Asian Journal of Chemistry; Vol. 25, No. 10 (2013), 5568-5570

ASIAN JOURNAL OF CHEMISTRY

http://dx.doi.org/10.14233/ajchem.2013.OH24

Effects of Alumina Micro-Powder Additions on Properties of Special Silicon Mullite Brick†

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AJC-13264

Special silicon mullite brick for transition belt of cement kiln was prepared using bauxite clinke, white fused alumina, silicon carbide, guangxi white clay, alumina micro-powder additions as main starting materials and sulfite pulp waste liquid as binder. Effects of alumina micropowder additions on cold physical properties, thermal shock resistance and abrasion resistance of silicon mullite brick were studied. The samples were characterized by XRD and SEM. The results showed that with alumina micro-powder addition increasing, cold strength, bulk density of special silicon mullite brick were first enhanced and then declined; apparent porosity first decreased and then increased; thermal shock resistance of the brick was first enhanced and then declined. When the addition of alumina micro-powder was 4 %, the silicon mullite brick had the best properties and the brick as ideal lining materials of the transition belt of large-scale cement rotary kiln.

Key Words: Alumina micro-powder, Silicon mullite brick, Cold properties.

INTRODUCTION

In recent years, the use of silicon mullite brick in cement rotary kiln has gotten rapid development, especially the large-scaling of kiln, for refractory material, has put higher requirements on physical and chemical performance[1]. In the process of production, refractory material of kiln body suffer varying degrees of damage in some large and medium-sized precalciner kiln (PCK), which influence the running period of cement kiln, that become the major factor that restrict to enhance the operation efficiency of cement kiln. In a word, the working life of refractory material has become the leading role in kiln's system operation efficiency. Specially, the temperature change frequently in transition zone, now used magnesia-alumina spinel also is not easy to hitch kiln coating and has high thermal conductivity that make the outer wall of kiln barrel a higher temperature. Due to it expansion on heated, it neither help save energy, but also affect the normal operation of equipment[2]. Transition zone is the most easily damaged parts of kiln liner, the temperature change frequently in this part and sometimes kiln coating hitch, sometimes it drops. The lining brick often direct expose in the high temperature airflow and this part is also the highest concentration of stresses[3], especially the large kiln, which has large material flow and fast kiln rate, the lining brick suffer stronger destructive power. So the kiln lining of transition zone must have higher thermal shock resistance, higher anti-corrosion ability and abrasive resistance. For the material's performance requirements, it even exceeds the sintering zone. Therefore the preparation of the excellent performance special silicon mullite brick to adapt to the transition zone process characteristics of high strength and wear resistant.

EXPERIMENTAL

Main experimental raw material: High alumina bauxite chamotte, which is calcined from Shanxi rotary kiln; fused white corundum powder, corundum has high hardness, high melting point, stable chemical properties and nicer resistance to acid and alkali corrosive ability; silicon carbide, which is synthetized by petroleum coke and SiO_2, has high dissociation temperature and Moh's hardness. So we choose corundum and silicon carbide to strengthen base material, which is the conditions that products to obtain large volume density, high hot strength, corrosion resistance, wear resistant and other performance. The introduction of admixture alumina micro-powder is to improve product performance. The binder select spent pulping liquor of specific gravity 1.20 to 1.25 and the addition is 3-4 %. The chemical composition of raw materials are shown in Table-1.

Production technology: Each sample formulation is shown in Table-2. Use sulfurous acid spent pulping liquor as binder and the size composition principle is small at both ends and big in the middle and the proportion is shown in Table-2.

†Presented to the 6th China-Korea International Conference on Multi-functional Materials and Application, 22-24 November 2012, Daejeon, Korea

TABLE-1
CHEMICAL COMPOSITION OF RAW MATERIALS (wt %)

Raw material	Al_2O_3	Fe_2O_3	SiO_2	CaO	MgO	Na_2O	SiC
High alumina chamotte	85.24	1.45	9.21	0.52	0.1	0.29	-
Fused white corundum powder	99.6	-	0.11	-	-	0.04	-
Silicon carbide		1.5					90.9
Lime mud from Guangxi	33.2	1.15	49.3				
Alumina micro-powder	99.22	0.05	0.03	0.12	0.01	0.07	

Take variety of materials into the mixing rolls, use 400 t friction press to suppress standard brick, dry at 120 ºC, 24 h and sintering at 1450 ºC and keep 3 h.

