

Influence of milling time on the performance of ceramic ball grinding media prepared from refractory waste

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Aluminosilicate refractory waste was used to prepare high performance ceramic ball grinding media with 75% Al_2O_3 . The influence of milling time on the particle size of raw materials, the sintering temperature, and performance of ceramic balls were studied. Results show that with increasing milling time the particle size of the raw materials decreased, resulting in a decrease of ceramic ball sintering temperature and an increase in wear resistance and volume density. The wear rate and volume density of balls prepared by milling the materials for 72 h were 0.01886 %/h and 3.374 g/cm³, respectively.

Key words: *aluminosilicate refractory waste; particle size; ceramic grinding media; wear resistance*

1. Introduction

Large amounts of refractory wastes are discarded during the production and application of refractories, and after refractories reach the end of their service life. In particular, more than 3 million tons of refractory waste is produced per year in China [1]. The majority of such refractory waste is accumulated inside or outside factories, causing severe problems with storage, disposal, and the environment, while only small amounts of the waste are now used as raw materials for refractories, cement, glass, ceramics, steels, etc., because of the potential undesirable influence of the waste on the quality of the product and its large crystal and high hardness, which make it difficult to mill and lead to increasing recycling costs. At present, reusing refractory waste

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has been widely studied, because of the scantiness of natural mineral resources, high landfill costs and the aim to reduce environmental pollution [1–4]. Since there are few reports on reusing aluminosilicate refractory waste as raw materials for high performance ceramics [5], the present paper reports the influence of raw material milling time on the sintering temperature and performance of as-prepared ceramic balls.

2. Experimental procedure

Alumina ceramic balls with 75% Al_2O_3 were prepared by reusing aluminosilicate refractory waste as a raw material. The waste (84.4 wt. %) was mixed with CaO – MgO – Al_2O_3 – SiO_2 quaternary system fluxing agents, then milled for 6 h, 12 h, 24 h, 48 h, and 72 h at a weight ratio of powder:media:water = 1:(3–6):(1–1.5) and dried at 100 °C for 24 h, respectively. The obtained powders were shaped by cold isostatic pressing at 65 MPa for 3 minutes. The ceramic blanks were sintered at 1270 °C, 1285 °C, 1300 °C, 1325 °C, or 1350 °C for 2.5 h. The chemical compositions of the waste and ceramic balls are given in Table 1. The particle size distributions of the original waste and obtained powders were measured by a LS-POP (III) OMEC particle size analyser. The water absorption ratio of the balls was measured according to Chinese National Standard GB/T3810.3-1999. The volume density of the balls was measured according to the Archimedes principle. The microstructure of ceramic balls was observed with a JSM-5610LV scanning electron microscope (SEM).

Table 1. Chemical composition of refractory waste and ceramic balls

Composition	Al_2O_3	SiO_2	TiO_2	CaO	MgO	Fe_2O_3	Other
Waste [%]	80.92	12.14	2.91	0.61	0.30	2.92	0.2
Ceramic ball [%]	75.01	17.26	2.18	1.39	0.99	2.48	0.69

A kind of alumina ceramic ball with 90% Al_2O_3 was selected as the reference ball, which was an imported alumina ceramic ball grinding media used in the architectural and sanitary ceramics industries. The prepared balls and reference ball were milled together with corundum media in a ball mill for 24 h, then dried at 300 °C for 30 minutes and weighed. The wear rate of the balls was calculated from the formula:

$$W = \frac{m_0 - m_1}{24m_0} \times 100\% \quad (1)$$

where W is the wear rate of the ceramic balls (%/h), m_0 and m_1 are the ball masses before and after milling.

3. Results and discussion

3.1. Influence of milling time on the particle size of raw materials

Figures 1 and 2 show the particle differential distributions of the original waste and raw materials, respectively, milled for various times. Figure 1 indicates that the average particle size and maximum particle size of the original waste was 38 μm and 169 μm , respectively. Figure 2 shows that with increasing milling time the particle distribution of the raw materials became narrow and the particle size decreased.

