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Characterization of coarse particles in alumina powders by a wet sieving method

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Abstract

The wet-sieving method was applied to determine the content of coarse particles in commercial alumina powders by four independent research organizations participated in the Round-Robin Testing. The results showed that those powders clearly contain 700 to several thousands ppm of coarse particles with the size >25 μm, whereas they could not be detected with the conventional particle size analyzer because of their low concentration. SEM and polarized light microscope observations revealed that the coarse particles contained several types of aggregates which survived during the production processes of alumina by the Bayer process.

Keywords: Al_2O_3; Coarse particles; Sieving; Testing

1. Introduction

Raw powder is a critical factor affecting the microstructure and properties of resultant ceramics[1] and needs to be fully characterized for producing ceramics of high quality. The coarse particles such as aggregates and agglomerates are very important characteristics in raw powder.[2] Papers showed their very detrimental effects on densification and properties of ceramics.[3] The coarse particles create large defects in the microstructure,[4–6] one of which may behave as a fracture origin, reducing the strength of ceramics.[7] They also reduce the densification rate as much as a factor of 10 or causes deformation of products.[8] Methods for characterizing coarse particles, agglomerates or aggregates, have been proposed, but they provide no direct information on their quantity.[10,11] Clearly an accurate characterization tool for coarse particles is crucial for commercial production of ceramics.

Accurate measurement of coarse particles has been difficult with conventional equipment, however, at least in the highly demanding level required in industries. In industries, the rigorous grinding is the most practical way to ascertain their elimination in the raw powders.[12] However, the results obtained are often far from satisfactory. There is a high demand in industry to develop a reliable method to evaluate the coarse particles in high accuracy, preferably with a simple apparatus and procedure. The wet-sieving method appears to be the most simple and direct way.

This paper presents a method for quantitatively evaluating the particles of extremely large size contained in ceramic raw powders, using industrial grade alumina powders as a model case. Four independent research organizations participated in the Round-Robin Testing

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for the examination in accordance with the same experimental procedure. The method is based on the quantitative wet-sieving, since this is the most direct method to evaluate large particles in powders.[13] SEM and polarized light microscope are also applied for examining the structure of large particles separated.

2. Experiments

Commercial alumina powders from three sources were used in this study (Table 1). For the experiment, 1 kg of each powder was weighed and mixed in a blender for 1 h in order to avoid the unpredictable variation of the measurement between the testing lots sampled from the powder. Particle size distribution of each raw powder was measured with a X-ray sedimentation-type particle size analyzer (Sedigraph 5100, Micromeritics Instrument Corp., USA). For the quantitative analysis of coarse particles in each raw powder, approximately 30 g of the powder was placed into a glass bottle of known mass to within 0.1 mg and dried in an electric oven at 110 °C for 2 h. After drying, the samples were cooled in a desiccator and were held equilibrated at room temperature for at least 10 min. The weight of the dried powder, Wp, was determined by subtracting the weight of a glass bottle from the total weight measured to within 0.1 mg.

The powder was carefully put into 200 ml of the dilute aqueous solution of sodium hexamethaphosphate (125 ppm) in a Teflon beaker (250 ml) while stirring continually by using a magnetic-stirrer. Ultrasonic agitation was then conducted to the suspension for 5 min using a ultrasonic homogenizer of 300 W in order to promote the dispersion of the powder in a solution. For the ultrasonication, the tip of the submersible ultrasonic horn was set at approximately half the depth of the suspension.

The method of separation of coarse particles is illustrated in Fig. 1. Sieves with openings of 25 and 53 μm of known weight (Ws) were used for the experiment as shown in Table 1. The sieve was set on the water bath with the mesh screen of the sieve just under the water surface. The sample suspension prepared was poured onto the sieve slowly so that none of the suspension was lost. The suspension in the beaker was transferred onto the sieve completely by rinsing the beaker with distilled water. Subsequently, a large amount of distilled water (approximately 10 l) was poured onto the sieve so that only coarse particles were left on the mesh screen. The sieve with coarse particles was weighed to within 0.1 mg (Wscp), after dried at 110 °C for 2 h and cooled in a desiccator. The content of coarse particles, X, in the raw powder was then determined according to the equation:

$$X = (Wscp - Ws)/Wp \times 10^6 \ \text{(ppm)}$$

The shape and structure of coarse particles separated were examined with SEM and polarized light microscope. For the examination with polarized light microscope, the coarse particles were placed on a glass slide and a few drops of methyleneiodide were added to achieve high optical transparency.