TABLE-2
SAMPLES PROPORTION (wt %)

Sample number	A1	A2	A3	A4	A5
High alumina chamotte	60	60	60	60	60
Silicon carbide	15	15	15	15	15
Fused white corundum powder	15	13	13	11	9
Lime mud from Guangxi	10	10	8	8	8
Alumina micro-powder	0	2	4	6	8

Characterization: We detect apparent porosity and bulk density of the sample after firing accordance with the GB/T 2997-2000, accordance with the GB/T5072-1985 to detect the cold crushing strength after firing samples, accordance with the YB/T376.1-1995 to detect refractoriness under load after firing samples, accordance with GB/T18301-2001 to take refractory wear test at normal temperature, use XD-3-type diffraction analyzer a sample phase composition, use S-3000N electron microscope to analysis sample microstructure.

RESULTS AND DISCUSSION

Influence of alumina micro-powder at normal temperature: Table-3 lists the samples' performance with different add contents of alumina micro-powder at normal temperature. Table-3 also showed that the volume density of the sample was increased and then decreased with increasing alumina micro-powder. Porosity presented the trend of decreasing first and then increasing apparently. Strength data (Table-3) show that the compressive strength first increase and then decreased, but the strength of sample has obviously improved, which is better than the alumina micro-powder that hasn't added the alumina micro-powder. This is mainly smaller particle size of alumina micro-powder, the right amount of alumina micro-powder filling in the aggregate that play the role of micronized fill. This improved the structure of the matrix, has changed the constitute of matrix particles, has reduced the porosity and has improved density. Alumina micro-powder also promotes the sinter of the material substrate portion so that the compressive strength has significantly improved. Table-3 also showed that the optimal performance of adding amount of alumina micro-powder is 4 % and that the volume density is 2.94 g/cm^3 and the compressive strength has reached 190 MPa.

Influence of added alumina micro-powder to refractoriness under load: Further, the refractoriness under load and thermal shock resistance of samples were also measured. From Table-3 it can be seen that the refractoriness under load of the silicon mullite brick is above 1650 ºC. The sample A3 reaches 1700 ºC and the thermal shock resistance were more than 30 times. It was far from meeting the transition zone of the cement kiln. It was mainly because of the brick surface temperature of the cement kiln transition zone at about 1400 ºC was enough to resistance high temperature thermal shock. Due to Al_2O_3 and SiO_2 form the solid solution leads the materials to generate the right amount of micro-cracks, which can effectively buffer the thermal stress that has reached the purpose to improve the thermal shock resistance of materials. Because of addition of silicon carbide in the preparation of silicon mullite brick, which has low coefficient of thermal expansion and high thermal conductivity, made the thermal shock resistance of the product significantly improved. It greatly enhanced the service life of special silicone mullite brick. The thermal conductivity of the silicon mullite brick is low than alkaline product, therefore silicon mullite brick can used as the lining brick of transition zone and the nice thermal barrier effect makes the kiln shell outside surface temperature 100 ºC lower average than of basic brick, so the energy -saving is obvious.

Table-3 also showed the normal temperature adhesive resistance test results that as increasing alumina micro-powder, the amount of sample abrasion is first gradually decreased and then increased. The abrasion resistance of materials depend on sample's strength and structure of compactness. Strength and high density materials have higher wear resistance. The lowest amount of wear of sample A was 3.2 cm^3 at normal temperature. The lining of cement rotary kiln has long been rolling friction of materials. The application of silicon mullite played an important role in the transition zone of cement rotary kiln with its good abrasion resistance.

TABLE-3
EACH PERFORMANCE TEST RESULTS OF SAMPLES

Performance index		Samples				
		A1	A2	A3	A4	A5
w/%	Al_2O_3			>67		
	SiO_2 + SiC			>25		
Refractoriness under load (0.6%)/ºC		1650	1670	1700	1680	1675
Cold compressive strength (MPa)		138	156	190	186	165
Apparent porosity (%)		20.7	18.6	14.3	16.5	17.2
Volume density (g·cm^{-3})		2.44	2.65	2.94	2.82	2.72
Thermal shock resistance (1100 ºC water-cooling)/ times)		22	26	31	27	28
Cold wear-resisting/cm^3		6.22	4.56	3.2	4.2	4.3
Thermal conductivity1000 ºC w/m·k		2.35	2.12	1.72	1.85	1.96

