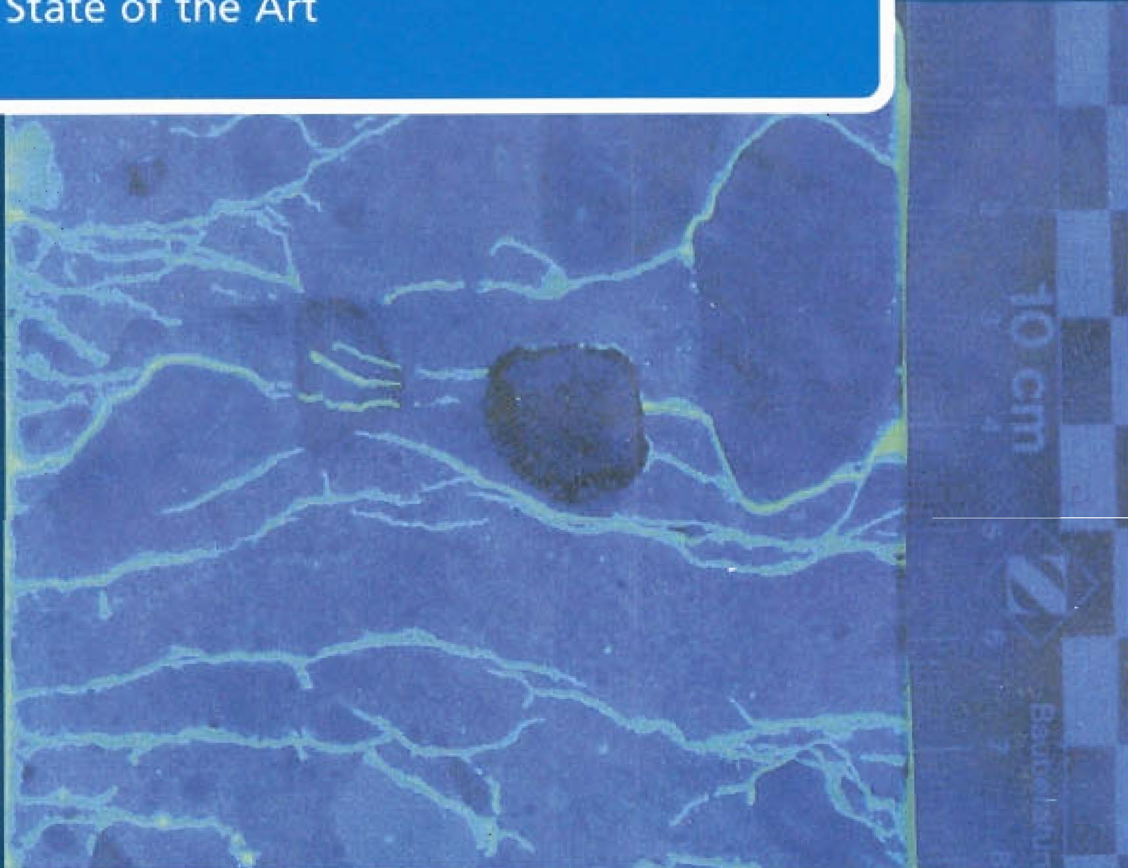
HETEK

Method for test of the Frost Resistance
of High Performance Concrete
State of the Art



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Abstract

The State-of-the-Art report contains an evaluation of selected existing freeze/thaw test methods: ASTM C 666, modified ASTM C 671, modified SN 640 461, SS 137244, and Rilem Draft Recommendation (CDF, and Cube test). The methods are described and their relevance is evaluated with a view to high performance concrete. The principles of frost deterioration are dealt with in so far as they have direct influence on the evaluation of the methods.

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1 Introduction

The Danish Road Directorate has made a research and development contract with seven consortia and individual companies. The subject of the contract is the establishment of guidelines for the execution of concrete structures with 100 years' service life, focusing on the technology of the contractor. Experience from large construction works proves that the execution phase is important to the achievement of the requested durability of concrete structures exposed to aggressive environment.

The development contract is financed by the Danish Agency for Development of Trade and Industry which demands that the task be defined, contracted and managed by a public agency, in this case the Road Directorate.

Task 2 of the development contract: "Test Methods for determining the Frost Resistance of High Performance Concrete" is awarded to a consortium consisting of Dansk Beton Teknik A/S, Dansk Teknologisk Institut, and Dansk Betoninstitut a/s. The task is carried out in cooperation with the Danish Road Institute, VD.

High performance concrete is defined as concrete with an expected service life of 100 years in aggressive environment [VD, 1995]. The consortium has implemented the following definitions of 'High Performance Concrete':

Concrete that can last for 100 years in an aggressive environment, having an equivalent W/C ratio of 0.35-0.45, and complying with the present Danish specifications regarding materials, mix compositions etc.

Hence, high strength concretes with high contents of silicafume, are not dealt with in the present work.

The first activity is a State-of-the Art Report with a critical evaluation of existing test methods through a study of literature with a critical and summarizing review of existing national and international knowledge to be used as the basis of the future work.

1.1 Background

Analysis of Danish and foreign standards and concrete specifications clearly demonstrates that the prevailing method of securing the frost resistance of a concrete material has been the requirements to the properties or origin of the aggregates (e.g. rock granite, class A material etc.), to the density (w/c ratio) of the cementiteous paste, and to the quality and amount of the entrained air void system.

When the rules were followed in practice, this procedure has under normal conditions resulted in the required quality level, i.e. the structures have for a long period been resistant towards freeze/thaw, salt, and humidity.

Moreover, requirements to direct testing of the frost resistance of the structures made have not been common. I.a. RILEM TC117-FCD: "Freeze-Thaw and Deicing Resistance of Concrete" states that the reason for this apparent disinclination for a direct testing of the quality of the concrete probably is the poor reliability and reproducibility of the frost test methods known so far, and the general lack of documentation for the correlation of relations between test results and actual properties.

However, the above mentioned Technical Committee also finds that it is now imperative to develop and substantiate test methods for direct testing of the characteristics of a concrete structure.

The European directive on building producers enables the use of a variety of constituent materials in concrete which may be exposed to an environment to which it has not traditionally been exposed. Furthermore, the introduction of new, considerably more dense types of concrete, high performance concrete, raises the question whether air entrainment is necessary in such concretes and whether the demands to the characteristics of the air void structure that are often used today (e.g. the air void spacing factor <0.20 mm) are sufficient for high performance concrete.

Unfortunately, no mathematical/physical models for the description of conditions as to saturation, freezing and deterioration of concretes with different diffusion and sorption characteristics have been developed. Therefore direct testing with application of the most relevant test methods is considered the only applicable procedure at the present time.

1.2 Objectives

The main objective of the project is to reevaluate existing methods and, if necessary, develop new methods for determination of the frost resistance of high performance concrete.

The State-of-the-Art report contains an evaluation of selected existing frost test methods, ref. Section 10 for detailed information on the references:

* ASTM C 666, A: Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing

* Modified ASTM C 671: Standard Test Method for Critical Dilation of Concrete Specimens Subjected to Freezing

* Modified SN 640 461: Test II for resistance to frost and de-icing salts. (Dubrolobov-Romer)

* SS 137244: Concrete Testing - Hardened Concrete - Scaling at Freezing (the Borås method)

* Rilem Draft Recommendation: 117-FDC Freeze-Thaw and Deicing Resistance of Concrete:

  1 Tests with water (CF) or with sodium chloride solution (CDF)
  2 Slab test (almost the same as SS 13 72 44)
  3 Cube test.

The methods are described as to test specimens, apparatus, measuring principle, conditioning, temperature intervals, and gradients and available knowledge regarding precision (repeatability and reproducibility), test duration, and economy.

The relevance of the various test methods is evaluated with a view to the use of high performance concrete.

The basic principles of frost deterioration are dealt with in so far as they have a direct influence on the evaluation of the existing test methods. The deterioration mechanism(s), including the controlling parameters, are described with a view to uncovering the possibilities of modelling frost deterioration.

There exist numerous other test methods than those described in the report. Two of these methods, that have been quite much used are:

* The critical degree of saturation method; a tentative RILEM-method [Fagerlund, 1977]. In a simplified version it has been much used by Swiss Federal Laboratories for Materials Testing and Research (EMPA) [EMPA, 1987], [EMPA, 1989], [Studer, 1992]

* Vuorinen's method; a dilation test which has been frequently used in Finland [Vourinen, 1969].

These two test methods have not been described in the present work due to the limitations of the contract.

However, the concept of critical saturation is included in several of the sections due its fundamental importance.

2 Damage Caused by Frost Attack on Concrete

Deterioration of concrete exposed to freezing conditions occurs when there is sufficient moisture present. The fundamental mechanism of frost damage is related to the volume increase occurring when water freezes to ice. Selected proposed mechanisms for freeze/thaw deterioration are dealt with in Section 6.

If this happens in a confined space the surroundings are exposed to pressure. If the tensile strength of the surroundings is exceeded, cracks are formed. The presence of air in capillary pores or in air bubbles reduces the pressure because they act as expansion chambers for unfrozen water reducing the hydraulic pressure and/or as strain release for growing ice bodies, ref. Section 6.

In concrete, water may in practice freeze in three different places:

a) In the capillary pores of the cement paste
b) In the void spaces such as air voids and defects (e.g. cracks)
c) In the porous aggregate.

2.1 Freezing of Concrete

In mature concrete with frost-resistant aggregates the signs of frost damage are:

* Surface scaling
* Internal cracking.

It is often impossible to diagnose the cause of concrete distress on the surface crack pattern alone. Map-cracking may be due to shrinkage, alkali-silica reaction (ASR), frost attack, sulphate attack, or delayed ettringite formation. Delamination may also be caused by at least three different processes: ASR, frost attack, and reinforcement corrosion.

In Denmark the diagnosis "frost-attack" has traditionally been given after excluding other possible causes such as ASR. If ASR occurs at the same time it is very difficult to diagnose the combination of ASR and frost-attack. Tests have indicated that cracking due to ASR might severely reduce the frost resistance [Trägård and Lagerblad, 1994].

2.1.1 Surface Scaling

Surface scaling is the loss of paste and mortar from the surface of concrete. Generally, pieces less than a few mm in thickness are lost. By the process coarse aggregate particles may be exposed and finally lost. Scaling might be combined with the occurrence of pop-outs, ref. below.

Typically, the concrete near the surface is more porous and contains surface cracks which facilitate rapid moisture ingress. If the inner concrete remains intact, the rate of scaling will decrease, whereas defects in the interior may cause increasing scaling rate.

Scaling is considerably accelerated by the presence of de-icing salt, ref. Section 4.2.2. The consequences of scaling include change in appearance and surface friction properties, and in severe cases the loss of significant cover over reinforcement. Scaling can therefore reduce the estimated service life with regard to reinforcement corrosion [Fagerlund, Somerville, and Tuutti, 1994].

2.1.2 Internal Cracking

Internal cracking may not be visible on the concrete surface.

As in all other concrete deterioration the theoretical "ideal crack pattern" will be modified by the presence of reinforcement in the concrete. The cracks tend to follow the line of least resistance. For instance in a pretensioned concrete railway sleeper interior expansion may cause map-cracking on the shoulders at each end while single, longitudinal cracks are found in the main body of the sleeper.

Delamination cracks are internal cracks in the concrete body parallel to the exposed surface. The concrete can be damaged by the delamination cracks to a considerable depth (often exceeding 100 mm). The cracks are discrete and sub-parallel to the surface and often with a crack distance of around 10 mm. The crack planes may frequently pass through aggregate. Between the crack planes, the concrete generally appears undamaged and without deterioration. In advanced stages, the crack planes are interconnected by smaller cracks, thus decreasing the size of unbroken concrete parts and eventually causing complete disintegration.

Since the delamination cracks are generally formed parallel to the exposed surface, they may be detectable in cored samples only. However, in the corners (joints) of concrete slabs, the delamination cracks parallels the vertical terminations of the plates and thus form vertical systems which are easily seen on the horizontal surface. The latter type of crack patterns are often named D-cracking [Cordon, 1966] or D-line cracking [Pigeon and Pleau, 1995].

The formation of D-cracking is connected to high moisture contents [Cordon, 1966] and may be superimposed by the effect of varying load capacity of the slab [Henrichsen, 1996].

In recent American literature D-cracking is referred to occur in concrete made with frost-susceptible aggregates in e.g. highway pavements, parking lots and sidewalks [ACI C 201, 1995]. The typical crack pattern visible on the surface of the concrete is referred to caused by expansion and cracking of coarse porous aggregate particles in the interior of the concrete. D-cracking is characterized by cracks through both the coarse aggregate and the mortar portion of the concrete. Away from the cracks, both the mortar and the coarse aggregate are strong and show no signs of deterioration.

The above statement is contradicted by the fact that D-cracking has been observed also in concretes with sound granitic aggregate. Furthermore, the existence of cracks passing through aggregate is governed by the relationship between the strength of the cement paste, the bond strength and the internal strength of the aggregate (the latter influenced by e.g. internal cracks).

Cracking is the visual appearance of internal frost damage. Frost damage will of course also affect the cohesion and strength of the concrete. Measurements have indicated that the loss in compressive strength might be as high as 30%, the loss in splitting tensile strength 50%, and the loss in bond to the reinforcement 80% or more [Fagerlund, Janz, and Johannesson, 1994].

2.2 Freezing of Porous Aggregates

Single porous coarse aggregate particles close to the surface may cause pop-outs on the concrete surface. The pop-out consists typically of a cone-shaped piece of concrete where the fracture passes through paste and dense aggregate particles. The porous particle causing the pop-out is located centrally at the bottom of the pop-out and is normally a porous limestone or a porous opaline limestone. The particle expands and cracks due to freezing of water inside the particle [Bache, 1991]. Alkali-silica reaction may cause similar pop-outs. See also Section 4.1.4 regarding effect of aggregates on frost damage.

2.3 In-situ Observations

According to Fagerlund [Fagerlund, 1993] scaling is observed on concrete structures exposed to de-icing salt or sea water, especially in moist parts, e.g. joints, tidal zone, splash zone, and areas without sufficient drainage. This is in accordance with Petersson who stated that internal cracking is observed in structures exposed to pure water, whereas scaling is observed in structures exposed to salt [Petersson, 1993].

Internal cracking is observed in structures with a moist interior, e.g. structures exposed to water pressure from behind and structures sucking moisture from the soil.

The surface of these structures are often drier than the interior due to evaporation. [Fagerlund, 1993].

Delamination is typical of concrete in moist environments such as, e.g., concrete pavements with insufficient drainage and bridge decks sealed below an inadequate membrane.

