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Properties of alumina based low-cement self flowing castable refractories

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Abstract

The main aspects that determine flowing properties of self flowing alumina based low-cement castables are determined. From the point of view of particle size distribution, different particle size distributions are compared by using Andreassen model according to n-value. The n-values are varied as 0.20, 0.21, 0.22, 0.23, 0.24 and 0.25 to define which one corresponds to the best flowing properties. The prepared castables are fired at different temperatures. Rheological and physical properties of prepared castable refractories were examined. It is found that the self flowing refractory which is prepared by using 0.23 n-value has the best self flowing properties. In addition to these studies, different alumina types which affect the aggregate system, and the roles of additives (calcined alumina, microsilica) are also examined by using $n = 0.23$ value. Finally SEM studies are performed to investigate the microstructure of these self flowing castables.

Keywords: Al_2O_3; Refractories; Castable

1. Introduction

In the last decades there has been an increasing trend among refractories towards replacement of bricks by castable refractories. Especially low-cement castables have been widely used in steel industry due to their superior rheological and physical properties. Initially, castable refractories were composed of only cement and aggregates. Then, the addition of deflocculants and fine fillers have followed with the aim to optimize the control of properties such as workability as a function of time.[1]

In order to compose self flowing refractory, components and the particle size distribution of the aggregate system have to be carefully selected since the particle size distribution is one of the most important factors that affect the refractory's rheological properties.[2]

In this study, particle packing behaviour was evaluated by the Andreassen model[3] according to n-values between 0.20 and 0.25. The flow properties were measured by the flow test comprising the flow cone and table. The purpose of this work is study the influence of particle size distribution, firing temperature, alumina aggregate types and other parameters on the rheological and physical properties of self flowing castables.

2. Experimental procedure

2.1. Raw materials and mixtures

The mixtures were composed with tabular alumina grades, white corundum, brown corundum, calcined bauxite, calcined alumina, reactive alumina, microsilica, and high alumina cement. Chemical composition of the basic materials is given in Table 1. The phases were determined by Philips PW 3710 X-Ray diffractometer.

Castable refractories which have different particle size distributions were prepared using the Andreassen equation with n-values between 0.20 and 0.25.

Andreassen equation[3]:

$$\mathrm{CPFT} = 100 \times \left(\frac{d}{D}\right)^n$$

CPFT, cumulative percent of grains; D, maximum used grain size; d, the grain size; n, particle size distribution parameter.

2.2. Sample preparation

All aggregate system components were mixed dry for 1 min in Hobart mixer. After water addition wet mixing was continued until getting flowing refractory about 4 min. A part of this flowing refractory was taken for the flow test and the other part was poured into the moulds of 50 mm × 50 mm × 50 mm without

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Table 1
Typical chemical compositions of raw materials

	Tabular alumina	White corundum	Brown corundum	Calcined bauxite	Calcined alumina	Reactive alumina	Microsilica	Cement
Al_2O_3	99.4	99.7	95	86	99.5	99.78	0.7	69–71
SiO_2	–	<0.3	1.5	7	–	0.03	97.5	0.8
CaO	–	–		–	0.01	0.02	–	27–29
TiO_2	–	–	3	4	–	–	–	0.1
Fe_2O_3, alkaline oxides, C	0.06	<0.3	0.5	3	0.49	0.17	1.8	1.1
Primary phases	α-Al_2O_3	α-Al_2O_3	α-Al_2O_3	α-Al_2O_3	α-Al_2O_3	α-Al_2O_3	–	CA, CA_2

CA, $CaO{\cdot}Al_2O_3$; CA_2, $CaO{\cdot}2Al_2O_3$.

vibration. After demoulding, the samples were cured for 24 h at room temperature and cured samples were dried at 110 °C/18 h. Then, they were fired at varying temperatures of 1100, 1300, 1500 °C for 5 h.

To determine the flowability of the castable samples, flow test C 230 was used which is a system including a flow cone and a flow table. Flow cone's outer diameter is 70 mm, inner diameter is 100 mm and the height is 70 mm. After wet mixing, metal flow cone was filled completely with self flowing castable without vibration. The spreading of castable on the table was measured after a period of 10 min as the flow value of the castable. Apparent porosity and bulk density of prepared samples were determined by the water immersion method (DIN 51056).

3. Results and discussions

3.1. The effect of the difference in particle size distribution

Fig. 1 shows the comparison of the variation in flow diameter with particle size distribution parameter (n) in Andreassen equation between 0.20 and 0.25. Tabular alumina was used as the refractory aggregate.

