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Non-destructive test methods application for structure analysis of ultra-high performance concrete after deterioration of cyclic salt-scaling

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1. Introduction

Concrete deterioration due to cyclic freezing and thawing is one of the most commonly occurring aggressive environments in Lithuania. However despite the fact there are only two standard test methods (for internal frost damage – LST L 1425.17 and for surface scaling – LST EN 1338) which allows estimating concrete condition. Nor internal frost damage (critical value: compressive strength reduction ≤ 5%) nor surface scaling (critical value: mass lost ≤ 1 kg/m^2) do not provide sufficient information about condition or changes in structure of concrete. Generally, if concrete specimens reached critical or nearly critical values, deterioration in structure is too severe and usually do not meet anymore certain exploitation requirements. In order to apply nondestructive test method for structure analysis of UHPC after certain cycles of salt-scaling it is necessary properly understand the structure of the material, destruction mechanism of frost damage and correctly interpret data of applied test method.

Many scientists agree with idea that resistance to frost damage of concrete mostly depends on: aggregates resistance to frost damage; cement matrix interaction with aggressive environment; interfacial transition zone; permeability of aggregates and cement matrix including interfacial transition zone. [1-3]. Another assumption with which agrees most scientists is, that proper porosity of concrete has positive effect to frost resistance. Proper porosity for each type of concrete has to be specific and resistance to frost damage mainly depends on: proper size of air pore, spacing factor, degree of saturation, rate of freezing, water to cement ratio and etc. [4-6]. W. Micah Hale investigated the need for air entrainment in high performance concrete (HPC) and founded that concrete with no air entrainment can be produced with adequate frost resistance when $w/c \leq 0.36$ and air content is 4%. [7]. J. T. Kevern founded that even without air entrainment pervious concrete has spacing factor less than 200 μm and that value is more than enough for freeze-thaw resistance [8]. Similar experiment results were obtained by Claus Germann Petersen [9]. John J. Valenza II states that concrete prepared with $w/c \leq 0.30$ does not require air entrainment to resist salt scaling, because there is very little bleeding and the surface strength does not deviate greatly from the overall strength, which is normally greater than that which indicates a low susceptibility to salt scaling. [10, 11]. Dipayan Jana founded that high performance concrete with $w/c \leq 0.30$ can be prepared and without air entrainment even than spacing factor is greater than 200 μm because HPC has very low permeability to moisture and a low amount of freezable water. [12]. Since porosity parameters of HPC ($w/c < 0.30$) do not have significant effect to frost resistance it can be assumed that aggregates, cement matrix and interfacial transition zone mostly affect deterioration of structure. L. Basheer noticed that permeability of concrete can be reduced by reducing average particle size of coarse aggregate. [13]. G. A. Lehrsch founded that the aggregates with particle size less than 3 mm practically does not absorb moisture [14]. According to literature review can be stated that concrete with low permeability to moisture, with all aggregates resistant to frost damage and less than 3 mm when $w/c < 0.30$ should be frost resistant and frost resistance mainly depends on cement matrix and interfacial transition zone. Many scientists agree with idea that pozzolanic materials (silica fume, fly ash, metakaolin, blast furnace slag) can improve structure of concrete and decrease permeability to moisture of cement matrix and interfacial transition zone. [15-17]. T.P. Chang founded that durability and structure of reactive powder concrete can be improved with two different curing regimes combining water-curing at 25°C with steam-curing at 85°C. That curing regime should increase up to 38% compressive strength of cylindrical specimens [18]. H. Famili states that thermal treatment not only improves structure of high strength self-consolidating concrete but also has positive effect to frost resistance [19].

UHPC is one type of concrete, which could meet all necessary durability requirements and be frost resistant. Notwithstanding these facts, but in durability terms UHPC is still a young material and there is no sufficient information about how material with very low permeability to moisture coefficient and low $w/c \leq 0.30$ ratio will behave in one or another aggressive environment and what kind of test methods could be applied for structure analysis. Jimin Guoa proposed for structure analysis of very high strength concrete to apply dynamic modulus of elasticity, dynamic modulus of rigidity, Poisson's ratio, ductility factor and Modulus of Rupture test methods. [20]. V. Vaitkevičius proposed for structure analysis of UHPC to apply dynamic modulus of elasticity, ultrasonic pulse velocity and fluorescence test methods [21]. Stefan Jacobsen during research on high performance concrete noticed, that dynamic modulus of elasticity after cyclic freeze-thawing can be recovered almost completely during subsequent storage in water but compressive strength will be recovered only 5%