As part of a recent reevaluation of the requirements of the Basic Concrete Specification [BBB, 1986] to frost resistant concrete selected Consulting Engineers were interviewed. The short interviews indicated the following [DTI, 1995]:

* Frost damage is in Denmark generally only classified as such if other deterioration mechanisms can be excluded and the concrete is moist and not air entrained

* Frost damage identified as above occurs in 5-10% of the structures investigated due to visible cracks or percolating water

* Delamination is the most frequently occurring frost damage, sometimes in combination with initial (thermal) cracks, alkali silica reactions, or percolating

* Frost damage is observed in areas with defective structural detailing or poor workmanship

* Surfaces facing South and West are often more damaged than others.

According to [Henrichsen, 1996] and [Pedersen, 1996] D-cracking is observed in Denmark in airport runways and flagstones/pavements.

3 Water and Ice

Below a brief introductory section on fundamentals for the transformation of water to ice is given.

The freezing of pure bulk water (1 atm) happens at 0°C and is accompanied by a volume increase of about 9% by volume ($9^{1/3}$=2.1% by length). The ultimate tensile strain of concrete is approximately 0.01%. Ice formation in a saturated concrete will thus cause damage.

Cooling of pure water at constant volume will not cause a total phase transition at 0°C. Only half of the water will become ice, ref, Figure 3.1. At -22°C the pressure will be 200 MPa, corresponding to a pressure increase of 10 MPa/°C. [Bache, 1991].

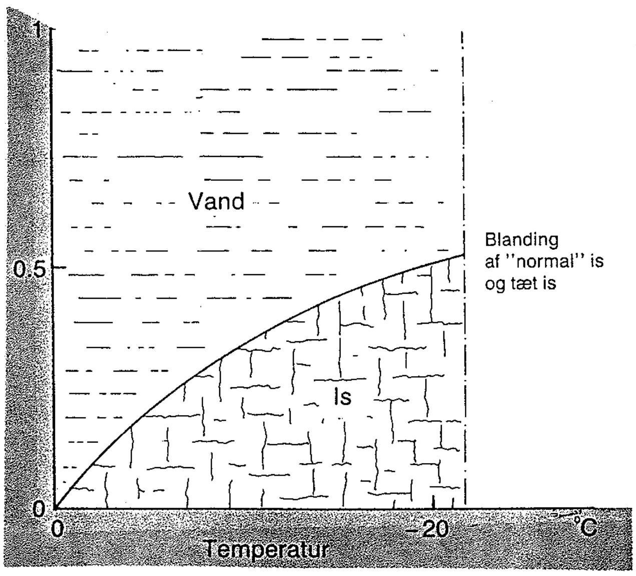

Figure 3.1 Volume ratio between water and ice during cooling at constant volume [Bache, 1991].

The ultimate tensile strain capacity of concrete is approximately 5 Mpa, corresponding to a freezing-point depression of approximately 0.5°C taking place before damage occurs.

The freezing-point depression due to soluble salts is approximately 2°C, ref. Section 5.1. Furthermore, water held in capillary pores is subjected to a freezing-point depression due to the lower free energy of the water held in these pores, ref. Section 5.1. Water held in gel pores will freeze at even lower temperature, if ever.

Thus, frost damage of critically saturated concrete is likely to occur at temperatures below -2.5°C.

During cooling super-cooling of the pore liquid may take place, ref. Section 5.2. The super-cooled liquid is thermodynamically unstable. Super-cooled liquid (typically in smaller pores) in contact with ice (typically in larger pores) will tend to flow towards the ice, ref. the ice lens growth theory, Section 6.3. It has been estimated by simplified theoretical calculations that ice lens growth can be hindered by a counter pressure of 1 MPa/°C. [Bache, 1991].

Freezing of water causes heat evolution (an exothermal process). The temperature of the system where growing ice is in contact with liquid pore solution will be at a constant temperature (i.e. freezing-point) until no more liquid with the actual freezing point is present. The heat formed by ice formation must be dissipated in the surroundings.

4 The Effect of Selected Parameters on Frost Damage

Below, the effect of selected material parameters and exposure conditions on frost damage is dealt with.

4.1 Material Properties

The dominant factors material properties the freeze/thaw durability of a concrete are:

* The amount of freezable water
* The availability of empty space
* The permeability of the concrete
* The strength of the concrete

4.1.1 Critical Water Saturation

The importance of the degree of saturation with respect to frost damage appears first to have been emphasized in [Hirschwalt, 1910]. Based on the theories of Powers, a critical degree of saturation has been proposed [Fagerlund, 1977]. The degree of saturation is the ratio between the absorbed, freezable water and the total available space (volume of water plus available air filled capillary pores and air voids). The critical degree of saturation is the limit above which the amount of absorbed water causes damage on freezing.

For ordinary concrete the normal range for the critical degree of saturation is 0.75 - 0.90. Increasing air void content results in a lower value of the critical degree of saturation. The critical degree of saturation is also lower the coarser the air void system [Fagerlund, 1977].

The critical degree of saturation can also be calculated theoretically on basis of the following information [Fagerlund, 1979], [Fagerlund, 1995-a]:

* The critical spacing factor between air voids
* The air void size distribution

Recently, the possibilities of evaluating the freeze/thaw durability of concrete based on calculations of critical degree of moisture saturation has been investigated [Hansen, 1995].

An increasing freezing rate will slightly reduce the critical degree of saturation, ref. Figure 4.1. This can also be explained theoretically [Fagerlund, 1992].

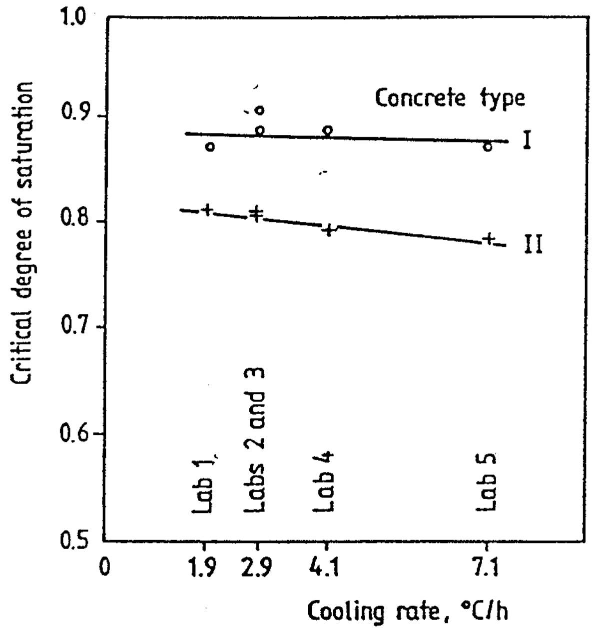

Figure 4.1 Effect of freezing rate on the critical degree of moisture saturation. Concrete I without entrained air and concrete II with 7% air [Fagerlund, 1992]

4.1.2 Microstructure

The rate of transport of water and the temperature at which the water freezes are influenced by the micro structure of the concrete, i.e.:

* The amount of pores, the pore size distribution, and the interconnection of pores
* The location, frequency, and interconnection of cracks and other defects.

The sizes of pores in the cement paste embraces several orders of magnitude. According to the origin and characteristics the pores are described as compaction pores (entrapped air), air voids (entrained air), capillary pores, and gel pores, ref. Figure 4.2.

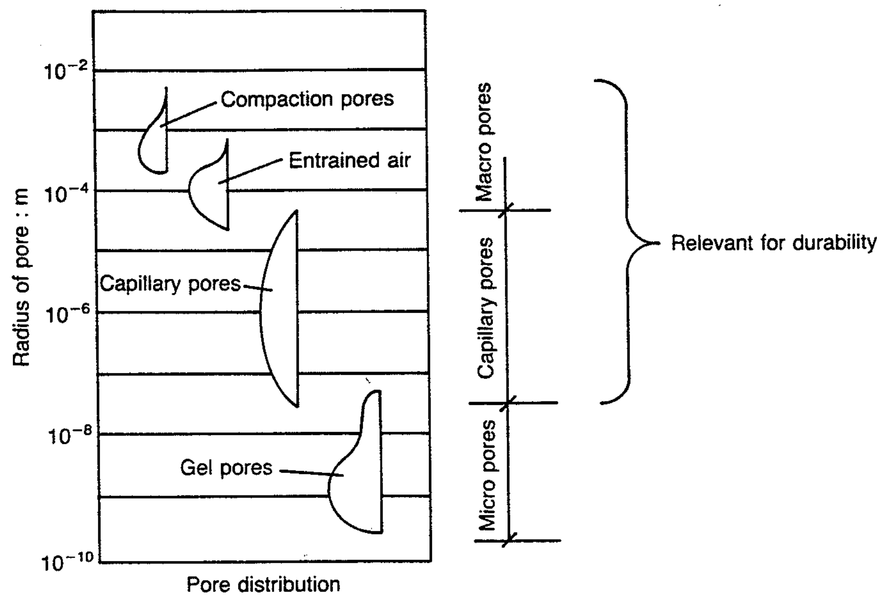

Figure 4.2 Pore size distribution [Setzer, 1977].

Based on the work reported in [Powers and Brownyard, 1948] it is assumed that water can be held in the finely porous material by three mechanisms. The physical condition of the water in the concrete is thus:

- Adsorbed in the gel pores
- Condensed in the capillary pores
- Free in larger cracks and pores.

The pore size distribution in the concrete is continuous, causing a gradual change of the physical state of the water. As an estimate, it is anticipated that capillary condensation will not occur at vapour pressures below 40% relative humidity. The BET theory gives the relationship between vapour pressure, amount of adsorbed molecules, and specific surface [Brunauer et al., 1938]. Capillary condensation is dealt with in Section 5.1.

For w/c=0.4 no freezing occurs above -20°C in a virgin paste [Fontenay, 1982]. Estimates based on entropy data indicate that pore water held below 60% relative humidity is not freezable at normal freezing temperatures [Marchand et al, 1994].

An estimate of the maximum crack width not causing freeze/thaw damage is given in [Fagerlund, 1993]. Water filled cracks of widths less than 0.2 mm are not expected to cause damage in a concrete with 2% air, 50 l/m^3 freezable water, and a critical spacing factor of 0.35 mm.

Air voids may become (partly) filled with crystals (e.g. calcium hydroxide, ettringite) during moisture exposure [Christensen et al., 1981] and due to prolonged setting of fresh concrete [Laugesen, 1996]. Furthermore, the finest air voids (diameter <10 μm) will become saturated. Also bigger air voids might become water filled due to dissolution of air [Fagerlund, 1993], [Fagerlund, 1996-b]. This will decrease the available space in air voids, thus increasing the risk of frost deterioration.

Drying and resaturation increase the amount of freezable water. This indicates that drying gives an increase in the volume of large pores and the continuity of the pore system [Fontenay, 1982]. Another possibility is that drying, by increasing the continuity of the pore system, reduces the amount of super-cooled water in isolated capillaries [Fagerlund, 1995-b]. Further drying below 58% appears not to alter the pore structure [Bager and Sellevold, 1986-b].

As drying is likely to occur in-situ, drying of exposed surfaces should be included in the conditioning of specimens for test of frost resistance, especially scaling tests.

The effect of drying on the result of freeze/thaw test results might be both positive and negative, ref. Section 8.

4.1.3 Strength

The stresses caused by ice-formation are so big that the concrete strength will be of very small importance when the concrete is more than critically saturated.

4.1.4 Aggregates

As for the concrete, the frost resistance of aggregates depends on the porosity (amount, pore size distribution, and continuity) and strength of the aggregates as well as the degree of saturation.

Experimental data suggests that most of the time the mechanism of frost action in aggregate particles is in good agreement with the hydraulic pressure theory [Powers, 1955]. The increase in volume during freezing causes either the aggregates to expand or to expel excess moisture. Damage occur when the pressure exceeds the tensile strength.

Based on calculation of the maximum hydraulic pressure developed and the elastic accommodation (possible relaxation] the critical aggregate size below which damage does not occur can be calculated [Pigeon and Pleau, 1995].

Aggregate particles inside the concrete can cause very severe internal damage when they aggregates are big and saturated [MacInnis and Lau, 1971].
The critical size is a function of the permeability and the tensile strength of the aggregate, the amount of freezable water, and the rate of cooling. Critical sizes of two have been

estimated to 1.2 cm (a chert-type aggregate) and 85 cm (a dolomite) (freezing rate 4°C/h) [Verbeck and Landgren, 1960].

4.1.5 Special Considerations regarding High Performance Concrete

Several typical characteristics of high performance concrete are expected to affect the frost resistance, e.g.:

* The low porosity, causing:
    * Low permeability, causing slow moisture transport during, predrying, re-saturation, freeze/thaw testing etc.
    * A smaller amount of moisture to obtain saturation

* Finer pores, causing:
    * Low permeability
    * Lower freezing-point of pore water
    * Larger possibility of super-cooling

* Self- desiccation, causing:
    * an initial low moisture content
    * possible formation of extensive microcracking

All of these characteristics should be considered when recommending methods for freeze/thaw testing of high performance concrete.

The frost resistance of high performance concrete, HPC, is a subject that has attracted intensive interest in the last few years. The practical experience with HPC is limited and laboratory investigations give conflicting results. Consequently there is no consensus amongst researchers on the topic.

If the hydraulic pressure damage mechanism, ref. Section 6.1, is dominant a closer spacing of air voids is needed in order to relieve the pressure because the capillary pores are narrower which leads to a smaller sphere of drainage for each air void. Thus, the requirements to the air void structure may differ from what is the experience with ordinary concrete.

It may, on the other hand, be assumed that a lower air content is sufficient to protect the high performance - paste due to the lower porosity of the paste.

Two schools of thoughts regarding frost resistance of high performance concrete are dominating the discussions today:

* One school claims based on the following argumentation that HPC is frost resistant even without air entrainment.

High performance concrete normally implies the use of low equivalent water cement ratio to create a dense binder with low permeability. In such concrete the amount of freezable water is low partly due to the low porosity with fine pore partly due to the fact that all capillary water is used during the hardening process. Since the concrete is expected impermeable to water the concrete should be frost resistant. Several laboratory tests have indicated this to be true.

* The other school argues that other experiments show, that concrete with even very low equivalent w/c - ratio is not always frost resistant. The reason could be that self desiccation causes micro cracking and that only a small amount of water is needed to create a high degree of water saturation which in a dense concrete increases the risk of frost damage.

The consequence of this is that proper air entrainment is needed to ensure the frost resistance of HPC. The US - SHRP project confirmed this conclusion [Zia, 1994].

