

# Influence of Al-W-B Recycled Composite Material on the Properties of High Performance Concrete

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**Abstract** – The aim of this study is to obtain high performance boron containing material with sufficient carrying capacity with increased porosity and lower density at the same time. The influence of the different concentrations of Al-W-B powder on the properties of the fresh and hardened HPC was investigated. In the concrete mix design, the allite containing White Portland cement CEM I 52,5 R, granite stone, sand, microsilica, on polycarboxylates based super plasticizer and Al-W-B powder were used. As a source of boron composite material (CM), previously grinded powder containing boron-tungsten fiber and aluminium matrix (CM Al-W-B) was used. Grinding was used for processing of CM Al-W-B powder.

**Keywords** – Al-W-B, high performance concrete, recycling, X-ray analysis.

## I. INTRODUCTION

Concrete is the most widely used construction material in the world [1]. Its consumption is around 10 billion tons per year [2]. The fact that radiation could be harmful has led to the development of a wide variety of shields to protect against it [3]. For nuclear radiation shielding, a larger quantity of the shielding material is required; therefore, a study on the propagation of radiation flux in the shielding materials is an essential requirement for shield design [4].

Presently, heavy weight concrete is extensively used as a shield in nuclear plants and radiotherapy rooms, as well as for transporting and storing the radioactive wastes. For this purpose, ordinary used concrete must have high strength and high density. Such concrete usually contains steel, hematite, magnetite and barite aggregates. The use of these elements in the concrete can increase the density of the product up to 3 to 5 (ton/m<sup>3</sup>) and more. It results in significantly higher density as compared to concrete made by using ordinary aggregates (sand, crushed stone, etc.) [5], [6].

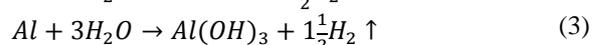
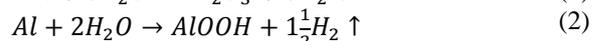
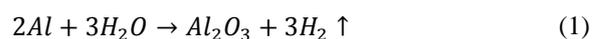
It has been found that it is not always possible to use heavy concrete in the building structures, especially in cases of tall (like skyscraper) or constructively complex buildings, because of possible deformations and collapse of built structure under disproportionately large load. Thus, in the framework of this work it was decided to create a concrete with the radiation shield properties, high strength and normal low density at the same time.

High performance concrete (HPC) is designed to ensure optimized performance characteristics for the given set of materials. The usage and the exposure conditions, consistent with the requirements of cost, service life and durability should be taken into account [7]. Architects, engineers, and

constructors all over the world have found that the application of HPC allows them to build more durable structures at comparable costs of HPC. HPC is in use in constructions in aggressive environments, marine structures, highway bridges and pavements, nuclear structures, tunnels, precast units, etc. [8], [9].

Aluminium matrix composites are lightweight materials with fine creep and fatigue behavior, as well as with the increased wear resistance as compared with fiber reinforced polymers. Moreover, the stiffness and the absolute strength of concrete can be significantly increased by composing with suitable reinforcement phases like particles, short fibers or continuous fibers. One of such composite materials (CM) is aluminium-tungsten-boron (Al-W-B). However, application and production of CM Al-W-B causes generation of industrial waste. The studied waste material is produced during the manufacturing process and application of finished materials (e.g. residues from cut). During the recycling process the hazardous prepreg of boron-tungsten fibers are produced. These fibers are thin, sharp and durable filamentous particles.

The studied Al-W-B contains Al as matrix in the alloy. When fine aluminium containing powder is added to water, H<sub>2</sub> gas generating reaction takes place. In addition to H<sub>2</sub> gas, such reactions can lead to the formation of alumina gel, boehmite (AlOOH or Al<sub>2</sub>O<sub>3</sub>·H<sub>2</sub>O) and/or gibbsite (Al(OH)<sub>3</sub> or Al<sub>2</sub>O<sub>3</sub>·3H<sub>2</sub>O) as following [10]:



Accordingly to Eq.(4), the standard free energy of reaction can be calculated from the standard-state free energies of formation as well. It is the sum of the free energies of formation of the products ( $\Delta G^\circ_{f,products}$ ) minus the sum of the free energies of formation of the reactants:

$$\Delta G^\circ = \sum \Delta G^\circ_{f,products} - \sum \Delta G^\circ_{f,reactants} \quad (4)$$

where  $\Delta G^\circ$  is the change in Gibbs free energy.