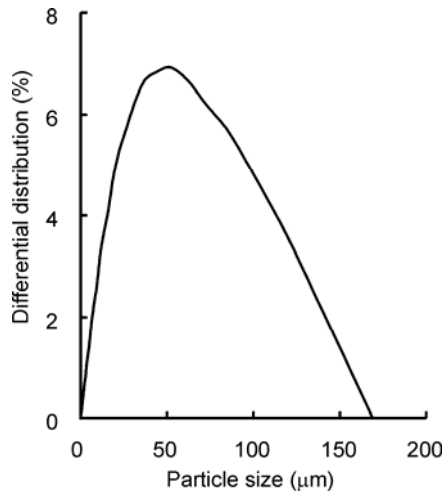


Fig. 1. Particle size differential distribution of the original waste

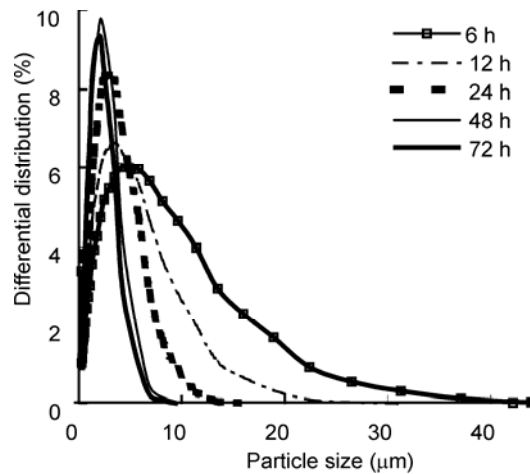


Fig. 2. Particle size differential distributions of the raw materials, with milling time changed from 6 to 72 hours

The average particle sizes of raw materials milled for 6 h, 12 h, 24 h, 48 h, and 72 h were 3.15 μm , 2.50 μm , 2.30 μm , 1.64 μm , and 1.42 μm , and the maximum particle sizes were 52 μm , 31.3 μm , 15.98 μm , 9.64 μm , and 9.64 μm , respectively. The results suggest that the waste was easily milled at the experimental ratio of raw materials:media:water of 1:(3–6):(1–1.5). The water ratio increased with milling - the longer the milling time, the more water was needed.

3.2. Influence of raw material milling time on ceramic ball sintering temperature and performance

The influence of raw material milling time on ceramic ball sintering temperature, water absorption, wear rate, and density are shown in Table 2, Figs. 3 and 4. The results indicate that when the raw material milling time was the same, the ceramic balls water absorption and wear rate both initially decreased and then increased (Table 2,

Fig. 3a)), while the volume density initially increased and then decreased (Fig. 3b). The sintering temperature was optimum when water absorption and wear rate were the lowest and the volume density was the highest.

Table 2. Influence of milling time on ceramic ball sintering temperature .

Milling time [h]	Water absorption ratio [%]				
	1270 °C	1285 °C	1300 °C	1325 °C	1350 °C
6	1.7353	0.2758	0.0083	0.0002	0.0015
12	0.1684	0.0084	0.0004	0.0006	0.0469
24	0.1171	0.0031	0.0004	0.0006	0.0342
48	0.0219	0.0004	0.0004	0.0008	0.0015
72	0.0010	0.0004	0.0007	0.0011	0.0290

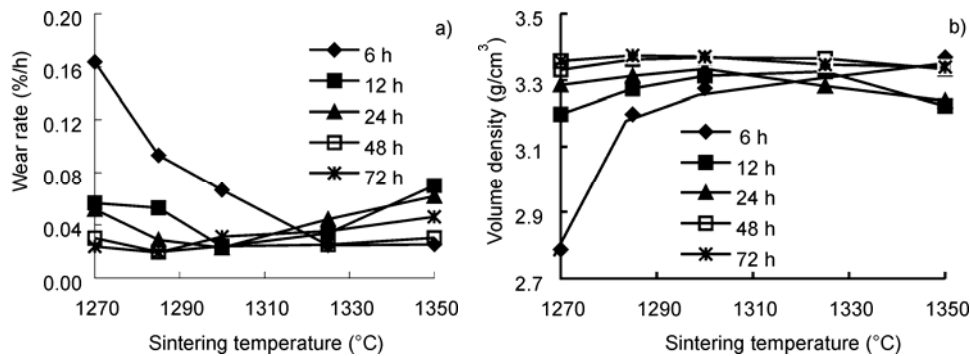


Fig. 3. Temperature dependence of wear rate (a), and density (b) of ceramic balls

