Effect of Alumina Fines on High Alumina Self-flow Low Cement Castables

R. Sarkar, A. Parija

Fines play a major role in the self flowing characteristics of castable which has been studied by using two different alumina fines, namely WTA (white tabular alumina) fine and reactive alumina in the self-flow low cement castable with distribution coefficient, q = 0,21 and 0,23 as per *Dinger* and *Funk* particle size distribution models. The castables were processed as per normal processing and properties were compared at three different temperatures. Results showed that reactive alumina containing composition showed improved properties than that of WTA fines batch for all the temperatures.

1 Introduction

Incorporation of various sophisticated processes to improve the quality of steel and automation to improve the productivity and efficiency have resulted revolutionary changes in steel manufacturing processes. Accordingly to cope up with the advancements in the user industries, refractory materials required to be upgraded to meet the severe challenges and to satisfy the critical operational parameters at high temperatures. Refractories are also required to perform better with less downtime, as being imposed by the user industries. Many inherent advantages of unshaped refractories have taken the attention of the refractory researchers, manufacturers and users to substitute for conventional shaped refractories by unshaped ones to meet the advanced challenges. Among the various unshaped refractories, castables lead in the areas of research, development, manufacturing and application.

For castables both flow and strength are required, which are contradictory in nature. Strength develops from increased packing efficiency and greater friction among different components but results in lesser flow. Hence conventional concept of packing does not work here. For castables, the type of packing plays a vital role. A different particle packing concept, named "Continuous Particle Size Distribution", is important for the castable development.

Furnas [1] stated that maximum packing density is obtained when particles of smaller size fill the voids between particles of greater size. According to *Andreassen* [2–4] particles cannot be of a particular size, instead they come within a certain narrow size range. Andreassen has given a model for continuous particle size distribution which was quiet popular but he did not assume any specific minimum particle size and assumed that the particles can be infinitesimally small which is practically not possible. This model was modified by *Dinger* and *Funk* [5], considering a specific minimum size of particles to make the model more acceptable.

Now, castables that can flow under its own gravitational force (weight) are called self-flow castables and they are important as no external energy is required to flow, to fill up the mold and to take the exact shape required of an intricate shaped mold. Self-flow castables contain a greater amount of fines while castables with lesser fines require vibration to take shape. The amount of fines content in a castable is decided by the distribution coefficients. If it is less, then the castable has greater amount of finer particles. Usually the castable is self-flowing in nature if the distribution coefficient is less than 0,25 [6].

Karadeniz et. al. have studied [7] self-flowing low cement castable using Andreassen model with distribution coefficients 0,20, 0,21, 0,22, 0,23, 0,24 and 0,25 and reported best properties for q values of 0,23 and 0,22 having 9 mass-% microsilica and 12 mass-% calcined alumina. *Altun* studied [8] self-flowing low cement alumina based castables using bauxite and brown fused corundum and reported better hot strength at 1000 and 1500 °C for the bauxite-based composition due to greater extent of mullite formation. Two different castable systems with different particle size distribution were studied [9] using silica sol between 12–18 %. Densification and cold strength was reported to be higher for the composition with lower intermediate fraction and hot strength was reported to be higher for the composition with higher intermediate sized particles content.

Greater densification and room temperature strength was reported [10] for the castable containing impure cement and only a little grossite phase (CA_2) other than the major

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Tab. 1 Details of the different starting materials

Constituent [mass-%]	WTA Grain	WFA Grain	Alumina Fines		Fume Silica	HAC
			TA	RA		
SiO_2	0,03	0,1	0,12	0,03	96,2	0,21
Al_2O_3	99,34	98,93	99,1	99,3	0,4	71,64
Fe_2O_3	0,035	0,06	0,2	0,03	0,1	0,11
TiO_2	–	Trace	0,06	Trace	–	–
CaO	–	0,1	–	0,02	0,2	26,91
MgO	–	–	–	0,01	0,1	0,32
Na_2O+K_2O	0,15	0,4	0,3	0,1	0,4	0,27

Property	WTA Grain	WFA Grain	Alumina Fines		Fume Silica	HAC
			TA	RA		
Particle size [µm]	–	–	>98 % <45	D50 = 2,5	>99 % <45	–
Bulk density [g/cm³]	3,61	3,77	–	–	–	–
Apparent porosity [%]	3,93	1,8	–	–	–	–
Specific surface area [m²/g]	–	–	–	3,1	20	0,44
Phase analysis	Corundum	Corundum	Corundum	Corundum	Amorphous	CA_2, CA

