

FROST TESTING NON-AIR ENTRAINED SCM HPC: SALT-SCALING AND INTERNAL DAMAGE

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ABSTRACT

Air-entrainment is difficult with high volumes of Supplementary Cementitious Materials (SCM). High Performance Concrete (HPC) with low w/b without air-entrainment can, however, survive severe deicer salt frost performance testing, eliminating all the hassle of producing air entrained concrete. Therefore, the surface scaling of low-CO₂-emission concretes was studied in freeze/thaw performance tests with a 3 mm layer of 3% NaCl on the surface. Four different non-air entrained silica fume-fly ash concrete mixes with w/b = 0.30 – 0.35 with 91d strengths >100 MPa were investigated after 3 different curing conditions (Normal, Dried, Wrapped). The w/b=0.30 CEM IIA reference mix without additional FA had lowest scaling following all curing conditions. However, all 4 mixes and 3 curing conditions suffered severe internal damage. Liquid Uptake (LU) during freeze/thaw related best to internal damage, whereas the correlation LU-scaling and strain-scaling were weaker. A parametric analysis of glue-spall stress as affected by internal cracking was made with increasing differential thermal expansion ($\Delta\alpha$) due to internal cracking and Poisson ratio (ν) equal to zero in severely cracked concrete assuming compression closes cracks without lateral deformation. An experimentally based model for elastic modulus (E) as function of cracking was employed. The analysis shows that glue spall stress reduced by reduced ice thickness due to LU while internal damage (reduced E and ν , increased $\Delta\alpha$) affects glue spall stress directly and indirectly by the same order of magnitude as 1 – 2 mm reduction of ice thickness. LU must be accounted for in modelling scaling.

Keywords: *Concrete, Supplementary Cementitious Materials, High Strength, Frost salt scaling, Cracking, Ice, Liquid Uptake*

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1. INTRODUCTION

A new ferryboat-free coastal highway in Norway is planned connecting the southwestern coastline with some 10 large bridges, of which several will be the world's longest of their kind [1] if/when built. There will very likely be requirements to sustainability including CO₂-emissions. The 3 methods for production of sustainable concrete for such structures are [2]: 1. replacement of cement with SCMs, 2. improved design (high particle packing, reduced volume of structures, prolonged service life etc), 3. CCS. Required technical quality must of course be maintained. The most available SCMs in Norway today are fly ash (FA), silica fume (SF), slag and limestone filler. Also calcined clay, natural pozzolana and local recycled and manufactured materials are relevant [2-4]. The service life relates directly to sustainability because all environmental burdens (emissions, consumption of primary raw materials and energy etc) must be calculated per unit service life of the structure. Concrete structures are exposed to deteriorating mechanisms (reinforcement corrosion, ASR, frost, leaching, sulfate attack, abrasion etc). In the northern hemisphere freeze-thaw and deicer salt surface scaling attack are important due to the widespread use of deicing chemicals on roads, bridges, parking lots, airfields etc., and due to the tidal zones of marine concrete. The deicer salt scaling is challenging but both the visible scaling and the invisible volumetric damage, termed internal cracking, should be considered [5,6].

Mass-ratio, air entrainment, type of binder and curing are the central material parameters for making frost durable concrete. In [3] Exposure classes XF3/XF4 ("freezing with high saturation without/with salt") specify Durability classes MF45/MF40 = effective mass-ratio 0.45/0.40 with air entrainment (≈ 4.5 vol-%). A protective air void system needs high air void specific surface ($\approx 30 \text{ mm}^{-1}$) and low air void spacing factor L ($\approx 0.2 \text{ mm}$) as quantified by ASTM C457 [7]. The limits depend on the details of the exposure and the concrete material (mass-ratio, type of binder and curing conditions). Air entrainment is, however, a hassle in concrete production with SCMs. Depending on the types and combinations of binder powder, admixtures, dosage sequence, mixing procedure, workability etc., the result can be unpredictable [8-12]. With appropriate combinations of admixtures, dosage- and mix sequences satisfactory air void parameters can still be achieved [13]. In practice air entrainment requires extra trial mixing, use of special admixtures and equipment, extra documentation, and it causes drop of strength compared to non-air entrained concrete. Therefore, developing frost durable SCM-concrete without air is desirable. This requires field experience, laboratory testing and/or various other types of experiments. In addition, of course quantitative models are needed based on understanding of the mechanism(s) acting when a deicing solution is freezing on a concrete surface. The mechanism amplifying concrete surface damage and the pessimum effect with salt was explained by Valenza and Scherer by the glue-spall model [14,15]. Glue-spall stress in the concrete surface parallel to the ice-interface (σ_{gs}) is due to thermal expansion mismatch, eq.(1) [14,15]:

$$\sigma_{gs} = \left(\frac{E_c}{1-\nu_c} \right) \left[\Delta\alpha\Delta T - \sigma_i \left(\frac{1-\nu_i}{E_i} \right) \right] \quad (1)$$

