



0008-8846(95)00173-5

HIGH STRENGTH CONCRETE - FREEZE/THAW TESTING AND CRACKING

Stefan Jacobsen¹, Hans Christian Gran¹, Erik J. Sellevold², Jon Arne Bakke²

1) The Norwegian Building Research Institute, Oslo, Norway, 2) The Norwegian Institute of Technology, Trondheim, Norway

(Refereed)

(Received May 2; in final form July 24, 1995)

ABSTRACT

Cracks due to rapid freezing and thawing in water (ASTM C666 procedure A) of non-air entrained high strength concretes have been investigated using a procedure designed to avoid creating cracks during specimen preparation. Polished sections impregnated by immersing virgin water saturated slices in ethanol containing fluorescent dye (Fluorescent Liquid Replacement - FLR) were inspected in ultra violet light in an optical microscope. The fluorescent impregnated polished sections showed no cracking in any of the concretes before frost exposure, and extensive cracking after deterioration. The amount of cracks on the polished sections showed a good relationship to Durability Factors (DF) measured in the rapid freeze/thaw test. The crack volume calculated using a simple square grid model of cracks correlated reasonably to measured volume increase at frost testing.

Key words: High strength concrete, freeze/thaw testing, cracks, microscopy, Fluorescent Liquid Replacement (FLR)

Introduction and background

The deterioration of concrete in rapid freeze/thaw testing (ASTM C666 procedure A or B) is normally measured as loss of resonance frequency, or as the corresponding calculated loss of relative dynamic modulus. To get a better understanding of how the test exposure affects the concrete, the crack pattern developed in concretes not able to withstand this type of testing can be inspected. Gran (1) used liquid replacement for fluorescent dye impregnation and microscopy of concrete: FLR - Fluorescent Liquid Replacement. FLR involves low risk of introducing cracks during preparation since no drying (heating, vacuum) is needed for impregnation. The impregnation consists of immersing concrete slices in alcohol containing dissolved dye. The colorant dye enters the pores of the specimen by liquid or solvent replacement. The results (1) showed no cracks in various virgin low-porosity hardened cement pastes with and without silica fume, whereas drying at 50 and 105 °C caused extensive cracking of the same pastes as judged by FLR. Bakke (2) investigated the effect of drying at 50 and 105 °C and vacuum pumping for a few

minutes to approximately 0.10 mbar on the extent of micro cracking. The experiments were performed on the 030-08 QD concrete of table 1 below. The results (2) showed that drying at 50 °C and 105 °C both lead to cracking, (most after 105 °C) when submerging the dried slices directly in alcohol after cooling. Parallel slices (dried at 50 and 105 °C) that were resaturated by immersion in water before liquid replacement showed no cracking. That is, apparent healing of the drying induced cracks took place, as judged by the FLR-technique. Vacuum pumping to 0.10 mbar also led to significant micro cracking, even after only 30 seconds at 0.1 mbar.

Other researchers too, have used dye impregnation systems to observe cracks in concrete. Marchand (3) used a red dye impregnation method developed by Hornain and Regourd (4) to study micro cracking due to different drying treatments. Hornain (5) has in a recent review on microscopy techniques discussed the observation of cracks in concrete. Kukko (6) studied cracks developed in non-air entrained high strength concretes by thin sections and image analysis, and also morphologies of cracks by SEM. The thin sections used in (6) were impregnated with fluorescent dye in vacuum, but possible cracking due to the vacuum treatment was not discussed. Some cracks were observed on the reference specimens before freeze/thaw testing, though rather few compared to after freeze/thaw exposure. In the present paper the FLR technique has been used to study the amount of cracks developed in non-air entrained high strength concretes exposed to rapid freezing and thawing in water.

Concretes and freeze/thaw deterioration

Details on the concretes (mixing, curing, testing, healing) are given in (10, 11, 14). Polished sections were prepared from 6 different concretes before and after exposure to ASTM C666 procedure A (rapid freezing and thawing in water). Table 1 shows the concretes investigated, their properties and how they were tested. The volume of cement paste was approximately 31 % for all mixtures, and no air entrainment was used. All concretes were investigated by the FLR technique before and after frost test.

