LUND UNIVERSITY

Fatigue effects associated with freeze-thaw of materials

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2000

FATIGUE EFFECTS ASSOCIATED WITH FREEZE-THAW OF MATERIALS

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Abstract

In a traditional freeze-thaw test, where the specimen is exposed to liquid water during thawing and/or during freezing, it is often observed that damage increses progressively with increasing number of freeze-thaw cycles. Sometimes, a certain "incubation" number of cycles is required before frost damage occurs. This behaviour is often believed to be an effect of fatigue. In this report it is shown that the damage progression observed is probably caused by a gradual water absorption during the test. Therefore, each new cycle is performed with a somewhat higher moisture level. Often a certain number of cycles ("incubation cycles") is required for frost damage to be initiated. Thereafter, due to the gradually increased water content during each new cycle, damage is increased with increasing number of cycles. This is not due to a fatigue effect of traditional type, but due to gradually higher internal stresses caused by the increased water level in the material.

In the paper it is also shown that a certain low-cycle fatigue exists, and has an influence on the damage progression, but this effect is limited to rather few freeze-thaw cycles coming directly after the cycle that initiated frost damage.

Equations for estimation of the progress of frost damage as function of the number of freeze-thaw cycles are given in the report.

FATIGUE EFFECTS ASSOCIATED WITH FREEZE-THAW OF MATERIALS

1. Introduction

There are two types of freeze-thaw damage:

1: *Internal damage* caused by a transgression of a certain maximum tolerable, critical, moisture condition inside the material.

2: *Surface scaling* mostly occurring when the surface of the material is exposed to a salt solution -de-icing salts or sea water- during freeze-thaw.

This paper only deals with internal freeze-thaw damage.

It is often believed that frost damage -at least in a freeze-thaw test- is caused by fatigue as a result of repeated freeze-thaw cycles. Each new cycle is supposed to add to cumulative internal damage similar to what occurs in normal mechanical fatigue with constant load cycles. Since the number of freeze-thaw cycles nomally is below 300 the fatigue should, however, be more of type low-cycle fatigue than high cycle fatigue. Also in the practical use of the material, the total number of severe freeze-thaw cycles seldom exceeds some thousands. Therefore, also in practice, frost damage should be of type low cycle fatigue provided there is any fatigue at all.

The reason behind the fatigue hypothesis is that one often notices a development of damage of the type shown in Figure 1 when the material is tested in so-called "open" freeze-thaw, i.e. in a test where the specimen has access to water, during freezing and/or during thawing, [1]. In some cases -curves D, P, C in Figure 1- damage increases progressively with the number of freeze-thaw cycles already form the first cycle. In other cases -all other curves in Figure 1- a certain number of freeze-thaw cycles are needed in order to initiate damage. Thereafter, damage increases progressively with increasing number of cycles.

It will be shown in this paper that this behaviour can be explained by a different phenomenon than ordinary fatigue, namely by a gradual water absorption during the test, causing bigger and bigger stresses during continued freeze-thaw. After a certain critical water content has been reached, damage begins and is increased by a low-cycle fatigue effect lasting for a limited number of cycles until the material is more or less totally destructed. This critical water content can be described as a *fatigue limit*.

