

Nondestructive testing of ultra-high performance concrete to evaluate freeze-thaw resistance

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1. Introduction

Rapid development of construction technologies creates a higher demand for materials, which have not only higher compressive strength, but also better durability properties. Freeze-thaw resistance is one of the most important durability properties of concrete and reinforced concrete structures used in Lithuania. Although freeze-thaw resistance of concrete products may be easily improved by adding air entraining additives, these chemical agents significantly reduce concrete's compressive strength [1].

Ultra-high performance concrete (UHPC) is one of the materials which could meet required strength and durability properties [2, 3]. In Lithuania UHPC is known as concrete which has compressive strength over 100 MPa [4]. By adding pozzolanic additives compressive strength can be easily increased up to 150-200 MPa [5]. Such high compressive strength may be achieved by the following measures: elimination of coarse aggregate makes the mixture more homogeneous [6]; higher content of fine aggregates improves the granular composition of the mixture [7]; components of the mixture are selected with similar modulus of elasticity to achieve a more uniform compressive deformation of concrete [8]; properties of concrete matrix are improved by adding pozzolanic additives [9]; W/C ratio reduced by adding high range water reduces (superplasticizers); thermal treatment of specimens also improves the concrete's tensile stress-strain behavior and the addition of steel or polypropylene fibers reduces brittle failure fracture of the concrete [10].

Concrete's freeze-thaw resistance depends of the resistance of each component to freezing and thawing [11]. The typical UHPC mixture consists of: sand, cement, microsilica, crushed quartz sand, fibers, superplasticizer and water. As the coarsest UHPC component is sand (the coarsest particle size is approximately 2 mm) this aggregate and other finer aggregates will have no effect on the freeze-thaw resistance of concrete because finer than 2-3 mm particles substantially reduce the water absorption of the aggregate [12], whereas dry or semi-dry aggregates are not susceptible to destructive freeze-thaw effect. UHPC concrete has very low porosity, often below 2%; therefore the cement matrix will have the greatest effect on concrete's freeze-thaw resistance [13]. Low water absorption and low capillary and closed air porosity (average size of pores 6×10^{-9} - 50×10^{-9} m [14]) increases the freeze-thaw resistance of concrete, but cyclic freezing and thawing has a cumulative effect, which causes microcracking of the cement matrix [15, 16]. The formation of microcracks is very difficult to observe, while visible cracks may have a

critical effect on the strength and reliability of the structure. Nondestructive testing is one of the methods that enable to observe the change in concrete's long-term strength and durability parameters. The aim of the experiment described in this paper is to predict UHPC freeze-thaw resistance and to determine the change in compressive strength of concrete subjected to recurrent freeze-thaw cycles and also to perform interim concrete quality control by applying ultrasonic and dynamic modulus of elasticity methods.

2. Materials used for the research

SiO₂ microsilica. SiO₂ microsilica manufactured by BASF was used for experimental research.

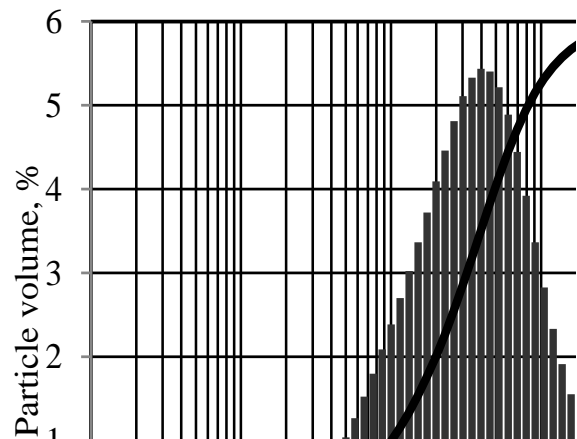


Fig. 1 Granular curve of SiO₂ microsilica

The main properties of SiO₂ microsilica: density 2120 kg/m³, bulk density (free-flow/compacted) 255/329 kg/m³, specific surface area 3524 m²/kg, hygroscopicity 158%, natural fall angle 54°. Chemical composition: SiO₂ (92.08%), Al₂O₃ (1.16%), Fe₂O₃ (1.24%), CaO (1.07%), MgO (0.80%), SO₃ (1.27%), K₂O (0.67%), Na₂O (1.13%). Fig. 1 exhibits the granular curve. The properties meet LST EN 12620:2003+A1:2008 standard requirements.