During firing, small pores moved toward large pores or excluded through the crystal boundary. On the other hand, the granules grew quickly, leading to some enclosed pores in the ceramics. At an elevated firing temperature, the pores inside ceramics became smaller, resulting in a decrease in water absorption and an increase in density before the temperature reached the optimum sintering temperature. When the firing temperature was higher than the optimum sintering temperature, the air pressure in the enclosed pores increased quickly, leading to an increased pore size and porosity, and to an expansion of the ceramics resulting in an increase in water absorption and a decrease in volume density. With decreasing pore size and porosity and increasing volume density, the ceramic ball wear rate decreased before the sintering temperature reached the optimum. On the contrary, the ceramic ball wear rate increased and wear resistance decreased with increasing sintering temperature after the temperature rose above the optimum sintering temperature. Figure 4a shows that the optimum sintering temperature of balls prepared from materials milled for 6 h, 12 h, 24 h, 48 h, and 72 h was 1325 °C, 1300 °C, 1300 °C, 1285 °C, and 1285 °C, respectively, suggesting that the sintering temperature decreases with increasing milling time. When sintered at the optimum tempera-

ture, both ball density and wear resistance increases (wear rate decreases) with increasing milling time (Fig. 4b). The density and wear rate of the compared sample were 3.64 g/cm^3 and 0.0537 %/h , respectively. The volume density and wear rate of the balls prepared from raw materials milled for 24 h and sintered at 1300°C were 3.334 g/cm^3 and 0.02272 %/h , respectively, and for the ceramic balls prepared from raw materials milled for 72 h and sintered at 1285°C were 3.374 g/cm^3 and 0.01886 %/h , respectively. These results indicate that the wear resistance of the prepared ceramic balls was higher than that of the reference balls. The wear rate of the prepared balls was only $(1/3)-(1/2)$ that of the reference ball.

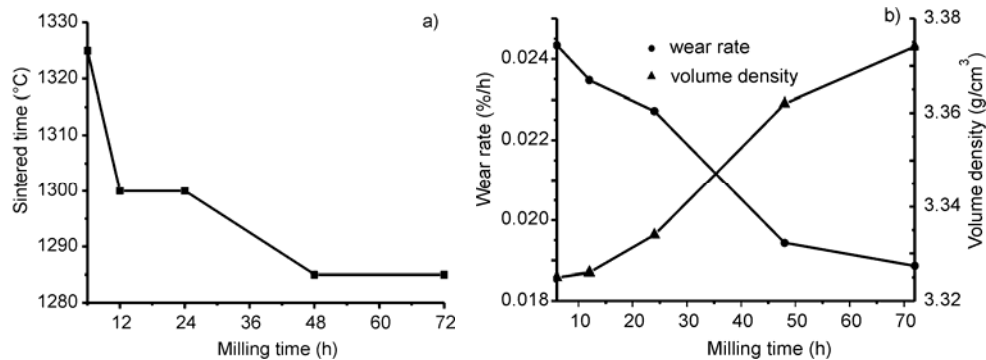


Fig. 4. Influence of milling time on ceramic ball sintering temperature, wear rate, and density

Experimental results indicate that the sintering temperature of the prepared ceramic balls decreases and performance increases with increasing raw material milling time, however increasing milling time increases ceramic ball production cost. Considering the ceramic ball economy performance, 24 hours is the optimum milling time.

3.3. Microstructure of ceramic balls

The cross-sections of ceramic balls without any treatment were analysed by SEM, and the results are shown in Fig. 5. Figure 5 shows that the average grain sizes of the balls prepared from raw materials milled for 6 h, 24 h, and 72 h were about $10 \mu\text{m}$, $5-6 \mu\text{m}$, and $3-4 \mu\text{m}$, and the pore diameters were about $10 \mu\text{m}$, $3 \mu\text{m}$, and $2 \mu\text{m}$, respectively. SEM microstructures indicate that with increasing raw material milling time, the as-prepared ball grain size, pore size, and porosity decrease, especially when the milling time is increased from 6 h to 24 h, which explains the decrease in balls wear rates and the increase in volume densities.

The prepared ceramic ball surfaces after milling with other balls and ultrasonic cleaning were observed under SEM, and the results are shown in Fig. 6. Comparing Fig. 6 with Fig. 5 indicates that the milled surface grain size was smaller than that inside the ceramics, suggesting that the balls were mainly worn by transcrystalline fracture.