Eq. (5) is the calculation result of change in free energy of formation of alumina gel as shown in Eq. (1). Eqs. (5) and (7) shows the change in Gibbs free energy of formation of boehmite and gibbsite as shown in Eqs. (2) and (3). The value of Gibbs free energy is obtained from table of standard thermodynamic values published at Chemistry-Reference web page [11]:

$$\begin{aligned} \Delta G^\circ(Al_2O_3) &= \{-1499,25 + 3 \cdot (0)\} \\ &\quad - \{2 \cdot (0) + 3 \cdot (-237,18)\} \quad (5) \\ &= -787,71 \text{ kJ/mol} \end{aligned}$$

$$\begin{aligned} \Delta G^\circ(AlOOH) &= \{-2287,32 + 1\frac{1}{2} \cdot (0)\} \\ &\quad - \{(0) + 2 \cdot (-237,18)\} \quad (6) \\ &= -1812,96 \text{ kJ/mol} \end{aligned}$$

$$\begin{aligned} \Delta G^\circ(Al(OH)_3) &= \{-1305,83 + 1\frac{1}{2} \cdot (0)\} \\ &\quad - \{(0) + 3 \cdot (-237,18)\} \quad (7) \\ &= -594,29 \text{ kJ/mol} \end{aligned}$$

When change in Gibbs free energy is negative, chemical reactions are spontaneous (thermodynamically favourable). The shape the fine powder increases the reactivity of aluminium. However, the presence of a passive layer containing oxides and hydroxides at the surface prevents the reaction between water and aluminium. Therefore, the hydration reaction of pure aluminium cannot proceed completely in water without any additives.

The amorphous  $Al(OH)_3$  is known for its amphoteric characteristic, resolving in either strong acid [Eq. (8)] or alkali [Eq. (9)] solution as follows:

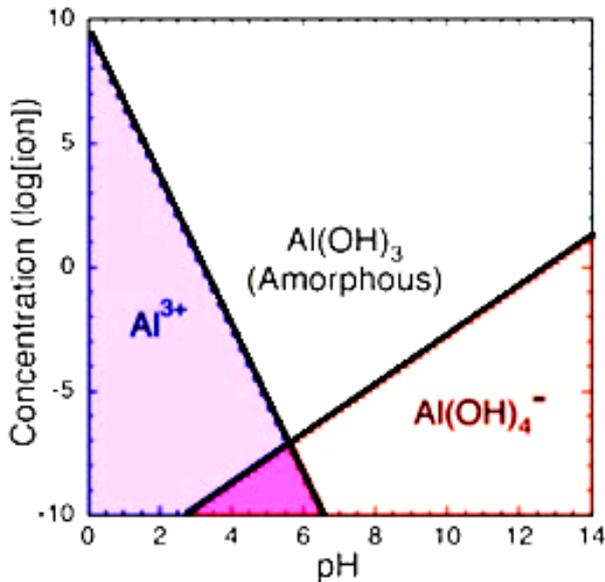
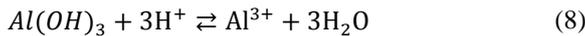
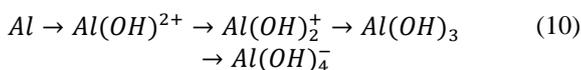


Fig. 1. Aluminium solubility diagram in equilibrium with amorphous  $Al(OH)_3$  in 25 °C [12].

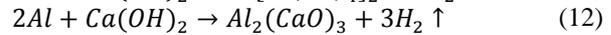
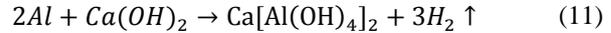
Fig. 1 shows the calculated aluminium solubility diagram in equilibrium with amorphous  $Al(OH)_3$  at 25 °C, based on equilibrium (8) and (9). The water molecules in the hydration shell are progressively replaced by hydroxyl ions, giving negative charge, according to the following sequence:



Hydrolysis scheme (10) would proceed from left to right as pH increased. The weight ratio of  $Ca(OH)_2$  is one of the

important factors to release the hydrogen gas from the corrosion reaction of aluminium powder [12], [13].

