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A study on mechanical and physical properties of monocalcium aluminate cement reinforced with nano-SiO_2 particles

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ABSTRACT

In this research the formation of monocalcium aluminate cement, for nanocomposite base, has been investigated using high energy ball milling followed by annealing. Then the influence of nano-SiO_2 particles-reinforcement on properties of hardened cement paste has been studied. The results showed that when a small amount of nano-SiO_2 particles (1 wt.%) are uniformly dispersed in the cement paste, the hydration process is improved and the mechanical and physical properties of the cement paste is better than that the plain cement. By further addition of nanoparticles, these properties decline slightly. The samples were also examined by X-Ray diffraction and scanning electron microscopy, to study the phase analysis and microstructure during the hydration process.

1. Introduction

In recent years, nanotechnology in cement and concrete has focused on the investigation of the structure of cement based materials with nanoparticle additives and their properties [1]. For better understanding of the structure of nanocomposite cement, the hydration process and mechanical and physical properties of cements have been investigated. There are many research works on the influence of nanoparticles on some properties of cement. Recent investigations showed that nano-SiO_2 in cement improved its workability and strength [2,3]. The effect of nano-TiO_2 was to improve the self-cleaning properties of cement and concrete helping to clean the environment [4,5]. Nano-Al_2O_3 increased the modulus of elasticity of cements [6] and also the effect of nano-Fe_2O_3 was to provide concrete with self-sensing capabilities as well as to improve its compressive and flexural strengths [2,7].

Monocalcium aluminate, $CaAl_2O_4$, is the main constituent of calcium aluminate cements. It is due to the special structure of this ceramic that it has its unique properties such as high strength and high temperature resistance [8] and that it has found a wide range of applications in the construction industry and, more recently, in optical, bio, and structural ceramics [9–11].

Calcium aluminate cements are suitable for making various composites with improved properties. Yi et al. [12] produced $CaO–Al_2O_3–TiB_2$ composites by the combustion synthesis technique. These materials have matrices based on binary calcium-aluminate compounds. Also, Jonas et al. [13] studied Thermal expansion properties of $CaAl_4O_7$ based refractory compositions containing MgO and CaO additions.

According to the effects of nanoparticles on cements and their properties, it seems that calcium aluminate cement can be suitable for making various nanocomposites with nanoreinforcement additives and their workability can be improved. No research work was found on the influence of nanoparticles on calcium aluminate cements. So the aim of this work is to study the effect of nano-SiO_2 particles on physical and mechanical properties of monocalcium aluminate.

2. Experimental

2.1. Raw materials

The starting materials used for $CaAl_2O_4$ formation were reactive alumina from Lafarge Company with 99.8% purity and calcium oxide from Merck Company with 99.99% purity. Moreover, nano-SiO_2 particles with a particle size of below 20 nm and 99.8% purity were prepared by Cosmos Plastics and Chemicals Company as reinforcement for nanocomposite.

2.2. Preparation of monocalcium aluminate cement base

For the formation of $CaAl_2O_4$ phase, Al_2O_3 and CaO were mixed with Al_2O_3/CaO = 1/1 mole ratio and milled for 100 h. The milling operation was carried out in a planetary ball milling system in air, at room temperature. The milling speed was set at 500 rpm and ball to powder weight ratio was 10/1. The milled powder was calcined for 2 h in 1000 °C in air atmosphere in Nabertherm

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GmbH furnace. The formation of $CaAl_2O_4$ phase was characterized using a Philips X'PERT MPD X-ray diffractometer (XRD).

2.3. Preparation of paste specimen

Nano-SiO_2 particles were added to the obtained $CaAl_2O_4$ powder in 0, 0.5, 1, 1.5, 2 wt.%, then mixed with the required amounts of water ($W_{H_2O}/W_{powder} = 0.3/1$ weight ratio) for 4 s. The mixing operation of powder and water was carried out by a digital amalgamator (D-1697-4 model) with 50 Hz frequency. The homogen paste was then cast into cylindrical aluminium molds with diameter and height equal to 1 cm, according to ASTM C1133-97 for compressive test, and vibrated on a vibrating table. The hydrated samples were left in their molds for 24 h in a 100% relative humidity condition. Then the samples were demolded and kept under water for 7 days, as the most important compressive strength development is reached during the first 7 days [14].

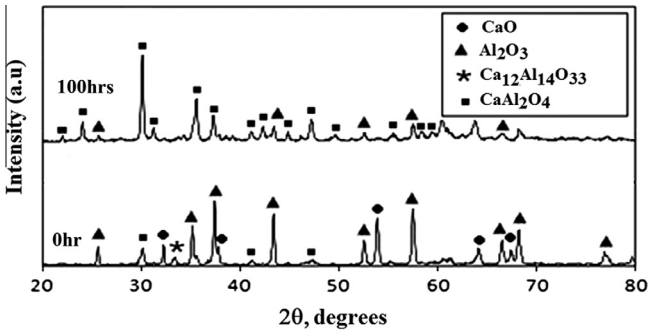

Fig. 1. XRD patterns for unmilled and 100 h milled samples annealed at 1000 °C.

