



Study on microstructure and bond strength of interfacial transition zone between cement paste and high-performance lightweight aggregates prepared from ferrochromium slag



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HIGHLIGHTS

- High-performance lightweight aggregates (HPLWA) are successfully fabricated by using ferrochromium slag, fly ash and bauxite as raw materials.
- The high-belite sulphoaluminate clinker is prepared and coated on the surface of HPLWA by one-step sintering technique to obtain an outstanding surface reactivity of aggregates.
- The ITZ properties including the hydration products and porosity profile were investigated by means of SEM and BSE imaging technique.
- The interfacial bond strength between HPLWA and cement paste is quantitatively analyzed by a three-point bending test.

ARTICLE INFO

Article history:

Received 8 June 2016

Received in revised form 22 February 2017

Accepted 10 March 2017

Available online 19 March 2017

Keywords:

Ferrochromium slag

High-performance lightweight aggregate (HPLWA)

Surface reactivity

Interfacial transition zone (ITZ)

ABSTRACT

In this study, high-performance lightweight aggregates (HPLWA) are successfully fabricated by using ferrochromium slag, fly ash and bauxite as raw materials. Besides, the high-belite sulphoaluminate clinker is prepared and coated on the surface of HPLWA by one-step sintering technique to obtain an outstanding surface reactivity of aggregates. The results show that the prepared HPLWA are made up of two parts, the inner porous ceramics which consist mainly of cordierite and spinel and the outer modified layers composed of belite and sulphoaluminate. The inner porous ceramics afford the HPLWA high compressive strength more than 6 MPa and low water absorption less than 2 wt%. Furthermore, the outer modified layers would weaken the compressive strength and increase the water absorption of HPLWA, but they endow a dense microstructure and high bond strength of interfacial transition zone (ITZ) between HPLWA and cement paste.

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1. Introduction

The concept of high-performance lightweight aggregates (HPLWA) was first proposed by Japanese scholars in 1990s to meet the needs of pumpable and castable lightweight aggregate concrete (LWAC) in engineering and construction practice. Characteristics required for HPLWA are lightweightness, high strength and low water absorption capacity [1]. HPLWA has a potential use in reducing constructing technical limits for ultra-high-rise buildings, long span structures and improving their seismic behavior [2,3]. However, there are not as much reports about the HPLWA as expected both in China and abroad. In fact, there are mainly three urgent problems facing the researchers or technical workers in the

preparation and application process of HPLWA, that is: 1) The utilized raw materials for preparing lightweight aggregates (LWA) are changing from natural raw materials such as clay, expanded shale and tuff [4–6] to industrial byproducts to relieve the growing shortage of natural materials and the increasing pollution of environment. These byproducts include fly ash, sewage sludge, incinerator bottom ash, various slags and so on [7–10]; 2) Currently, rapid cooling is the most common way of cooling in the preparation of LWA, which often brings the micro cracks inside LWA and weakens its mechanical and physical properties [11]. Accordingly, to meet the high strength and low water absorption characteristics of HPLWA, the raw material compositions and sintering process are needed to be elaborately considered. For instance, Markus [12] pointed out in his study that different heat treatments and cooling rates would result in the changes of strength and microstructure of LWA. Cheeseman [13] indicated that different sintering

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temperatures combined with various holding times may change the pore structures in the outer shell, and a closed pore structure would significantly reduce the water absorption of LWA. Hu [14] has proposed that lightweight aggregates consisting of cordierite ($2\text{MgO}\cdot 2\text{Al}_2\text{O}_3\cdot 5\text{SiO}_2$) phase present excellent thermal-shock resistance and maintain a high compressive strength in the repaid cooling process. 3) The further surface modification or pretreatment of LWA should be taken into account since the workability and interfacial transition zone (ITZ) properties of LWAC depend largely on the nature of aggregates [15,16]. It has reached a consensus that the pre-wetting treatment of LWA would not only improve the workability of fresh concrete but also provide internal curing for hardened concretes. Furthermore, Kong et al. [17] developed a novel method to further enhance ITZ of recycled aggregate concrete by surface-coating of pozzolanic materials, such as fly ash, slag and silica fume around recycled aggregates. Morin et al. [18] has proposed that thin polymer coatings on aggregates reveals a significant effect on concrete cracking behavior and increase its fracture energy and characteristic length.

The interfacial transition zone (ITZ) has long been considered as a zone of weakness in concrete composite, in terms of both strength and the permeation of fluids. It is generally accepted that the ITZ between the aggregate and the cement paste has a complex structure; large flat $\text{Ca}(\text{OH})_2$ crystals form within this structure, perpendicular to the surface of the aggregate grains, which results in the formation of a highly porous structure in the ITZ, due to the accumulation of water films close to the aggregate surface [19,20]. In order to fully understand this porous ITZ and its role in the concrete composite, many techniques have been introduced to study the properties of the ITZ. These techniques include the backscattered electron (BSE) imaging technique [21], nanoindentation testing [22], spectrum analysis [23], scanning electron microscope (SEM) [24], X-ray diffraction (XRD) [25] and so on, among which the nanoindentation testing is accepted as the most direct and efficient approach to study the microstructure of the ITZ. The BSE imaging technique is also a mature method to study the pore structure of the ITZ currently. Furthermore, the interfacial bond strength between the normal aggregate and the cement paste can also be tested by means of compressive strength, tensile strength or bending strength test [26–28]. However, there are few reports about the quantitative test of interfacial bond strength between the LWA and the cement paste.