In eq.(1) E_c and E_i are the elastic moduli of concrete and ice, ν_c and ν_i are the Poisson's ratios of concrete and ice, and $\Delta\alpha$ is the difference between the thermal expansion coefficient of ice and concrete and σ_i is the stress of ice according to eq.(2) [14,15]:

$$\sigma_i = \frac{0.5t_c \left[\frac{E_c}{1-\nu_c} \right] \Delta\alpha\Delta T}{0.5t_c \left(\frac{E_c}{E_i} \right) \left[\frac{1-\nu_i}{1-\nu_c} \right] + t_i} \quad (2)$$

In eq.(2) t_c and t_i are the thicknesses of concrete and ice. High tensile stress in the concrete surface develops at flaws, gaps or ice-brine pockets in the ice, initiating and propagating fracture into the concrete. Quite different types of experiments [16-18] showed that de-icer salt scaling increases with increasing ice layer thickness as expected from eq.(1) and (2). Çopuroğlu and Schlangen [17] in addition developed a numerical model from the Delft lattice model simulating ice cracking during freezing and that cracks could propagate into the concrete initiating scaling. The protective effect of air voids in the glue spall model is to reduce thermal expansion mismatch according to Sun and Scherer [19] since air-voids compress concrete when freezing while highly saturated due to cryo-suction. The powerful suction was observed in freeze-warping of concrete-steel composites with steel on top [20]. Air entrained concrete warped convex, opposite of the concave warping without air voids [20]. A recent numerical study by Bahafid, Hendriks, Geiker et al [21] showed how discontinuities in ice on a (non-air entrained) concrete surface gave high local concrete surface tensile stresses in varying combinations of ice- thickness and -gap width during freezing. Tensile stresses increased in the concrete under flaws in the ice, with increasing ice thickness and when the flaws (or gaps) between adjacent ice islands on the concrete surface became narrower. Maximum concrete surface tensile stress always occurred under the ice gaps and was large enough to fracture concrete like in [14,15].

There are some important similarities between internal cracking and salt scaling during frost exposure. Entrained air voids protect against both, and there is an accelerated Liquid Uptake (LU) both during freeze/thaw with water and with salt solution [22]. However, a much clearer relation LU-internal frost damage is seen than LU-deicer salt scaling [6]. Still, it was claimed that LU causes salt scaling damage by swelling of the cement paste and scaling of thin flakes [23,24]. Furthermore, the protective effect of air voids against deicer salt scaling reduces in severely dried and re-saturated surfaces [25], for never-dried surfaces [26, 27], for surfaces of fly ash concrete with prolonged water curing [6] and after carbonation of slag concrete [28]. The effects [6,22-28] are not readily explained by the glue-spall model. The scope of this study is to investigate some of these problems, namely salt-frost durability of concrete with SCMs without air entrainment and how the glue spall stress is affected by simultaneous internal cracking. Four different HPC mixes with relevant SCM binders for the E39 project were produced and exposed to three different realistic curing conditions. Surface scaling (kg/m²), internal cracking (length change) and LU (weighing) were monitored during freeze/thaw slab testing with 3 % NaCl- solution on the concrete surface and a parametric study of the effect of internal cracking on glue-spall stress was made.

2. EXPERIMENTS: CONCRETE MIXES AND FROST TESTING

First the particle packing of the mixed natural-crushed granitic aggregate from Årdal Norway (0-8 mm sand and 8-16 mm coarse with 0.4 % absorption) was maximized. Minimum bulk aggregate void space was measured to be ≈ 300 liters/ m^3 bulk aggregate by varying the ratio sand/coarse. Workability was then optimized using 335 liters of filler modified cement paste / m^3 concrete with the most dense-packed aggregate mix. Co-polymer water reducer dosage was adapted to give stable fresh concrete mixes with high workability. The main binder was Anlegg FA cement (CEM IIA-V-42.5N) from Heidelberg, Norway with 75.5% clinker, 15 % fly ash ground with the clinker, 5.5% gypsum, and 4% limestone filler. A dry condensed silica fume from Elkem, Norway and additional low lime Fly ash were also used. Four final concrete mixes were prepared. **Mix 1** (Reference mix) (030AnlFA): w/b=0.30, Anlegg FA (incl 16% FA) + 3% SF, **Mix 2** (0.35FA8SF): w/b=0.35, Anlegg FA (35% FA) + 8% SF, **Mix 3** (0.30 FA8SF): w/b=0.30, Anlegg FA (35% FA) + 8% silica, and **Mix 4** (0.30 FA16SF): w/b=0.30, Anlegg FA (35% FA) + 16% SF. SlumpFlow was 550-675 mm, Slump 250 – 260 mm and T500 was 7.8 – 15.7 sec, so workability was viscous and heavy flowing. Fresh air void contents were 1.4 – 1.9 vol-% and 91-day compressive strengths > 100 MPa for all 4 mixes. Environmental performance indicators were calculated with an LCA-tool used by the Norwegian ready mixed concrete industry. Mix 2 had lowest GWP from cradle to gate according to relevant ISO- and EN-standards with GWP ≈ 200 kg/ m^3 almost giving the rating “Low carbon extreme” in the Norwegian Concrete Association environmental performance classification scheme.

