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Improved compressive strength of alkali activated slag upon heating

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ABSTRACT

This paper presents a study on thermal stability of alkali activated slag (AAS) prepared from electric arc furnace slag (EAFS) using a mixture of alkaline sodium hydroxide and sodium silicate solutions. The samples were investigated by means of XRD, SEM, TG/DTA and porosity analysis. Compressive strengths of AAS samples before and after exposure to elevated temperatures ranging from 600 °C to 1000 °C were determined. The significant microstructural modifications highlighted by changes in porosity due to the sintering process are responsible for the strengthening of AAS sample after heating at 600,800 and 1000 °C. Moreover, in AAS sample after heating to and above 600 °C wüstite transforms to spinel.

1. Introduction

EAFS is non-hazardous solid waste generated during the iron and steel scrap remelting in electric arc furnaces. Growing global steel demand and increasing share of steel production in electric arc furnaces impose the necessity of finding the appropriate route for EAFS valorization. Currently, this slag is considered as a possible additive in the production of asphalt mixtures [1], or for conventional concrete production [2,3]. Moreover, the silica and alumina content renders the EAFS an attractive source material for the alkaline activation process. This process involves a chemical reaction between oxides of silicon, aluminum and highly alkaline activator yielding a new material with amorphous or semi-amorphous structure, called alkali activated cement [4]. This reaction product of slag alkaline activation is a calcium silicate hydrate (C–S–H) gel, which is similar to the reaction product of the cement hydration process. It is highly amorphous and characterized by a high degree of aluminum substitution for silicon [5] which results in the formation of calcium (alumino) silicate hydrate (C–(A)–S–H) gel [6]. This is the main difference between the reaction products of the cement hydration process and the alkaline activation of slag. The structure of C–(A)–S–H gel is strongly dependent on the type of alkali activator, which greatly affects the properties of AAS [7].

The understanding of the AAS behavior, when exposed to an aggressive environment, is critical in the prediction of the material life time and its behavior during exploitation. Hence, the aim of this paper was to investigate the AAS performances at high temperatures.

2. Experiments

The EAFS used in this investigation was supplied from the Steel Mill Nikšić in Montenegro. The mean particle size of the grinded slag was $d_{50}=24\ \mu m$ and its chemical compositions in terms of main oxides were: 46.5% CaO; 23.5% FeO; 12.2 SiO_2; 6.5% MgO; and 7.24% Al_2O_3. The slag was alkali activated at solid to liquid mass ratio of 4. The alkali activator was prepared by mixing sodium water glass (molar ratio $SiO_2/Na_2O=3.2$) and 10 M NaOH solution, in the mass ratio of 2. The obtained paste was cast in a cylindrical plastic mold, oven cured for 48 h at 65 °C. After demolding, the samples were cured an additional 14 days at ambient temperature.

Subsequently, the AAS was subjected to heating at 600, 800 and 1000 °C. As soon as the target temperature was attained, it was maintained for an additional 60 min before the furnace was shut down to allow the specimens in the furnace to cool down to room temperature. All samples (including control samples) were tested for compressive strength using a universal testing machine according to MEST EN 1354:2011 standard and subjected to porosity investigations using an ASAP 2020 instrument. A pressure

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transducer in the range of 0–1000 mm Hg ($\pm$ 0.15% accuracy) was used to monitor the vapor pressure in the system. X-ray diffraction (XRD) patterns of EAFS and AAS were obtained on a Philips PW 1710 diffractometer using monochromatized CuKα radiation (λ = 1.54178 Å) and step-scan mode (2θ range was from 4° to 90° 2θ, step 0.02° 2θ, time 0.8 s). Scanning electron microscopy (SEM) analysis of AAS was examined using a FEI 235 DB focused ion beam system, equipped with the EDAX (Energy-dispersive X-ray spectroscopy) energy dispersive spectrometer (EDS). TG/DTA measurements were performed in air atmosfere using a TA-SDT 2060 instrument. Approximately 20 mg of the sample was crushed and placed in a platinum crucible and heated to 1200 °C at a constant heating rate of 15 °C/min.

3. Results and discussion

Microstructure of the AAS shown in Fig. 1a is characterized by the presence of unreacted slag particles (denoted by A) partially covered and bonded in the reaction product of the alkaline activation of slag (denoted by B). The EDAX findings of B region (Fig. 1b) reveal the presence of C–(A)–S–H phase as a main reaction product of alkaline activation because of the high Si and Ca content and much lower Al content, [8]. Moreover, the low Ca/Si ratio of 0.97, typical for AAS, was observed [7].