As shown in Eqs. (11) and (12), in water – Portland cement system (alkaline medium), aluminium also reacts with free calcium hydroxide  $Ca(OH)_2$  by generating  $H_2$  gas as follows [14]:



Calcium hydroxide acts as a catalyst to promote the hydrogenating reaction.

Reactions (1)–(3) and (11)–(12) can lead to de-watering process in fresh concrete. Thus the increase of aluminium containing powder concentration in wet Portland cement concrete can increase the necessary amount of water to provide flowability. Generated  $H_2$  gas can be responsible for formation of opening permeable channels and increased porosity in production of concrete.

Boron has two naturally occurring and stable isotopes,  $^{11}B$  (80.1 %) and  $^{10}B$  (19.9 %).  $^{10}B$  is often used low-energy neutrons absorbing element with low density at the same time. As it is difficult to obtain pure boron by using the common extraction methods, the use of boron containing composites is more economical and practical. In works [15]–[17] the effect of boron containing materials (such as natural mineral and recycled wastes) on the properties of concrete has been investigated. There is another point to consider that concrete, used in nuclear applications, must have adequate engineering properties (compressive strength, shrinkage, workability, tensile strength and modulus of elasticity) as main factors of importance in large stationary installations.

Preliminary tests of this study have shown that the addition of Al-W-B powder to a concrete mixture significantly reduces the mechanical strength of the concrete. In order to fully assess the effects of powder on the concrete structure, it was decided to use HPC as a reference concrete. In this study an optimal mix design of HPC from the authors' previous research work [18] was selected. The influence of the Al-W-B powder in different dosages on the properties of HPC was investigated. In the concrete mixtures Aalborg White Portland cement CEM I 52,5 R (WPC), granite stone, sand, microsilica, SP and Al-W-B powder in different dosages were used.

## II. MATERIALS AND METHODS

### A. Materials Used in Study

WPC produced at Aalborg cement factory (Denmark) was used in the given study. The chemical composition and physical properties of the WPC are summarized in Table 1. Morphology of CM Al-W-B particles was in the range from approximately 2  $\mu m$  to 100  $\mu m$  (see Fig. 3).

Two groups of aggregates with particle sizes of 0–1.8 mm (sand) and 0–5.0 mm (crushed granite stone) and a certain distribution of fraction (shown in Table II) were used for preparation of HPC. Maximum aggregate size was 5 mm. The specific gravities of the fine (sand) and coarse (crushed granite stone) aggregates were 2.64 and 2.70  $g/cm^3$ , respectively. Microsilica “Elkem-microsilica – 920 D” was used as

microfiller with high pozzolanic activity. Superplasticizer (SP) "Semflow MC", based on polycarboxylates (specification shown in

Table III), was used in HPC mixtures.

As a source of boron, CM Al-W-B was used. A method of disintegration was used for processing of CM Al-W-B powder as described in paper [19]. The grinding was carried out in several stages in order to obtain a powder with the determined particle size. Scanning Electron Microscopy (SEM) combined with EDS-Energy Dispersive X-Ray Spectroscopy was used to establish the composition of CM AL-W-B. It was found that the CM Al-W-B powder material consists of B - 47.05 %; Al - 43.26 %; W - 5.06 %; Mg - 2.78 %; Mn - 1.16 %, Fe - 0.63%. In this powder B and W were 99.9 pure, but an aluminium alloy matrix consists of Al (90.34 %), Mg (5.81 %), Mn (2.42 %), Fe (1.32 %). Resemble composition powder waste materials have been investigated and described in the previous works [19], [20]. Particle sizes of CM AL-W-B are in the range from approximately 2 μm to 100 μm (see Fig. 3).

