AA36 - Effect of Submicron Reactive Alumina on the Rheological and Physico-Mechanical Properties of Microsilica Free Low Cement Castables

Ramkrishna Halder[1], Subhashree Sethi[2], Poomalai Saravanan[3] and Nageswar Kapuri[4]
1. Deputy Manager
3. Senior General Manager
4. Assistant Vice President
Hindalco Innovation Centre - Alumina (HIC-A), Belagavi, India
2. Assistant Manager
Hindalco Industries Limited, Belagavi Works, India
Corresponding Author: ramkrishna.halder@adityabirla.com

Abstract

Alumina is an integral part of the castable formulation, and it plays a very significant role in the castable matrix because of its superior high temperature properties. Hindalco Industries Limited (HIL) at Belagavi produces 80 to 90 different grades of specialty alumina, which are used for various refractory and ceramic applications. Hence, it was thought prudent to investigate the effect of the rheological and physico-mechanical properties on microsilica free low cement refractory castable.

The effect of submicron reactive alumina and its different proportions on the rheological and physico-mechanical properties on low cement high alumina castables were studied. The castable batches were prepared with different proportions of submicron reactive alumina by using calcium aluminate cement as a binder. The physico-mechanical properties of the prepared castables, i.e., flow, setting time, apparent porosity (AP), bulk density (BD), cold crushing strength (CCS), and modulus of rupture (MOR) were studied. The morphological analysis was carried out using scanning electron microscopy (SEM). The results are compared to find out the effectiveness of the selected alumina and its proportions to achieve the optimum properties in the refractory castable.

Keywords: Submicron reactive alumina, Castable, Rheological and physico-mechanical properties, Cold crushing strength.

1. Introduction

Alumina is a widely used material in the modern refractory industries due to its superior thermo-mechanical properties. The reactive aluminas are finely milled calcined aluminas which contain nearly 100 % alpha phase, especially low Na_2O containing material. The material is grounded up to their primary crystal size by a highly efficient grinding process to make it easily sinterable, which helps to develop high strength in the product, normally at relatively lower firing temperature compared to the normal calcined alumina. The global reactive alumina market size is anticipated to be USD 985 million in 2022 and it is expected to touch USD 14 756 million, at a CAGR of 6.98% during the forecast period [6].

The demand of monolithic refractories is increasing day by day because of its many advantageous properties over the shaped refractories in terms of production cost, installation efficiency, safety, and reduced manpower. Castable is a major group in the field of refractories. They are often supplied as a dry mix, water/solvent is added during installation to provide appropriate placement properties. The high alumina cement bonded refractory castables have generally the complex heterogeneous microstructures and their physical and thermo-mechanical properties are highly temperature dependent because of the complex hydration and dehydration processes [1]. The reduction of cement leads to higher refractoriness due to CaO-reduction. However, the opportunity of cement reduction is limited because of requirements for early strength development. On the other hand, the reduction of water leads to a higher packing density and hence reduces the porosity in the refractory [2]. This effect results in higher strength, lower high temperature shrinkage and better resistance to corrosion and infiltration of corrosive material like steel making slag.

Micro-fillers in the castable application enables a gap filling mechanism between two particles, causing a reduction in water requirement [3]. To get a dense matrix either in low cement or ultra-low cement castable, the addition of water is very important. A lower amount of water is always good to get better physical properties like strength and other high temperature properties. Reactive alumina is one of the major components of these micro-fillers. However, the choice of the reactive alumina or matrix alumina in new generation high performing castables is not limited to chemical purity but mainly to the physical properties, such as specific surface area (BET) and particle size distribution (PSD). The combination of reactive alumina with other matrix components, such as tabular alumina 20 µm, allows the control of the particle size distribution to achieve the desired rheological properties. The sintering activity of the reactive alumina can also largely influence the final performance of castable. Here an effort has been made to find out the effect of reactive alumina on the water demand/flow behaviour of the castables and its impact with different proportions in the final properties (like CCS and MOR) of such type of castable formulation.

2. Experimental

The selected calcined and reactive alumina grades were analysed in terms of chemistry (XRF), particle size distribution (PSD), and specific surface area (SSA) and the detailed analysed results are presented in Table 1. Also, the physical and chemical properties of other raw materials used for the preparation of castables have been presented in Table 2. The particle size distribution of alumina was measured using a Micrometrics, Sedi-graph 5100 and the specific surface area was measured by BET method using an advanced Quantachrome Nova 4000e. The fired castable sample was also analysed in SEM (HITACHI SU 1500) to see the micrograph (Fractography).

Subsequently, three castable batches (B1, B2 and B3) were prepared by varying the proportions of submicron reactive alumina and calcined alumina in the low cement castable formulation with 95 % alumina (aggregates with fines) plus 5% high alumina cement. The physical properties like flow, apparent porosity (AP), bulk density (BD), modulus of rupture (MOR), and cold crushing strength (CCS) were measured.