Tab. 2 Particle size distribution of the castables with q value 0,21 and 0,23

Particle Size [mm]	Percentage of Particles	
	q = 0,21	q = 0,23
–3 to +2	10,03	10,58
–2 to +1	15,28	15,96
–1 to +0,5	13,22	13,6
–0,5 to +0,3	8,58	8,73
–0,3 to +0,15	10,26	10,31
–0,15 to +0,001	42,63	40,82

corundum phase obtained on firing at 1600 °C. In a different study [11] high alumina self-flow (distribution coefficient 0,21) castables were found to have mullite and grossite phases other than the major corundum phase for silica sol and alumina cement containing castables respectively. Addition of nano-alumina (d_{50} = 43 nm) in self-flow castable was reported [12] to reduce the flow value due to greater attraction between nano alumina and other matrix particles. Again nanoparticles promote sintering and corresponding strength also. *Otroj* and *Daghighi* [13] reported increased strength at lower temperature (1300 °C) due to enhanced solid phase sintering in presence of nanosized particles and also CA_6 formation. *Badiee* and *Otroj* studied [14] the effect of nano-titania addition in high alumina self-flow castable and reported a decrease in self- flowability and working time but increase in mechanical properties with increasing amount of nano-titania.

In the present work self-flow low cement high alumina was designed as per the Dinger and Funk model with distribution coefficients of 0,21 and 0,23 and using two different alumina fines, namely technical alumina (TA) and reactive alumina (RA), to study the effect of the alumina fines on the different properties of the castable.

2 Experimental

High alumina self-flow castables were prepared using white tabular alumina (WTA) (*Almatis*/IN), white fused alumina (WFA) (Chinese source) grains/aggregates, alumina fines (Almatis/IN), high alumina cement (Almatis/IN), silica fume (*Elkem*/IN) and additives like deflocculant, set retarder and organic fibers. Details of the physicochemical properties of the starting materials are provided in Tab. 1. Tab. 2 shows the percentages of the different particle size fractions that are required for making the castable compositions having two different distribution coefficients (0,21 and 0,23) calculated from the formula as proposed by Dinger and Funk [10].

Four different castable batches were prepared using alumina fines TA and RA with q values of 0,21 and 0,23 (namely 21TA, 23TA, 21RA and 23RA respectively). For all the compositions, 4 mass-% high alumina cement was used as well as 5 mass-% fume silica, 0,3 mass-% deflocculant (ammonium polymethacrylate), 0,1 mass-% anti-setting agent (citric acid) and 0,05 mass-% organic (polypropylene) fiber. All the castable compositions were processed under similar conditions.

2.1 Preparation of the castable

First, all the raw materials were dry mixed in a planetary mixer (*Hobart*/GB, model N50), then water was added till the mix attains self flowing consistency. Mixed compositions were characterized for self-flowability, using a flow cup, as per *Fang et al.* [15]. Water mixed castable compositions were placed in the cup and flow values were measured after allowing to flow for 30 s onto a flow table by drawing the cup upward. Measurement of flow was done after allowing the material to flow (spread). Flow values, an average of eight different directions for each composition, were presented.

Next the mixed batches were poured into lubricated iron moulds of dimension 50 mm³. The excess mix was scrapped off and smoothened by a trowel. The mixes were allowed to aged for 24 h inside the mould under humid condition. Next they were demolded and allowed to be air dried for 24 h followed by oven drying at 110 °C for 24 h. The dried samples were then fired at 950 °C and 1550 °C with 2 h soaking time at peak temperatures. Dried and fired cubes were characterized for bulk density (BD) and cold crushing strength (CCS).

The characterization was done as per *Bureau of Indian Standard (BIS) Specifications*, IS 1528–1974, Part XII, Part VIII and Part IV, reaffirmed on 2002. Each data represented here are an average of five individual measurements. Phase analysis of the 1550 °C fired samples was also done to evaluate any phase changes occurred during firing.

