastic state.

Since water expands as it freezes, there is an effect on the concrete's normal pattern of temperature-induced volume expansion and contraction. This fact was used in the dilation experiments carried out by Buck and others *[Buck, 1976; Hoff & Buck, 1983; Loov et al, 1984; Möller, 1956; Nykänen, 1956]*. It also provides the basis for ASTM C671 — Standard Test Method for Critical Dilation of Concrete Specimens Subjected to Freezing *[ASTM, 1989a]*, which tests for the frost resistance of concrete by determining the period of time required for water-immersed specimens to produce critical dilation when subjected to freezing. Dilation is measured on a length change vs temperature graph as the maximum difference between the steadily decreasing normal contraction line and the expansion (dilation) of the specimen that occurs when water in the specimen freezes. Dilation can be expressed in terms of average strain by dividing by the original specimen length. Various values ranging from 50×10^{-6} to 200×10^{-6} have been reported for the critical dilation which indicates freezing damage. Buck *[1988]* also noted that a frost resistant specimen will show some limited dilation, but will not show increasing dilation as the temperature decreases, whereas a specimen which is not frost resistant will show increasing dilation as temperature continues to decrease. Figure 2.2 illustrates these concepts.

PLEASE + 2 volt



Dilation obtained by measuring change in specimen length and dividing by original specimen length.

FIGURE 2.2: Dilation Concept for Frost Resistance

In a construction-site case study *[Matti, 1986]* where premature freezing actually did occur there were some visual signs of freezing damage. The concrete had a dark, wet look. Fossilized ice crystals or "crow's feet" were visible at the bond surface with the aggregate. Krylov *[1985]* refers to this as "fancy frosting", where a trace of the melted ice film is visible on the paste walls after the aggregate particles have been removed. Fagerlund *[1985]* also mentions "the occurrence of imprints of ice needles or ice crystals in the hardened concrete, especially at the interfaces between coarse aggregate particles and the cement paste."

In the case study mentioned above, only the surface layer of the concrete and particularly the corners were seriously damaged. This is typical of on-site freezing damage, as the outer layer of the concrete sections is most vulnerable to a freezing environment. The surface layer damage extends beyond loss of strength. Dusting problems *[PCA, 1987]*, loss of abrasion resistance *[CPCA, 1984]*, and spalling problems *[Pink, 1967]* are also related to premature freezing.

Prematurely frozen concrete may also bond poorly with the reinforcement in the concrete *[Krylov, 1985]*. Houde's test results *[1990]* showed a 40% loss in bond strength in reinforced beam specimens which were exposed to various freezing temperatures directly after casting. Suprenant and Basham *[1985]* used a finite element heat-transfer computer program to show that any metallic embedment with a cross-sectional area greater than 1 in^2 (650 mm^2) should be heated to prevent any localized freezing damage that would lead to a poor bond with the reinforcement.

Thus the effects of premature freezing are quite widespread and serious. They affect many aspects of the concrete's subsequent in-service performance in terms of both mechanical behaviour and durability.

2.3 Freezing Damage Mechanisms

Various explanations for these freezing effects have been suggested, but there is not yet a complete understanding of the damage mechanisms. There are two broad categories of mechanisms. The first, ice lensing or frost heave, occurs in plastic concrete or concrete of very low maturity, whilst the second category is prevalent in more mature concretes. Much of the research concerning this second group of damage mechanisms has been involved with trying to explain the freeze-thaw behaviour of concrete.

Collins *[1944]* first applied the ice lens or frost heave concept from soil mechanics to concrete. As the temperature in the concrete falls below the freezing point, water freezes in large pores and at the paste-aggregate interface. Due to a differential in the free energy between ice and water, these ice crystals attract large amounts of water and grow, forming ice lenses or needles. (Temperature gradients in the concrete are an important driving force for this water migration.) If the concrete has not attained sufficient strength to resist the expansive forces associated with ice formation, the internal structure of the concrete can be disrupted.

This disruption of the concrete matrix will correspond to a decrease in strength and an increase in porosity. The imprints of these ice lenses or needles are the "fancy frosting" or "crow's feet" described on page 10. In hardened and polished specimens, these imprints will appear as cracks. Houde *[1990]* investigated the crack pattern and density of frozen specimens. He related the crack density to increased porosity and decreased compressive strength. As ice lenses often occur at the paste-aggregate interface, they decrease the bond strength and thus the strength of the concrete.

It is generally accepted that ice lensing is the main damage mechanism in very immature concrete and is the mechanism responsible for severe freezing damage. A conclusive measure of the level of maturity at which ice lensing ceases to be a problem has not yet been determined. According to Fagerlund *[1985]*, ice lensing will stop being of significance when one of the following two conditions is met:

1. The loss of heat from the ice front is larger than the supply of heat to the ice front, stopping ice lens growth. This condition is governed primarily by the concrete permeability. Powers *[1956]* calculated that if the permeability coefficient is less than $5x10^{-8}$ cm/s frost heaving can no longer take place. He estimated that most concretes reach this permeability level 2 to 3 days after casting, but obviously this is very dependent on the concrete mix design and curing regime.

2. The pressure opposing ice lens growth is sufficient to prevent further growth, or to cause penetration of the constriction leading into the adjoining pore in the concrete (a phenomenon that does not cause any volume increase). This opposing pressure is supplied by the cohesion of the concrete. Fagerlund argues that theoretically this condition is the critical one and the cohesion corresponding to a tensile strength of 0.1 MPa is sufficient. This level of tensile strength occurs roughly at the time of final set.

Even if ice lensing cannot occur because the concrete is mature enough, there will still be water migration and freezing taking place in the concrete. There is controversy regarding the mechanisms which cause this to occur and what actually causes the damage to the concrete. Powers *[1956]* introduced the hydraulic pressure theory in which a destructive stress is created by the concrete resisting water flow away from the region of freezing. Recall as water freezes, it expands — 9% in an unconfined state — thus as a pore fills with ice the unfrozen water will try to escape. Powers also introduced the osmotic pressure theory to explain why water sometimes moves towards a freezing site. As the water (which is actually a weak solution) freezes, the nearby unfrozen water becomes more concentrated. Osmotic pressure is thus created and water will move to the freezing site to maintain equilibrium. One source *[Maclean et al, 1981]* claims that this process is at work in low water-cement ratio concretes, whereas the hydraulic pressure model is the main mechanism in high water-cement ratio concretes.

Ramachandran et al *[1981]* summarized the thermodynamics of frost damage in porous solids. They used thermodynamic models to explain the water migration from the finer pores to the coarser pores where ice crystals are in the process of forming. One important statement from their summary is that "in a porous system, the thermodynamic equilibrium between water in the small pores and ice can be achieved by either reducing the chemical potential of the water by placing it under tension (formation of menisci) or increasing the chemical potential of the bulk ice by increasing its pressure."

Litvan *[1973]* thought some damage is caused by ice formation, but that the water migration is the main source of damage. MacInnis and Beaudoin *[1973]* concluded from their work that, at low levels of maturity, the hydraulic pressure created in the liquid by the formation of ice is the major mechanism responsible for freezing damage.

Fagerlund *[1985]* considered the hydraulic pressure theory significant and noted that, in immature concretes with low strengths, the air void spacing created by air entrainment would be insufficient to prevent damage. He claimed that the air spaces required are "created by self-desiccation caused by the "contraction" of non-evaporable water. Air spaces thus created are evenly distributed with very low spacing over the entire concrete volume provided the concrete is not exposed to water at an early age." He and others have used this concept to obtain the degree of hydration required for freezing resistance.

Whatever the exact mechanism, it is readily apparent that the amount of freezable water in the concrete and its ability to move within the concrete are very important. The water-cement ratio, degree of hydration, and presence of external sources of water will all affect the amount of water or degree of saturation. The nature of the capillary pore system will affect the permeability of the concrete and hence the amount of water migration and resultant stresses. Air-entrainment provides closely-spaced air voids to which water migrates and where ice crystals grow without creating stresses.

The previous mechanisms have dealt with the mechanical damage due to water freezing and migration. Freezing temperatures will also slow down and perhaps stop the hydration process. Most sources claim cement hydration essentially stops at -10°C to -12°C and thus it is assumed concrete does not gain strength at lower temperatures. Kayyali et al *[1980]* were concerned with how the interruption of cement hydration affects the deposition of hydration products. They subjected plain and air-entrained cement-paste specimens to 18 or 36, three-hour, freezing and thawing cycles at an early age. In the plain paste specimens they discovered large, easily-cleaved portlandite ($Ca(OH)_2$) crystals. While the total porosity was not affected, there was a greater proportion of large voids in the frozen specimens. These stress-intensifying voids and weak portlandite crystals decrease the strength of the cement paste. With the air-entrained paste, the air voids provide a site for the segregation of portlandite during early freezing and thus crystals of a smaller size form. The air-entrained pastes do not experience the same decrease in compressive strength and fracture toughness as the plain pastes.

2.4 Criteria to Prevent Premature Freezing Damage

There is a need for criteria to ensure that damage due to premature freezing is avoided. The following four concepts have been discussed:

1. minimum strength,
2. minimum prehardening time,
3. critical dilation criteria, and
4. freezable water.

As the freezing damage mechanisms rely on the ability of the concrete to withstand the pressures generated by water migrating and freezing, there should be a minimum concrete strength which the concrete must attain to withstand the stresses generated by these pressures. (Strength is also a measure of degree of hydration and thus is related to the amount of freezable water remaining in the concrete.) The measure of strength most commonly used is compressive strength. Hoff and Buck *[1983]* reviewed many of the minimum compressive strength values that have been determined. These included strengths of 2.4 to 4.4 MPa in Sweden, 6.9 to 10.3 MPa in Canada, 4.8 to 8.8 MPa in the Soviet Union, 5.9 MPa in Japan and 14.5 MPa in Switzerland. One of the most commonly quoted values is 500 psi (3.5 MPa), the value given in the current ACI Standard Specification *[ACI Committee 306, 1989b]* and which has been in the ACI Standards and Recommendations since 1948 *[Carino, 1988]*. According to ACI once the concrete has achieved this strength it is safe from premature freezing damage regardless of the type and length of curing prior to freezing. RILEM[1] (International Union of Testing and Research Laboratories) recommends a slightly higher minimum compressive strength of 5.0 MPa (based on cube strength) *[Krylov, 1985]*. One of the lowest minimum strengths (2.0 MPa) suggested was by Pink *[1967]* in his recommendations for winter concreting in Britain. The RILEM recommendations say that this minimum strength should be increased to a maximum of 70% of the design strength if the concrete is saturated and exposed to a freeze-thaw environment during service. The Japanese standard also increases its minimum strength of 5 MPa to as high as 20 MPa for saturated concrete *[Koh, 1985a]*.

In Hoff and Buck's review, Bergstrom *[1976]* is quoted as saying "the assumption that there is a general strength at which concrete is frost proof is a fairly rough approximation." This is a very appropriate comment when one considers the wide range of critical strengths which have been suggested. There has been a great deal of debate about what is an acceptable minimum strength, but it is apparent that there will not be one value at which all concrete is safe. Numerous factors, including the amount of freezable water, the structure of the capillary pore system, the presence of dissolved solids in the pore water, and the rate of freezing will affect the amount

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[1]RILEM established a technical committee on winter concreting in 1976 with experts from 13 countries. They published the latest "International Recommendations on Concreting in Cold Weather" in 1985 in an attempt to bring together the knowledge and practices from many countries in one document.

of stress created by premature freezing in any situation. Therefore, concrete at a certain strength level may be strong enough to withstand freezing in one situation, but not strong enough under different conditions.

The other criterion commonly used to ensure that there is no premature freezing damage is the minimum prehardening time (mpt). Under this criterion the concrete must be cured (above a certain temperature) for a required length of time. The mpt concept is based on the fact that, after a certain amount of time, the concrete will have achieved a certain strength and permeability, and hydration will have progressed to a point where the amount of freezable water is below a harmful level. As with the minimum strength concept, there are too many variables to determine one value for the mpt. The various aspects of the mix proportions (type and amount of cement, water-cement ratio, etc.), curing temperature and nature of freezing will all affect the mpt required. Thus any code will have to use a conservative value for the mpt to ensure that all concretes will be safe. This is the type of criterion currently being used in both ACI and Canadian Standards Association (CSA) Standards and Recommendations[1] *[ACI Committee 306, 1989a, 1989b; CSA, 1990].*

In the ACI Standard, the basic protection period to avoid freezing damage is 3 days at a minimum temperature of 5°C to 13°C, depending on the section size. This protection period can be reduced to 2 days if Type III cement, an accelerator, or 60 kg/m^3 of extra cement is used. The protection period can also be reduced to a

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[1]ACI has published both a Standard Specification (ACI 306.1-87) and Recommendations (ACI 306R-78 revised 1983) for Cold-Weather Concreting. The Standard Specification is a short document intended for incorporation into project specifications. It contains the minimum requirements needed to produce satisfactory results whereas the Recommendations contain more detailed information on recommended practices and procedures for cold weather concreting.

minimum of 24 hours if the in-situ strength is greater than 500 psi (3.4 MPa). The ACI Recommendations qualify these protection periods stating they are for normal weight, air-entrained concrete that will not be frozen in a saturated condition. The above requirements are only for protection against freezing damage; they do not ensure adequate strength gain for form removal or early loading. The ACI Recommendations also include protection periods for adequate strength gain depending on when the structure will be loaded. In the CSA Standard, the basic curing period is 3 days at greater than 10°C or until 35% of the 28-day design strength is attained. For "extra durability" (i.e. for concrete which will be exposed to freeze-thaw, severe abrasion or severe air pollution while in service) the basic curing period is extended by 4 days or until 70% of the design strength is achieved.

Maturity methods have often been used to calculate minimum maturities and hence prehardening times once a minimum compressive strength has been determined *[ACI Committee 306, 1989b; ASTM, 1989b; Pink, 1967, 1985; Koh, 1985a, 1985b]*. The maturity factor is a function of time and temperature, most commonly expressed in North America as:

$$M = \Sigma(C+10)\Delta t$$

where C = temperature in degrees Celsius, and

Δt = duration of curing at temperature C in hours.

A strength-maturity factor relationship for a particular concrete is determined by testing a series of cylinders in the laboratory. Thus if a time-temperature record of the on-site concrete is available the in-situ strength can be estimated. Or similarly, if a minimum strength is required and the curing temperature is known, a minimum protection period can be estimated. Although strength-maturity relationships are widely researched and used, there continues to be some controversy about the validity of many maturity functions at low temperatures *[CPCA, 1984; Houde, 1990]*.

Another approach is the critical dilation criterion which is concerned with the volume change of concrete as it freezes. The current ASTM Standards *[ASTM, 1989a]* contain ASTM C671 "Test Method for Critical Dilation of Concrete Specimens Subjected to Freezing" which involves monitoring the length change of a specimen undergoing freezing cycles. The criterion for frost resistance depends on a sharp increase in dilation from one cycle to the next. Buck *[1988]* has proposed a change to the procedure and criterion based on his earlier research *[Buck, 1976]*. He proposes that only a single cycle of freezing be used and that the criterion be an absolute dilation value. (Dilation less than 50 microstrain would indicate frost resistance whilst dilation exceeding 200 microstrain would indicate lack of frost resistance; anything between these two levels would be unclassified and additional cycles could be used.) These changes would make the test method more applicable for premature freezing frost resistance. For the critical dilation criterion to be of practical use it must be converted to a minimum strength or prehardening time for a particular concrete in a particular situation. In this way the minimum strength, or time, can be monitored in the field to determine when the concrete is safe from freezing damage.

Theoretically, if the amount of freezable water in the concrete is known, one could determine if the freezing of this water can be accommodated by the elastic volume change and the air-void system of the concrete. By knowing the amount of heat released at freezing and the latent heat of fusion of water, one can calculate the amount of freezable water. However, Hoff and Buck *[1983]* reviewed a number of papers that indicate practical application of this concept is not simple.

This "freezable water" concept brings the degree of saturation of the concrete into the picture. It has been shown that when the concrete has a high degree of saturation, it is very susceptible to freezing damage. Even fog curing *[Roshore, 1967]* or sprinkling with water *[CPCA, 1984]* can increase the damage. Thus water curing is highly discouraged in cold weather concreting. The current ACI and CSA Standards *[ACI Committee 306, 1989a; CSA, 1990]* require that any water curing be terminated 24 and 12 hours respectively, before the temperature protection period ends to allow the concrete sufficient time to dry out. Of course, the concrete can also be saturated accidently. Care must be taken to keep the concrete clear of unexpected precipitation or snow and ice melted by surrounding construction.

2.5 Preventing Premature Freezing

To ensure that the mpt or strength criterion is satisfied in the field various curing methods have been developed to prevent freezing. These can be divided into two categories:

1. curing without applied heat, and
2. curing with applied heat.

The first category includes thermos curing and antifreeze admixtures. Thermos curing relies on keeping the concrete from freezing with the concrete's initial internal heat and the heat produced by cement hydration. This can be assisted by various insulating blankets, forms, and enclosures as well as preheating the concrete materials before mixing. One research project considered the use of heated fresh concrete where the initial concrete temperature ranged as high as 60°C *[Kukko, 1985]*.

The use of cold weather or antifreeze admixtures to lower the freezing point of the pore water and ensure strength gain at sub-zero temperatures is quite common in the Soviet Union and Europe, but not in North America. A 1985 report *[Turenne, 1985]* claims there is no recorded use of such admixtures in Canada. This type of admixture is strongly discouraged by the ACI Recommendations *[ACI Committee 306, 1989b]*. However, a recent American article *[Brook et al, 1988]* indicates that the North American concrete industry is taking another look at developing such admixtures. Commonly used admixtures include sodium nitrite, sodium sulphate, calcium nitrate, and potassium carbonate (potash) together with various accelerators, retarders or workability agents, depending on the properties of the admixture *[Ivanova et al, 1985; Kivekäs & Leivo, 1985]*. Some of these admixtures can be used at temperatures as low as -20°C. Although the initial cost of concretes with antifreeze admixtures can be 75% greater than ordinary concretes *[Kivekäs & Leivo, 1985]* there is potential for large cost savings due to reduced protection and heating requirements. There have been durability concerns with respect to some admixtures. One unique admixture in today's environmentally-conscious world was developed in the Soviet Union and is a byproduct made from the waste materials of a chemical fertilizer factory *[Matyszewski et al, 1985]*.

Other approaches include:

1. the development of super-rapid hardening cements with normal setting times *[Krylov, 1985]*;
2. regulated set cements that develop high strengths at one to two hours after mixing *[Hoff, 1976]*; and
3. the use of accelerators for high early strength.

With these techniques a substantial amount of internal heat is generated soon after casting which will prevent freezing during the following critical time period. Zachara *[1985]* discovered that hardening of concretes made from sintered aluminous cements takes place at temperatures as low as -15°C and can even occur at -20°C if subjected to delayed vibration. Thus there is potential for improved frost resistance with these cements.

The second category of curing methods involves curing with additional applied heat to keep the concrete from freezing. Various electric and gas heaters, using forced air, steam or radiation, in conjunction with protective enclosures are commonly used. Electric blankets and heated forms are effective in some applications. Induction heating by applying a low voltage current to the reinforcing rods or the concrete itself has been tried *[Ghosh & Mustard, 1983]*, along with imbedded electrical resistances. The Soviets have tried covering the concrete with a layer of electrically conductive polymer containing embedded electrodes *[Abramov et al, 1985]*.

2.6 Improving Frost Resistance

As well as preventing freezing of the concrete, the problems associated with premature freezing can be tackled by improving the frost resistance of the concrete. One traditional way of doing this is to use air-entraining agents. Small, regularly-spaced air bubbles in the concrete provide sites for water to migrate to and for ice crystals to grow without the imposition of stress. The benefits of air-entrainment with respect to improving freeze-thaw frost resistance are well documented and accepted. Similar benefits have been observed with premature freezing of air-entrained concrete. The ACI Standard Specification *[ACI Committee 306, 1989a]* requires that any concrete which will be exposed to freezing in a wet condition during construction shall be air-entrained irrespective of whether it will be exposed to freezing while in service. However, there are many factors which make it difficult to control air-entrainment and ensure that bubbles with the correct size and spacing exist in the concrete. Ramachandran et al *[1981]* suggest these problems could largely be avoided if the preformed bubble reservoirs could be added in the form of particles. They describe two inventions which use this principle:

1. hollow plastic microspheres *[Sommer, 1977]* and
2. porous particles *[Litvan, 1978]* made from materials such as commercially fired clay bricks, diatomaceous earth and vermiculite.

Structural, and especially insulating, lightweight concretes are more resistant to heat loss than normal weight concrete *[ACI Committee 306, 1989b]*. Other aspects of mix design, particularly with respect to lowering the water-cement ratio can also improve the frost resistance of the concrete. Acceleration of strength gain through cement composition (i.e increasing tricalcium silicate component) or accelerating additives will allow the concrete to be exposed to freezing temperatures earlier. However, accelerating strength gain, which is usually accompanied by increased porosity and permeability, can be detrimental to long-term durability.

Aggregates can play a role in frost resistance. If the aggregate's pore structure is such that the pore water readily freezes, either the aggregate will expand elastically or the water will flow out from the aggregate under pressure *[Ramachandran, 1981]*. Both processes will affect the surrounding concrete matrix. Thus aggregates should be chosen with care, and attention should be given to their moisture condition prior to mixing.

2.7 Determining the Extent of Premature Freezing Damage

From a practical point of view, one of the most important aspects of premature freezing is how to determine whether frozen concrete has been damaged. Aside from some surface damage which is occasionally observed (see page 10), there will not be any visible signs of damage. Pink *[1967]* claims that it will be obvious if the concrete was frozen while plastic, but not if it had stiffened prior to freezing. In the case study *[Matti, 1986]* described previously, ultrasonic pulse velocity methods were used successfully to determine the location of damaged concrete. Similarly, the Schmidt Hammer and Windsor Probe tests have been used to check the uniformity of the concrete with good results *[Sarja, 1985; Turenne, 1985]*. Note that these tests are evaluating the surface concrete where freezing damage is most likely to occur. Field-cured specimens may be of some assistance, but the temperature conditions within the specimens will not be the same as that of the larger cast concrete sections and thus may not reflect the same amount of damage. Cored specimens and pull-out tests may also be used for strength. With these strength methods the apparent loss in strength may be only a delay in rate of strength gain rather than permanent damage. Unfortunately, the effects of freezing on the concrete's durability are not determined by any of the commonly used test methods described above.

2.8 Use of Fly Ash with Respect to Premature Freezing

One of the latest trends in concreting is to use a supplementary cementing material like fly ash. Extensive research has been carried out into the use of fly ash — how it affects workability, mechanical properties, durability, etc. A change in the cementious material will (theoretically at least) also affect the resistance of a concrete to premature freezing. Fagerlund *[1985]* suggests several reasons why:

1. "the pore structure will undergo changes which means that changes will also appear in the permeability and in the freezable water.
2. the amount of self-desiccation, i.e. the amount of "chemical shrinkage" of the non-evaporable water might be changed. This will affect the evolution of the air-space volume created by self-desiccation in the cement paste."
3. the reactivity of the cement is reduced. Therefore the rates of cement hydration, strength gain and heat evolution are lower, requiring longer protection times.

Many specifications do not permit the use of fly ash in cold weather concreting without approval of the engineer *[Day, 1991]*. It would seem that this is largely due to concern regarding the slow rate of strength gain in fly ash concretes which may be slowed further in cold temperatures. Gardner *[1990]* had results that showed the long-term strength development of Type I/ 25% fly ash concrete cured at 0°C is roughly 10 MPa less than that of similar concretes cured at 20°C. The reduction in strength is severe at ages less than 14 days. Day *[1991]* presented similar results. The strength reduction was even more significant in concretes with a 50% fly ash replacement level.

Day also carried out a preliminary study into the effects of freezing on fly ash concretes. Air-entrained and non air-entrained, plain and fly ash concretes with 20% and 30% replacement were used. They were frozen at -20°C for 48 hours, after curing periods at room temperature ranging from 8 hours to 7 days. His results showed:

1. at the 3 day testing age, slightly lower strengths with frozen specimens as compared to normally-cured specimens;
2. at 28 and 90 days, no significant strength losses, small strength gains with frozen concretes in some cases; and
3. in the long-term, no apparent difference between plain and fly ash concretes or between air-entrained and non air-entrained concretes.

This research provided the starting point for the following research project, "The Effects of Premature Freezing on Plain and Fly Ash Concrete Strength Development". The literature reviewed in this chapter suggested some unanswered questions and concerns to consider in a project on premature freezing. The following chapter will discuss how these influenced the objectives and procedures of the research.

CHAPTER 3: RATIONALE AND OBJECTIVES

Damage due to premature freezing is one of the reasons there is a great deal of reluctance to use fly ash as a supplementary cementing material in cold weather concreting. Yet, very little is known about the effects of premature freezing on fly ash concretes. Further research to understand the problem is required.

It is apparent from the previous chapter that there are many different effects of freezing which could and should be considered, but of course it is impossible to cover all these effects in one project. Loss of strength is always a primary concern because any significant permanent loss could make the concrete unfit for its intended use. Delays in strength gain, as opposed to permanent losses, will also affect safety and timing during construction.

Compressive strength testing of cylinders or cubes has traditionally been the most common method of measuring strength and assessing concrete quality. Specimens for compressive strength testing are relatively easy to prepare and test. The development of standard procedures, which are in widespread use, has ensured that compressive strength continues to be the industry standard. There are, though, questions regarding the reliability of compressive strength to measure changes in fracture behaviour *[Kayyali, 1979]*. Bergstrom claimed that the modulus of rupture (i.e. flexural strength) is more sensitive to deterioration than compressive strength *[Bergstrom, 1955]*. Houde's results *[Houde, 1990]* contradict this statement — compressive strength was affected more than modulus of rupture in his tests. Both compressive and flexural strength testing were used in this project to see which is more sensitive to freezing.

To understand the strength testing results and to provide information for any future studies on durability loss or other effects, it is important to measure the basic properties of the various concretes used. Thus fresh concrete tests (slump, air content, and yield) were carried out. Initial and final setting times were obtained using penetration resistance tests on mortar sieved from the concrete. The relationship between these setting times and the age at freezing may be important in assessing the amount of damage and indicating the damage mechanism responsible. Point-count tests to determine paste content, air content and air void spacing factor in the hardened concrete were also performed.

Another important aspect of the testing program was the monitoring of internal temperatures with thermocouples. The objectives and reasoning for this temperature monitoring were:

1. to determine the actual concrete age at freezing. The specimen age at placement in the cold room is not the same as the age when concrete actually freezes (i.e. reaches 0°C). There is a delay as the concrete cools down from the pre-freezing temperature and as heat is generated by cement hydration.
2. to monitor for temperatures other than 0°C which may be critical for freezing damage. Only "free" water freezes at 0°C — the freezing temperature for the rest of the water depends on how it is confined in the pore structure. Cement hydration has virtually stopped at -10°C — this may be another temperature to consider.
3. to monitor temperature gradients with respect to time (rate of cooling) at various locations in the specimens. The rate of freezing influences

freezing damage. The influence of various factors including mix design variables, concrete age at freezing and degree of saturation on the gradient was studied.

4. to monitor temperature gradients within specimens. Temperature differences in the concrete are a driving force for moisture movement. Again, the influence of various factors on these temperature gradients was considered.

5. laboratory constraints require the use of small specimens, rather than the full-scale elements actually found on the construction site. Ultimately, temperature monitoring can be used to see how these small samples are simulating the temperature conditions (gradients, etc.) in such full-scale elements. Temperatures must also be monitored in order to do any maturity calculations.