TABLE I  
PROPERTIES OF WPC

Composition		
Component	Wt %	
CaO	68.9	
SiO <sub>2</sub>	24.8	
Al <sub>2</sub> O <sub>3</sub> + Fe <sub>2</sub> O <sub>3</sub>	2.20	
MgO	0.52	
SO <sub>3</sub>	2.20	
K <sub>2</sub> O + Na <sub>2</sub> O	0.27	
LOI	0.14	
Physical and mechanical properties		
Specific gravity (kg/cm <sup>3</sup> )	3150	
Setting time, initial (min)	120	
Compressive strength (MPa)	2 days	32
	7 days	54
	28 days	70

TABLE II  
SIEVING ANALYSIS OF MINERAL AGGREGATES

Sieve mesh size, mm	Sieved particles, mass %	
	Granite stone	Sand
8	100.0	100.0
5	68.0	100.0
3.15	53.0	100.0
2.24	50.0	100.0
1.18	40.0	100.0
0.6	38.0	72.0
0.3	25.0	43.0
0.150	7.0	15.0
0.075	4.0	5.0
0.063	3.0	1.0

TABLE III  
SPECIFICATION OF SP

Density	1.05 kg/dm <sup>3</sup> ± 10 g/dm <sup>3</sup>
Na <sub>2</sub> O equivalent	<2 %
Chloride content	<0.05 %
pH	7.0
Active component	Polycarboxylate ether
Overdose side effects	Curing delays, segregation
Dose	0.2–2.5 %

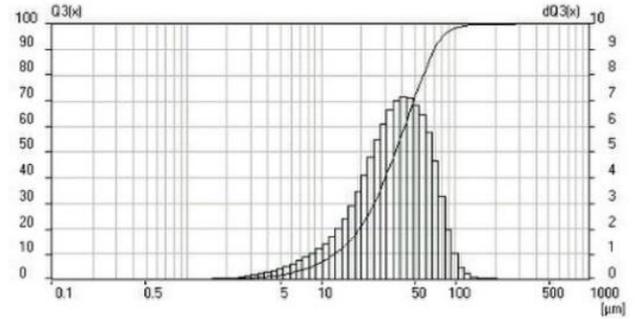


Fig. 2. Al-W-B powder particles size distribution histogram.

X-ray analysis of CM Al-W-B powder has shown the presence of aluminium, boron oxide and aluminium-iron composition (see Fig. 4). One signal (marked as X) was not understood in this research.

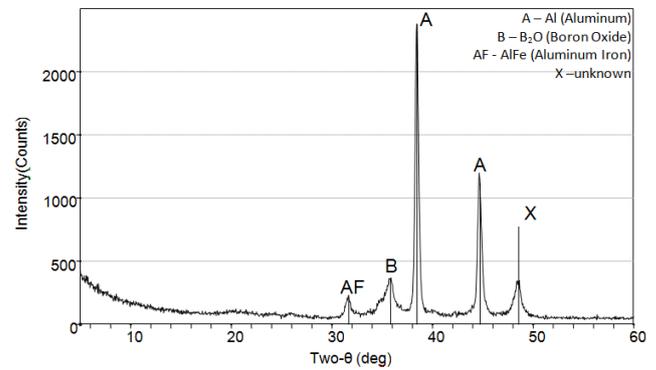


Fig. 3. Phase composition of Al-W-B powder.

**B. Methods**

Four groups of HPC samples were produced and compared. Recipes of HPC used in this study are summarized in Table IV. Due to fine particle sizes of Al-W-B, it was decided to replace only finer fraction (from 0.063 μm to 0.150 μm) of used granite and sand with Al-W-B.

SP was added to mixing water in the above mentioned ratios by weight of cement. Laboratory size paddle mixer was used for production of concrete. Concrete mixtures were prepared by using the following procedure: (1) the components of the mixture were mixed in dry state for 2 minutes; (2) a half of water was added to the mixture; (3) mixing was continued for 1 minute; (4) the second half of water with SP was added to the mixture; (5) mixing was continued for 2 minutes.

After mixing of fresh concrete, the obtained W/C ratio was recorded and flow tests were done. After completion of these tests, the fresh concrete was casted into molds without any vibrations and cured at the temperature of 20±2 °C and a relative humidity of 50–55 %. For each sample of HPC and HPCC, five (cubes with dimensions of 100x100x100 mm) specimens were cast. Additionally, to visually estimate the increase in volume after 1 hour from the moment of adding the water, three samples (prism with dimensions of 160x40x40 mm) for each mix design of HPCC were made.

