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A method for the reduction of deformation of high-strength lightweight cement concrete

Aleksandr Sergeevich Inozemtcev
Researcher, Scientific and Educational Center Nanomaterials & Nanotechnology, Moscow State University of Civil Engineering, Moscow, Russian Federation

Evgenij Valerjevich Korolev
Director, Scientific and Educational Center Nanomaterials & Nanotechnology, Moscow State University of Civil Engineering, Moscow, Russian Federation

This paper presents research into the deformation properties of high-strength lightweight concrete (HSLC) containing hollow aluminosilicate microspheres. A method of increasing the fracture toughness of the HSLC by using a modifier as a coupling agent on the surface of the microparticles of aggregate is proposed. Use of the hollow microspheres is proposed to produce lightweight concrete with high performance characteristics: increasing the content of spherical microparticles in the concrete composition promotes the formation of a close-packed structure with low deformation. The coefficient of fracture toughness of the HSLC was found to be comparable to that of fine-grained high-strength normal concrete and is limited by the strength characteristics of the micrometric aggregate particles. The aim was to create an active iron–silica shell on the surface of the hollow spheres, which interacts with the major components and products of cement hydration and reinforces the phase boundary. The proposed method reduced longitudinal and transverse deformations of the HSLC by 1·20–1·60 and 1·37–2·21 times respectively. The elastic modulus of the HSLC was determined to be 6·0–8·5 GPa, with a Poisson's ratio of 0·08–0·14. The nanomodifier reduced the intensity of cracking under the influence of shrinkage stresses of HSLC by 56·9%.

Notation

E	elastic modulus
$k_{\mathrm{ft},1}$	coefficient of fracture toughness for concrete aged 28 d
$k_{\mathrm{ft},2}$	coefficient of fracture toughness for concrete after operational impact
R'	flexural or compressive strength of water-saturated sample at 6 d
R''	flexural or compressive strength of dried sample for relative humidity of 40–60% and temperature of 30–40°C
R_{com}	compressive strength (cube) of concrete aged 28 d
R_{fs}	flexural strength of concrete aged 28 d
R_{pr}	prism strength
R_{sp}	specific strength (relationship of R_{com} to $\rho/\rho_{\mathrm{water}}$)
v_{f}	volume fraction of hollow microspheres
ε_1	relative longitudinal deformation at 30% of breaking load
ε_2	relative transverse deformation at 30% of breaking load
μ	Poisson's ratio
ρ	average density
σ	load equal to $0{\cdot}3R_{\mathrm{pr}}$

Introduction

The task of combining the positive features of lightweight and heavy cement-based concrete is a recognised global trend (Costa *et al.*, 2012; Kılıç *et al.*, 2003; Korolev and Smirnov, 2013; Tanyıldızı, 2014; Wilson and Malhotra, 1988). Extensive experience (Daniel *et al.*, 2014; Kockal and Ozturan, 2011; Ming Kun *et al.*, 2014; Sajedi and Shafigh, 2012) in engineering construction and thermal insulation materials has been accumulated around the world. In Russia, the use of hollow glass and aluminosilicate microspheres has been suggested for creating lightweight concretes (Inozemtcev and Korolev, 2013; Oreshkin, 2008; Oreshkin *et al.*, 2014; Semenov *et al.*, 2014) and practical applications of lightweight concrete containing microspheres include the road plates used during reconstruction of the bridge over the River Volga (Ponomarev, 2009). Inozemtcev (2014) and Inozemtcev and Korolev (2015) showed that increasing the volume fraction of hollow microspheres promotes the formation of a close-packed structure in which the microsized filler particles are able to bridge cracks and thus increase the specific strength of the material. However, in practice, implementation of the combination of low average density and high strength in high-strength lightweight concrete (HSLC) requires that special attention be paid to the structure and deformation of the material under the influence of operating loads.