TABLE 1: Concrete mixtures investigated

Mix		w/c+s	s/c+s	Type of cement	Aggregate	f_c start MPa	DF 2)	No.of cycles
030-08 QD	(11)	0.30	0.08	HS 1	Quartzdiorite	141	41-100	300
030-08 LS		0.30	0.08		Limestone	110	50-92	275-300
030-08 LWA1		0.30	0.08		Macrolite	41	23-99	140-300
035-08 LWA2W	(10)	0.35	0.08	HS 2	Leca 800 1)	72	2	35
040-00	(10)	0.40	0	OPC	Granite	74	11	70
040-05		0.40	0.05		Granite	83	10	70

1) Water saturated 2) Durability Factor according to ASTM C666 procedure A

Preparation of polished sections

Dye impregnated polished sections for observation in ultraviolet light were prepared (1). In this technique areas impregnated with the fluorescent dye are enlightened, whereas non-impregnated areas appear dark in ultraviolet light. Impregnated (porous) regions normally include cement paste, voids and cracks. The light intensity in impregnated areas depends on cement paste porosity, the extent and quality of polishing after impregnation and the ability of the impregnation resin (or alcohol) to penetrate.

Preparation of the polished sections consisted of first sawing a slice of concrete perpendicular to the length axis of the concrete beam. The slices were normally sawn from the centre (middle) portion of the 10 by 10 by 35 cm beams in a thickness of 2 - 3 cm immediately after termination of freeze/thaw testing, one slice for each variable. In addition, reference specimens were taken from the same concretes before freeze/thaw exposure. The slices were polished on one side and submerged for 3 - 7 days in ethanol. The ethanol contained 1 % by mass of dissolved fluorescent dye. That is, the wet slices were submerged in virgin condition, without any drying prior to the impregnation by liquid replacement. After 3 - 7 days excess dye on the surface of the concrete was removed by polishing. During the course of this polishing, the concretes were inspected in the microscope regularly. The polishing is important since surface dye must be removed, whereas impregnation in cement paste and cracks must remain. In high strength concretes with very dense paste, this can be a difficult task since the cement paste is impregnated to rather small depths. The measurement of depth of impregnation can be done by microscopic investigation of a surface sawn perpendicular to the surface exposed to the fluorescent liquid, as shown by Gran, (7). If one only wants to study the amount of cracks, it will therefore sometimes be easier to polish the specimens until the dye filled cracks are visible, ignoring the study of the cement paste between the cracks. In ultraviolet light the cement paste and aggregate will then appear dark whereas the cracks will appear illuminated. In the present study some of the specimens were polished so that the cracks appeared enlightened, whereas the paste was rather dark. Quantification of the amount of cracks (crack number) was performed by using a modified version of a test method developed by The Danish Institute of Technology (8). The so called crack number was measured by counting amount of cracks within a given area. The test method was modified slightly by taking into account the length of the cracks by counting how many cracks that crossed 30 parallel lines with 1 mm distance and length 25 mm each (30 mm by 25 mm area). A Leitz Laborlux polarised light microscope was used at 50 x magnification for measurements of crack densities, whereas efforts to determine crack widths were performed at higher magnification. Also other methods for the registration of cracks exist: Hornain and Regourd (4) and Ringot (9).