The test results revealed that the best flow was obtained with $n = 0.23$ value. Presumably, with this n-value, the friction between the grains is very low and particle packing is very good. Adjustment of particle size distribution is a prime factor in obtaining self flow ability so that choosing particle size distribution parameter 0.23 produced the most favorable results.

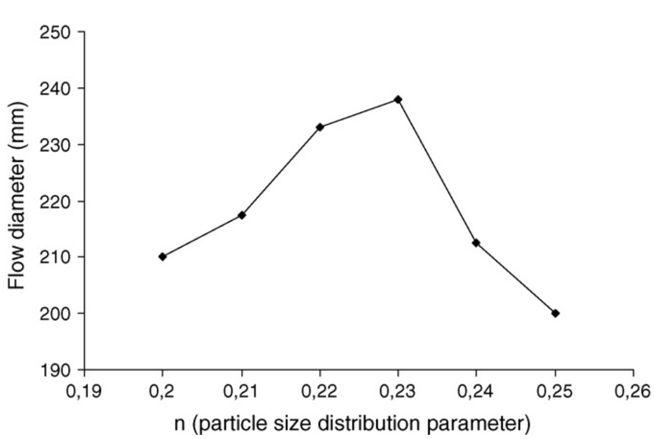

Fig. 1. The comparison of change in flow diameter with particle size distribution parameter.

3.2. The effect of firing temperature

3.2.1. Cold crushing strength

Prepared samples were fired at 1100, 1300 and 1500 °C for 5 h after drying at 110 °C for 18 h and their cold crushing strength were determined. The effect of firing temperature on the castable refractory's cold crushing strength for 0.23 n-value is shown in Fig. 2.

When aggregate system is reacted with water, hydrate phases form which in turn decrease the refractroy's strength. Firing results in the sintering and the formation of ceramic bonds between the particles and consequently the strength has increased.

Therefore, Fig. 2 indicates that increase in firing temperature results in the development of the cold crushing strength. At the firing temperature of 1100 °C, corundum (α-Al_2O_3), anorthite ($CaO{\cdot}Al_2O_3{\cdot}2SiO_2$) and mullite ($3Al_2O_3{\cdot}SiO_2$) phases were observed in the structure of the refractory. Formation of corundum and mullite phases contribute to the increase in strength. When firing temperature was increased to 1300 °C, in addition to these phases, hibonite ($CaO{\cdot}6Al_2O_3$) phase was observed to form with a decrease in the content of the anorthite. After 1500 °C firing temperature, corundum, anorthite, mullite and

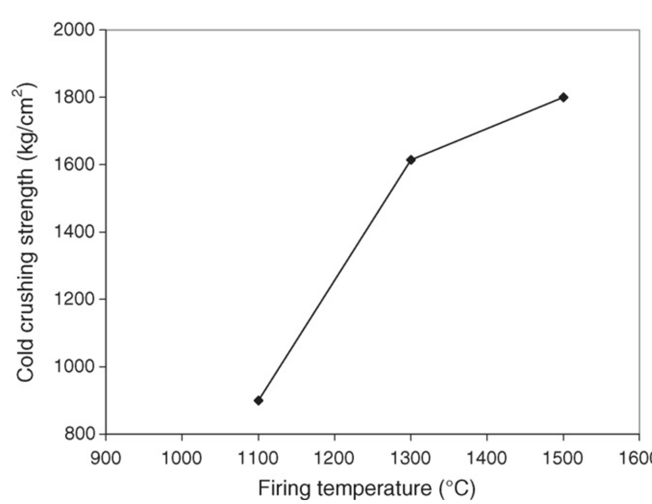

Fig. 2. The effect of firing temperature to cold crushing strength for 0.23 n-value.

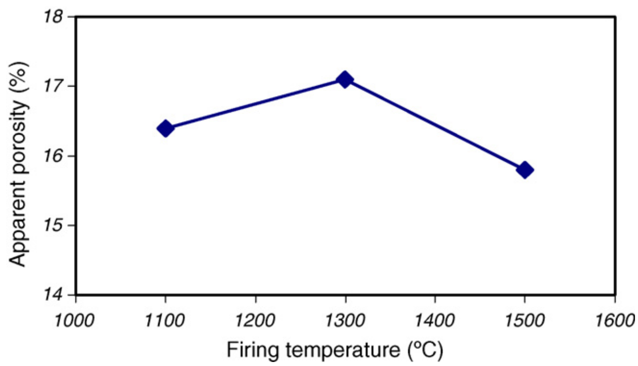

Fig. 3. The effect of firing temperature to the refractory's apparent porosity for 0.23 *n*-value.

hibonite phases were found to exist with further decrease of anorthite.