Materials and methods

Portland cement CEM I 42·5 R ('Mordovcement') was used for preparation of test concrete samples. The chemical composition of the cement is shown in Table 1. The mineral part of the concrete mixture produced included siliceous materials with different dispersions. The quartz sand used (fraction 0·160–0·630 mm) consisted of 20–30% fraction 0·160–0·315 mm and 70–80% fraction 0·315–0·630 mm. Stone powder was produced by grinding quartz sand (specific surface area of 700–800 m^2/kg) and microsilica MK-85 particles (particle size less than 10^{-6} m) with a silicon dioxide (SiO_2) content of more than 85%.

Inotek aluminosilicate hollow microspheres were chosen as the functional filler for the lightweight concrete; their properties are presented in Table 2.

A complex nanosized modifier based on an iron hydroxide and silicic acid sol was used for modification of the filler surface.

Component	
Tricalcium silicate (C_3S): %	61·5
Dicalcium silicate (C_2S): %	16·1
Tricalcium aluminate (C_3A): %	6·2
Tetracalcium aluminoferrite (C_4AF): %	6·2
Silicon dioxide (SiO_2): %	23·4
Aluminium oxide (Al_2O_3): %	5·0
Iron oxide (Fe_2O_3): %	4·0
Calcium oxide (CaO): %	60·4
Magnesium oxide (MgO): %	1·1
Sulfur trioxide (SO_3): %	2·8
Chlorides (Cl^-): %	0·003
Calcium sulfate ($CaSO_4$): %	5·4
Alkali metal oxides (R_2O): %	0·75
Loss on ignition: %	1·7

Table 1. Mineralogical and chemical composition of Mordovcement clinker

Property	
Appearance	Grey powder
pH	6–7
Density: kg/m^3	600–800
Compressive strength: MPa	13–35
Fraction: 10^{-6} m	0–100
Wall thickness: % of diameter	10
Humidity: %	<0·5
Oil adsorption: g/g	16–18
Silicon dioxide (SiO_2): %	58–62
Aluminium oxide (Al_2O_3): %	32–38

Table 2. Properties of hollow aluminosilicate microspheres

Preparation of the nanomodifier, as suggested by Grishina and Korolev (2012), included three stages. In the first stage, a true solution of ferric chloride is prepared and hydrolysed in order to produce iron hydroxide (III) sol (average particle size of 6·5–8·5 nm) in the second stage. In the third stage, sodium ions in an aqueous hydrosilicate solution are added to interact with the negatively charged nanoparticle sol of iron hydroxide (III). This leads to the formation of silica sol with an average particle size of 15–20 nm). This multi-component modifier creates a shell on the surface of the microspheres with reactivity to cement and the products of cement hydration.

A polycarboxylate-based superplasticiser (Melflux 1641F, BASF Construction Polymers (Trostberg, Germany)) was used at a dosage of 0·8% by weight of cement for each composition to decrease the water/cement ratio and improve workability of the concrete mixture.

The concrete compositions were prepared by combining and mixing the components in an automatic mortar mixer according to DIN EN 196-1-2005 (DIN, 2005). The concrete samples were cured at a temperature of 20°C and humidity of 80–85%. The structure of the HSLC is shown in Figure 1.

Physical and mechanical properties were determined for cube samples (edge length 70 mm) and prisms (40 × 40 × 160 mm) according to ASTM C39/C39M-10 (ASTM, 2010). ASTM C469/C469M-14 (ASTM, 2014) was used to determine the elastic modulus and Poisson's ratio (for 70 × 70 × 280 mm prisms). The concrete structure was examined using a Senterra Raman spectrometer; further information on technical parameters can be obtained from the literature (SECNN, 2015).