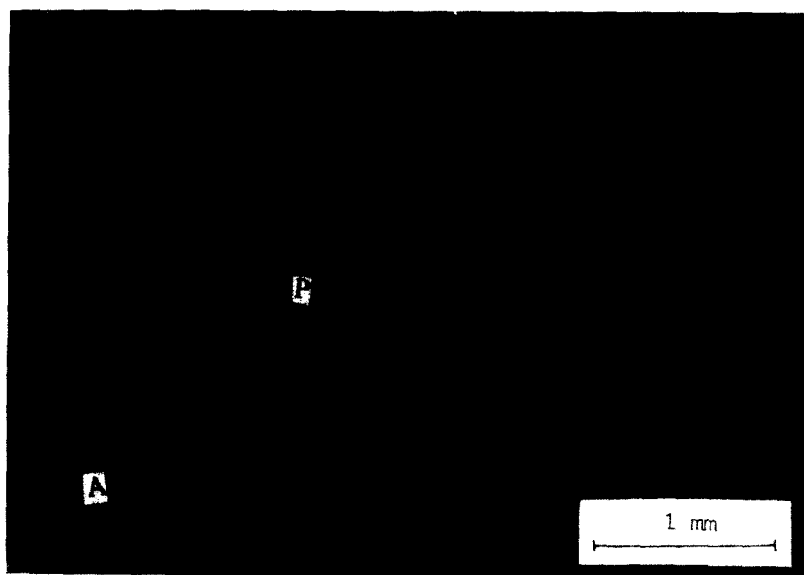


FIG. 1

040-00 Crack pattern after frost crack no. 777 (P: paste, A: aggregate)

Results and discussion

After exposure to rapid temperature cycling the concretes were deteriorated to various degrees with Durability Factors varying from 2 to 100, see table 1. (100 represents no loss of resonance frequency after 300 cycles.) The large variation was partly due to variation in test procedure (11), and partly due to variation in concrete quality. Cracks were observed on polished sections taken from deteriorated concrete beams. On the most deteriorated concretes a dense pattern of cracks could be seen with typical distances between cracks about 400 - 500 μm (040-00 and 040-05). In figures 1 and 2 photos taken from plane sections with typical crack patterns observed after frost deterioration are shown. Cracks traversed paste and most bond zones between aggregate and paste. The approximate half distance between cracks in the most deteriorated concretes seems to be in the neighbourhood or a little higher than the critical air void spacing factor normally required for these kinds of concretes to survive the ASTM C666 procedure A test: 200 - 250 μm . No cracks were observed on any of the concretes before exposure to rapid freezing and thawing in water. This is important since microcracking has been observed on thin sections of virgin high strength concretes. That kind of cracking might therefore be connected to preparation operations if they include drying by vacuum or heating. This has been discussed and investigated in more detail elsewhere (1, 2, 7, 12).

In figure 3 a plot of Crack number vs. Durability Factor is shown. From the figure it can be seen that a certain scatter appears. Two polished sections per variable may have improved the correlation. Still a fair relationship between Durability Factor and the logarithm of the Crack number exists. The points connected by lines represent specimens from each of the three different types of freeze/thaw cycle used in (2, 11). In (2, 11) it was found that type of cycling had an effect on cracking. Figure 3 indicates that few cracks result in relatively large damage for concretes with higher Durability Factors. Concretes with lower Durability Factors and more

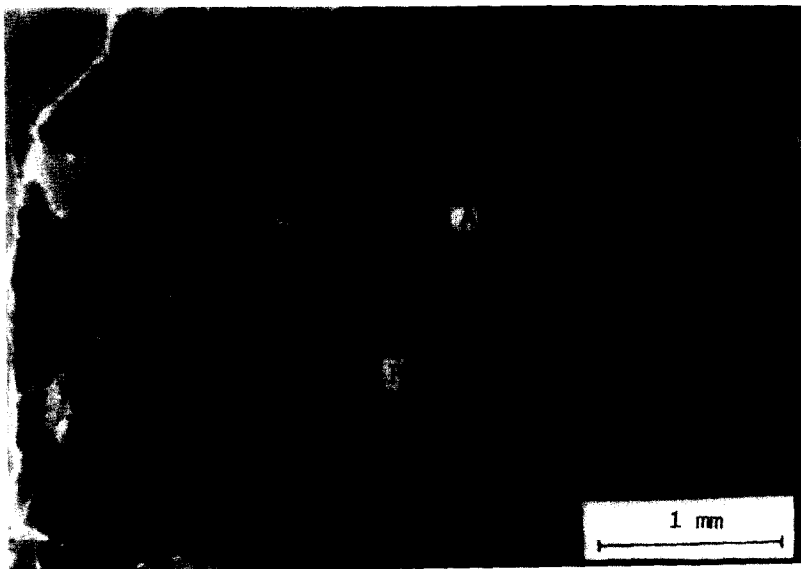


FIG 2

040-05 Crack pattern after frost, crack no. = 665 (P: paste, A: aggregate)