2. Closed freeze-thaw versus open freeze-thaw

2.1 Open freeze-thaw. Gradual water uptake

In a traditional open freeze-thaw test the specimen is frozen either in air or immersed in water, and it is almost always thawed in water. This means that the specimen is free to absorb water during the test. Normally the water content increases gradually since the thawing phase normally causes bigger absorption than the freezing phase causes drying. Examples of the gradual water absorption during a test is seen in Figure 2, [2].
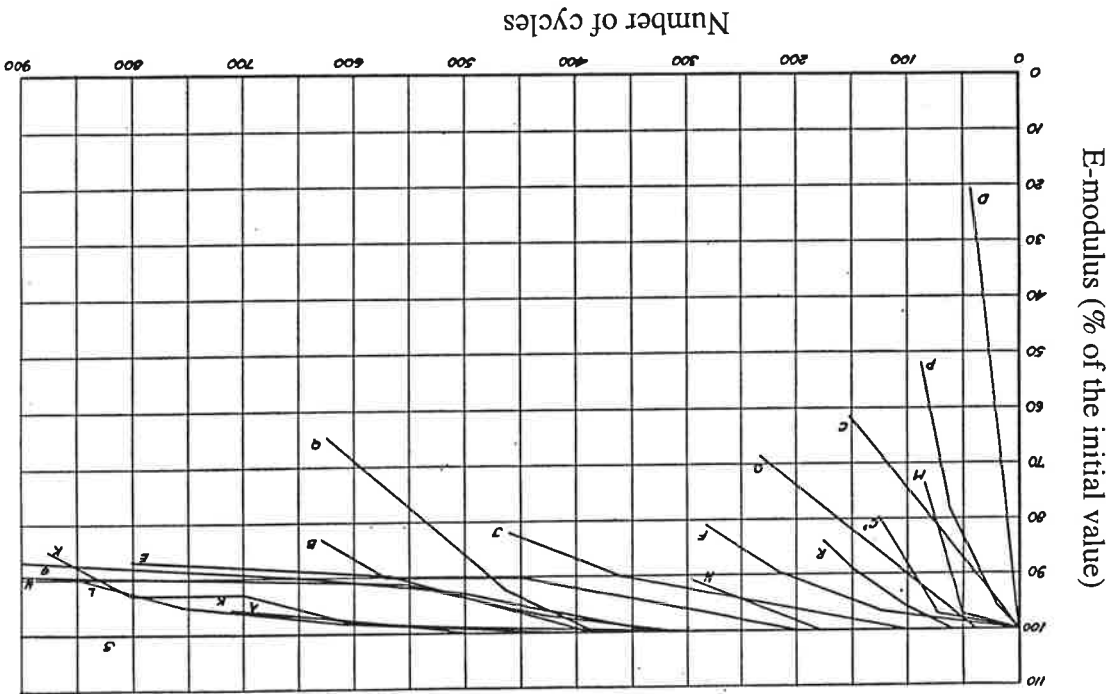
The mechanism behind this water absorption is not fully clarified. It, however, occurs in the initially air-filled pore system since this is the only place where water can be accommodated. It also occurs in cracks formed during freeze-thaw. At constant temperature, above zero, the water absorption can be caused by a dissolution of air enclosed in "air-pores". The mechanism is described in [3].

Number of cycles

E-modulus (% of the initial value)

Figure 1: Reduction in E-modulus of cement mortar specimens repeatedly frozen in air to −15°C and thawed in water at +5°C. 2 cycles per day; /1/.

Number of cycles
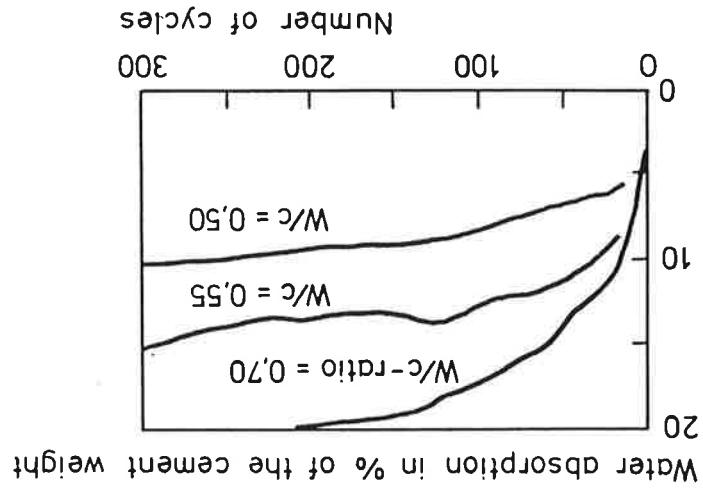
0   100   200   300

W/c = 0.50

W/c = 0.55

W/c-ratio = 0.70

Water absorption in % of the cement weight

Figure 2: Water absorption in three types of concrete during freeze-thaw in water; /2/.

During the thawing phase in water, this might be sucked into the material by different mechanisms. Since ice-bodies contained in bigger pores has lower free energy than unfrozen water in finer pores, there is a certain internal desiccation of the material during the freezing phase. During thawing the material is therefore able to suck water in order to restore saturation of the desiccated pores. The mechanism is only active in fine-porous materials containing a large fraction of unfreezable water.

Another possibility is that water is sucked into the material when ice inside the pores melts. The volume of the melted water is lower than the initial volume of ice. Another mechanism for water absorption during thawing is that when the specimen warms from its lowest temperature the contraction of the ice phase is bigger than the contraction of the solid material. Therefore, theoretically, there is a possibility of a certain absorption provided the specimen is placed in unfrozen water, [4].

When the material is frost damaged, the cracks formed will be rapidly filled by water. Therefore, one can assume that the absorption rate is somewhat bigger in the frost damaged material than it was before frost damage occurred.

A special case of open freeze-thaw is a salt scaling test where the material surface is exposed to a salt solution, normally NaCl. Then, ice bodies formed close to the surface will absorb water from the solution since this contains unfrozen liquid down to the eutectic temperature (-21°C for NaCl.) The driving force for water absorption is the free energy differential between liquid and ice, [5].