The coefficient of fracture toughness was evaluated by the MIIT method (Leszczynski, 1980; Scheikin, 1972) according

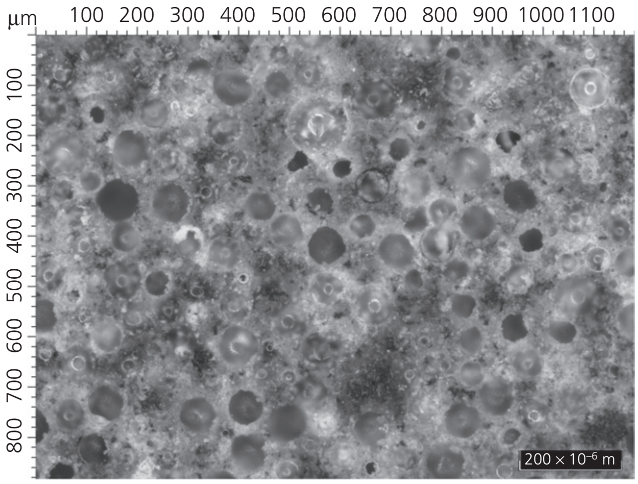

Figure 1. Example of structure of HSLC with hollow microspheres; average density 1400 kg/m^3

to the formula

$$k_{\mathrm{ft},2} = \frac{R''}{R'} \quad (1)$$

Results and discussion

Different operational factors can lead to the formation of cracks and destruction of the material when concrete is under load in constructions. The relation of tensile strength to compressive strength as a coefficient of fracture toughness is thus an important characteristic for structural concrete, and this research studied the HSLC compositions listed in Table 3.

The process of destruction depends on the adhesion properties of the cement stone and filler particles. To increase resistance to crack formation and growth it is necessary to improve adhesive strength at the interface boundaries. The nanoscale additive BisNanoActivus (Grishina and Korolev, 2012), based on iron hydroxide and silicic acid sol, has been proposed as an adhesive. This modifier intensifies interactions and provides for the formation of additional cement hydration products in the contact zone (Figure 2), thus strengthening the phase boundary.

Analysis of cement stone samples (A in Figure 2) showed well-defined peaks, identified as Raman emission peaks connected with carbonaceous bonds (278 cm^{-1}), ettringite (SO_4^{-2}) (989 cm^{-1}) and silicon–oxygen (Si–O) bonds (1085 cm^{-1}) (Sagrario and Lucia, 2011), with relative intensities of 547, 365 and 806 respectively. Analysis of the interface between the cement stone and microspheres (B in Figure 2) revealed only two peaks of decreased relative intensity (137 and 501 for peaks 4 and 5 respectively), specific to the Si–O stretching band. Internal strain of tetrahedral silicates $v_4[SiO_4]$ generates bonds in the region of 400–600 cm^{-1} (Machovic *et al.*, 2006; Peskova *et al.*, 2011), which can be used to describe the spectrum of the HSLC at the cement stone–microsphere phase boundary. The 462 and 517 cm^{-1} peaks may be interpreted as deformation oscillations of anti-symmetric bonds of O–Si–O. Comparison of relative intensities (bands 649; 643; 141; 401 and 1239 cm^{-1}) shows that corresponding height of the peaks is growing by 15, 11 and 35%, respectively. This provides evidence of an increase in the number of crystalline reaction products at the boundary.

The properties of the HSLCs were examined and compared with high-strength fine-grained heavy concrete. The results in Table 4 show that the developed HSLCs (compositions C1–C6) were of comparable fracture toughness to the fine-grained high-strength heavy concrete (composition C0). The table also shows that increasing the content of the hollow microspheres led to decrease in fracture toughness, which can be explained by the reduced thickness of the cement paste layers for a higher volume of microspheres (i.e. lower average density of concrete). Furthermore, the HSLCs with unmodified

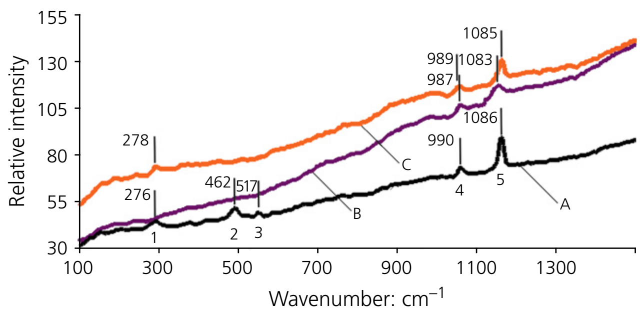

Figure 2. Raman spectra of cement stone (A), interface of cement stone/microspheres (B) and interface of cement stone/modified microspheres (C)