XRD and SEM analysis of samples: XRD pattern is shown in Fig. 1 of the sample A3. The analysis results shown that the main crystalline phases are mullite, carborundum and a small amount of glass phase. The hardness of these crystal phase are high, which ensure the dense and high strength of the sample. Adding alumina micro-powder will lead to SiO_2 in carborundum with highly activity that conduce to form a relatively dense network-shaped mullite binder phase in the matrix that make the product crushing strength and the volume density increased significantly.

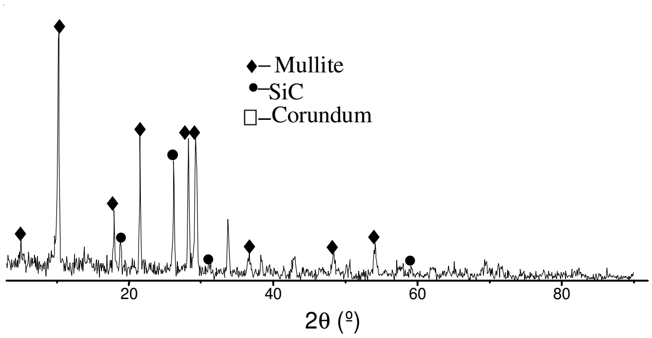

Fig. 1. XRD pattern of sample A3

Fig. 2a is the SEM photo of A0 sample that calcined at 1450 ºC. As it was not added alumina micro-powder, the structure is relatively loose, exist large pores, the bonding strength of the particles and the matrix is low, generating the columnar mullite. Since the mullite has little content, it cannot offset the contraction that caused by the liquid phase at high temperature. So it has large contraction and worse thermal shock resistance after buring. Fig. 2b is the fracture of macroscopic morphology of A3 sample calcined at 1450 ºC. It can be seen continuously distributed of alumina mirco-powder and the generated mullite in sample. Silicon carbide particles and mullite closely combine enhanced the ceramic matrix-bound that made the internal structure more compact and has improved the strength. Due to the addition appropriate amount of alumina micro-powder, that formed a large number of short columnar mullite crystals. At a certain temperature, the surface of the sample form a solid glaze membrane. Brick surface form a continuous SiO_2 dense layer to prevent erosion of the gas and the melt, so that make the sample anti-corrosion and enhance abrasion resistance. Combined with the high thermal conductivity and low thermal expansion coefficient of silicon carbide, the heat shock resistance stability of product is an ideal refractory of transitional zone. But when the micro-powder exceeds the optimum adding amount, despite the micro-powder added can improve the strength of the sample, due to the high temperature has generated excessive mullite that produce larger expansion so that thermal shock resistance of the material will deteriorate.

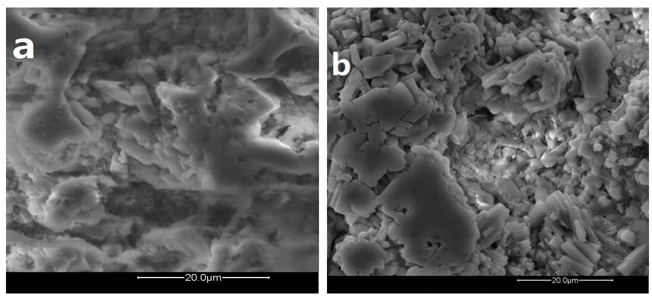

Fig. 2. SEM analysis of samples; a -sample A0; b-sample A3

Conclusion

With the increasing amount of alumina micro-powder, the volume density and compressive strength of the sample first increases and then decreases. When the amount of alumina micro-powder is 4 %, the sample has the best comprehensive performance. And the volume density reached 2.94 g/cm^3, the thermal shock resistance exceed 30 times, refractoriness under load reached 1700 ºC. It means that sample's performance improved.

XRD analysis showed the sample that add 4 % alumina micro-powder that the main phases are mullite, corundum and silicon carbide. As these crystalline phases have high hardness, which laid a foundation for the preparation of dense and high-strength silicon mullite brick.

ACKNOWLEDGEMENTS

This project was supported by National Natural Science Foundation of China (51002002).

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