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INFLUENCE OF DELAYED ETTRINGITE FORMATION ON THE MECHANICAL PROPERTIES OF AERATED CONCRETE

VPLIV ZAKASNELE TVORBE ETRINGITA NA MEHANSKE LASTNOSTI AERIRANIH BETONOV

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Delayed ettringite formation (DEF) is a chemical reaction with proven damaging effects on the mechanical properties of a hydrated concrete. Ettringite crystals can cause cracks and their widening due to the pressure on the crack walls caused by a positive volume difference in the reaction. Concrete may show improvements in strength at early stages, but a further growth of the cracks causes widening and spreading of these cracks through the concrete structure. In a hydrated concrete, crystals of ettringite can also cause a disintegration of the concrete. In this paper we investigate the potential to utilise a positive volume difference in DEF in order to improve the mechanical properties of a hydrated fine-grained concrete. Finely dispersed crystallization nuclei, achieved by adding an air-entraining agent (AEA) and a short vibration of specimens, is presented as the main prerequisite for such improvements. The control of the expansion and mechanical properties were performed on the concrete samples with and without AEA by inducing DEF. For a microstructure examination of the fine-grained concrete an optical microscope and a scanning electron microscope were used. We found that controlled DEF, which is guaranteed by adding AEA and with the formation of the uniformly dispersed air bubbles, which are the crystallization sites for ettringite crystals, improves the mechanical properties. The specimens with induced DEF were measured and found to have a 6 % increase in the compressive strength.

Keywords: delayed ettringite formation (DEF), concrete, aerated concrete, microstructure, mechanical properties

Zakasnela tvorba etringita (DEF) je kemična reakcija z dokazano škodljivim učinkom na mehanske lastnosti hidratiziranega betona. Kristali etringita lahko zaradi pozitivne prostorninske razlike med reaktanti in produkti povzročijo nastanek razpok, rast kristalov etringita na stenah razpok pa povzroči njihovo širjenje. Pri mladih betonih rast kristalov etringita poveča trdnost, v hidratiziranem betonu pa nastanek in napredovanje razpok, kar lahko povzroči tudi razpad betona. V tem članku predstavljamo idejo, kako bi lahko pozitivne volumenske spremembe, ki so posledica DEF v hidratiziranem, drobnozrnatem betonu izkoristili za izboljšanje mehanskih lastnosti. Pogoj za izboljšanje le-teh je zagotovljen z uporabo dodatka aeranta (AEA), s katerim v hidratiziranem betonu zagotovimo enakomerno razpršene zračne mehurčke, ki so nukleacijska jedra. Zgostitev betona smo izvedli z vibriranjem, ki lahko traja le kratek čas. V raziskavi smo prikazali način in postopek izdelave betona z enakomerno razpršenimi nukleacijskimi jedri iz zračnih mehurčkov. Kontrolo nabrekanja in mehanskih lastnosti smo opravili pri vzorcih betona z dodanim aerantom in brez njega z induciranjem zakasnele tvorbe etringita. Za raziskave mikrostrukture vzorcev drobnozrnatega betona smo uporabili optični in elektronski vrstični mikroskop. Ugotovili smo, da lahko z nadzorovano zakasnelo tvorbo etringita, ki je zagotovljena z dodanim aerantom in nastankom enakomerno razpršenih zračnih mehurčkov, ki so nukleacijska mesta za kristalizacijo etringita, izboljšamo mehanske lastnosti. Pri vzorcih z inducirano zakasnelo tvorbo etringita smo izmerili 6-odstotno večjo tlačno trnost.

Ključne besede: zakasnela tvorba etringita (DEF), beton, aerirani beton, mikrostruktura, mehanske lastnosti

1 INTRODUCTION

Delayed ettringite formation (DEF) in cementitious materials is considered as a harmful chemical reaction leading to a variety of damages.[1–3] The volume of the formed DEF crystals in a hardened concrete is larger than the volume of reactants and the main results are the forces arising from the growing crystals acting upon the walls of the crack. As a consequence, DEF cracks continue to grow wider and spread through the concrete structure.[4,5] A considerable recent research has led to a better understanding of the mechanisms of DEF.[6] In general, it is acknowledged that DEF is a result of various factors and conditions including excessive temperatures of above 70 °C, the presence of sulphates, existing cracks, moist conditions and so on.[7–10] Ekolu et al[11] summarise various control measures that could be used for the prevention of DEF including the use of chemical additives. However, preventative measures and improvements with respect to durability require further attention.

