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Original Article

Fine grinding of brittle minerals and materials by jet mill

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Abstract

Various variables affecting grinding, such as air pressure, minerals or materials hardness, feed size were investigated. The limitations of grinding of gypsum, barite, ilmenite, quartz and ferrosilicon were also elucidated by means of particle fineness size distribution and morphology of ground products. It was found that:

1) The density of particles, which are in the grinding zone affects the product fineness, i.e. higher feed rate results in a larger product size. The appropriate feed rate is suggested to be 0.2~0.5 g/s. Moreover, the density and hardness of minerals or materials tend to have an effect on the product fineness. Heavy minerals, such as barite or ilmenite, exhibit a finer product size than lighter minerals, like quartz. However, for quartz, the higher hardness also results in a larger $d_{50.}$

2) Air pressure is the most vital variable which affects the grinding by a jet mill. The d_{50} seems to relate to the applied air pressure as a power law equation expressed as following:

$$d_{50} = aP^{b} \quad ; \text{as} \quad P \neq 0$$

The a-value and b-value have been found to correlate to the feed size. The higher the air pressure applied the finer the product size attained. Moreover, air pressure has a greater effect on hard minerals than on softer ones.

3) Feed size seems to have a small effect on ground the product fineness of soft materials, such as gypsum and barite, but a significant effect on that of hard materials, such as ferrosilicon and quartz, in particularly by milling at low air pressures of 2~3 kg/cm^2.

4) For the breakage behavior and morphology of ground materials, it was also found that the minerals having cleavages, such as gypsum and barite, tend to be broken along their cleavage planes. Thus, the particle size distribution of these products becomes narrower. While quartz, ilmenite, and ferrosilicon have shattering and chipping breakage mechanisms, grinding results in angular shapes of the ground products and a wider size distribution. Blocks or platelets and agglomerations may occur during grinding of soft minerals, like gypsum, especially at lower and higher air pressures, respectively.

Keywords: jet mill, grinding of brittle minerals and material, effect of jet mill parameters, quartz, ferrosilicon

1. Introduction

Since recently, fine grinding and ultra fine grinding are important for preparing very fine powders, in some cases below 5-10 microns, of cement, fillers (Schwarz, 1998), resin, pigments, chemicals (Zugner *et al.*, 2006), pharmaceutical and cosmetic ingredients (Choi *et al.,* 2004; Chikhalia *et al.*, 2006; Fukunaka *et al.*, 2006; Hasegawa *et al.*, 2006; Schlocker *et al.*, 2006), toners (Tanaka and Kamiya, 2006), plastic, black powders, foods, ceramics (Russell, 1989; Lecoq *et al.*, 2003), electronic materials (Kequka, 1996) and

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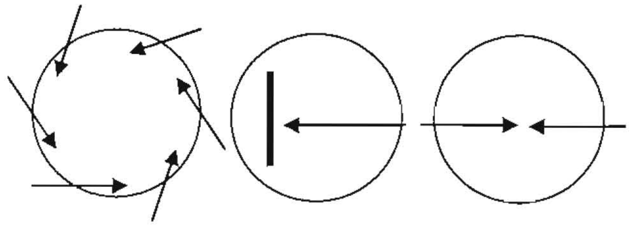
minerals (Seishin Enterprise, 1991; Morasut *et al.*, 2001). Among the industrial mills currently employed for producing high quality fine materials, jet mills occupy an important place (Alfano *et al.*, 1996; Midoux, 1999). A jet mill is a static machine which does not have any grinding media. The milling component of the jet mill consists of a chamber with one nozzle or more. The particles to be ground are accelerated by pressurized gas or steam jets, and the grinding effect is produced by inter particle collision or by impact against solid surfaces (Tuunila and Nystrom, 1998). Compared to grinding media mills, jet mills offer some advantages such as high fineness combined with a narrow particle size distribution. Furthermore, high gas throughputs result in lower milling chamber temperatures and as there are, no agitated built-in elements, the jet mill is insusceptible to dust explosions. In addition, high turbulences in the milling chamber are leading to higher heat transmission and higher mass transfer, and there is no product contamination through wear caused by autogenously grinding (Seishin Enterprise, 1991; Vogel, 1991; Muller *et al.*, 1996).