4.2 Exposure

4.2.1 Temperature

The temperature affects the amount of ice formed and the rate of flow of water.

Temperature fluctuations during the first four months of the Danish reference year are given in Figure 4.3. 0°C is passed 2x71 times, and the temperature reaches -21°C once and is lower than -15°C 6 times.

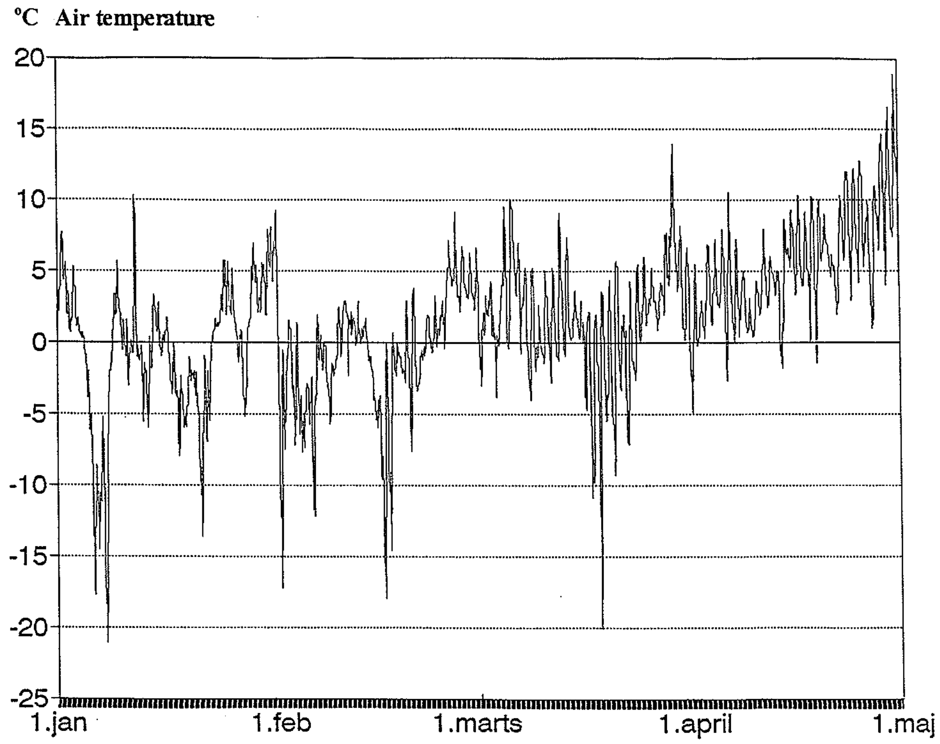

Figure 4.3 Temperature during January to April in the Danish reference year [Nielsen, 1995].

According to [Beton-Bogen, 1985] the rate of temperature lowering of outdoor air in Denmark is 0-3°C/h. The highest value measured is 6.5°C/h, which as a mean is reached once per 12 year. According to [Fontenay, 1982] the maximum rate of temperature lowering obtained in Denmark is 3.3°C/h.

An increasing number of freezing-point passages facilitates the moisture ingress when exposured to salt, ref. Section 4.2.2. An increased rate of freezing and thawing increases the hydraulic pressure, ref. Section 6.1, and it also increases the risk of temperature induced stresses, whereas an increased duration of freezing facilitates the growth of ice.

The rainfall during the same period of the reference year is 146 mm, ref. Figure 4.4.

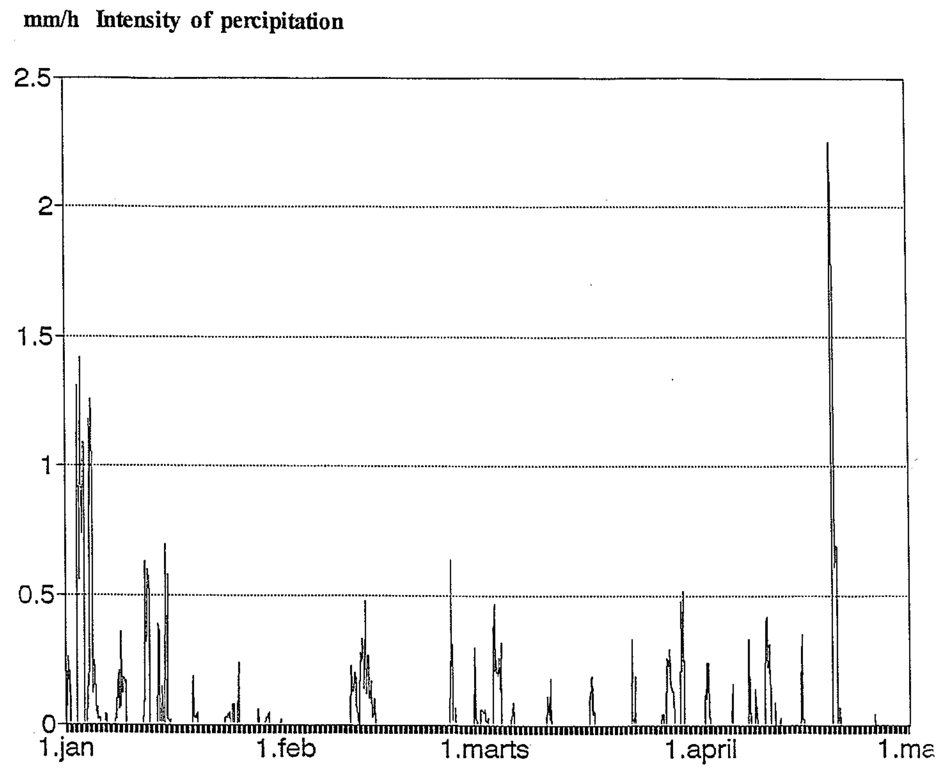

Figure 4.4 Rainfall during January to April in the Danish reference year [Nielsen, 1995].

The rate of freezing appears only to have a marginal effect on the critical degree of saturation, ref. Figure 4.1.

4.2.2 Salt

Exposure to salt/salt solutions increases the freeze/thaw damage, this is illustrated in Figure 4.5. However, there appears to be disagreement as to whether it is the salts dissolved in the pore solution or those present in the solution surrounding the specimen that enhance the deterioration by scaling [Marchand et al., 1994]. Lindmark found that the inner salt concentration had little effect on the frost resistance [Lindmark, 1993].

The effect of salt on the frost resistance has never been fully clarified. There are many hypotheses, a few are presented below.

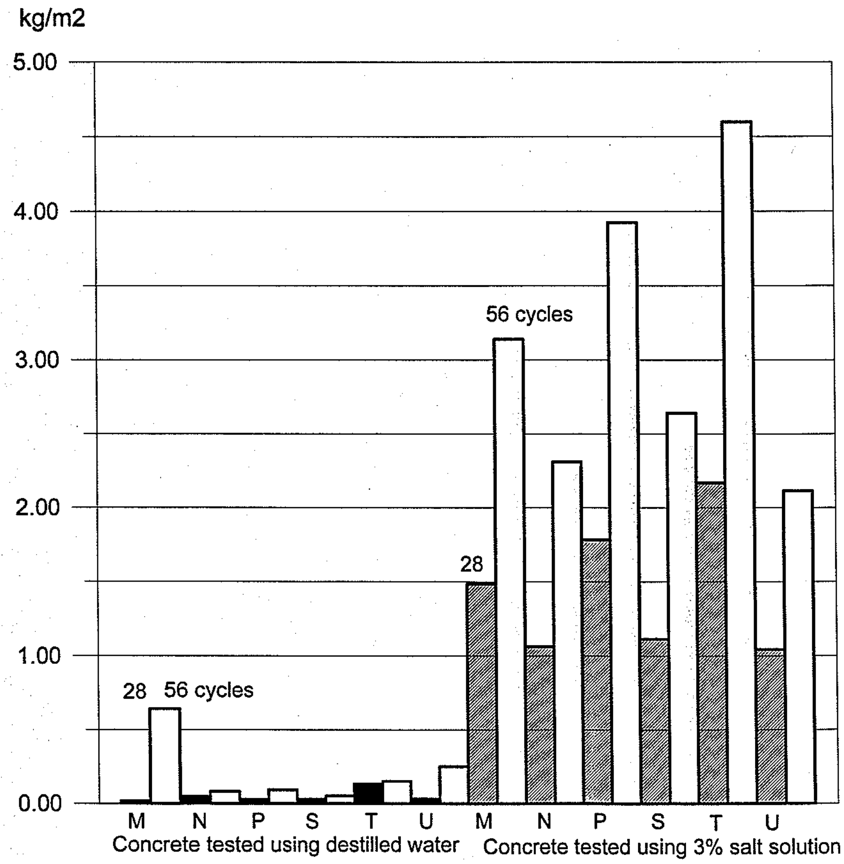

Figure 4.5 Surface scaling of selected concretes (M, N, P, S, T, U) tested according to the Borås method, with water or salt solution [Pedersen and Hougaard, 1994].

For each deicing chemical there appears to be a pessimum concentration at which the damage is greatest. The existence of pessimum concentrations has been explained by the combined action of the total amount of freezable water and the size of the osmotic pressure, and has for four deicing chemicals been found to be 2-4% [Verbeck and Klieger, 1957]. Concerning sodium chloride, exposure to a 3% sodium chloride solution appears to cause most damage. However, it is plausible that the most dangerous salt concentration can be different for different concretes, depending on the binder type and w/c. For very low w/c it seems as if there is small effect of salt concentration [Lindmark, 1993]

The dominating effect of salt might be the increased absorption in the surface layer [Fagerlund, 1993]. Concrete tends to suck water when subjected to repeated freezing and thawing. According to [Fagerlund, 1994] water from inside the concrete might be transferred towards the surface due to concentration differences causing internal drying. Exposure to a salt solution has been reported to cause an increased absorption [Powers, 1945], [Fagerlund, 1992]. See also Section 8.3.2.

The mechanism has recently been described as follows [Geiker and Thaulow, 1996]: Soluble salts will reduce the freezing-point of a liquid. The freezing point of the pore liquid is -1 to -2°C, whereas a 3% sodium chloride solution freezes at -2°C. During melting, the temperature will only increase after all the ice with the actual melting point has melted, ref. Section 3. Thawing of water is accompanied by a volume contraction. If pure ice is present on the surface when the ice in the pores melts, the pores will maintain their original degree of saturation. However, if the surface is exposed to a liquid salt solution the pores may become re-filled. The determining factor is thus the difference in freezing-point between the pore liquid and the liquid of exposure.

Ingress of salt has been said to decrease the amount of freezable water. Due to the very small difference in concentration of salt water and pore liquid this effect is questioned.

The eutectic temperatures and the minimum temperatures for which the de-icing salt is active in practice are listed in Table 4.1 for selected de-icing salts used by the Danish Road Directorate. Potassium is normally only used mixed with sodium chloride.

Table 4.1 Eutectic temperatures and the minimum melting temperatures in practice of selected de-icing salts.

Salt	Eutectic temperature, °C	Minimum melting temperature in practice, °C
Sodium chloride	-21.2 (23.3% sol.)	-6 to -10
Potassium chloride	-51.6 (29.8% sol)	-20 to -25
Urea	-11.7 (33.6% sol.)	-4 to -7

The application of de-icing salt may cause a thermal shock to the concrete due to heat from the concrete absorbed by the melting ice. A temperature profile in an ice-covered concrete during thawing with sodium chloride is given in Figure 4.6.

The significance of thermal shock has never been clarified. It is quite clear, however, that no thermal shock is required for salt scaling to occur. This is found by laboratory testing of concrete in sodium chloride solution. Scaling is obtained despite the fact that the specimen is stored the whole time in the same solution. [Fagerlund, 1996-b].

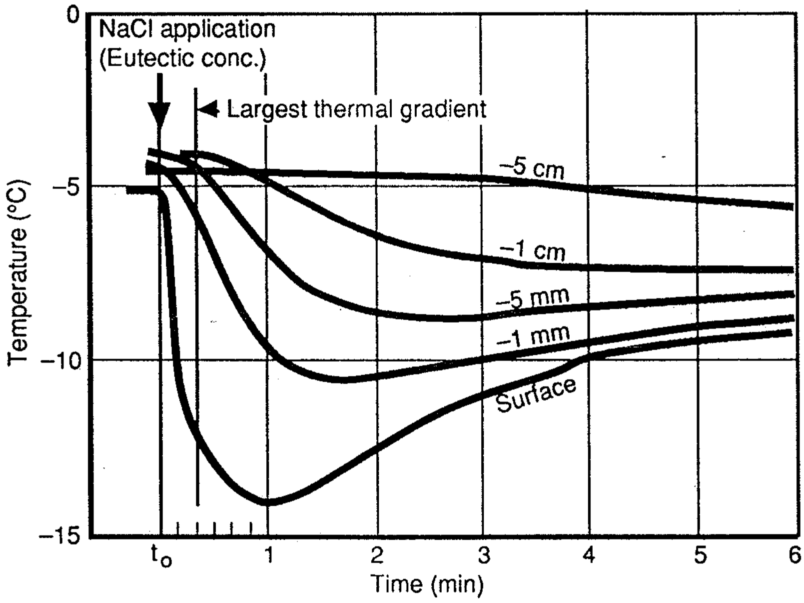

Figure 4.6 Temperature profile in ice-covered concrete during thawing with sodium chloride [Rösli and Harnik, 1980]

5 Selected Equations

Selected theoretically based equations are mentioned below with the purpose of facilitating a later qualitative and a semi-quantitative evaluation of methods for testing the frost resistance of concrete.

5.1 Thermodynamic Issues

Parameters affecting the free energy of the pore solution, soluble salts or capillary condensation, will affect the properties of the solution.

5.1.1 Factors affecting The Properties of the Pore Solution:

5.1.1.1 Chemical Composition of Pore Solution

The pore solution of concrete contains water soluble salts, especially salts of the alkali metals sodium and potassium, Na and K. The chemical potential of a solution is less than the chemical potential of the pure solvent by an amount -RTlnx, where R is the gas constant, T the temperature, and x the molar ratio of the solvent. Several related properties of the solution have their origin in this decreased value of the chemical potential, e.g. freezing-point depression and osmotic pressure.

5.1.1.2 Capillary Condensation

At a given vapour pressure, water can be adsorbed in sufficiently small pores by the process of capillary condensation.

The relationship between pore size and the relative vapour pressure, ρ, is described by the Kelvin-La Place equation for cylindrical pore shape:

$$2\,\sigma_{lg}/r_k = -\,RT\ln\rho/v$$

where σ_{lg} is the interfacial tension between liquid water and water vapour, r_k the Kelvin radius ($1/r_k$ the curvature of the meniscus), and v the molar volume of the liquid water.