This internal temperature monitoring should provide some useful information for explaining other test results.

Many aspects of the experimental procedures (described in detail in Chapter 4) were kept the same as Day's earlier project *[Day, 1991]* so that a comparison of results was possible. Preliminary tests carried out in the summer of 1990 were used to help develop the details of the procedures. The large number of specimens required in the main testing program meant that multiple batches of the same concrete mix were necessary. The procedures used for each step, from materials selection to testing, helped maintain consistency from batch to batch.

A key element of the procedure is placing the concrete (at various ages) in a cold room at -20°C for 48 hours. This freezing regime simulates an accidental freezing due to inadequate equipment, equipment failure or unexpected weather over a weekend time period. It is apparent from previous research that freezing at relatively early "concrete age" is required to see any significant damage. Thus freezing at 8, 17 and 24 hours from mixing was selected.

The degree of saturation at freezing has been shown to influence the freezing effects. Moist-cured or water-soaked specimens typically experience a greater amount of damage than air-cured specimens. Thus some of the specimens were soaked before freezing to simulate water-cured concrete or concrete accidentally soaked by rain or the melting of surrounding snow and ice. The preliminary work for this project indicated that the soaking regime could also affect the long-term strength of normally-cured (unfrozen) specimens. Thus, for each set of soaked specimens which were frozen another set of specimens was soaked and cured without freezing to use in strength comparisons.

The concrete mix designs used reflect what is currently being used in the construction industry and meet current CSA requirements *[CSA, 1990]*. As this was research into the effects on fly ash concretes some of the mixes obviously contained fly ash. For comparison, control mixes without fly ash were also used.

Previous research has indicated air-entrainment will significantly reduce the detrimental effects of premature freezing. Thus, both non air-entrained and air-entrained concretes were tested. Typically, the non air-entrained concretes are for interior use where there is no possibility of exposure to a freeze-thaw environment during service. However, it is not unrealistic for them to be exposed to freezing during construction.

Mix designs were selected that produce concretes with similar fresh concrete properties and long-term strengths. Thus, concretes requiring the same construction procedures and with the same structural applications are compared. The mix designs used in this project are for low-strength structural concretes; they have relatively low cement contents and high water-cement ratios. These are concretes which, judging by previous results, tend to be affected more seriously by premature freezing (i.e. testing the "worst case scenario").

Consideration of the above details created a research project which provides information on the impact of premature freezing on concrete strength development. The inclusion of variables like air-entrainment, age at freezing and soaking gives some insight into what factors affect the amount of damage. Temperature monitoring and qualitative analysis of the fracture mode will provide further insight into the strength testing results. As with any research project, the scope of the testing program and the simulation of actual site conditions were limited by laboratory and time constraints. The details of the mix designs, procedures and testing program are given in the following chapter.

CHAPTER 4: EXPERIMENTATION

This chapter summarizes the materials, mix proportions and experimental procedures used. It concludes with an overview of the testing program.

4.1 Materials

The coarse aggregate used was a limestone/quartzite river gravel available locally in the Calgary area. It had a 14 mm nominal maximum size, a dry-rodded unit weight of 1635 kg/m^3, a specific gravity (SSD) of 2.56 and an absorption value of 1.0%. A grading curve for the aggregate and the CSA requirements for a Group I aggregate are given in Figure 4.1 *[CSA, 1990]*. The coarse aggregate was brought to saturated-surface-dry (SSD) condition by a soaking and draining regime prior to mixing.

The fine aggregate was also a locally available limestone/quartzite river gravel. To maintain consistent grading the sand was sieved into three proportions:

fine	< 0.315 mm,
intermediate	> 0.315 mm and < 1.25 mm, and
coarse	>1.25 mm.

It was then recombined in fixed proportions: 15% of the fine material, 70% of the intermediate material and 15% of the coarse material. The resulting grading curve and the corresponding CSA requirements are shown in Figure 4.2 *[CSA, 1990]*. This recombined sand had a fineness modulus of 2.43, a bulk specific gravity of 2.65 and an absorption value of 1.6%. In order to bring the fine aggregate to SSD condition, moisture was added to it after placement in the mixer and prior to the addition of the other mix materials.

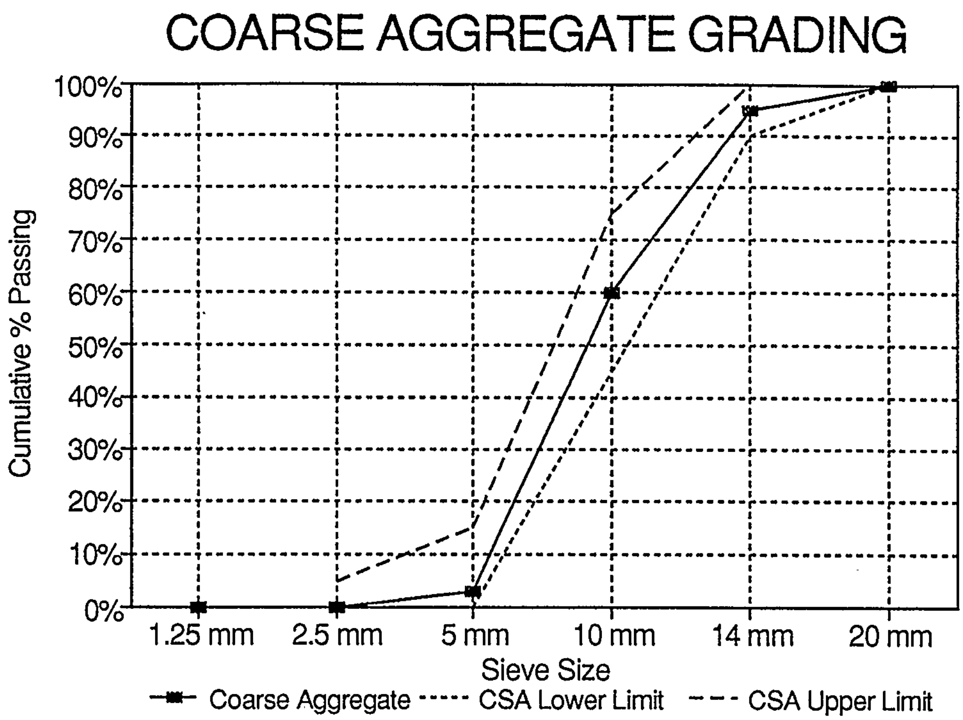


FIGURE 4.1: Coarse Aggregate Grading Curve with CSA Requirements

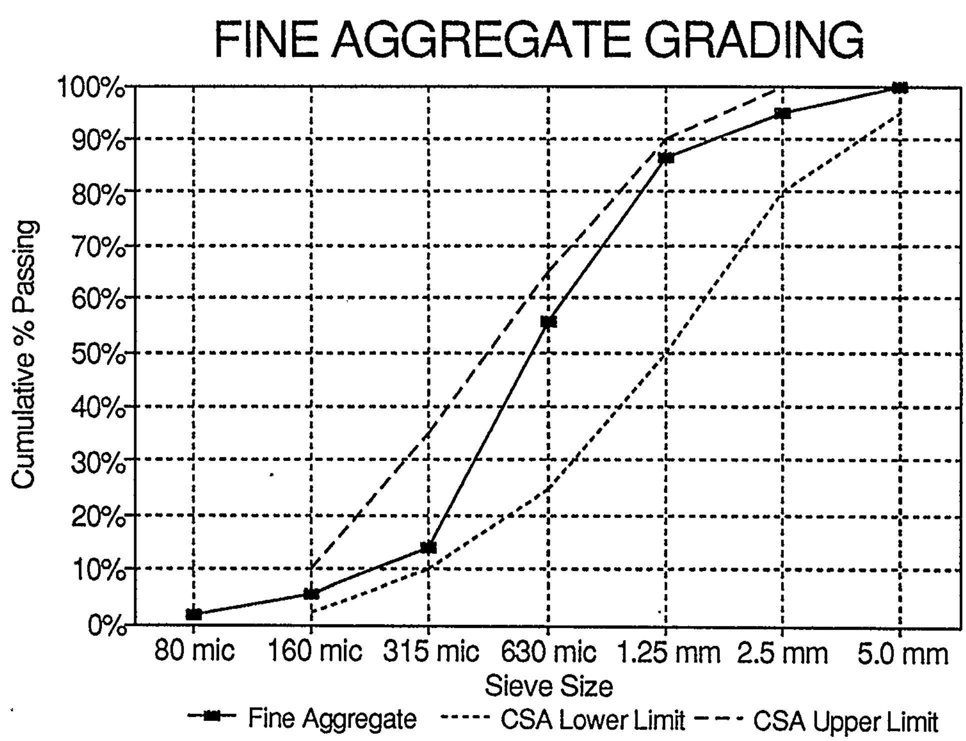


FIGURE 4.2: Fine Aggregate Grading Curve with CSA Requirements

Type 10 Portland cement and a CSA Class C fly ash from Alberta were used. The chemical and physical properties of these cementious materials are given in Table 4.1 *[Day, 1991]*.

TABLE 4.1: Properties of Cement and Fly Ash

Chemical Composition	Type 10 Cement	Fly Ash
SiO_2	20.8%	57.8%
Al_2O_3	4.0%	23.0%
Fe_2O_3	3.3%	3.5%
CaO	61.6%	9.9%
MgO	4.3%	1.5%
Na_2O	0.15%	2.3%
K_2O	0.66%	0.50%
SO_3	2.7%	0.3%
Physical Properties	**Type 10 Cement**	**Fly Ash**
Loss on Ignition	1.7%	0.5%
Specific Gravity	3.15	2.04
Spec. Surface — Blaine	4030 cm^2/g	2800 cm^2/g
— Nitrogen		16100 cm^2/g
Pozzolanic Activity Index (cement)		91
% Retained on 45μm Sieve		26%

A neutralized vinsol resin air-entraining agent was used when air-entrainment was required. No other admixtures were used. Calgary tap water was used as the mix water.

4.2 Concrete Mix Proportions

The mix proportions were selected to meet the following criteria:

1. a slump of 80 ± 20 mm,
2. a 28-day compressive strength of 35 ± 5 MPa,
3. if air-entrained, an air content of 6.5 ± 0.5%,
4. a fly ash replacement level of 25% by weight (when applicable), and
5. CSA requirements *[CSA, 1990]*.

Trial mixes were used to refine the calculated mix proportions until the criteria were met. The final mix proportions are given in Table 4.2. The calculated mix proportions have been adjusted to actual mix proportions by accounting for the difference between the theoretical unit weight and the measured unit weight during the test program. In the mix code notation CON refers to the control mixes without fly ash and FA refers to the mixes containing fly ash. The next letter, N or A, refers to no air-entrainment or with air-entrainment.

The w/c′ ratios for the non air-entrained mixes CON-N and FA-N are 0.48 and 0.47 respectively. For the air-entrained mixes CON-A and FA-A, the w/c′ ratios are 0.40 and 0.38.

The 28 day strength using standard testing procedures (ASTM C39-86) was determined during the trial mixes. The results are also given in Table 4.2

TABLE 4.2: CONCRETE MIX PROPORTIONS

MIX CODE (1)	CALCULATED PROPORTIONS (2) C. Agg	F. Agg	Cement	Fly Ash	Water	AEA(3)	THEOR. YIELD	ACTUAL YIELD(4)	ACTUAL PROPORTIONS (2) C. Agg	F. Agg	Cement	Fly Ash	Water	AEA(3)	28 DAY STRENGTH
CON-N	1021	757	325	0	157	0	2260	2350.3	1027	761	327	0	158	0	34.2 MPa
CON-A	1047	692	386	0	154	36	2279	2291.5	1053	696	388	0	155	36	34.1 MPa
FA-N	1021	753	244	81	153	0	2252	2350.3	1027	757	245	81	154	0	38.9 MPa
FA-A	1047	714	290	96	145	78	2292	2196.0	1053	718	292	97	146	78	34.1 MPa

NOTES:

1. Mix Code Notation:
   - CON - without fly ash
   - FA - with fly ash
   - N - no air-entrainment
   - A - with air-entrainment
2. All proportions given in kg/m^3 with the exception of AEA given in mL/100kg of cement+fly ash.
3. AEA - air entraining agent.
4. Actual yields based on average for batches used in testing program.

4.3 Experimental Procedures

The following procedures were followed to mix, cast, cure and test the specimens.

4.3.1 Mixing:

The dry fine aggregate (15% coarse, 70% intermediate and 15% fine) was placed in the mixer. The amount of water required to obtain SSD conditions in the fine aggregate was added and the mixture thoroughly agitated. A small sample of this sand was taken for oven drying and subsequent moisture content determination. A small sample of the drained coarse aggregate was taken and excess moisture (above SSD) was calculated. (The sample was retained for total moisture content determination.) The coarse aggregate (adjusted for excess moisture content), cement, fly ash and water (also adjusted for excess moisture content) were added to the mixer. If air-entraining agent was required, it was added to one litre of the mix water and added at this time. The concrete was mixed for a few minutes and then a slump test (ASTM C231-89a) was performed. The concrete was mixed for a further minute before being discharged. Finally, unit weight (ASTM C138-81) and air content (ASTM C231-89a) tests were conducted on the fresh concrete.

4.3.2 Casting:

The cylinders (75x150 mm disposable wax-coated cardboard moulds) and beams (75x75x300 mm plastic moulds) were cast in two layers. A vibrating table was used to consolidate each layer. When internal temperatures were monitored, thermocouples were inserted in the specimens immediately after casting and the specimens were vibrated by hand to ensure that the thermocouples were completely encased by concrete. With some mixes additional specimens were cast. For setting time tests, mortar was sieved from the fresh concrete using a 4.75 mm sieve and cast into penetration resistance specimens in accordance with ASTM 403-80. If point-count specimens were required, 150x150 mm cylinders were cast, in modified cardboard moulds, in two layers.

4.3.3 Curing:

The initial curing regime depended on whether or not the specimens were to be soaked before freezing. If the specimens were not to be soaked, they were sealed in plastic bags and were left sitting in the laboratory where the temperature was 22±2°C. If the specimens were to be soaked, they were covered with plastic until 4 hours after the addition of water to the concrete batch. They were then carefully placed in a water bath at room temperature. Just before the required freezing time the specimens were removed from the water bath and sealed in plastic bags.

At the selected freezing time (measured from the addition of water), half of the specimens were chosen at random and placed in a cold room. The temperature in the cold room was lowered to -20°C. (In one exception the cold room temperature was lowered to -40°C for the first 16 hours and then raised to -20°C.) After 48 hours the specimens were removed from the cold room and returned to the laboratory. During this 48 hour period, the remaining half of the specimens were left to cure in the laboratory. After another 24 hours, all the specimens were demoulded and placed in a fog room at 100%RH and 24.5°C until their testing age (measured from the start of fog room curing). The curing regime for the 8 hour freezingis illustrated in Figure 4.3. The 17 and 24 hour freezings would be the same except for the additional delay until the start of freezing.

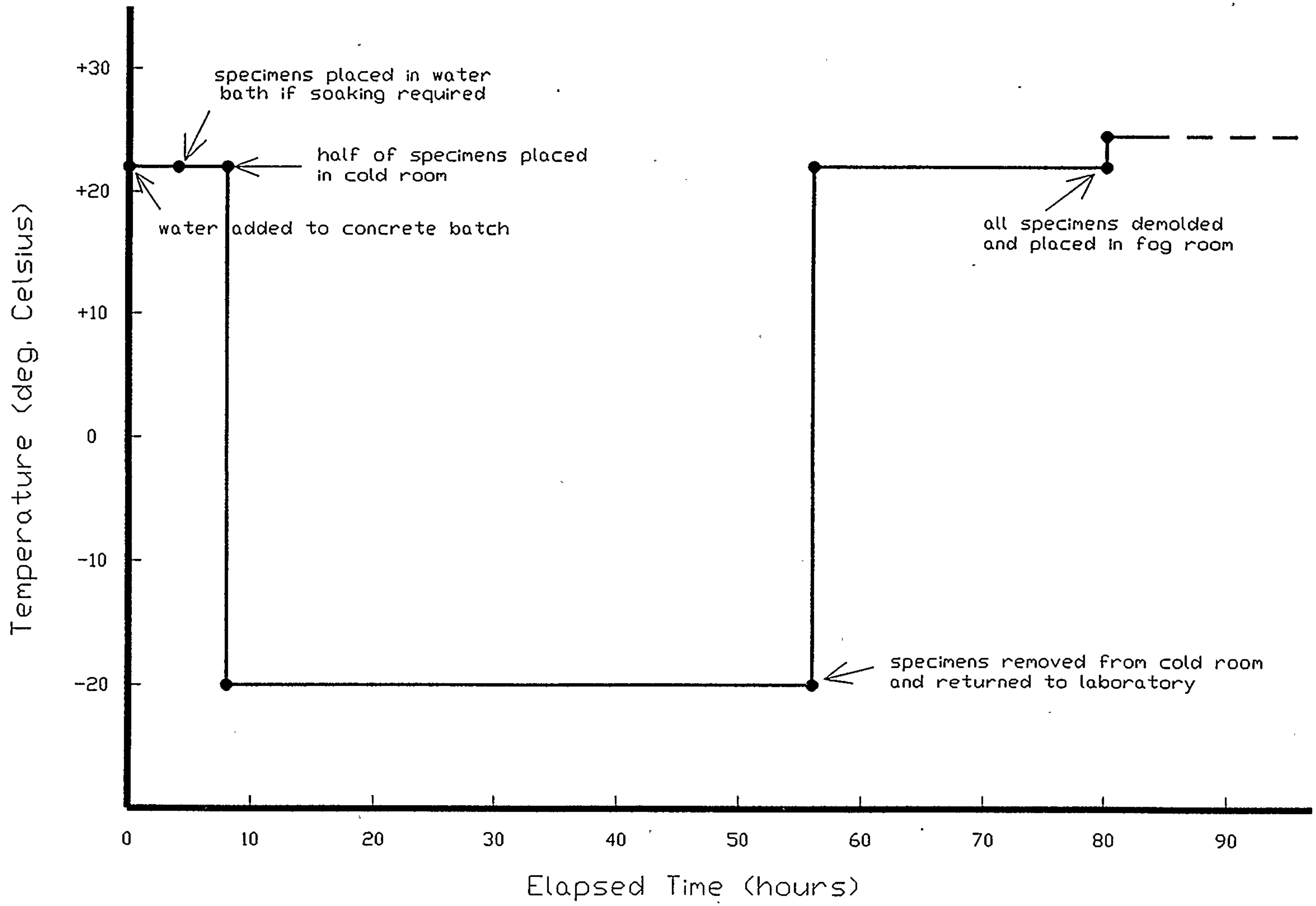


FIGURE 4.3: Curing Regime for 8 Hour Freezing

4.3.4 Testing

The penetration resistance specimens were tested in accordance with ASTM C403-80 to determine initial and final setting times.

The cylinders were tested for compressive strength in accordance with ASTM C39-86. The cylinders were capped with a sulphur/fly ash capping material. The beams were tested for flexural strength in accordance with ASTM C78-84 using third point loading. The flexural moulds had two beams side by side — thus each beam had an inner side which was cast parallel to another beam and an outer side. For consistency in the standard flexural tests, the outer side was placed in tension.

Testing for flexural and compressive strengths was carried out at 3, 7, 28 and 90 days from placement in the fog room. For each testing age, three normally cured and three frozen specimens were tested. Both sets of specimens had come from the same batch of concrete and had undergone identical curing regimes apart from 48 hours in the cold room for the frozen specimens.

Point-count specimens were sliced from the large cylinders and tested in accordance with the Modified Point-Count Method in ASTM C457-82a to determine the paste content, entrapped and entrained air contents, and air void spacing factor in the hardened concrete.

4.4 Testing Program

Fresh concrete tests (slump, unit weight and air content) were performed on all concrete batches. Two setting time tests and one point-count test were carried out for each of the four mixes.

The internal temperature monitoring is summarized in Table 4.3. To obtain the temperature gradients within the specimens, monitoring occurred at several locations. The thermocouple locations for the various monitoring schemes are illustrated in Figure 4.3. Monitoring typically began shortly after casting and continued until removal from the cold room.

An overview of the strength testing program is given in Table 4.4. Sets of specimens from all four mixes were subjected to the freezing regime at 8 hours in the soaked and dry condition. As well, sets from the CON-N and FA-N mixes were subjected to freezing at 17 and 24 hours in the dry condition. The 8 and 24 hour sets had flexural and compressive strength specimens. The 17 hour set had only compressive strength specimens.

In conjunction with this strength testing, both compressive and flexural specimens were tested in order to determine the population statistics. Some flexural specimens were also tested to study the effect of putting the inner or outer side in tension. One set of specimens was tested after freezing initially at -40°C rather than -20°C.

Observations of the fracture surface in the flexural specimens were made to determine if the failure was primarily at the paste-aggregate interface or through the aggregate.

TABLE 4.3: Internal Temperature Monitoring Program

Mix	Curing (1)	Thermocouple Locations (2)
CON-N	D-F8	along beam
	S-F8	along beam
CON-A	D-F8	along beam, across cylinder
	S-F8	along beam, across cylinder
FA-N	D-F8	along beam, across beam
	D-N	along beam, across beam
	S-F8	along beam
	D-F24	along beam
FA-A	D-F8	along beam

Notes:
1. D = dry, S = soaked
   F# = age at placement in cold room
   N = normal curing (i.e. no freezing in cold room)
2. Thermocouple locations described in Figure 4.4.

TABLE 4.4: Strength Testing Program

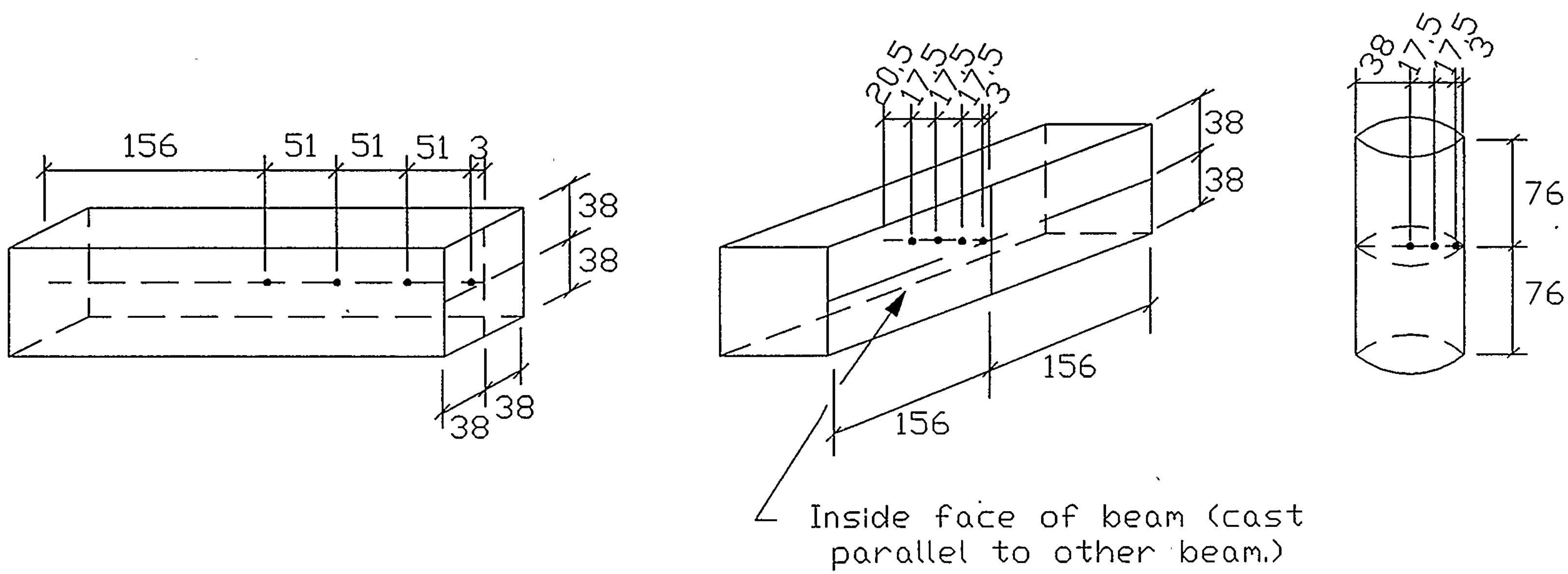
Mix	Age at Freezing			
	8 hours		17 hours	24 hours
	D	S	D	D
CON-N	C&F	C&F	C	C&F
CON-A	C&F	C&F	-	-
FA-N	C&F	C&F	C	C&F
FA-A	C&F	F	-	-

Notes:
1. Age at freezing is the age at placement in the cold room.
2. C = compressive strength testing
   F = flexural strength testing
3. D = dry, S = soaked

Along Beam

Across Beam

Across Cylinder



All dimensions in millimetres.

FIGURE 4.4: Thermocouple Locations

CHAPTER 5: RESULTS - SUMMARY AND DISCUSSION

The results from the various components in the testing program are summarized and discussed in this chapter. They are presented in the following order:

1. fresh concrete properties,
2. setting time tests,
3. internal temperature monitoring,
4. point count tests,
5. strength testing, and
6. fracture mode analysis.

5.1 Fresh Concrete Properties

The results from the slump, air content, and unit weight tests are summarized in Table 5.1. The individual batch results are given, as well as the average for each concrete type. The curing regime that each batch was exposed to is also included. Abnormalities in the fresh concrete properties assist in explaining the strength testing results which are presented later in this chapter.

Previous quality control testing *[Day, 1991]* has indicated that with the moisture control procedures used during mixing, the actual water-cement ratio (w/c) of a particular batch is within 0.01 of the target w/c with 95% confidence. Thus the apparent variability in the slump results is most likely due to the slump testing procedure, not due to a varying w/c from batch to batch. In particular, the third FA-N batch had a slump of 140 mm, but the workability was not significantly different from the other FA-N batches.

TABLE 5.1: FRESH CONCRETE PROPERTIES

Mix Code	Batch (1)	INDIVIDUAL BATCH RESULTS Slump (mm)	Air Content (%)	Yield (kg/m^3)	AVERAGE Slump (mm)	Air Content (%)	Yield (kg/m^3)
CON-N	D-F8	95	2.4	2359	70	2.5	2350
	S-F8	50	2.2	2346			
	D-F17,F24	65	3.0	2346			
CON-A	D-F8	70	6.2	2292	70	6.3	2292
	S-F8	65	6.4	2291			
FA-N	D-F8* (2)	85	1.8	2342	95	1.9	2350
	D-F8	80	1.8	2345			
	S-F8	140	1.9	2346			
	D-F17,F24	85	2.0	2368			
FA-A (3)	D-F8	60	7.0	2196	60	7.0	2196
	S-F8	45	4.5	2320			

Notes:
1. Batch Notation: D = dry, S = soaked, F# = age at freezing
2. This batch was frozen at -40°C rather than the standard -20°C.
3. Average does not include values from second batch as air-entrainment did not work properly due to a problem with the mixer.

Due to a problem with the concrete mixer, the second FA-A batch was not mixed for as long as the standard procedure required. This meant that the air-entraining agent had insufficient opportunity to work properly and the air content (4.5%) was lower than desired. The low air content explains the low slump and high unit weight measurements for this batch. These values are not included in the averages.

Replacing some of the cement with fly ash significantly increases the amount of air-entraining agent required to attain the desired air content. It is also more difficult to maintain consistent air content levels with the fly ash concretes.