after 22-29% reduction. [22]. Therefore it could be assumed that dynamic modulus of elasticity test method cannot be always reliable. For salt-scaling Héctor L. proposed ultrasonic pulse velocity test method and to measure the length change in the specimen [23]. Mahmoud Nili tried to establish a correlation between mass loss of salt-scaling and change of compressive strength [24]. Yang Quanbing founded relationship between mas loss of salt-scaling and spacing factor [25]. Bertil Persson also offered to measure length change after cyclic salt-scaling [26]. According to literature review there is no so much test methods for structure analysis of concrete after cyclic salt-scaling, despite these facts many scientists forget, that application of test method mainly depends on functional relationship of applied test method and investigated deteriorated property of concrete. Thus main aim of the experiment was to determine possibility of non-destructive test methods application for structure analysis and to find functional relationship between applied test methods and mass loss of salt-scaling.

2. Materials used for the research

Cement. Portland cement CEM I 52.5 R was used in experiment. Main properties: paste of normal consistency– 29.3%; soundness (Le Chatielier) – 1.0 mm; initial setting time – 145 min; compressive strength (after 2/28 days) – 38.6/65.3 MPa. Mineral composition: C_3S – 57.26; C_2S – 15.41; C_3A – 8.68; C_4AF – 10.15. Chemical composition of Portland cement is shown in Table 1 and particle size distribution presented in Fig. 1.

Silica fume. Silica fume, also known as microsilica (MS) or condensed silica fume is a by-product of the production of silicon metal or ferrosilicon alloys. Main properties: density – 2120 kg/m^3, bulk density (free-flow/compacted) – 255/329 kg/m^3, hygroscopicity 158%, natural fall angle 54º. Chemical composition of silica fume is shown in Table 1 and particle size distribution presented in Fig. 1.

Table 1

Chemical compositions of Portland cement, silica fume and glass powder

Components	Quantity, %		
	CEM I 52.5 R	Glass powder	Silica fume
SiO_2	20.61	72.76	92.08
TiO_2	-	0.04	-
Al_2O_3	5.45	1.67	1.16
Fe_2O_3	3.36	0.79	1.24
MnO	-	0.02	-
MgO	3.84	2.09	0.80
CaO	63.42	9.74	1.07
SO_3	0.80	0.10	1.27
Na_2O	0.20	12.56	1.13
K_2O	1.00	0.76	0.67
P_2O_5	-	0.02	-
$Na_2O_{eq.}$	0.86	13.06	1.57
Loss of ignition	1.00	1.00	-

Sand. In experiment ordinary sand was used. Main properties: fraction 0/2; average density – 2670 kg/m^3, bulk density – 1625 kg/m^3, impurities ≤ 1.5%.

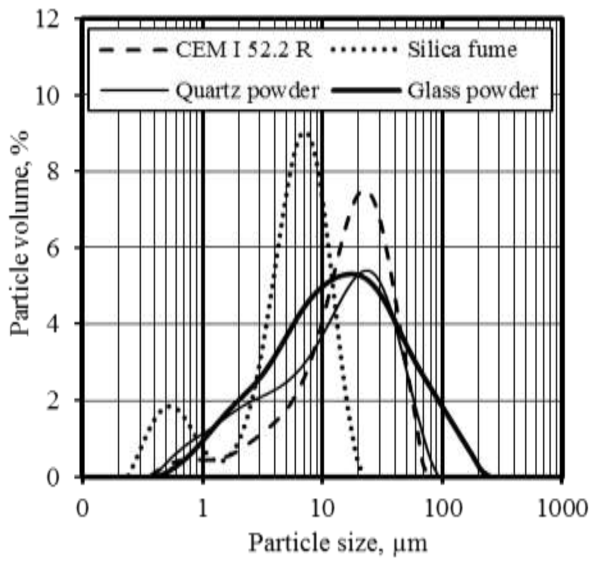

Fig. 1 Particle size distribution of cement, glass powder silica fume and glass powder

Glass powder. Glass powder was produced by various tares of glass. Main properties: specific surface area – 1485 cm^2/g; bulk density – 1245 kg/m^3; density – 2266 kg/m^3. Chemical composition of glass powder and is shown in Table 1 and particle size distribution presented in Fig. 1.

