Powder Technology 303 (2016) 27–32

Contents lists available at ScienceDirect

Powder Technology

journal homepage: www.elsevier.com/locate/powtec

Effect of rare-earth Lu_2O_3 on the wear resistance of alumina ceramics for grinding media

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ARTICLE INFO

Article history:
Received 19 January 2016
Received in revised form 4 September 2016
Accepted 7 September 2016
Available online 09 September 2016

Keywords:
Alumina ceramics
Grain refinement
Lutetium oxide
Wear resistance
Grinding media

ABSTRACT

Wear resistance of grinding media is crucial for the quality of powders. The purpose of this work is to improve the wear resistance of grinding medium in an Al_2O_3-$CaCO_3$-SiO_2-MgO-Lu_2O_3 (ACSML) system. The effect of Lu_2O_3 content on bulk density and wear rate is discussed. The phase composition and microstructure of this material are analyzed. The results show that adding a trace amount of Lu_2O_3 to alumina can evidently improve wear resistance by grain refinement and enhancing density. The wear rate of grinding medium is as low as 0.00044‰, and the wear resistance has been improved by 31% than the sample without Lu_2O_3. However, excessive Lu_2O_3 can lead to deterioration of wear resistance, which due to grain growth and existence of $Al_5Lu_3O_{12}$.

1. Introduction

The processing of ball milling is very important in many industries like chemical, mineral, metallurgy, silicate, pharmaceutical, pesticide and food. Grinding media are indispensable material during ball milling. The wear resistance of grinding media is crucial to product quality. There is a strict limit to the content of contaminant (contaminant is unwanted material that polluted the sample) in some products, especially in food and pharmaceutical industry. Control of contaminant is a key factor in guarantee for drug safety. The United States Pharmacopeia divides impurities into three categories and the lowest content of three is <0.0005% [1]. Besides, in silicate industry, demands for high-purity alumina ceramic have increased with developing technology, such as insulator used for high pressure and extra-high voltage systems, electric component with low dielectric loss and good surface finish, ceramic tube used for lamps, and precision components in aerospace domain. The contaminant content is the key issue in preparing high-purity alumina ceramic, too. However, grinding media are one of the main pollution sources. So it is very important to improve the purity and wear resistance of the grinding media.

The current research of grinding media focuses on ball size [2], shape design, material choice [3], the kinetics of grinding media wear, effect of grain size and porosity, wear mechanism, etc. Numerous studies have shown that polycrystalline alumina with sub-micrometer grains has good wear resistance [4]. In order to optimize wear resistance, the alumina grain size should be small, regardless of the alumina content [5]. The wear resistance of polycrystalline alumina materials not only has a strong dependence on the microstructure and on the average grain size [6,7], but also is relevant to the chemical composition of the ceramic [8,9]. Besides, there are a lot of researches on the effect of rare earth on the performance of ceramics. The researchers discovered that rare earth oxides can reduce the sintering temperature [10], refine grains [11,12], increase the size of grain boundary grooves [13], and improve density [14,15], strength [16], hardness [17], thermo stability [18], creep resistance [19], etc. Nevertheless, the effect of rare earth on wear resistance mechanism has not been systematically investigated, especially for high alumina doped with Lu_2O_3. In the present study, we investigate the effect of Lu_2O_3 on wear resistance of high alumina ceramic (>98 wt%). Density, wear rate, phase composition and microstructure of this material are investigated.

2. Experimental

High alumina ceramic was prepared in an Al_2O_3-$CaCO_3$-SiO_2-MgO-Lu_2O_3 (ACSML) system. Samples were prepared using a commercial alumina powder (>99.8% purity and a mean particle size of 0.65 μm). The alumina powder was mixed with magnesia, calcium carbonate, silicon dioxide and lutetium nitrate hydrate. Mixed additions of SiO_2 and MgO allow a good densification and a good homogeneity of the microstructure [20,21]. The sum of Al_2O_3 and Lu_2O_3 was 99 wt%. The content of $CaCO_3$ and MgO is the same that is 0.33 wt%. The content of SiO_2 is 0.34 wt%. When the Lu_2O_3contents were 0, 0.0001, 0.001, 0.01, 0.1 and 1 wt%, the samples were referred to as Samples 1, 2, 3, 4, 5, and 6. The

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http://dx.doi.org/10.1016/j.powtec.2016.09.018

powders were ball-milled for 24 h, and then shaped to obtain green compacts (sphere of 30 mm in diameter) by cold isostatic pressing at 100 MPa. They were sintered at 1475 °C for 1 h in air. After these processes, bulk density and wear rate were tested. Bulk density was measured by the Archimedes method. Wear rate was tested in accordance with Chinese Building Materials Industry Standard JC/T 848.1-2010 (alumina grinding ball). The process is as follows: Weigh a sample (M_1) and measure diameter (D_x). Then put about 1 kg samples and 1 L unfiltered water into a polyurethane pot, and then be milled for 24 h. The speed of ball mill is 80 rpm. Dry it and weigh again (M_2). Wear rate is calculated by the equation:

$$W = KD(M_1 - M_2/M_1) \tag{1}$$

In this equation, W is wear rate (‰); K is a constant (4.17×10^{-4} mm^{-1}); D is the mean diameter (mm) of samples; M_1 is the weight of before wear (g); and M_2 is the weight of after wear (g). Based on the industry standard, wear rate of alumina grinding ball (alumina content is about 99 wt%) should be <0.15‰.