The cubes of hardened concrete were used for determination of compressive and flexural strength. Fine powder of hardened HPC was prepared for X-ray phase analysis.

Hardened samples were taken out from the molds 24 h later and cured for 2, 7 and 28 days until the time of testing. The specimens were immersed in water at 20±2 °C until the test age.

The effects of SP on the workability and consistency of the fresh concrete mix design were measured using the flow cone test according to LVS EN 12350-2:2009. The compressive strength tests were performed by using “Controls-50-C4632” according to LVS EN 12390-3:2002. X-ray phase analysis was done using "Rigaku Ultima+" (Cu Kα).

The morphology of the hardened samples of concrete was investigated by using VHX-2000 optical microscope (Keyence Corporation) equipped with lens VH-Z500R/W. Images of surfaces were captured in 3D mode (large depth-of-field & super resolution imaging function). Constant illumination (3200 K), brightness and magnification of 500X were selected for objective comparison of the surfaces of specimens.

TABLE IV  
MIX DESIGN OF HPC AND HPCC'S

Material	Bulk density (kg/m <sup>3</sup> )	Dosage (kg/m <sup>3</sup> concrete)			
		HPC 2.5	HPCC 2.5-1	HPCC 2.5-2	HPCC 2.5-3
Cement CEM I 52.5 R	3150	650	650	650	650
Sand	2640	494	483	478	347
Granite stone	2800	3474	3400	3364	2442
Microsilica	2400	100	100	100	100
Super plasticizer	1050	16.25	16,25	16,25	16,25
Al-W-B	143	0	5	10	200

Note: "2.5" in designations means the amount of SP (2.5 % of cement weight).

### III. RESULTS AND DISCUSSION

#### A. Effect of SP on the Properties of Fresh Concrete

Results given in Table V show that the necessary W/C ratio should be increased to ensure similar flowability by increasing the concentration of Al-W-B powder in HPC. It was observed that the significantly increased concentration of Al-W-B (up to 200 kg/m<sup>3</sup> concrete) did not drastically change the necessary W/C ratio. It is possible that the balance of reaction between

Al powder and wet Portland cement takes place at very low concentration of used Al-W-B powder, leading to inhibition of reaction when higher amount of Al-W-B is applied. In further works it is planned to investigate this effect in more detail.

Cone tests of fresh concrete in all cases were symmetrical, indicating that the quality of mixed concrete is acceptable.

TABLE V  
THE EFFECT OF AL-W-B ON THE PROPERTIES OF FRESH CONCRETE

Sample	Obtained W/C ratio	Cone flow test (mm)	Accordance to SF class
HPC-2.5	0.28	285	SF2
HPCC-2.5-1	0.29	281	SF2
HPCC-2.5-2	0.30	283	SF2
HPC-2.5-3	0.31	281	SF2

Note: SF (cone flow test) classes are set in accordance with standard LVS EN 12350-2:2009

After 1 hour from the moment of water addition, it was visually observed that concrete with Al-W-B swells as shown in Fig. 5. An average expansion in volume was 2 % (HPCC 2.5-1), 4 % (HPCC 2.5-2) and 12 % (HPCC 2.5-3). Due to the friction between concrete surface and mould wall, an expansion was happening unequally by forming the round surface of the top edge.



Fig. 4. Swelling of HPCC 2.5-3.

#### B. Effect of SP on the Properties of Hardened Concrete

##### 1. Compressive strength

Results given in Table VI show that in general the addition of Al-W-B decreases the strength of HPC in all cases.

TABLE VI  
EFFECT OF AL-W-B ON COMPRESSIVE STRENGTH OF HPC

Sample	Compressive strength, MPa		
	2 days	7 days	28 days
HPC 2.5	88.3	94.9	115.8
HPCC 2.5-1	61.4	58.2	65.4
HPCC 2.5-2	56.4	68.6	67.3
HPCC 2.5-3	4.4	9.4	12.5

##### 2. X-ray analysis

In accordance with the result of X-ray analysis for three different dosages of Al-W-B (see Fig. 6), it was observed that:

1) Intensities of specific peaks for crystalline SiO<sub>2</sub> (quartz) overall decreases by increasing the amount of Al-W-

B. Some exceptions with higher intensities (e.g. at  $2\theta = 38$ ) can be explained with the formation of the crystalline compositions with a structure similar to  $\text{SiO}_2$ .