Quartz sand. In experiment quartz sand was used. Main properties: fractions: 0/0.5 and 0/2; density 2670 kg/m^3, bulk density 1600 kg/m^3, impurities ≤ 0.5%.

Chemical admixture. In experiment was used superplasticizer based on polycarboxylic ether (PCE) polymers. Main properties: appearance: dark brown liquid, specific gravity (20°C) – 1.010÷1.070 g/cm^3, alkali content – 2.5%, chloride content – 0.1%.

Additional materials. Fluorescent dye PFINDER 902 and fluorescent dye developer PFINDER 970 designated for surface defect detection was used for experimental research.

3. Testing procedures

Sample preparation and curing. Fresh concrete mixes were prepared in laboratory vibrating mixer, which allows production of homogeneous mixes with very low w/c. Mixing procedure was performed according to [21].

Table 2

Compositions of ultra-high performance concrete

Composition	w/c	Water, l	Cement, kg/m^3	Micro fillers, kg/m^3		Aggregates, kg/m^3			Super plasticizer, l
				Silica fume	Glass powder	Sand	Quartz sand		
						# 0/2	# 0/0.5	# 0/2	
No.1	0.24	176	735	99	412	-	96	866	36.76
No.2	0.24	176	735	-	512	-	96	866	36.76
No.3	0.24	176	735	-	512	962	-	-	36.76

Homogeneous mixes were cast in moulds, compacted for 30 seconds on a vibrating table CM 539 and kept for 24 hours at 20°C/95 RH. After 24 hours specimens were demoulded and stored in water at 20±2°C for 7 days. Then hot water curing was applied to specimens at 80°C for 3 days (thermal regime 2+67+3). After thermal treatment all specimens were restored in water at 20±2°C until 28 days of age.

Salt-scaling and porosity parameters. Salt-scaling of concrete was performed according to EN 1338:2003 standard [27]. For experiment were used cylinders (*d* = 50 mm and *h* = 50 mm) and prisms (7x7x21 cm).

Structure analysis. For structure analysis were used ultrasonic pulse velocity, dynamic modulus of elasticity, fluorescence and compressive strength test methods. Ultrasonic pulse velocity was measured according to EN 12504-4:2004 standard [28] and dynamic modulus of elasticity was measured by EN 14146:2004 standard [29]. Compressive strength was determined after 28 days according to EN 12390-4:2000 standard [30]. Surface cracks were detected by fluorescence test method with optical microscope OLYMPUS BX51TF.

4. Results and discussions

Compositions and main properties of hardened concrete are shown in Tables 2 and 3 respectively. Main goal of the experiment was to determine possibility of non-destructive test methods application for structure analysis and to find functional relationship between applied test methods and mass losses of salt-scaling.

Table 3

Physical/mechanical properties of concrete

Characteristics	Composition		
	No.1	No.2	No.3
Density, kg/m^3	2407	2434	2422
Compressive strength (28 days), MPa	166	171	224
Relative water absorption, %	1.61	1.72	1.94
Mass loss of salt-scaling (40 cycles), kg/m^2	0.0249	0.0058	0.0034

Ultrasonic pulse velocity, dynamic modulus of elasticity, compressive strength and fluorescence test methods were applied for structure analysis of UHPC after cyclic salt-scaling. Relationships between applied test methods and mass losses of salt-scaling were determined. Properties of material were determined before experiment and after 40 cycles of salt-scaling. Surface scaling test method was performed in 3% NaCl solution.

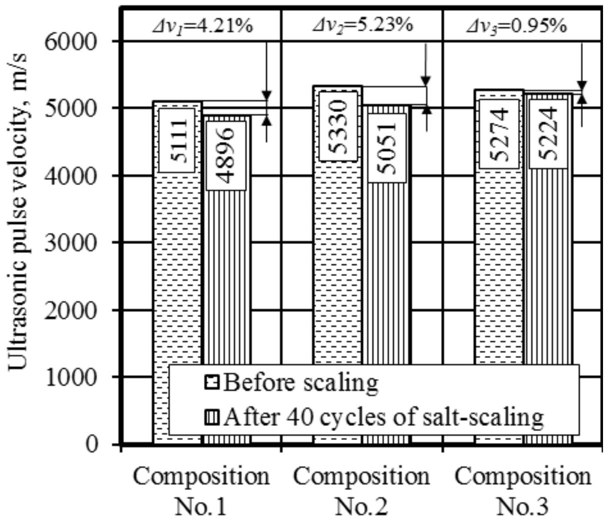

Fig. 2 Ultrasonic pulse velocity of concrete: before experiment and after 40 cycles of salt-scaling

Notwithstanding of UHPC composition, decrease of ultrasonic pulse velocity (Fig. 2) and mass losses of salt-scaling at 40 cycles (Table 3) were insignificant.