The fresh concrete results indicate that the mix proportions yield concretes that:

1. meet the mix criteria specified in Section 4.2 of the previous chapter, and
2. have similar fresh concrete properties.

Although the workability of the mixes, as measured by slump, is basically the same, from a practical viewpoint, the air-entrained mixes are easier to work with and are less prone to bleeding and segregation. It should be noted that the fly ash mixes had a slightly lower water content (and hence w/c′) than the comparable control mix while maintaining the same workability.

5.2 Setting Time Tests

Figure 5.1 is a graph of penetration resistance plotted against the elapsed time from mixing for each mix type. From this graph the time taken to reach a penetration resistance of 3.5 MPa and 27.6 MPa can be determined — i.e. the initial and final setting times can be determined. These are given in Table 5.2.

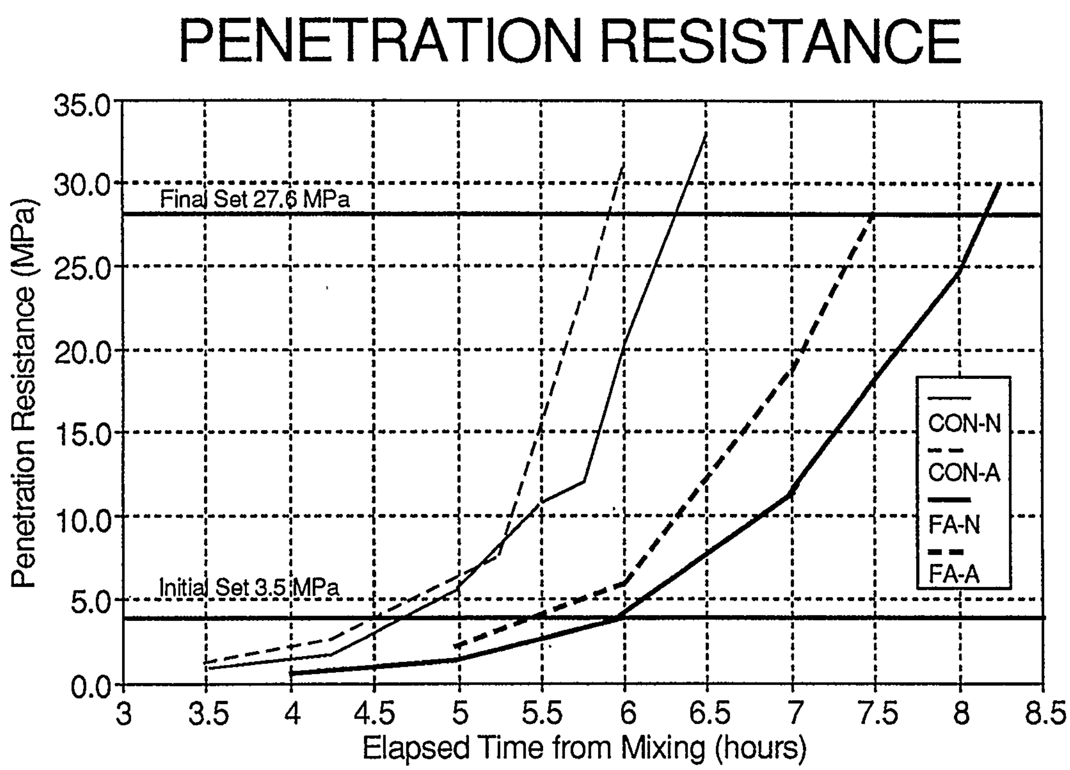


Figure 5.1: Results of Penetration Resistance Tests for Setting Times

TABLE 5.2: Initial and Final Setting Times

Mix	Initial Setting Time (hours)	Final Setting Time (hours)
CON-N	4.7	6.3
CON-A	4.6	5.9
FA-N	6.0	8.2
FA-A	5.5	7.5

The results follow typical trends for concretes. The fly ash mixes (FA-N and FA-A) have longer setting times than the corresponding control mixes (CON-N and CON-A). This is because fly ash has a slower rate of hydration than Type 10 cement. The non air-entrained mixes (CON-N and FA-N) have longer setting times than the corresponding air-entrained mixes (CON-A and FA-A) primarily because the total cementious material content (cement + fly ash) is lower in the non air-entrained case.

FA-N is the only mix which had not reached final set at the time of placement in the cold room (8 hours after adding water to batch). But as the internal temperature monitoring described next in this chapter will indicate, at the final setting time of 8.2 hours for this mix, the specimens' internal temperature had not fallen below 0°C.

5.3 Internal Temperature Monitoring

Internal temperature monitoring was carried out using thermocouples embedded in the specimens. A variety of mixes under different curing conditions was monitored as described in Section 4.4. A complete set of results (for the first 24 hours after mixing) is given in Appendix A.

5.3.1 Typical Temperature Behaviour

A typical set of results is given in Figure 5.2. These results are from the FA-N mix placed (in the dry condition) in the cold room at 8 hours. Temperatures were monitored from the surface to the centre of the beam specimen, at four locations along its length.

The temperature curves can be divided into four phases. In the first phase (from casting to placement in the cold room) the concrete temperature rises above the

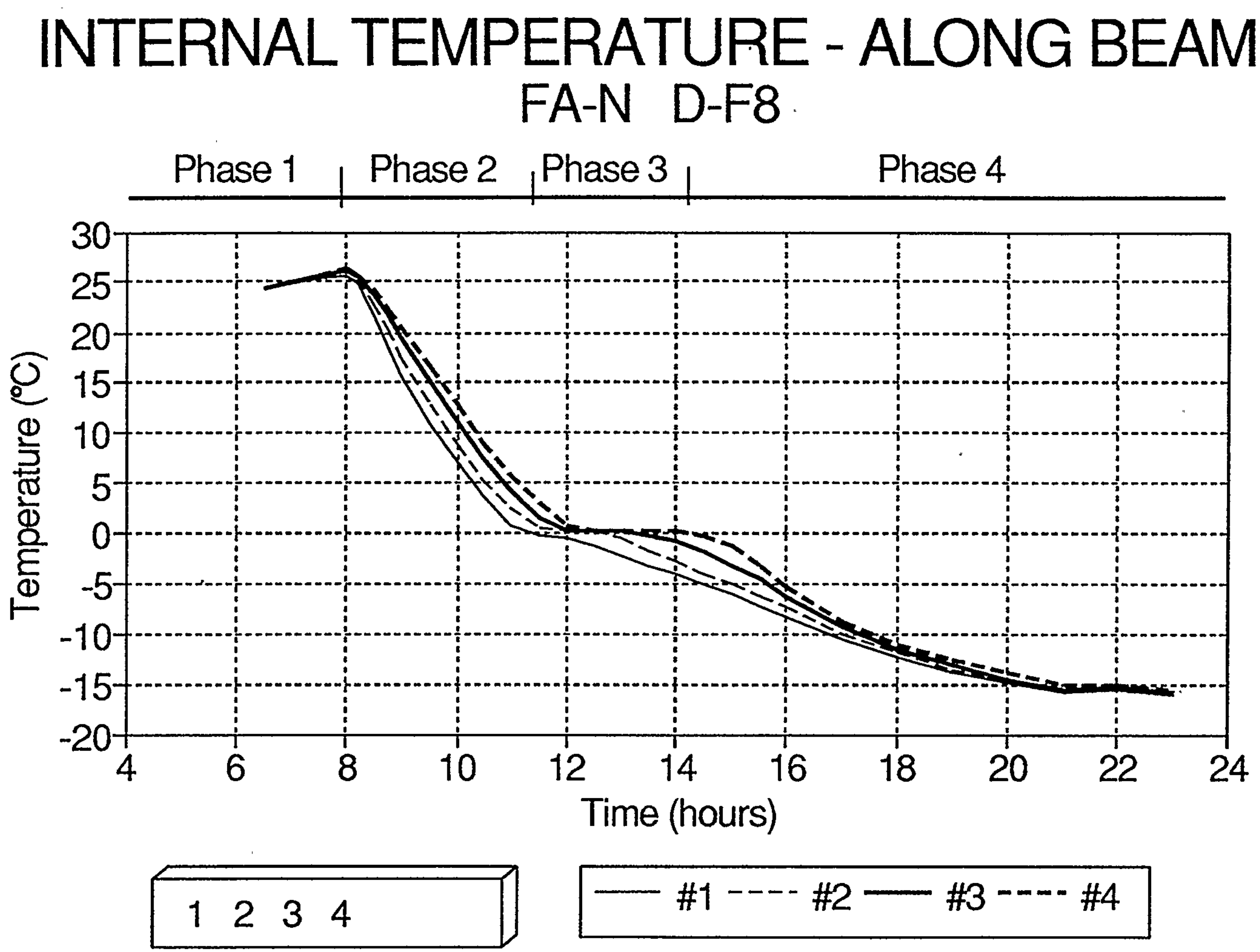


FIGURE 5.2: Typical Temperature Monitoring Results

ambient temperature of the laboratory, because of the heat produced by cement hydration. In the second phase, (following placement in the cold room), the temperature falls to 0°C. The rate of cooling is fairly constant during this phase. During the third phase, the concrete temperature stays at 0°C for a period of time. This phase lasts longer at the centre of the beam and corresponds to the freezing of "free water" in the concrete. In the final phase, the temperature decreases to the ambient temperature of the cold room (-20°C). The rate of cooling during this stage is slower than in phase 1 and gradually decreases. This general pattern of temperature behaviour occurs in all specimens.

In the example of Figure 5.2, the temperature rises to 26°C at the end of phase 1. Phase 2 takes 3.5 hours for the surface of the specimen and 4.5 hours for the centre. Phase 3 lasts from 0.5 hours at the surface of the specimen to 2 hours at the centre. In phase 4, the specimens take another 15 hours to reach the cold room temperature.

5.3.2 True Age at Freezing

With placement in the cold room at 8 hours, the surface of the beam specimens reaches 0°C in 3 to 4.5 hours depending on the mix design and curing conditions. The centre of the beam reaches 0°C in 4 to 5 hours, but does not fall below 0°C until 6 to 6.5 hours. "Age at freezing" can be defined as either the age at which the surface layer of the concrete reaches 0°C, or as the time period over which all the concrete falls below 0°C. In either case, it is clear that age at freezing should be differentiated from age at placement in a freezing environment. In the case of the cylinders (Figures A.12 and A.13), "freezing" occurred much quicker; 0°C being reached in 2 hours and the temperature falling below 0°C at 2 to 3 hours from placement in the cold room.

The specimens reach -10°C (the temperature at which hydration, and hence strength gain, stops) in 16 to 18 hours.

5.3.2 Temperature Differences and Gradients

Another area of concern is the temperature differences and gradients that develop within the concrete because of the different cooling rates within the specimen. The graphs in Appendix A can be used to determine the temperature difference between the surface and the centre of the specimen. An average temperature gradient can be calculated by dividing by the distance between the thermocouples as shown in Figure 4.4.

A FA-N beam specimen was monitored under the normal curing condition (i.e. no freezing). Its temperature curves are given in Figure A.5. The maximum temperature difference between the surface and the centre is 0.7°C. This corresponds to an average gradient of 5°C/m along the length of the beam. Temperatures were monitored across the beam width, as well as along the beam length. These results indicate that the inner half of the beam (the side closest to the other beam in the mould) has higher temperatures than the outer half.

Phase 1 for the frozen specimens is the same as normal curing. Thus, the temperature differences between the surface and centre thermocouples are of the same magnitude as that of the normally-cured specimens.

In phase 2, the temperature difference gradually increases. The maximum temperature difference ranges from 2.5 to 6°C depending on mix and curing conditions.

These differences correspond to a 15 to 40°C/m average gradient along the beam length.

In phase 4, the temperature difference reaches a maximum between 2 and 5°C (15 and 35°C/m gradient). As the temperature of the concrete stabilizes at the ambient cold room temperature, all temperature differences in the specimens disappear.

5.3. Effect of Mix Type

Figure 5.3 shows the temperatures monitored by the centre thermocouple in beam specimens cast from each of the four mix types, and frozen under the D-F8 condition. The same temperature pattern is shown by all four mixes. The small differences between curves can be explained largely by the varying temperatures at the end of phase 1 (placement in cold room). For example, the CON-A mix has a higher temperature at placement in the cold room than the other mixes. It has a higher cement content than the CON-N mix and no fly ash, and thus it initially has a greater heat of hydration than the other concretes. This highest initial temperature combined with a continuing greater heat of hydration means the CON-A mix is the slowest to cool.

As far as the temperature differences within the specimens are concerned, the graphs in Appendix A show that the FA-N mix has the largest differences and FA-A has the smallest differences.

5.3.5 Effect of Soaking

Soaking, prior to placement in the cold room, has the effect of lowering the prefreezing temperature of the concrete 5°C from that of a normally-cured concrete.

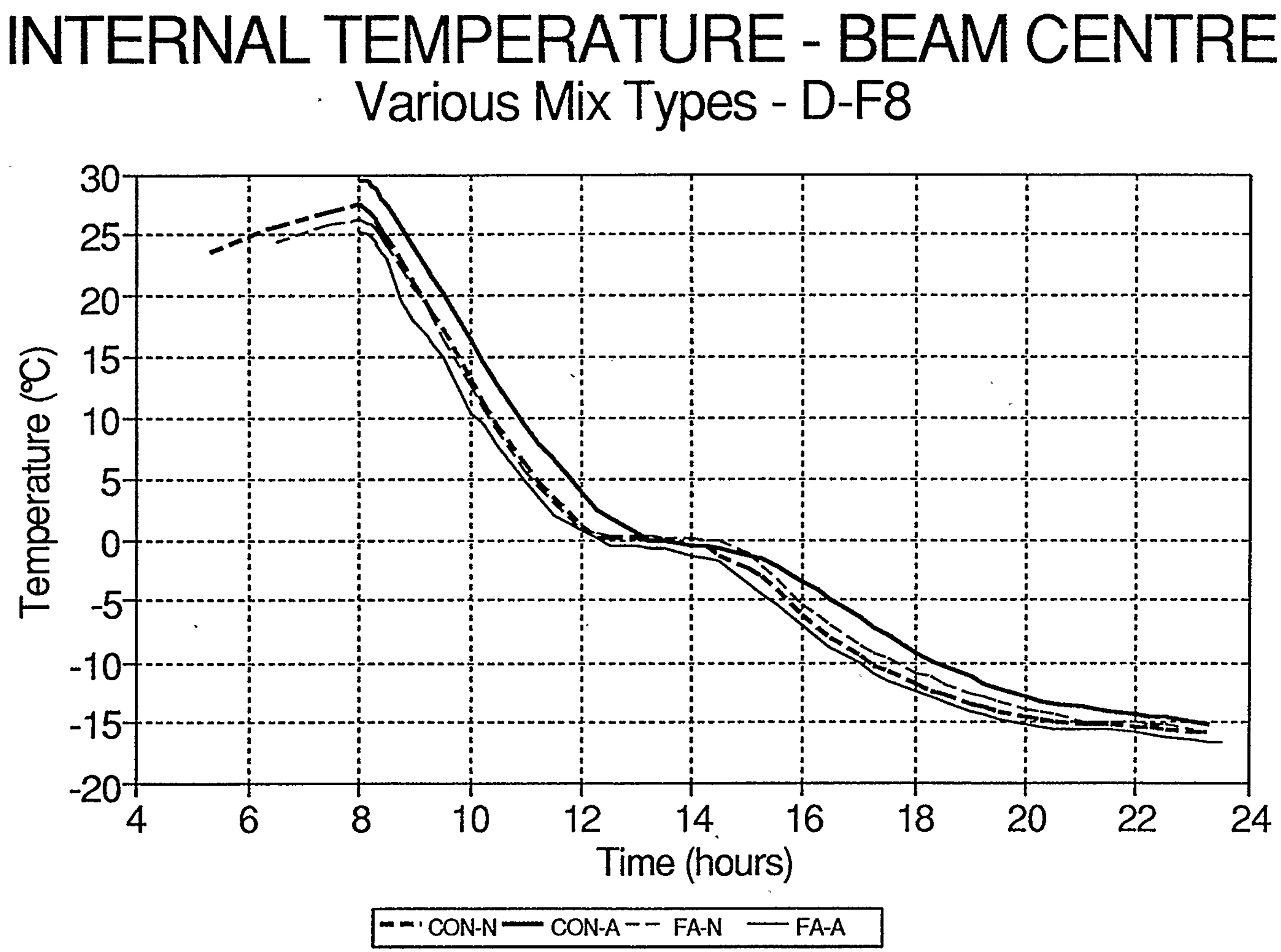


FIGURE 5.3: Effect of Mix Type on Beam Centre Temperature

Because they are at a lower temperature when placed in the cold room, the soaked specimens have slightly lower temperatures than the comparable dry specimens at the same age throughout cooling. The basic temperature profile, however, is the same. This behaviour is illustrated in Figure 5.4. It contains the centre thermocouple temperatures from FA-N beam specimens cured under various conditions.

The graphs in Appendix A show that soaking does not affect the temperature differences within the concrete or the length of time the temperature remained at 0°C.

5.3.6 Effect of Age at Freezing

Temperature monitoring was carried out on FA-N beam specimens which were placed in the cold room at 8 hours, and 24 hours, from mixing. The results from the centre and surface thermocouples are given in Figure 5.5. In order to have a comparable time scale, "Time" has been adjusted to time elapsed from placement in the cold room. The surface temperatures from both specimens follow the same temperature profile. However, for the centre location, the specimen frozen at 8 hours remains at 0°C for longer (2 hours) than the specimen frozen at 24 hours. Cement hydration has proceeded further in the 24 hour specimen and there is considerably less "free water" to freeze. Because of the delay at 0°C, the specimen frozen at 8 hours reaches the cold room ambient temperature slightly more slowly than the other specimen.

5.3.7 Effect of Specimen Type

As indicated in Section 5.3.1, cylinder specimens reach 0°C and -20°C more quickly than beam specimens. The size of the cylinders is smaller than the beams

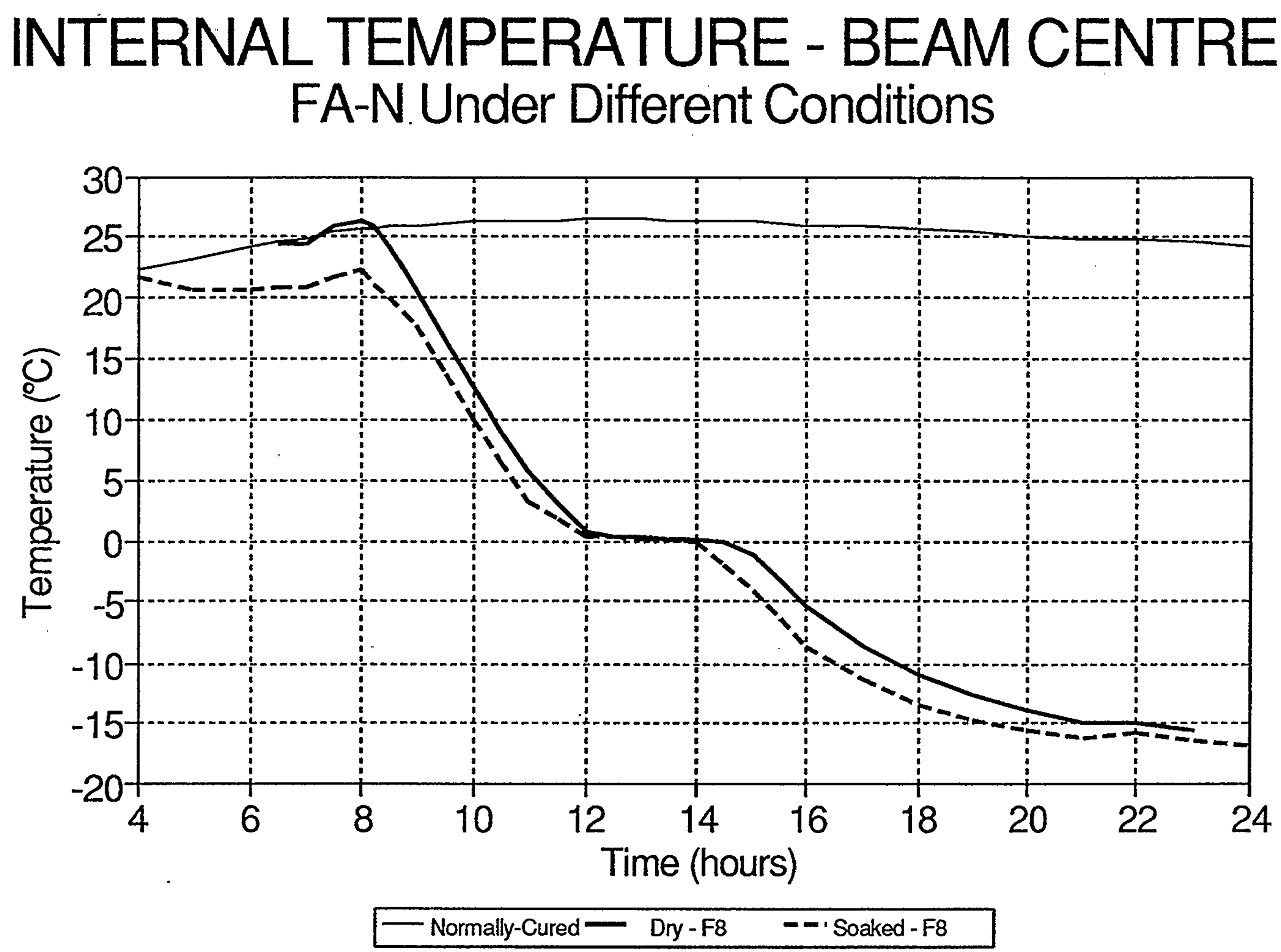


FIGURE 5.4: Effect of Soaking on Beam Centre Temperature

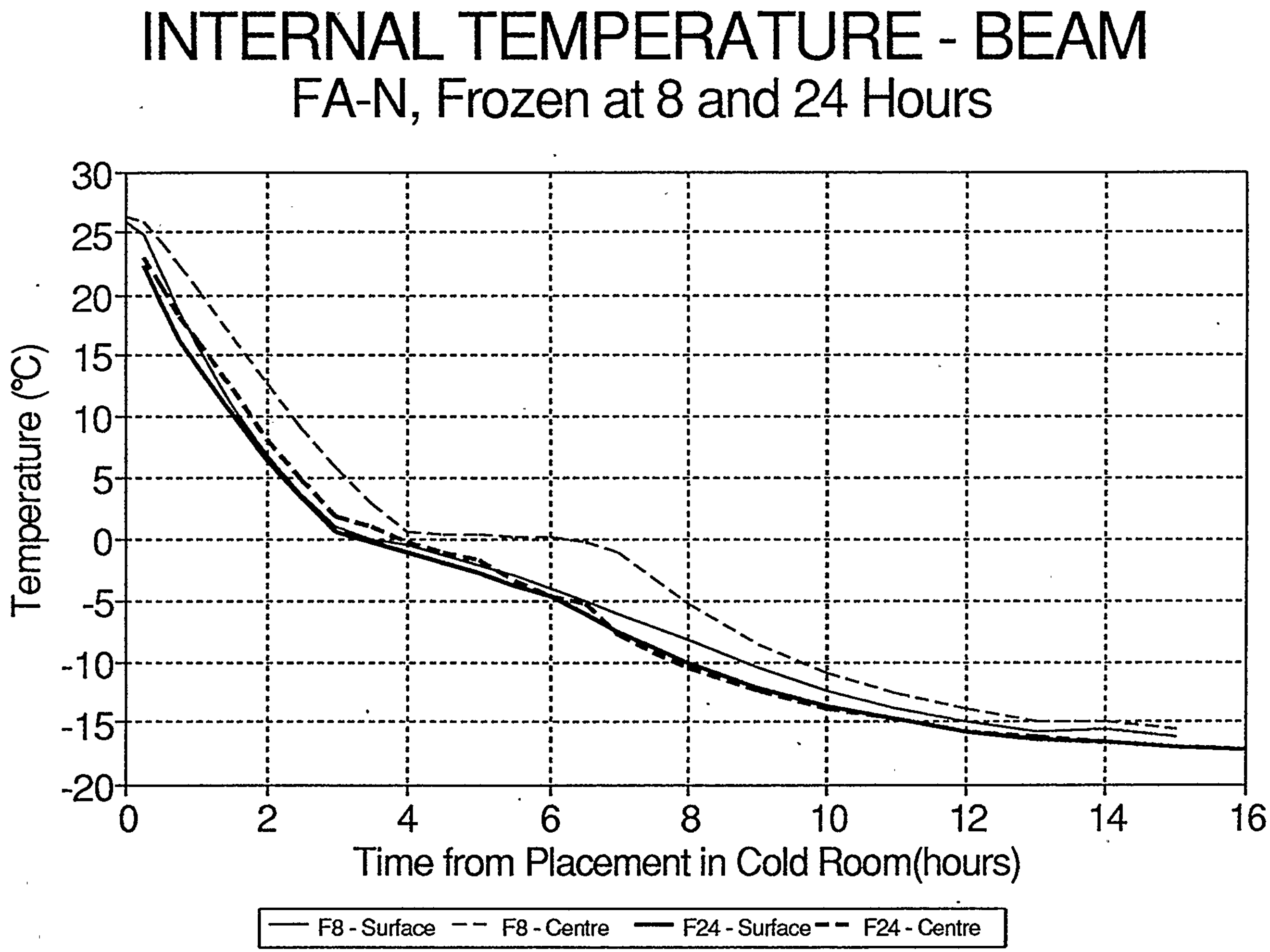


FIGURE 5.5: Effect of Age at Freezing on Beam Temperatures

(cylinder volume is 40% of beam volume) — therefore total heat of hydration is less. The temperature differences, which do not exceed a maximum of 1.4°C across the cylinder, are less than the differences present in the beam specimens. These differences correspond to gradients which are of the same magnitude as those along the beam, but less than those across the beam.

5.4 Point-Count Tests

Using the modified point-count test method, the paste content, entrapped air content, entrained air content and air-void spacing factor have been determined for the hardened concrete. These results are given in Table 5.3.

The total air contents are higher than those measured in the fresh concrete by the air meter test. These two methods do not typically give the same air content *[ASTM, 1989c]*. The entrapped air content is different as the specimens were consolidated differently. The fresh concrete sample was rodded and prepared immediately after mixing when workability is the highest. However, the cylinder, from which the point-count specimen was sliced, was vibrated and prepared midway through the casting of specimens when workability had decreased.

According to CSA *[1990]*, air-entrained concrete has a satisfactory air-void system if the average spacing factor is less than 0.23 mm, with no single test greater than 0.26 mm. The fly ash mix (FA-A) meets this criterion, but not the control mix (CON-A).

TABLE 5.3: Point-Count Test Results

Quantity	CON-N	CON-A	FA-N	FA-A
Paste Content	30.3%	32.6%	29.3%	28.8%
Air Contents:				
Entrapped	3.9%	2.5%	4.8%	4.6%
Entrained	0.0%	5.0%	0.0%	4.6%
Total	3.9%	7.4%	4.8%	9.2%
Air-void Spacing Factor (1)	0.57	0.26	0.41	0.18

Notes:
1. The air-void spacing factor is calculated based on the total air content.

5.5 Statistical Information for Strength Testing

The results of the population statistics tests will be discussed before the strength testing results. These population statistics are used to estimate the accuracy of the results.