Here, the matrix of the castable formulation was made with tabular alumina, reactive and calcined alumina, and high alumina cement, in order to avoid any adverse effect of other raw materials, like microsilica, sillimanite sand etc. on the reactivity of the final castable, especially at the elevated temperature. The castable formulation (Table 3) was designed with a reactive and calcined alumina by varying their proportions to see the effect of the reactive alumina on the performance of the castable properties.

Table 1. Physico-chemical properties of the selected aluminas.

Parameters	Calcined alumina	Reactive alumina
Al_2O_3	99.6	99.8
Na_2O	0.25	0.08
SiO_2	0.02	0.04
Fe_2O_3	0.02	0.02

Parameters	Calcined alumina	Reactive alumina
CaO	0.026	0.02
MgO	0.01	0.05
d_{10} (µm)	0.95	0.24
d_{50} (µm)	4.20	0.49
d_{90} (µm)	7.94	2.90
SSA (m^2/g)	1.6	7.4

Table 2. Properties of other raw materials used for castable formulation.

Parameters	Tabular alumina	High alumina cement
Al_2O_3	99.4	70.1
Na_2O	0.32	0.24
SiO_2	0.08	1.49
Fe_2O_3	0.025	0.12
CaO	0.055	25.9
MgO	0.034	0.02
Sp. Gravity	-	2.91
BD (g/cm^3)	3.62	-
AP %	1.34	-

Table 3. Castable formulation used for the experiment.

Raw materials used	Castable batches		
	B-1	B-2	B-3
Tabular Alumina (0.2–6 mm)	75 %	75 %	75 %
Tabular Alumina -325#	5 %	5 %	5 %
Calcined Alumina	15 %	12 %	9 %
Reactive Alumina	0 %	3 %	6 %
High Alumina Cement	5 %	5 %	5 %
Total	100 %	100 %	100 %
Dispersant	0.2 %	0.2 %	0.2 %

The castable batches were prepared by mixing in a Hobart mixer. The flow properties of the castable (after 15 s vibration) were measured in the vibrating table (frustum cone with 50 mm height, vibration at 0.75 mm amplitude and 60 Hz frequency). Subsequently, the batches were cast into the moulds of 160 × 40 × 40 mm size (for MOR and CCS). The cast samples were then cured and dried at 110 °C for 24 hours in an oven to measure the room temperature properties. The cast bars were also fired at different temperatures and subsequently, modulus of rupture (MOR) and cold crushing strength (CCS) were measured using 60-tonne capacity universal testing machine as per standard method (IS 10570-2011). The bulk density (BD) and apparent porosity (AP) of all the samples were measured by water displacement method using Archimedes principle.

3. Results and Discussions

Figure 1 shows the flow properties of the castable, made with different proportions of reactive alumina (0 %, 3 %, and 6 %). The higher proportions reactive alumina reduced the water requirement and improved the rheological properties because of its better packing efficiency in the castable formulation. Also, the bulk density increased with an increase in reactive alumina proportions by filling the interstitial voids in the castable matrix and formed a compact structure, and subsequently reduced the porosity which is evident from Figure 2.

It could be seen that the MOR and CCS values (Figure 3 and 4) increased with an increase in temperature. However, a dip could be seen at 1100 °C, mainly because of an inadequate driving force for sintering at this temperature for such kind of high alumina castable. This is mainly due to the removal of structural water from high alumina cement and lower level of ceramic bond formation at this temperature.

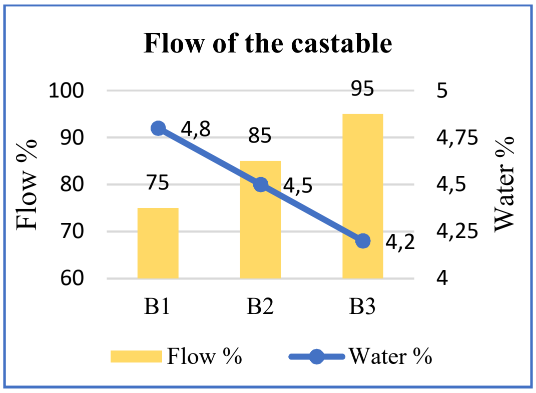

Figure 1. Flow properties of the castable.

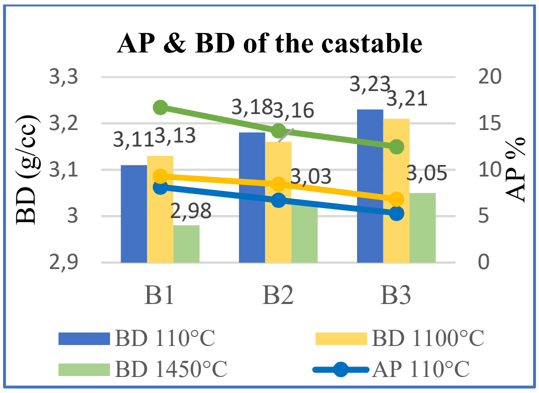

Figure 2. AP and BD of the castable.