Assuming the Kelvin radius, r_k, is equal to the pore size, R, (contact angle = 0), $\sigma_{lg,water}$ = 0.076 N/m, v = $18\text{x}10^{-6}$ [m^3/mole] the relationship between the size of the pores and the relative humidity at which condensation appears can be calculated, ref. Figure 5.1 a. Capillary condensation causes a change in chemical potential and a freezing-point depression of the affected pore solution.

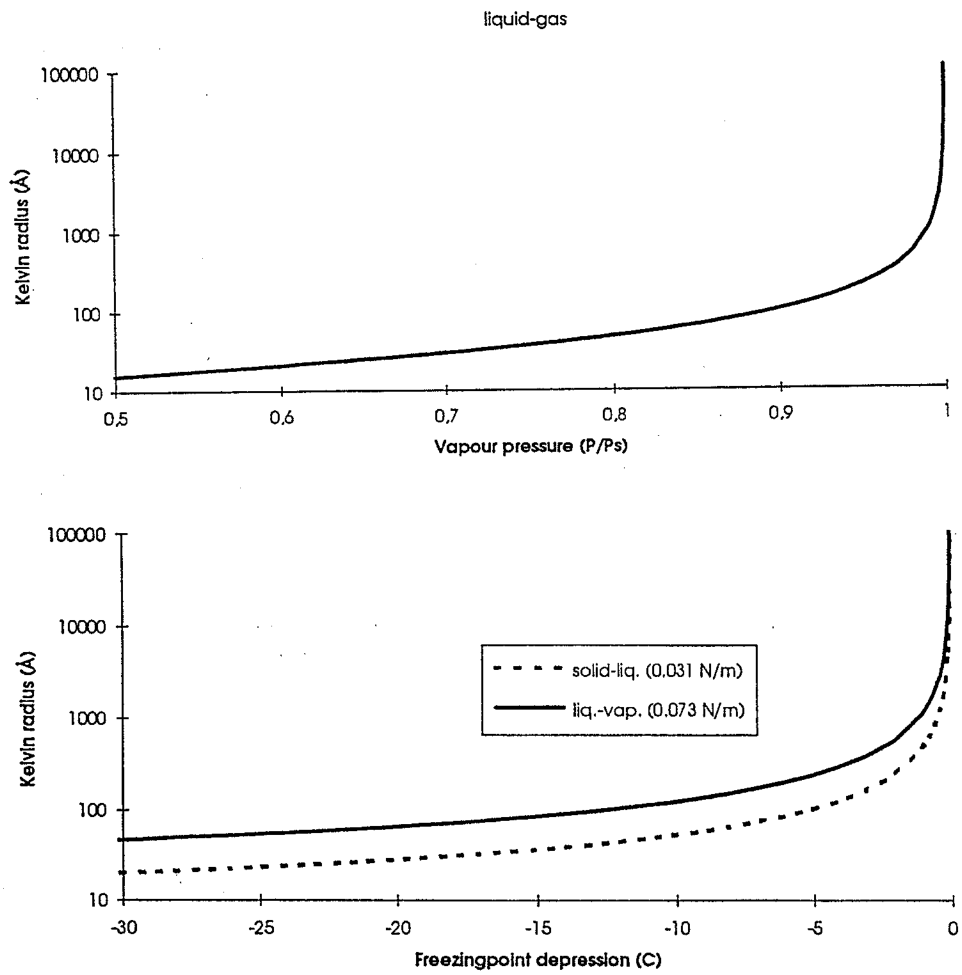

Figure 5.1 Relationship between the size of the pores and a) the relative humidity at which condensation appears and b) the temperature at which ice formation (from pure water) is possible [Jakobsen, 1995].

5.1.2 Freezing-Point Depression

5.1.2.1 Freezing-Point Depression due to Solubles

The freezing-point depression, θ, of a solvent solution with an ionic solute is:

$$\theta = n\,K_f\,\gamma\,m$$

where n = number of ions produced by dissociation, $K_{f,\,water}$ = 1,86 [°C kg/mole], m = molality of the solute [mole/kg], and γ the activity coefficient. $\gamma_{KOH,\,m=0.5} = 0.7$. [Castellan, 1964].

The main solute in pore liquid is potassium salts. The potassium concentration in pore liquid is typically 0.2 - 1.0 mole/kg [Thaulow, 1996], corresponding to a freezing-point depression of approximately 0.5-2.5°C.

5.1.2.2 Freezing-Point Depression in Capillary Pores

The lower pressure in capillary pores causes a decrease in the free energy of the liquid and thus a lowering of the freezing-point [Castellan, 1964]. Ignoring surface adsorbed layers the relationship between the size of the pores and the change in freezing-point, τ, is [Fontenay, 1982]:

$$2\ \sigma/r_k = -\ln\tau\ h_f/v$$

where σ is the interfacial tension between liquid water and ice (0,031 N/m), h_f the heat of fusion (heat absorbed at transformation from solid to liquid water: 333.6+2.22T [kJ/kg], T in °C) [Jakobsen, 1995]. The tensile stress in the unfrozen layer may be governed by the surface tension between either water and ice or water and vapour. The correlation between pore size and freezing point is illustrated in Figure 5.1 b. The result depends on which surface tension is used. There are also other possibilities of expressing the relation between pore radius and freezing point [Defay, Prigogine, Everett, 1966]. It has never been clarified which expression is correct.

5.1.3 Osmotic Pressure

The osmotic pressure, π, of a solution relative to the pure solvent is depends on the concentration of solutes:

$$\pi = n\ \gamma\ c\ R\ T$$

where c [mole/l] is the molar concentration.

The osmotic pressure of the pore liquid and of typical Danish sea water relative to pure water ($[Na^+]$ = 12 [g/kg]) is approximately 12 atm and 5 atm, respectively.

5.2 Super-Cooling

Super-cooling is delayed freezing, and can be observed as hysteresis between freezing and thawing temperatures. Super-cooling is due to a combination of delayed nucleation and the so-called ink-bottle effect. Freezing of cement paste at a rate of 3.3°C/h has been observed to cause a super-cooling of 5-7°C [Fontenay, 1982].

Freezing of super-cooled pore liquid may happen momentarily and affect a large amount of the pore liquid. This 'shock-freezing' has been reported to cause damage [Grübl and Sotkin, 1980]. According to [Fagerlund, 1993], super-cooling is normally not assumed to cause damage. This is seen in dilation tests where the nucleation of super-cooled water is seen as a small and rapid increase in length. The expansions is, however, considerably smaller than the fracture strain. [Fagerlund, 1996-b].

Part of the effect attributed to super-cooling, ref. above, might be due to the liquid-gas surface tension governing freezing, but the liquid-solid surface tension governing melting [Sellevold, 1979].

5.3 Thermal Induced Stresses

Thermal induced stresses may cause cracking if the tensile strength is exceeded. Thermal stresses may be due to:

* Thermal incompatibility between aggregates and matrix
* Temperature difference between centre and surface of the test specimens

5.3.2 Thermal Incompatibility

Selected coefficients of thermal expansion are listed in Table 5.1

Table 5.1 Thermal coefficient of expansion (linear).

Material	Thermal coefficient of expansion, $x10^{-6}/°C$
Cement Paste	10-30 Note 1, 11-16 Note 2
Concrete	10 Note 1
Water	16 at 5°C, 88 at 10°C Note 4
Ice	51 Note 5
Limestone	0.9-12.2 Note 2, 2.8 Note 3
Sandstone	4.3-13.9 Note 2
Quartzite	10.7 Note 3
Flint, Møn	13.9 Note 3; Chert 7.4-13.1 Note 2
Granite, Rønne	4.1 Note 3
Granite, Red Sweden	4.6 Note 3
Granite, Norway	5.0 Note 3
Diabase, Halmstad	8.5 Note 3
Basalt, Norway	9.0 Note 3

Notes: 1: [Beton-Bogen, 1995], 2: [Neville, 1981], 3: [Nielsen, 1970], 4: [Handbook, 1982), 5: [Håndbog, 1978]

Cracking in the paste will occur when the ultimate tensile strain capacity (ϵ approximately 100×10^{-6}) is exceeded. Ignoring effects as creep and relaxation the minimum temperature fluctuation, ΔT, causing cracking can be estimated from:

$$\Delta T < \epsilon / \Delta\alpha$$

where $\Delta\alpha$ is the difference in thermal coefficient of elongation. $\Delta\alpha = 5 \times 10^{-6}$ results in $\Delta T < 20°C$.

It has been stated that a difference in thermal expansion coefficients above $5.5 \times 10^{-6}\ °C^{-1}$ may affect the durability of concrete exposed to freezing and thawing [Neville, 1981].

Tests reported in [Sellevold, Jacobsen, and Bakke, 1993] where concrete specimens were exposed to cyclic temperature variations between approximately +40°C and 0°C with about the same rate as on an ordinary freeze/thaw test resulted in no destruction of the concrete, indicating that ice formations is required for frost damage to occur.

5.3.2 Temperature Difference

Increased cooling/heating rate and specimen size will increase the risk of thermal cracking. Rapid cooling may cause surface cracking, whereas rapid heating may cause internal cracking. The influence of temperature change on the stress development has been estimated, ref. Figure 5.2. Cracking may occur when the tensile stress/tensile strength ratio exceeds 1. Thus, to limit the risk of temperature induced cracking the cooling and heating rate should be kept below 50°C/h for a maximum surface - centre distance of 150/2 mm.

In this calculation concrete is assumed to be a homogeneous, isotropic material (i.e. no difference between paste and aggregates).

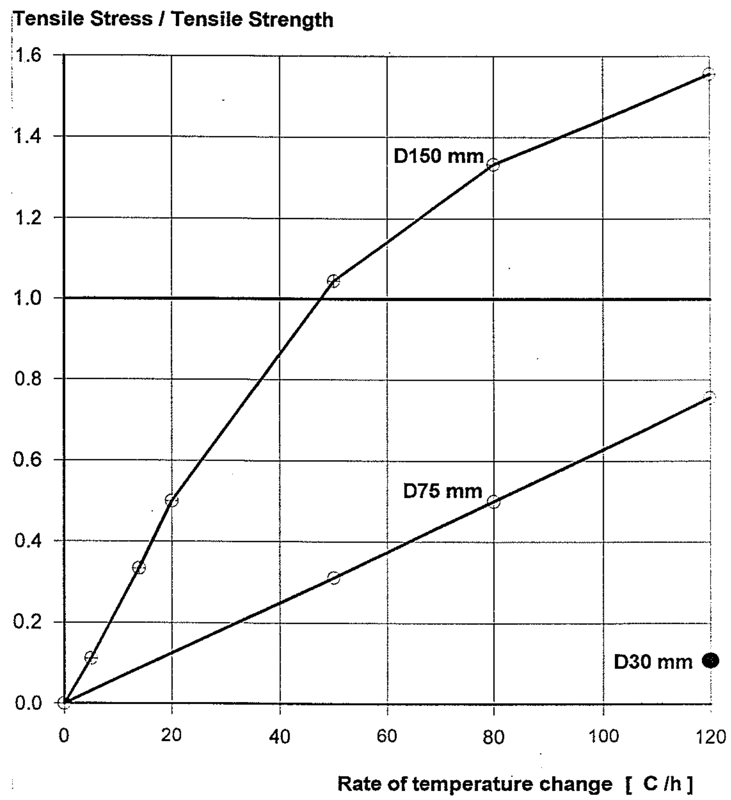

Figure 5.2 Effect of the rate of temperature change on the risk of cracking (stress/strength) for selected specimen sizes (diameter 30, 75, and 150 mm, infinite length assumed) [Pedersen, 1996].

5.4 Moisture Ingress

The moisture content in concrete increases due the action of one or more of the following mechanisms.

* Diffusion of water vapour
* Osmotic pressure
* Permeation
* Capillary suction
* Cyclic thawing and freezing
* Diffusion of air from air voids.

Osmotic pressure is dealt with in Section 5.1.2. Diffusion of vapour is not considered relevant here because it will only cause low water content inside the concrete, neither is permeation.

Capillary suction and freeze/thaw cycles produce so high moisture contents that frost damage might occur.

Diffusion of air from air voids is a very slow process but results in very high moisture contents in concrete that is stored in water for a long time.

In concrete surfaces not permanently exposed to water the moisture content may fluctuate due to alternating drying and wetting.

The rate of moisture ingress depends on the exposure, the materials structure, and the actual moisture content and distribution. Thus, i.a. the pre-conditioning of test specimens affects the rate of ingress.

Models for the various transport mechanisms are given, e.g. in [Kropp and Hilsdorf, 1995].

A theory has been developed for calculation of the moisture content in concrete at varying outer moisture conditions (liquid water and/or vapour) [Fagerlund and Hedenblad, 1993].

5.4.1 Capillary Suction

Owing to irregularities of the pore system, real materials differ from the theoretically based formula. Therefore, empirical relations are established to describe the uptake of a liquid as a function of time. For short term contact to a liquid, the rate of the uptake is referred to as initial absorption rate, a, [$g/m^2\ s^n$]:

$$a = m/A\ t^n$$

where m is the amount of absorbed liquid [g], A the area in contact with water [m^2], t the time, and n is a constant, typically 0,5.

5.4.2 Cyclic Thawing and Freezing

Cyclic thawing and freezing is observed to increase the rate of moisture ingress, ref. above. At present a qualitative model is available that explains the effect [Geiker and Thaulow, 1996]. Quantification of this model is needed.

5.4.3 Diffusion of Air from Air Voids

According to [Fagerlund, 1993] in each air void an air bubble is enclosed during the capillary absorption process. This bubble is exposed to an over-pressure due to the current air-water meniscus. Consequently, the solubility of air is increased and the air is therefore gradually dissolved in the pure-water and diffused to a coarser air bubble or the surface of the concrete. Air is replaced by water. Therefore, the water content gradually increases to very high and dangerous levels. The process has been described and quantified.

6 Freeze/Thaw Damage, Selected Hypotheses

Several models explaining the freeze/thaw deterioration of concrete have been proposed, e.g.

* The hydraulic pressure theory
* The osmotic pressure theory
* The microscopic ice lens growth theory
* Thermal incompatibility between matrix and aggregates
* Shock freezing.

None of the proposed mechanisms can explain all the in-situ and experimental observations. There is no consensus regarding mechanisms responsible for damage in cement paste [ACI C 201, 1995].