In open freeze-thaw the water content in the specimen will therefore gradually increase, and thereby the internal stresses during freeze-thaw will gradually increase.

2.2 Closed freeze-thaw. No water uptake

In closed freeze-thaw the material is protected from moisture uptake or loss. Thus, the moisture present before the first freeze-thaw is unchanged during all consecutive freeze-thaw cycles. The consequence of this is that either the material is damaged, which is the case when the moisture content is above a certain critical level, or that it is undamaged, which is the case when the moisture content is below the critical value. It will be shown below that the number of freeze-thaw cycles -the fatigue effect- is of significant importance only when the moisture content is above the critical.

3. The effect of moisture at closed freeze-thaw

In closed freeze-thaw there is no moisture change in the material during the entire test. A test can be performed in the following manner: A series of specimens of the same material is adjusted to different individual moisture conditions, either by drying from saturated condition, or by absorption from dried condition. After that, they are sealed in order to hinder moisture exchange with the surroundings. Hysteresis effects might cause a certain moisture gradient across the specimen volume during the moisture adaptation phase, especially when the adaptation is made by drying. This effect can be diminished by a conditioning procedure where the specimen is cyclically warmed and cooled in its sealed condition. Another possibility is to adapt the specimens by using a pressure plate apparatus in which water is either forced out by an external air pressure, or in which water is sucked in by a gradually lowering of the outer air pressure. The test principles are described in [6]. In both cases, water leaves or enters the specimen homogeneously over the entire volume.

A suitable mechanical property for detecting internal damage, like the dynamic E-modulus, is determined for each specimen before freeze-thaw. After that, the specimens are exposed to a number of freeze-thaw cycles in sealed condition. The selected mechanical property is determined after a limited number of cycles. The fatigue effect can be observed by testing after different number of freeze-thaw cycles.

Finally the *damage* -like the residual E-modulus- is plotted versus the moisture content. Then, diagrams of the type in Figures 3 to 6 are obtained. In these diagrams moisture condition is expressed in terms of a degree of saturation defined as:

$$S = w_e/V_P \tag{1}$$

where w_e is the total amount of water in the specimen (kg, or litres) and V_P is the total pore volume in the specimen (litres).

Figure 3 shows results for *cement mortar*, [7]. Below S=0.77 there is no frost damage irrespectively of the number of freeze-thaw cycles. Above S=0.77 damage is progressively increasing with increasing value of S. Besides, in this moisture region, damage increases with the number of freeze-thaw cycles.

Figure 4 shows results for a *concrete*, [8]. In this case the limit between undamaged and damaged materual corresponds to S=0.85. Above this value the damage increases with increasing S. The number of freeze-thaw cycles is of no importance; 78 cycles give about the same damage as 9 cycles.

Figure 5 shows results for two *concrete* types, [9]. The critical moisture contents are 0.80 and 0.90. Only 6 cycles were used. Despite this, the damage is severe when the critical moisture value is transgressed. The damage is proportional to this transgression.

Figure 6 shows results for a *sand-lime brick*, [10]. As in the case of cement mortar -Figure 3- damage increases with increasing number of cycles, but only provided the degree of saturation is above 0.80. This value is independent of the number of freeze-thaw cycles. Damage is proportional to the transgression of this value.

Figure 7 shows the freezing expansion of *concrete* specimens pre-conditioned to different degrees of saturation and freeze-thaw tested in isolated condition for different number of freeze-thaw cycles, [11]. There is a clear indication of a critical degree of saturation of 0.90. This value is independent of the number of freeze-thaw cycles.

Many more results of similar type can be found in [8], [10], [12].

Comprehensive experimental tests of different types of materials therefore indicate that:

1: there exists a moisture content that is fairly well-defined and that marks the border between frost resistance and frost damage. This moisture content seems to be almost uninfluenced by the number of freeze-thaw cycles, at least to about 100 cycles. The critical moisture content is individual for each material. It is comparable to the *fatigue limit* in normal mechanical fatigue, since moisture contents below the critical moisture content are too low to be able to cause damage.

2: frost damage is directly proportional to the amount by which the critical moisture content is transgressed, indicating that the internal stresses increase in proportion to the amount of frozen water above the critical.

3: the proportionality constant between damage and transgression of the critical moisture content increases with the number of freeze-thaw cycles. Few additional cycles above the first give big additional damage. Thus, frost attack above the critical moisture content can be

considered a type of *low-cycle fatigue*. Below the critical moisture content there is no significant fatigue. If fatigue in this region should exists, it should be of limited practical importance since the number of freeze-thaw cycles in nature is too small to cause high-cycle fatigue.