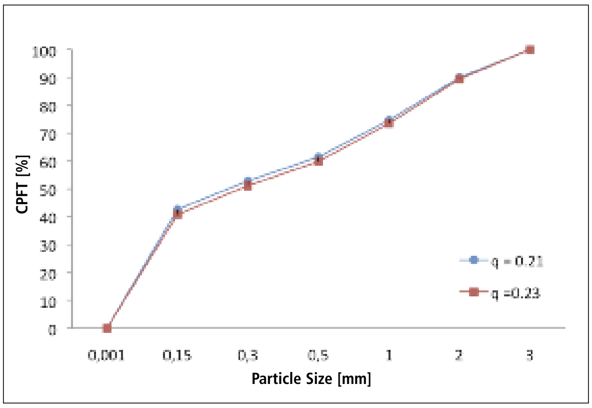

Fig. 1 Plot of cumulative percent finer than [%] of different sizes of castable components against their sizes

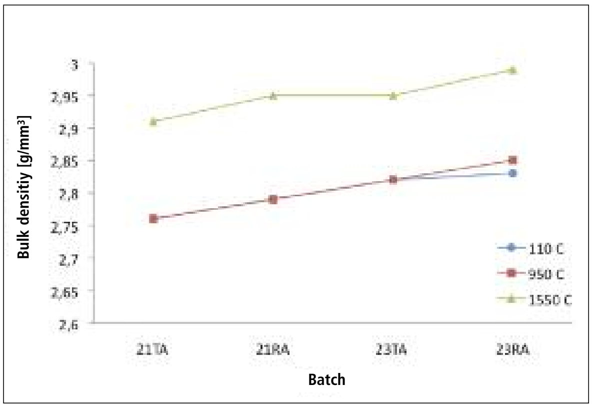

Fig. 2 Variation of bulk density of the castables processed at different temperatures

3 Results and discussion

Fig. 1 shows the plot of CPFT (cumulative percent finer than) against particle size for the distribution coefficient q = 0,21 and 0,23. The highest particle size of 3 mm has a CPFT of 100 % and the lowest particle size being 1 µm shows 0 % CPFT. The composition with q value 0,21 has a higher extent of fines content. There is an abrupt change in the slope of the plot in the finer size range due to high fine content in the compositions (low q value). Tab. 2 shows that the percentage of finer (< 0,15 mm) fraction is higher for the composition with q = 0,21.

Physicochemical properties of the starting raw materials indicate (Tab. 1) that the materials all are pure and contain a minimum amount of impurities. Among the two different aluminas RA is marginally purer and finer than the TA alumina fines.

Tab. 3 shows the water requirement and self-flow values of the different castables. The use of RA was found to reduce the water demand of the castable drastically and also resulted in better self-flowing values. Again, the increase in the q value has resulted in a decrease in the water requirement and also a decrease in the self-flow value for both the fine alumina types. Increase in q value means decrease in fine content, resulting in lesser water requirement from the surface wetting point of view. Again, higher q value means greater extent of coarser fraction, resulting in lower flowability.

Tab. 3 Water requirement and flow value of different castables

	21TA	21RA	23TA	23RA
Water [%]	7	5.4	6.8	5
Flow value [mm]	155	170	140	160

3.1 Bulk density study

Fig. 2 shows the variation in bulk density (BD) of different compositions studied vs. the firing temperature. Density values were found to increase with the increase in q value and also in presence of RA. Higher q value means greater extent of coarser fractions, which has a greater bulk density values than the finer fraction. Again, better flowability resulted in better filling and compaction. Also lower water requirement for the RA containing compositions resulted in a less porous structure and correspondingly resulted in higher BD values.

Reduction in BD values was not observed on increasing the firing temperature from 110 and 1000 °C due to breakage of hydraulic bonds. Significant extent of increase in density values was observed on further increase in temperatures due to the sintering of the compositions. A maximum BD value of 2,99 was observed for the 23RA batch on firing at 1550 °C

3.2 Cold crushing strength study

Fig. 3 shows the variation of the cold crushing strength (CCS) values of different castables compositions against firing temperature. Degradation of strength at intermediate temperature due to the breakage of hydraulic bond in cement containing castables was not observed for all the compositions. Better compaction, use of organic fibers etc. might have prevented the loss in strength. Strength was also found to be higher for compositions containing RA and also increase in the q-value. Better compaction

