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Durability Characterisation of Glass Fibre Reinforced Concrete by Resistance to Freezing and Thawing

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Precast concrete panels are becoming widely used in residential buildings. In this case concrete is acting both as decorative and protective envelope of the whole building. Due to structural and technological reasons, outer skin layer is quite thick- usually 80mm, which can be reduced to 15mm by using innovative and economical composites, such as glass fibre reinforced concrete (GRC). As there is lack of knowledge in scientific literature about this composites behaviour in freeze- thaw conditions, main aim of this article is to investigate freeze- thaw durability of GRC. Silica fume and metakaolin were used as additives, dosing 2,5%, 5% and 7,5% from mass of cement and proved to enhance long term ductility of glass fibre reinforced composite after freeze- thaw tests. Also, flexural stain energy is used as a new concept in concrete freeze- thaw durability to investigate fibre reinforced composites ductile/ brittle behaviour. Resistance to freeze- thaw of glass fibre reinforced concrete (GRC) modified with silica fume and metakaolin was tested according to freeze/ thaw test method with de-icing salt after 112 cycles. Surface mass loss was obtained, flexural strain energies calculated from load- deformation curves and ductile/ brittle behavior of GRC composite explained. SEM micrograph analysis was used to identify possible flaws on the surface of filaments and fiber-matrix contact zone due to freeze- thaw influence.

Keywords: glass fiber reinforced concrete, ductility, static fatigue, freeze- thaw.

Introduction

As concrete in the 21st century took second place as the most consumed material after drinking water, its impact on main ecological issues such as CO_2 emissions, deforestation, and depletion of raw material resources became ever more apparent. While Intergovernmental Panel on Climate Change (IPCC) predicts an unprecedented chain of social disasters due to global warming by the 2050's, current design codes require only 50 years of service life for residential concrete structures (IPCC report, 2018; EN 1992-1, 2004). Yet first major problems usually arise much earlier. According to The American Society of Civil Engineers (ASCE) calculations, the average maintenance-free life for a concrete structure built today is approximately only 18,5 years (Abanilla et al., 2006). On-going demand for new concrete calls for mining of non- renewable materials such as limestone,

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clay, sand and various coarse aggregates. Finally, traditional reinforcing steel requires relatively thick concrete protective cover to sustain a defensive shield from electrochemical reactions that cause rebar corrosion (Biswas et al., 2020; Echevarria, 2018).

The overall life span of the building can be increased, and raw materials saved by optimizing structural behavior and the durability of concrete members (Kulas 2015; Moya et al., 2014; Gregory, 2011). For example, conventional precast concrete wall elements usually have three main components: an inner structural layer of 140 mm, an insulation of 200 mm and 80 mm of the outer decorative layer. As the outer layer of concrete has no structural functions, it can be optimized by using self-compacting fiber-reinforced composites, such as glass fibre reinforced concrete (GRC). If the thickness of the outer skin layer for precast elements is reduced down to 15-20 mm, a significant amount of materials can be saved and self- weight of outer walls reduced by 30% (White et al., 2011; Lameiras, 2013). Pigments can be used more efficiently in GRC than in normal concrete due to less concrete volume because only the outer surface of the concrete is actually visible.

GRC has been used over half a decade as building material in various applications, mostly for decorative concrete panels (Rickard, 2015; Teraura, 2015). Refined by 17% of ZrO_2, alkali resistant (AR) glass fibres show no significant symptoms of chemical corrosion in aggressive alkaline environments such as concrete during long periods of time (Holubova et al., 2017; Helebrant et al., 2017; Mills, 1981). Self-compacting concrete can be produced by adding 3% of AR glass fibres from the total weight of concrete matrix with flexural strength between 10-16 MPa, which is 5-8 times stronger in bending then regular concrete without reinforcement. But strength is often not the best definition for mechanical properties of materials. A measure of material's ability to undergo significant plastic deformations before rupture is called ductility and is far more definitive than strength. Due to large post- crack deformations, GFRC is considered as a ductile material, while plain concrete is brittle in case of tension and bending (Gopalaratnam 1995; Ali et al., 1975; Correia et al., 2006; Enfedaque et al., 2011).

The carbonization reaction in Portland cement is often leading to efflorescence and blushing of decorative concrete. For this reason, silica fume and metakaolin are often used as color enhancing micro fillers due to their pozzolanic reaction with $Ca(OH)_2$ (portlandite). $Ca(OH)_2$ also plays a negative role in GRC durability because growing sharp portlandite crystals cause flaws on the surface of the glass filaments and weakens them (Orlowsky et al., 2005; Purnell et al., 2018). Young Portland cement matrix can have pH up to 12,6 where pores are filled with a high concentration of hydroxyl (OH-) ions which are breaking the Si-O-Si bonds in the surface of glass filaments. Carbonization on the other hand, is described as a positive but very slow process in GRC literature because $Ca(OH)_2$ is transformed to $CaCO_3$ which is filling the pores and densifying concrete matrix (Arabi et al., 2018; Majumdar et al., 1991). Micro fillers are reducing amounts of portlandite phase in the concrete in such way enhancing the mechanical durability of GRC composite (Bartos et al., 1996; Brandt et al., 2003; Peled et al., 2005; Marikunte et al., 1997).