4: there seems to exist a sort of *maximum possible damage* which is independent of the number of freeze-thaw cycles, and that is proportional to the amount by which the critical degree of saturation is transgressed.
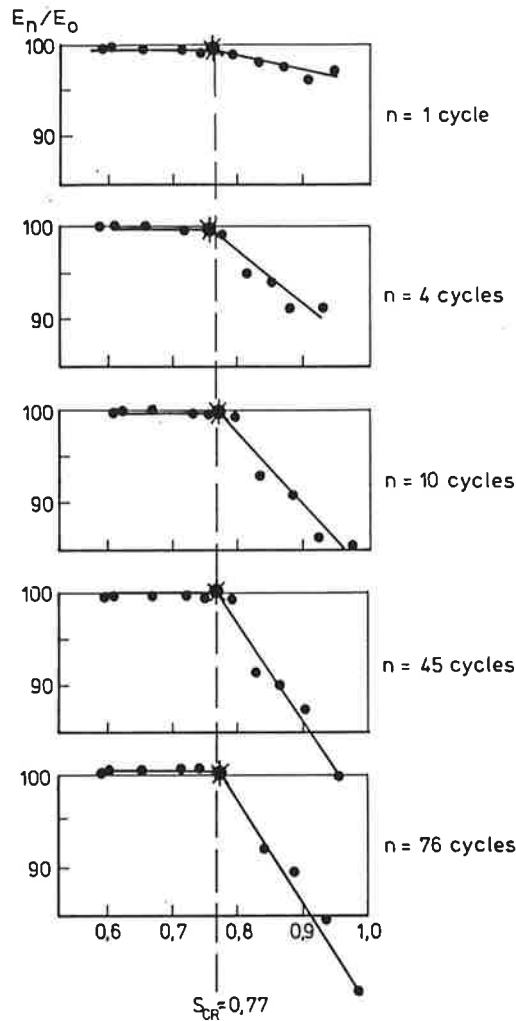

Figure 3: Effect of the degree of saturation and the number of freeze-thaw cycles on a cement mortar tested by closed freeze-thaw; /7/.
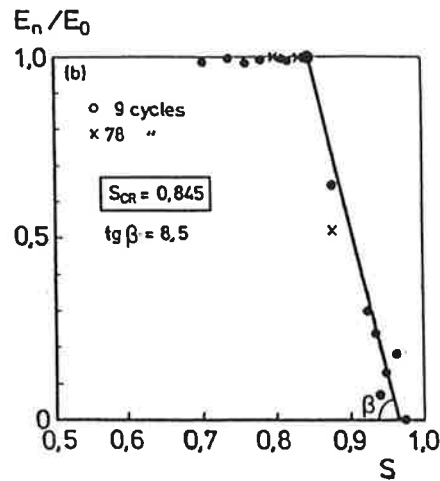
Figure 4: Effect of the number of freeze-thaw cycles and the degree of saturation on a
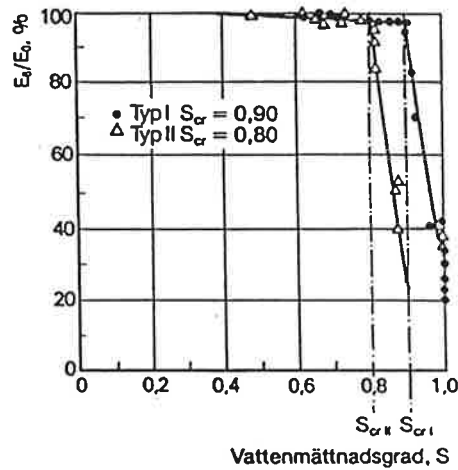concrete tested by closed freeze-thaw; /8/

Figure 5: Effect of the degree of saturation on the E-modulus of two types of concrete at a
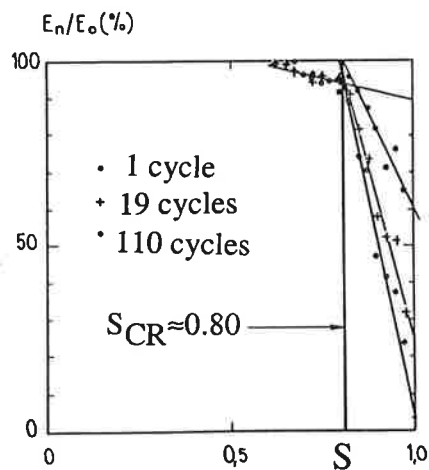closed freeze-thaw test; /9/.