In practice, concrete and mortar mixes are normally based on Portland cement clinker, where the chemical process of hydration of the clinker minerals yields hydrates and hydroxides. However, because of the presence of gypsum, the chemical reaction between tricalcium aluminate (C_3A), gypsum ($CaSO_4 \cdot 2H_2O$) and water forms ettringite crystals ($3CaO\ Al_2O_3\ 3CaOSO_4 \cdot 32H_2O$). The volume difference in this reaction is positive and ettringite crystals grow fast, growing quickly on the unhydrated cement particles, which can slow down the hydration[12]. The presence of ettringite in a liquid cementitious system is unproblematic but its formation or re-formation in an already hydrated concrete can lead to extensive damages.[6] Due to the resulting volume difference, particularly in the presence of sulphates, an expansive force within the concrete can cause its disintegration (sulphate corrosion). It is well known that cements with a low C_3A content are more resilient to sulphate corrosion, but that also depends on the form of C_3A.[13] For instance, crystalline C_3A is more reactive than its amorphous version.

A positive volume difference as a result of an early ettringite formation (EEF) in cementitious materials rich with $C_4A_3\overline{S}$ (calcium aluminate sulphate, expansive cement) can be used to compensate for the shrinkage during the drying.[14] In this case, $C_4A_3\overline{S}$ hydrates within a few hours or days causing a uniform distribution of ettringite and a homogeneous expansion of the hardened concrete at early stages. However, it is less known that an ettringite formation and expansion in hardened cementitious materials can also be used for their controlled strengthening.

The formation of a new phase characterized by a substantial volume expansion for the purpose of strength improvement is well known in the mainstream material science. Such strengthening is based upon the creation of an internal compressive stress on the contact between the existing matrix and the new-phase particles and depends on their shape, size and their overall dispersion. It is desirable that the newly formed particles are small, spherical and located sufficiently apart from each other to avoid overlapping the stress fields. In the case of metallic materials we know of the dispersion strengthening of the cooper matrix with the ZrO_2 particles[15,16]. A strength improvement of the Al_2O_3 ceramics with finely dispersed ZrO_2 particles is one example of this kind of internal stresses in an Al_2O_3 matrix created by applying an external-force-trigger polymorphic transformation of a tetragonal crystalline structure of ZrO_2 into a monoclinic crystalline structure. An increased volume creates substantial compressive stresses in a matrix surrounding the transformed particles leading to a several-fold increase in the compressive strength as well as in the resistance to the spreading of the cracks. The studies that apply to this type of mechanism for a strength improvement of concrete are rare.

2 EXPERIMENTAL WORK

2.1 Description of the experiment

Experimental work included a selection and research of concrete components, an investigation of a cement paste and testing of the samples of a fine-grained concrete. Experimental work was determined with an experimental design research with the aim of confirming that DEF in a hydrated concrete improved the strength properties and also that an addition of an air-entraining agent (AEA) creates, by volume, uniformly distributed nucleation sites for the formation of ettringite crystals and a target microstructure.

2.2 Materials

2.2.1 Selection of fine-grained concrete components

For the sample preparation we used the cement known as CEM I 42.5 R. In order to ensure the formation of the AF_m phase, we used fly ash, for which we defined the components and the specific surface area. The role of fly ash was twofold – chemical and physical; its chemical role was possible because fly ash is actively involved in the pozzolanic reaction, while its physical impact was in place because it works as a nucleation site and as filler. For the aggregate we used chemically neutral standard sand with its properties being in line with EN ISO 196-1 and ISO 697. The purpose of adding a chemically neutral aggregate was to prevent an alkali-silica reaction (ASR), which would result from the reactive minerals in the aggregate. The most important precaution was the use of the petrographic components of the aggregates, which will not react with alkali.[17] The used additive – an air-entraining agent – was the anion-based abietic acid. The components were mixed with clean drinking water.