3.2.2. Apparent porosity

Fig. 3 shows the effect of firing temperature on the refractory's apparent porosity for $n = 0.23$ value. Increase in firing temperature first causes an increase in the apparent porosity but at 1500 °C firing temperature a decrease of apparent porosity was observed. The explanation was assumed to be that high firing temperatures caused the melting of the fine grains which in turn had an effect in closing the porosity of the refractory structure.

3.3. Properties of refractories prepared with different alumina raw materials

Table 2 shows the properties of samples prepared with different alumina raw materials. Tabular alumina seems to be the most advantageous raw material for the self flowing castable refractoy for $n = 0.23$ value. Also it can be said that white and brown corundum can be alternative for tabular alumina to produce self flowing castable. However, for the castables with calcined bauxite, in order to obtain good flow behaviour, the application of vibration is necessary.

3.4. The effect of addition of microsilica

3.4.1. The flow diameter

The effect of the microsilica on flow diameter of castable refractory for *n*-value of 0.23 is shown in Fig. 4. It can be observed that the increase in microsilica content also increased the flow diameter but when the content is increased to 12%, the flow diameter decreased. In this type of systems if fines and superfines are added in increased quantities, flocculation tends to occur which causes a decrease in the flow behaviour. Microsilica is an attractive alternative in improving particle packing, reducing water consumption and leading to low-porosity castables with suitable mechanical strength by filling the porosity in the refractory structure.

Table 2
The properties of prepared with different alumina raw materials for 0.23 *n*-value

	Tabular alumina	Brown corundum	White corundum	Calcined bauxite
Flow diameter (mm)	238	220	223	127.5
Density (g/cm^3)	2.98	3.12	3.1	2.86
Apparent porosity (%)	17.1	15.8	16.1	16.7
Water demand (%)	5.19	5.58	5.4	6.01

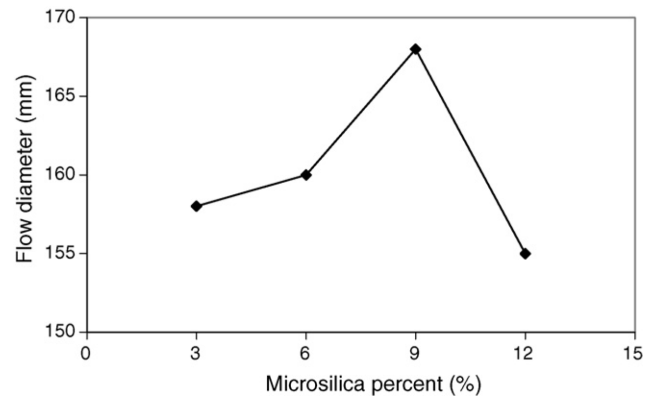

Fig. 4. The effect of microsilica on flow diameter of the castable refractory.

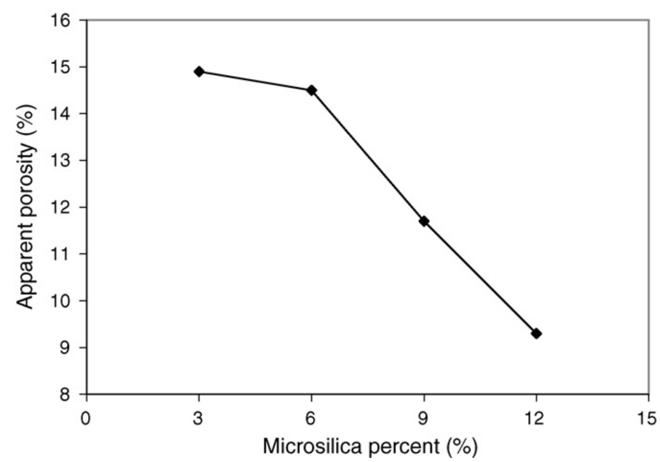

Fig. 5. The effect of microsilica to the apparent porosity of the refractory.

3.4.2. Apparent porosity

Fig. 5 shows the effect of microsilica on the apparent porosity of the refractory for $n = 0.23$ value. Microsilica is found to

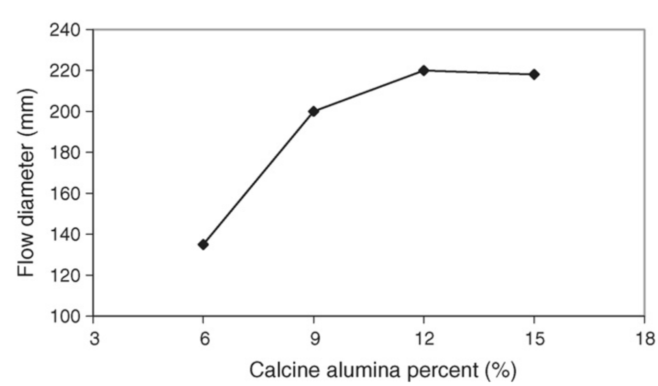

Fig. 6. The effect of the calcined alumina on flow of the refractory.

decrease the apparent porosity due to its equiaxed and superfine morphology, the porosity in the structure is closed.