In most of the scientific literature considering the durability of GRC, wet-dry cycling is discussed as mostly unfavorable weathering conditions for this composite. Standard test methods are mostly based on Litherland which offers to keep GFRC specimens in warm 50°C water for 90 days which represents 20 years of natural weathering in UK climate (Litherland 1985). Many scientists doubt the adequacy of this test method when reactive micro fillers such as silica fume and metakaolin are used due to complex pozzolanic reactions in elevated temperatures, that usually are not common in real climate. Purnell tested specimens with a modified matrix and found that they were acting very unusual, for the first 28 days strength was growing and after that started dropping rapidly (Purnell et al. 1999; Purnell et al., 2005; Purnell, 2001).

Freeze-thaw durability of GFRC is poorly investigated, even though it is one of the major climatic forces acting on concrete facades in many European countries. Enfedaque A. tested freeze-thaw resistance of GRC according to scaling at freezing/ thawing test method where one cycle is freezing specimens at -20°C in for 6 hours and thawing in +20°C warm water for 6 hours. After 50 cycles in the climatic chamber, GRC with OPC formulation lost about 40 % of initial flexural and 80% of strain capacity, becoming a more brittle material with less bending strength (Enfedaque et al., 2012).

Up to this day, the main criteria for assessing GRC durability was the modulus of rupture (M.O.R) after an accelerated ageing test which is simply the maximum flexural strength in MPa of the specimen being tested. However, not only the flexural strength but also the behavior during fracture is important, particularly the flexural toughness, because cracking is mainly caused by the lack of fracture toughness. This property of composite such as GRC can be expressed numerically by calculating the fracture energy G_f, which represents the work done by external forces for propagation of the crack until final fracture occurs (Hillerborg et al. 1975; Hillerborg, 1985).

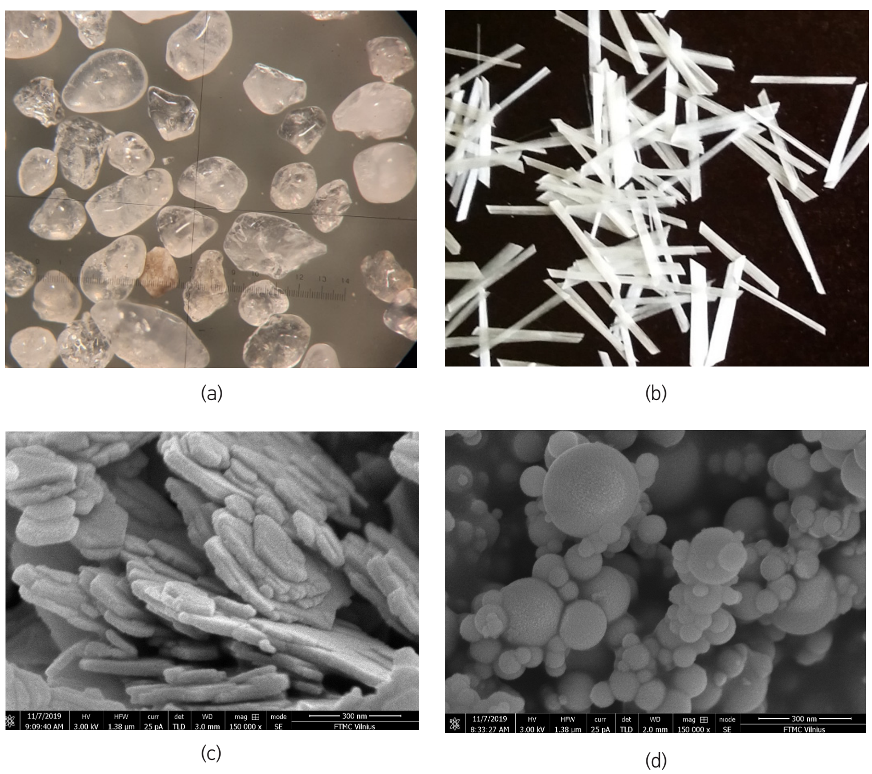
In this research paper, freeze- thaw durability test results are described for GRC modified with metakaolin and silica fume.

Materials and Test Methods

Materials

Materials used for the production of reference GRC concrete samples include type CEM I 52,5R ordinary Portland cement (OPC), quartz sand, polycarboxylic ether- based superplasticizer, alkali resistant (AR) glass fibres and water. Metakaolin and silica fume were used for typical GRC matrix modification in different quantities (2,5%, 5% and 7,5% from the mass of OPC).