2.2.2 Characterization of the samples and the investigation procedure

Concrete-mixture components were determined on the basis of the standard consistency of the cement paste. We prepared two different series of the samples. The compositions of the mixtures are shown in **Table 1**. The samples were mixed with a laboratory mixer to meet the requirements of EN 196-1. Fresh concrete was built in the standard moulds with the dimensions of 4/4/16 cm using a vibrating table with a vibration time of 5 s, a frequency of 50 Hz and amplitude of 0.75 mm. The samples were monitored for 28 d in a climatic chamber at a temperature of (20 ± 2) °C and a relative humidity of (98 ± 2) %. In all the samples we performed the measurements of density, compressive and flexural strengths in the intervals of (7, 14, 28 and 56) d. On the set of the samples that contained fly ash, after 28 d of treatment in the climatic chamber, we performed a Duggan test. This test is used to achieve an accelerated delayed ettringite formation. The test consists of soaking the samples in demineralised water at (20 ± 2) °C and drying them at a temperature of (81 ± 2) °C. The test cycles are shown in **Figure 1**. After the Duggan[18] test the prisms were put through a laboratory conditioning in a desiccator for 48 h in the time between the above phases,

Table 1: Compositions of fine-grained concrete mixtures
Tabela 1: Prikaz komponent drobnozrnatega betona

Specimen	Aggregate (g)	Cement (g)	Fly ash (g)	Water (g)	AEA (g)
AI	1350	450	–	218.2	6.8

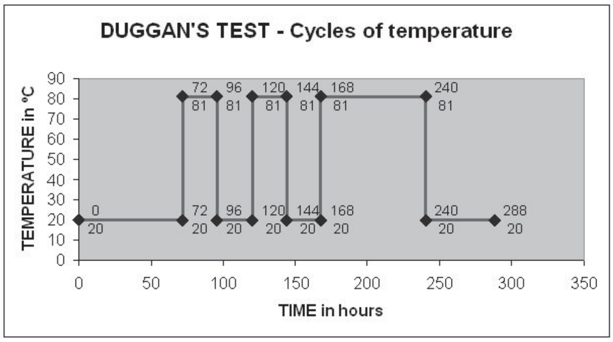

Figure 1: Cycles of Duggan test
Slika 1: Prikaz ciklov Dugganovega testa

and were then once again immersed in demineralised water for 24 h in order to fill the capillaries and voids with water. In these samples we controlled the delayed ettringite formation by measuring the expansion caused by ettringite-crystal growth.

At the moment when the expansion was no longer observed, in micrometers, we stopped the measurements. We considered that the delayed ettringite formation had been completed. The measurements of density and strength were then performed on the samples. These measurements were repeated on the samples where the Duggan test was not carried out, so we could obtain the comparisons of these values.

3 RESULTS AND DISCUSSION

3.1 Results of the investigations of concrete components

Cement, standard sand and an air-entraining agent were used as the benchmark for the standard-quality results. Therefore, declared properties are not included. Fly ash contains silicates, carbonates and phosphates of calcium, magnesium, iron and aluminum and other elements. Illite/kaolinite clays, apart from illite and kaolinite minerals, also contain α-quartz, Fe_2O_3 and $CaCO_3$.[19] The results of the laboratory analysis for fly ash are shown in **Table 2**.

3.2 Characterization of the macrostructure

With an optical microscope Olympus SZX we observed significant break areas of the hydrated concrete. The control of the dispersion bubbles of the air-entraining agent showed that they were mostly distributed uniformly. The bubbles had a diameter between 25 µm and 50 µm. The average measured distance between the bubbles was 0.1 mm to 1.2 mm.

3.3 SEM characterization

With the electron scanning microscopes JEOL JSM 5610 and QUANTA 200 3D, we observed the characteristic fields of the air-entraining bubbles. In these bubbles we discovered ettringite crystals. This proved our assumption that the bubbles of the air-entraining agents are nucleation sites. The morphologies of individual ettringite crystals are very similar. Ettringite crystals grow in bunches, and they are all needle-like and thin. An examination of a large number of bubbles showed that sample A has fewer ettringite crystals, as shown in **Figure 2**. The samples that were labelled as BI and BI-DT showed many more bunches of ettringite crystals. These crystals grow mainly on the porous sites in the air bubbles (**Figure 3**). On the samples that were labelled as BI-DT, we noticed more microcracks in the bubbles than on the samples labelled as BI. In these microcracks we also observed the presence of ettrigite crystals that were very thin and needle-like, suggesting their rapid growth (**Figure 4**).