The results of XRD analysis (Fig. 2) have shown that EAFS and AAS before annealing are similar and mainly X-ray amorphous. The main crystal phase is wüstite ($Fe_{1-x}O$), followed by larnit, β-Ca_2SiO_4, and gehlenite, $Ca_2Al(AlSiO_7)$. There is a possibility that small amounts of iron in AAS, but because it is a very small amount its unambiguous identification based on XRD measurements was not possible. In the thermally treated sample, besides larnite and gehlenite, a new spinel phase can be observed, suggesting that wüstite, $Fe_{1-x}O_4$, was transformed to magnetite, Fe_3O_4, during thermal treatment.

The results of thermal analysis are shown in Fig. 3. The TGA/DTA curves of EAFS indicate that above 600 °C phase transformation of wüstite into magnetite occurs, which is accompanied by mass increase and appearance of an endothermic peak on the DTA curve (Fig. 3a). The mass increase is attributed to the oxidation of Fe(II) to Fe(III) during the heating in air atmosphere. AAS shows typical thermal behavior for alkali activated materials upon heating (Fig. 3b).

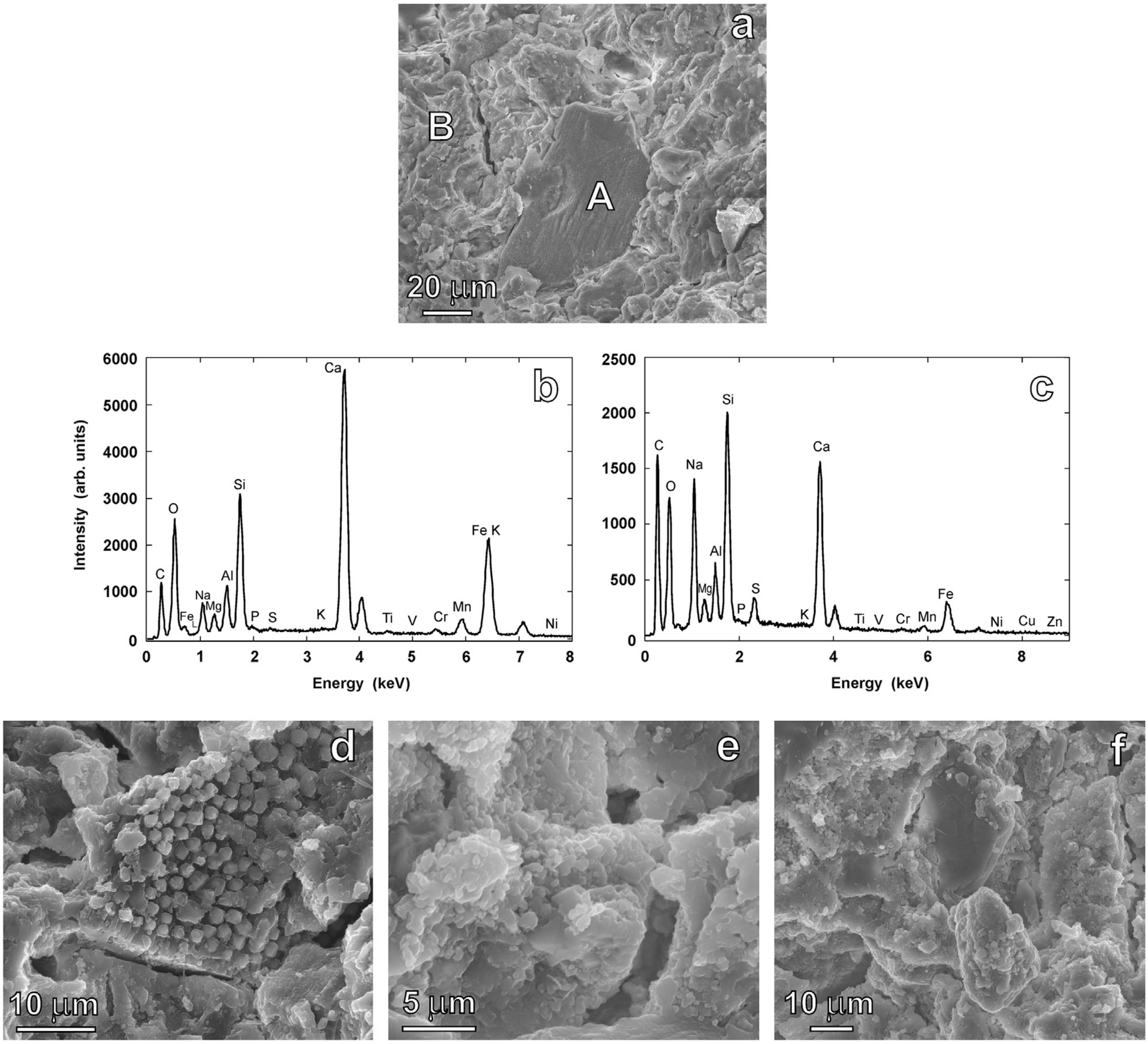

Fig. 1. SEM-EDS of AAS (a–c) before and (d–f) after anealing.