Granite. Granite produced by UAB Granitas and crushed to 0/2 fraction was used for the experimental research. The main properties: density 2670 kg/m³, bulk density 1600 kg/m³, clay and dust particle content 0.5%. Chemical composition: SiO₂ (72.04%), Al₂O₃ (14.42%), K₂O (4.12%), Na₂O (3.69%), CaO (1.82%), FeO (1.68%), Fe₂O₃ (1.22%), MgO (0.71%), TiO₂ (0.30%), P₂O₅ (0.12%), MnO (0.05%). Fig. 2 exhibits the granular curve. The properties of the filler meet LST EN 12620:2003+A1:2008 standard requirements.

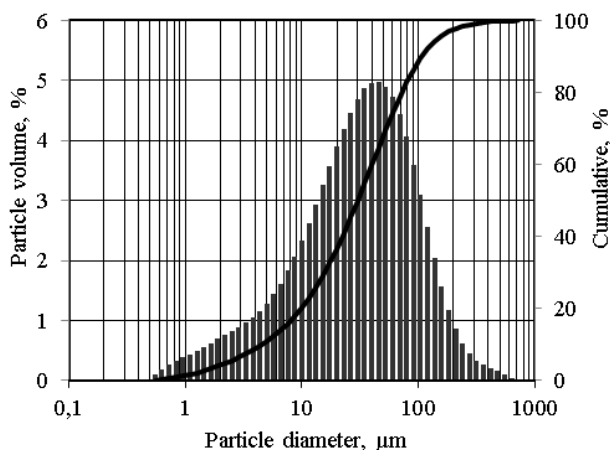


Fig. 2 Granular curve of crushed granite

Sand. Sand from Zatyšiai quarry of average coarseness (fraction 0/2 mm, average density 2670 kg/m^3 , bulk density 1625 kg/m^3 , air entraining potential 39.1%, clay and dust content 1.5%) was used for experimental research. Chemical composition of the sand: SiO_2 (82.39%), Fe_2O_3 (1.32%), TiO_2 (0.21%), Al_2O_3 (3.89%), CaO (3.74%), MgO (0.54%), R_2O (1.40%), SO_3 (0.15%), impurities (6.37%). The properties of the aggregate meet LST EN 12620:2003+A1:2008 standard requirements.

Quartz sand. Quartz sand from Anykščiai quarry (0/1 and 0/2 fractions, density 2670 kg/m^3 , bulk density 1600 kg/m^3 , clay and dust content 0.5%) was used for experimental research.

Cement. Portland cement CEM I 52,5R produced by the *Cemex* factory located in Sweden was used for the research. The specifications of the cement: specific surface area $370 \text{ m}^2/\text{kg}$, paste of normal consistency 27.3%, initial setting time 130 min., final setting time 215 min., compressive strength 30.6 MPa (after 2 days) and 57.6 MPa (after 28 days), flexural strength 6.0 MPa (after 2 days) and 8.1 MPa (after 28 days). Mineral composition of Portland cement: C_3S -61%, C_2S -12%, C_3A -7%, C_4AF -13%. The properties were determined accordance to LST EN 197-1:2001/A3:2007 standard.

2.1. Chemical admixtures

Superplasticizer *Glenium ACE 30* produced by *BASF* was used for experimental research. It is a polycarboxylate ether-based superplasticizer. Technical information: active substance: polycarboxylate ethers, appearance: dark brown liquid, density: $1.06 \pm 0.02 \text{ g/cm}^3$, hydrogen ion concentration (pH): 6.5 ± 1.5 , maximum chloride content (by weight): 0.10%, maximum equivalent alkali content expressed as Na_2O (by weight) 1.0%, optimum ambient temperature $+20^\circ\text{C}$.

Superplasticizer *Glenium ACE 430* produced by *BASF* was used for experimental research. It is a polycarboxylate ether-based superplasticizer. Technical information: active substance: polycarboxylate ethers, appearance: dark brown liquid, density: $1.06 \pm 0.02 \text{ g/cm}^3$, hydrogen ion concentration (pH): 5.5 ± 1.5 , maximum chloride content (by weight): 0.10%, maximum equivalent alkali content expressed as Na_2O (by weight) 0.6%, optimum ambient temperature $+20^\circ\text{C}$.