A comparison of the microstructures of the specimens from the fine-grained concrete mixes AI, BI and BI-DT, presented in **Figures 2, 3** and **4**, clearly demonstrates that, as expected, similar AEA-induced nuclei exist in AI, BI and BI-DT specimens. Although ettringite crystals can be found in the concretes produced by using pure Portland cement, no visible ettringite crystals were detected in any of the numerous prisms from the AI mix. However, ettringite crystals did appear

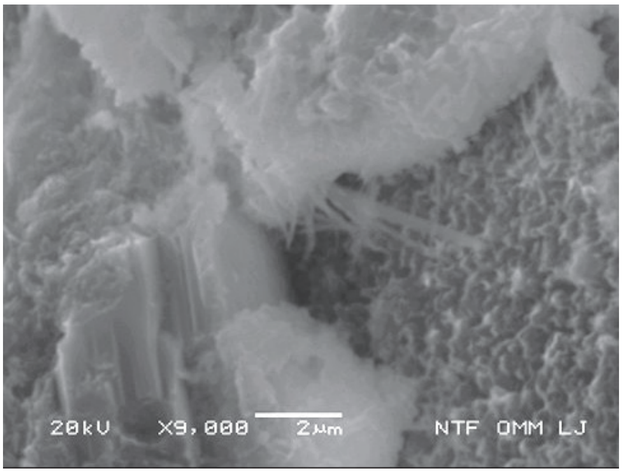

Figure 2: Micro fractography of sample AI with ettringite crystals; SEM, SEI
Slika 2: Mikrofraktografija vzorca z oznako AI s kristali etringita; SEM, SEI

Table 2: Results of the laboratory analysis for fly ash
Tabela 2: Rezultati laboratorijskih analiz elektrofiltrskega pepela v masnih deležih, *w*/%

Component part	Content, *w*/%
Loss on ignition	0.41
Insoluble residue	16.67
SiO_2 impure	13.08
SiO_2 pure	47.62
SiO_2 soluble	0.64
SiO_2 total	48.26
SiO_2 active	35.18

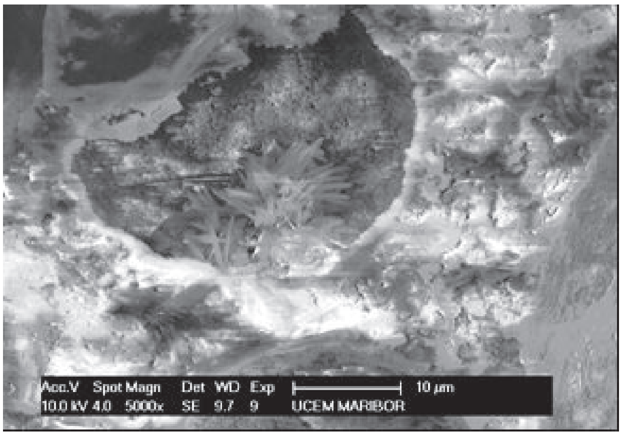

Figure 3: Micro fractography of sample BI with ettringite crystals; SEM, SEI
Slika 3: Mikrofraktografija vzorca z oznako BI s kristali etringita; SEM, SEI

in the specimens of all the other fine-grained concrete mixes. The microstructures of the specimens from the fine-grained AI concrete mix show very few ettringite crystals in the AEA nuclei themselves, but they were detected in the microcracks (**Figure 2**). In line with the findings established by Myneni et al.[20] these crystals have a thin, needle-shaped morphology and the length of approximately 2 µm, revealing a rapid growth. The fly ash in the fine-grained BI concrete mix may well be a source of soluble calcium required for an ettringite formation, as reported by Solem and McCarthy[21], Zhang and Reardon[22] or Chrysochoou and Dermatas,[23] because its crystals were found in large quantities in the microcracks and within the AEA-induced nuclei. **Figures 2** and **3** show that ettringite crystals have a thin, needle-shaped morphology, while those found in the microcracks are only approximately 2 µm long as opposed to the 10-µm-long crystals found in the nuclei (**Figures 3** and **4**). The microcrack that appeared on the surface of a nucleus in **Figure 4** can be associated with shrinkage of the matrix during the hydration[24]. The comparison between various different BI specimens shows that ettringite crystals start growing wherever there is enough space for the growth before further damaging the concrete, which enables further growth. The AEA-induced nuclei may, therefore, act as relief reservoirs enabling the growth of the substances like ettringite crystals in a hardened concrete with minimum or no damaging effects. Hime[25] even recommends air entrainment as a way to prevent DEF and reports on only a single incident where an air-entrained concrete suffered from DEF.