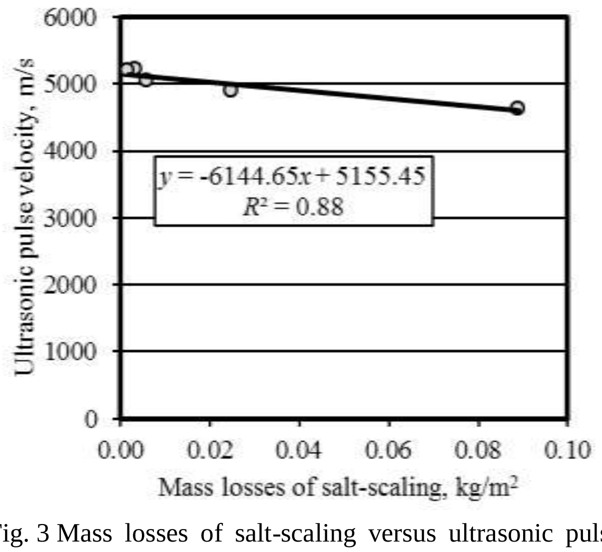

Fig. 3 Mass losses of salt-scaling versus ultrasonic pulse velocity at 40 cycles

Best results of compressive strength and salt-scaling resistance were noticed in composition with ordinary sand and glass powder (composition No. 3). In the same composition minimal reduction of ultrasonic pulse velocity (decreased by 0.95%) and mass loss of salt-scaling (decreased by 0.0034 kg/m^2) were observed.

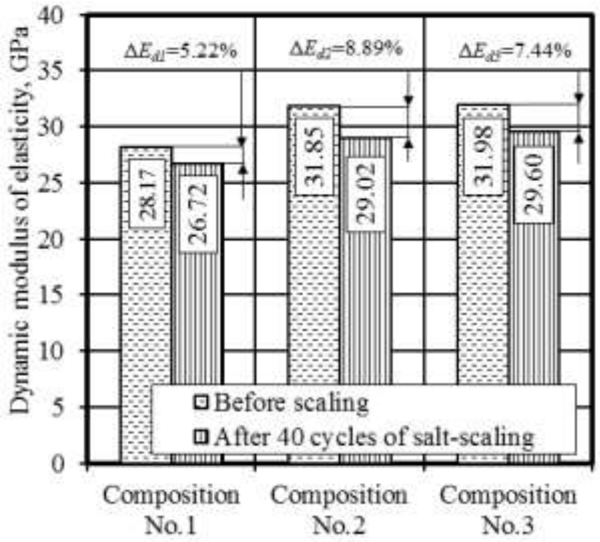

Fig. 4 Dynamic modulus of elasticity: before experiment and after 40 cycles of salt-scaling

Maximal decrease of ultrasonic pulse velocity (decreased by 5.23%) was noticed in composition (No.2) with quartz sand and glass powder. Maximal mass losses of salt-scaling (decreased by 0.0249 kg/m^2) were noticed in composition (No.1) with quartz sand, glass powder and silica fume. Interesting fact was noticed, that all compositions of UHPC and all properties of substituted materials were almost the same, however maximal salt-scaling were observed in composition (No.1) with silica fume. In the same composition (No.1) salt-scaling at 40 cycles was more than 4 times higher comparing with others (No.2 and No.3).