Nanoparticle suspension *X-SEED* produced by *BASF* was used for experimental research. It is a hydration

accelerator that stimulates the growth of calcium hydrosilicates (C-S-H). Technical information: appearance: white odourless nanoparticle suspension, density - 1.135 g/cm^3 , hydrogen ion concentration (pH) - 11.0, maximum chloride content (by weight) - 0.10%, maximum equivalent alkali content expressed as Na_2O (by weight) 4.0%, optimum ambient temperature $+20^\circ\text{C}$.

2.2. Additional materials

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

3. Testing procedure

Concrete mixtures were prepared from dry aggregates. Cement and aggregates were dosed by weight, water and chemical admixtures were added by volume (Table 2). Some chemical admixtures were dissolved in water and added together with water, other admixtures were added separately without water.

Table 1

UHPC mixing procedure

Time, sec.	Mixing procedure
60	Homogenization of sand, granite, microsilica and cement
30	Addition of the required water amount and 50% of the superplasticizer
60	Homogenization
120	Pause
30	Addition of the remaining superplasticizer
60	Homogenization

Super plasticizer *Glenium ACE 30* was used for the mixtures of composition 1, 2 and 3, super plasticizer *Glenium ACE 430* was used for the mixture of composition 4. Sand from Zatyšiai quarry was used for the mixture of composition 1, quartz sand from Anykščiai quarry was used for the mixtures of composition 2, 3 and 4.

The concrete was mixed in a vibrating mixer. The vibrating mixer, due to its unique design and mixing intensity, has better capability to produce more homogeneous concrete mixture of high viscosity and with the lowest water and cement ratio. The main parameters of the vibrating mixer: oscillation frequency: 30-500 Hz, capacity: 4 liters. The mixing process is started at the lowest frequency and is raised to the highest frequency over 2 minutes. The mixing procedure is described in Table 1.

Prisms ($40 \times 40 \times 160 \text{ mm}$) and cylinders ($d = 50 \text{ mm}$, $h = 50 \text{ mm}$) were formed for the research to determine concrete properties. The specimens were compacted for 30 seconds on a vibrating table CM 539 (vibration frequency 50 Hz, amplitude 0.5 mm) and were left for setting in laboratory environment ($20 \pm 2^\circ\text{C}$) for 24 hours, afterwards they were hardened in water ($20 \pm 2^\circ\text{C}$) for 5 days, steam cured in the oven for 24 hours (steam curing procedure 2 + 19 + 3 h, isothermal phase temperature 80°C) and left to harden in water ($20 \pm 2^\circ\text{C}$) for the remaining 21 day. Before testing all specimens were dried in laboratory oven (for 48 hours in temperature $100 \pm 5^\circ\text{C}$).

Composition of concrete mixtures, 1 m³

Composition	Water, l	C, kg	SiO ₂ , kg	Granite, kg		Sand, kg		Pl, l	X-SEED, l
				0/2	0/1	0/2	0/1		
No. 1	229	735	99	417	-	972	31.61	-	
No. 2	224	735	99	417	972	-	31.61	-	
No. 3	258	735	99	412	96	866	37.76	11.03	
No. 4	244	785	106	383	-	893	54.94	11.77	

Note: C - cement content, SiO₂ - SiO₂ microsilica, 0/2 – fraction of granite aggregate, 0/1 and 0/2 fractions of sand and quartz sand, PL: superplasticizer, X-SEED: nanoparticle suspension.

After 28 days of setting the cylinders were polished in order to obtain the absolutely parallel surface. The parallel of the side, with reference to the end faces of the specimens was verified according to the LST EN 12390-1:2003/AC:2005 standard. The compressive strength of test specimens was verified according to the LST EN 12390-7:2009 standard.