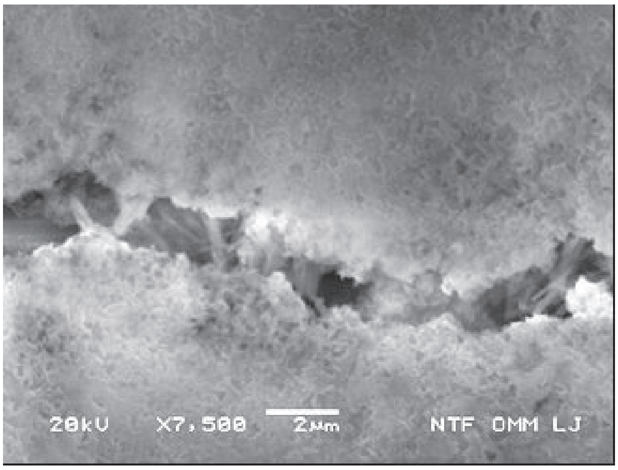

Figure 4: Micro fractography of sample BI-DT with ettringite crystals; SEM, SEI
Slika 4: Mikrofraktografija vzorca z oznako BI-DT s kristali etringita; SEM, SEI

3.4 TGA analysis

A simultaneous thermal analysis (STA) and a thermal gravimetric analysis (TG) were performed on the cement-paste samples of concretes A and BI in a static air atmosphere. Measurements were performed with a device Netzsch STA 449C Jupiter. We measured the change in the mass of the samples at 25 °C. Each measurement lasted 60 h. The maximum change in the mass, which was 19.88 %, was found in the cement-paste sample of concrete AI after a period of 446.8 min (**Figure 5**). The change in the mass of the cement-paste sample of concrete BI was lower and amounted to 19.66 % after a period of 438.2 min (**Figure 6**). The TG measurements have shown that, by modifying the formation of a concrete sample, the changes in

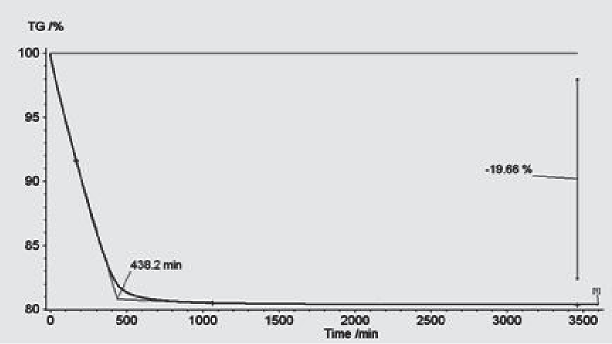

Figure 5: Results of a TG analysis for sample AI
Slika 5: Rezultati TG-analize za vzorec AI

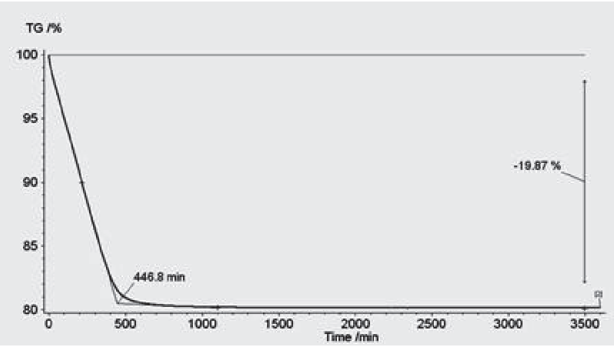

Figure 6: Results of a TG analysis for sample BI
Slika 6: Rezultati TG-analize za vzorec BI

the fly ash did not transgress the setting time of the concrete, which must be longer than 360 min and shorter than 540 min.

3.5 XRM analysis

X-ray mapping (XRM) analyses were performed with the electron scanning microscope JEOL JSM 5610 on the samples that are the fracture sites of the hydrated fine-grained concrete labelled as BI-DT. We analysed a particular field with the crystals of ettringite as shown in **Figure 7**. Based on the results, it was suggested that, in the areas of air bubbles in the cement matrix, the sulphate attack had been completed. These results confirmed that the process of delayed ettringite formation had been completed too. The chemical elements – sulfur and aluminum, which are characterized by their chemical reactions, are present only in the sites with ettringite crystals.