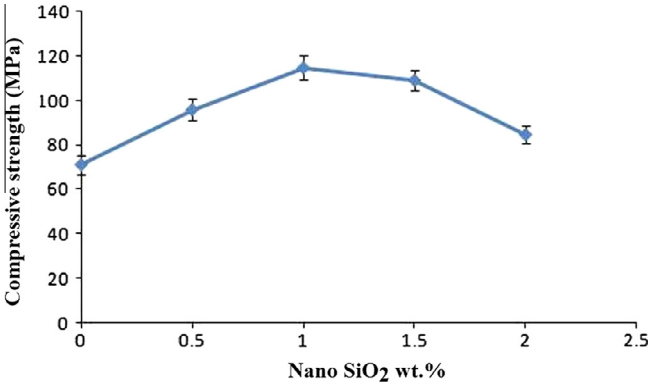

Fig. 2. Compressive strength of hydrated samples with 0, 0.5, 1, 1.5, 2 wt.% nano-SiO_2 particles.

2.4. Test methods

The hydrated samples were tested for certain mechanical and physical properties. Compressive strength of samples was determined by the instrument (Hounsfield, H25 KS model). Five specimens were tested for each age.

Bulk density and apparent porosity were also determined according to ASTM C0020. Fracture toughness was determined by nanoindentation (CSM instrument, CPX) that was equipped with atomic force microscopy (AFM, CSM Bruker). Indentation testing is commonly used for evaluating material toughness, and shows the fracture resistance to the scale of crack pattern. To determine the fracture toughness of samples, K_{IC}, Eq. (1) proposed by Oliver and Pharr [15] was used.

$$K_{IC} = a^*(E_{IT}/H_{IT})^{1/2}(F/c^{*3/2}) \quad (1)$$

where E_{IT} and H_{IT} are the Young's modulus and the hardness of the test material, which are obtained by nanoindentation test, equipped with Indentation 4.06 software, F is the indentation load, a^* is the empirical constant and c^* is the half length of the resultant indentation crack, which was obtained by AFM test equipped with Image Pro Plus software.

Finally, the formation of hydrated phases was characterized by XRD and the microstructure of hydrated samples was investigated by scanning electron microscopy (SEM, Philips XL30).

3. Results and discussion

3.1. Monocalcium aluminate formation

Fig. 1 shows the XRD pattern for unmilled and 100 h milled starting powder mixture annealed at 1000 °C for 2 h. In the unmilled mixture the amount of $CaAl_2O_4$ phase is low but when the mixture is milled for 100 h, a high level of strain energy is supplied and when the sample is calcined, the activation energy needed to form $CaAl_2O_4$ is provided and the $CaAl_2O_4$ single phase is obtained. The secondary phases such as $Ca_{12}Al_{14}O_{33}$ were initially formed but because of 1:1 mole ratio mixture of CaO and Al_2O_3, after prolonged reaction time, only the $CaAl_2O_4$ phase remained and secondary phases were converted to $CaAl_2O_4$. In this research $CaAl_2O_4$ was formed at a temperature 400–450 °C lower than the temperature usually used in the traditional solid state methods. $CaAl_2O_4$ single phase that was obtained from these processes was used as the base matrix of nanocomposite.

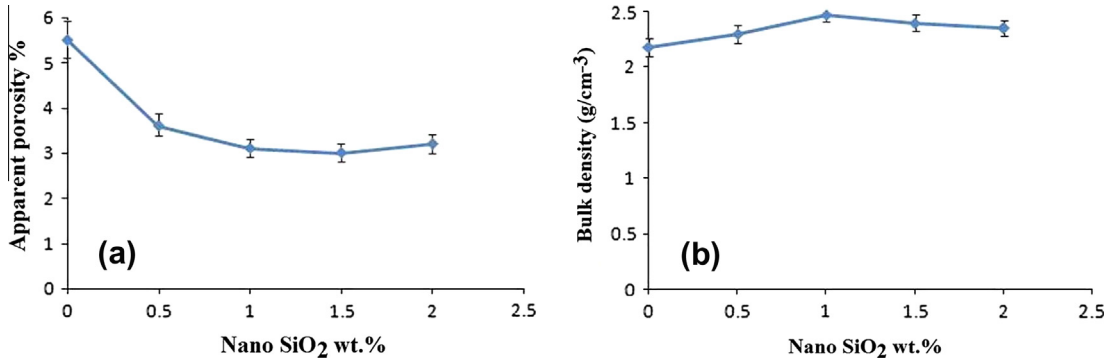

Fig. 3. (a) Apparent porosity and (b) bulk density of hydrated samples.