The air porosity indicators were verified according to the GOST 12730.4-78 standard. Relative water absorption of test specimens was calculated and pore size indicators were determined according to the same standard. λ is the average pore size indicator, α is pore uniformity indicator and computable closed and open porosity of concrete. When all the values are known the concrete's freeze-thaw resistance factor is calculated from Eq. (1).

$$K_s = \frac{P_u}{0.09P_a} \quad (1)$$

where K_s is freeze-thaw resistance factor, P_u is air porosity of concrete (air content in concrete), %, P_a is open (capillary) porosity of concrete, %.

The freeze-thaw resistance of concrete specimens was determined according to the LST L 1428.17 standard. 5% NaCl solution was used in the experimental research. The test specimens were examined by ultrasonic method according to the LST EN 12504-4 standard. Knowing that there is a functional relationship between ultrasonic pulse velocity and compressive strength, the ultrasonic pulse velocity in concrete specimens was measured after a certain number of freeze-thaw cycles [17-19]. The ultrasonic pulse velocity was measured by putting two transducers on the opposite faces of the specimen (direct transmission) and calculated from Eq. (2).

$$V = \frac{L}{T} \quad (2)$$

where V is ultrasonic pulse velocity, m/s; L is distance between sensors (specimen length), m; T is time of impulse travel, s.

The change of dynamic modulus of elasticity in test specimens was determined according to the LST EN 14146 standard. The dynamic modulus of elasticity was measured using *ERUDITE MKIV* instrument by triggering mechanical oscillation of longitudinal waves in test specimens (40×40×160 mm concrete prisms). Longitudinal wave oscillation frequency ranged from 500 to 10000 Hz. Basing on the type of longitudinal oscillations the dynamic modulus of elasticity was calculated from Eq. (3).

$$E_{din}^{is} = 4 \times 10^{-6} f_0^2 \rho l^2 \quad (3)$$

where f_0 is fundamental longitudinal frequency of oscillation, Hz; ρ is specimen density, kg/m³; l is specimen length, m.

The optical fluorescence microscopy method was used to detect the surface cracks in the test specimens. This method enables to visually detect 0.1-500 μ m defects in the test specimen. Optical microscope *OLYMPUS BX51TF* was used to identify the cracks.

4. Test results

Porosity indicators and freeze-thaw resistance of the concrete was calculated according to the methodology described in the previous section. Research results are presented in Table 3.

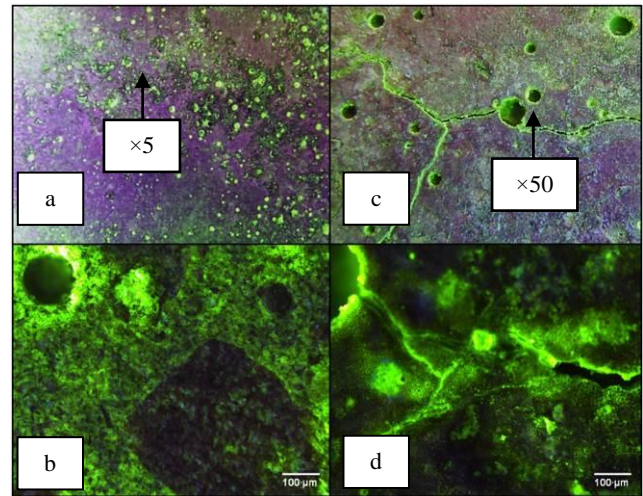


Fig. 3 Surface of test specimens a) no cracks and b) ×50 magnification; c) present cracks and d) ×50 magnification

The goal of the experiment was to determine which of nondestructive test methods provides the most reliable information about the deterioration of concrete due to recurrent freeze-thaw cycles. The results of ultrasonic impulse velocity, dynamic modulus of elasticity and compressive strength tests are presented in Table 4.

After 200 freeze-thaw cycles in 5% NaCl solution both the ultrasonic impulse velocity and the dynamic modulus of elasticity decreased, although there were no visible structural changes in all test specimens. The optical fluorescence microscopy method was used for visual examination and defect detection by applying the fluorescent dye

PFINDER 902 to the surface of the test specimens. Fig. 3 shows the surface of concrete test specimens exposed to ultraviolet radiation. We can see that test specimens of composition 3, which demonstrated the best freeze-thaw resistance, do not exhibit any cracks when exposed to UV radiation (a), whereas $\times 50$ magnification reveals only capillars and caverns of open pores. Test specimens of composition 4, which demonstrated the poorest freeze-thaw resistance, exhibit pronounced cracks seen both with naked eye and by $\times 50$ magnification. This method, does not provide information about the internal changes in the structure of concrete and is only an ancillary method.