3.6 pH measurement

The fine-grained hydrated concrete was sampled by breaking off a piece. This piece of concrete was crushed and sifted through a sieve with an aperture diameter of 0.02 mm. 50 g of the sample was mixed with a laboratory stirrer with 10 ml of distilled water. The solution was filtered, before being tested, through a filter paper N°40. The measurement procedure was taken from the literature.[26] The measurements were performed with a pH meter Mettler Toledo S20 on three samples of each type of concrete in the intervals of (1, 2, 3 and 4) min. For each sample we performed three measurements and calculated the mean value. The results of the mean values are presented in **Table 3**. Based on the measurements, we found that the pH of the hydrated concrete with fly ash was reduced due to the Duggan test, although this value did not change significantly. It is known that the pH of a concrete with fly ash is lower than in the cases of the concretes without it. This is due to the binding of $Ca(OH)_2$ with the reactive silicates contained in fly ash.[27] The results confirmed that, with an addition of fly ash, the concrete's pH was decreased below the critical value of 9. This means that the corrosion resistance of concrete is guaranteed.

Table 3: Measurements results for the pH values

Tabela 3: Rezultati meritev pH-vrednosti

Sample	pH (mean value)
AI	12.33
BI	11.88
BI-DT	11.86

3.7 Expansion measurement

After a required 28-day curing period 6 prisms of the BI mix were exposed to a Duggan test in order to achieve the accelerated ettringite formation. The prisms were then placed into a standard apparatus for determining the length change of the hardened cement paste, mortar and concrete, constructed according to ASTM C490-86. During these measurements the apparatus itself was placed in a climatic chamber with a constant temperature of (20 ± 2) °C and a relative humidity of (98 ± 2) % (**Figure 8**).

Ettringite formation was then monitored by measuring the length change (expansion) with a Mahr's MarCator 1080/12.5/0.005 mm digital micrometer. The results were recorded with an analogue/digital converter connected to a workstation. The expansion development was regularly measured in 15-minute intervals with a measurement accuracy of 0.005 mm, although the intervals could well be longer considering the slow pace of DEF.

Figure 9 shows the change in the length for six fine-grained concrete prisms from the BI mixes that were exposed to the Duggan test and the final 24-hour immersion in demineralised water. The change in the

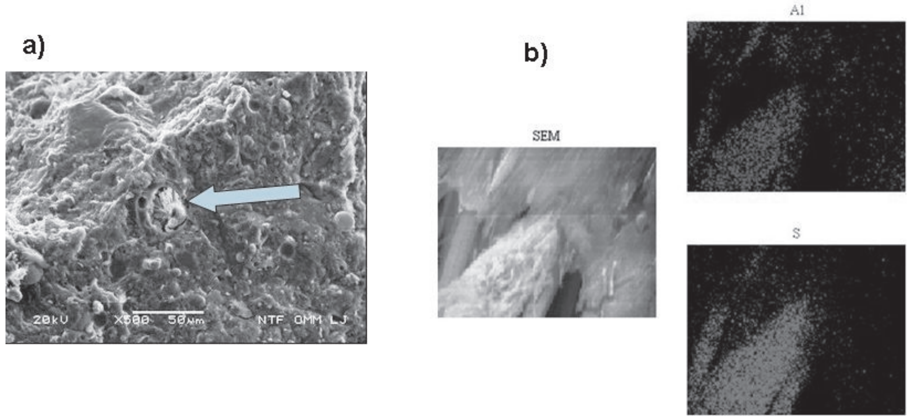

Figure 7: XRM analysis of an ettringite crystal in an air bubble of AEA: a) the arrow on the micro fractograpy SEM, SEI, shows the analyzed crystal, b) the chemical elements S and Al are present only in ettringite crystal

Slika 7: XRM-analiza kristalov etringita v zračnem mehurčku aeranta: a) puščica na mikrofraktografiji SEM, SEI kaže analizirani kristal, b) kemijska elementa S in Al sta prisotna samo na kristalu etringita

Figure 8: Apparatus for measuring an expansion of hardened concrete according to ASTM C490-86, placed in a climatic chamber