Table 1
Alumina powders and the size of opening of the sieve used for separation of coarse particles

Grade	Alumina-I	Alumina-II	Alumina-III
Crystalline phase	α	α	α
Al_2O_3,%	99.9	99.8	99.8
D_{50}, μm	1.73	2.71	0.47
Opening of the sieve, μm	25	25	53

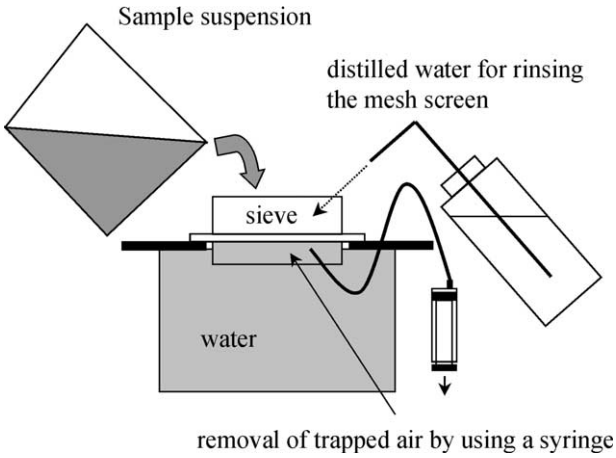

Fig. 1. The method of separation of coarse alumina particles.

3. Results

Fig. 2 shows the particle size distributions of alumina raw powders measured with a X-ray sedimentation-type particle size analyzer. The average particle sizes of the raw powders measured are 1.73, 2.71 and 0.47 μm, respectively for powders I, II and III as listed in Table 1. However, large particles with the size <20 μm were hardly detected in these powders as presented in Fig. 2. As is the case of almost all types of analyzer, this analyzer provides information on particles of major size range, but not of extreme size region.[13] The error noise of any analyzer exceeds an equivalent coarse particles of a few thousand ppm.

Table 2 and Fig. 3 show the contents of coarse particles in these powders determined through four independent organizations. The sieve with an opening of 25 μm was used for separation for powders I and II, whereas the sieve of 53 μm was used for powder III. Those powders clearly contain 700 to several thousands ppm

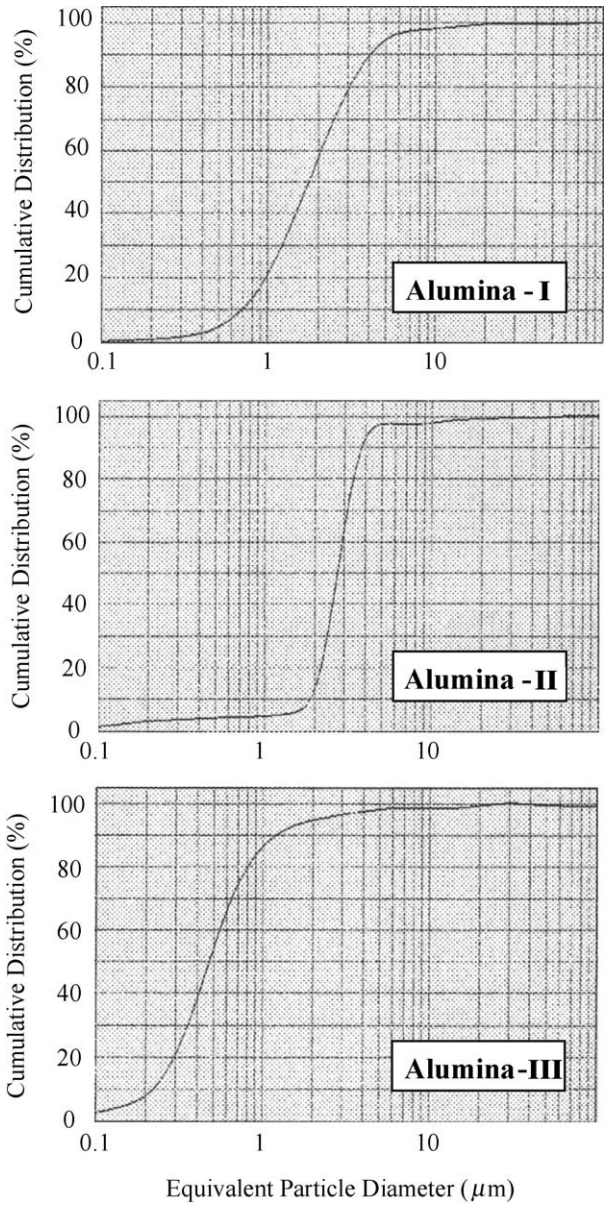

Fig. 2. Particle size distribution of each alumina powder measured by X-ray sedimentation particle size analyzer.

of coarse particles of the size <25 μm. Almost the same results were obtained among the organizations participated in the Round-Robin testing for all powders, except a slight deviation in some cases. Clearly, the method proposed in this study can be applied for evaluating the amount of coarse particles in ceramic raw powders quantitatively.