The results from any batch of concrete will have a certain degree of variability. This "batch" variability is due to many factors including:

1. incomplete mixing of the concrete materials in the mixer,
2. inconsistent consolidation when casting specimens, and
3. variations in the testing procedures such as rate of loading and positioning of the specimen in the test apparatus.

There are additional factors which contribute to the "batch-to-batch" variability.

To get an estimate of the "batch" variability, a large number of specimens must be tested and the standard deviation calculated. Teychenne et al *[1975]* claim "it is generally accepted that the variation in concrete strengths follows the normal distribution and that at a given level of control the standard deviation increases as the specified characteristic strength increases up to a particular level and is independent of the specified strength above this level. Standard deviation being independent of the specified characteristic strength above 20 MPa." The concrete used in this research is above that strength level, and thus it should be appropriate to find a standard deviation that is applicable for all mixes and testing ages.

For flexural strength, two sets of 20 specimens were tested with the following results:

Mix, Curing Condition, Testing Age	Standard Deviation
CON-A, D-N, 28-day	0.256 MPa
FA-N, S-F8, 3-day	0.206 MPa

Using the standard F-statistic and hypothesis testing *[Walpole and Myers, 1985]*, at a 0.10 level of significance, one cannot reject the null hypothesis, that the standard deviations are equal.

A confidence interval for the flexural strength results can be determined. This confidence interval is calculated using:

i. the average of the two standard deviations (0.231 MPa),

ii. the average of 3 specimens as the strength estimate, and

iii. a 90% confidence level.

The interval is:

(estimate of strength - 0.22 MPa) < true strength < (estimate of strength + 0.22 MPa)

An important aspect of examining the strength testing results is to compare the frozen and normally-cured specimens by expressing the frozen strength as a percentage of the normally-cured strength. Hypothesis testing can be used to determine if there is a significant difference between the frozen and normally-cured strengths. At a 0.10 level of significance, and at a typical normally-cured strength of 6.0 MPa, there is insufficient evidence to reject the null hypothesis that the frozen strength is equal to the normally-cured strength when the percentage value is between 95% and 105%. This illustrates that small differences in strength gains cannot be considered statistically significant. The 0.10 level of significance means that the probability of a Type I error (i.e. claiming strengths are not equal when they are) is 10%. In this instance, the probability of a Type II error (i.e. claiming strengths are equal when they are not) is 6% if the percentage value is actually 90%.

For compressive strength, two sets of 20 specimens were tested with the following results:

Mix, Curing Condition, Testing Age	Standard Deviation
CON-N, D-N, 28-day	1.82 MPa
CON-N, D-N, 3-day	1.20 MPa

Using the F-statistic, at a 0.10 level of significance, one must reject that the standard deviations are equal. The 1.20 MPa value should be used for the short-term (3- and 7-day) test results and the 1.82 MPa value should be used for the long-term (28-day and 90-day) test results.

To remove concerns that other mix types or differences in curing (i.e. soaking or freezing) could affect the standard deviation, the standard deviations from each set of tests were calculated. For example, for the 28-day tests these ranged from 0.72 to 3.26 MPa with a pooled standard deviation of 1.78 MPa. It is appropriate to use 1.82 MPa as the estimate for standard deviation of the compressive strength test results at this age.

Confidence intervals for the compressive strength results can be calculated. For the 28-day testing age, the confidence interval is calculated using:

i. a standard deviation of 1.82 MPa,

ii. the average of 3 specimens as the strength estimate, and

iii. a 90% confidence level.

The interval is:

(estimate of strength - 1.7 MPa) < true strength < (estimate of strength + 1.7 MPa)

As with flexural strength results, hypothesis testing can be used to see if there is a significant difference between frozen and normally-cured specimens. At a 0.10 level of significance, at a typical 28-day strength of 35 MPa, one must accept the null hypothesis that the frozen strength is equal to the normally-cured strength if the percentage value is between 93% and 107%. The probability of a Type I error (i.e claiming strengths are not equal when they are equal) is 10% at the 0.10 level of significance. The probability of a Type II error (i.e. claiming strengths are equal when they are not) is 24% if the true percentage value is 90% or 3% if the true percentage value is 85%.

The variability and statistical significance associated with the testing results should be kept in mind when studying the following strength results.

5.6 Flexural Strength Results

5.6.1 Testing Inside versus Outside of Beam

As explained in the experimental procedures section (Section 4.3), the beam moulds contained two beams cast side-by-side. The beams stayed in these moulds until 24 hours after removal from the cold room. Thus the flexural strength results may be influenced by whether the inside (the side parallel to the other beam) or the outside of the beam was placed in tension.

To investigate this possibility, 20 beam specimens were cast from the FA-N mix and frozen in the soaked condition at 8 hours. At 3 days after placement in the fog room, 10 specimens were tested for flexural strength with the inside in tension, and 10 specimen were tested with the outside in tension. The results are:

	Average Strength	Standard Deviation
Inside in Tension	3.07 MPa	0.213 MPa
Outside in Tension	3.24 MPa	0.199 MPa

Using hypothesis testing, at a 90% confidence level, one can conclude that there is a significant difference between the strengths.

In all of the flexural strength testing results reported hereafter, the outside of the beam was placed in tension. This eliminates one source of variation in the testing results.

5.6.2 Flexural Strength Results

The flexural strength results are summarized in Table 5.4. For each mix type and curing condition, both normally-cured and frozen strengths are given. With FA-N, the D-F8* results are for specimens that were frozen at -40°C rather than the standard -20°C. Strength values were measured at 3, 7, 28, and 90 days from the start of fog-room curing. The percentage value is the frozen strength expressed as a percentage of the normally-cured strength.

The upper graphs in Figures 5.6 to 5.9 display the strength gain information. Within each graph, the results from the different curing conditions are presented. The right bar of each pair is the normally-cured strength and the left bar is the frozen strength.

The lower graph in figures 5.6 to 5.9 displays the percentage value information.

For any curing condition, both the normally-cured and frozen sets of specimens were:

1. cast from the same batch of concrete,
2. cured under identical conditions except for 48 hours in the cold room for the frozen specimens, and
3. were tested at the same age under identical conditions.

Thus the effects of batch-to-batch variability have been removed. When comparing the two sets of specimens, any difference in strength gain is due solely to the effects of the freezing period.

TABLE 5.4: FLEXURAL STRENGTH RESULTS

1. All strengths given in MPa.
2. Percentage value is the frozen strength expressed as a percentage of normal strength.
3. Testing age in days from start of fog curing.

MIX: CON-N

Testing Age	D-F8			S-F8			D-F24		
	Normal	Frozen	Percentage	Normal	Frozen	Percentage	Normal	Frozen	Percentage
3	5.04	4.85	96.2%	5.03	4.48	89.1%	4.43	4.63	105%
7	5.18	5.10	98.5%	5.59	5.18	92.7%	4.91	5.32	108%
28	6.51	6.27	96.3%	6.42	6.15	95.8%	5.59	6.39	114%
90	6.85	6.72	98.1%	6.76	6.72	99.4%	6.08	6.51	107%

MIX: CON-A

Testing Age	D-F8			S-F8		
	Normal	Frozen	Percentage	Normal	Frozen	Percentage
3	4.64	4.25	91.6%	4.90	4.59	93.7%
7	4.94	4.48	90.7%	5.27	4.92	93.4%
28	5.50	5.35	97.3%	6.05	5.85	96.7%
90	-	-	-	6.13	6.36	108%

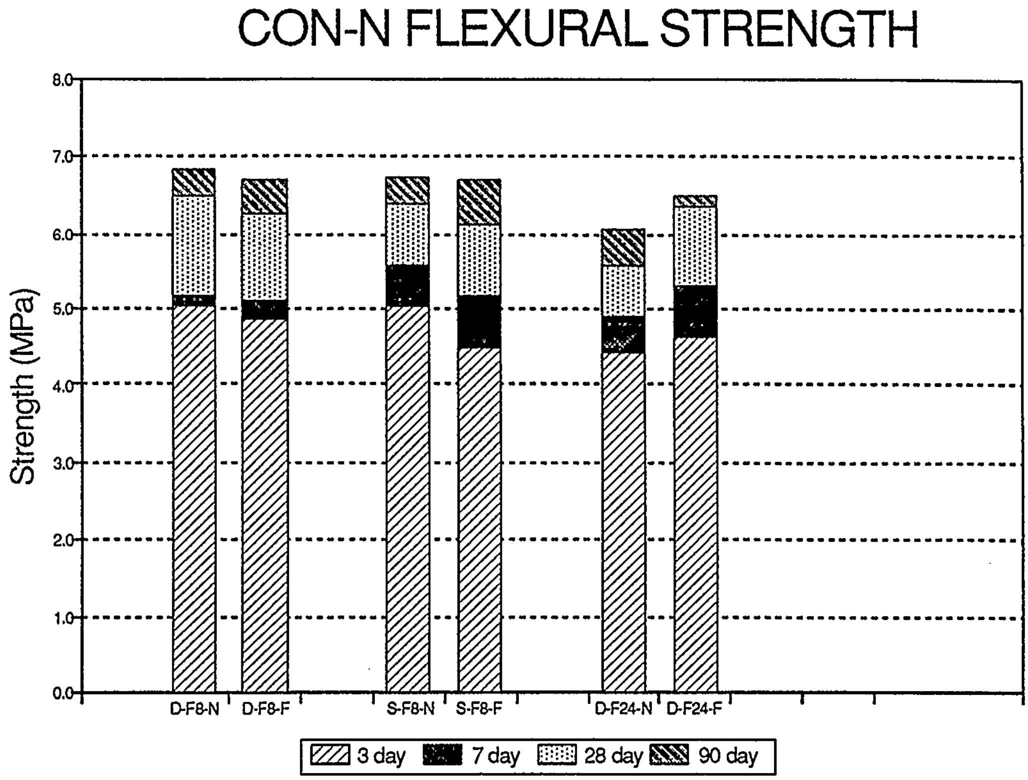
MIX: FA-N

Testing Age	D-F8 (1)			S-F8			D-F24			D-F8* (2)		
	Normal	Frozen	Percentage	Normal	Frozen	Percentage	Normal	Frozen	Percentage	Normal	Frozen	Percentage
3	4.47	3.57	79.9%	4.01	2.93	73.1%	4.22	3.93	93.1%	4.15	2.72	65.5%
7	5.03	4.18	83.1%	4.98	4.09	82.1%	4.63	4.70	102%	4.88	3.35	68.6%
28	6.02	5.20	86.4%	6.63	5.81	87.6%	6.59	6.18	93.8%	6.36	5.26	82.7%
90	7.53	7.04	93.5%	7.41	6.99	94.3%	7.08	7.54	107%	7.28	5.68	78.0%

1. 28-day strength measured at 21 days for D-F8.
2. Frozen at -40 C rather than -20 C.

MIX: FA-A

Testing Age	D-F8			S-F8		
	Normal	Frozen	Percentage	Normal	Frozen	Percentage
3	3.49	3.16	90.5%	4.32	4.15	96.1%
7	4.17	3.82	91.6%	5.02	4.91	97.8%
28	5.32	5.64	106%	6.43	6.42	99.8%
90	5.98	6.58	110%	7.55	6.92	91.7%



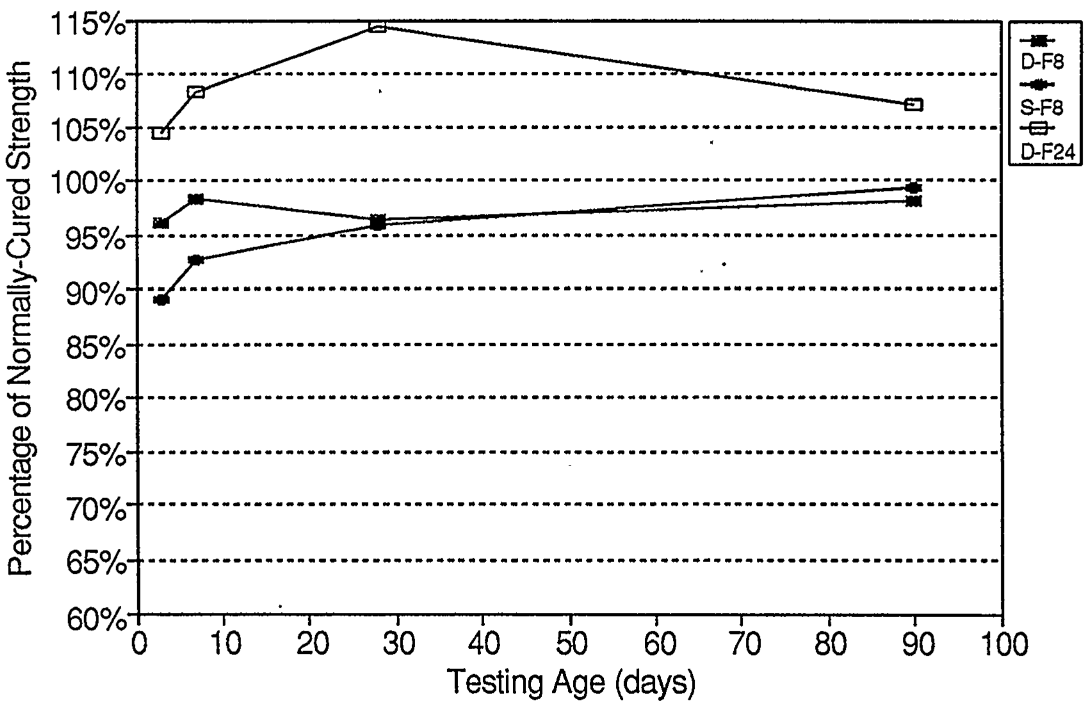


FIGURE 5.6: Fexural Strength Results for CON-N

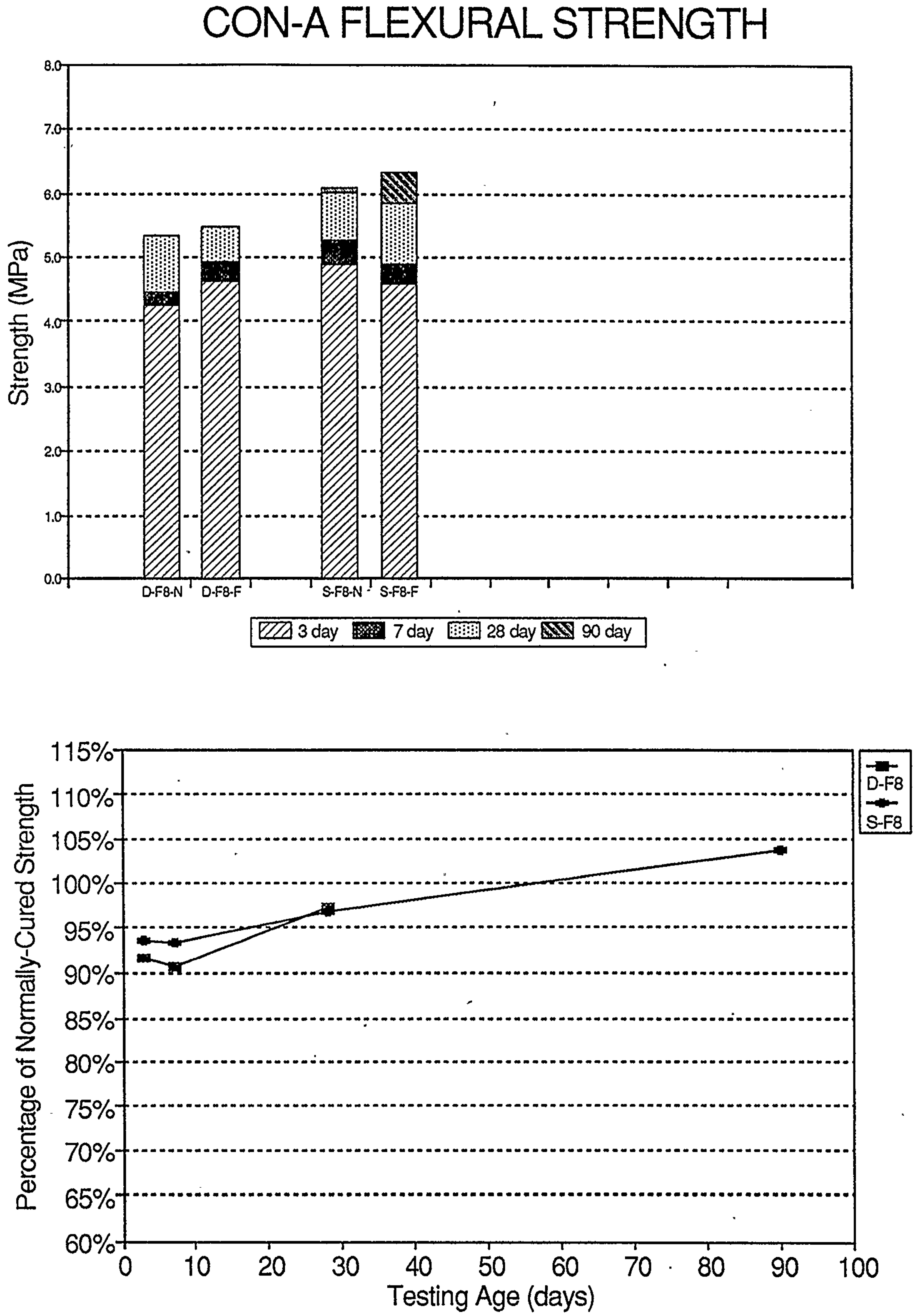
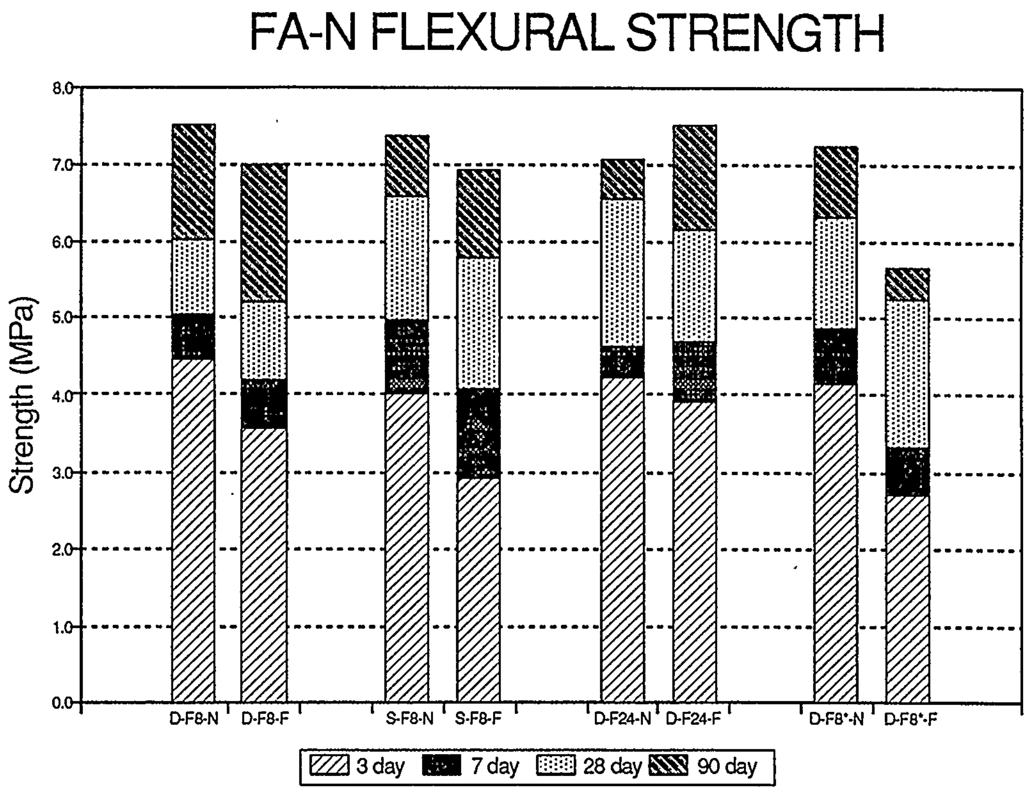


FIGURE 5.7: Flexural Strength Results for CON-A



Note: 28-day strength for D-F8 measured at 21 days.

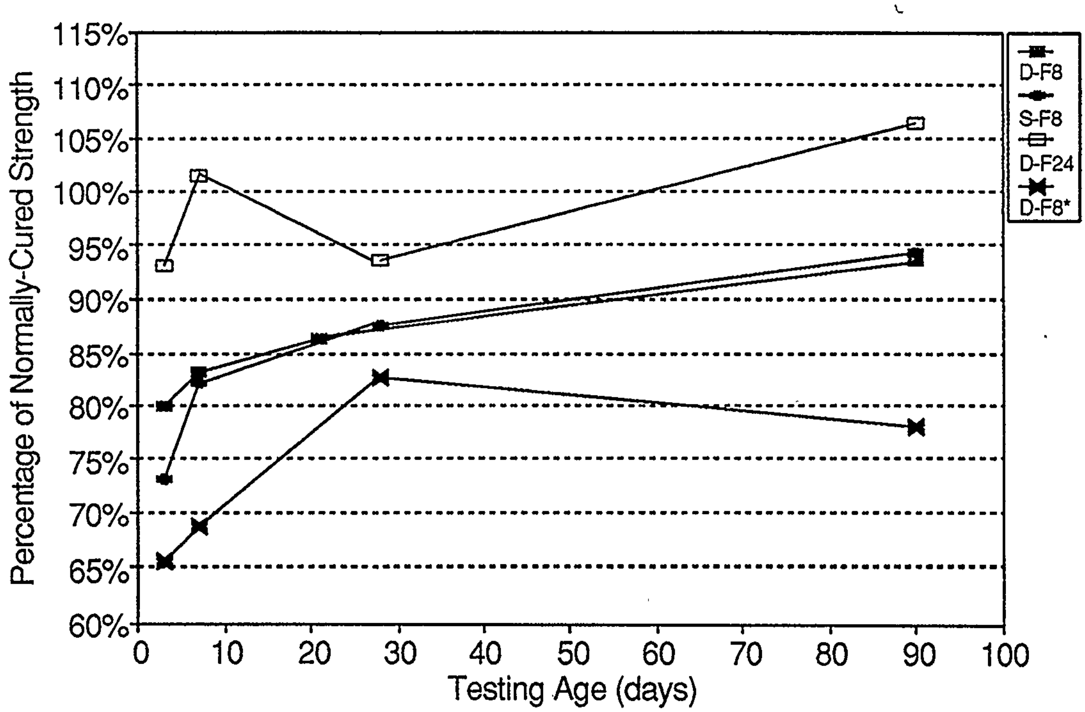


FIGURE 5.8: Flexural Strength Results for FA-N

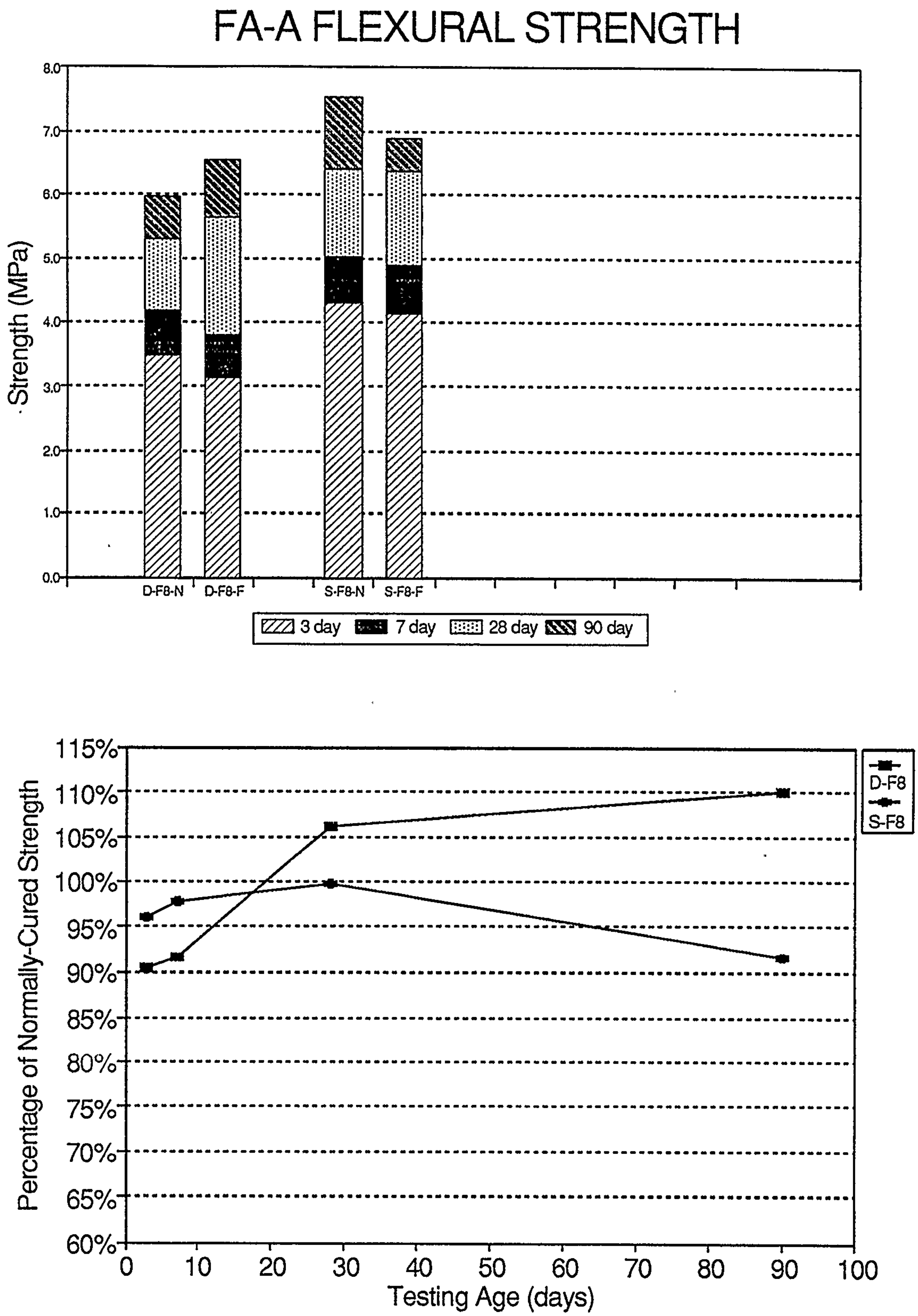


FIGURE 5.9: Flexural Strength Results for FA-A

5.6.3 Normally-cured Strength Gain Curves

The flexural strength gain curves for the normally-cured specimens exhibit typical behaviour for plain and fly ash concretes. Although they have the same ultimate strength, the fly ash concretes initially gain strength slower.

The difficulty in obtaining the proper air-entrainment in one of the FA-A batches explains the difference between the normally-cured strengths from the D-F8 and S-F8 curing conditions as shown in Figure 5.9. The lower air content in the S-F8 batch results in a higher strength.

The soaking regime does not have a significant effect on the strength development.

5.6.4 Effect of Testing Parameters during Freezing

5.6.4.1 Effect of Testing Age

At 3 days, the frozen flexural strength is usually significantly lower than the normally-cured strength. But as the testing age increases, this difference decreases. With the standard -20°C curing regime, the flexural strength was never less than 92% of the normal strength at 90 days. In fact, in some cases the frozen concrete is stronger than the normally-cured concrete at the later test ages.

5.6.4.2 Effect of Replacing Cement with Fly Ash

FA-N (Figure 5.8) shows slightly greater flexural strength reduction (between 5 and 15% depending on the testing age) than CON-N (Figure 5.6). This difference is significant at early testing ages.