Phase composition of samples was initially identified by comparing the X-ray powder diffraction (Cu K_α) patterns with the standard chart. The test was carried out in an "X'Pert PRO" multi-purpose X-ray diffractometer (PANalytical B.V., Almelo, Netherlands) with 40 kV voltage and 40 mA current. X-ray patterns were taken by measuring 2θ from 5° to 90°, at a step size of 0.02° and a dwell time of 5 s per step. The results of the powder diffraction patterns were analyzed with X'Pert High Score Plus software. The microstructure of the sintered samples was characterized using field emission scanning electron microscope (FESEM) equipped with energy dispersive spectroscopy (EDS), model S-4800, Hitachi, Japan makes. The samples were coated with golden prior to examination. In order to explore the mechanism, an additional experiment was done. Put Lu_2O_3 powder to coat on single-crystal alumina. And then, they were sintered at 1600 °C for 3 h. The interface between Al_2O_3 and Lu_2O_3 was observed by FESEM.

3. Experimental results

3.1. Wear rate and bulk density

Fig. 1 shows the relationship among Lu_2O_3 content, wear rate, and bulk density. The curves of wear rate and bulk density vary parabolically with the increase of Lu_2O_3 content. With the increase of Lu_2O_3 content, the bulk density curve increases first and then decreases, but the wear rate curve decreases first and then increases. When Lu_2O_3 content is <0.01%, the density and wear resistance of samples are better than that of Lu_2O_3-free sample.

Wear rate of 99% alumina grinding ball should be <0.15‰ according to Chinese Building Materials Industry Standard. The wear rate of Sample 1 without Lu_2O_3 is 0.00064‰. Wear rates of samples fall sharply with the increasing of Lu_2O_3 content. The wear rate of Sample 4 reaches the minimum (0.00044‰). The wear resistance of Sample 4 has been improved by 31% than the sample without Lu_2O_3 and been enhanced by 284 times over a product with good resistance on the market (its wear rate is 0.125‰.). However, the wear rate of Sample 5 rises rapidly and is over than that of Sample 1.

The results indicate that trace amounts of Lu_2O_3 can effectively improve the wear resistance and bulk density of alumina ceramic. The appropriate adding quantity is 0.001–0.01 wt%. Excessive Lu_2O_3 will result in poor wear resistance of the alumina ceramic.

3.2. Phase composition

Sample 1 is Lu_2O_3-free sample. The wear resistance of Sample 4 is the best. The wear resistance starts becoming bad from Sample 5. And the wear resistance of Sample 6 is the worst. So the Samples 1, 4, 5, and 6 were chosen for testing. The phase composition of Samples 1, 4, 5, and 6 was analyzed by XRD. Fig. 2 shows the XRD results. In Samples 1, 4, and 5, Al_2O_3 is the main crystal phase and $MgAl_2O_4$ is the secondary phase. In Sample 6, $Al_5Lu_3O_{12}$ appears in addition to Al_2O_3 and $MgAl_2O_4$.

3.3. Microstructure

Samples 1, 4, 5, and 6 were cut and polished for FESEM testing. The microstructures of the cross-section are shown in Fig. 3. Submicron alumina ceramic exhibits substantially higher wear resistance compared to the larger grain size of alumina [22] (the grain size of submicron alumina ceramic is between 100 nm and 1 μm). In Fig. 3, average grain sizes of the four samples are <1 μm. They belonged to submicron alumina ceramic. With the increasing of Lu_2O_3 content, the average grain size decreases firstly and then increases.

A comparison among four samples shows that the average grain size of Sample 4 with 0.01 wt% Lu_2O_3 is the smallest and the microstructure

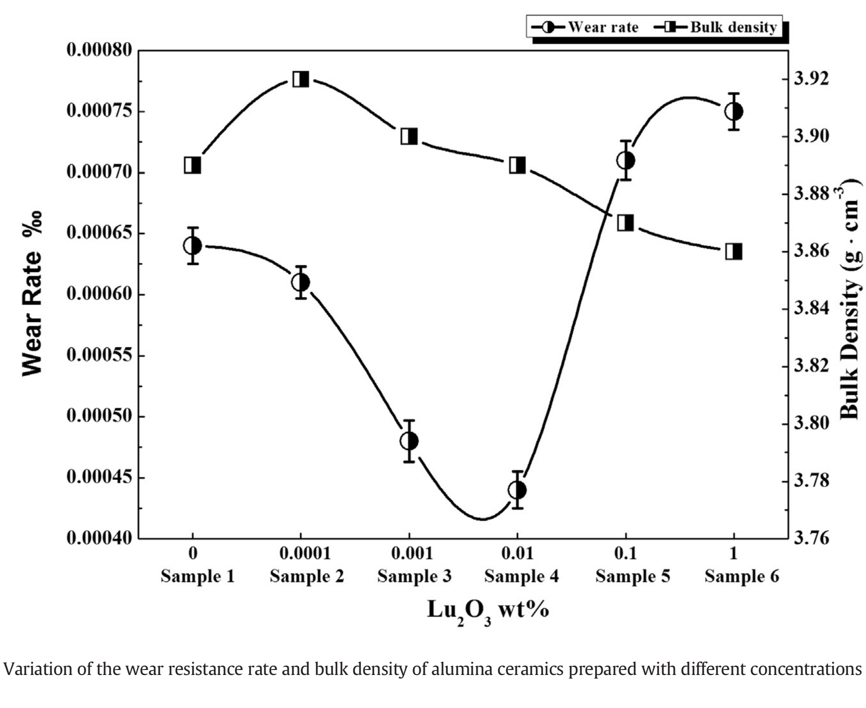

Fig. 1. Variation of the wear resistance rate and bulk density of alumina ceramics prepared with different concentrations of Lu_2O_3.