Fig. 4(a)–(f) show the SEM micrographs of coarse particles found in alumina powder I. The coarse particles separated are hard agglomerates or "aggregates". Observation of more than 150 coarse particles showed that they could be classified into several types;

Type-1: Porous vermicular aggregates composed of

Table 2
The amount of separated coarse particles in commercial alumina powders measured by four independent organizations

Powder	Alumma-I	Alumma-II	Alumma-III
Opening of screen	25	25	53
Content	ppm	ppm	ppm
Organizations			
A	3023	963	6511
	2052	786	4382
			3706
B	2251	781	5524
	2605	949	5083
	2375	762	5218
C	2341	721	3234
	2464	1212	3101
	2563	1213	3549
D	2724	624	3102
	2850	774	3569
	3034	638	3550
Average	2571	857	4002
S.D.	301	195	853

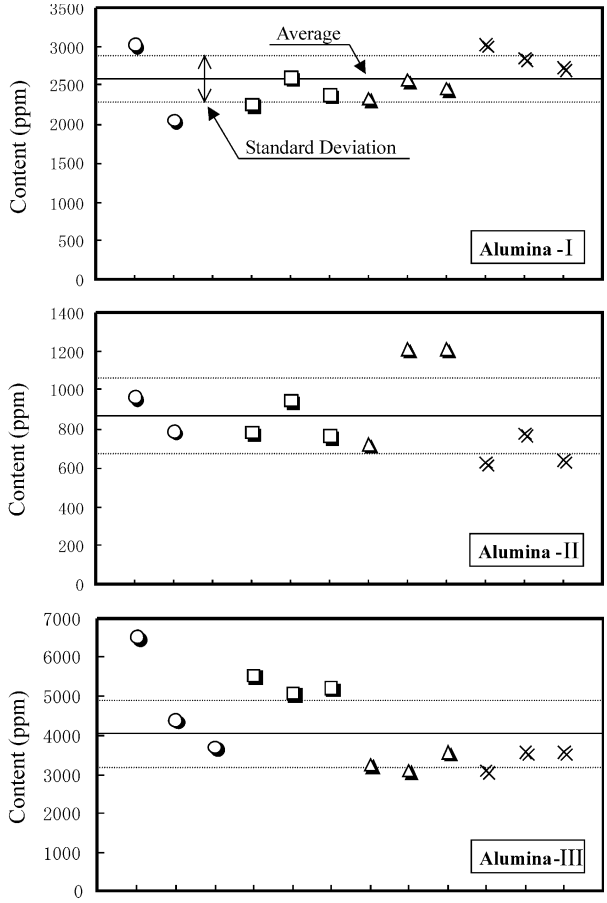

Fig. 3. The measured amount of coarse particles in commercial alumina powders.