Taking into account, that changes in the microstructure of a material affect such properties as the modulus of elasticity and compressive strength, the change in ultrasonic impulse velocity and the dynamic modulus of elasticity were measured in test specimens subjected to freeze-thaw cycles. It was observed that saturation, granular composition of the aggregates, temperature and density had a significant effect on the ultrasonic pulse velocity. In order to eliminate the influence of saturation the specimens were left to dry in the laboratory oven at of $100 \pm 5^\circ\text{C}$ for 48 hours.

Table 3

Physical properties of concrete

Concrete properties	Concrete composition			
	No. 1	No. 2	No. 3	No. 4
Density, kg/m^3	2416	2384	2400	2359
Uniformity of pores, α	0.25	0.40	0.42	0.35
Average pore size, λ	0.30	0.20	0.37	0.46
Capillary porosity, %	2.34	2.68	3.51	5.46
Air porosity, %	10.49	12.37	12.03	13.10
Overall porosity, %	12.83	15.05	15.54	18.57
Freeze-thaw resistance factor, K_f	50	51	38	27
Relative water absorption, %	1.07	1.26	1.63	2.65

Table 4

Physical-mechanical properties of concretes

Compo-sition	Ultrasonic pulse velocity,		Dynamic modulus of		Compressive strength, MPa	
	0 cycles	200 cycles	0 cycles	200 cycles	0 cycles	200 cycles
No.1	5016	4911	29.530	27.153	144	131
No.2	4841	4773	29.339	28.110	136	127
No.3	4869	4801	31.339	31.073	135	131
No.4	4375	4310	30.308	28.647	85	76

The ultrasonic pulse velocity was measured in concrete prisms ($40 \times 40 \times 160$ mm) and cylinders ($d = 50$ mm, $h = 50$ mm). The results of the measured time of travel are presented in Fig. 4. The observations revealed that the ultrasonic pulse velocity decreased after 200 freeze-thaw cycles in 5% NaCl solution. The ultrasonic pulse velocity decreased gradually in test specimens of composition No.1, whereas in test specimens of other compositions the ultrasonic pulse velocity decreased randomly. Knowing of the functional relation between the

ultrasonic pulse velocity and compressive strength it could be claimed that the decrease in the ultrasonic pulse velocity is caused by the reduced compressive strength.

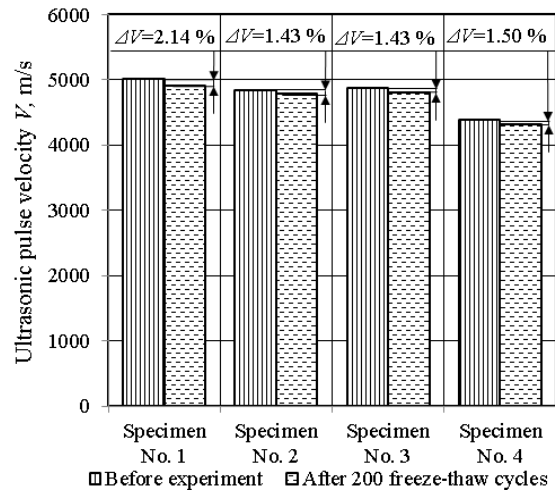


Fig. 4 The function of freeze-thaw cycles and ultrasonic pulse velocity

The dynamic modulus of elasticity was measured in concrete prisms ($40 \times 40 \times 160$ mm). The test results are presented in Fig. 5. Every material has its own natural frequency of oscillation, which is steady and does not depend on external factors as long as the structure of the material does not change. The observations have revealed that the dynamic modulus of elasticity changed randomly in different freeze-thaw stages, although the final dynamic modulus of elasticity slightly decreased in all test specimens. It was noticed, that the influence of dynamic modulus of elasticity depends of residual saturation in tests specimens and change in microstructure on-going hydration in the test specimens.