Rapid hardening cement class CEM I 52,5R was chosen due to the demand of the pre-cast industry to have good early strength with bulk density 1200 kg/m^3 , specific gravity 3103 kg/m^3, specific surface area 4200 cm^2/g (Blaine method), compressive strength 41,1MPa after 2 days and 69,0MPa after 28 days, water demand 30,2% and initial setting time- 90 minutes. Fine quartz



(a) (b) (c) (d)

Fig. 1

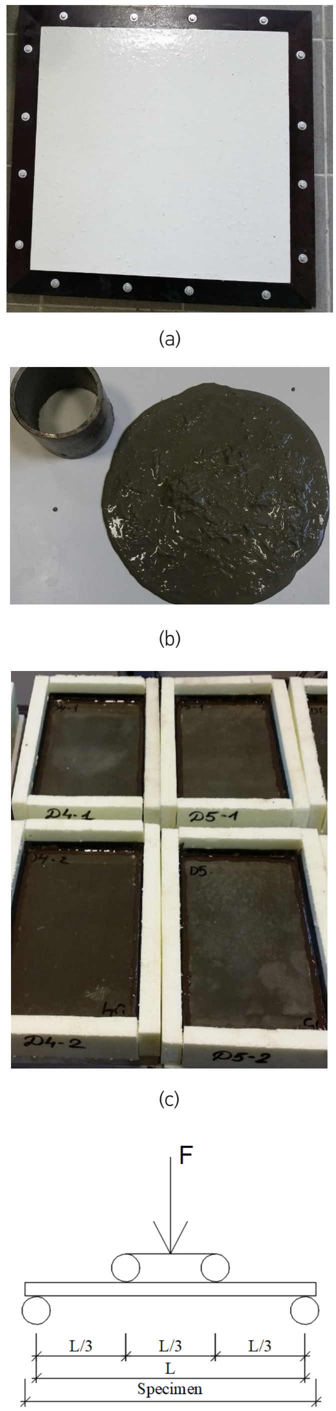
Materials used: a) quartz sand particles (fraction ~500μm); b) glass fibres in the concrete matrix; c) metakaolin (magnitude 150000x); d) silica fume (magnitude 150000x) sand with D_{max}=1,25 mm (Fig. 1a) was used because of good workability due to smooth- shaped particles and no susceptibility to alkali-silica reactions, which could influence freeze- thaw test results. Quartz sand contained >98,5% SiO_2 and <0,6% Al_2O_3, bulk density was 1640 kg/m^3, specific gravity 2650 kg/m^3 and water absorption- less then 0,5%. Silica fume was in the form of powder, with bulk density 600 kg/m^3, specific gravity 2200 kg/m^3, specific surface area 300000 cm^2/g (according BET method). Metakaolin was also in the form of powder, with bulk density 480 kg/m^3, specific gravity 2500 kg/m^3, specific surface area 150000 cm^2/g (BET method).

12 mm length AR glass fibers were used with a high percentage of ZrO_2- 17% (Fig. 1b), which makes them alkali- resistant to aggressive cementitious matrix. Tensile strength of glass filaments were 1400 MPa, deformation modulus 74 GPa. Melting point 1100°C, thermal resistance up to 350°C. One fibre contains 200 individual filaments with diameter 18 μm each.

Test methods

Seven different concrete formulations were tested after 0 and 120 freeze- thaw cycles according to EN 13198 Method B with de-icing salt. Flexural strength was tested before and after freeze- thaw cycles, flexural strain energies calculated from load- deformation curves and ductile/ brittle behavior of GRC composite explained. SEM micrographs were analyzed and glass filament surfaces checked for possible flaws from frost damage.

Batches of self-compacting GRC premix were mixed with a high shear mixer, with speed up to 800 RPM. In the beginning, water is added together with superplasticizer, after that sand poured to the mixing bucket. After slow mixing for 20- 30 seconds, cement is added and the whole matrix mixed for 2 min at maximum 800 RPM. Finally, fibres are added and dispersed into the matrix with 400 RPM for the 60s. Fresh concrete is poured into to smooth plywood molds and concrete boards with size 525x525mm and 15mm thickness were made (Fig. 2a). Concrete slump is tested with cylinder according to EN 1170-1 (Fig. 2b) and entrained air quantity is measured with 8 liter air entrainment meter “TESTING” in accordance with the air pressure compensation principle. Hardened boards are placed into a curing chamber for temperature 20°C and 60% relative humidity for 28 days. Cured boards are cut into 4 plates 260x150 mm. Two of them are prepared for freezing and scaling tests according to EN13198 (Fig. 2c). The last two are cut into six 50 mm wide coupons and after 28 days of curing tested for initial flexural capacity before freeze- thaw by 4- point bending scheme according to EN 1170-4, with loading control 10 N/s (Fig. 2d).