5.6.4.3 Effect of Age at Freezing

CON-N and FA-N mixes had specimens frozen at both 8 hours and 24 hours from mixing. With both mixes and at all testing ages, the 24 hour frozen/normal percentage value is usually significantly greater than the 8 hour value. The concrete is less affected by freezing as the age at freezing increases.

The specimens frozen at 24 hours have long-term strengths that exceed the normally-cured strengths. Already at the early testing ages, there is sufficient time for the frozen strength to match or exceed the normally-cured strength.

5.6.4.4 Effect of Soaking

The effects of freezing on flexural strength are not altered by whether the specimens are frozen in the dry or soaked condition. This is evident in Figures 5.6 to 5.9.

5.6.4.5 Effect of Air-Entrainment

With the plain concretes (Figures 5.6 and 5.7), there is no difference between the non air-entrained and air-entrained mixes. With the fly ash concretes (Figures 5.8 and 5.9), the non air-entrained mix has a permanent strength loss due to freezing, whereas the air-entrained mix has a permanent strength gain.

5.6.4.6 Effect of Freezing Regime

The FA-N mix (Figure 5.8) was subjected to two different freezing regimes in the D-F8 mode. The temperature at placement in the cold room was -20°C in one case and -40°C in the other case. With the -40°C freezing, there is a greater strength loss at early ages and a significant strength loss of 22% in the long-term. This is the largest strength loss of any of the flexural testing situations.

5.7 Compressive Strength Results

Table 5.5 and Figures 5.10 to 5.13 summarize the compressive strength testing results. Again, the upper graph contains the strength gain information and the lower graph is the frozen strength expressed as a percentage of the normally-cured strength.

5.7.1 Normally-cured Strength Gain Curves

The compressive strength gain curves for the plain and fly ash mixes show typical strength gain patterns. Although they have the same ultimate strength, the fly ash mixes gain strength at a slower rate.

In Figure 5.12, the FA-N batch used for the S-F8 curing condition appears to have a lower strength gain than the other batches. Results from an earlier stage of this research indicate the soaking regime would not account for this difference. This batch did have a higher slump than the other batches, but this is felt to be due to a testing abnormality, not because the water content was inadvertently higher. (Note that flexural strength for this batch was not reduced.)

The CON-N, D-F8 strength results given in Figure 5.10, at 3 and 7 days, were influenced by a problem with the bearing face of the top bearing block deviating from a plane surface. Whilst this problem has affected the strength values measured, it does not affect the percentage value as both sets of specimens were tested under identical conditions.

The soaking regime does not significantly affect the strength development.

TABLE 5.5: COMPRESSIVE STRENGTH RESULTS

1. All strengths given in MPa.
2. Percentage value is the frozen strength expressed as a percentage of normal strength.
3. Testing age in days from start of fog curing.

MIX: CON-N

Testing Age	D-F8			S-F8			D-17			D-F24		
	Normal	Frozen	Percentage	Normal	Frozen	Percentage	Normal	Frozen	Percentage	Normal	Frozen	Percentage
3	26.9	22.4	83.4%	24.6	18.4	74.6%	25.8	26.6	103%	25.8	26.1	101%
7	25.2	23.0	91.6%	30.2	27.6	91.3%	26.6	25.3	95.2%	26.6	26.4	99.4%
28	38.2	37.9	99.4%	34.0	35.6	105%	32.7	36.8	113%	32.7	32.6	99.8%
90	44.8	41.1	91.7%	43.3	36.5	84.3%	37.4	35.6	95.1%	37.4	40.6	109%

MIX: CON-A

Testing Age	D-F8			S-F8		
	Normal	Frozen	Percentage	Normal	Frozen	Percentage
3	23.6	22.0	93.2%	24.1	22.4	93.0%
7	26.9	26.3	98.0%	25.3	26.2	104%
28	32.5	33.1	102%	38.2	37.8	99.0%
90	40.0	42.9	107%	41.2	43.3	105%

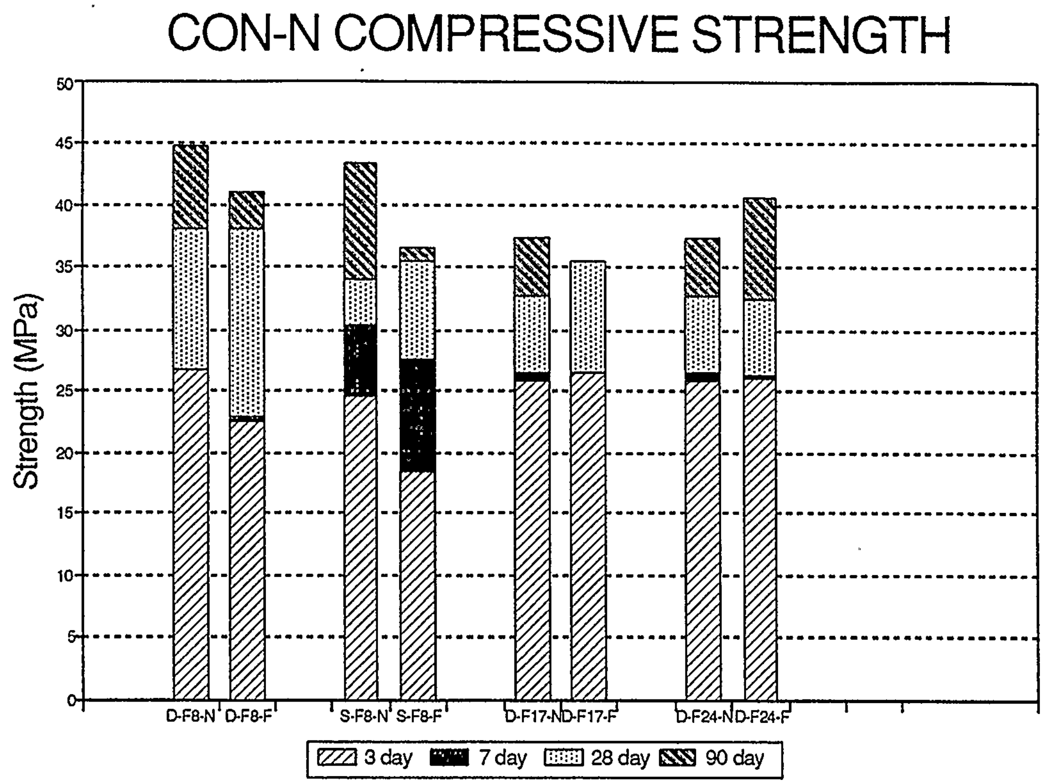
MIX: FA-N

Testing Age	D-F8 (1)			S-F8			D-17			D-F24			D-F8* (2)		
	Normal	Frozen	Percentage	Normal	Frozen	Percentage	Normal	Frozen	Percentage	Normal	Frozen	Percentage	Normal	Frozen	Percentage
3	21.0	15.1	72.0%	17.4	10.8	62.2%	22.4	19.3	85.8%	22.4	21.0	93.4%	17.3	10.7	61.9%
7	26.6	19.1	71.9%	19.0	13.6	71.7%	23.8	25.3	106%	23.8	24.8	104%	22.9	14.9	64.9%
28	36.7	30.3	82.8%	31.9	24.4	76.5%	38.0	37.2	98.0%	38.0	38.9	103%	28.5	22.4	78.5%
90	46.8	40.8	87.1%	34.8	28.0	80.4%	43.6	44.6	102%	43.6	48.1	110%	41.9	30.5	72.7%

1. 28 day strength measured at 21 days for D-F8.
2. Frozen at -40 C rather than -20 C.

MIX: FA-A

Testing Age	D-F8		
	Normal	Frozen	Percentage
3	17.1	14.4	84.4%
7	19.4	18.8	97.2%
28	27.1	29.1	108%
90	30.8	35.3	115%



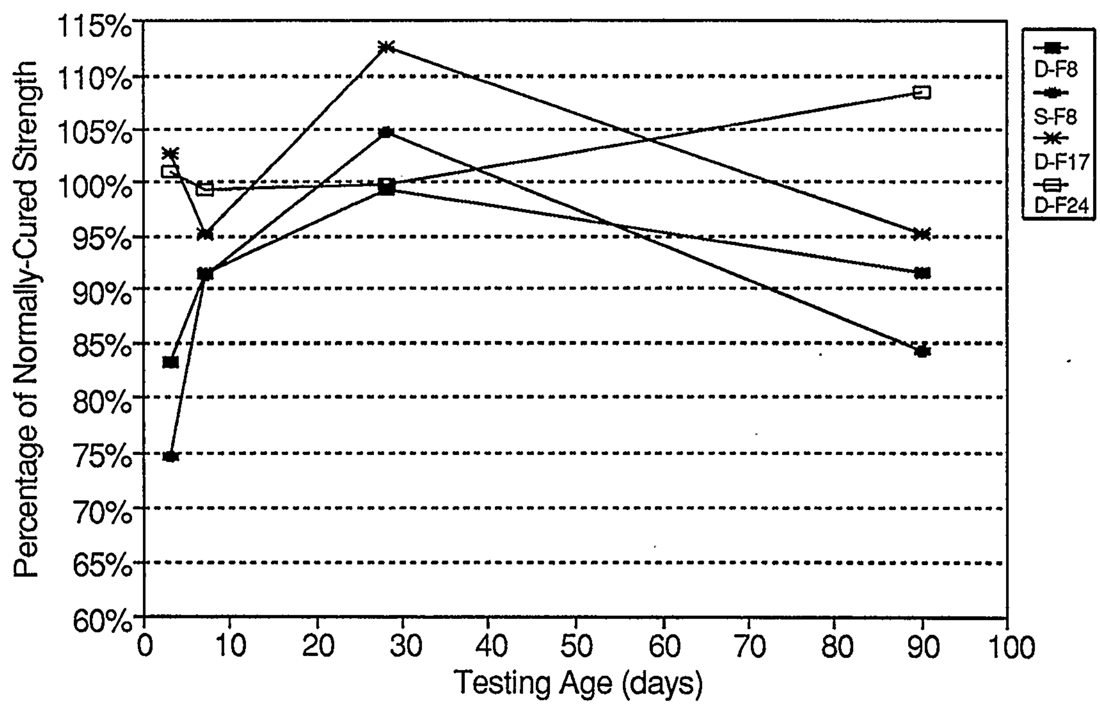
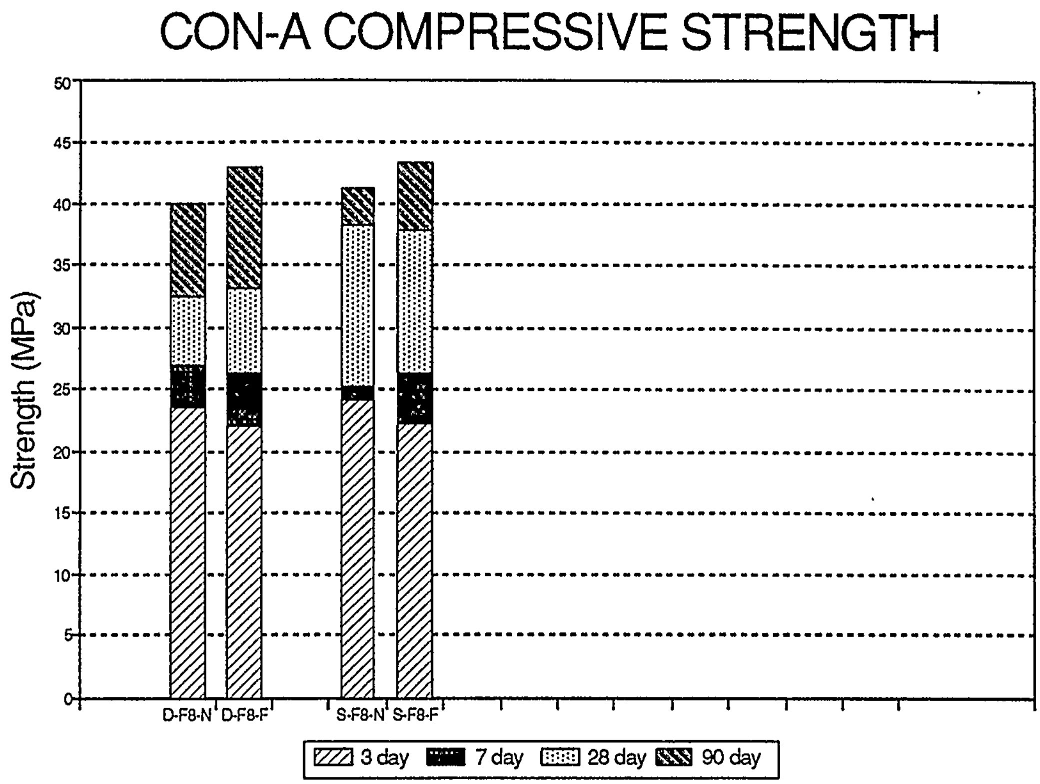


FIGURE 5.10: Compressive Strength Results for CON-N



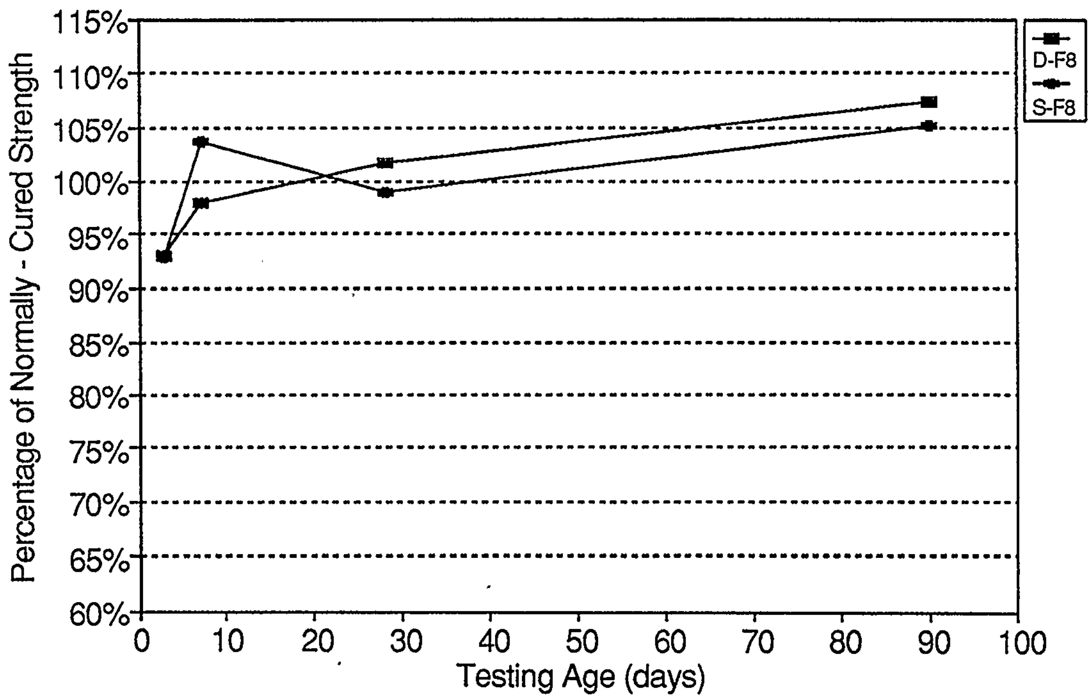
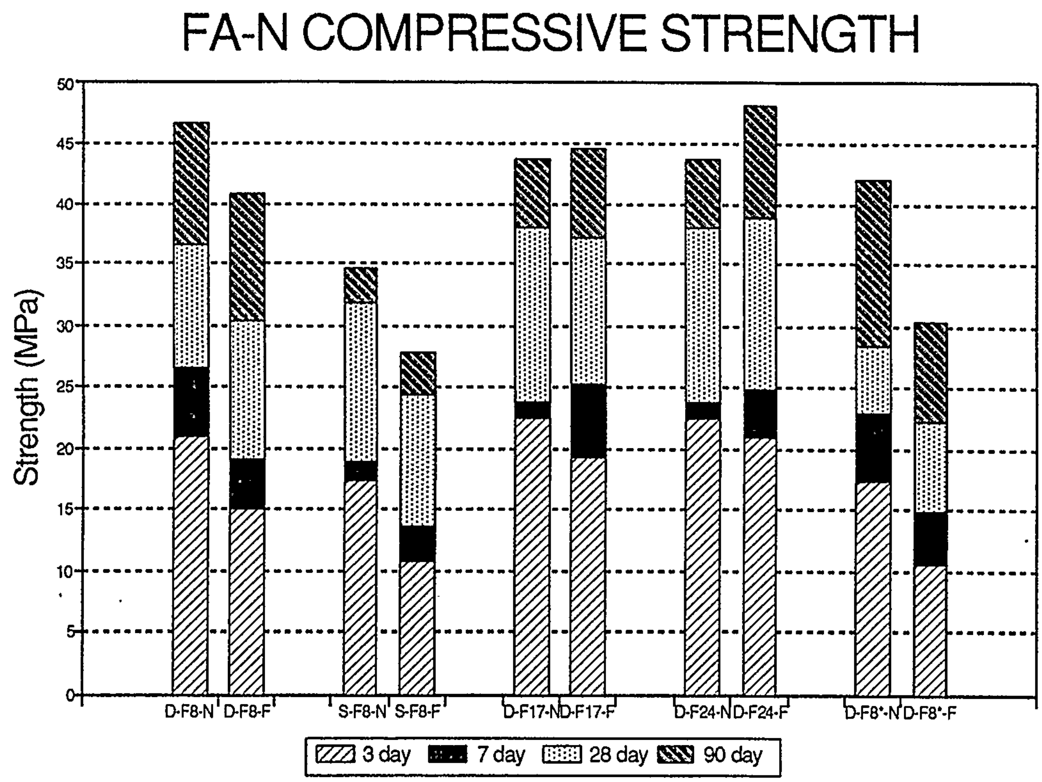


FIGURE 5.11: Compressive Strength Results for CON-A



Note: 28-day strength for D-F8 measured at 21 days.

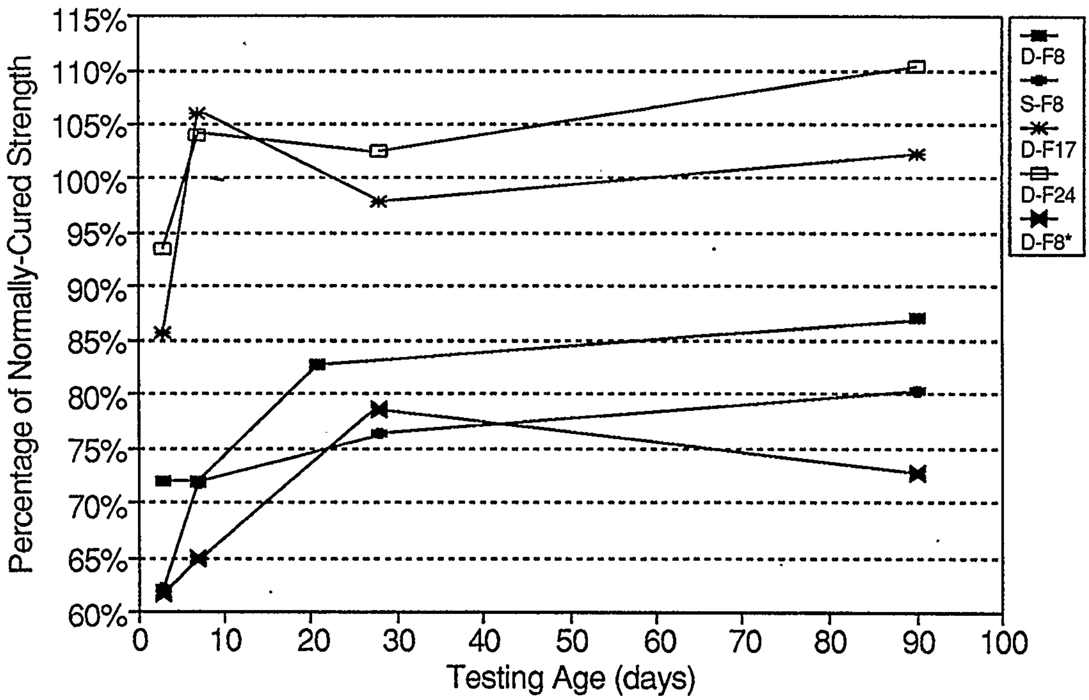
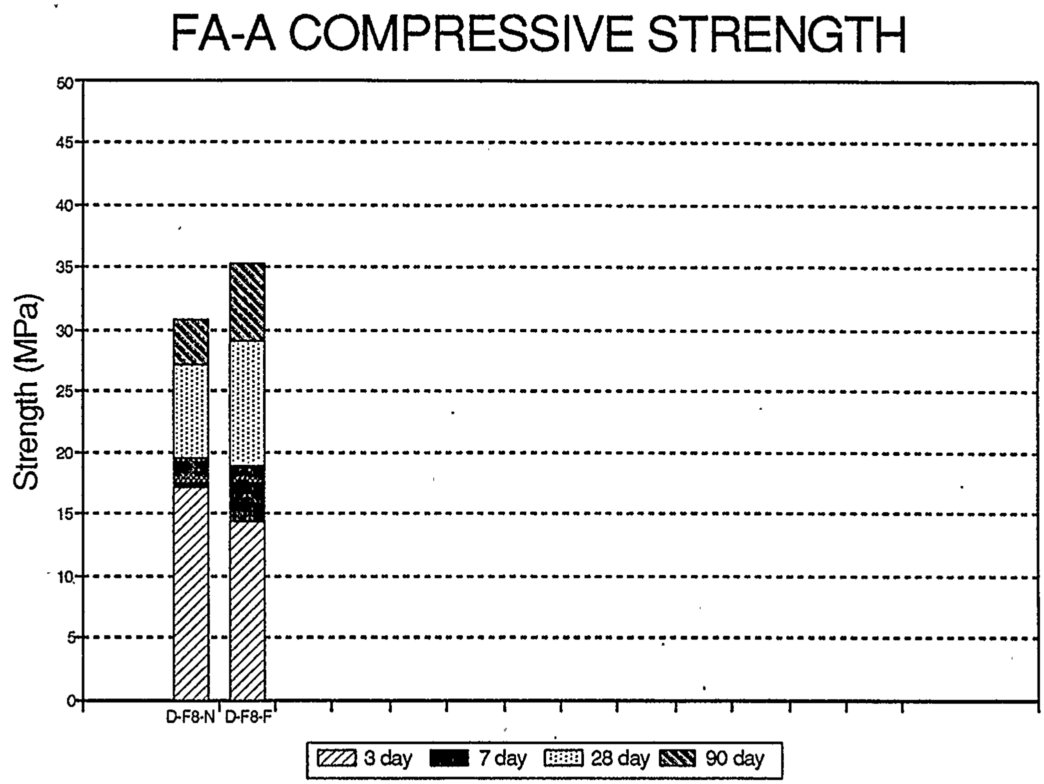


FIGURE 5.12: Compressive Strength Results for FA-N



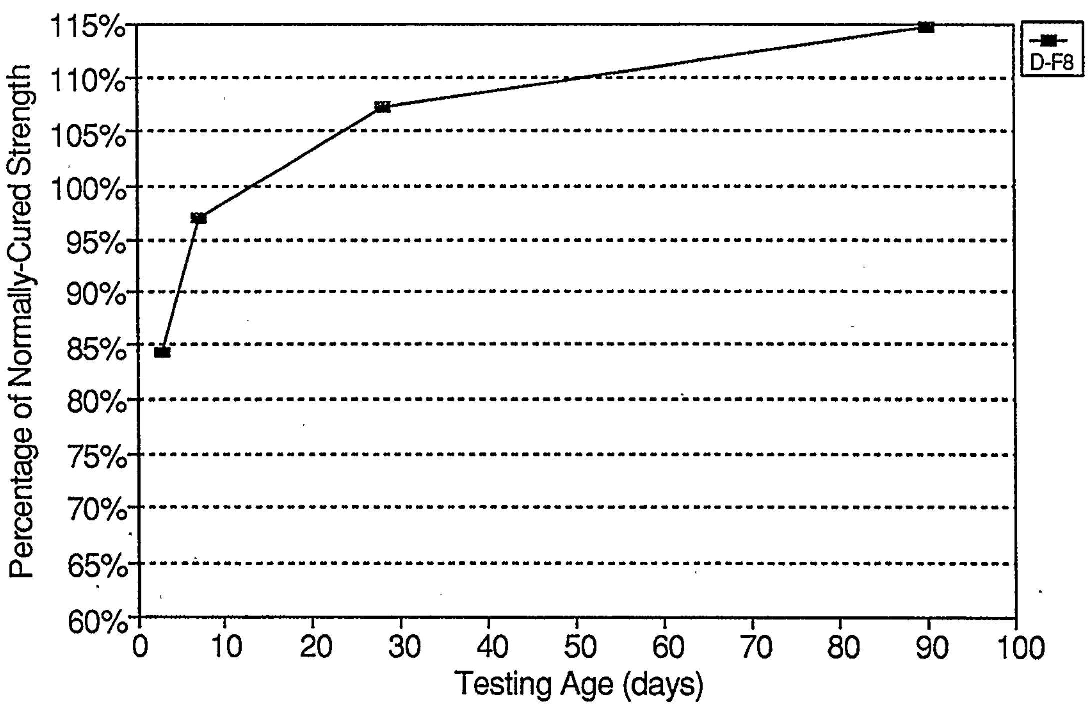


FIGURE 5.13: Compressive Strength Results for FA-A

5.7.2 Effect of Testing Parameters during Freezing

5.7.2.1 Effect of Testing Age

In general, at early testing ages, the compressive strength of the frozen concrete is significantly lower than the normally-cured concrete strength. However, in the long-term, this strength loss is reduced or eliminated. In some instances, the long-term strength of the frozen specimens is significantly higher than that of the normally-cured specimens.

The CON-N mix (Figure 5.10) is the exception to this general trend. The 90-day percentage values are significantly lower than the 28-day values for some of the batches. There is not a readily apparent explanation for this behaviour.

5.7.2.2 Effect of Fly Ash Replacement

With the non air-entrained mixes (Figures 5.10 and 5.12), there is a greater strength loss in the fly ash mix when frozen at 8 hours, but not when frozen at 17 and 24 hours. FA-N frozen at 8 hours is the only mix to suffer permanent strength loss due to freezing. With the air-entrained mixes (Figures 5.11 and 5.13), the fly ash and plain mixes follow basically the same behaviour.

5.7.2.3 Effect of Age at Freezing

The CON-N and FA-N mixes were frozen at 8, 17 and 24 hours. Figures 5.10 and 5.12 show, in general, increasing the age at freezing decreases the compressive strength loss due to freezing. There is not a significant difference between the behaviour of the 17 and 24 hour frozen concrete. In the long-term, the 24 hour frozen concrete was stronger than the normally-cured concrete.

5.7.2.4 Effect of Freezing Regime

The FA-N mix was frozen both using the standard -20°C curing regime and another where the initial cold room temperature was -40°C. Freezing at the lower temperature increases the freezing damage (15% more at 90 days) as shown in Figure 5.12.

5.7.2.5 Effect of Soaking

There is no significant difference between the effect freezing has on soaked versus dry specimens. On average, the soaked specimens have a 2.8% greater strength loss. This is evident in Figures 5.10 to 5.12.

5.7.2.6 Effect of Air-Entrainment

With the fly ash mixes, the non air-entrained mix (Figure 5.12) has significantly lower frozen/normal percentage values than the air-entrained mix (Figure 5.13). The non air-entrained mix has a permanent strength loss due to freezing, whereas the air-entrained mix has a permanent strength gain due to freezing.

