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Mechanochemically synthesized high alumina cement and their implementation as low cement castables with some micro-fine additives

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ABSTRACT

High-energy ball milling viz. mechanochemical process is being utilized to mechanically activate ceramic powders for low temperature solid state reactions. The process can help to select low-cost commercially available oxides and can produce powders with nanometer size granules. On the other hand, high alumina cement provides high service temperature when used as refractory castable. Therefore, the effects of high-energy ball milling and subsequent calcinations on the formation of high alumina cementing phases using mixtures of Al_2O_3 and $CaCO_3$ were investigated. Nano-meter sized high alumina cement (HAC) powders were synthesized by mechanochemical treatment of Al_2O_3 and $CaCO_3$ in weight ratios 7:3 and 8:2. This paper compares the calcined high alumina cement obtained by mechanically activated precursor mix for 1, 2 and 3 h. Low cement castables were prepared from calcined Chinese bauxite as aggregate matrix, prepared HAC acting as hydraulic binder and micro-fine additives as pore filling agents. The bonding of high alumina cement as well as sinterability in these castable was studied with ZrO_2, α-Al_2O_3 and SiC as micro-fine additives. Castables formulated by prepared high alumina cement demonstrate remarkably improved bulk density and apparent porosity as when compared with those prepared by commercially available cement. Casting water demand was also reduced, as a result quick setting behavior was observed. The addition of mechanochemically processed cements in refractory castables improved the thermo-mechanical properties to a significant extent.

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1. Introduction

High alumina cements (HACs) or calcium aluminate cements (CACs) are the only options as a refractory cements due to their high refractoriness and are most suitable for high temperature applications [1–4]. Their hydraulic strength development is due to water bonding reactions of the calcium aluminates to form a water-resistant hydrated phases. It is a cold hydraulic bonding system. The alumina content in high performance HAC exceeds 70% Al_2O_3 and remaining is mainly CaO content. High performance concretes are possible from calcium aluminate cements and also, ultra-high strength concretes have been proposed [5–13]. The flexural strength of macro-defect-free (MDF) concrete samples based on high alumina cement show much higher values [14–16]. The difference in refractoriness between HAC and Portland cement is due to the presence of C_2S and C_3S as the main constituents of Ordinary Portland Cement (OPC), which have low eutectic points. While HAC on the other hand with high Al_2O_3 (low CaO and SiO_2) possesses a high melting point and is used as refractory cement. CA, CA_2, CA_6 and $C_{12}A_7$ are the main constituents in HAC. In addition to CA ($CaAl_2O_4$), the HAC contains major amounts of CA_2 ($CaAl_4O_7$), $C_{12}A_7$ ($Ca_{12}Al_{14}O_{33}$) phases and minor amount of un-reacted alumina. Very little amounts of C_3A ($Ca_3Al_2O_6$) is observed when samples are heated above 1500 °C. The amount of the Ca-rich phase $C_{12}A_7$ is found to decrease with time as it reacts with alumina to form CA_2 or CA whereas, the amount of CA_2 formed decreases comparatively slowly with time. HAC is used as binding materials for monolithic applications and a significant advancement in monolithic technology is the development of refractory concretes or castables [17–19]. Castables, a type of monolith, are complex refractory formulations, requiring high-quality precision-sized aggregates, modifying fillers, binders, and additives. The use of reduced cement contents in monoliths such as low cement castables and ultra-low cement castables has grown significantly over the past few years. They may be cast in molds to form specific products (pre-cast shapes) or cast “in place”, as when forming a lining for a kiln furnace.