Figure 6: Effect of the number of freeze-thaw cycles on the E-modulus of a sand-lime brick
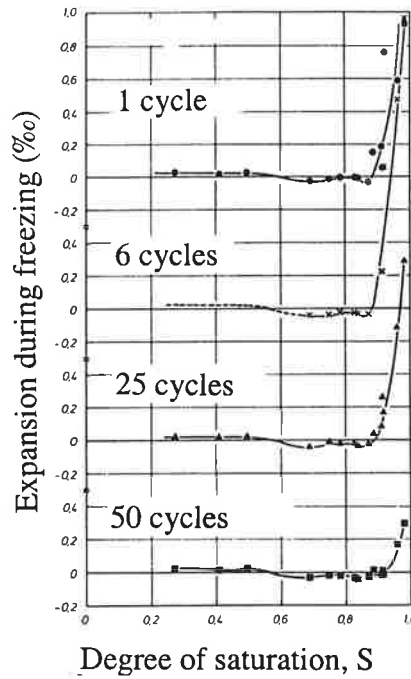at a closed freeze-thaw test; /10/

Figure 7: Effect of the number of freeze-thaw cycles and degree of saturation on expansion during closed freeze-thaw of a concrete; /11/.

4. The fatigue effect in closed freeze-thaw

The experimental findings described above indicate that damage, D, can be described in the following manner:

$S \leq S_{CR}$: $D=0$ (2a)

$S_{CR} < S \leq 1$: $D = K_N(S-S_{CR})$ (2b)

where K_N is a "coefficient of fatigue" that depends on the number of cycles. K_N is the slope of the damage curve in diagrams ot type Figure 3 to 6. For the cement mortar in Figure 3 the fatigue coefficient is plotted in Figure 8. Evidently, K_N approaches an asymptot when the number of freeze-thaw cycles is increased. For the cement mortar in Figure 3 this happens when the number of cycles is about 80. For freeze-thaw cycles above this value, the fatigue coefficient is almost constant, indicating that no more frost damage occurs when the water content is kept constant, irrespectively of the number of freeze-thaw cycles above 80.

For the sand-lime brick in Figure 6, the value of K_N is plotted in Figure 9. The curve has the same general shape as for the cement mortar.

The coefficient of fatigue for both the cement mortar and the sand-lime brick can be expressed in the following way:

$$K_N = \frac{A \cdot N}{B+N}$$ (3)

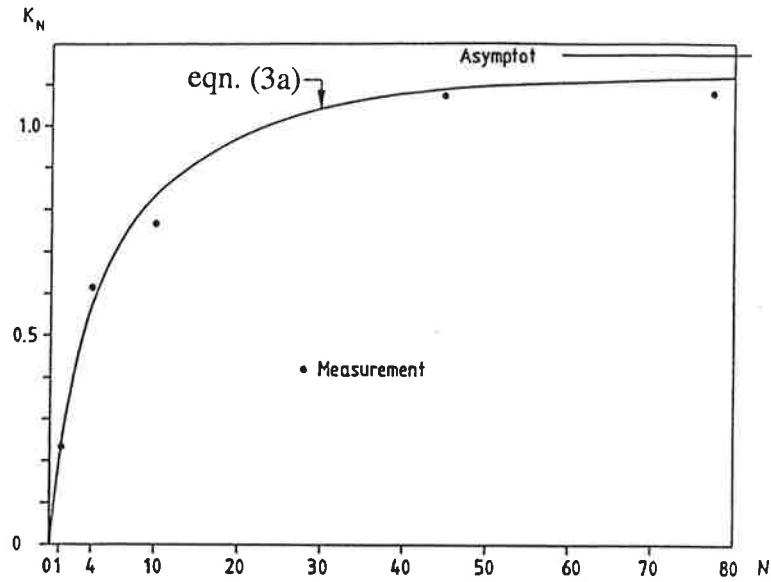where A and B are constants. A is the level of the asymptot.

Figure 8: The fatigue coefficient K_N for the cement mortar in Figure 3.
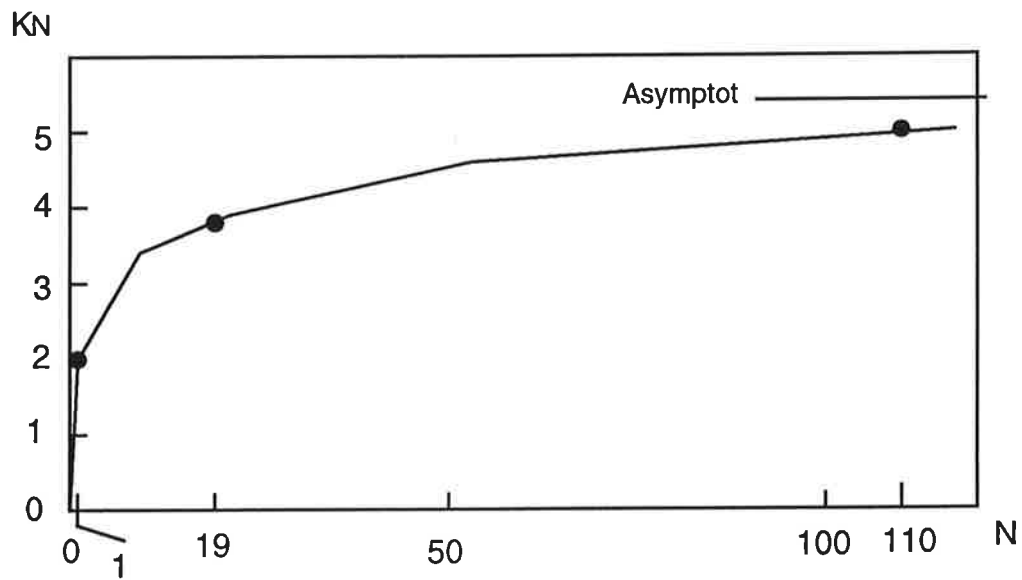