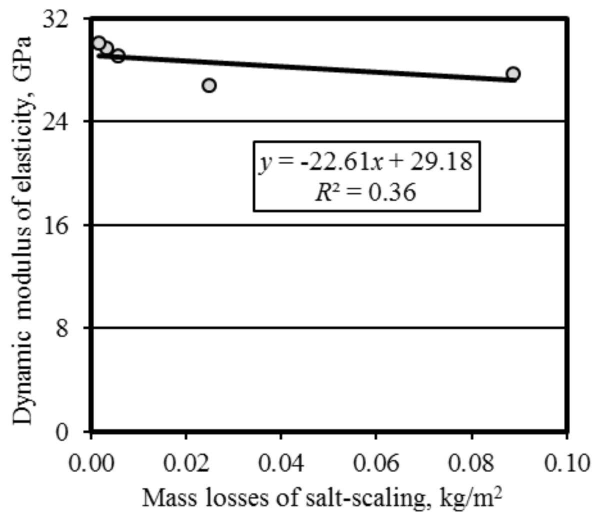

Fig. 5 Mass losses of salt-scaling versus dynamic modulus of elasticity at 40 cycles

Reduced ultrasonic pulse velocity indicates that cracking initiated in structure of UHPC. Relationship between mass losses of salt-scaling and ultrasonic pulse velocity at 40 cycles (Fig. 3) shows strong correlation coefficient (R^2=0.88). Experiments results allow assume that ultrasonic pulse velocity is one type on non-destructive test method, which could be applied for structure analysis after cyclic salt-scaling.

Interesting fact was observed with dynamic modulus of elasticity test method. During experiment was obtained higher reduction of dynamic modulus (Fig. 4), which allow assume, that dynamic modulus of elasticity test method is more sensitive than ultrasonic pulse velocity.

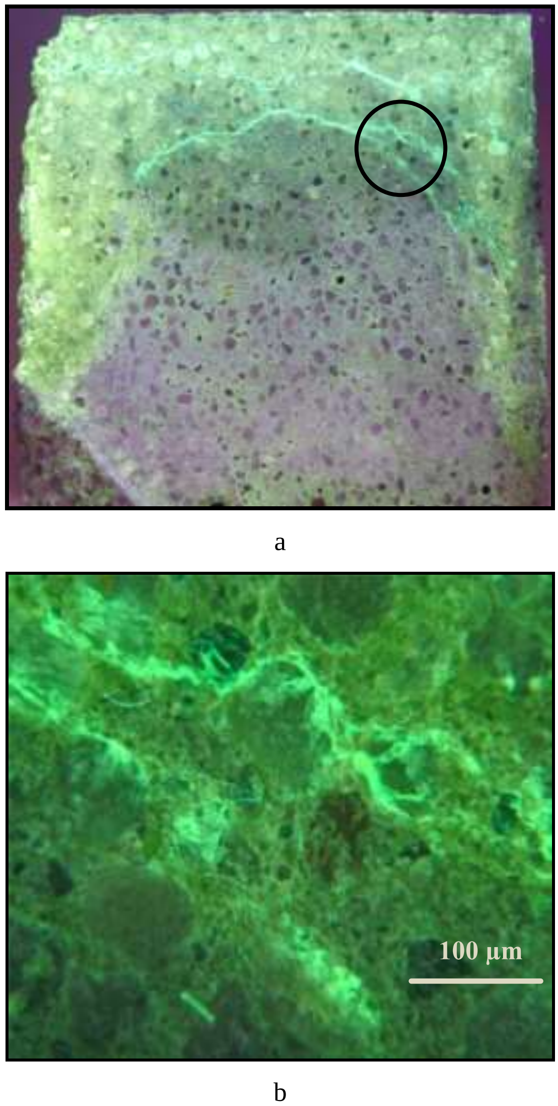

Fig. 6 Defects of structure identified by fluorescence test method: a - without magnification and b - with 50x magnification

Although the tendencies with both non-destructive test methods remained similar, however relationship between mass losses of salt-scaling and dynamic modulus of elasticity at 40 cycles was more than 2 times weaker (correlation coefficient R^2 = 0.36) comparing with ultrasonic pulse velocity test method (correlation coefficient R^2 = 0.88). According to the results of experiment, could be stated, that dynamic modulus of elasticity is not precise enough and should not be applied for structure analysis of UHPC after cyclic salt-scaling.

Different functional relationships strength between mass losses of salt-scaling and applied non-destructive test methods probably related due to distinct sensitivity to detect cracks in concrete structure. Cyclic salt-scaling initiated surface destruction process, which differently distributed in cross section of specimen. These experiments results demonstrate, that event after cyclic salt-scaling destruction process could initiate cracking in deeper layers. That assumption was confirmed by fluorescence test method (Fig. 6).