(a) (b) (c) (d)

Fig. 2

Test specimens and test methods: a) boards 525x525x 15 mm; b) slump test; c) preparation for freezing and scaling tests; d) 4-point bending scheme

Freeze- thaw tests are done in a 3% NaCl solution, which covers the whole testing surface- 39300 mm^2 per specimen. The standard EN 13198 requires 28 freeze- thaw cycles with de-icing salt and total test area greater than 7500 mm2 for GRC. Specimens are placed into a climatic chamber for 56 and 112 cycles, where one cycle lasts 24 hours with temperature changing from -20^{0}C to +20^{0}C. 112 cycles with NaCl can be interpreted as 2 times higher than XF4 exposure according to EN 206-1. After 56 and 112 cycles, plates are cut into three coupons and flexural capacity is tested, deflection and loading data collected in spreadsheet format.

For the 4-point bending scheme formula (1) is used to calculate flexural strength values and to draw stress-strain curves, when distance between load points is L/3:

$$\sigma=\frac{F\cdot L}{b\cdot h^2} \tag{1}$$

Where, σ – flexural stress (MPa), F – force (N), L – distance between supports (mm), b - width of specimen (mm), h – thickness of specimen (mm).

Fracture energy calculation is based on the fracture work principle, which is in general expressed as a product of force and distance. In this case, it is assumed that the work done by the external forces is fully used for the propagation of the crack. External work is transformed into fracture energy G_f and calculated according to formula (2), recommended by RILEM. As the mass of the specimen is significantly less than acting forces, work done by gravitation (self- weight of speci-men) can be neglected.

$$G_f=\frac{W_0}{A_{lig}} \tag{2}$$

Where, G_f – fracture energy of GRC (N/m), W_0- work done by the testing machine, which is the area under the load-deflection curve (Nm), A_{lig}- fracture area of specimen (m^2).

SEM micrograph images were obtained from specimen fracture zones with Helios Nanolab 650 microscope. For good electrical conductivity, the analysed surface was cowered with a 20-30 nm thickness carbon layer. Clearest images were obtained at 3kV voltage and 0,8nA current. For chemical composition analysis EDS detector Oxford Instruments, Xmax 20 mm^2 and INCA 4.15 software were used and results were obtained at 20kV voltage and 1,6nA current.

Concrete compositions

Eight different compositions were made according guidelines of EN 15191 and mix proportions are given in Table 1. The most common matrix composition was used as a reference (REF1), which consists of equal parts of cement and quartz sand 1:1, water/ cement ration 0,36 and 1%

Table 1
Relative compositions

Materials	REF1	S2,5	S5	S7,5	M2,5	M5	M7,5
CEM I 52,5R	1	1	1	1	1	1	1
Superplasticizer	1,1%	1,1%	1,1%	1,1%	1,1%	1,1%	1,1%
W/C	0,36	0,36	0,36	0,36	0,36	0,36	0,36
Quartz 0/1.25	1	1	1	1	1	1	1
Silica fume	*	2,5%	5%	7,5%	*	*	*
Metakaolin	*	*	*	*	2,5%	5%	7,5%
Glass fibre	2,9%	2,9%	2,9%	2,9%	2,9%	2,9%	2,9%

of superplasticizer. Fiber quantity was the same through all batches- 3% from the weight of all dry materials. The other six compositions were based on REF1, only adding different quantities of reactive micro fillers- silica fume and metakaolin. Quantities of these additives were 2,5%, 5% and 7,5% from the mass of cement or 20, 40 and 60 kg/m^3.

Results and discussion

Fresh concrete properties

The addition of micro fillers influenced the workability and porosity of fresh concrete. The highest 19 cm slump according EN1170-1 slump test method was achieved with reference composition (REF1) without micro fillers. Increasing quantities of additives to 7,5% decreased slump to 13 cm and silica fume showed higher impact (Fig. 3a). Directly after workability testing, entrained air quantities were measured. Metakaolin showed minimum influence, while silica fume gradually increased entrained air quantity from 2,3% (REF1) to 4,8%(S7,5) (Fig. 3b).

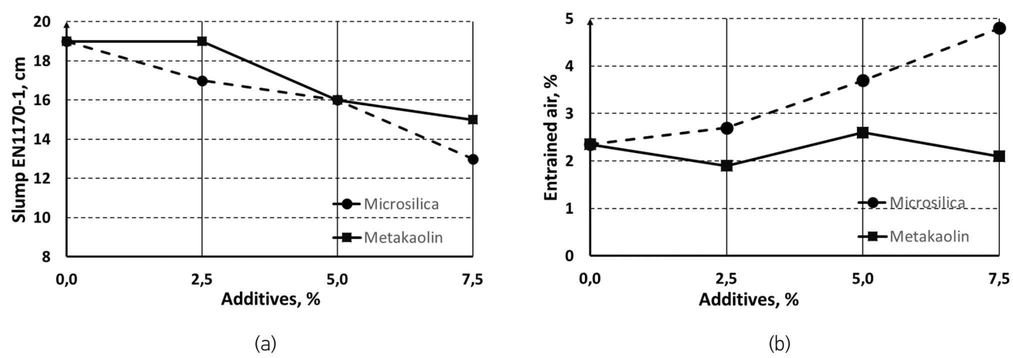


Fig. 3

Fresh concrete test results: a) workability according EN1170-1; b) entrained air quantities

Scaling at freezing/ thawing

As GRC composites are mostly used for thin decorative concrete products, their long-term visual characteristics should be one of their major properties. And in the cold climate zones, one of the main factors of concrete deterioration is the frost damage.