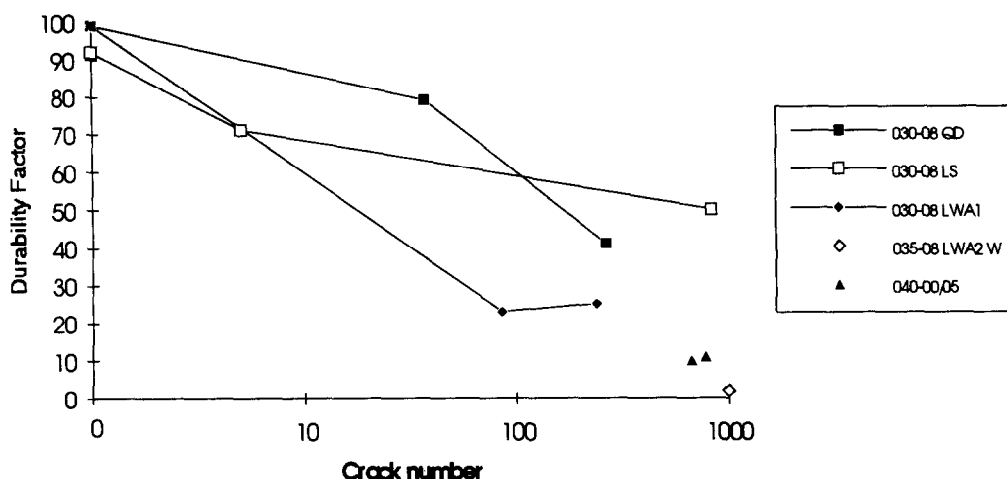


FIG. 3

Durability Factor vs. crack no. ($DF = -26.93 \cdot \log(\text{crack no}) + 96.5$, $r = -0.90$, 12 sets of data)

cracks, on the other hand, may cover a larger range of crack numbers. The results therefore indicate a rather low threshold value of cracking/damage beyond which the damage decelerates for this particular freeze/thaw test and concretes. Such a threshold value between damage and development of permeability was observed by Breyse, Gerard and Lasne (13).

Some larger cracks were seen on the surface of the most deteriorated specimens. The polished sections revealed rather dense microcrack patterns, as shown in figures 1 - 2, over most of the cross section of the beams. The rather small crack widths were somewhat difficult to judge with the optical microscope. The order of magnitude was about $10 \mu\text{m}$, but may vary somewhat. This has been studied in more detail by scanning electron microscopy (SEM) in (14). For the 040-00 and 040-05 concretes the volume of the freeze/thaw specimens were measured at each measurement of relative dynamic modulus of elasticity. The volume increase of the beams at stop of freeze/thaw test for 040-00 and 040-05 were 0.8 and 1.4 % by volume, as measured by weighing in air and water, (10). Crack volume calculated assuming a square crack pattern with sides 450 by 450 μm and crack width 5 μm , gives a crack volume of $(5+5) \times 450 \mu\text{m}^2 / (450 \times 450) \mu\text{m}^2 = 0.022$ i.e. 2.2 %. The assumed crack pattern is simplified but still indicates that the volume increase of the specimen corresponds roughly to the volume of cracks.