Frost testing was done on $\approx 5 \times 15 \times 15$ cm slabs cut from 15 cm cubes [29] with 3 types of curing. **Normal:** Cubes stored 7d in water and 14 d in the climate room (at 65%RH, 20 °C, 45 g/ m^2 ·h evaporation rate from free water surface). Then the cubes were sawn into 47 mm thick slabs and further 7d exposure in the climate room while prepared with non-absorptive butyl tape to make the dam and then + 3d with pure water on the test surface. Then water was replaced with 3 mm 3 % NaCl solution before freeze/thaw testing for 56 cycles. **Dried:** Cubes stored 7d in water, 14 d in the same climate room, then sawing and further 14 weeks exposure in the climate room, prepared with butyl tape, and then 3d with pure water on the test surface. Then water was replaced with 3 mm 3 % NaCl solution before freeze/thaw testing for 112 cycles. **Wrapped:** Cubes stored 7d in water, 14d in the same climate room, sawing, and 14 weeks packed in plastic-covered aluminum foil, then prepared with butyl tape before 3d with pure water on the surface. Then water was replaced with 3mm 3 % NaCl solution, before freeze/thaw testing for 112 cycles. During frost-deicing testing of the 3 x 16 slabs measurements were made of scaling of concrete from the top surface (kg/ m^2). Internal damage was measured by monitoring length change horizontally across the 15 cm width of 2 of 4 parallel slabs using a dilatometer on invar studs drilled and glued into the 47 mm thick vertical slab surfaces at half-height and mid-width. LU was monitored by weighing slabs in SSD state with the non-absorptive butyl after each scaling- and length measurement and adding scaled mass corrected for loss of evaporable water.

3. RESULTS, DISCUSSION AND OUTLOOK

3.1. Freeze/thaw

Table 1 shows that Mix 1 is quite scaling resistant after Normal and Dried curing. Common accept criteria of the test are $m_{56} < 0.5$ or $m_{56} < 1 \text{ kg/m}^2$ and acceleration factor (m_{56}/m_{28}) < 2 . Mix 2 – 4 and Wrapped curing perform less well. There are several Wrapped mixes that have quite low scaling m_{56} , but the acceleration is highest in Wrapped curing so all these mixes fail, see the (m_{56}/m_{28}) and (m_{112}/m_{56}) acceleration factors.

Table 1. Scaling (kg/m^2) and acceleration (m_{2n}/m_n), all values average 4 parallel slabs

Sample ID	Curing Conditions	m_{28}	m_{56}	m_{112}	m_{56}/m_{28}	m_{112}/m_{56}
		Average at 28 th cycle	Average at 56 th cycle	Average at 112 th cycle		
Mix 1	Normal	0.20	0.37	NA	1.79	NA
	Dried	0.14	0.21	0.26	1.43	1.25
	Wrapped	0.02	0.08	1.32	3.67	15.17
Mix 2	Normal	1.19	2.47	NA	2.08	NA
	Dried	1.72	1.98	2.45	1.15	1.23
	Wrapped	0.18	1.07	3.97	5.73	3.69
Mix 3	Normal	0.63	1.27	NA	2.00	NA
	Dried	0.63	0.98	1.37	1.54	1.39
	Wrapped	0.05	0.23	1.11	4.09	4.67
Mix 4	Normal	0.54	0.98	NA	1.81	NA
	Dried	1.53	1.76	NA	1.14	NA
	Wrapped	0.20	0.92	2.29	4.63	2.46

The measurements of internal damage (details not shown) showed that all specimens had internal damage with expansive strain > 0.001 already after 28 cycles, i.e. $>$ approximately 10 times the tensile fracture strain of concrete. Strain increased at continued cycling. One mix had expansion > 0.01 (= 1%) after 56 cycles and at least 6 mixes had expansion > 0.01 after 112 cycles. There was a tendency that also development of internal damage was more accelerated after wrapped curing, just like the acceleration of scaling.