Freeze-thaw tests were done for all compositions and took 112 days according to the test method given in EN13198 Annex B, where one cycle is equal to 24 hours. After every 7 cycles scales from the concrete surface were collected and weighted, specimens cleaned, and surface cowered with 5mm of 3% NaCl solution. Total mass loss is represented in Fig. 4a and 4b after 56 and 112 cycles.

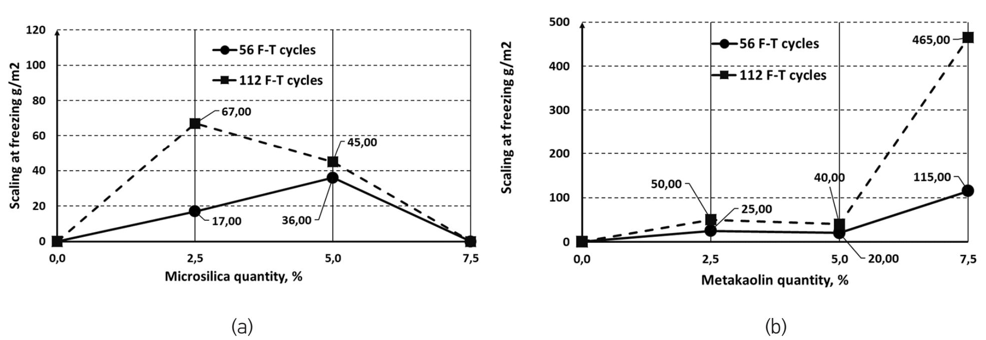


Fig. 4

Scalling at freezing/ thawing, g/m^2 according to EN13198 in 3%NaCl solution with different quantities of reactive micro fillers: a) silica fume; b) metakaolin

When the direct freeze- thaw test with de-icing salt is used, the maximum mass loss according to EN13198 shall be 1500g/m^2 after 28 freeze- thaw cycles. None of the compositions tested exceeded 500 g/m^2 even after 112 freeze- thaw cycles, showing excellent durability of GRC composite in freeze- thaw conditions (Fig. 5a). Adding silica fume between 2,5%- 5% had a negative effect, resulting in surface mass loss up to 67 g/m^2 (Fig. 5b). On the other hand, a higher amount- 7,5% showed no visible surface flaws, which might be explained by air entraining effect (4,8%) in the fresh concrete mix which increases resistance against freeze- thaw cycles. Metakaolin showed opposite results than silica fume. It can be safely used in quantities 2,5 and 5% with a maximum 50 g/m^2 mass loss after 112 cycles, while 7,5% metakaolin addition had a significant impact on the concrete surface deterioration- 465 g/m^2 (Fig. 5c).

(a) (b) (c)

Fig. 5

Photos of specimens after 112 freeze-thaw cycles: a) REF1, with no additives; b) S5 with 5% silica fume; c) M7,5 with 7,5% metakaolin

Flexural strength

In GRC technical literature flexural strength if often expressed as modulus of rupture (M.O.R.), which represents maximum stress point which is reached during a 4-point bending test just before the first crack appears. After the crack, the specimen does not fully break, but deformation goes on while stress (acting force) is decreasing.

Measure of material's ability to undergo high plastic deformations before rupture is called ductility. A lot of scientific research show that GRC is initially ductile material with a tendency to become brittle after long term weathering, which depends on many factors, such as concrete composition and weathering conditions.

This research showed that severe freeze- thaw conditions did not decrease the initial GRC flexural strength, which even increased from 10 MPa to 12 MPa (Fig. 6). The addition of silica fume and metakaolin had no significant impact on initial flexural capacity.

Positive effects of investigated reactive micro fillers appeared after stress- deformation curves were analyzed- metakaolin and silica fume increased ductility of GRC. This applied both for initial