After self healing of the 040-00 and 040-05 beams, measured by regained resonance frequency after 3 months storage in water, polished sections were made in the same manner as described earlier, one from each of the two beams. On these polished sections no cracks could be observed. The type of cracks that were filled with dye dissolved in alcohol immediately after deterioration could apparently not be filled after subsequent self healing. It is important to note that the almost 100 % recovery of the resonance frequency and pulse velocity only gave a minor recovery of lost compressive strength on frost deterioration. The strength loss on deterioration was 22 - 29 %, but the recovery was only 4 - 5 %. The non-destructive methods do therefore not reflect the concrete strength. However, the results of the self healed plane sections indicate that the healing hindered the penetration of alcohol and/or the fluorescent dye into the cracks. This has been studied further in (14).

An other important aspect of the results is the very dense crack patterns observed. Such high crack densities have not been observed by the authors during many years of investigations at The Norwegian Building Research Institute on cores taken from concrete structures in Norway damaged by various types of exposure. The observed self-healing might be an explanation for this.

Conclusion

An investigation of cracks in concrete due to frost deterioration after rapid freezing and thawing in water has been performed by microscopy on polished sections impregnated by a fluorescent dye dissolved in ethanol (FLR technique). The technique is designed to avoid creating cracks during specimen preparation.

The results show that the non-air entrained high strength concretes investigated were crack free before freeze/thaw exposure. After internal damage due to rapid freezing and thawing in water, cracking was observed on the polished sections. Measured crack densities correlated fairly with the measured Durability Factors. On the most deteriorated concretes crack patterns with average grid size of 400 - 500 μm were observed. Cracks followed around most of the aggregate particles. Estimated crack volume correlated reasonably well with measured volume increase of concrete beams after frost deterioration. The observed crack densities are very high compared to what has been observed by the authors on field concretes. Self healing may be an important factor for this.

Acknowledgement

The research was carried out with financial support from The Norwegian Research Council/Norcon programme, Public Roads Laboratory, Norcem and Norwegian Contractors. The first author thanks The Norwegian Research Council for the scholarship provided.

References

- (1) Gran, H.C.: Fluorescent liquid replacement technique. A means of crack detection and water.binder ratio determination in high strength concretes, *Cem.Conc.Res* Vol.25 No.5 pp.1063-1074 (1995)
- (2) Bakke, J.A.: Diploma thesis, The Norwegian Institute of Technology/The Norwegian Building Research Institute, (1993) (In Norwegian).
- (3) Marchand, J.: PhD-thesis École Nationale des Ponts et Chaussées, 326 p. (1993)
- (4) Hornain, H. and Regourd M.M.: 8th Int.congr.on the chemistry of cement, Rio de Janeiro, V-4, pp.53-59 (1986) (In French)
- (5) Hornain H.: Conf. in Tribute to M.Regourd, University of Sherbrooke, Canada 31 p., (1994) (In French)
- (6) Kukko, H.: VTT Publication 126, Finland, (1992)
- (7) Rønning, T.F. and Gran, H.C.: High strength concrete materials development report 5.10, STF70 A92055, SINTEF, Trondheim Norway (1991) (In Norwegian with English summary)
- (8) DTI (Danish Institute of Technology) Test method TI-B5 Denmark 1987 (In Danish)
- (9) Ringot E.: *Cem. Conc. Res.* Vol.18, pp.35-43 (1988)
- (10) Jacobsen, S.: PhD-thesis in preparation, The Norwegian Institute of Technology, (1995)
- (11) Sellevold, E.J., Jacobsen S., Bakke J.A.: The Int. Worksh. on Freeze/thaw and Deicing Salt Scaling Res. of Conc. Ed.: J.Marchand and M.Pigeon, Laval University, Québec, Canada, pp. 155-165 (1993)
- (12) Rønning, T.F.: Elkem Materials, data sheet 3.2.4, Properties (1989)
- (13) Breyse D., Gérard B. and Lasne M.: ACI-SP 145, pp.1013-1032, (1994)
- (14) Jacobsen, S., Marchand J., Hornain, H.: SEM-observations of the microstructure of frost deteriorated and self-healed concretes, paper submitted for publication in *Cem. Conc. Res.*