The measurements of Liquid Uptake (LU) during freeze/thaw were in line with previous experience [6] etc.: LU correlates clearly to Internal damage and a bit less clearly to scaling. The correlation between internal damage and scaling was similar to the correlation LU-scaling.

3.2. Parameter studies of glue-spall stress

All concrete material parameters of eq.(1) and (2) are affected by internal cracking. A parametric study was performed of the resulting effect on σ_{gs} . Then scaling depth was estimated as a relative

crack penetration depth by the fracture mechanics of the glue spall model [14,15]. Since LU can become very high the ice thickness will reduce during freeze/thaw so that the glue spall stress will reduce, eq.(1)-(2). $LU = 1 \text{ kg/m}^2$ is common, meaning ice thickness reduction $\approx 1 \text{ mm}$. At the same time internal cracking will increase the residual expansion, increasing the differential thermal expansion. The latter was calculated based on determining an effective residual coefficient of thermal expansion of the concrete. The Poisson ratio of the concrete will be reduced by the internal cracking. Ice contracting on cooling will cause strain parallel to the concrete surface that will tend to close concrete cracks without affecting lateral strain at severe internal cracking, hence ϑ_c is likely close to zero. E-modulus drops by the internal cracking and was calculated from measured strain and [30]. Figure 1 shows some results.

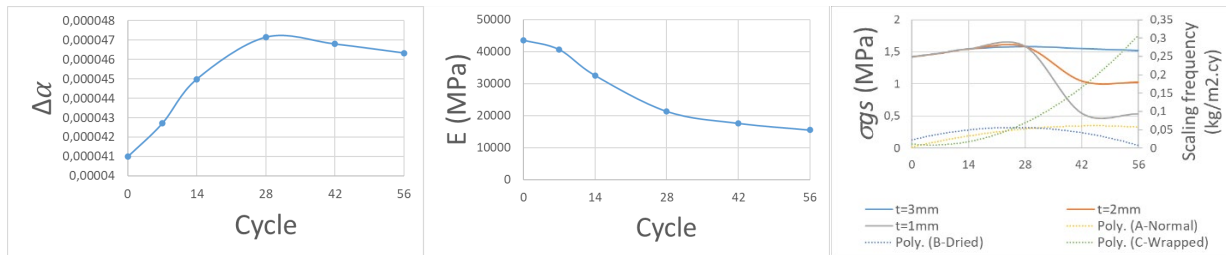


Figure 1 a) residual differential thermal expansion, b) concrete E-modulus, and c) glue spall stress at varying ice thickness and polynomial trend of scaling frequency, average 4 mixes, each with 3 curing conditions.

Figures 1a and 1b show development of differential thermal expansion and E-modulus with our strain data. Figure 1c shows glue spall stress as function of cycling on the left-hand axis using realistic material parameters and varying ice thickness. The right-hand axis of Figure 1c shows average polynomial trend of scaling frequency for the 4 mixes for each of the 3 types of curing based on details behind Table 1 (not shown here) with linear, accelerated, and decelerated progress for Normal, Wrapped and Dried cure for all 4 mixes. Both the low stresses in Figure 1c compared to [21] and the acceleration warrant more work on modelling. Due to the high LU at high internal cracking we chose to highlight the effect of ice thickness. Such cracking with high penetration of fluid must be reported specifically according to [29] and was not caused by leakage between concrete and butyl-tape in this work. Parameter studies of the above-mentioned relative crack penetration depth revealed, not surprisingly, that cracks propagate from ice and longer into concrete when there is internal damage in the concrete. Several other aspects of simultaneous scaling, internal cracking and LU were analyzed indicating how internal cracking and LU affect glue spall stress in different ways [31]. In a recent study by Maus et al [32] CT-scanning revealed that the saline ice on the concrete surface is micro-columnar and permeable. Hence LU during freeze/thaw [6, 22] should be modelled with this kind of permeable ice, and not with impermeable ice like in [6,33]. Also, the loss of protective effect of air voids after prolonged water curing of FA-concrete [6], after severe and no drying, after carbonation of slag-concrete etc [6,23-28] needs investigation. Internal damage can then affect glue spall stress to different degrees depending on how $\Delta\alpha$ is affected, see eq.(1) and (2). Another important aspect is water filling of air – voids which will affect frost test performance [6, 27, 34, 35].

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