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A Preparation System for Grinding Brittle Materials to Submicron and Nano Particles via Mechanical Comminution

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Abstract. Among industrial and laboratory-scale mills currently employed for producing submicron and nano particles, the high energy media mills occupy an important place. The strength characteristic of ultra-fine particles of brittle materials is analyzed, and the size effect of ultra-fine particle is considered. A kind of high energy media mills i.e. a stirred media mill designed and manufactured. Based the stirred media mill, the corresponding process route by wet grinding and the preparation system via mechanical comminution for grinding submicron and nano particles of brittle materials are proposed and built. Experiments were made through the stirred media mill with different size of the grinding media and at different levels of rotational speed of the mill and the grinding time, and at a fixed filling ratio with unitary size of the grinding media. From the experiment results, it is expected that the preparation system and the corresponding process route based on the stirred media mill is suitable to grind particles to submicron and nano size range for brittle materials.

1. Introduction

With the development of technology, the requirements of the fineness and particle size distribution for powder are increasingly stringent, and the demand for submicron and nano particles is growing in the fields of materials engineering [1]. The preparation of submicron and nano particles via mechanical comminution is not confined to a certain material as the preparation technology of liquid phase method and gas phase method, but has extensive adaptability to materials [2]. The preparation of submicron and nano particles by mechanical comminution has important practical significance because of its simple process and high output, especially in the preparation of submicron and nano particles of mineral materials, which has unparalleled advantages compared with other methods. Wet grinding in stirred media mills is suitable for producing ultra-fine particles for its enhanced stress intensity and the stress frequency acting in grinding cylinder [3]. The ultra-fine particles, which is extremely difficult to be prepared in dry grinding system, can be easily obtained with wet grinding process [4]. The increase trend towards ultra-fine milling makes it worthwhile to study the wet-grinding process with stirred media mills.

In this study, a preparation system for grinding submicron and nano particles of brittle materials by mechanical method is built, while a process route for wet grinding based on a stirred media mill is proposed with a consideration of strength characteristic and the size effect of ultra-fine particles of brittle materials. The stirred media mill was designed and manufactured, which can use small grinding



media, and can work at different levels of operation condition. The experimental results obtained from grinding quartz indicate that the preparation system can be used for grinding brittle materials into the particle sizes of submicron and nano range.

2. Breakage Mechanism of Brittle Materials into Submicron and Nano Particles

2.1. The Strength Characteristic of Brittle Particles

At the most fundamental level, comminution reduces to the breakage of individual particles that occurs through contact with other particles or with the grinding media, or with the solid walls of the mill [5]. The elementary comminution event can be regarded as a single particle subject to a stress field. The nature of the stress field around and within individual particles, the material properties of the particulate material, and the size and distribution of micro-flaws within the particles govern the size and shape distribution of the fragments and the surface area produced.

The stresses acting on an elemental area mn perpendicular to the z -axis within a spherical particle due to two equal and opposite concentrated forces [6], as shown in figure 1, can be obtained by superposing the stresses due to the two concentrated forces P acting on O_1 and O_2 :

$$\sigma_r = \frac{P}{2\pi\rho_1^2} \left[-\frac{3r^2(R-z)}{\rho_1^3} + \frac{(1-2\mu)\rho_1}{\rho_1 + R - z} \right] + \frac{P}{2\pi\rho_2^2} \left[-\frac{3r^2(R+z)}{\rho_2^3} + \frac{(1-2\mu)\rho_2}{\rho_2 + R + z} \right] \quad (1)$$

$$\sigma_\theta = \frac{(1-2\mu)P}{2\pi\rho_1^2} \left[\frac{(R-z)}{\rho_1} - \frac{\rho_1}{\rho_1 + R - z} \right] + \frac{(1-2\mu)P}{2\pi\rho_2^2} \left[\frac{(R+z)}{\rho_2} - \frac{\rho_2}{\rho_2 + R + z} \right] \quad (2)$$

$$\sigma_z = -\frac{3P}{2\pi} \left[\frac{(R-z)^3}{\rho_1^5} + \frac{(R+z)^3}{\rho_2^5} \right] \quad (3)$$

$$\tau_{rz} = -\frac{3rP}{2\pi} \left[\frac{(R-z)^2}{\rho_1^5} - \frac{(R+z)^2}{\rho_2^5} \right] \quad (4)$$

where σ_r is radial stress, σ_θ is circumferential stress, σ_z is longitudinal stress, τ_{rz} is shear stress, μ is Poisson's ratio, R is radius of the particle, ρ_1, ρ_2 are the distances of point A within the particle to O_1, O_2 respectively, and second subscripts refer to the stresses caused by the forces at O_1 and O_2 respectively.

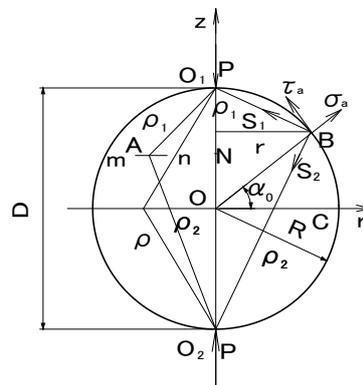


Figure 1. Two concentrated forces acting on a spherical particle.