Figure 8: The fatigue coefficient K_N for the sand-lime brick in Figure 6.

For the *cement mortar* the following equation is valid:

$$K_N = \frac{1.2 \cdot N}{4 + N} \tag{3a}$$

For the *sand-lime brick* it is valid:

$$K_N = \frac{5.4 \cdot N}{8.3 + N} \tag{3b}$$

Provided the same type of equation is valid also for the concretes in Figure 4 and 5, and provided B=4, the following equations for K_N are valid:

The concrete in Figure 4:

$$K_N = \frac{13 \cdot N}{4 + N} \tag{3c}$$

Concrete Type I in Figure 5:

$$K_N = \frac{12 \cdot N}{4 + N} \tag{3d}$$

Concrete Type II in Figure 5:

$$K_N = \frac{12.5 \cdot N}{4 + N} \tag{3e}$$

All equations (3a) to (3e) assume that "damage" is expressed as:

$$D = (E_n - E_0)/E_0 = \Delta E / E_0 \tag{4}$$

where E_n is the E-modulus after N cycles, E_0 is the E-modulus before freeze-thaw, and ΔE is the change in E-modulus caused by freeze-thaw. If another measure of damage is used, the values of the coefficients A and B should of course be changed.

According to eqn. (2b) and (3) the maximum possible damage at a given degree of saturation and at a very large number of sealed freeze-thaw cycles is:

For $S \leq S_{CR}$:

$$D_{max} = 0 \tag{5a}$$

For $S_{CR} < S \leq 1$:

$$D_{max} = A(S - S_{CR}) \tag{5b}$$

The absolute maximum damage occurs when S=1 and the number of freeze-thaw cycles is very big. It is

$$D_{max,abs} = A(1 - S_{CR}) \qquad D \leq 1 \tag{5c}$$

This means that the maximum possible damage for the materials in Figures 3 to 6 is:

Cement mortar in Figure 3:
$D_{max,abs}=0.27$
Concrete in Figure 4:
$D_{max,abs}=1$ (total destruction)
Concrete Type I in Figure 5:
$D_{max,abs}=1$ (total destruction)
Concrete Type II in Figure 5:
$D_{max,abs}=1$ (total destruction)
Sand-lime brick in Figure 4:
$D_{max,abs}=1$ (total destruction)

For the three types of concrete it was assumed that the coefficient B is the same as for the cement mortar (B=4). If the value for sand lime brick is used instead, B=8, the value of A would be a bit higher meaning that the fatigue effect is a bit bigger than given by eqn. (3c) to (3e). The·asymptot A would in this case have the following values:

For the concrete in Figure 4:   A=17   (instead of A=13)
For the concrete Type I in Figure 5:   A=17   (instead of A=12)
For the concrete Type II in Figure 5: A=17.5 (instead of A=12.5)

Thus, the effect of the choice of the value of B is not so important.

The experiments and the calculation show that in almost all cases very severe damage occurs when the material is completely saturated. Besides, also small transgressions of the critical moisture content gives big damage; see Figure 3, 4, 5, 7. Therefore, the comparably small damage also at full saturation in the cement mortar is noteworthy; see Figure 6. The reason is not clear. There are at least two possibilities:

1: When calculating the degree of saturation, the total porosity was maybe not used, but a porosity not including the whole air-pore system. It is difficult to experimentally determine the total porosity if the material is not completely dried before vacuum saturation. Normally, drying at +105°C is required. Besides, the vacuum saturation might have been incomplete due to difficulties in evacuating all air in the pore system during the vacuum treatment. Both effects will lead to higher calculated degrees of saturation than the real.