5.8 Comparison of Flexural and Compressive Strength Results

The flexural and compressive strength results basically show the same trends. If there is no permanent strength loss, the percentage curves follow the same pattern and normally do not deviate more than 5% from each other. However, with the FA-N, mixes which do show a permanent strength loss, the compressive strength loss is 8% greater on average than the flexural strength loss.

5.9 Comparison with Preliminary Project

Some of the compressive strength testing in this research project was similar to Day's earlier project *[Day, 1991]*. Although the mix designs were slightly different (most significantly, a 28-day target strength of 40 MPa and a fly ash replacement level of 30% were used in the earlier project), the curing and testing procedures were the same. The two sets of results can be compared on the basis of the frozen strength as a percentage of normal strength.

Table 5.6 contains this comparison. The results never differ by more than 10.0% and the results from this project were on average 2.0% higher. This is an insignificant difference considering the accuracy of the results and thus this comparison gives confidence in the reproducibility of the testing results.

TABLE 5.6: Comparison with Previous Project

Mix	Curing Condition	Testing Age (days)	Previous Results	Current Results
CON-N	D-F24	3	90.9%	93.2%
		7	94.1%	98.0%
		28	103%	102%
		90	104%	107%
CON-A	D-F8	3	106%	101%
		7	105%	99.4%
		28	109%	99.8%
		90	103%	109%
FA-N	D-F24	3	80.4%	84.4%
		7	87.2%	97.2%
		28	100%	108%
		90	111%	115%
FA-A	D-F8	3	94.6%	93.4%
		7	99.4%	104%
		28	102%	103%
		90	104%	110%

Notes:

1. D = dry, F# = age at freezing

5.10 Fracture Mode Analysis

After the flexural strengths of the beam specimens were tested, the resulting fracture surface was analyzed. The surfaces were given a fracture mode "rating" based on the proportion of failure occurring at the paste-aggregate interface versus failure through the aggregate. This rating was made using the following scale:

0 all bond failure at the paste-aggregate interface
↓
4 half the aggregate failed at the bond interface, half failed through the aggregate
↓
8 all failure through the aggregate

Graphs for the various mixes and curing conditions are given in Figures 5.14 to 5.18.

In all cases, there is more bond failure at the early (3- and 7-day) testing ages. As the concrete matures, there is more failure through the aggregate rather than at the paste interface.

A significant difference between the frozen and normally-cured specimens can be seen in some of the non air-entrained fly ash (FA-N) mixes. With the 8 hour freezing, under dry and soaked conditions (D-F8 and S-F8), the frozen specimens have a greater proportion of bond failure than the normally-cured specimens. This is also apparent with the FA-N specimen frozen at -40°C (D-F8*). Figure 5.19, containing photographs of the beam fracture surfaces tested at 90 days, clearly shows this difference. In Figure a, CON-N cured under D-F8, there is no difference in the fracture mode of the frozen and normally-cured specimens. However, Figures b,c, and d, corresponding to FA-N under D-F8, S-F8 and D-F8* conditions respectively, show a difference between

FA-N under D-F8, S-F8 and D-F8* conditions respectively, show a difference between the fracture surfaces. Smooth aggregate sockets, where paste-aggregate failure has occurred, are more plentiful in the frozen specimens.

This concludes the summary and discussion of the various testing results. The following chapter contains some general conclusions and recommendations based on the results presented here.

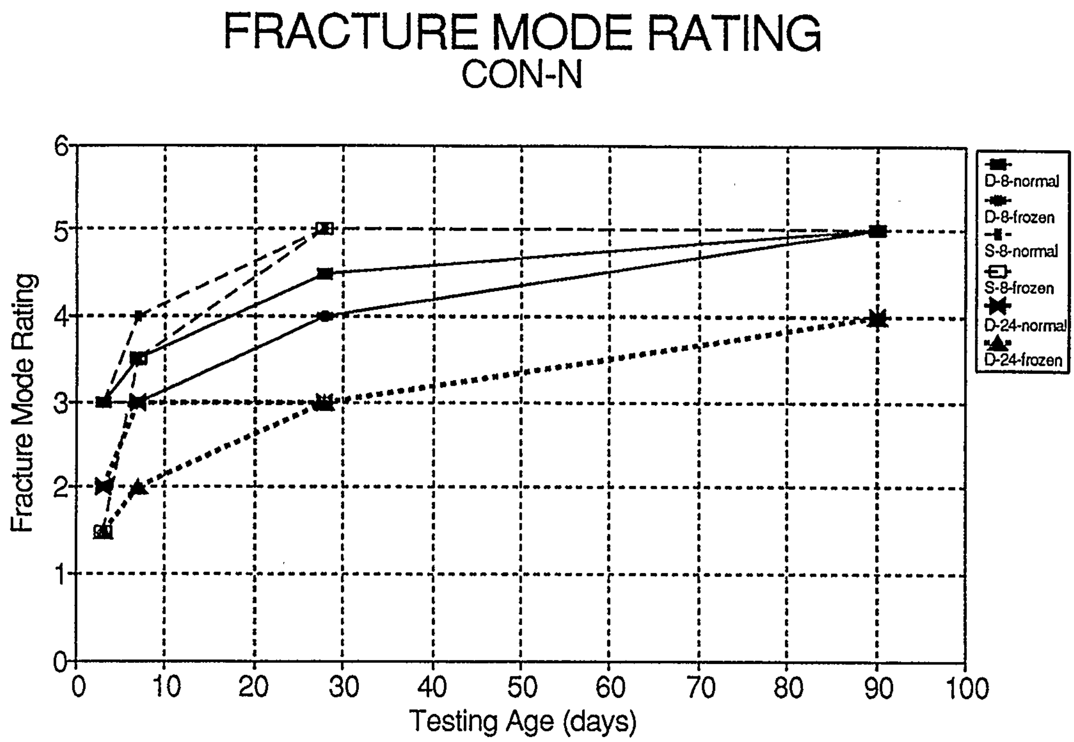


FIGURE 5.14: Fracture Mode Analysis for CON-N

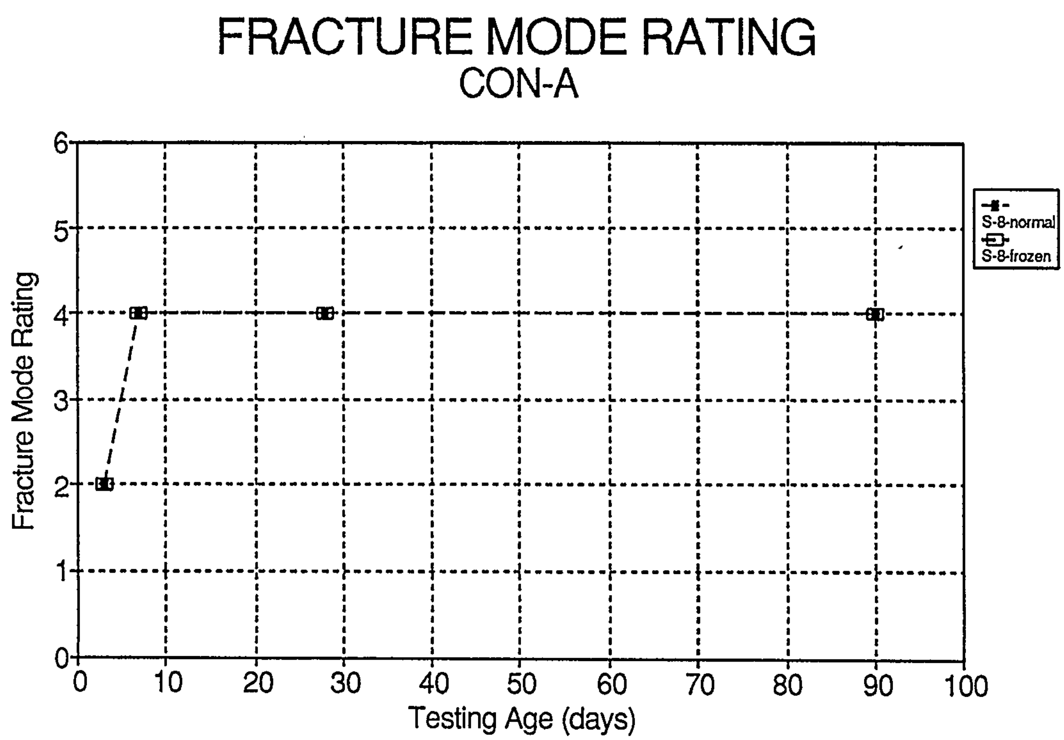


FIGURE 5.15: Fracture Mode Analysis for CON-A

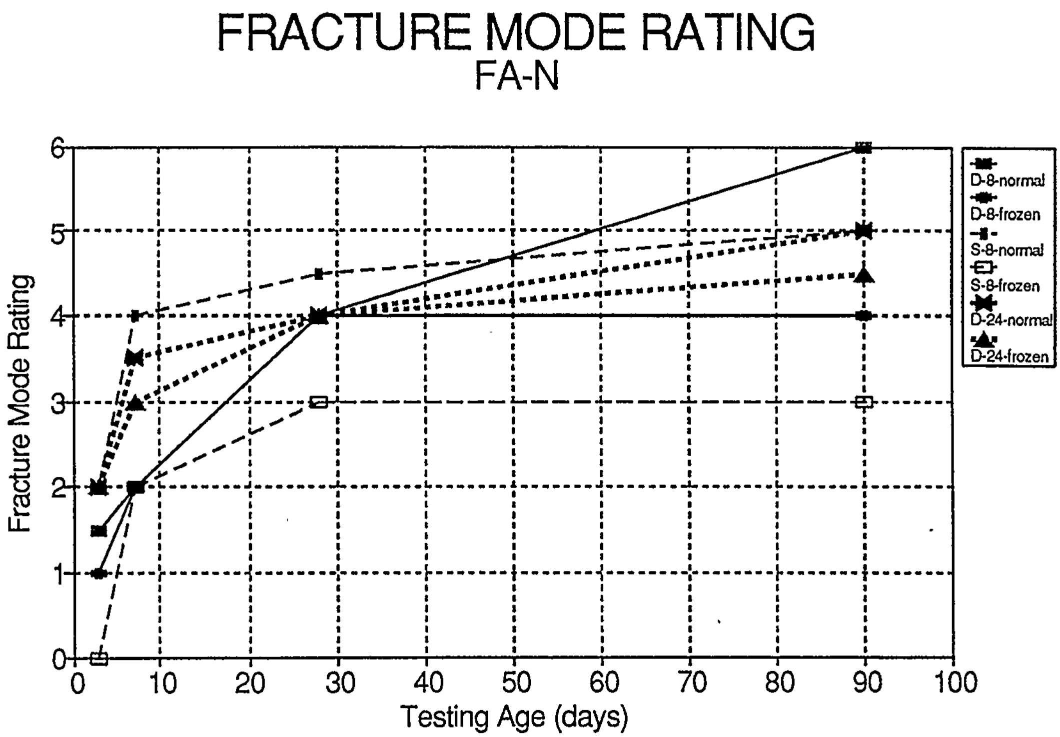


FIGURE 5.16: Fracture Mode Analysis for FA-N

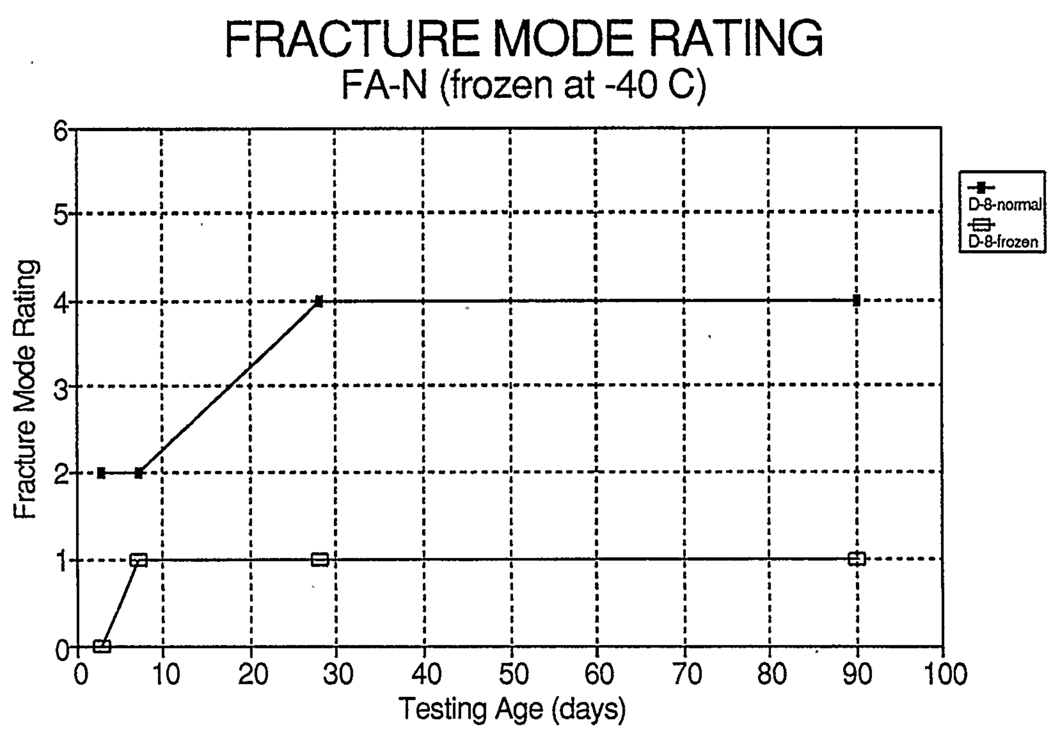


FIGURE 5.17: Fracture Mode Analysis for FA-N at -40 C

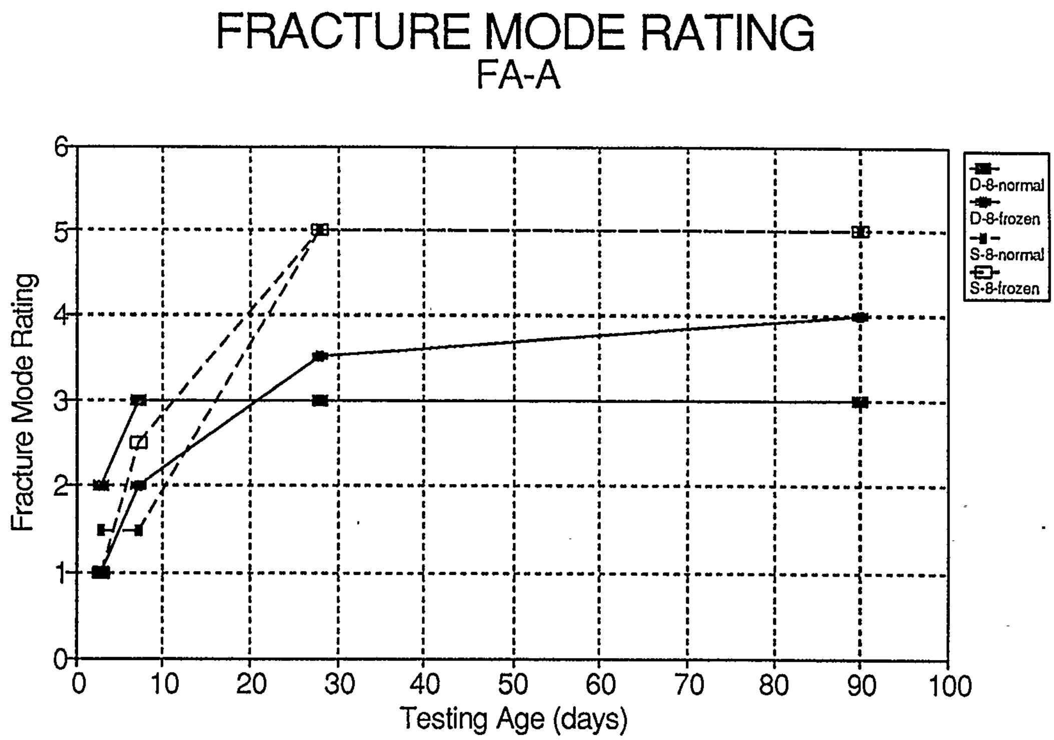
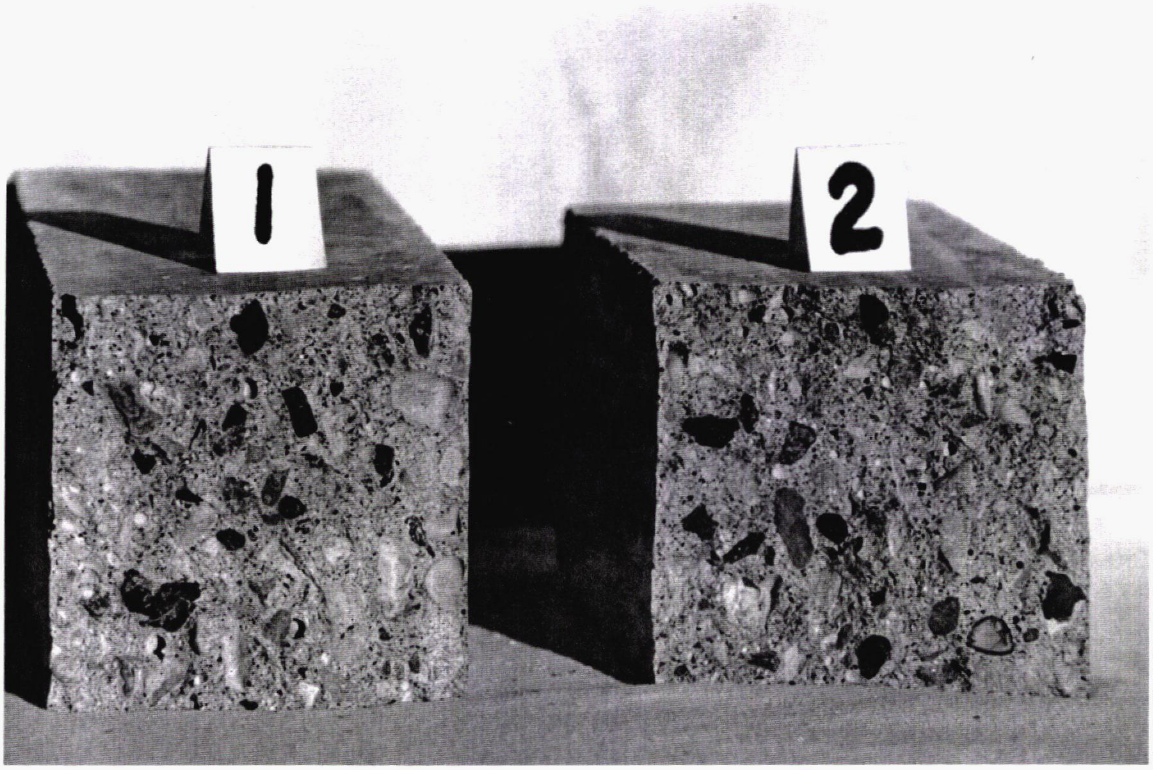
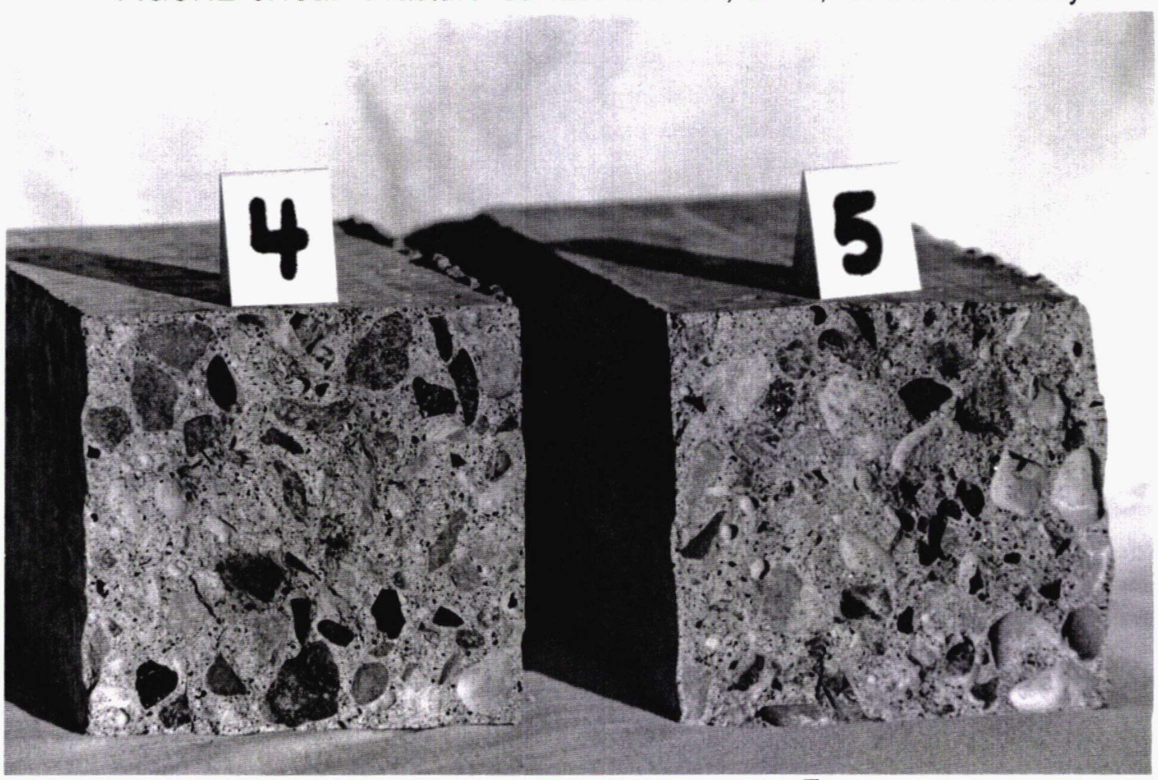


FIGURE 5.19: Fracture Mode Analysis for FA-A



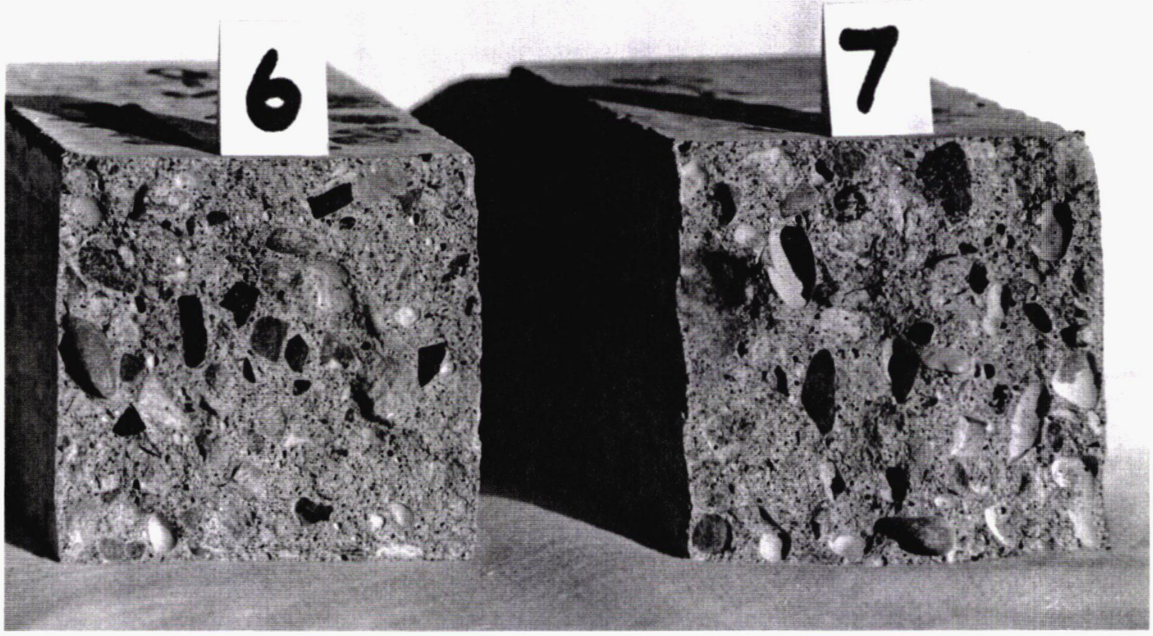
Normal Frozen

FIGURE 5.19a: Fracture Surface CON-A, D-F8, tested at 90 days



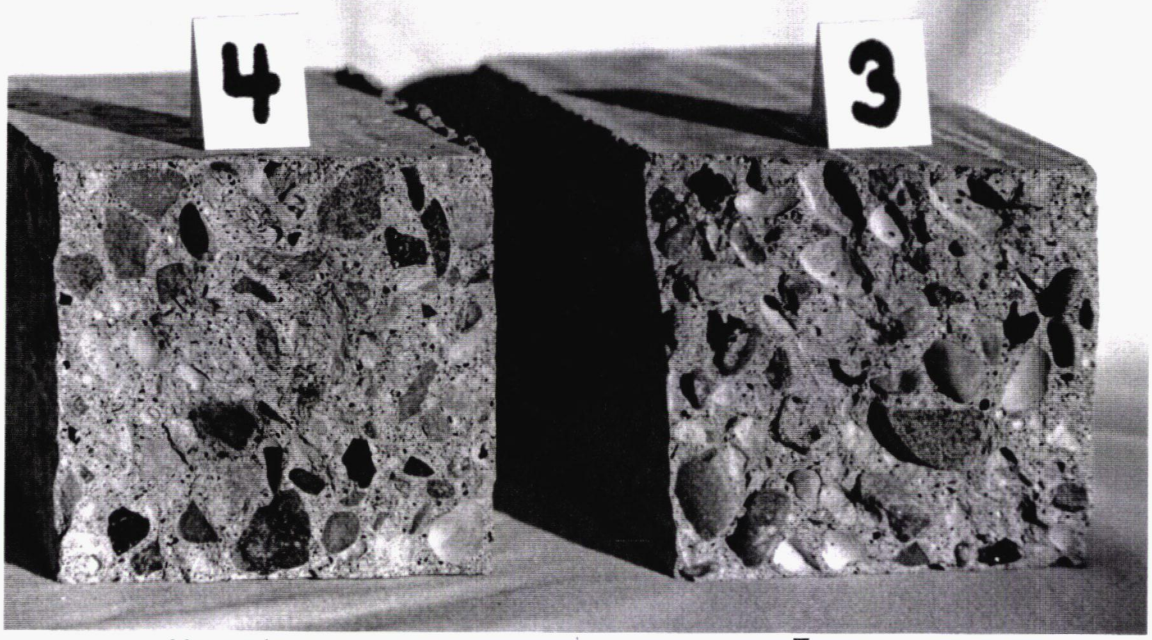
Normal Frozen

FIGURE 5.19b: Fracture Surface FA-N, D-F8, tested at 90 days



Normal Frozen

FIGURE 5.19c: Fracture Surface FA-N, S-F8, tested at 90 days



Normal Frozen

FIGURE 5.19d: Fracture Surface FA-N, D-F8*, tested at 90 days

CHAPTER 6: CONCLUSIONS AND RECOMMENDATIONS

The research program summarized in the preceding chapters provides insight into the effects of premature freezing on plain and fly ash concrete strength development. The following conclusions can be made from the testing results:

1. The strength development of fly ash concrete is more susceptible to premature freezing than that of comparable plain concrete. When frozen 8 hours after mixing, non air-entrained fly ash concrete has a greater permanent strength loss than plain concrete. Even when there is no permanent strength loss, the early strength development of fly ash concrete is affected more by freezing.