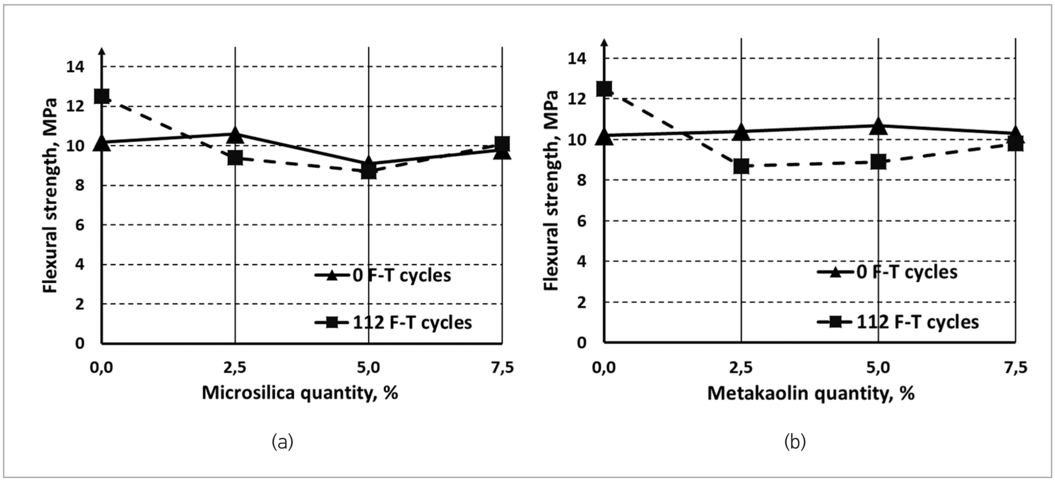


Fig. 6

Flexural strength (M.O.R.) before freeze- thaw and after 112 cycles: a) with silica fume; b) with metakaolin

and after freeze- thaw states and can be characterized as maximum deformation mm at 3 MPa residual stress (Fig. 7). Optimal quantities were 5% and 7,5% for silica fume with 4 mm deformation and 7,5% for metakaolin with 4.9 mm deformation after 112 freeze- thaw cycles.

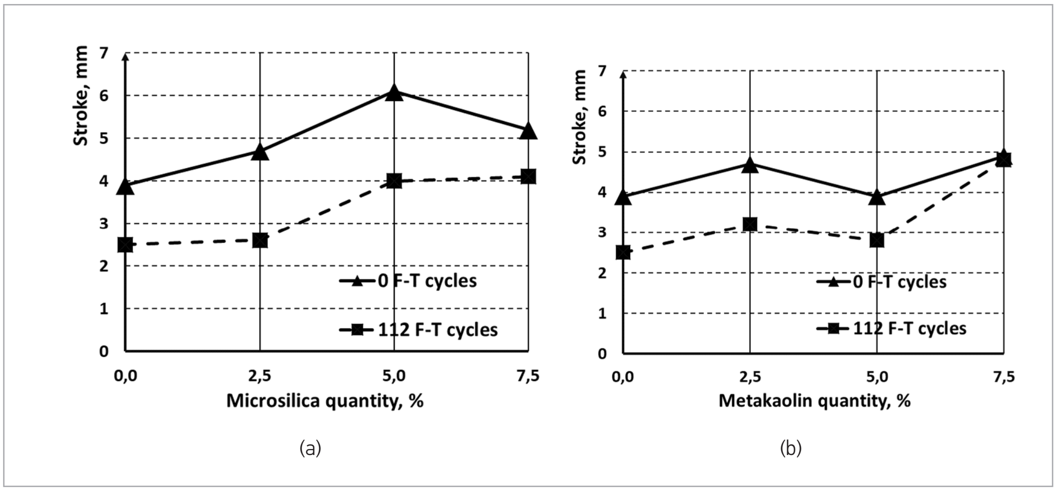


Fig. 7

Deflection (deformation) before freeze- thaw and after 112 cycles: a) with silica fume; b) with metakaolin

Flexural strain energy

For ductile materials such as GRC, flexural strain energy can describe mechanical behavior much more accurately than strength and deflection. Having spreadsheet data of stress- deformation curves (Fig. 8) it is not hard to calculate strain energies for each tested specimen according to formula 2. Work done by the testing machine is divided by the fracture area where work is equal to the area under the curve.

Flexural strain energy results are given in Fig. 9 for all compositions before freeze- thaw and after 112 cycles. REF1 composition has the lowest initial strain energy 1183 N/m, which after 112 freeze- thaw cycles dropped to 620 N/m, which is 47%. Addition of reactive fillers increased post free freeze- thaw flexural strain energies up to 1149 N/m for silica fume and 1291 N/m for metakaolin, which is close to initial value 1183 N/m before weathering tests and without micro fillers.