The basic principles of frost deterioration are dealt with in so far as they have a direct influence on the evaluation of the existing test methods. Below, a brief introduction to the above proposed mechanisms are given; shock-freezing and thermal incompatibility being dealt with in Section 5.2 and 5.3.

6.1 The Hydraulic Pressure Theory

Powers, in his early work, proposed that ice nucleates and grows in capillary pores; forcing them to dilate or expel excess water from the freezing sites. Elevated hydraulic stresses arise as water is expelled due to the relatively low permeability of the cement paste. It was recognized that distance from the void boundary, the degree of saturation, and the rate of freezing would influence the magnitude of hydraulic stress build-up [Powers, 1945].

Based on the hydraulic pressure theory the correlation between the permeability of the matrix, the air void structure, and the rate of freezing was established. To minimize the risk of frost damage (for a given concrete composition and freezing rate) the distance between the air bubbles should be below a certain critical spacing factor [Powers, 1949], [Powers and Helmuth, 1953]. The correlation between the rate of freezing and the critical spacing factor is illustrated in Figure 6.1. The critical spacing factor is normally deter mined by comparing the result of freeze/thaw tests with the measured spacing factor.

Since different test methods introduce different amounts of water in the specimen the critical spacing factor determined will be a function of the test method characteristics. A "moist test" gives a lower value than a "dry test". This is further discussed in [Fagerlund, 1992].

From the value of the critical spacing factor, the required air content can be calculated [Powers, 1949].

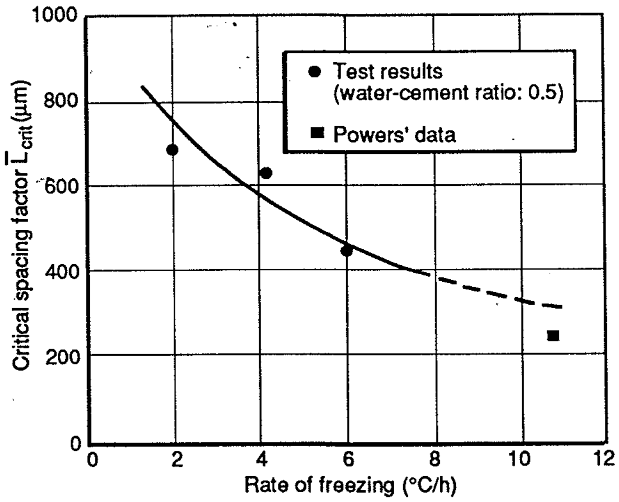

Figure 6.1 Correlation between critical spacing factor and freezing rate (testing according to ASTM C 666) [Pigeon et al., 1985].

Although the rate of ice formation is lower at decreased temperature, the increased expansion at lower temperatures, often observed, is explained by a lower permeability caused by ice formation closing some of the pores [Fagerlund, 1993].

The hydraulic pressure theory can not explain several features of frost damage, e.g. the effect of the minimum freezing temperature, the duration of freezing, damage due to freezing of non-expanding liquids, the effect of deicing salt [Marchand et al., 1994], and the effect of the total amount of ice formed [Jakobsen, 1995]. The freezing rate has been found to have no effect on salt scaling [Sellevold and Farstad, 1991].

The existence of a critical spacing factor predicts the existence of a critical degree of saturation [Fagerlund, 1979], ref. Section 4.1.

6.2 The Osmotic Pressure and Ice Lens Growth Theories

The hydraulic pressure concept was modified when experiments showed significant evidence that moisture was moving toward rather than away from reezing sites. There are two possibilities, osmotic pressure and ice lens growth. Below the theories are descriped separately, althoug they could well supplement each other [Fagerlund, 1996-b].

6.2.1 The Osmotic Pressure Theory

An osmotic pressure gradient, arising from solute rejection as relatively pure ice grows from alkaline pore solution leaving the remaining pore solution with a higher concentration of solutes (soluble salt), was proposed to be the cause of the movement of water and pressure build up. Continued freezing in pores would gradually increase the solute concentration in the remaining pore solution, hence increasing the osmotic pressure gradient (ref. Section 5.1) drawing water from capillaries and gel pores e.g. [Powers and Helmuth, 1956].

The osmotic pressure theory can explain the effect of the duration of the freezing period.

6.2.2 The Ice Lens Growth Theory

Progressive ice accretion in cracks during periods of sustained low temperatures slightly below 0°C may generally dominate the deterioration of concrete in many environments [Litvan, 1978].

Super-cooling of the pore liquid may take place during cooling of the concrete, ref. Section 5.2. The super-cooled liquid is thermodynamically unstable. Super-cooled liquid (typically in smaller pores) in contact with ice (typically in larger pores) will tend to flow towards the ice, thus facilitating ice lens growth.

Ice lens growth has also been explained by larger ice crystals feeding on smaller crystals [Kukko, 1992]. The phenomena is known as Ostwald ripening.

Ice lens growth is favoured by increased duration of the freezing period.

7 Selected Test Methods

Below, selected test methods are described and briefly evaluated. A more detailed discussion of the effect of the various test parameters is given in Section 8. To provide an overview, the test parameters have been summarized in Table 7.1 (appended).

7.1 ASTM-C 666, Resistance of Concrete to Rapid Freezing and Thawing

Principle

Concrete samples surrounded by 1-3 mm tap water are exposed to 300 freeze/thaw cycles with moderate to relatively large temperature gradients. Possible types of damage are internal cracks and, to some extent, surface scaling. The cracks reduce the fundamental frequency of transverse vibration. The square of this is by the method assumed to be proportional to the dynamic E-modulus, E_{dyn}. The cracks also increase the length of the test specimens.

According to the test method description, the test procedure is not expected to markedly influence frost resistant concretes, defined as:

* Concretes being not critically saturated or
* Concretes being produced with frost resistant aggregate, having a proper air void system and having reached appropriate maturity to avoid critical degree of saturation under normal circumstances.

Test Specimens

Cast prisms or cored cylinders with a diameter of 76-127 mm and a length of 279-406 mm.

Conditioning

Cast samples are water cured for 14 days and mounted with strain gauge studs. Cored samples are protected against drying, mounted with strain gauge studs, and saturated in lime water until 'constant weight', i.e. approximately 3 days.

Test Procedures

Each specimen is placed in a container surrounded by 1-3 mm tap water. The container is placed in a bath containing e.g. glycol and is exposed to temperature cycles (2-5 hours), each: +4.4 °C → -17.8 °C → +4.4 °C. The temperature is measured centrally in a dummy.

E_{dyn} of the test specimen is measured with a resonance frequency tester according to ASTM C 215. The length is recorded with a dial gauge. E_{dyn} and length of each test specimen is recorded at the initiation of the testing and at intervals of no more than 36 cycles.

The testing proceeds until 300 cycles, but should be terminated if E_{dyn} decreases to 60 % of the initial value - expressed in terms of the following relation: $E_{dyn,n}/E_{dyn,o} = f_n^2/f_0^2$ where f is the fundamental frequency and n is number of cycles - or if the length change exceeds 0.10 % of the initial length. The relative E_{dyn} and the relative length change are calculated and given as test results.

Evaluation of Test Results

The method does not declare conformity criteria for the test results.

Advantages

The method is widely used, mainly in the USA and Canada, and hence much data can be provided.

The application of rather large test specimens sustains that the material is representative.

The determination of E_{dyn} is performed with acceptable accuracy. The length measurements have high precision [Pigeon et al., 1986]. The change in E_{dyn} and the relative length change during the freeze/thaw test correlates well.

The testing limits of E_{dyn}=60 % and length change of no more than 0.10 % (not to mistaken with conformity criteria) correlate well with a visual evaluation of the degree of damage [Laugesen, 1996].

Disadvantages

To avoid alien stresses in test specimens during freezing and thawing, it is prescribed to omit samples containing rebars. When cores are taken from reinforced structures, excessive coring may thus be needed to obtain cylinders of sufficient size without rebars.

The measurement of E_{dyn} may give erratic results:

* If local variations in the crack formation develop during the test.
* If the transmission is reduced, e.g. by placing the oscillator or the transducer upon an aggregate or a part of the paste/mortar which is partly spalled off the test sample.
* If there is surface scaling, the fundamental frequency increases due to the loss in weight and moment of inertia. Thus, one might believe that the concrete is less damaged in its interior than it is [Fagerlund, 1996-b].

To reduce the possible sources of error, the test specimen should be placed in the exact same way during all measurements. Furthermore, it has to be secured that the contact points are of acceptable quality.

Duration

Conditioning: approximately 3 days, testing procedures: 42 days. Total duration: 45 days.

Cost Level

DKK 10,000 per set of 6 cores.

7.2 Modified ASTM C 671, Critical Dilation

Principle

A long term water-stored concrete specimen is subjected to a slow freezing during which the length change of the specimen is continuously recorded. If the internal expansion caused by freezing pore water gives rise to intrinsic cracking, a discontinuity will appear on the length-versus-time contraction curve. This takes place at a temperature corresponding to the initial freezing point in the concrete pore system, normally around -5 °C for ordinary concrete and at much lower temperature for dense concrete [Fagerlund, 1996-b].

The test method is based on *ASTM C 671-94: Standard Test Method for Critical Dilation of Concrete Specimens Subjected to Freezing*. In the applied version some changes and simplifications have been introduced in order to apply with the [Øresundsundskonsortiet, 1994] and in order to make the test easier to perform. The modified version has been applied in [Hansen, 1995]. These changes include the use of air as cooling media, where kerosine or silicone oil is prescribed in the ASTM method.

Furthermore, the temperature range for the cooling process has been extended from 1.7 °C → -9.4 °C to 20 °C → -20 °C and finally only one freeze/thaw cycle is prescribed, whereas the original ASTM method operates with several subsequent freeze/thaw cycles of each test specimen with 2 week intervals, and water storage of the test specimens between each cycle.

Test Specimens

75 mm in diameter, 150 mm in length cast concrete cylinders. Other dimensions or drilled cores can also be tested, however comparisons must be based on identical specimen size. According to [Øresundsundskonsortiet, 1994], a test series contains 12 specimens, two tested at a time at each of 6 terms.

Conditioning

Prior to testing, the specimens are stored in water at 20 (±2)°C. The test of cast concrete cylinders is typically carried out at different maturity ages, e.g. 3, 8, 12, 16, 20 and 24 weeks. Before start of freeze/thaw exposure, the specimens are taken out of the water, dried, and wrapped in plastic film in order to avoid loss of water by evaporation during the testing.

Test Procedure

The test specimens are fitted with axially centred gauge studs and placed in a measuring frame in order to be able to register the length change during the freeze/thaw procedure. The measuring frame is constructed with invar steel rods and equipped with an appropriate length measuring device connected to a data logger. Each measuring frame is calibrated with an invar steel bar with known thermal characteristics in order to determine the movements of the frame during the freeze/thaw cycle. The test is carried out in an insolated cabinet with circulating air, which is cooled from +20°C to -20°C at a constant rate of 2.8 (±0.5) °C/hour. At the end of the cooling period, the specimens are reheated from -20°C to +20°C at a rate of 2.8 (±0.5)°C/hour. Hence, the whole test period has a duration of approximately 28 hours. The temperature is monitored and controlled by means of temperature sensors placed at the surface and centre of a dummy concrete specimen situated in the immediate vicinity of the actual test specimens.

The temperature and length change development corrected for the contraction of the test frame is illustrated on a graph. The frost resistance of the concrete can be evaluated from the shape of the curve for the cooling process.

Evaluation of Test Results

If a discontinuity, i.e. an abrupt change or even an expansion, of the length vs temperature curve at temperatures below the freezing point is observed, the concrete cannot be characterised as frost resistant.
However, it is not stated what the magnitude of this discontinuity must be to fail conformance, ref. below. A reasonable conformance criterion might be a certain fraction of the fracture strain, e.g 0.005 % or 0.01 % [Fagerlund, 1996-b].

Advantages

The method is quick. An evaluation of the frost resistance of a given concrete can be given after a test with a duration of 28 hours. It should be noted, though, that the required maturity is minimum 3 weeks and at proceeded testing up to 24 weeks [Øresundskonsortiet, 1995].

Disadvantages

Advanced equipment for temperature control and length change measurements is required.

The interpretation of the results can be difficult, as experience with the modified method is limited. Furthermore, no quantitative requirements to the allowed magnitude of dilation currently exist.

The duration of the test period (28 hours) can give rise to practical problems when repeated tests are performed.

Duration of Test
A test specimen can be tested within two days. For testing of new concrete the total test period including conditioning can be up to 24 weeks.

Cost Level
DKK 12,000 per set of 12 specimens.

7.3 Modified SNV 640461 (Dobrolubov/Romer)
Principle
Small concrete test specimens are exposed to 350 rapid freeze/thaw cycles with large temperature gradients by automatic lowering the samples into 20 °C tap water and -20 °C chloride solution, respectively. According to the method, the concrete is not frost resistant, if the freeze/thaw cycles result in internal cracking of the test specimens giving way to expansion. The relative length change of the test specimens is recorded.

The test method has been applied in Denmark for approximately 20 years, partly with the following modifications:

* Test specimen length: *60 mm* changed to *70 mm*.
* Length measurement: *Projection measure* changed to *axial measure*.

The length measurement method was modified in order to improve the repeatability of measurements.

Test Specimens
Concrete prisms 30 mm x 30 mm x 70 mm, cut from larger cast or cored samples. A test series contains 6 prisms of the same concrete.

Conditioning
At a maturity of at least 28 days, the prisms are cut and mounted with strain gauge studs centrally on the ends of the elongated prism. The samples should be protected against drying. Subsequently, the prisms are water saturated for 5 days or until constant weight.

Test procedure
A total of 350 freeze/thaw cycles are carried out. During each cycle, the test specimens are placed in a cooling bath with -20 °C saturated $CaCl_2$ solution for 20 minutes and subsequently in a heating bath with +20 °C running tap water for 10 minutes.

The length of the test specimens is recorded at the initiation of the test and after each 50 freeze/thaw cycles. The relative expansion is calculated for each term. The mean expansion value of the 6 prisms are for each term plotted in a diagram also containing the conformity criteria.

Evaluation of Test Results
The conformity criteria after 350 cycles are:

* Expansion < 0.10 %: High frost resistance
* Expansion 0.10-0.20 %: Moderate frost resistance
* Expansion > 0.20 %: Low frost resistance.

Advantages
The test is the fastest of the multi cycle freeze/thaw test methods. The small size of the test prisms facilitates testing of many concretes at a time.
The conformity criterium correlate reasonably well with a visual estimate of the degree of deterioration.