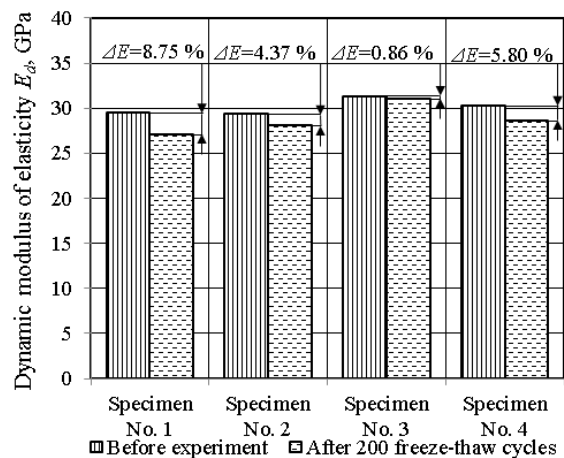


Fig. 5 The function of freeze-thaw cycles and dynamic modulus of elasticity

The comparison of the changes in ultrasonic pulse velocity and dynamic modulus of elasticity after 200 freeze-thaw cycles in 5% NaCl solution has revealed that the ultrasonic pulse velocity changed from 1.43% (composition No.2) to 2.14% (composition No.1) compared to the initial values, whereas the dynamic modulus of elasticity changed from 0.86 % (composition No.3) to 8.75% (composition No.1).

The comparison of ultrasonic pulse velocity (Fig. 4) with the dynamic modulus of elasticity (Fig. 5) at different freeze-thaw stages has revealed that the functional relationship between the dynamic modulus of elasticity and the compressive strength (correlation coefficient $R = 0.826$) is much stronger than the relationship between the ultrasonic pulse velocity and the compressive strength (correlation coefficient $R = 0.430$).

The test specimens were compressed after 200 freeze-thaw cycles. The compressive strength of all test specimens has decreased (Fig. 6). The same was demonstrated by the lower dynamic modulus of elasticity and ultrasonic pulse velocity. Test samples of composition No.4 demonstrated the poorest strength properties, the drop in compressive strength after 200 freeze-thaw cycles in 5% NaCl solution was 10.47%. Test specimens of composition No.1 demonstrated the best strength properties, although the change in strength after 200 freeze-thaw cycles in 5% NaCl solution was 9.02%. The least change in the compressive strength (2.78%) was observed in the test specimens of composition No.3.

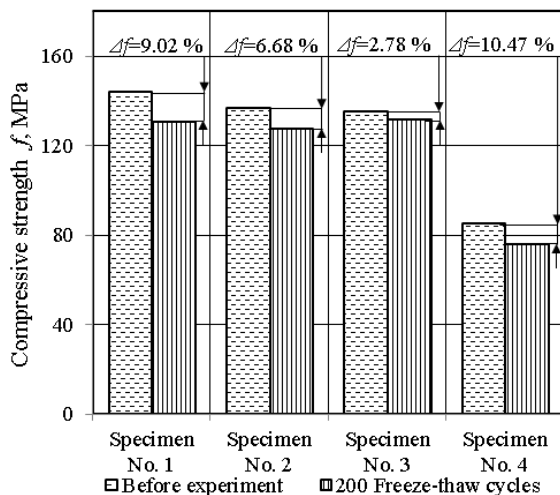


Fig. 6 The compressive strength of concrete after 200 freeze-thaw cycles

5. Conclusions

1. 200 freeze-thaw cycles in 5% NaCl solution that correspond to approximately 800 freeze-thaw cycles in natural water have revealed little visible change, however the microdefects in concrete's structure have reduced the compressive strength of the test specimens from 2.78% (composition No.3) to 10.47% (composition No.4), the ultrasonic pulse velocity changed from 1.43% (composition No.2) to 2.14% (composition No.1) and the dynamic modulus of elasticity changed from 0.86% (composition No.3) to 8.75% (composition No.1).

2. The interim control of freeze-thaw resistance was done by the calculation of the dynamic modulus of elasticity. The researchers have observed that these methods produce sufficiently precise evaluation of the decrease in the compressive strength of test specimens, however the dynamic modulus of elasticity has a stronger functional relationship with the compressive strength than ultrasonic pulse velocity, therefore we may state that in our case the calculation of the dynamic modulus of elasticity was more reliable method for measuring the freeze-thaw

resistance.