Composition	Target density: 10^3 kg/m^3	Content: % mass[a]					
		C	MS	MMS	QS	MA	W
C0	2·4	29·7	—	—	43·2	17·8	9·3
C1	1·3	48·0	21·8	—	2·8	9·9	17·5
C2	1·4	44·6	18·5	—	9·2	11·4	16·2
C3	1·5	41·6	15·6	—	14·9	12·8	15·2
C4	1·3	48·0	—	21·8	2·8	9·9	17·5
C5	1·4	44·6	—	18·5	9·2	11·4	16·2
C6	1·5	41·6	—	15·6	14·9	12·8	15·2

[a]C, cement; MS, microspheres; MMS, modified microspheres; QS, quartz sand; MA, mineral additive (including stone powder and microsilica); W, water (taking into account content of plasticiser at 0·8% by weight of cement)

Table 3. Compositions of fine-grained high-strength heavy concrete (C0) and HCLCs (C1–C6)

Composition	v_f	ρ: 10^3 kg/m^3	R_{fs}: MPa	R_{com}: MPa	$k_{ft,1}$
C0	0	2·32	19·1	115·1	0·166
C1	0·532	1·25	4·9	45·3	0·108
C2	0·485	1·35	6·6	47·6	0·139
C3	0·438	1·46	7·8	54·0	0·145
C4	0·532	1·28	5·0	49·7	0·101
C5	0·485	1·37	5·7	51·4	0·110
C6	0·438	1·48	7·3	62·1	0·116

Table 4. Coefficients of fracture toughness

microspheres (C1–C3) showed higher (6–20% higher, depending on average density) values of fracture toughness than the compositions with nanomodifier.

The decrease in fracture toughness of the HSLCs is partially due to the relation

$$2. \quad k_{ft,1} = \frac{R_{fs}}{R_{com}}$$

The interaction of the nanomodifier components with the cement and hydration products (Figure 2) ensures growth of compressive strength with a constant value of flexural strength. This is because the hollow filler microparticles are spherical in form and thus have good resistance to compressive loads (Table 2) but, at the same time, the fragile thin walls of the microspheres are not able to resist significant bending loads.

The same is true for the concretes containing modified microspheres. Active silica on the surface of the microspheres intensifies the local interaction with portlandite: silicon dioxide reacts with calcium hydroxide to form calcium hydrosilicates directly in the zone of binder/filler contact. Thus, dense strong bonds are formed in the most defective areas and this reduces the role of microspheres as structural defects. The formation and propagation of cracks occurs at applied loads and a high volume content of filler promotes the branching of microcracks and prevents the formation of larger trunk cracks and destruction of the concrete. Therefore this parameter (formula 2) cannot exhaustively or adequately characterise the fracture toughness or the efficiency of the method proposed to improve it.

The data presented in Table 5 show that the proposed complex nanomodifier, applied to the surface of the microspheres, plays a functional adhesive role and increases strength values of HSLC by 12–20% regardless of the average density of the concrete. This is due to the concrete's increased resistance to crack formation and growth. The intensification of cement hydration processes on the surfaces of the microspheres leads to additional amounts of calcium hydrosilicates formed on the interface boundary, thus hardening this zone. Because the microparticles have a high specific surface area and a good degree of dispersion in the mixture, much of the microsphere surface forms strong bonds with the cement stone throughout the whole volume, thus leading to reduced deformations and increased concrete strength.