2. The difference between the 3-day and 90-day strength testing results emphasizes the importance of differentiating between short-term and long-term effects. Thus, if concrete is prematurely frozen, strength testing will have to be either delayed until at least 28 days or some form of accelerated strength testing will have to be used in order to determine if there is any permanent strength loss.

3. With the later freezing ages and with air-entrained concrete, the ultimate strength of the frozen concrete is greater than that of the normally-cured concrete. It is apparent that, if no damage occurs, there is an opportunity for the enhanced ultimate strength development which occurs with curing at cool temperatures to take place.

4. Despite being more susceptible to premature freezing, from an ultimate strength point of view, current CSA curing requirements are adequate for fly ash concretes at the 25% cement replacement level. With a pre-freezing curing period of 17 hours, there is no permanent strength loss. Using the maturity concept, this corresponds to a curing period, at the CSA minimum curing temperature of 10°C, of 29 hours - much less than the 72 hours required by the CSA code. (Recall that these mix designs were selected to produce concretes which would be susceptible to premature freezing. Most concrete in actual use would require even less curing.) Although curing may be sufficient for ultimate strength, there is still the problem of slow initial strength development. Care has to be taken with respect to early form removal, or load application, when concrete is exposed to freezing temperatures.

5. The soaking regime used in this research did not significantly affect the strength results. This does not discredit the claim that saturated concrete is more susceptible to freezing damage. It does indicate that the soaking regime used did not bring the concrete to a critical degree of saturation. As the soaking regime did not affect the length of time the concrete remained at 0°C while cooling, soaking could not have substantially increased the amount of "free water". This soaking regime should produce a degree of saturation similar to that which would occur due to water ponding or spraying. This would suggest that the fears regarding moist curing are not justified.

6. The specimens which incur permanent strength loss are also the ones which have the greatest amount of paste-aggregate bond failure — a typical mode of failure when freezing damage occurs.

7. The results from the standard (-20°C) and non-standard (-40°C) curing regimes differ. This emphasizes the importance of the freezing regime in determining the effects of premature freezing and the limitations associated with using only one regime in the laboratory.

8. When a permanent strength loss occurs, compressive strength is affected more than flexural strength. It is unclear whether this is because:
    i. compressive strength is more sensitive to freezing, or
    ii. the compressive specimens are smaller and thus experience a greater rate of cooling.

9. The temperature monitoring results indicate that it is important to differentiate between age at exposure to freezing temperatures and the age at which the concrete actually freezes. This difference will be even more significant with larger specimens where "freezing" will take even longer to occur.

10. The air-entrained mixes perform better than the non air-entrained mixes. It is unclear whether this is due:
    i. directly to the air-entrainment, or

ii. to the increased cement content and decreased w/c′ required to give the air-entrained mixes the same workability and strength properties as the non air-entrained mixes.

This research program has answered some questions regarding the effects of premature freezing on plain and fly ash concrete strength development, but it also raises some new questions. As well, it has laid the groundwork for further research. A set of procedures and mix designs have been developed which are practical in the laboratory and yield consistent results. Many of the basic concrete properties (fresh concrete properties, setting time, 28-day strength, air-void spacing factor, etc.) have also been measured. The following paragraphs summarize some important areas requiring further investigation and suggest some methods for carrying out that research:

1. Adequate strength development is not the only criterion which must be satisfied for satisfactory concrete performance. The effects of premature freezing on durability are also very important. A testing program similar to the one carried out here, but with the emphasis on durability tests such as freeze-thaw resistance and scaling resistance, is essential in determining if durability is impaired even when strength development is not. Scaling resistance is particularly important because premature freezing often affects the surface concrete more than the interior.

2. In this research project, saturated-surface dry (SSD) coarse aggregate was used. Failure at the paste-aggregate interface played a significant role in the fracture of

specimens with permanent strength loss. The saturated aggregate may be a major source of the water which migrates to and disrupts the bond at the paste-aggregate interface. Some of the test series should be repeated using aggregate with a moisture content representative of that found under normal storage conditions. This may reduce the weakening of the paste-aggregate interface and thus reduce the strength loss.

3. The nature of the freezing regime can affect the results, but it is unclear exactly what aspects of the freezing regime, and corresponding temperature conditions within the concrete, are affecting the results. The cooling rates and gradients which developed in these beam test specimens were all quite similar. By varying the specimen size and cold room temperature, a wider spectrum of temperature conditions can be studied. It would be useful for further research to find the features of a critical freezing regime that would maximize damage.

4. Before making recommendations regarding the CSA and ACI code requirements, a wider range of fly ash concretes should be tested. In particular, the cement replacement level and w/c′ parameters could be varied in a series of tests. The mix designs used in these tests could also be developed to answer the question raised in point 10 of the conclusions as to whether air-entrainment or reduced w/c′ improved premature freezing resistance.

5. The minimum curing times presented in the CSA and ACI codes are based on a minimum curing temperature. Room temperature was used during the pre-

freezing curing period in this research project. It would be beneficial to repeat some of the test series using this minimum temperature as the curing temperature. This would further check the adequacy of code requirements.

6. The soaking regime used did not affect these strength development results, but it may increase other effects like reduced durability and thus should be used in further studies. A soaking regime in which the specimens are immersed in water during the time when they are freezing could also be tried. This external source of water would theoretically contribute to ice lensing during freezing.

In conclusion, the effects of premature freezing on plain and fly ash concrete strength development have been studied in some detail. Beyond comparing the behaviour of the two concrete types, the various curing conditions and tests used contribute to the understanding of premature freezing. The results indicate that there is merit in extra caution when using fly ash concretes in winter concreting, but they also suggest an outright ban of fly ash in winter concreting is not justified. Further research is required before conclusive statements can be made regarding codes for the use of fly ash in cold weather concreting.

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APPENDIX A

INTERNAL TEMPERATURE MONITORING RESULTS

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INTERNAL TEMPERATURE - ALONG BEAM
CON-N D-F8