2: The freezable water in the cement mortar is considerably lower than the total water content. Therefore, the "effective" degree of saturation, only considering the freezable water, is smaller than the degree of saturation defined by eqn. (1). The effective degree of saturation is defined:

$$S_{eff} = w_f/(w_f+V_a) \tag{6}$$

where w_f is the amount of freezable water in the specimen (kg, or litres), and V_a is the volume of air-filled pores (litres). Example: If S is 0.95 and only 40% of the water is freezable S_{eff} is only 0.88. This means that a high value of S might not have as negative effect on frost resistance as might be expected if the material has a high fraction of unfreezable water.

4. Interpretation of results of an open freeze-thaw test

The freeze-thaw results shown in Figure 1 for open tests can now be interpreted in the following way, see Figure 10:

1: When the test starts, the initial moisture content, S_o, is below the critical value. Consequently no damage occurs during the first cycles; points 1, 2, 3 ,4 in Figure 10.

2: Due to water uptake during and between the freeze-thaw cycles, the water content is gradually increasing; Figure 10(a). Finally, the critical moisture content is reached in the whole or parts of the material, point 5 in Figure 10. The time and cycles needed for this to happen depends on the water uptake during each cycle, ΔS, and on the difference between the initial moisture content S_o and the critical moisture content S_{CR}. Thus, a higher number of cycles are needed for materials which are highly frost resistant than for materials with low degree of frost resistance. This explains why the different materials in Figure 1 start to deteriorate after different numbers of cycles.

3: Already one cycle after the critical moisture content has been reached, the water content is so high that frost damage occurs; point 6 in Figure 10. The amount of frost damage depends on the amount by which the critical water content is transgressed, and is given by the expression $D=K_N(S-S_{CR})=[A \cdot 1/(B+1)] \cdot (S-S_{CR})$.

4: Due to frost damage, the water absorption during each cycle probably increases in comparison with the absorption before the critical moisture content was reached. This is visualized by the steeper water uptake curve in Figure 10(a).

5: Due to the extra water absorption and the increasing number of cycles, damage after each number of cycles is determined by different damage lines; point 7 is on the line for 10 cycles, point 8 is on the line for 20 cycles, point 9 is on the line for 50 cycles, etc.

Thus, the gradual damage observed in open freeze-thaw tests is not a consequence of fatigue, but of a gradual increase in moisture content above the critical. There is a certain low-cycle fatigue, but it is limited to the first cycles.

Frost damage as function of the number of freeze-thaw cycles can be described by:

1: *Before start of damage*, i.e. for $S \leq S_{CR}$ and $N_{tot} \leq N_o$ it is valid:

$$S_o + N_{tot} \cdot \Delta S_1 \leq S_{CR} \tag{7}$$

That is, the total number of freeze-thaw cycles before frost damage occurs is:

$$N_o = (S_{CR} - S_o)/\Delta S_1 \tag{8}$$

where ΔS_1 is the water absorption during each freeze thaw cycle before frost damage occurs.

This means that the total number of freeze-thaw cycles, that a material can sustain, increases with increasing difference between the critical water content and the water content at the start of the freeze-thaw test. This is the reason why a higher air content in concrete gives a higher frost resistance. The higher air content reduces the value of S_o since only the capillary pore system is filled by water during pre-storage before start of the freeze-thaw test. Similarly, a hard-burnt clay brick is more frost resistant than an under-burnt brick made of the same raw material, because the pore structure of a hard-burnt brick is such that it contains many pores - "impermeable pores"- that cannot become water-filled during ordinary water storage; [10].

Normal pre-storage before start of a freeze-thaw test only causes "capillary saturation", while coarser "impermeable" pores stay air-filled. However, during the freeze-thaw test, also these pores might become gradually water-filled.

2: *After start of damage*, i.e. for $S_{CR} \le S \le 1$ and $N_{tot} > N_o$ it is valid, eqn. (2b):

$$D = K_N \cdot [(S_{CR} + N \cdot \Delta S_2) - S_{CR}] = K_N \cdot N \cdot \Delta S_2 \tag{9}$$

Or, by inserting eqn. (2b):

$$D = \frac{A \cdot N^2}{B+N} \cdot \Delta S_2 \tag{10}$$

where ΔS_2 is the water absorption in a frost damaged concrete during each freeze-thaw cycle. N is the number of freeze-thaw cycles causing frost damage ($N = N_{tot} - N_o$).

In eqn. (10) it is assumed that each new freeze-thaw cycle causes the same amount of water absorption irrespectively of the amount of frost damage. Probably, it would be more reasonable to assume that the water absorption is depending on the degree of damage. One possibility is to assume a lineraly increasing water absorption with increasing number of cycles:

$$\Delta S_2 = \Delta S_1 + C \cdot N \tag{11}$$

where C is a constant.