To evaluate the fracture characteristics of the HSLCs and the influence of modifier on these characteristics, deformation values at 30% of the breaking load were obtained experimentally. The results are presented in Figure 3 and Table 6. The relative longitudinal and transverse deformations of the HSLCs are greater than those of the fine-grained high-strength heavy concrete (C0) by 1·20–1·60 and 1·37–2·21 times respectively. This is due to the presence of microspheres with a low elastic modulus. The hollow particles crush even when exposed to relatively low loads, but C1 and C4, with an average density 1290 kg/m^3, show less longitudinal and transverse deformation than other compositions with a higher density. This can be considered as proof of the hypothesis that hollow microspheres are natural structural defects; an increase in their number leads

Composition	ρ: $\times 10^3$ kg/m^3	R_{com}: MPa	R_{sp}: MPa	R_{pr}: MPa
C0	2·34	122·2	52·3	93·4
C1	1·29	45·3	35·1	41·8
C2	1·38	49·4	35·8	44·2
C3	1·48	54·9	37·0	47·0
C4	1·29	52·2(+15·2%)	40·5(+15·5%)	47·4(+13·4%)
C5	1·39	55·2(+11·7%)	39·7(+11·1%)	49·7(+12·4%)
C6	1·50	65·8(+19·9%)	43·9(+18·7%)	56·5(+20·2%)

Table 5. Strength properties of the studied concretes. The values in parentheses show the change in nanomodified HSLC with respect to the HSLC with the same average density without nanomodifier

to intensive branching of cracks and, as a net result, causes extra resistance to cracking.

Figure 3 and Table 6 show that the HSLCs (C1–C6) had smaller values of elastic modulus than the fine-grained high-strength heavy concrete (C0). At the same time, the Poisson's ratios of C1–C6 were more than or about equal to the C0 Poisson's ratio. Modification of the surface of the microspheres by means of nanoscale additive thus had a positive effect in terms of reducing deformations and fragility fracture. Compositions C4, C5 and C6, prepared with nanomodified microspheres, showed less relative longitudinal and transverse deformations than the reference compositions C1, C2 and C3, with differences of around 7–12% and 8–16% respectively. The elastic modulus was increased by 7·6–14·1% due to compaction of the structure because of additionally formed bonds of calcium hydrosilicates at the phase boundary (Figure 2). Due to the intensive reductions in transverse deformations (compared with longitudinal deformations) a reduction in Poisson's ratio by around 5–10% was observed for compositions with average densities of 1300–1400 kg/m^3.

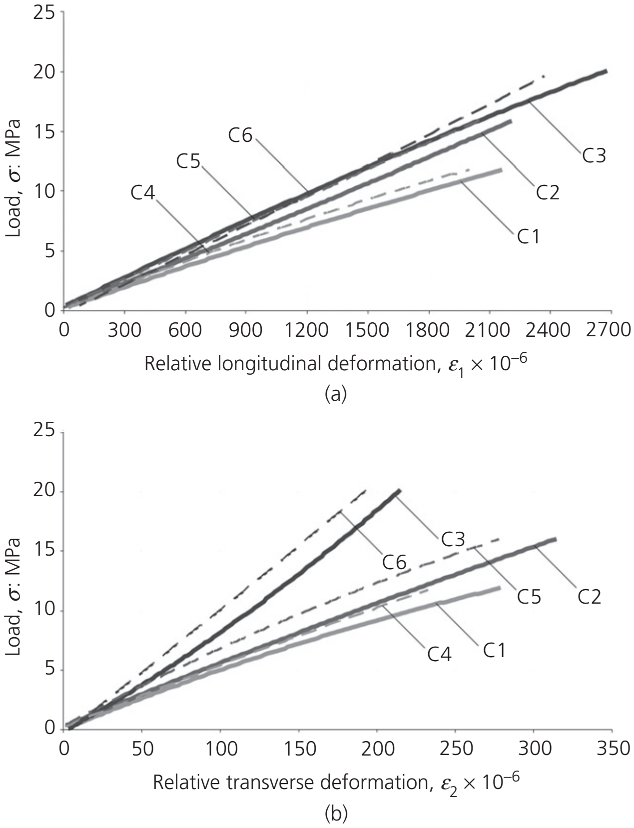

Figure 3. Longitudinal (a) and transverse (b) deformations of HSLCs and nanomodified HSLCs