3.5. The effect of calcined alumina

Fig. 6 shows the effect of calcined alumina on the flow behaviour of the self flowing castable. Calcined alumina increases the flow diameter, but as mentioned above as this type of material is added to the system more, the flocculation starts to occur.

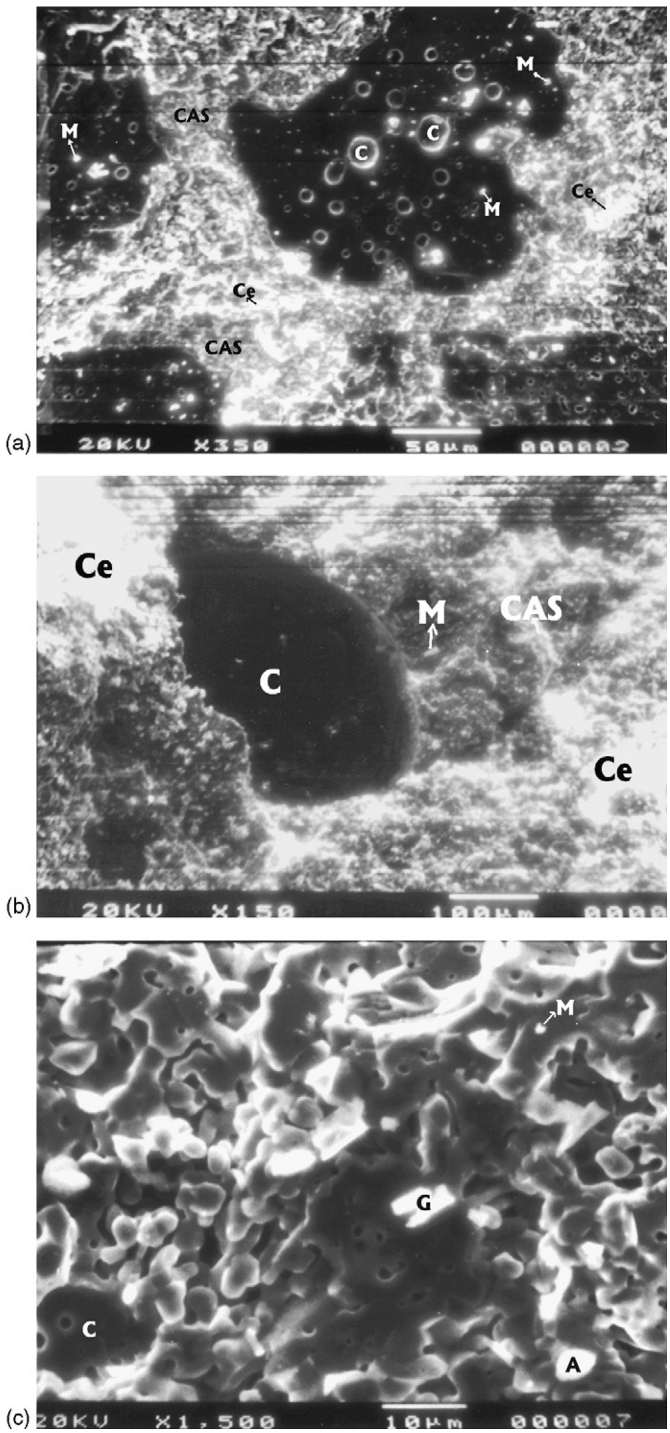

Fig. 7. SEM photomicrographs of tabular alumina based self flowing castable fired at different temperatures at 5 h: (a) 1100 °C; (b) 1300 °C; (c) 1500 °C. C, corundum; A, anorthite; M, mullite; G, gehlenite; H, hibonite; Ce, cement; CAS, $CaO{\cdot}Al_2O_3{\cdot}SiO_2$.