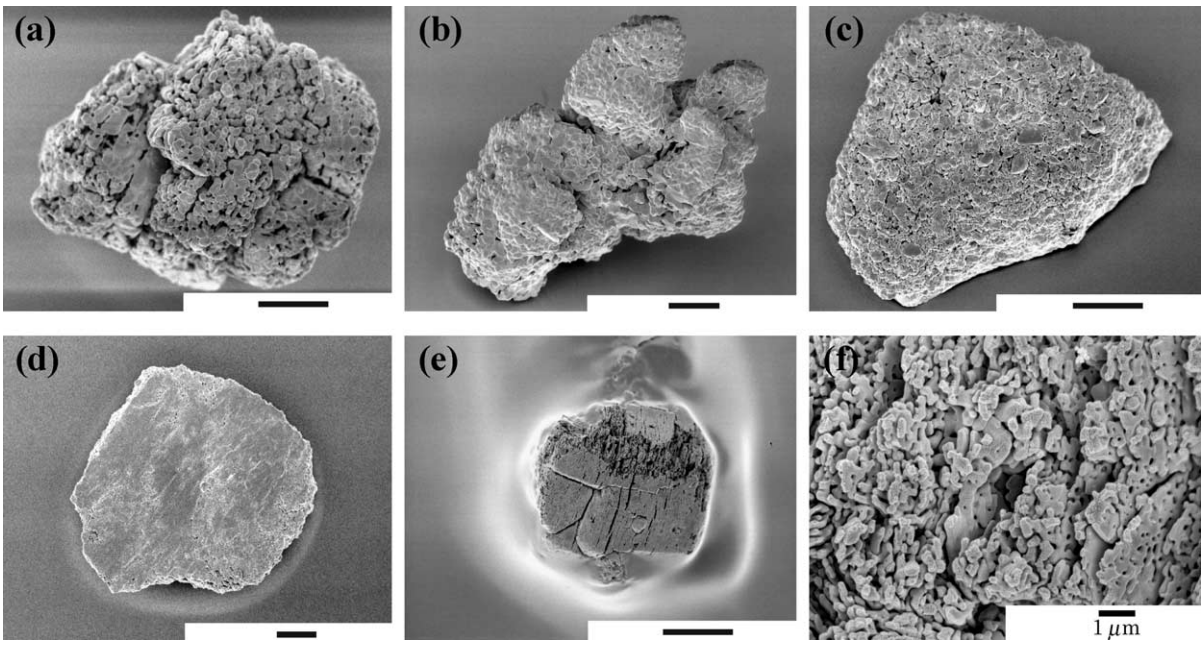

Fig. 4. SEM photographs of separated coarse particles found in alumina powder-I: photographs from (a) to (e) show various types of coarse particles (bar; 10 μm), and (f) shows the high magnification image of the Type-5 aggregate.

particles with the size of several micrometers [Fig. 4(a)].
Type-2: Dense aggregates composed of particles of several micrometers [Fig. 4(b)].
Type-3: Dense aggregates in which fine particles of submicron size fill the spaces between the particles of several micrometers [Fig. 4(c)].
Type-4: Aggregates apparently sintered to form a dense body [Fig. 4(d)].
Type-5: Aggregates consisting of several regions, within each of which elongated plate-like particles are oriented to a specific direction [Fig. 4(e),(f)].

The size of coarse particles ranged 25–80 μm. The SEM micrograph taken at high magnification for the Type-5 aggregate [Fig. 4(f)] shows the porous structure of plate-like particles. Type-1 is the most frequent and is about 50% of all coarse particles.

Figs. 5 and 6 show the crossed-polarized light micrographs of coarse particles for two angles of rotation. A characteristic pattern of changing color is noted with rotation of the particle in this micrograph. For a particle, the small regions mutually change in color with the rotation at every 45°. Clearly, the coarse particle consists of several regions, for which primary particles align to different directions like Type 5 aggregates. Coarse particles which are composed of primary particles with nearly the same orientation are also found. They showed almost the same color for entire region for any angle of rotation as presented in Fig. 6.

4. Discussion

The present results conducted by four independent activities clearly demonstrated that the wet-sieve analysis is a reliable method to determine the content of coarse particles contained in commercial alumina powders. The method requires no special equipment and is easily applicable for unexperienced researchers. It may provide a standard tool for quantitatively determining the content of coarse particles in ceramic raw powders.

The structure of coarse particle is responsible for the slight difference in results among organizations. As shown in Fig. 4, the coarse particles in commercial alumina powders were essentially aggregates of several types. Breaking down of these coarse particles into small particles should depend on the methods or conditions for grinding, suggesting that the small discrepancy of results can happen by the different output power of ultrasonic homogenizer and/or location of the horn in the suspension. Well stirring during ultrasonic homogenization should be very important, since coarse particles tends to settle on the bottom of the beaker, where the ultrasonic energy is low.