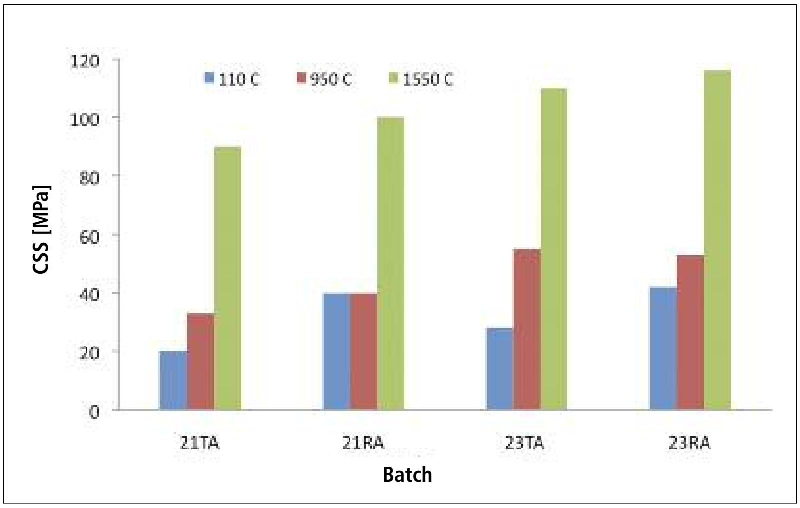

Fig. 3 Variation of CCS of the castables processed at different temperatures

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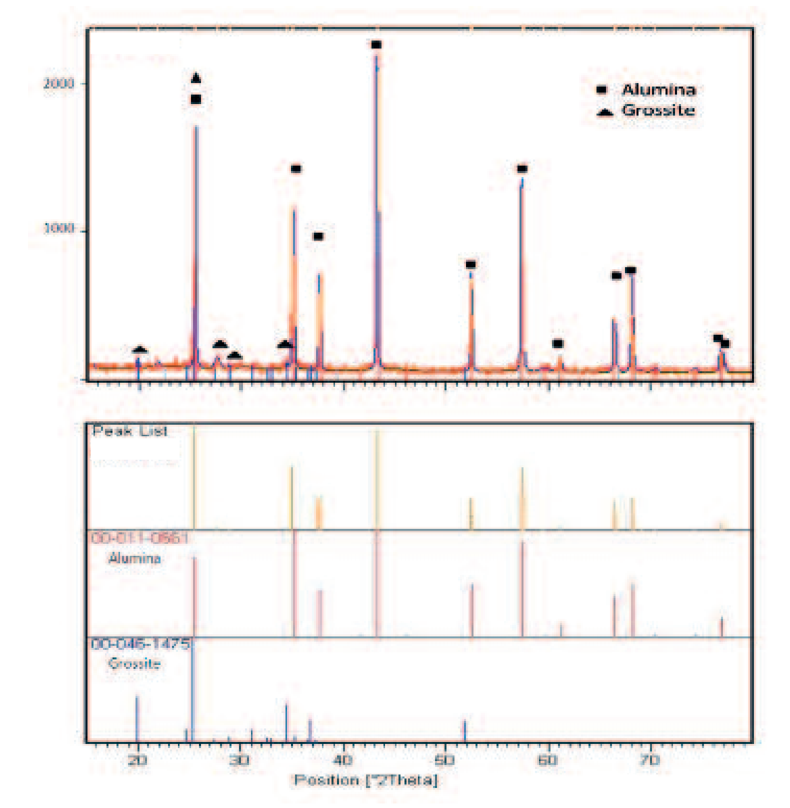

Fig. 4 XRD plot of the 21RA castable processed at 1550 °C

from higher flowability may be the reason for both the observations.

3.3 Phase analysis study

Phase analysis by powder XRD study of all the batches fired at 1550 °C was done; XRD plot of batch 21RA is shown in Fig. 4 as a representative one. For all the compositions corundum was found as the major phase and grossite (CA_2) as the minor one. No hibonite phase (CA_6) formation was observed. Again, no peak of calcium aluminate (CA) phase was found (which was present in the cement). This may be due to the reaction between fine alumina and the CA phase at high temperatures in the matrix of castables forming greater extent of grossite (CA_2) phase. And this reaction might have caused better bonding in the matrix resulting in better densification and strength values.

4 Conclusions

- Use of reactive alumina resulted in lesser water demand with better self-flowability.
- Increase in q-value marginally reduces the water demand and also resulted in lower flow values.
- Use of reactive alumina has resulted in higher density and strength values.
- Reaction between CA phase and fine alumina forming only CA_2 phase is the reason for high strength development at higher temperatures.

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