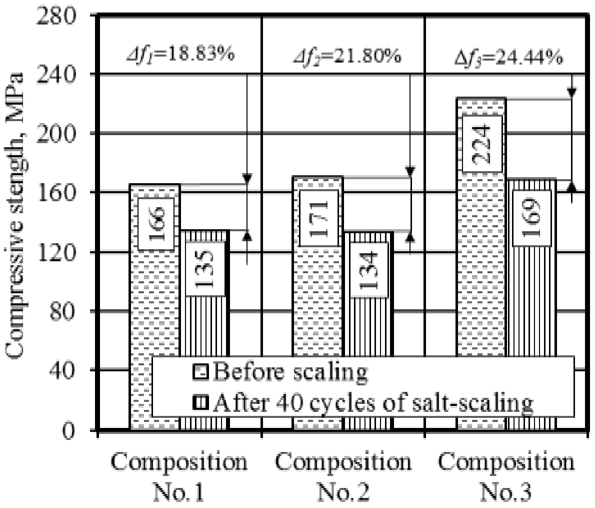

Fig. 7 Compressive strength of concrete: before experiment and after 40 cycles of salt-scaling

Micro-cracks after cyclic salt-scaling disproportionally distributed in cross-section of concrete and decrease in deeper layers. Different functional relationships strength between salt-scaling and applied test methods could be explained due to structure inhomogeneity of concrete in cross-section.

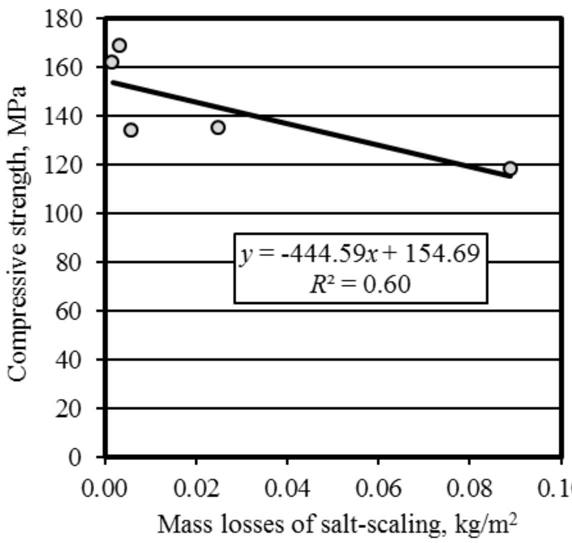

Fig. 8 Mass losses of salt-scaling versus compressive strength at 40 cycles

Another relationship was made between mass losses of salt-scaling and compressive strength (Fig. 8). Tendencies also were obtained very similar as with ultrasonic pulse velocity (Fig. 3) or dynamic modulus of elasticity test methods (Fig. 5). It could be noticed, that functional relationship of applied test method (Fig. 8) is insufficient for structure analysis of concrete after cyclic salt-scaling (correlation coefficient $R^2 = 0.60$). Although compressive strength method is not classified as non-destructive test methods, but also should not be used for structure analysis of UHPC after cyclic salt-scaling.

Another relationship was made between mass losses of salt-scaling and relative water absorption (Fig. 9). It could be observed that with increasing relative water absorption and salt-scaling also proportionally increased (correlation coefficient $R^2 = 0.89$). Although with this simple test methods cannot be applied to predict mass losses of salt-scaling, but could be simplest way to find out which composition will have lower resistance to deleterious environment.