2.2. Size Effect of Ultra-Fine Particles

It is known that the fracture strength due to the tensile stress of brittle particles undergoes very clear size effects [7]. The strength of brittle particle has a structure sensitive property, which increases as the

size decreases. This phenomenon may be explained by the definition of statistical size of self-similarity, for which the most dangerous flaw proves to be of a size proportional to the structural size.

Assuming that the flaws are obeyed the Weibull's distribution and the number of cracks increases as the specimen' volume increases, the fracture strength depending on the specimen' size is given by:

$$\sigma_t / \sigma_{t0} = (R_0^3 / R^3)^{1/w} \quad (5)$$

where σ_t is critical fracture stress, σ_{t0} is tensile strength of a specimen with a radius of R_0 , R_0 is radius of the specimen and w is Weibull's coefficient of uniformity.

3. A Preparation System for Submicron and Nano Particles Grinding of Brittle Materials

3.1. Process Route of Wet Grinding

To grind particles into the submicron and nano size range, it is necessary to raise the stress strength, the stress intensity and the probability of particle capture. Therefore, in the process route of wet grinding, requirements for small grinding media, high filling ratio for materials and media, high rotor speed, short residence time of material and large circulating slurry flow should be satisfied. Combined with material circulation process, pumping mode and homogenization method, a process route of wet grinding as shown figure 2 is proposed.

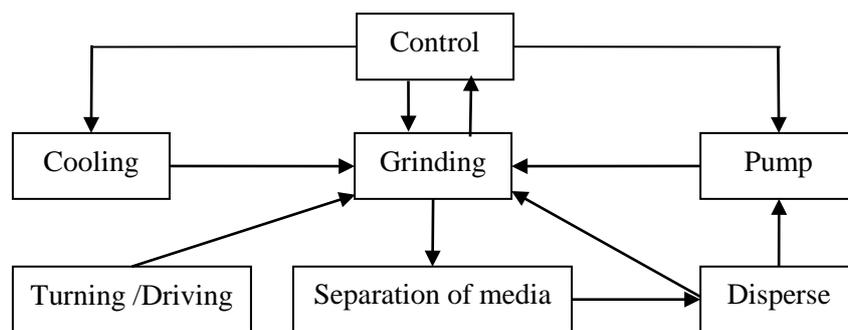


Figure 2. Process route for the preparation system for grinding submicron and nano particles.

3.2. Preparation System for Grinding Submicron and Nano Particles of Brittle Materials

As high energy media mill based on stirring method may fulfill the requirements, the preparation system for grinding submicron and nano particles of brittle materials (figure 3) is built with a stirred media mill as the core, together with a peristaltic pump at the left of the stirred media mill for the circulation of slurry flow, a control box at the right of the stirred media mill for the adjustment & control of the system.



Figure 3. The preparation system for grinding submicron and nano particles via mechanical comminution.

3.3. The Stirred Media Mill

A stirred media mill of wet grinding was designed and manufactured with an outside cooler for the mechanical seal, an inner separator of grinding media and a disperser. Figure 4 and 5 are the front view and top view of the stirred media mill, respectively. The stirred media mill is composed of a turning device, a riving mechanism, a fluid grinding apparatus and a frame. The grinding apparatus is consisted of a grinding disk, a cylinder, cover and base of the cylinder, a grinding disk, a chamber, a feeding hole and an outlet. The stirred media mill is working by: (1) the motor controlled by frequency converter drives a hollow shaft through a belt transmission which increases the rotating speed, (2) the hollow shaft brings along the grinding disk rotating at high-speed, (3) the grinding disk propels the grinding media and particles moving, (4) the particles subject to impact, extrusion and shear between the medium and the cylinder and among the media to obtain the final ultra-fine product.

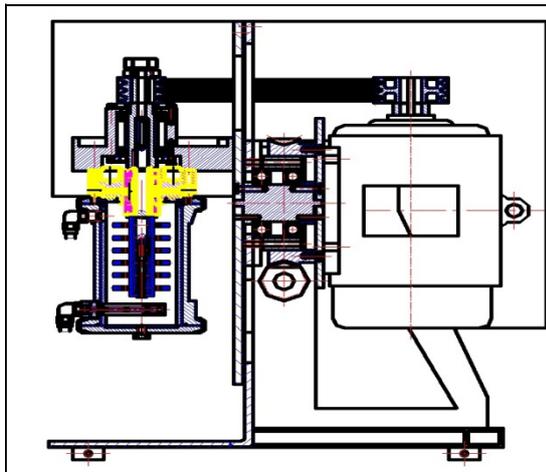


Figure 4. Front view of the stirred media mill.