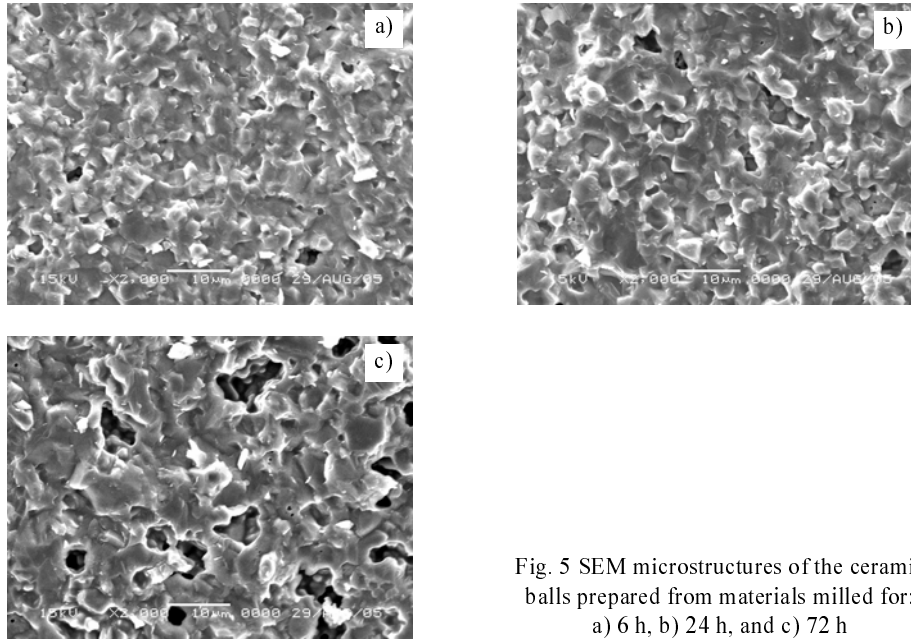


Fig. 5 SEM microstructures of the ceramic balls prepared from materials milled for: a) 6 h, b) 24 h, and c) 72 h

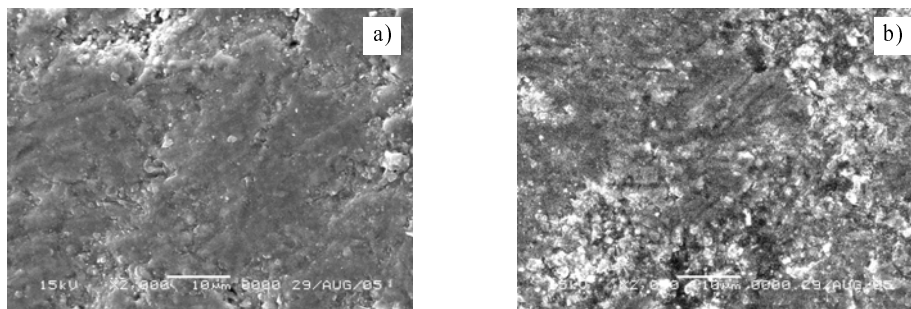


Fig. 6. SEM microstructures of the surfaces of ceramic balls prepared from materials milled for: a) 6 h, b) 72 h

During the milling process the large grains on the surface were first broken and worn off, and the remains were still combined as original adjacent crystals, then part of the remaining grain was further broken, and the remaining grains became smaller. The transcrystalline fracture explains the low wear rate of the prepared ceramic balls.

4. Conclusions

High performance alumina ceramic balls with 75% Al_2O_3 were prepared from aluminosilicate refractory waste. The particle size of aluminosilicate refractory waste decreased with increasing milling time, resulting in the decrease of sintering temperature and an increase in the density and wear resistance of the as-prepared ceramic

balls. The optimum milling time at the weight ratio of powder:media:water of 1:(3–6):(1–1.5) was 24 hours. The wear rate of the reference ball, with 90% Al_2O_3 , was 0.0537%/h, and the wear rate of the balls prepared from waste milled for 24 hours (and sintered at 1300 °C) and 72 h (sintered at 1285 °C) was 0.02272 %/h and 0.01886 %/h, respectively. The wear mechanism of the prepared balls was transcrystalline fracture.

Acknowledgements

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