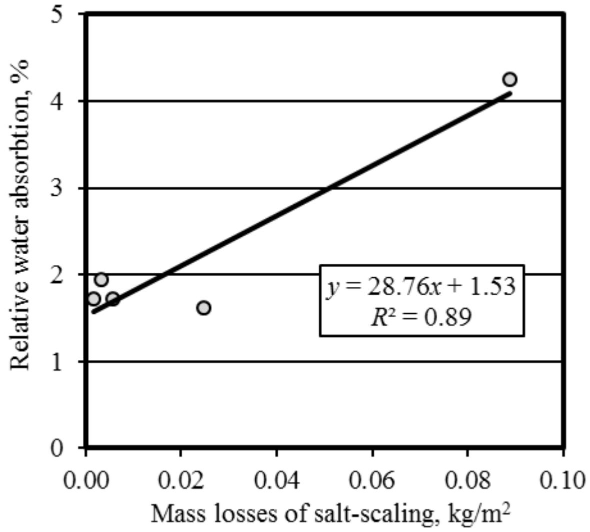

Fig. 9 Mass losses of salt-scaling versus relative water absorption at 40 cycles

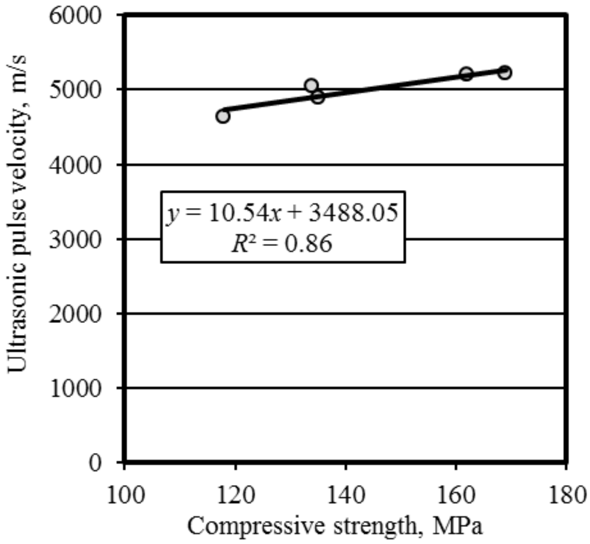

Fig. 10 Compressive strength versus ultrasonic pulse velocity at 40 cycles

In order to find out how applied test methods correlate with each other, also were made others functional relationships (Figs. 10-12). The strongest functional relationship was observed between ultrasonic pulse velocity and compressive strength test methods (correlation coefficient $R^2 = 0.86$). It could be assumed, that applied test methods could be used for structure analysis after cyclic salt-scaling, however relationship between mass losses of salt-scaling and compressive strength test method was insufficient.

Also were made relationships between compressive strength and dynamic modulus of elasticity or dynamic modulus of elasticity and ultrasonic pulse velocity also showed insufficient correlation. Although ultrasonic pulse velocity test methods showed strongest correlation with salt-scaling, which could suggest, that applied method could be used for structure analysis of UHPC after cyclic salt-scaling, however one test method which do not correlate with other applied methods is not accurate and sufficient for proper structure analysis.

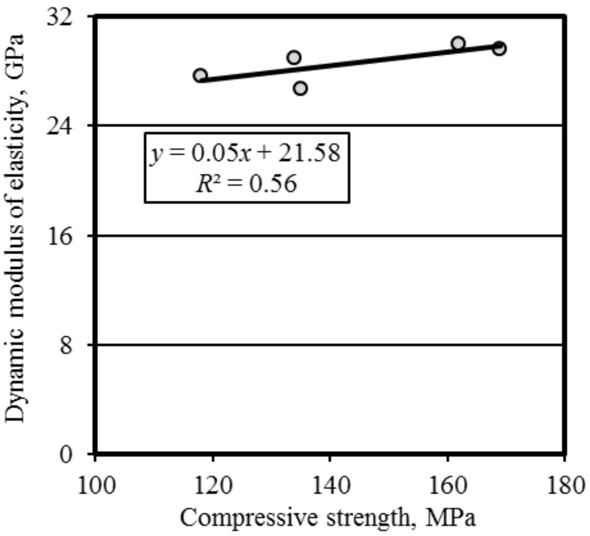

Fig. 11 Compressive strength versus dynamic modulus of elasticity at 40 cycles

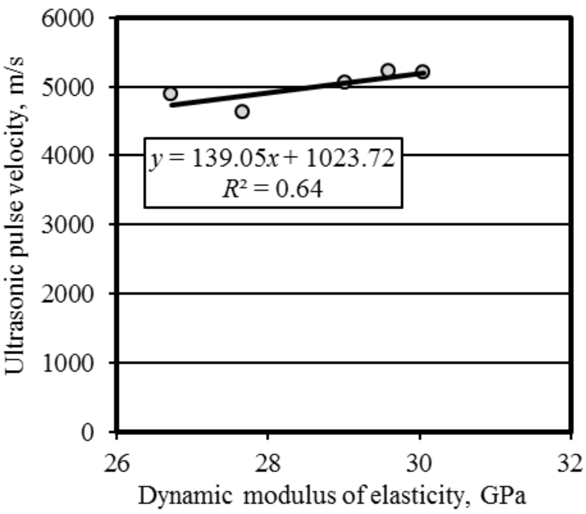

Fig. 12 Dynamic modulus of elasticity versus ultrasonic pulse velocity at 40 cycles

Several test methods were applied for structure analysis of ultra-high performance concrete after deterioration of cyclic salt-scaling. All applied test methods were not sensitive enough for structure analysis had very little change of ultra-sonic pulse velocity or dynamic modulus of elasticity and had very high deviation coefficient. Experiments results gives doubts about used methods applicability.