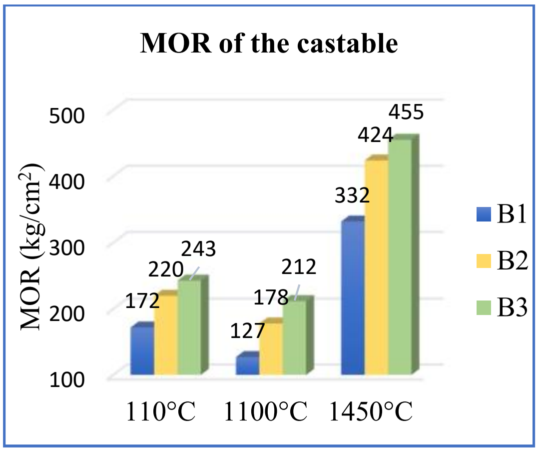

Figure 3. MOR of the castable batches.

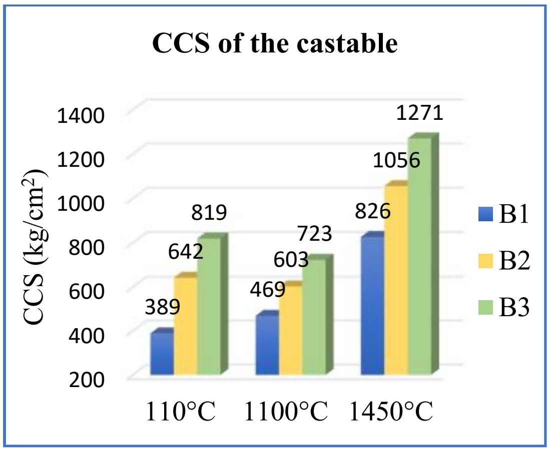

Figure 4. CCS of the castable batches.

Normally, the presence of reactive alumina has beneficial effect for high temperature refractory products, and thereby responsible for the higher strength development at elevated temperature. The SEM analyses of 1450 °C heated specimens are shown in Figure 5a, 5b and 5c. It is very evident that batch B-1 displayed an intergranular fracture in the absence of reactive alumina whilst a relatively denser matrix was formed with minimal pores or cracks and clearly displayed a transgranular fracture in case of batch B-2 and B-3. This dense matrix was primarily responsible for very high strength and modulus of rupture at elevated temperature.

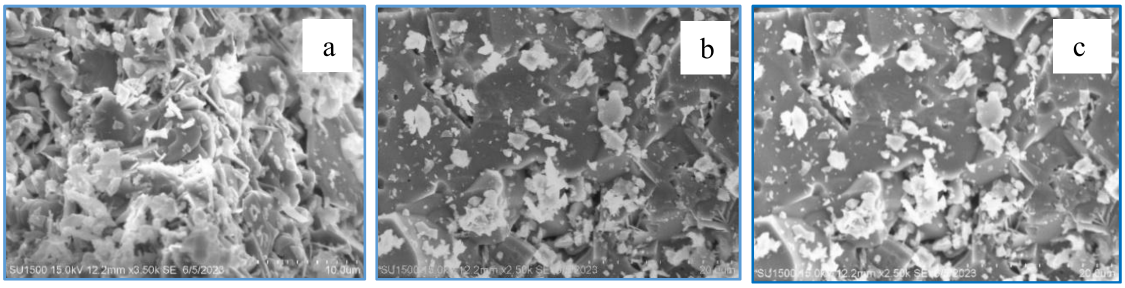

Figure 5. (a): SEM of castable batch B-1, (b): SEM of Castable batch B-2, (c): SEM of castable batch B-3.

4. Conclusions

It is obvious from this study that the reactive alumina in low cement castables, plays a very crucial role in providing the desired flow and castable properties.

The finer reactive alumina, helped to improve the particle packing and thereby improving the densification of the castable, leading to higher mechanical properties both at room and elevated temperatures. Here, the castable formulation with reactive alumina 3 % and 6 % showed very good rheological properties and flow increased by 13 % and 26 % respectively. Also, the thermomechanical properties increased by 28 % and 54 % at higher temperature (1450 °C) due to its very dense matrix which was also reflected in the SEM micrograph. Higher level of reactive alumina (> 6 %) in the castable formulation was not evaluated in the present scope of study, in order to contain the cost of the product within the desired range. However, it is good to have such kind of data for comparative evaluation purpose.

5. References

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