The experience with the test method in Denmark indicates that it can be used to approve concretes as being frost resistant: Concretes evaluated by the test as having high frost resistance have so far performed well in the structures.

Disadvantages
Small cut test specimens cause a high content of coarse aggregate to be directly exposed. The role of the transition zone between paste and aggregate may thus be exaggerated in comparison to its influence in concrete structures. However, it can not be excluded, that the poor performance observed in test specimens originally containing various plastic debonding phenomena in the microstructure [Laugesen, 1996] also infers a low frost resistance in the actual structures. If that is the case, the described disadvantage of small specimen size may be an advantage.

Test specimens are susceptible to drying (even in normal laboratory atmosphere), e.g. during the cutting and mounting of strain gauge studs. Test results have indicated that drying of the prisms has reduced the frost resistance [Laugesen, 1996]. The test specimens should thus be carefully protected against drying.

The method can not be used for rejection of concretes as being not frost resistant, since concretes evaluated by the test as having low or moderate frost resistance, may in concrete structures show either good or poor frost durability [Puckman et al, 1988], [Henrichsen, 1995].

The modified length measurement (in the dimension of the length measured), has shown not to correlate perfectly with the original method (projected measurement) [Laugesen, 1989]. It is inferred that the differences in placing of gauge studs cause the apparent delay in development of expansion, since the initial expansion of the test specimens are measured only with the modified method.

Testing in saturated $CaCl_2$-solution has been questioned for two reasons:

1. Concrete is attacked chemically [Verbech and Klieger, 1956], [Petersson 1984].
2. The concrete is dried intrinsically due to the very low vapour pressure of saturated $CaCl_2$. The drying effect is very big [Petersson 1991].

Experience from petrographic analysis does not confirm the chemical attack as being a main deterioration mechanism [Laugesen, 1996].

Duration

Conditioning: approximately 5 days, testing: 8 days. Total duration: 13 days.

Cost Level

DKK 5,000 per set of 6 prisms.

7.4 SS 13 72 44 (The Borås method) Scaling at Freezing

Principle

The method is originally modified from ASTM C 672 in order to imitate the natural temperature variations as regards velocity. Freeze/thaw exposure is carried out one-dimensionally on the upper horizontal surface, while the remaining surfaces are insulated against humidity and heat transfer. The exposed surface is covered with water or a saline solution and the action is obtained by cooling and heating during cycles of 24 hours. The exposure may result in scaling of the surface. Furthermore, frost pop-outs may form due to deterioration of stones. The amount of scaled material expressed as weight per unit area is registered after given numbers of freeze/thaw cycles.

Test Specimens

Cast or drilled samples. The total area exposed should be over 50000 mm^2.

Curing

Cast specimens: In water for 6 days + 12 days at 60 (±20) % RH and 20 °C. Subsequently 2+7 days in climate chamber at 65 % RH and 20 °C. 3 days covered with water from tap on the exposure surface.

Cored specimens: 7 days in climate chamber as above + 3 days covered with water as above.

Test Procedure

The test specimens are wrapped in a rubber cloth, which is glued on to the surfaces not to be exposed. The rubber cloth is then thermally insulated. The rubber cloth is placed in such a way that a boundary is established to the free surface that is covered with water. The surface is covered with either a 3% NaCl solution (method A) or pure water (method B) to a height of 3 mm immediately before the specimens are placed in the freeze/thaw chamber. Evaporation from the water surface is prevented by a plastic cover.

The temperature cycles shall be within the following range of temperature variations:

* Cooling from +20 °C to -4 °C during a period of 4.5 hours = 5.3 °C/h
* Cooling from -4 °C to -18 °C during a period of 7.5 hours = 1.9 °C/h
* Constant low temperature -18 °C for 4 hours
* Heating from -18 °C to +20 °C during a period of 8 hours = 4.8 °C/h

The temperature is registered by a thermocouple in the middle of the water/saline solution of a sample, e.g. a concrete dummy.

The scaling of material from the exposed surface is registered after 7, 14, 28, 42, and 56 cycles and in case of extended procedure after a further 70, 84, 98 and, 112 cycles.

According to the Swedish Bridge Norm (BRO 94), 112 cycles shall allways be used when the concrete contains microsilica, due to the fact that such concrete often exhibit accelerated scaling occuring after 56 cycles.

Evaluation of Test Results

The conformity criteria for ordinary concretes are based on mass of scaling at 28 days (m_{28}), 56 days (m_{56}), and, optionally, at 112 days (m_{112}):

* Very good: m56 average < 0,10 kg/m^2

* Good: m56 average < 0,20 kg/m^2 or
  m56 average < 0,50 kg/m^2 and m56/m28 < 2 or
  m112 average < 0,50 kg/m^2

* Acceptable: m56 average < 1,00 kg/m^2 and m56/m28 < 2 or
m112 average < 1,00 kg/m^2

* Unacceptable: the above not complied with.

Advantages
The method represents an action frequently occurring in practice. The test specimens are big. The estimation of the results is quantitative.

When the test procedure is followed the reproducibility and the repeatability of the method is acceptable [Pedersen. 1996]. Apparently the method is not influenced by possible reinforcement bars in the test specimen. The test equipment and control systems are ordinary laboratory equipment.

Disadvantages
No natural ageing of the surface, such as carbonation, could take place during the test. Therefore, the test result might be too pessimistic for concretes for which carbonation is postive with regard to salt scaling (e.g. OPC-concretes) [Petersson, 1995], and too optimistic for concretes for which carbonation is negative (e.g. slag cement concretes) [Stark & Ludwig, 1995; Matala, 1995]. Since an optimistic result is dangerous the test is not suitable for concrete containing slag cement.

Only the surface is assessed and possible internal crack formations is not accounted for. This could be done by measuring the dynamic E-modulus by means of ultra sonic velocity [Jacobsen et al., 1996].

Special Conditions
Preparing the test specimens, it is of importance to secure that the rubber clothing is tight at the edge of the surface to be exposed. This to ensure that no water can pass along the side of the specimens. Furthermore, it is necessary during the test to inspect and maintain the thin layer of water at the surface. If the freeze/thaw system is not controlled by the actual temperature recorded in the exposure salt solution at one specimen, the number of specimens in each freeze/thaw chamber should be held constant, e.g. by use of dummies. This should be documented.

Duration
Curing for 10 days, testing for 56 days or 112 days. Total duration 65 days (or 121 days).

Cost Level
DKK 4,500.- per set consisting of 5 test specimens

7.5 RILEM Draft Recommendation: 117-FDC Freeze/Thaw and Deicing Resistance of Concrete: 1: Tests with Water (CF) or with Sodium Chloride Solution (CDF)

Principle

Concrete samples partly immersed in salt water are exposed to 28 freeze/thaw cycles with moderate temperature gradients. The freeze/thaw procedure causes the exposed surface to crumble or scale if the concrete is not frost resistant.

Test Specimen

Cast 'half-cubes, 150 mm x 150 mm x 75 mm, produced by casting in a cube form modified by a vertical teflon insert placed in the centre of the form. The plane of each half cube that is cast against this teflon plate is the surface to be exposed. The surface planes perpendicular to the exposed surface are sealed by a plastic coating.

For samples cored from structures, this method is intended for testing of the exposed surface. No dimensions are given for cored samples. The maturity must at least be as for the cast cubes.

Conditioning

6 days water curing after de-moulding. Air conditioning for 21 days at 20 °C and 65 % RH. Subsequently, the test specimen is resaturated by immersing the test surface of the sample in demineralised or distilled water for 7 days. During the resaturation, the sample weight is recorded.

Test procedure

The samples are placed in a test container with the exposure surface immersed in a solution of 3 % NaCl in demineralised or distilled water. The closed test container is partly submerged in a temperature bath with e.g. glycol and exposed to temperature cycles: cooling: +20 °C → -20 °C during 4 hours; constant temperature at -20 °C for 3 hours; heating: -20 °C → +20 °C during 4 hours; constant temperature at +20 °C for 1 hour. The temperature is measured in the temperature bath.

The test proceeds until 28 cycles. After (4, 6,) 14, and 28 freeze/thaw cycles the scaled material is dried and weighed. The scaled material is removed from the exposed surface of the test specimen by cleaning in an ultra sonic bath. This is done during the time interval when the temperature is above 15 °C.

Evaluation of Test Results

Conformity criterium after 28 freeze/thaw cycles: scaling < 1500 g/m^2.

Advantages
The temperature cycle is well determined due to cooling/heating in well stirred glycol bath.

Tests according to the actual method has not been performed by the present authors. Quite some work has been carried out describing the precision of the test [Hartman 1992], [Setzer and Auberg 1995], [Setzer et al. 1995].

Consequently no further advantages /disadvantages of the test method have been described here.

Disadvantages
Scaling might also occur on the side surfaces under the plastic coating. Thus, the scaling per bottom surface area might be exaggerated [Fagerlund, 1996-b].

Duration
Conditioning: 14 days, testing: 14 days. Total duration: 28 days.

Cost Level
DKK 4,500 per set of 4 cubes.

7.6 RILEM Draft Recommendation: 117-FDC Freeze/Thaw and Deicing Resistance of Concrete: 2: Slab test
This test method is identical to SS 13 72 44, Borås, except for a more well defined conditioning at the sample maturity of 7-19 days:

* Slab test: 65 (±5) %RH.

* Borås: 60 (±20) %RH.

No conformity criteria are given in this test method.

For description of test, see Section 7.4.

7.7 RILEM Draft Recommendation: 117-FDC Freeze/Thaw and Deicing Resistance of Concrete: 3: Cube Test
Principle
Concrete samples immersed in 3 % salt solution or in tap water are exposed to 56 freeze/thaw cycles. The cooling gradient is moderate to low, the heating gradient is high. The amount of surface scaling caused by the freeze/thaw procedure is measured.

Test Specimens
Cast cubes, 100 mm x 100 mm x 100 mm. Alternatively cored samples from structures.

Conditioning
Water curing for 6 days after de-moulding. Air conditioning for 20 days at 20°C and 65 % RH. Resaturation during 1 day, by immersing the test specimen in a closed test container with a 10 mm thick layer of tap water or 3 % salt solution surrounding the sample (20 mm on the surface). The absorption during this 1 day resaturation is measured.

Test Procedure
The closed test container with the concrete sample is exposed to 56 freeze/thaw cycles with temperature range from +20°C to -15°C. The controlling temperature is measured centrally in a concrete dummy. On cooling, the test container is immersed in a temperature bath for 16 hours: +20°C → 0°: 2 hours, 0°C → -15°C: 14 hours. Subsequently, the test container with the sample is transferred to a heating bath of 20°C for 8 hours.

The testing proceeds for 56 freeze/thaw cycles. After 7, 14, 28, 42, and 56 cycles, the mass of the scaled and dried material is measured.

Evaluation of Test Results
The test method does not declare conformity criteria.

Advantages
No information regarding specific advantages have been found.

Disadvantages
Scaling is not homogeneous. Normally, most of the scaling occurs at the bottom surface and at the bottom part of the 4 side surfaces, whilst the top surface often is unharmed or only scaled to a minor extent. Thus, the reported loss expressed in kg/m_2 cannot be directly translated to 'depth of erosion' [Fagerlund, 1996-b].

Duration
Conditioning: 20 days in air, 1 day in water (fresh or salt); testing: 56 days. Total duration: 77 days.

Cost Level
DKK 4,000 per set of 4 cubes.

8 Discussion

In the following, the effect of the variation of selected test parameters is discussed with the purpose of evaluating relevant existing methods for the determination of the frost resistance of high performance concrete.

Furthermore, the accuracy of the methods as well as the damage types observed are dealt with.

For the discussion of test methods, the following names will be applied:

ASTM C 666
Critical Dilation
Dobrolubov/Romer
Borås
CDF
Slab Test
Cube Test

Since the Slab Test is identical to the Borås test method, only the latter is mentioned in the following section.

8.1 Reproducibility and Repeatability

The reproducibility and repeatability of the freeze/thaw methods may be described based on Round Robin Tests. A Large number of such tests have been performed and some reported, e.g. Borås Test [Andalen and Lundgren, 1992], Cube Test [Siebel and Reschke, 1993], and CDF Test [Setzer and Auberg, 1995].

Generally, the round robin freeze/thaw tests show relatively poor results, although recent tests carried out on pavement blocks show improvements by using enhanced temperature control systems [Pedersen 1996]. The large variations may be caused by one or more of the following:

* Lack of detailing within the descriptions opens for variation in procedures
* Poor laboratory workmanship/equipment (i.e., testing not carried out strictly according to the test method)
* Inhomogeneous test material.

On the other hand, the reporting of round robin tests may have omitted disagreeable results, caused by e.g. 'new laboratories' participating in the test.

Thus, an objective evaluation of the test method may very difficult.

Test methods with lack of detailing must be revised or rejected.

Poor laboratory workmanship should be improved by better descriptions of the test methods maybe in combination with the use of 'check lists' which should be part of the test methods.

Inhomogeneous material, i.e. concrete with variation in properties between test specimens, is undesirable in connection with determining the reproducibility and repeatability of a test method. Inhomogeneous concrete has been shown to have marked influence on freeze/thaw test results, causing variations of expected identical samples ranging from few g/m^2 to several kg/m^2 [Andalen and Lundgren, 1992], [Pedersen, 1996]. It may be expected that such variations in test specimens reflect possible variations in the actual frost resistance of the structures. Consequently, the freeze/thaw test results should include information on such variations.

8.2 Damage Types in Test Specimens

Scaling and internal cracking are two damage types observed in concrete specimens subjected to freeze/thaw testing. This observation correlates with observations from exposed structures (see section 2). The damage types have been reported often to occur independently of each other [Pigeon, 1987], [Jacobsen and Sellevold, no.4].

Test methods mainly causing surface scaling are Borås, CDF, and the Cube test. However, in recent developments within concrete mix designs, it has been experienced that the traditional 'scaling methods' may result in internal cracking as well as scaling [Geiker, 1995], [Jacobsen and Sellevold, 1994].

Test methods mainly resulting in internal cracking are ASTM C 666, Dobrolubov/Romer, and Critical Dilation. Apart from macro scale fine and coarse cracks, one special crack type observed in the test specimens freeze/thaw tested by these methods is characterized by (micro) cracks completely surrounding the aggregate particles and passing through the paste [Johansen et al, 1994]. This has been confirmed in specimens tested after ASTM C 666 [Jacobsen et al., 1995] and Dobrolubov/Romer [Laugesen, 1996].

The deterioration, e.g. of the micro-structure, studied in specimens tested with these methods may, however, differ from that of in-situ structures showing internal cracking or delamination [Laugesen, 1996].