Slika 8: Aparat za meritve nabrekanja otrdelega betona, izdelan skladno z ASTM C490-86, v klimatski komori

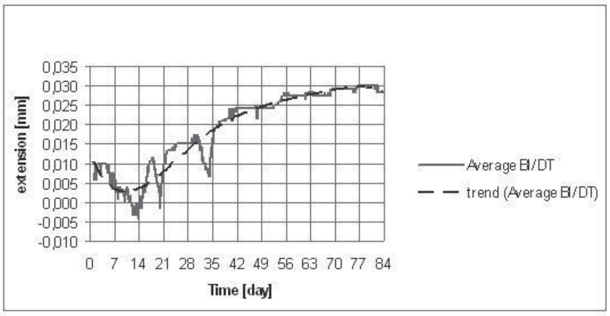

Figure 9: Results of the expansion measurements for the samples labelled as B-DT after the Duggan test

Slika 9: Rezultati meritve nabrekanja za vzorce z oznako B-DT po Dugganovem testu

Table 4: Densities and mechanical properties of the hardened fine-grained concrete (mix AI)

Tabela 4: Vrednosti gostote in mehanskih lastnosti za otrdeli drobnozrnati beton (mešanica AI)

Time interval t/d	ρ/(kg/m^3)	f_m/MPa	f_c/MPa
7	1806	2.5	18.0
14	1804	4.0	14.8
28	1883	5.2	17.3
56	1885	5.3	19.5
121	1887	5.4	20.5

length of the prisms was measured daily and stopped after 83 d when the measurements did not show any further expansions. The result of the measurements of the length change (expansion) of prismatic samples was evaluated as the average value which is 0.0125 %. All the samples were visually inspected and no cracks were found.

3.8 Mechanical properties

The density of the hardened fine-grained concrete (ρ), its compressive (f_c) and flexural strengths (f_m) were measured on 6 additional prisms for each of the three mixes after the standard periods of (7, 14 and 28) d, and, additionally, after 56 d and 121 d when compressive and flexural strengths should reach a plateau. The mechanical properties of the fine-grained concrete were examined with a material testing machine Zwick Roell and a method according to EN 196-1.

Table 5: Densities and mechanical properties of the hardened fine-grained concrete (mix BI)

Tabela 5: Vrednosti gostote in mehanskih lastnosti za otrdeli drobnozrnati beton (mešanica BI)

Time interval t/d	ρ/(kg/m^3)	f_m/MPa	f_c/MPa
7	1801	2.7	11.6
14	1807	4.1	14.3
28	1803	5.2	17.7
56	1817	5.3	18.8
121	1818	5.6	21.0

Table 6: Density and mechanical properties of the hardened mortar prisms (BI-DT: mix BI with Duggan test)

Tabela 6: Vrednosti gostote in mehanskih lastnosti za otrdeli drobnozrnati beton (BI-DT: mešanica BI z Dugganovim testom)

Time interval t/d	ρ/(kg/m^3)	f_m/MPa	f_c/MPa
121	1810	6.0	22.5

Tables 4, 5 and **6** show the measured densities (ρ), flexural (f_m) and compressive strengths (f_c) of the hardened fine-grained concrete prisms for the mixes AI, BI and BI-DT. The compressive strength of the BI prisms, after 121 d, is by 6.6 % lower than that of the BI-DT prisms. A comparison of the results showed that the concrete with fly ash has a slightly lower initial strength and an increased final strength.[28]

4 CONCLUSIONS

Controlling DEF by using AEA as a nucleation agent results in a slight increase of the compressive strength of the fine-grained concrete. Small and thin crystals of ettringite, resulting from a series of chemical reactions that take place in the hydrated concrete, caused a swelling of the concrete. The local stress concentration at the nucleation sites, which are the air bubbles of an air-entraining agent where ettringite crystals grew, did not cause an extension of the microcracks that could lower the compressive strength of the concrete.

The ettringite-crystal growth in the porous parts of the walls of the air bubbles caused a change in the microstructure of the concrete. This change represents a transformation of the existing porous microstructure in line with the tiny crystals condensed in the microstructure.

The result of these changes in the microstructure of the nucleation sites is a reinforced cementitious matrix. The strength improvement is a result of hardening of the cementitious matrix, causing an increase in the compressive strength of the concrete.

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