2) Intensities of the specific peaks for mineral portlandite (free  $\text{Ca}(\text{OH})_2$ ) overall decreases by increasing the concentration of Al-W-B. As in case of  $\text{SiO}_2$ , some exceptions can be explained with the formation of similar crystalline structures.

- Overall, the intensities for crystalline mineral tobermorite ( $\text{Ca}_5(\text{OH})_2\text{Si}_6\text{O}_{16}\cdot 4\text{H}_2\text{O}$ ) increase by increasing the concentration of Al-W-B. This effect can be explained with the reaction between free  $\text{Ca}(\text{OH})_2$ ,  $\text{SiO}_2$ , water and aluminosilicates from cement.

- Overall intensities for crystalline mineral svyatoslavite ( $\text{CaAl}_2\text{Si}_2\text{O}_8$  or  $\text{CAS}_2$ ) or calcium aluminodisilicate increase by increasing the concentration of Al-W-B. In this case it is possible that the free alumina ( $\text{Al}_2\text{O}_3$ ) or aluminium from the added Al-W-B powder stayed in reaction with free  $\text{SiO}_2$  (from microsilica),  $\text{Ca}(\text{OH})_2$  and cement minerals in concrete mixtures.

Some peaks in this research works are not defined by using the analytical software. Composites in this research can contain more minerals than shown in this X-ray analysis. There are at least two possible options – 1) crystalline composites with very similar structure exhibit low intensity peaks in x-ray analysis; 2) minerals are in glassy phase and cannot be determined by using this method.

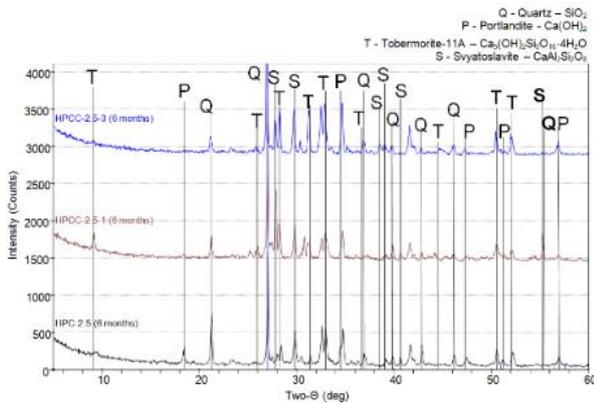


Fig. 5. The influence of Al-W-B dosage on mineral composition of HPC.

As shown in Fig. 7, X-ray analysis shows the influence of Al-W-B on crystalline composition of HPCC2.5-1 after different times of curing (3 days and 6 month). The overall intensities for specific peaks of portlandite ( $\text{Ca}(\text{OH})_2$ ), hatrurite ( $\text{CaSiO}_5$ ) and quartz ( $\text{SiO}_2$ ) decrease for longer cured concrete samples. It is a logical result of concrete hardening process in which original cement minerals create hydrated minerals in the presence of water.

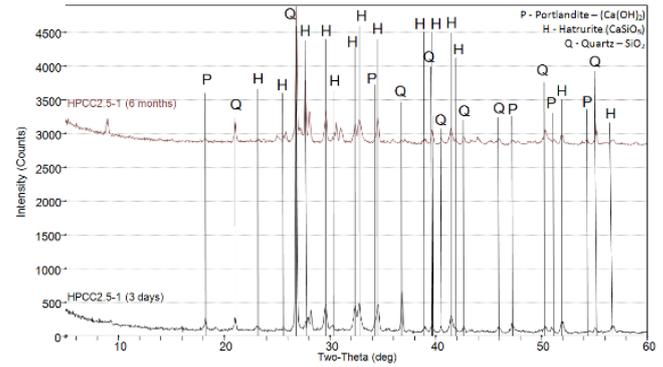


Fig. 6. The influence of Al-W-B on mineral composition of HPCC 2.5-2 depending on the time of curing.