8.3 Influence of Variation in Test Parameters

The freeze/thaw test methods cause varying type and degree of deterioration of the test specimens. In order to compare and evaluate the test methods, the influence of the following selected parameters on the test results are been discussed:

* Conditioning of test specimens
* Water absorption of test specimens during the freeze/thaw testing
* Cooling/heating rate
* Temperature limits of the freeze/thaw cycles
* Temperature control
* Specimen size
* Measurement techniques for damage registration

8.3.1 Conditioning of Test Specimens

The conditioning of the test specimens has frequently been shown to influence the results of freeze/thaw testing. The term 'conditioning' has in the literature been applied for one or more of the following procedures (ref. Table 7.1):

* Storage before de-moulding
* Water storage
* 'Air storage'
* Pre-drying
* Re-wetting

8.3.1.1 Early Storage

Variations in the (duration of water curing)/(duration of 'air storage') ratio have been observed to affect the scaling [Andersson and Petersson, 1987]. Differences in the early storage have influenced the round robin tests on Borås testing [Pedersen, 1996].

Regarding the Borås method, it should be noted that it differs from the Slab Test at only one point: the latter having a more well defined RH regime during the 'air curing' at a maturity of 7-19 days, ref. Table 7.1.

The influence of the RH-variability of the early Borås 'air storage' (7-19 days after casting) appears not to have been described.

8.3.1.2 Pre-drying

No pre-drying is specified for ASTM C 666, Critical Dilation and Dobrolubov/Romer, ref. Table 7.1.

The 3 scaling methods (Borås, CDF and Cube test) all prescribe conditioning in air: 14 days of 'air storage' (the first 12 at rather inexact conditions: 60±20 % RH for Borås) + 7 days of pre-drying at well specified temperature and relative humidity (20±2 °C and 65±5 % RH). The temperature requirement is easy to fulfil, and problems in obtaining the specified conditioning temperature has not been reported.

An additional requirement regarding evaporation measured from a free water surface of 45(±15) g/m^2h is specified. This latter demand is in contrast to the theoretical evaporation being 66 g/m^2h at 20 °C, 65% RH and a wind velocity of 0 m/s. According to practical experience, the evaporation measured from standardized evaporation bowls gives the following results:

* In (large) climate rooms the evaporation measured is often lower than 30 g/m^2h unless a certain wind velocity is applied [Petersson, 1996], [Mork, 1996], [Jacobsen. 1996]
* In climate cabinets with some air movement to secure uniform conditions the evaporation is recorded to be 80-110 g/m^2h [Laugesen, 1996].

Explanations of the too low evaporation rate in the larger climate rooms may be 1: that the climate locally does not fulfil the specifications (with 20 °C and 65 (±5)% RH) and/or 2: that the atmosphere above the surface of the water bowl has increased RH and hence decreased evaporation. Both explanations indicate that some variations in the actual conditioning of test specimens may be present, both within the climate room, and between climate rooms.

[Jacobsen at al., 1995] have measured the weight loss of test specimens stored in climate rooms/cabinets under various conditions. It was shown that the weight loss did rather correlate with RH/wind speed than with evaporation from the standard bowl (see Figure 8.1). It was also shown that samples conditioned at 50-65 % RH and moderate wind speed obtained the highest frost resistance measured by the Borås method.

The large effect of the drying procedure on scaling is not clarified. According to [Fagerlund, 1996-b], this may be due to:

1. Increase in the amount of freezable water, or
2. Decrease in the amount of water in coarser pores, interfaces, aggregate, etc.

Effect 1 is negative, effect 2 is positive. At mild drying, effect 2 is probably dominant. At hard drying, effect 1 is probably dominant. Thus, small changes in the drying climate might have very big effect on the result of the test.

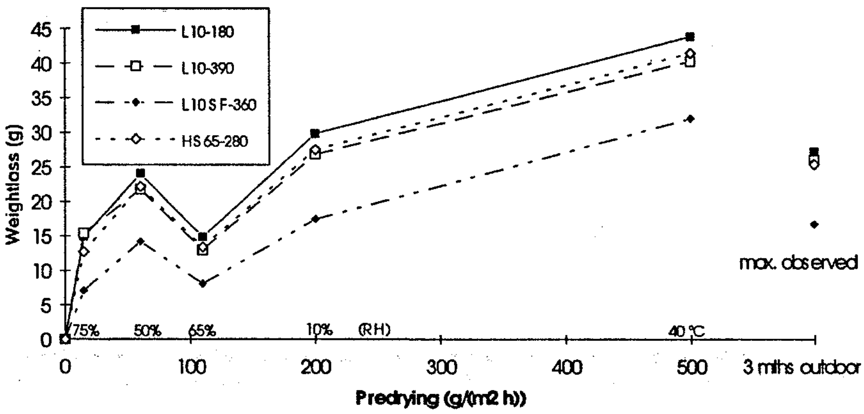

Figure 8.1 Weight loss of test specimens due to pre-drying at various RH-conditions [Jacobsen at al., 1995].

8.3.1.3 Resaturation

The degree of saturation is the single factor that has the largest effect on the result of a freeze/thaw test. The degree of saturation is determined both by the treatment before start of freeze/thaw - i.e. method of drying and resaturation - and by the 'wetness' of the test, i.e. the period during which the specimen can absorb water [Fagerlund, 1996-b].

Resaturation is specified in all the test methods (except for Critical Dilation, prescribing moist cured saturated specimens). The resaturation temperature is 20-23 °C.
The duration of the resaturation could be given as:

* Given time period
* Time until constant weight (dw/dt= x)
* Time until given weight gain

For pre-dried test specimens (Borås, CDF and Cube test) the resaturation time is specified to 3 days (72±2 h).

For the test methods without a specified pre-drying (testing of cored material according to ASTM C 666, Critical Dilation, and Dobrolubov/Romer) the resaturation of test specimens is specified: water curing until 'constant weight', 'constant weight' not being defined. The resaturation period is often 3-5 days, being the time where the absorption/time curve generally levels out (see Figure 8.2).
Consequently, the effective resaturation (amount of water absorbed) may vary, also as a function of the actual moisture content of the concrete, at the moment of coring/receiving cores at the laboratory, and specimen size.

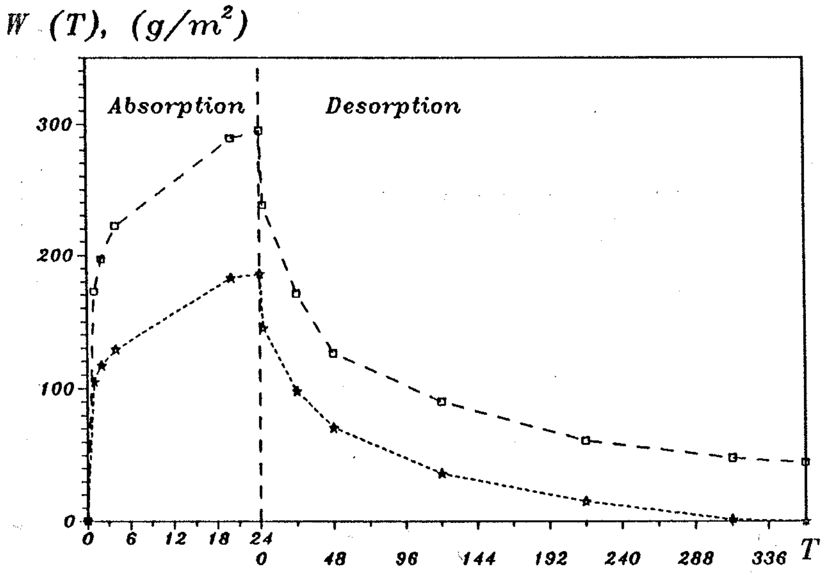

Figure 8.2 Typical change in water content of test specimens subjected to absorption (immersion in water) and desorption (20°C and 50% RH) [Aarre, 1994].

For concrete with low capillary porosity and the size of test specimens used in most of the test methods, the short term resaturation period of 3-5 days, is expected to influence only the outermost part of the specimen. Defects in microstructure, such as cracks and larger porous systems may, however, cause saturation to a marked depth or even throughout the test specimen. Consequently, variation in microstructure may be directly responsible for variation in frost resistance due to increased absorption during both resaturation and testing [Laugesen, 1996].

In order to reduce the variability of the resaturation of test samples, the following arguments in the discussion of 'constant time' versus 'constant weight' have to be considered:

* Resaturation for a fixed time might result in different saturation levels for different concretes, e.g. for different w/c ratios,
* Resaturation for a fixed time might result in different saturation levels of similar concretes if the pre-drying is not well defined (which may be a problem, as suggested above).
* Determination of the absorption, i.e. obtaining exact surface dry conditions at the weighing, is somewhat uncertain.
* The rate of weight gain of test specimens during absorption is gradually reduced with time giving problems in stating the point where 'constant weight' is achieved (ref. Figure 8.2).

The resaturation liquid is specified with some variations as seen below:

* ASTM C 666: Tap water saturated with lime
* DR: Not specified
* Borås: Tap water
* CDF: 3 % NaCl solution with distilled/demineralized water
* Cube Test: 3 % NaCl solution with tap water, (alternative: pure tap water)

Apart from the possible influence regarding salt solution or pure water as resaturation liquid (ref. section 4.2.2), it has been stated that variations in tap water quality, such as 'hardness', have proved to influence the test results [Mork, 1996].

8.3.2 Water Absorption of Test Specimens During the Testing

8.3.2.1 Constantly water cured specimens

For cast test specimens being water cured until the moment of initiating the test, no (marked) water absorption during repeated freeze/thaw cycles has been observed for ASTM C 666 [Pigeon et al., 86] and for Borås [Jacobsen et al., 1995]. It must be considered, however, that also very small water absorption might cause big frost damage in cores where the concrete was very close to critical saturation when the test started [Fagerlund, 1996-b].

8.3.2.2 Pre-dried specimens and specimens cored from structures or larger castings

For the test methods requiring curing in air, and/or if the conditioning includes potential drying of the test specimens, absorption during the freeze/thaw test can be observed which is profoundly larger than the absorption without freeze/thaw cycles. It has been shown for Borås and CDF [Jacobsen and Sellevold, 1994]. In these tests most concretes had a low degree of frost resistance and therefore were severely damaged in the test. In a concrete with high frost resistance the effect of salt on the absorption might be smaller or none [Fagerlund, 1996-b].

Increased absorption rates during freeze/thaw testing has also been recorded for ASTM C 666 [Laugesen, 1996].

Furthermore, it has been shown that the rate and degree of absorption are further increased by use of salt water, for Borås [Jacobsen and Sellevold, 1994], for CDF [Setzer, 1995] and for Cube test, see also the discussion in section 4.

Recording of water absorption during testing is not prescribed in any of the test methods, but can be performed for most of the test methods, e.g. Borås and CDF [Jacobsen and Sellevold, 1994], and ASTM C 666 [Laugesen, 1994].

8.3.3 Cooling/Heating Rate

The cooling/heating rate of the applied test methods (see Figure 8.3) have varying influence on scaling and on internal cracking. It has been assumed that rapid cooling mainly causes internal cracks, whereas slow cooling (in salt solution) mainly causes scaling.

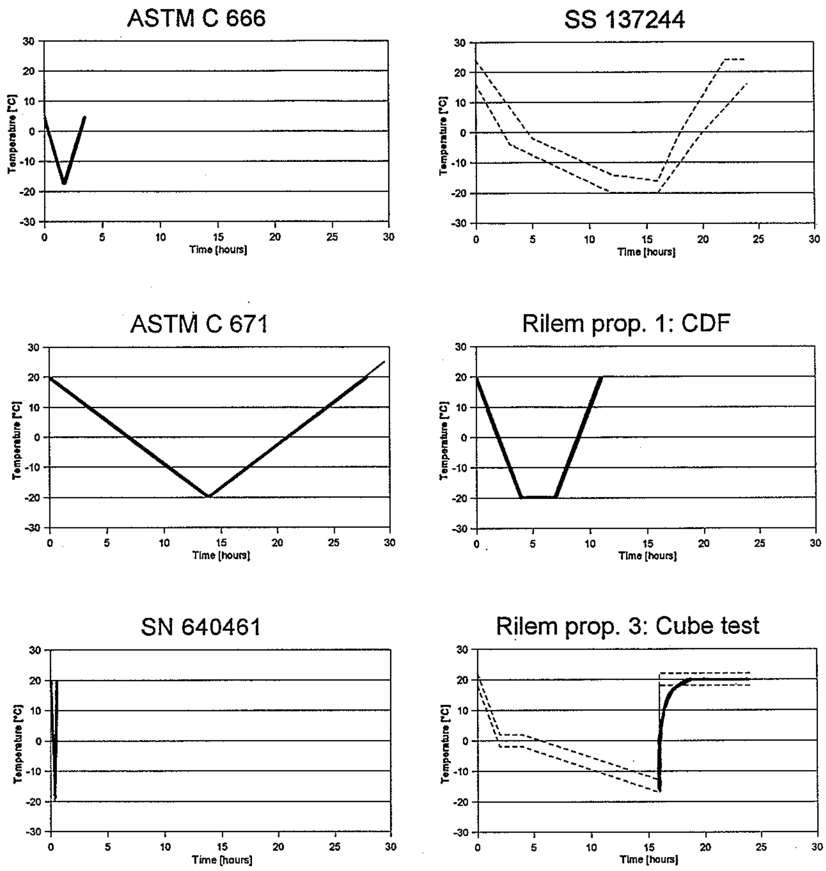

Figure 8.3 Temperature/time curves for the freeze/thaw cycles of the described test methods

The frost/salt scaling (CDF) of non-air entrained concrete is increased by reducing the rate of cooling and by increasing the period at the minimum temperature [Jacobsen et al., 1995-b]. Yet, for test specimens from an air entrained concrete (with very good scaling durability) no significant effect of different freeze/thaw cycles could be seen.

Internal cracking is not as clearly affected by varying cooling/heating rates for non air entrained concrete. For air entrained concrete increase in rate of cooling correlates with internal cracking [Jacobsen et al., 1995-b], [Pigeon et al., 1986], ref. Figure 6.1.

A detailed analysis of the rate of freezing is performed in [Fagerlund, 1992]. According to this analysis, the major effect of the freezing rate is indirect and is determined by its effect on the possibility it gives the concrete to absorb water during freeze/thaw. Thus a freezing cycle that is 'moist' is always more harmful than a freezing cycle that is 'dry' irrespectively of the rate of freezing [Fagerlund, 1996-b].