In this study, HPLWA were prepared by using the ferrochromium slag, a typical industry waste generated during the manufacture of the stainless steel [29], as the main raw material. The cordierite phase was also introduced into this system to improve the mechanical properties of the prepared HPLWA. Besides, a modified layer (also known as high-belite sulphoaluminate clinker) was designed and coated on the surface of the HPLWA to realize its further surface modification. The ITZ properties including the hydration products and porosity profile were investigated by means of SEM and BSE imaging technique. Furthermore, the interfacial bond strength between HPLWA and cement paste is also quantitatively analyzed by a three-point bending test.

2. Experimental procedures

2.1. Materials

The utilized raw materials for fabricating HPLWA were ferrochromium slag (Leshan, Sichuan Province, China), commercial available fly ash and bauxite. Besides, the limestone, clay, anhydrite and bauxite were used for the preparation of high-belite sulphoaluminate clinker used as the outer modifying layer. Additionally, ordinary Portland cement (namely 42.5 grade) conforming to Chinese standard GB175-1999 was used for further study of the microstructure and bonding strength of ITZ between HPLWA and cement paste.

The chemical compositions of raw materials aforesaid are measured by quantitative X-ray fluorescence spectrum analysis (XRF; Axios-Advanced, PANalytical Corporation, The Netherlands), and listed in Table 1. It can be noted that the ferrochromium slag mainly consists of silicon (Si), magnesium (Mg), and aluminium (Al), which are similar with the chemical compositions of cordierite ($2\text{MgO}\cdot 2\text{Al}_2\text{O}_3\cdot 5\text{SiO}_2$). So it would be appropriate to use ferrochromium slag as main raw material to synthesize cordierite by adding some other materials being rich in Si and Al. In this study fly ash and bauxite have been chosen as the additional materials. The main mineral compositions of ferrochromium slag, fly ash and bauxite are also analyzed by X-ray diffraction (XRD, X'Pert Pro, PANalytical Corporation, The Netherlands), and displayed in Fig. 1. It shows that the ferrochromium slag mainly consists of forsterite, hercynite and also small amount of enstatite and unreacted chromium ore (Fig. 1a); the fly ash mainly consists of quartz, mullite and a small quantity of calcite (Fig. 1b); the bauxite consists of aluminium oxide, mullite, rutile and also small amount of aluminium phosphate (Fig. 1c).

2.2. Sample preparation

Based on the stoichiometric composition of cordierite ($2\text{MgO}\cdot 2\text{Al}_2\text{O}_3\cdot 5\text{SiO}_2$), a series of ferrochromium slag (55.00 wt%), fly ash (33.75 wt%), bauxite powder (11.25 wt%), and extra 5 wt% powdered carbon used as expansion agent were weighted and mixed in a cement mortar mixing machine with a controlled amount of water (about 22 wt%). The mixture was then rubbed to 10–15 mm green pellets by hands. Additionally, the outer modifying layer comprising of 62.11 wt% limestone, 29.37 wt% clay, 5.77 wt% anhydrite and 2.75 wt% bauxite was prepared and coated on the surface of green pellets using water as binding agent. The mass ratio between outer modifying layer and inner pellets is 0.24 to 1.00. The pellets as well as modified pellets were subsequently dried at 105 ± 2 °C in a baking oven in order to remove the free water, and then sintered for 30 min in a muffle furnace at temperatures ranging from 1250–1300 °C. The sintered pellets firstly cooled in the furnace to a temperature of about 800 °C, and then rapid cooled in the air to room temperature to avoid the known crystal transforming of $\beta\text{-C}_2\text{S}$ [30]. The heating rate was fixed at 5 °C/min, and a dwell time for 20 min at tem-

Table 1
Chemical composition of raw materials measured by quantitative XRF.

Materials	Chemical composition (wt%)										
	SiO ₂	CaO	Al ₂ O ₃	Fe ₂ O ₃	MgO	Cr ₂ O ₃	TiO ₂	SO ₃	MnO	Na ₂ O + K ₂ O	Loss
Ferrochromium slag	35.12	1.57	22.21	4.21	27.77	7.37	0.73	0.46	0.23	0.15	/
Commercial alumina	0.04	/	99.6	0.03	/	/	/	/	/	0.26	/
Silica powder	99.9	/	0.01	0.04	/	/	/	/	/	/	/
Clay	54.98	0.01	21.69	7.09	1.92	0.03	0.91	0.03	0.05	3.50	9.62
Limestone	0.29	61.83	0.09	0.11	0.19	/	/	0.02	/	0.01	37.41
Anhydrite	4.17	36.45	1.11	0.84	4.94	/	/	44.67	/	0.32	6.93
Bauxite	24.00	/	69.61	2.15	0.05	0.05	3.20	/	/	0.18	0.20

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