3.6. Microstructural evaluation

3.6.1. Microstructural evaluation on firing

Fig. 7a is the micrograph of tabular alumina based self flowing castable fired at 1100 °C. Corundum (α-Al_2O_3) is the main matrix of the refractory. It is revealed that there is extensive CAS formation as the binder liquid phase. Also it is shown that the mullite ($3Al_2O_3{\cdot}2SiO_2$) crystals was formed in the structure of the refractory by the reaction of alumina and the microsilica. In addition to this, cement grains, blanks and blistering are observed in the microstructure of the refractory. The micrograph of structure fired at 1300 °C is shown in Fig. 7b, where the same phases as at 1100 °C firing were found to exist. Fig. 7c shows the micrograph of the refractory which was fired at 1500 °C. In this case, existence of low melting glassy phases like gehlenite ($2CaO_2{\cdot}Al_2O_3{\cdot}SiO_2$) and anorthite ($CaO{\cdot}Al_2O_3{\cdot}SiO_2$) are seen.

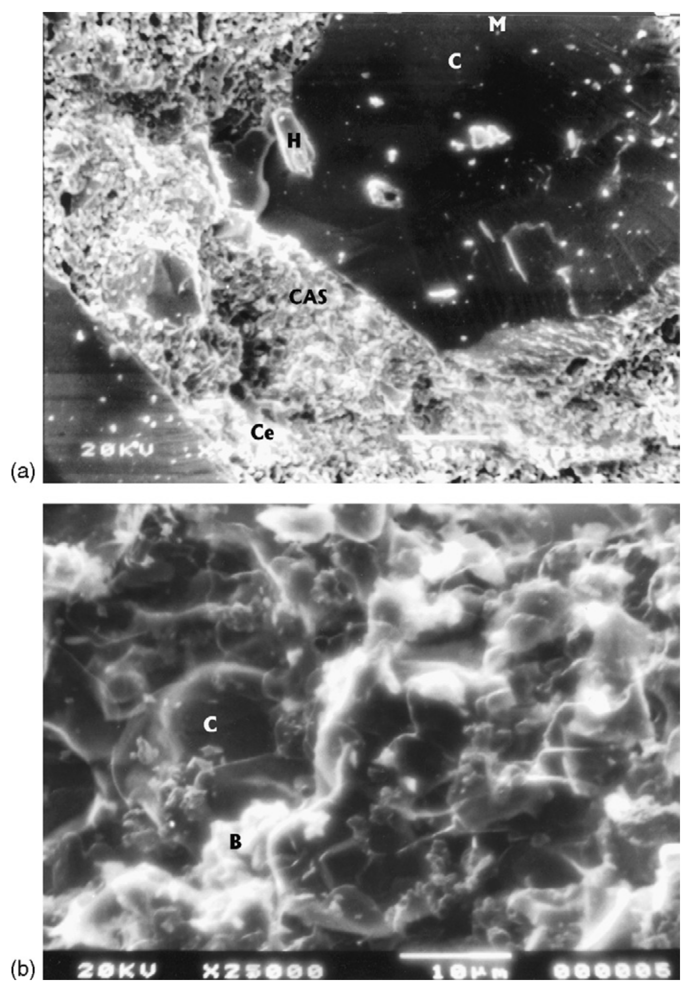

Fig. 8. SEM photomicrographs of different alumina types fired at 1300 °C at 5 h: (a) white corundum; (b) calcined bauxite. H, hibonite ($CaO{\cdot}6Al_2O_3$); B, complex compound with CaO, Al_2O_3, Fe_2O_3, TiO_2, SiO_2.

3.6.2. Microstructural evaluation of alumina types

Fig. 8a is the micrograph of white corundum based self flow castable refractory. The main matrix is corundum and it has silicate phases as shown before. In addition to these phases hibonite phase is seen in the structure which takes shape by the reaction of calcium oxide and alumina above 1300 °C. This phase have an important role that it improves the mechanical strength of the refractory. Fig. 8b shows the micrograph of the calcined bauxite based self flowing castable refractory. Since the bauxite raw material includes impurities in its structure, compounds formed by these impurities (B) are observed in the figure.

4. Conclusions

Based on the above given results, it can be concluded that the properties of self flowing castables develop with:

- the use of 0.23 and 0.22 for *n*-value;
- the use of microlica to 9%;
- the use of calcined alumina to 12%;
- the use of tabular alumina, white corundum and brown corundum;
- the use of high firing temperature.

Acknowledgement

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References

1. The Technical Association of Refractories, *Refractories Handbook*. The Technical Association of Refractories, Japan, 1998.
2. Myhre, B., *The Effect of Particle Size Distribution on Flow of Refractory Castables*. Elkem Refractories, International Ceramics, Norway, 1994.
3. Dinger, D. R. and Funk, J. E., Particle packing II: review of packing of polydisperse particle systems. *Interceramic*, 1992, **41**, 95–97.