For concrete in service, various kinds of local cracks are formed due to shrinkage effects and many other external factors. An additional criterion for evaluation of the coefficient of fracture toughness was thus chosen – the so-called MIIT method. Table 7 lists the values of fracture toughness determined by this method, characterised by the flexural and compressive strength. Analysis of Table 7 shows that the formation and growth of cracks due to internal stresses (e.g. extreme changes in operating conditions, variable wetting and drying)

Composition	R'_{fs}: MPa	R'_{com}: MPa	R''_{fs}: MPa	R''_{com}: MPa	$k_{ft,2}$ from R_{com}	$k_{ft,2}$ from R_{fs}
C1	4·92	44·6	1·42	41·0	0·919	0·289
C2	6·50	48·4	1·75	44·7	0·924	0·269
C3	7·04	53·7	1·87	49·3	0·918	0·266
C4	5·28	50·2	2·13	45·7	0·911	0·403
C5	6·07	52·1	2·56	47·2	0·906	0·422
C6	7·31	60·3	3·22	56·0	0·928	0·440

Table 7. Coefficient of fracture toughness determined by MIIT method

Composition	σ: MPa	$\varepsilon_1 \times 10^{-6}$	$\varepsilon_2 \times 10^{-6}$	E: GPa	μ
C0	28	1669	142	19·2	0·085
C1	12	2163	279	5·55	0·129
C2	16	2210	314	7·25	0·142
C3	20	2678	214	7·47	0·080
C4	12	2002(−7·4%)	233(−16·4%)	6·00(+8·0%)	0·116(−9·7%)
C5	16	2053(−7·1%)	278(−11·5%)	7·80(+7·6%)	0·136(−4·7%)
C6	20	2347(−12·3%)	195(−8·6%)	8·52(+14·1%)	0·083(+4·2%)

Table 6. Deformation properties of the studied concretes. The values in parentheses show the change in nanomodified HSLC with respect to the HSLC with the same average density without nanomodifier

is less intense in the nanomodified HSLCs. With use of the proposed nanomodifier, it is possible to increase the fracture toughness by 39·4–65·4% (if the latter is defined through calculation of flexural strength). However, this calculation method for the coefficient of fracture toughness is not really adequate and is also of low information content – the ranges of the obtained values correspond to the range of measurement errors (0·8–1·9%).

However, the proposed modification method of application of a colloidal solution of iron hydroxide and silicic acid (which are reactive to cement and hydration products) to the surface of hollow microspheres can be considered an effective way of reducing deformations due to hardening of the interface boundary between the cement stone and hollow filler.

Conclusions

- Hollow microspheres are prospective fillers for high-performance lightweight concrete. Increasing the content of spherical microparticles in the concrete composition promotes the formation of a close-packed structure that shows small deformation. The coefficient of fracture toughness of the high-strength lightweight concretes (HSLCs) studied was comparable to that of fine-grained high-strength heavy concrete (more than 0·1) and was limited by the strength characteristics of the micrometric particles of aggregate.
- A method of increasing the fracture toughness of HSLC containing aluminosilicate microspheres was proposed. This method consists of application of a colloidal solution of iron hydroxide and silicic acid (which are reactive to cement and hydration products) to the surface of hollow microspheres. Reinforcement of the phase boundary occurs as a result of this application.
- The variation in longitudinal and transverse deformations of HSLC with volume content of the hollow microspheres was examined and the influence of the nanomodifier on the elastic modulus and Poisson's ratio of the studied concretes was shown. The proposed modification method led to reductions in HSLC longitudinal and transverse deformations of 1·20–1·60 and 1·37–2·21 times respectively. Application of the nanomodifier reduced the intensity of cracking under the influence of shrinkage stresses by 56·9%.

It is necessary to search for combined solutions that will provide the reinforcement and/or damping effect to reduce deformations and to increase the elastic modulus and Poisson's ratio of HSLC to make them close to values for fine-grained high-strength heavy concrete.

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