3. The change in the test specimen properties is examined over the entire volume of the specimens both by using the ultrasonic pulse velocity and the dynamic modulus of elasticity methods. Such examination provides more information about the structural changes in deeper layers of the tested material. Although these methods produce sufficiently precise evaluation of the decrease in the compressive strength, the ultrasonic method is more convenient for testing the constructions in-situ, whereas the calculation of the dynamic modulus of elasticity could be used in laboratory testing.

4. The experimental forecasting of freeze-thaw resistance produced very high values of freeze-thaw resistance factor (K_f). Presumably the UHPC should have very high freeze-thaw resistance. However, the majority of test specimens lost 5% of their initial strength after 200 freeze-thaw cycles in 5% NaCl solution. Therefore it could be stated that according to GOST 12730.4-78 standard freeze-thaw prediction of UHPC is incorrect and inaccurate.

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V. Vaitkevičius, E. Šerelis, Ž. Rudžionis

NEARDANČIŪJŲ TYRIMŲ METODŲ TAIKYMAS VERTINANT YPAČ STIPRIŲ BETONŲ ATSPARUMĄ ŠALČIUI

R e z i ū m ė

Pastaruoju metu vis didesnis dėmesys skiriamas tokioms medžiagoms, kurios pasižymėtų puikiomis stiprumo ir ilgaamžiškumo savybėmis. Viena tokių medžiagų, kuri tenkina šias savybes – tai ypač stiprus betonas (YSB). Nors YSB pasižymi puikiomis mechaninėmis bei ilgaamžiškumo savybėmis ir tiktų naudoti įvairios paskirties statybinėms konstrukcijoms, tačiau kaip ir visos medžiagos, po kurio laiko veikiant įvairiems aplinkos veiksniams praranda savo pradines projektines savybes. Šiame straipsnyje, iširta viena iš svarbiausių betono ilgaamžiškumo savybių, kuri šiuo metu aktualiausia Lietuvoje eksploatuojamoms

statybinėms konstrukcijoms ir medžiagoms, tai atsparumas šalčio poveikiui. Vienas iš būdų, kuriais galima tirti YSB savybių kitimą, tai neardomųjų metodų taikymas. Kiekvienas metodas turi savo trūkumų ir privalumų, todėl esant įvairiems aplinkos veiksniams pasižymės skirtingais tikslumais ir turės skirtingą patikimumą. Ilgaamžiškumo savybės tirti panaudotas ultragarsinis ir dinaminio tamprumo modulio metodas. Šių metodų patikimumas patikrintas nustatant betono stiprį gniuždant. Paviršiniams defektams bandiniuose užfiksuoti panaudotas fluorascensinis metodas. Tyrimai prognozuojant ir nustatant ypač stipraus betono atsparumą šalčiui pasaulinėje praktikoje yra mažai publikuoti, o Lietuvoje neatliekami.

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NONDESTRUCTIVE TESTING OF ULTRA-HIGH PERFORMANCE CONCRETE TO EVALUATE FREEZE-THAW RESISTANCE

S u m m a r y

Recently increased attention has been given to materials, which could meet certain strength and durability requirements. Ultra high performance concrete (UHPC) is one of the materials which possess such properties. UHPC has excellent durability and mechanical properties and could be suitable for various construction applications, however all construction over time in aggressive environment loses its original designed properties. In this article researched one of the most important durability parameter – freeze-thaw resistance of concrete. This durability parameter is significant for constructions and materials used in Lithuania. Nondestructive test methods are one of the ways, which are suitable to investigate durability properties of UHPC. Each of the methods has its advantages or disadvantages and depending on environment will presents different reliability results. Durability properties of UHPC were investigated by ultrasonic and dynamic modulus of elasticity methods. The reliability of these test methods was verified by determining the compressive strength, and surface defects were visually inspected by fluorescence method. There are a few publications about freeze-thaw resistance of UHPC and fewer articles about concretes prediction to severe frost damage, however such research in Lithuania is not done at all.

Keywords: Ultra-high performance concrete, UHPC, freeze-thaw resistance of UHPC, Nondestructive testing, ultrasonic pulse velocity, dynamic modulus of elasticity.

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