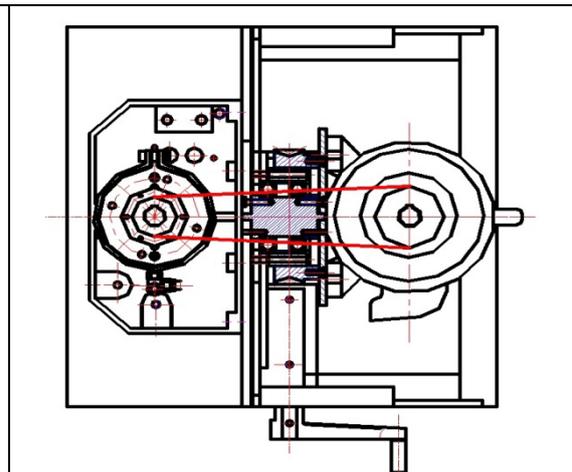


Figure 5. Top view of the stirred media mill.

4. Results and Discussion

4.1. Experimental and Materials

Quartz powder is used in the experiments. Some properties of this material are list in Table 1. Raw materials for feed into the stirred media mill were from the final product of a jet mill.

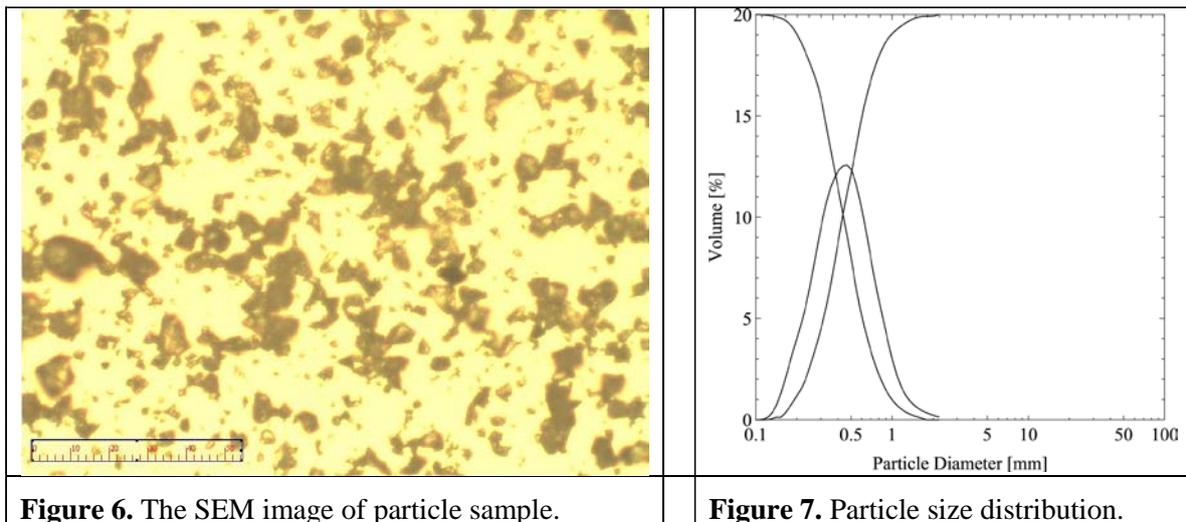
Table 1. Properties of the experimental material.

Material	Density	Mohs hardness	Fineness of raw material	Shape of raw particle
Quartz	$2.65 \times 10^3 \text{ kg/m}^3$	7.0	$D_{90} \leq 5.0 \mu\text{m}$	polyhedron

The raw material was fed into the stirred media mill manually with the grinding media at a media filling ratio and a material filling ratio. In each experiment, the total mass of feed material was fixed, the circulation of slurry flow and grinding time was adjustable. The size of the grinding media was in the interval of [0.2 mm, 2 mm], and unitary size of the grinding media was used in each experiment

4.2. Comminution Results and Influencing Factors

Experiments were made through the stirred media mill with different size of the grinding media, and at different levels of rotational speed of the mill, the the circulation of slurry flow and grinding time. Quartz was used in the experiment to achieve the finest product, and the size distribution of this product was analyzed by SEM through Nova nanosem 430 and by particle size analyzer through Malvern SKC-3000. The comminution results of the quartz product as shown in Figure 6 and Figure 7 were obtained when the rotational speed of the dual cone impact mill was 4500 rpm and with the grinding time of 10 hours.



The fineness of the particle product is affected by the size of the grinding media, filling ratio, rotational speed of the mill, the circulation of slurry flow and grinding time and so many others, but the size of the grinding media plays a key factor on the fineness of the particle product among others. A mean particle size of the submicron and nano range was obtained with the grinding media at the size of [0.2mm, 2mm].

5. Conclusions

The high energy media mills occupy an important place in producing submicron and nano particles, for its high the stress strength, the stress intensity and the probability of particle capture, which can satisfy requirements for small grinding media, high filling ratio for materials and media, high rotor speed, short residence time of material and large circulating slurry flow.

The strength characteristic and the size effect of ultra-fine particles of brittle materials are analyzed. The preparation system for grinding submicron and nano particles of brittle materials via mechanical comminution is built, while the process route of wet grinding using the stirred media mill is proposed. The stirred media mill was designed and manufactured, which can use the grinding media at the size of [0.2mm, 2mm], and can work at different levels of rotational speed of the mill, filling ratio, the circulation of slurry flow and grinding time. The experimental results obtained from grinding quartz indicate that the preparation system and its process route based on the stirred media mill can be used for grinding brittle materials to the particle sizes of submicron and nano range.

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