Visual inspection by fluorescence test method revealed that deterioration by surface scaling is not homogeneous. The greatest damage was observed on the frozen surface and gradually decreased in deeper layers. Surface scaling probably affected all cross section; however deterioration degree in each layer was different. Although concrete by nature is inhomogeneous material and in different circumstances applied methods should be perfect for structure analysis, however after cyclic surface salt-scaling concrete should be considered as composite material, which has several layers with its own properties. Therefore applied test methods were not accurate enough and inappropriate for structure analysis of ultra-high performance concrete. The question arises if there is at all any suitable test method for structure analysis when concrete is affected by cyclic salt-scaling?

5. Conclusions

1. Dynamic modulus of elasticity and compressive strength test methods due to insufficient functional relationship between applied methods and mass losses of cyclic salt-scaling should not be applied for reliable structure analysis of UHPC.

2. Ultrasonic pulse velocity is one type of non-destructive test methods, which could be applied for structure analysis of UHPC after cyclic salt-scaling deterioration (correlation coefficient R^2 = 0.88), however one test method is not sufficient.

3. Mass losses of researched compositions after 40 cycles of salt-scaling were between 0.0034 kg/m^2 to 0.0249 kg/m^2. Composition with ordinary sand and glass powder showed best salt-scaling resistance.

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NEARDANČIŲ TYRIMO METODŲ TAIKYMAS TIRIANT YPATINGAI STIPRAUS BETONO STRUKTŪRĄ PO CIKLIŠKO PAVIRŠINIO ŠALDYMO

R e z i u m ė

Eksperimento metu buvo pritaikyti neardantys tyrimo metodai siekiant ištirti ypatingai stipraus betono struktūros pokyčius po 40 paviršinio šaldymo ir atšildymo ciklų 3% NaCl tirpale. Ypatingai stipraus betono struktūros pokyčiams identifikuoti buvo pritaikytas ultragarsinis, dinaminio tamprumo modulio ir stiprio gniuždant nustatymo metodai. Siekiant įvertinti metodų tinkamumą sudarytos priklausomybės tarp taikyto tyrimo metodo ir masės nuostolių po 40 paviršinio šaldymo ciklų 3% NaCl tirpale. Paviršiniai struktūros mikro įtrūkimai buvo identifikuoti fluorascensiniu metodu. Pagrindinis straipsnio tikslas nustatyti, kuris neardantis tyrimo metodas yra tinkamiausias tiriant struktūros pokyčius įvykusius po paviršinio šaldymo ir atšildymo. Pasaulinėje praktikoje yra paskelbta tik keletas mokslinių tyrimų ir publikacijų apie ypatingai stipraus betono atsparumą paviršiniam cikliškam šaldymui ir atšildymui, ir dar mažiau atlikta tyrimų, kuriais būtų galima įvertinti ypatingai stipraus betono struktūros pokyčius neardančiais tyrimo metodais, Europoje šie tyrimai iki šio dar nebuvo atlikti.

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NON-DESTRUCTIVE TEST METHODS APPLICATION FOR STRUCTURE ANALYSIS OF ULTRA-HIGH PERFORMANCE CONCRETE AFTER DETERIORATION OF CYCLIC SALT-SCALING

S u m m a r y

Non-destructive test methods for structure analysis of ultra-high performance concrete were applied after 40 cycles of salt-scaling. Salt-scaling test method was performed using 3% NaCl solution. The main aim of the experiment was to find new way to observe structure changes in ultra-high performance concrete products, which could help for scientists better understand deterioration mechanism of concrete after cyclic salt-scaling. Salt-scaling of UHPC were investigated by ultrasonic pulse velocity, dynamic modulus of elasticity and compressive strength methods. Surface defects were visually identified by fluorescence test method. There are a few publications about salt-scaling resistance of UHPC and fewer articles about nondestructive test methods how to evaluate structure of concrete after certain cycles of salt-scaling, however such research in Europe is not done at all.

Keywords: ultra-high performance concrete, mass losses of salt-scaling, non-destructive test methods.

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