Fig. 8

Stress- deformation curves after 112 cycles: a) REF1; b) S5; c) M7,5

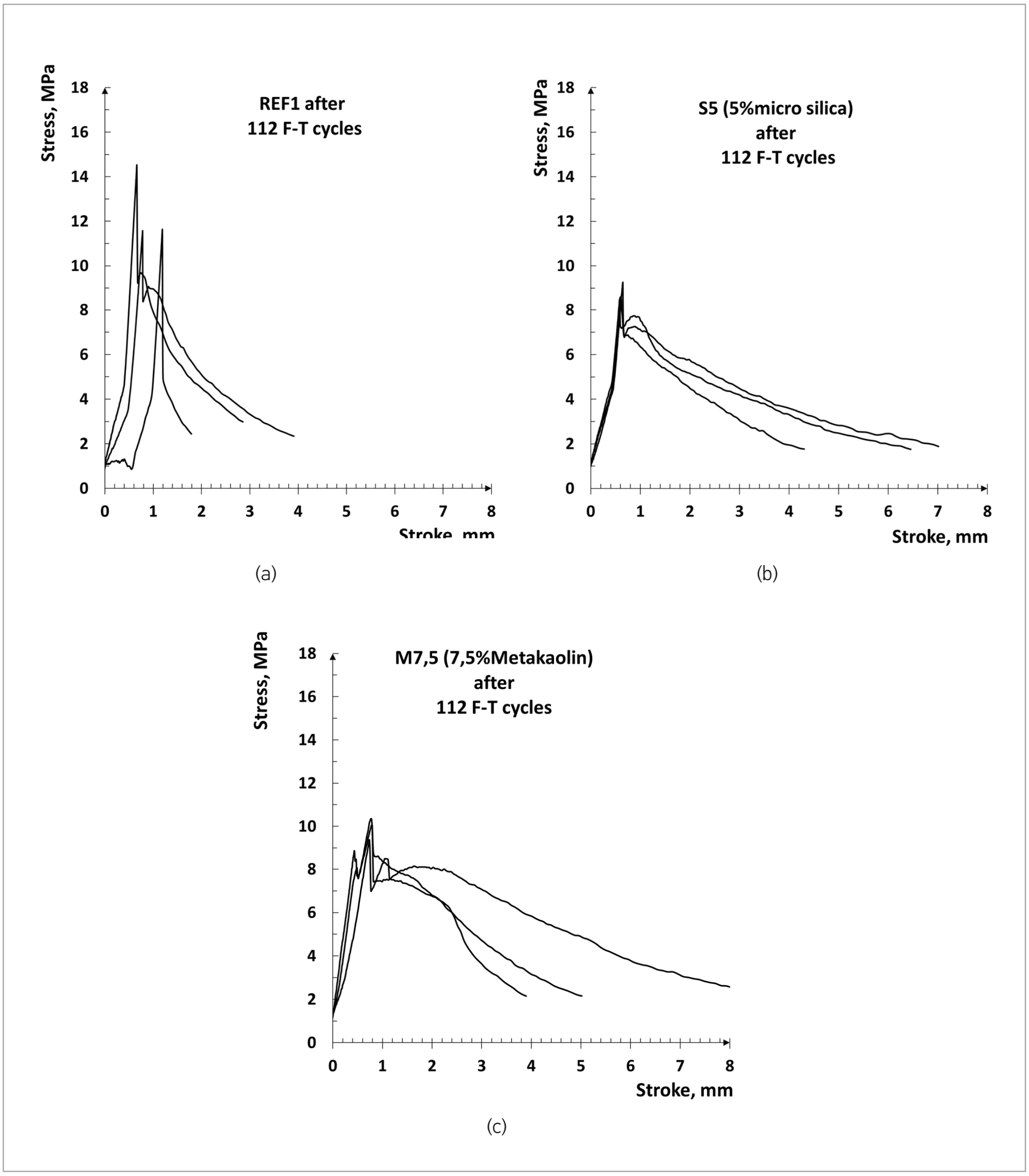
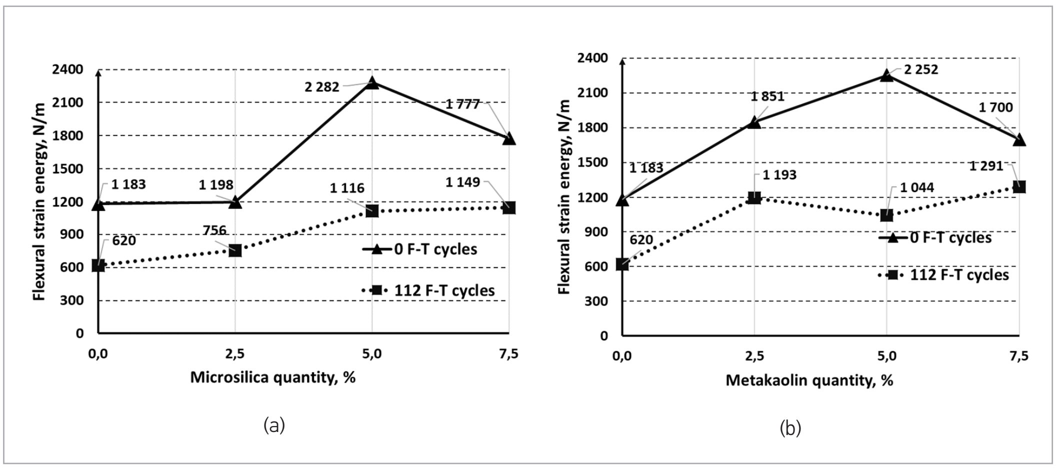