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The main technical advantages of low cement castables (LCCs) and ultra-low cement castables (ULCCs) are their excellent physical properties, such as high density, low porosity, high cold/hot strengths, high abrasion and corrosion resistance. The working life of HAC in steelmaking and other ceramic industries is greatly dependent on the material's ability to withstand high temperatures without undergoing significant deformation and corrosion. Therefore, one of the approaches used throughout the later decades is to improve the performance of HAC by reduction of the liquid content formed at elevated temperatures on high-alumina refractory castables [6]. Low-melting point eutectic phases are often formed in these castables because of the reaction between Al_2O_3, SiO_2, and CaO. Outstanding gains in refractoriness have been obtained through the reduction of the amount of CaO, SiO_2 and increasing the Al_2O_3 content in the HAC. Conventionally, high alumina cements are obtained by fusing or sintering a mixture of suitable proportions of argillaceous and calcareous materials such as CaO or $CaCO_3$ and alumina (Al_2O_3) at temperatures above 1500 °C and subsequent grinding. The resultant product is a fine powder and typically has very low specific surface area (<1 m^2/g). The completion of such reactions depends on the particle size, specific surface area and the mixing of the reactant powders. Even after repeated firing grinding cycles to eliminate all of the unreacted materials, the product batch frequently contains unreacted starting materials. Recently, mechanochemical reaction has emerged as a variable method to produce nano scale materials. This technique generally influences texture and structure leading to a decrease of the particle size and simultaneously increases the micro strain due to the contribution of the grain boundaries formed during the process [20]. Mechanical activation can also be employed in order to improve the kinetics of adsorption, catalysis and mineral synthesis, as well as to customize the mineral surfaces with respect to structure and composition. Modifying the structure, composition and reactivity of the mineral surface gives rise to new applications and products [21]. It has been reported by many researchers that the mechanochemical synthesis processing can be designed in such a way as to synthesize nano-crystalline particles dispersed within a soluble salt matrix. The chemical precursors react, either during milling or in the subsequent heat treatment stage [22–29]. In the present work, a high-energy ball milling process has been applied to the mixtures of Al_2O_3 and $CaCO_3$. The reaction between Al_2O_3 and $CaCO_3$ toward cementing phase formation is obtained at much lower temperatures (~1000 °C). High energy ball milling process has a significant effect to obtain high reactivity of mixes prepared by commercial raw materials. The reaction of the oxides is initiated at temperatures as low as 900 °C. Two compositions of calcium aluminate powders containing 70 and 80 wt.% Al_2O_3 were selected. The prepared cements were characterized for their structural, mechanical and cementing properties. Finally, the calcinations temperature and time were optimized to obtain desired phases in the HAC. The effect of high-energy ball milling on the fineness of the mixtures, microstructure development of the milled powders, cementing properties and phase formation was investigated. The prime cementing phases observed, were CA, CA_2 and CA_6. Furthermore, low cement castables were prepared from calcined Chinese bauxite, prepared HAC and micro-fine additives. Here we use α-alumina, zirconia and silicon carbide as additives in castable matrix. α-Alumina is most stable and technically useful crystalline form. α-Alumina has a crystal structure based on hexagonal close packing of oxygen ions with aluminum ions occupying two out of every those octahedral voids. Inter atomic bonding though generally taken to be ionic, has significant covalent component. As a result commercial alumina is difficult to sinter and requires high temperature above 1700 °C for any significant densification. It is therefore, necessary to obtain alumina in a reactive form with submicron particle size to carryout sintering at temperature of 1550 °C. Zirconia is considered very high temperature sustaining engineering material because it shows good chemical stability, high compressive strength, good fracture strength at high temperature and low thermal expansion coefficient. This complex refractory formulation was chosen, keeping in mind the severe operating conditions of secondary steelmaking and ever demanding clean steel process requirements. Alumina-zirconia based monoliths are being used in sliding gate plates, submerged entry nozzles and ladle shrouds. Due to continuous casting operations these complex refractory shapes suffer degradation due to high abrasion. Only alumina-zirconia based materials are found to survive such operations. Carbon is sometimes added to prevent wettability from molten metal but it has very low oxidation resistance. So, submicron sized silicon carbide was used in the present work to deliver similar performance. Silicon carbide is very hard material and its addition improves thermal shock which is very much required in sub-entry nozzles. It also gets oxidized at temperatures higher than 1400 °C, therefore its addition was kept to only 1 wt.% in all castable matrixes. In the current work, X-ray diffraction results confirmed several crystalline calcium-aluminate phases. Physico-mechanical properties such as apparent porosity (AP), bulk density (BD), hot modulus of rupture (HMOR), cold modulus of rupture (CMOR) and cold crushing strength (CCS) of sintered castables were studied. The sintered castables were also characterized for their microstructure using (scanning electron microscopy) SEM.

2. Experimental procedure

2.1. Materials

The starting raw materials aluminum oxide, calcium carbonate, and zirconium dioxide were of A.R. grade and procured from Loba Chemie Pvt. Ltd., Mumbai, India. The materials were used as received. CA-25 C and CA-14 M high alumina cement used for comparative study was provided by Almatis, Kolkata, India. The Chinese bauxite, reactive alumina and silicon carbide powder were supplied by Shiva Minerals Pvt. Ltd., Rajgangpur, India. The calcined bauxite contained minimum 88.60%, 4.78%, 1.58%, 4.0%, 0.26%, 0.08% and 0.70% by weight Al_2O_3, SiO_2, Fe_2O_3, TiO_2, CaO, Na_2O and others respectively.

2.2. HAC powder preparation and phase analysis

Commercially available Al_2O_3 and $CaCO_3$ powders were used as the starting materials with 7:3 and 8:2 wt.% of Al_2O_3 and CaO. The milling operation was carried out in a QM-1SP4 type planetary ball milling system in air at room temperature for 1, 2 and 3 h. A total number of 80 stainless steel balls with a diameter of 10 mm and 2 stainless steel balls with a diameter of 20 mm were used as a milling medium in a 500 ml stainless steel vial. The milling speed was set at 600 rpm. All the samples were prepared in a glove box before the milling procedure to avoid moisture absorption. The mechanochemically prepared powders were isothermally heat treated (calcined) at the rate of 5 °C/min in air atmosphere with 1 h holding time at 1000 °C.

The phase composition and crystal structures of the prepared powders were investigated at room temperature using X-ray powder diffraction measurements. X-ray powder diffraction data were recorded at ambient conditions on a high resolution laboratory X-ray powder diffractometer at 40 kV and 40 mA. All the X-ray samples were contained in low absorbing glass capillaries of 0.3 mm diameter and sealed in a glove box under argon atmosphere using a hot wire. Data were taken from 15 to 50° at 1°/min. The samples were spun during measurement for better particle statistics. A qualitative phase analysis using the PDF-2 database (ICDD, 2003) was

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