The minimum measurable value is approximately 30 ppm for coarse particles in ceramic raw powder in the present method. The value can be reduced further if large quantity of raw powder is used for analysis. In a common commercial balance, the minimum reading and the maximum sample weight are 0.1 mg and 200 g, respectively. With a specimen of 100 g, and practically

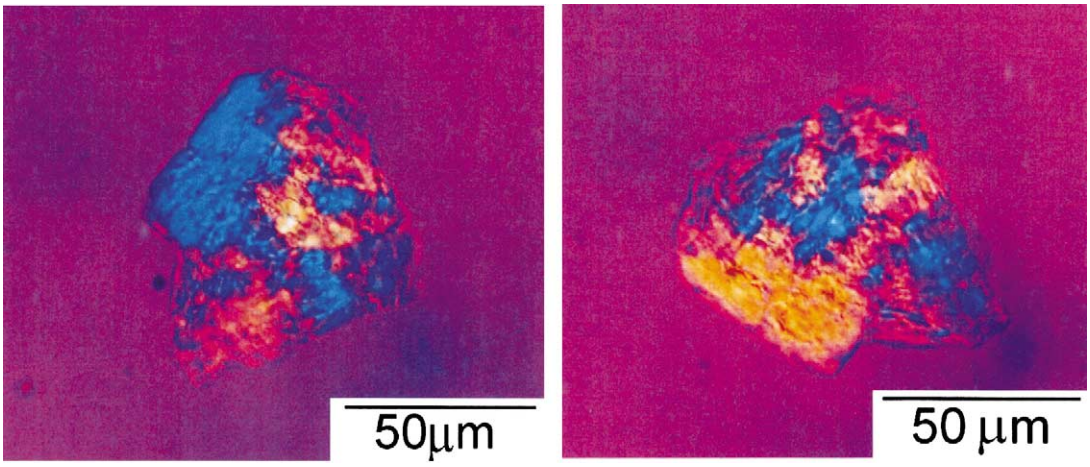

Fig. 5. Crossed-polarized light photographs of separated coarse particles.

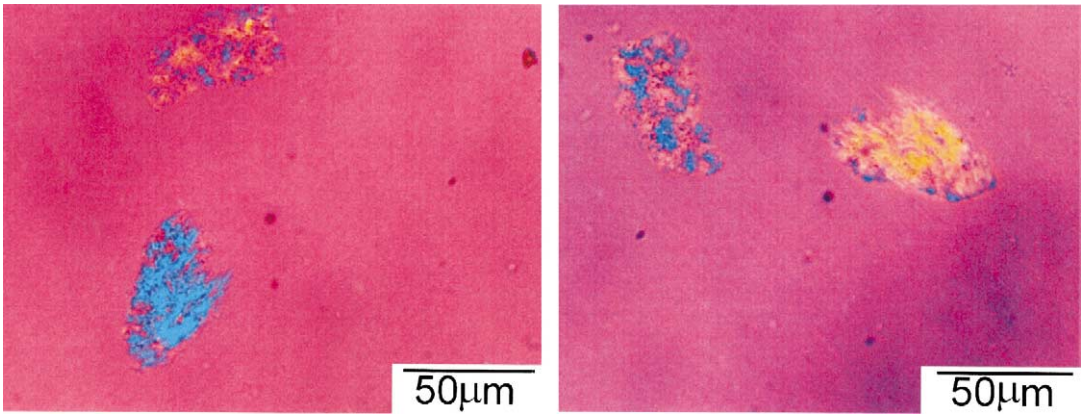

Fig. 6. Crossed-polarized light photographs of separated coarse particles.

reliable reading of 1 mg, it is possible to determine the coarse particle of 10 ppm.

It is easy to understand the presence of coarse particles in the present powders. The powders used in this study are so-called low soda aluminas, and are made by the Bayer process. Aggregates and their origin in the alumina have been reported.[14] The aggregates are formed in the decomposition of aluminum trihydroxide, $Al(OH)_3$, the intermediate product. Some of them clearly survived the grinding/classification processes.

The manufacturers of raw powder clearly leaves the final grinding for producers of ceramics. Their products appear fine in a normal examination involving a commercial particle size analyzer, and satisfy the minimum requirements which commercial products must fulfill. The fulfillment of the minimum requirement, however, does not guarantee the production of good ceramics. Coarse particles of 1000 ppm can fully dictate the microstructure and the quality of ceramics. In a typical production of alumina ceramics, the average particle size of ceramics is 10 times of that of the starting powder, i.e. the volumes of some particles increased 1000 times in sintering and grain growth. The quality of ceramic depends on the grinding of ceramic producers.

5. Conclusions

The present study proposed a method for quantitatively evaluating the particles of extremely large size contained in ceramic raw powders, using industrial grade alumina powders as a model case. Four independent activities indicated that the wet-sieving was a reliable method to determine the content of a small amount of coarse particles in raw powders. The coarse particles separated were essentially aggregates which survived during the production processes of alumina by the Bayer process.

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