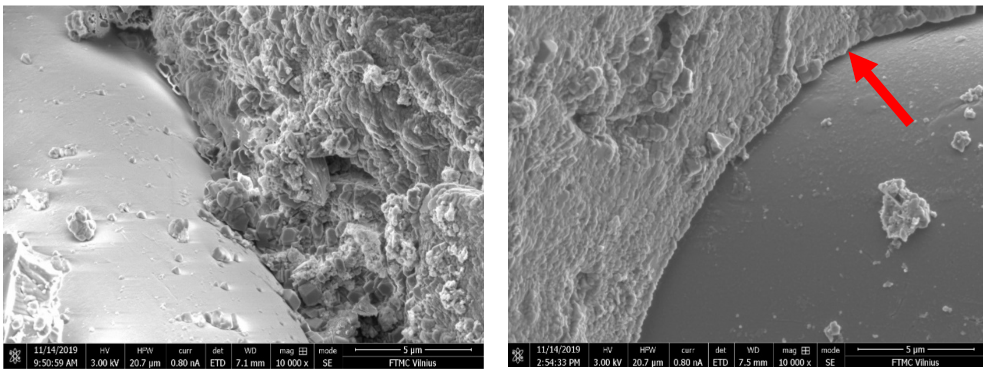
Fig. 9

Flexural strain energies before and after 112 freeze-thaw cycles: a) silica fume; b) metakaolin



SEM investigation

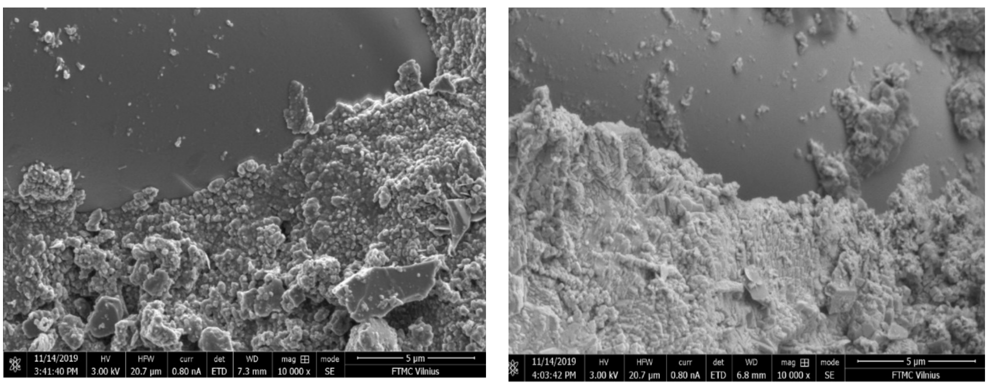
SEM micrographs of specimen fracture zones explained the influence of micro fillers on the durability of GRC in freeze- thaw conditions. Gaps between filament surface and the matrix, visible in Fig. 10 b showed defilamentation of filaments in the interfacial zone for specimens without micro fillers (REF1). On the other hand, compositions S7,5 and M7,5 with 7,5% silica fume and metakaolin showed better cohesion between matrix and glass surface (Fig. 11a and 11b). Surfaces of filaments that are in direct contact with the matrix had few hydration products in REF1 samples without micro fillers. Brittle portlandite phases are more common in REF1 than in compositions with 7,5% micro fillers. Reaction of $Ca(OH)_2$ with silica fume and metakaolin produced new C-S-H phases which increased the fibre- matrix bond and resulted in higher flexural strain energies. Even after 112 freeze-thaw cycles with de-icing salt, glass filaments had no significant flaws or cracks on their surface, but some minor surface roughness could be noticed for specimens without micro fillers



(a) (b)

Fig. 10

SEM micrographs of fibre- matrix interface (REF1) without micro fillers: a) before freeze-thaw; b) after 112 freeze- thaw cycles



(a) (b)

Fig. 11

SEM micrographs of glass filament surface after 112 freeze- thaw cycles: a) S7,5 with 7,5% silica fume; b) M7,5 with 7,5% metakaolin

Conclusions

Obtained results of this research could be used in optimization of conventional precast concrete wall panels with decorative concrete outer layer, reducing self- weight of the elements, quantity of raw materials (sand and cement) used for their production. GRC composites showed very good freeze- thaw resistance after 112 cycles in de-icing salt solution. No weight loss was found in test specimens without micro fillers and with 7,5% silica fume. 2,5% and 5% silica fume resulted in minor defects- up to 67 g/m^2. Metakaolin had a higher negative impact, especially when used in higher quantity (7,5%), having a maximum of 465g/m^2 of lost mass from the surface which might be associated with loss of workability and should be investigated further. Stress- deformation curves showed brittle behavior of GRC after freeze- thaw cycles without micro- fillers. Adding silica fume and metakaolin improved long term ductility of GRC in freeze- thaw conditions. This was explained by better fibre- matrix bond due to formation of new C-S-H phases.

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