### 3. Investigation of surfaces with optical microscope

As shown in Fig. 7, the roughness of concrete surface increases by adding the higher concentration of Al-W-B. No cracks on the surface of HPC 2.5 were observed by using the selected observation method (see Fig. 7a). Some coloured particles of granite were observed on this surface. Some micrometer sized cracks and remaining particles from Al-W-B powder were observed on the surface of HPCC 2.5-1 (see Fig. 7b). Roughness caused by micrometer size collapsed and remaining bubbles was observed on the surface of HPCC 2.5-2 (Fig. 7c). Surface of HPCC 2.5-3 (see Fig. 7d and Fig. 8) contains large (with diameter up to  $600\ \mu\text{m}$  and depth up to  $300\ \mu\text{m}$ ) opened pores as a result of bubble collapsing close to the surface of concrete.

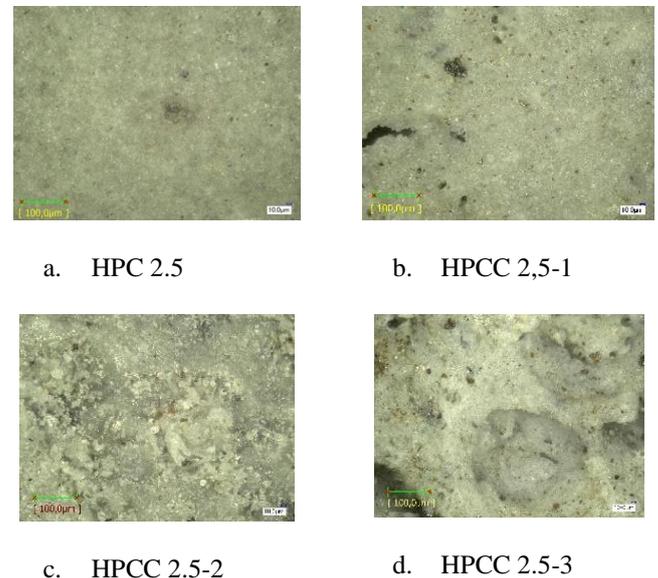


Fig. 7. Pictures of HPC and HPCC surfaces, obtained by optical microscope (500X).

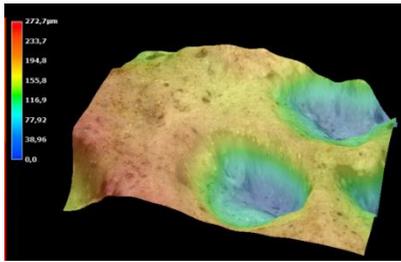


Fig. 8. 3D picture of HPCC 2.5-3 surface, obtained by optical microscope (500X).

#### IV. CONCLUSION

1) The necessary W/C ratio increases along with increasing the concentration of Al-W-B from 0 to 30 kg/m<sup>3</sup> concrete due to dewatering process caused by reaction between aluminium powder and wet cement.

2) Flowability of fresh concrete decreases by increasing the concentration of Al-W-B from 0 to 30 kg/m<sup>3</sup> concrete.

3) After 1 hour from water adding, the volume of fresh concrete increases by increasing the concentration of Al-W-B from ~0% (HPC 2.5) to 12% (HPCC 2.5-3). It was noticed that the reaction between aluminium and wet Portland cement generates H<sub>2</sub> gas leading to foaming (swelling) of concrete.

4) Generally, compressive strength of 28 days cured concrete decreases by increasing the concentration of Al-W-B from 15.83 MPa (HPC 2.5) to 12.52 MPa (HPCC 2.5-3).

5) X-ray analysis has shown that the concentration of mineral syvatoslavite increases by increasing the concentration of Al-W-B.

6) Investigation with optical microscope has shown that the surfaces of samples of HPCC are less smooth and are more contaminated with colored particles (by Al-W-B) than the samples of HPC.

7) Only the surface of HPCC 2.5-3 has clearly visible opened pores on unaffected (original) surface.

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