This gives the following development of damage:

$$D = \frac{A \cdot N^2}{B+N} \cdot (\Delta S_1 + C \cdot N) \tag{12}$$

Example

The following data are valid for a certain material tested by open freeze-thaw:

S_o: 0.60
S_{CR}:   0.85
ΔS_1:   0.003
ΔS_2:   0.007
A: 1.5
B: 4
C: 0

The number of freeze-thaw cycles before frost damage is:

$N_o = (0.85 - 0.60)/0.003 = 83$ cycles

Development of damage is described by:

$$D = \frac{1.5 \cdot N^2}{4+N} \cdot 0.007$$

This gives the following development of frost damage.

N_{tot}	N	D (%)
83	0	0
90	7	5
100	17	14
150	67	66
185	102	100

This development of damage is shown in Figure 11. It resembles damage curves observed in a real test; cf. Figure 1.
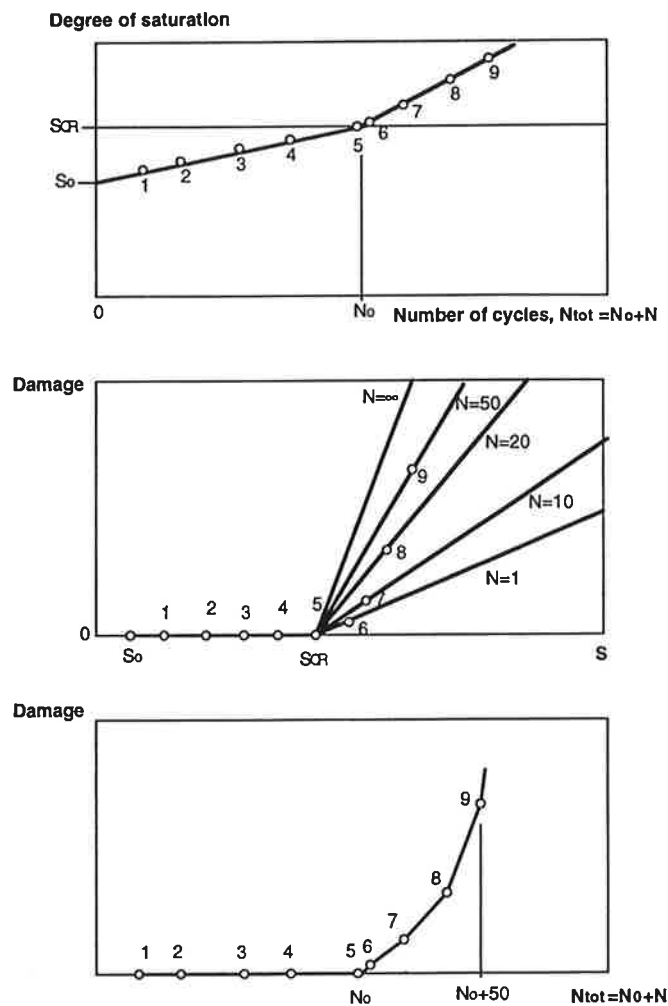

Figure 10: Principles of water absorption and development of damage in an open freeze-thaw test.
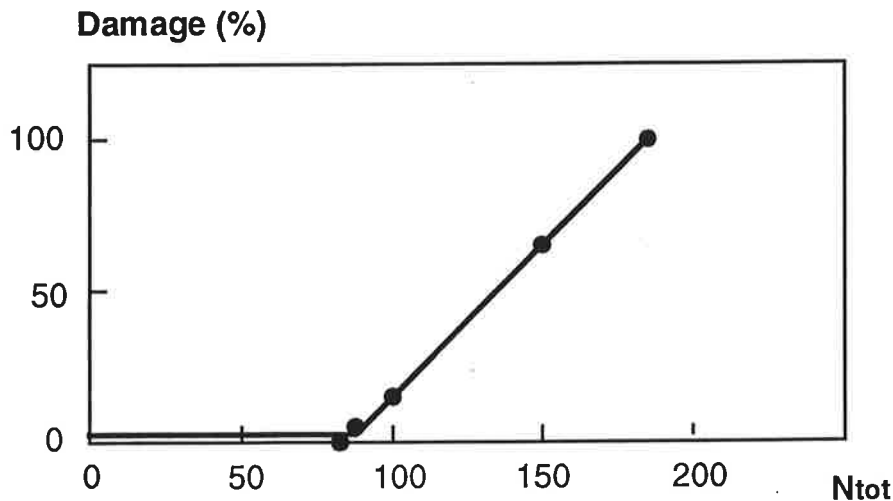
Damage (%)

Figure 11: Plot of data from the Example.

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