8.3.4 Temperature Limits of the Freeze/Thaw Cycles

All the test methods discussed, except ASTM C 666, prescribe a minimum temperature of -17 to -20 °C and a maximum temperature of approximately 20 °C. From microcalorimetric measurements [de Fontenay, 1982], [Bager and Sellevold, 1986-a] and from critical dilation testing, it appears that for normal concrete the main freezing takes place at -5 to -10 °C. For dense concrete, the most severe freezing takes place at lower temperature where more ice is formed and the poresystem partly blocked by ice [Fagerlund, 1996-b].

However, for Borås testing -19 °C has been observed to be a critical temperature, since specimens cooled to temperatures just below or above this temperature show difference in amount of scaling [Mork, 1996]. [Lindmark, 1993] found that the scaling of a given concrete was proportional to the square of the minimum temperature: Scaling = const $/\theta_{min}/^2$. Thus, freezing at -14 °C only gave about 40 % of the scaling that was obtained at -22 °C. This should be further investigated. No information on the effect on frost damage of minimum freezing temperatures appears to be available for the other freeze/thaw methods dealt with.

8.3.5 Temperature Control

The temperature is recorded in different positions:

* In test specimen or in the test liquid retained at the specimen
* In the cooling/heating agent.

8.3.5.1 Temperature Measurement in Test Specimen, or in the Test Liquid Retained at the Specimen

The temperature lowering are in ASTM C 666, Critical Dilation, Borås, and Cube Test directed to follow a given cooling rate or a well defined line in the time/temperature diagram. However, the (possible) formation of ice within the test specimen influences its temperature due to the heat of crystallization. Freezing of super-cooled pore liquid causes rapid temperature rise, e.g. [Grübl and Sotkin, 1980].

If the (impossible task of staying on the) prescribed time/temperature curve should be followed, the cooling temperature should consequently be instantly corrected. Temperature control based on measurements within the test specimen (or in the test liquid) will thus be dependant on the performance of the actual test specimen. Such delicate control is not considered possible, especially not at multispecimen testing.

The temperature control in the test specimen is for the given test methods generally carried out by measuring in a concrete dummy of identical dimensions as the test specimens. At prolonged exposures, the quality of such dummies may decrease, causing the temperature development to differ from that of the test specimens. Furthermore, the methods do not specify that the dummy should be made of same concrete nor have similar moisture content as the ones freeze/thaw tested.

To overcome the problems of specifying temperature control, the test methods accepts a variation in the time/temperature curve. This, however, opens for laboratory differences which may be critical to the freeze/thaw test results.

Consequently, it appears that specifying temperature cycles based on measurements in test specimen or in test liquid introduces possible variations in the test results.

8.3.5.2 Temperature Measurement in the Cooling/Heating Agent

Temperature control of the cooling/heating based on measurements in the cooling/heating agent (liquid or air) is prescribed in the methods Dobrolubov/Romer and CDF. To ensure a homogeneous and reproducible temperature transfer to the test specimen, the cooling/heating capacity of the agent should be well defined, e.g. by a prescribed stirring or heat exchange rate. This is not sufficiently well described in the methods.

Temperature control based on measurements in the cooling agent, demands strict limits on test specimen size in order to secure reproducibility of the actual exposure, influencing both the temperature and stress/strain sequences during the freeze-thaw cycles, ref. Section 8.3.6. The presently allowed variations of the test specimen sizes apparently do not secure this.

8.3.6 Specimen Size

The size of test specimens may influence the test results due to varying temperature developments, and hence variations in stress/strain which could possibly have direct influence on the deterioration. However, temperature cycling experiment performed by Sellevold and Jacobsen clearly indicate that temperature gradients are insignificant for damage [Fagerlund 1996-b]. The eigenstresses of the test specimens for Dobrolubov/-Romer testing have been shown to have no influence on the test results [Wilk and Dovbrolubov, 1982]. Apparently the influence for other test methods have not been investigated. Initial considerations regarding the stress development in test specimens has been performed [Pedersen, 1996] ref. Figure 5.2.

8.3.7 Measuring Techniques for Damage Registration

The properties measured are:

* Change in dynamic modulus of elasticity (E_{dyn})
* Length change
* Amount of scaled material.

8.3.7.1 Dynamic Modulus of Elasticity, E_{dyn}

As described for the ASTM C 666 test method, the E_{dyn} of a test specimen depends on the degree of internal cracking. The measurement of E_{dyn} may give erratic results:

* If local variations in the crack formation develop during the test.
* If the transmission is reduced, e.g. by placing the oscillator or the transducer upon an aggregate or a part of the paste/mortar which is partly spalled off the test sample.
* If there is surface scaling, the fundamental frequency increase due to the loss in weight and moment of inertia. Thus, one might believe that the concrete is less damaged in its interior than it is [Fagerlund, 1996-b].

To reduce the possible sources of error, the test specimen should be placed in the exact same way in all measurements. Furthermore, it has to be secured that the contact points are of acceptable quality.

Since the change of E_{dyn} of a test specimen correlates generally well with its length change, the latter being the most consistent, ASTM C 666 contain a 'security' against erratic measurements.

8.3.7.2 Length Change

Length measurements performed with a dial gauge at standardized conditions, e.g. 20 °C, is easily carried out and have high reproducibility. Applied with axial measurements, appropriate (conical) gauge studs, and corresponding concave dial gauge studs, a high precision can be obtained.

Continuous length recordings during cooling/heating with LVDT-equipment may cause problems in relating the measured values to absolute values. For Critical Dilation, where the LVDT is cooled/heated along with the test specimen, this may cause systematic errors. The evaluation of the results of length change during cooling should therefore be handled with care. However, the relative movement during the cooling may still correctly show a possible existence of a dilation, ref. calibration by invar rod, Section 7.2

8.3.7.3 Amount of Scaled Material.

The amount of surface scaling is recorded by gentle removal with squirt-bottle and brush (Borås and Cube test), and subsequent drying and weighing. The handling may be responsible for variations in amount of collected material and hence in the test result. Especially for moderately damaged specimens, such variation in handling may influence the test result. For frost resistant specimens, the handling will hardly have any effect and for severely damaged specimens, the amount of scaling will any way be above any conformity criteria.

The removal by ultra sonic bath (CDF) appears readily to decrease the uncertainties of the manual cleaning. Yet it may well show, that the effective 'stirring action' of the ultra sonic bath will be difficult to standardize: how much water should be in the bath ?, how many samples can be run at the time ?, etc.

Variations in the procedures of collecting scaled material may be a source of error. This is especially relevant for test specimens of modern type concretes showing quite deep deterioration interchanging with parts of almost unaffected exposed surface.

9 Summary and Conclusions

Concerning performance testing of frost resistance of high performance concrete the following should be considered:

* The test shall resemble the environmental exposure, but be somewhat more severe
* The type of damages should resemble damage types observed in-situ
* The test shall have a high degree of reproducibility
* The ranking of materials shall be according to experience
* A correlation between test results and performance in-situ shall be established to allow for conformity criteria to be given.

In-situ observations indicate the existence of two often independently acting types of damage: Scaling and internal cracking. Thus, at the present the need of two test methods causing scaling and internal cracking, respectively, is assumed.

Special considerations regarding the freeze/thaw test methods are:

* To simulate in-situ conditions, the test shall include drying as part of the conditioning
* Both the rate of cooling, the minimum temperature, and the duration of the freezing period applied in the test method should be chosen as to obtain the most severe frost attack within a likely regime
* In-situ cooling or heating rates causing thermal stresses larger than the tensile strength are not expected (except temperature shock due to application of deicing). The specimen size and the rate of cooling should be chosen accordingly
* The minimum temperature should probably be below -19°C for concrete in the Nordic climate
* The temperature control should be based on temperature measurements in the cooling/heating agent surrounding the specimen. The cooling capacity should be sufficient to eliminate the possible effect of heat of crystallization.
* The registration of damage should be relevant. Measurement of absorption during conditioning and testing as well as evaluation of final cracking may be relevant supplementary information. Measurement of initial absorption may even act as an alternative to actual performance testing
* The method description should be unambiguous, and check lists are believed to be of high value.

The relevance of the below test methods with a view to the use on high performance concrete has been evaluated:

* ASTM C 666, A: Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing
* Modified ASTM C 671: Standard Test Method for Critical Dilation of Concrete Specimens Subjected to Freezing
* Modified SNV 640 461: Test II for resistance to frost and de-icing salts. (Dubrolobov-Romer)
* SS 137244: Concrete Testing - Hardened Concrete - Scaling at Freezing (the Borås method)
* Rilem Draft Recommendation: 117-FDC Freeze-Thaw and Deicing Resistance of Concrete:
    1. Tests with water (CF) or with sodium chloride solution (CDF)
    2. Slab test (identical to Borås)
    3. Cube test.

Evaluation of the methods with regard to fulfilment of the above preliminary requirements, ref. Table 9.1, indicate that none of the methods are directly applicable.

Table 9.1 Fulfilment of preliminary requirements

	ASTM C 666	Mod. Critical dilation	Mod. Dobr./ Romer	Borås	CDF	Cube Test
Primary damage: Scaling	-	-	-	+	+	+
Primary damage: Internal cracking	+	+	+	-	-	-
Pre-drying	-	-	-	+	+	+
Minimum temp. below -19°C	-	+	+	-	+	-
Well defined (cast/cored) specimen size	-/-	+/-	+/+	+/-	+/-	+/-
External temp. control	-	-	+	-	+	-
No risk of thermal stresses	+	+	+	+	+	+
Well defined registration of damage	+	-	+	+	+	+
Conformity criteria	-	-	+	+	+	-
Correlation to in-situ performance (Acceptance/Rejection)	?/?	?/?	+/-	?/?	?/?	?/?
Acceptable accuracy	?	?	?	?	?	?

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Test Methods

* ASTM C666, A: Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing, 1992.

* ASTM C671: Standard Test Method for Critical Dilation of Concrete Specimens Subjected to Freezing, 1994.

* SNV 640 461: Norms for frost- and frostsalt resistance of concrete, Chapter 2: Test II for resistance to frost and de-icing salts (Dubrolobov-Romer). Official translation in Bulletin of Betonstrassen AG, June 1985, Wildegg, Schwizerland.

* SS 137244: Concrete Testing - Hardened Concrete - Scaling at Freezing (Borås metoden), March 1995.

* Rilem Draft Recommendation: 117-FDC Freeze-Thaw and Deicing Resistance of Concrete:
  1. Tests with water (CF) or with sodium chloride solution (CDF). Materials and Structures 1995, 28,175-182.
  2. Slab test. Materials and Structures 1995, 28, 366-371.
  3. Cube test. Materials and Structures 1995, 28, 366-371.

Table 7.1 Comparison of selected freeze-thaw test methods.

Parameter	ASTM C 666	Modified ASTM C 671 (Critical Dilation)	Modified SNV 640461 (Dobrolubov/Romer)	SS 137244 (Borås)	Rilem proposal 1: CDF	Rilem proposal 3: Cube test
Cooling rate Heating rate	+4.4 →-17.8: 9-22 ℃/h -17.8 →+4.4: 9-22 ℃/h	+20 →-20: 2,8 ℃/h -20 →+20: 2,8 ℃/h	+20 →-20: 120 ℃/h[1] +20 →-20: 240 ℃/h[1]	+20 →-4: 5,3 ℃/h -4 →-18: 1,9 ℃/h -18 →+20: 4,8 ℃/h	+20 →-20: 10 ℃/h -20 →+20: 10 ℃/h	+20 →0: 10 ℃/h 0 →-15: 1.25 ℃/h[1] -15 →+20: 20 ℃/h[1]
Max. temperature	4.4 (±1.7)	20 ℃	20 ℃	20 (±4) ℃	20,0 (±0,5) ℃	20 (±2) ℃
Min. temperature	-17.8(±1.7) ℃	-20 ℃	-20 ℃	-18 (±2) ℃	-20,0 (±0,5) ℃	-15 (±2) ℃
Temperature measure-point	centre of dummy	centre of dummy	liquid of temperature bath	centre of exposure (test) liquid	liquid of temperature bath	centre of dummy
Exposure	full test specimen	full test specimen	full test specimen	one plane, from above	one plane, from below	full test specimen
Exposure media (→test liquid')	1-3 mm layer of tap water	air (sealed test specimen)	cooling: saturated CaCl solution heating: running tap water	3 mm layer of 3 % NaCl in tap water	10 mm layer of 3 % NaCl in distilled or demin. water	10 mm layer of tap water or 3 % NaCl in tap water
Freeze-thaw cycles	300	1	350	56	28	56
Duration of 1 cycle (hours)	2-5	29 (±5)	0.5	24	12	24
Measuring terms	per 36 cycles	constant measurement	per 50 cycles	at 7, 14, 28, 42, 56 cycles	at (4, 6,) 14, 28 cycles	at 7, 14, 28, 42, 56 cycles
Measuring parameters	E_{dyn}, length	length	length	scaling	scaling	scaling
Cleaning of surface(s)	-	-	-	squirt-bottle and brush	ultra sound bath	squirt-bottle and brush
Sample curing before start of conditioning: - mould: - water curing: - air storage:	 1 day 13 days -	 1 day 3,8,12,16,20,24 weeks -	 1 ≥28 days[2] -	 1 day 6 days[3] 12 days[3] + 2 days[4]	 1 6[4] 14	 1 6 -
Test duration (days) after e.g. cutting:[4] - pre-drying - re-saturation - freeze-thaw - total	 0 3[5] 42 45	 0 0 1 1	 0 5[5] 8 13	 7 3[5] 56 66	 7 7[6] 14 28	 20 1[5 or 6] 56 77
Cost level (DKK)	10,000 per set of 6 cores	12,000 per set of 12 specimens	5,000 per set of 6 prisms	4,500 per set of 5 samples	4,500 per set of 4 samples	4,000 per set of 4 samples

Note 1: Underlined values for cooling/heating rates are approximated, since the test methods prescribe alternating immersion of test specimens into liquids of fixed temperatures.
Note 2: The test method does not specify if the curing is in water or in air.
Note 3: Conditions: RH = 60 (±20) %. Evaporation from a free water surface = 45 (±15) g/m²h (ref. Section 8.3.1.2, discussion).
Note 4: Conditions: RH = 65 (±5) ℃. Evaporation from a free water surface = 45 (±15) g/m²h (ref. Section 8.3.1.2, discussion).
Note 5: Re-saturation with tap water.
Note 6: Re-saturation with salt solution, 3 % NaCl in tap water (Cube test) or in distilled/demineralised water (Rilem 1: CDF).