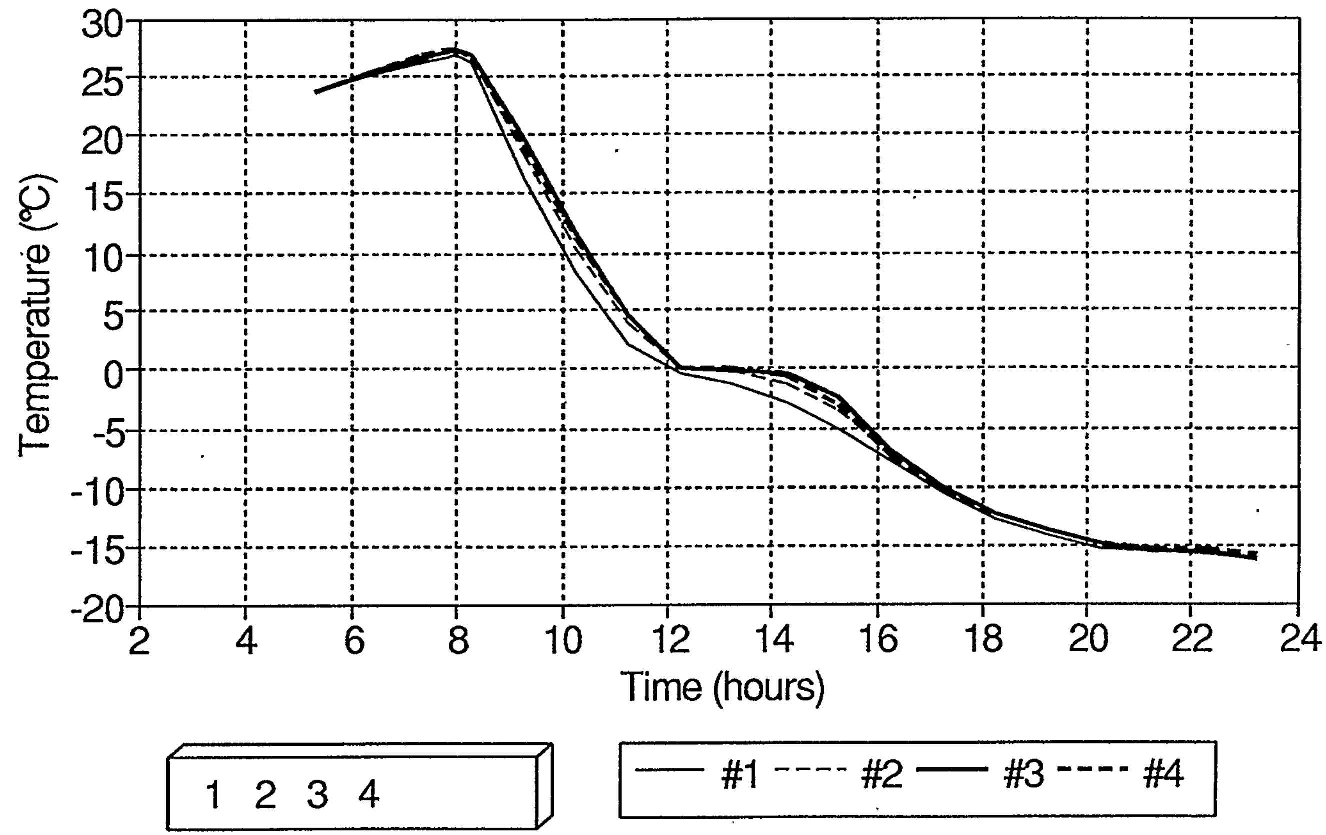

FIGURE A.1: Internal Temperature Monitoring Along Beam CON-N, D-F8

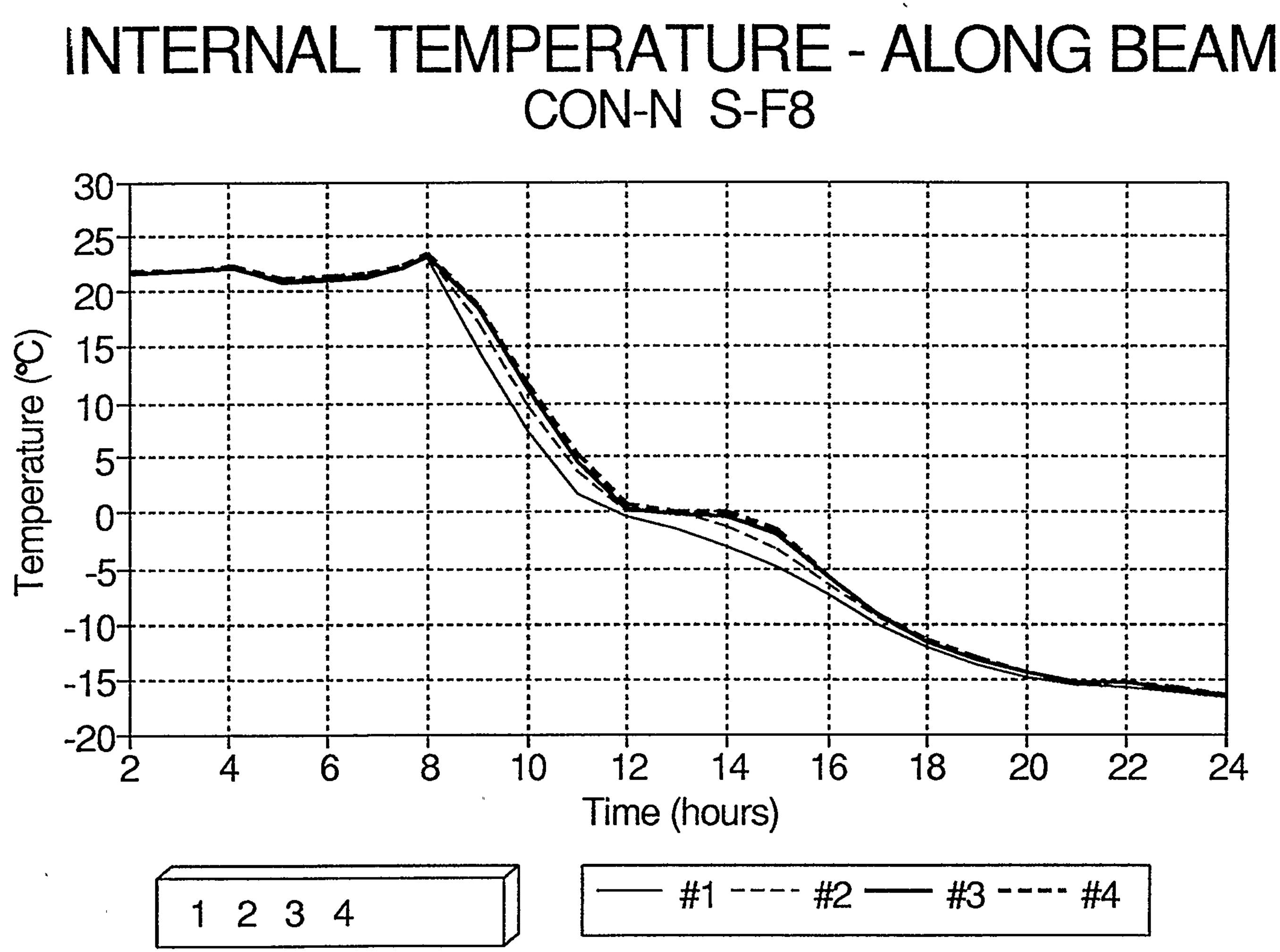

FIGURE A.2: Internal Temperature Monitoring Along Beam CON-N, S-F8

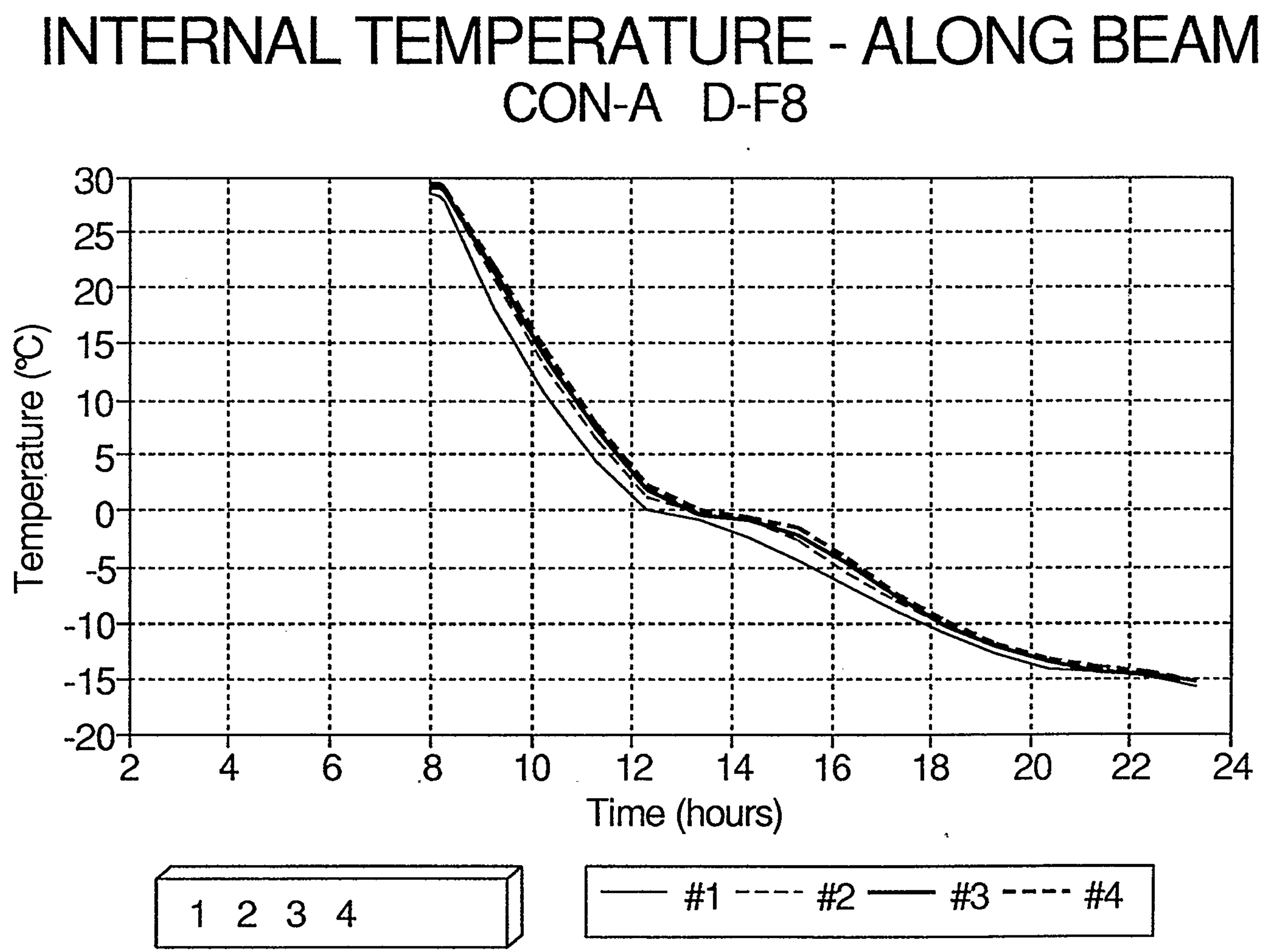

FIGURE A.3: Internal Temperature Monitoring Along Beam CON-A, D-F8

INTERNAL TEMPERATURE - ALONG BEAM

CON-A S-F8

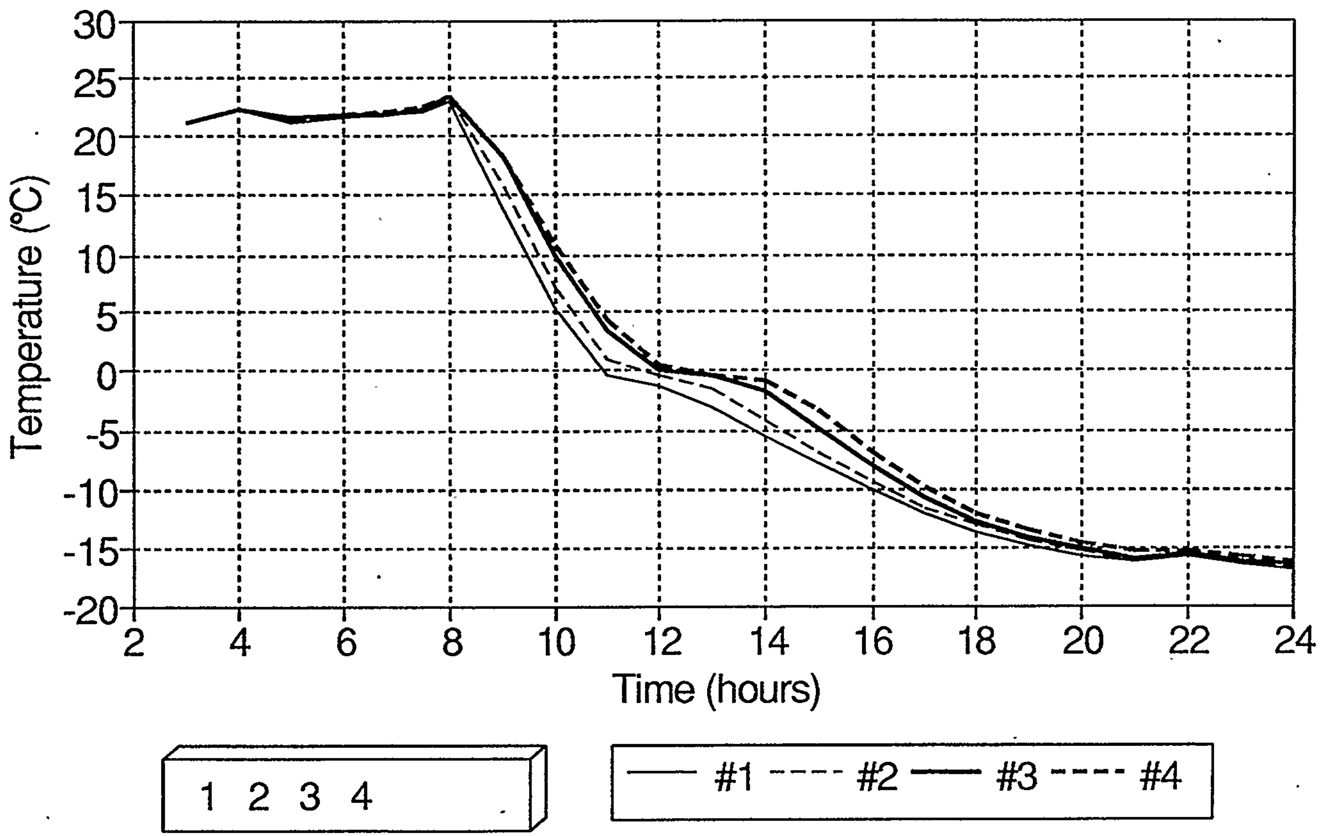

FIGURE A.4: Internal Temperature Monitoring Along Beam CON-A, S-F8

INTERNAL TEMPERATURE - ALONG BEAM

FA-N D-Not Frozen

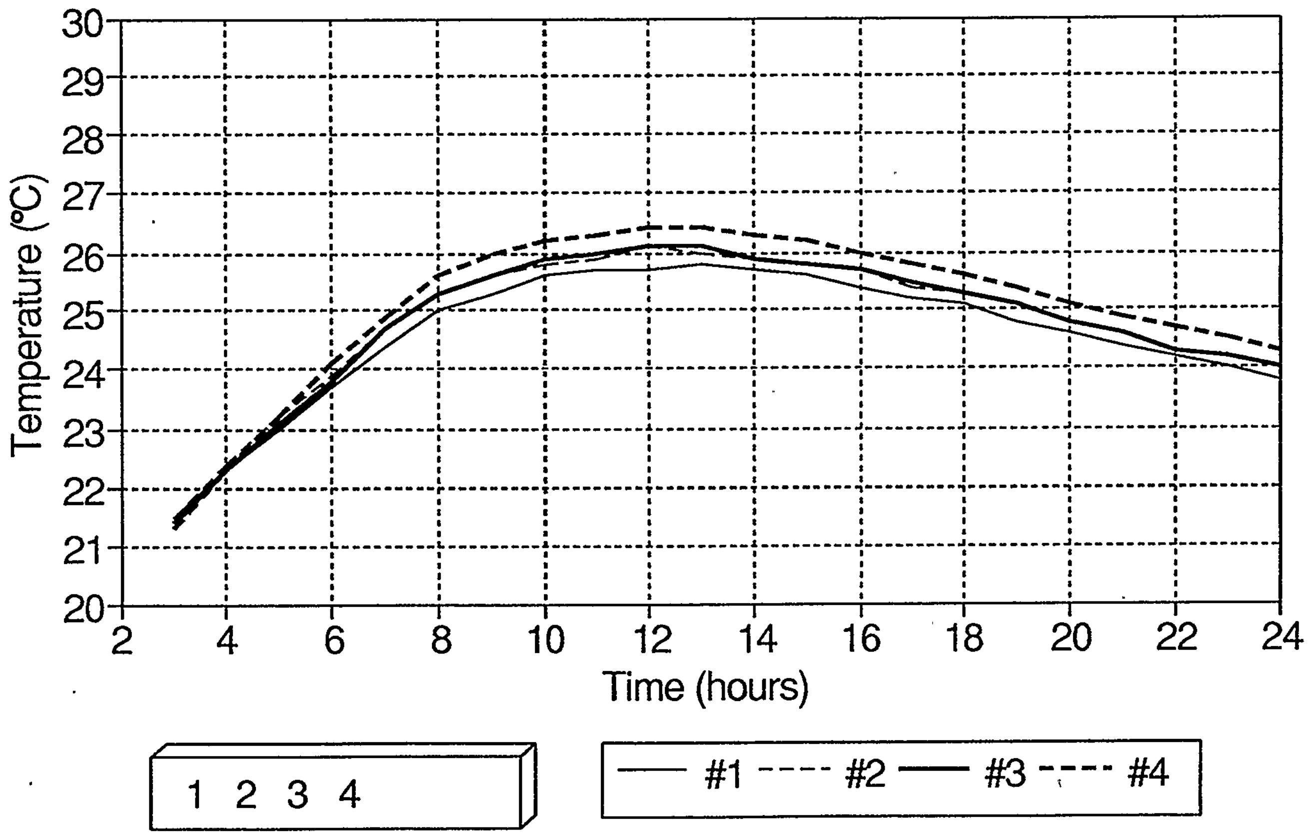

FIGURE A.5: Internal Temperature Monitoring Along Beam FA-N, D-N

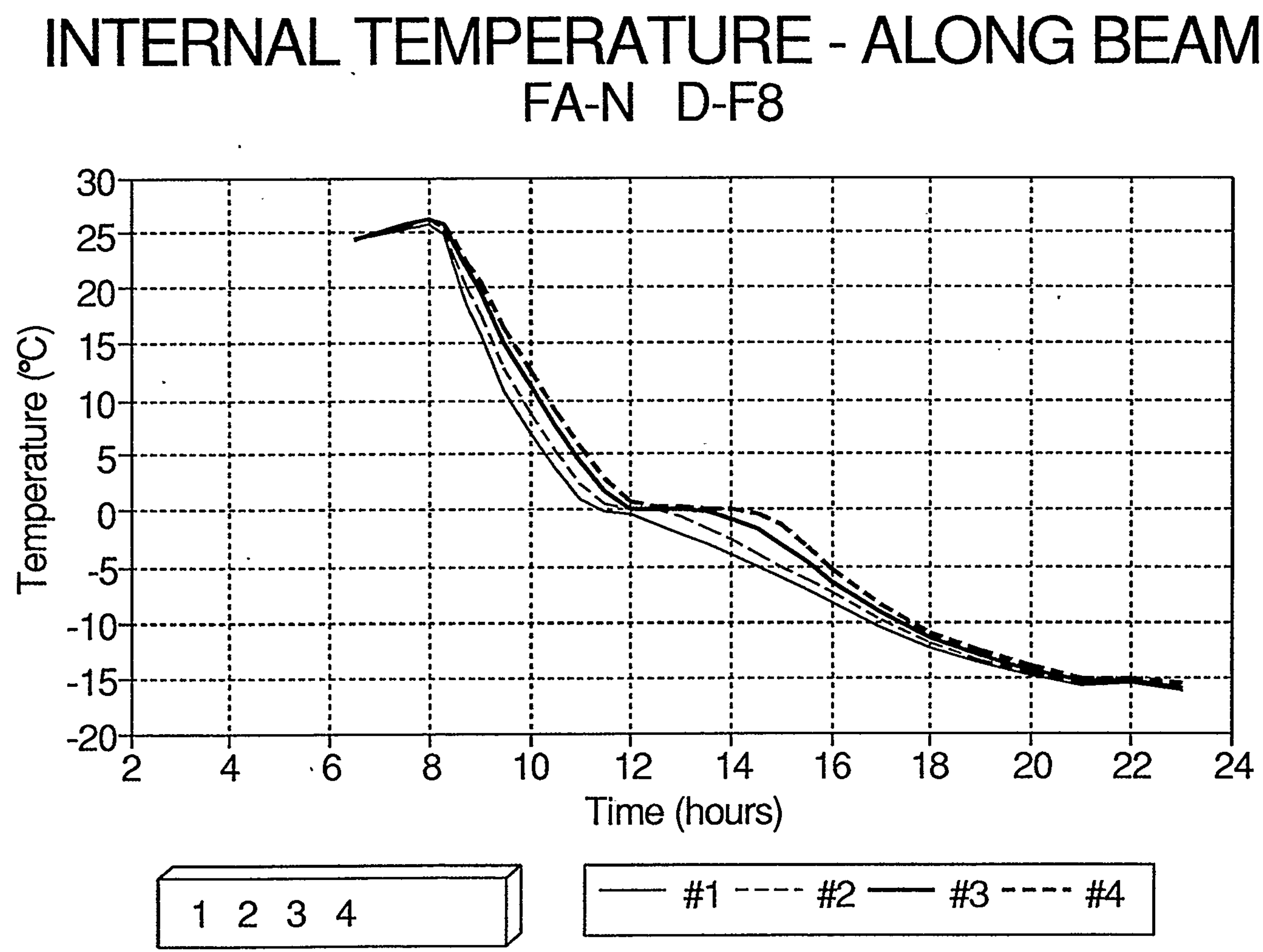

FIGURE A.6: Internal Temperature Monitoring Along Beam FA-N, D-F8

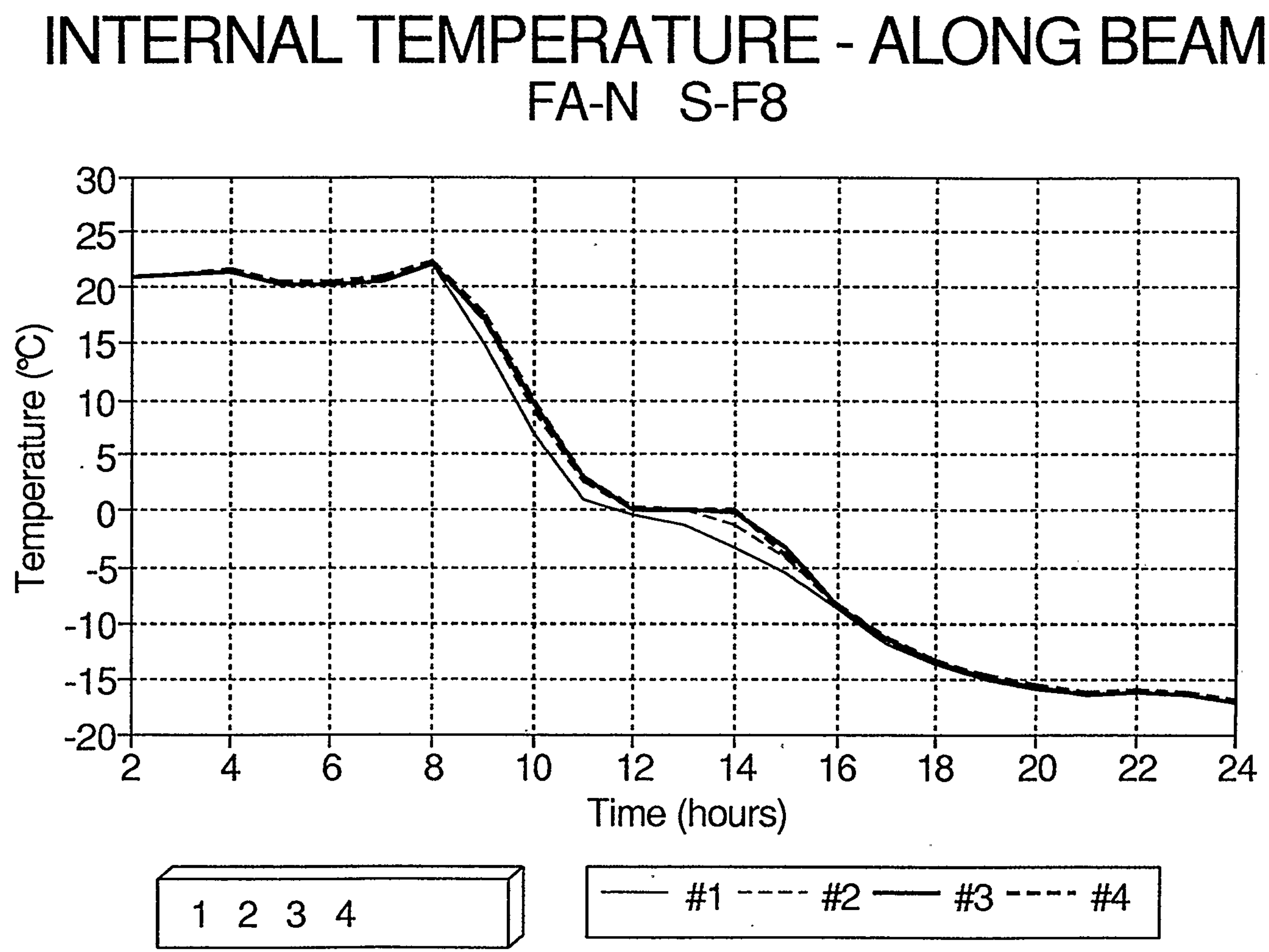

FIGURE A.7: Internal Temperature Monitoring Along Beam FA-N, S-F8

INTERNAL TEMPERATURE - ALONG BEAM

FA-N D-F24

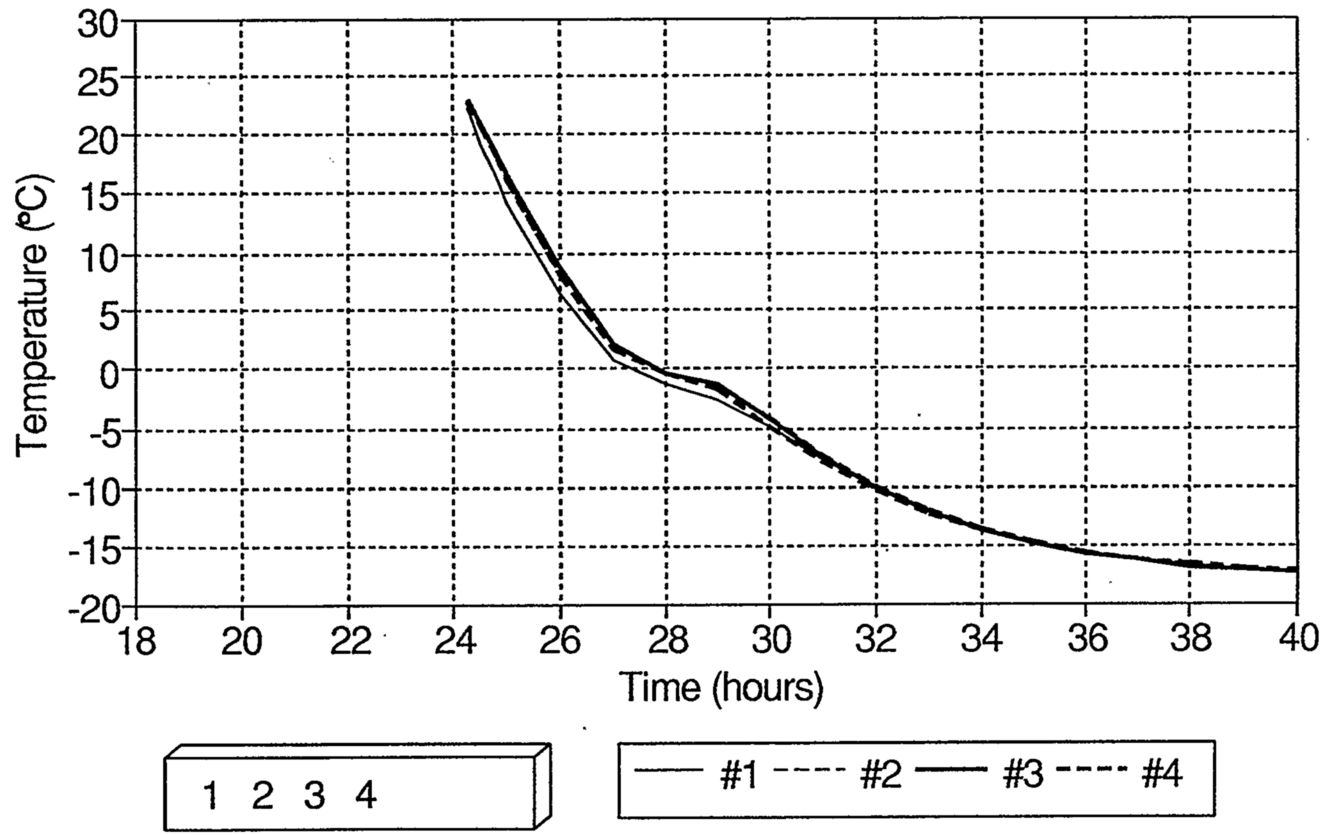

FIGURE A.8: Internal Temperature Monitoring Along Beam FA-N, D-F24

INTERNAL TEMPERATURE - ALONG BEAM
FA-A D-F8

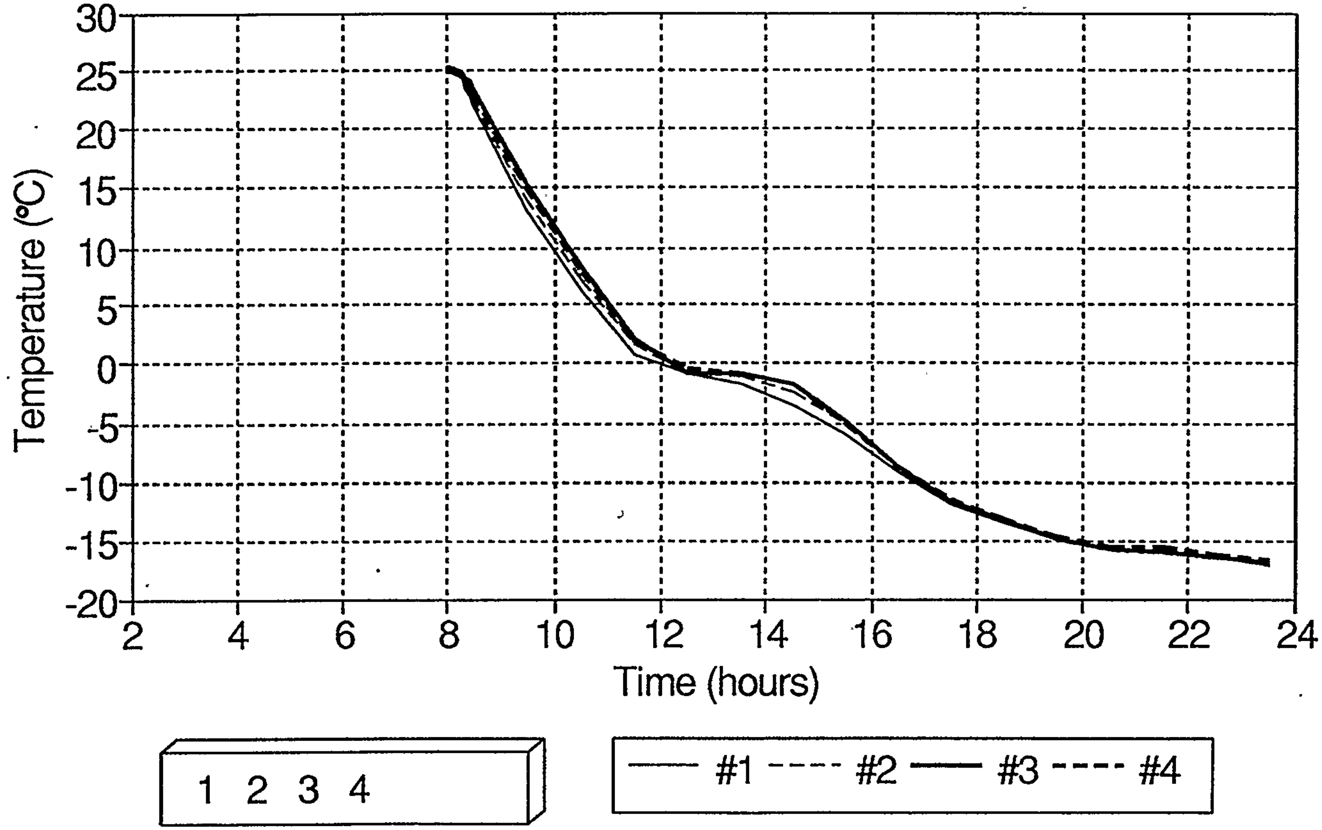

FIGURE A.9: Internal Temperature Monitoring Along Beam FA-A, D-F8

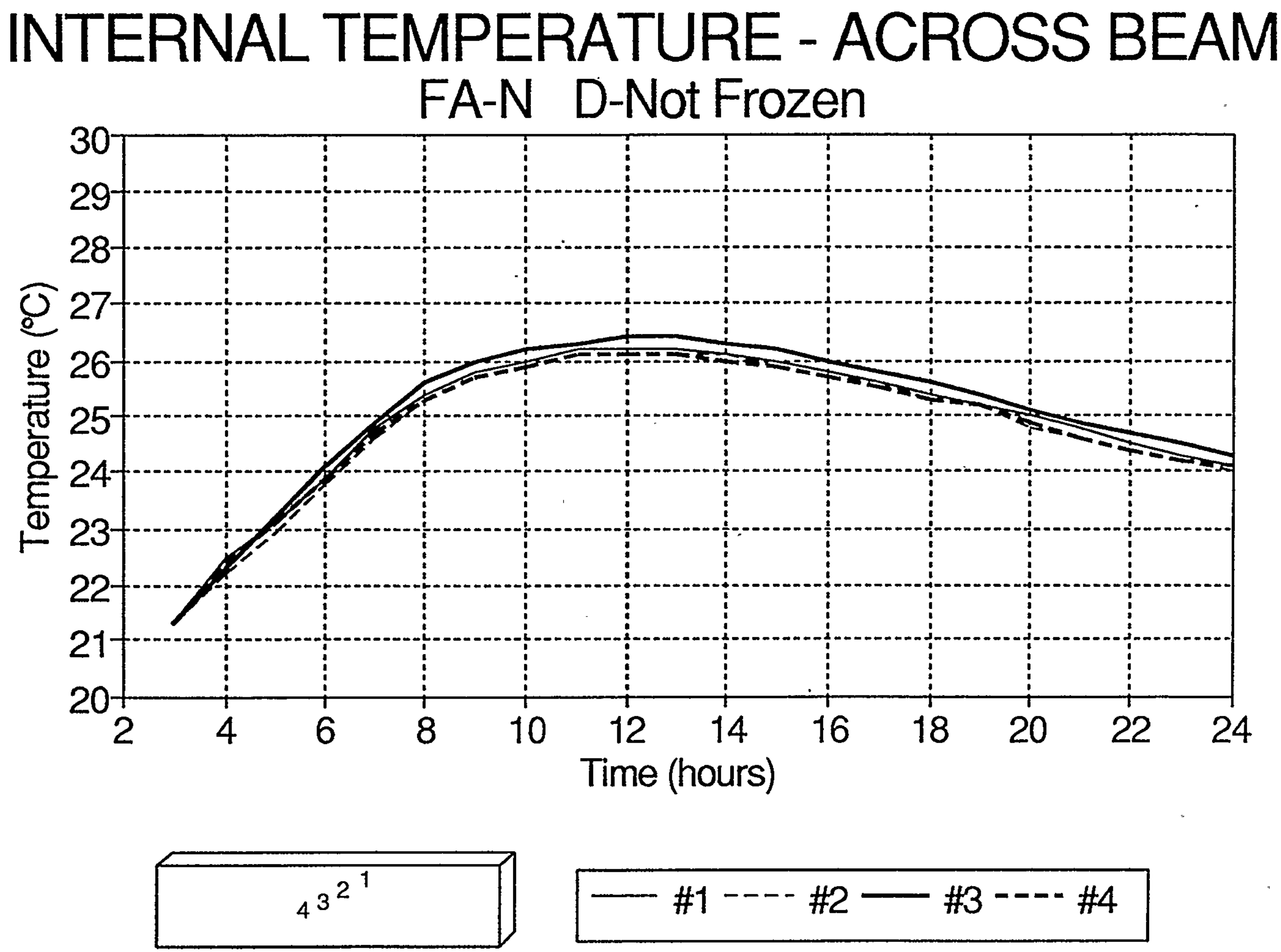

FIGURE A.10: Internal Temperature Monitoring Across Beam FA-N, D-N

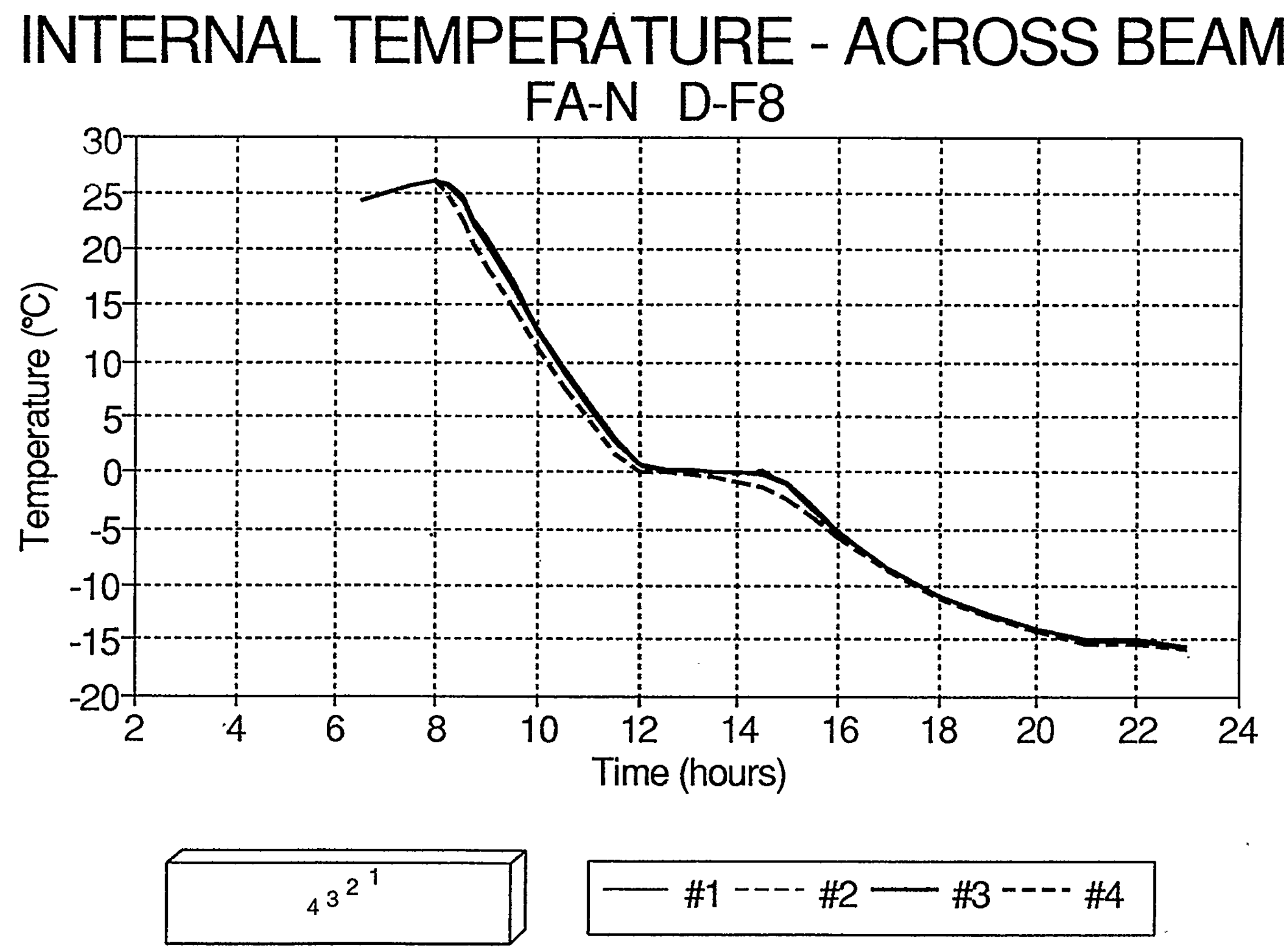

FIGURE A.11: Internal Temperature Monitoring Across Beam FA-N, D-F8

INTERNAL TEMPERATURE - CYLINDER
CON-A D-F8

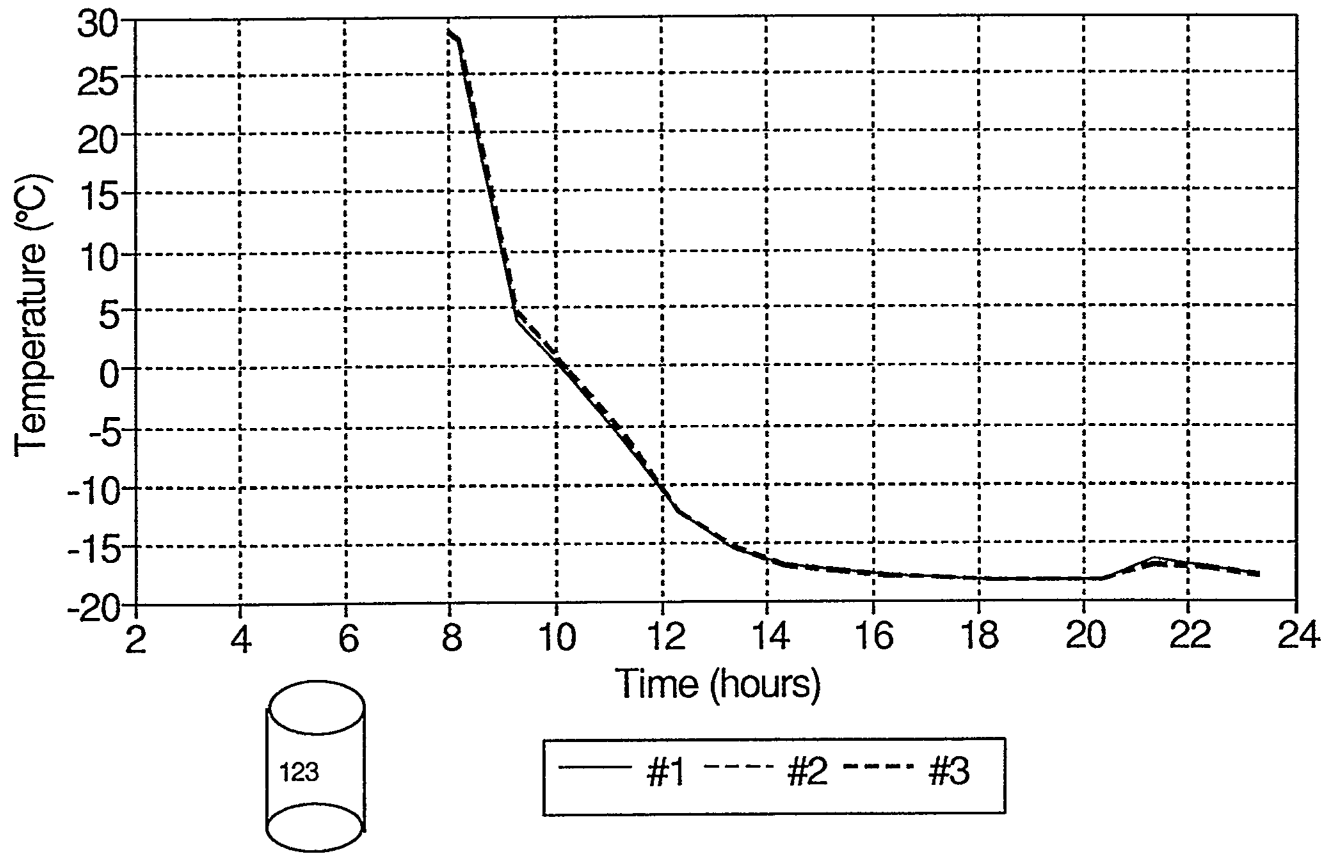

FIGURE A.12: Internal Temperature Monitoring Across Cylinder CON-A, D-F8

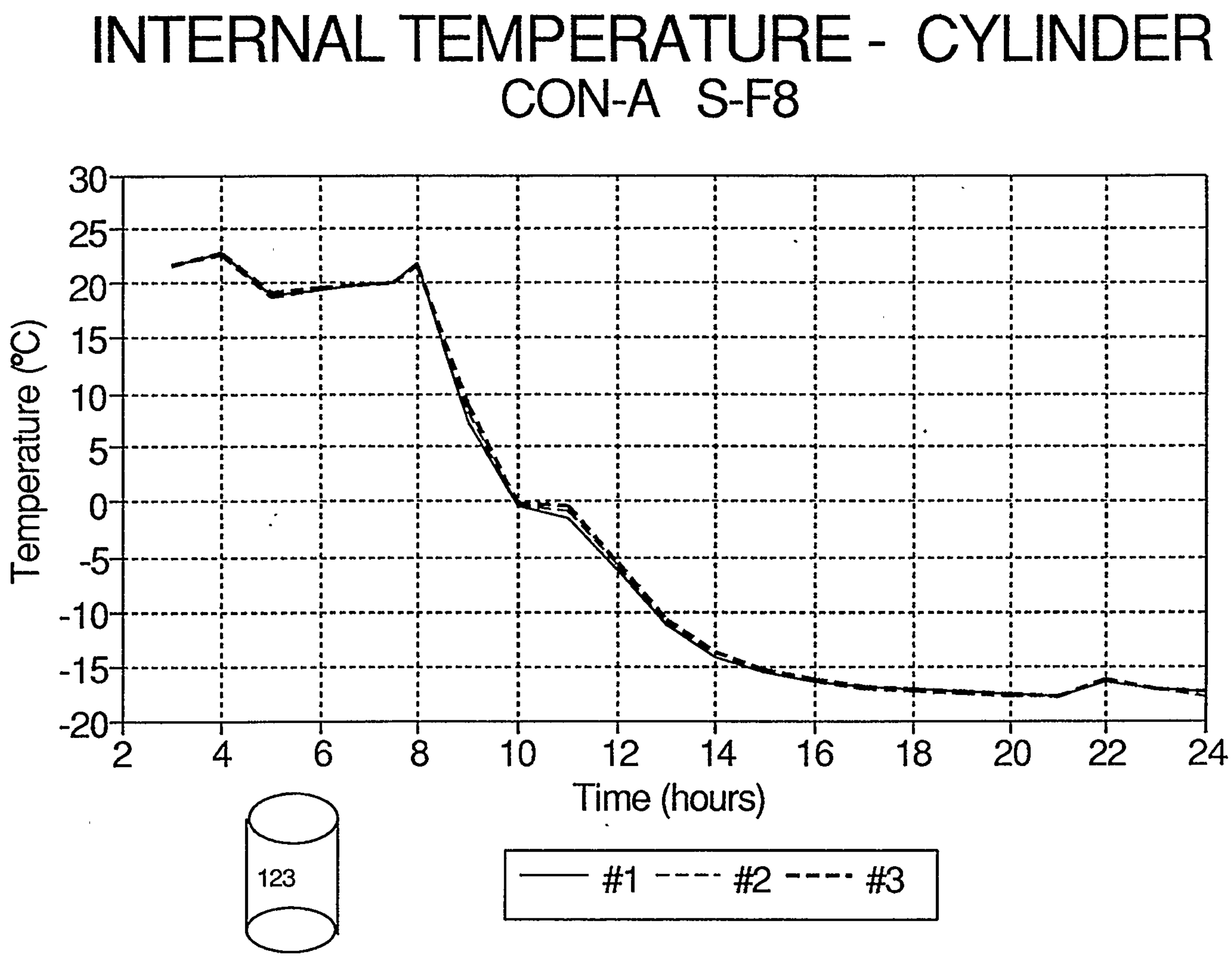

FIGURE A.13: Internal Temperature Monitoring Across Cylinder CON-A, S-F8