According to the principles of particles motion in Figure 1, jet mills are classified into spiral jet mills, target jet mills and opposed jet mills. Spiral jet mills comprise of a flat cylindrical milling chamber. The milling gas (air) with a pressure between 1~16 bars expands through 8~10 milling nozzles, tangential in the milling chamber, thus creating a spiral vortex. The feed material enters the milling chamber accelerated by an injector gas system. The comminution takes place by autogenously impact grinding between the particles. The ground particles leave the mill with the gas system through the vortex finder (Eskin *et al.*, 1999). In a target jet mill, particles are accelerated to impact on a target by feeding the gas-solid mixture through a supersonic nozzle and the ground products are classified by cyclones and collected in a vessel (Lecoq *et al.*, 1999). In an opposed mill, particles to be ground are fed into the grinding chamber by applying pressure to a gas-solid mixture through the opposite nozzles, and then projected violently against one another by the jets of compressed air (Berthiaux and Dodds, 1999; Eski and Voropayev, 2001).

The grinding efficiency of jet mill depends not only on the mill type but also on the velocity of the particles and the particles sizes (Mebtoul *et al.*, 1996; Tasirin, 1999), the collision angles of particles hitting each other or the grinding wall or target (Salman *et al.*, 1995; Verroorn and Austin, 1990; Kurten *et al.*, 1970), the milling zone (Eskin and Voropayev, 2001), feed rate and gas pressure (Kolacz, 2004; Gommeren *et al.*, 2000; Ramanujam and Ven Kateswarlu, 1969), minerals or materials properties (such as hardness, density and breakage behaviors (Lecoq *et al.*, 1999; Berthiaux and Dodds, 1999; Sikong *et al.*, 1990), particle size classification (Godet-Morand *et al.*, 2002; Zhen and Yuxin, 1998), particle shapes and nozzle characteristics (Midoux *et al.*, 1999; Gregor and Schonert, 1983; Wang *et al.*, 1998). Cui and his coworkers (2007) suggested that the impact velocity of particles V_p could be determined by the following equation:



(a) (b) (c)

Figure 1. Principles of particle motion in 3 types of jet mill, a) spiral jet mill, b) target jet mill and c) opposed jet mill (Thaler, 2000).

$$V_p = \eta\sqrt{2P/\rho} \quad (1)$$

Where P is the operating pumps pressure ρ is the density of fluid and η is a particle velocity coefficient with 0.75. In general, it is known that the grinding rate of a jet mill is proportional to velocity, either the particle velocity or the velocity of the suspending fluid. Mebtoul *et al.* (1996) reported that plastic behavior dominates for impacts at higher velocities (V_p>100 m/s). The velocity of particles decreases with the increasing solid volume fraction or feed rate, because the strain energy decreases by transformation to kinetic energy of the particle (rebound) corresponding to elastic behavior, whereas fracture energy and heat dissipation correspond to plastic behavior. The rate of grinding of particles attrition in fluidized beds was reported by Tasirin and Geldart (1999) as a power law function of the free jet velocity, namely in the form of:

$$R_{gr} \;\alpha\; V_p^{2\sim3} \quad (2)$$

The free jet velocity was calculated based on the cross-sectional area of the nozzle and by assuming that the pressure change along the tube was small (less than 10%). The grinding power supplied as kinetic energy for sonic nozzles proposed by Midoux *et al.* (1999), can be expressed by:

$$E_k = \frac{1}{2}M_g V_p^2 \quad (3)$$

Where M_g is the gas mass flow rate and directly proportional to the grinding pressure, the nozzle section and the molecular weight of the fluid employed for grinding shown in equation (4):

$$M_g = PA\sqrt{\frac{M_w}{RT}\left(\frac{2}{k+1}\right)^{\frac{k+1}{k-1}}} \quad (4)$$

Where k is the ratio of specific heats of the gas, M_w is the molecular weight of fluid, R is gas constant and T is the temperature. According to the suggestion of Schurr and Zhao (1994) the specific energy consumption (E_{sp}) including both grinding pressure and solid feed rate parameter is calculated by:

$$E_{sp} = \frac{E_k}{Q} \tag{5}$$

This compares different mills, or working conditions (type of gas, temperature, pressure) on the same system. In spiral jet mills, the specific surface area of the product (SSA) is related to E_{sp} by a power function:

$$SSA \ \alpha \ (E_{sp})^x \tag{6}$$

Another approach model for a formal force acting (f) on a particle in a particle-particle collision process, is proposed by Eskin and Voropayev (2001):

$$f = (1+\kappa)\frac{\pi d_s^{\ 2}}{4} \rho_s \varepsilon Vp^2 \tag{7}$$

Where κ is the coefficient of restitution for a particle-particle collision (κ=0 for particle collision within the milling zone perfectly plastic normally κ= 0.2), d_s is a particle diameter, ρ_s is a particle density and ε is particle volume concentration.

In this study, the effect of physical properties of minerals and material and operating parameters upon milling by an 'O' jet mill were investigated.

2. Experimental

2.1 Materials

Four different kinds of minerals and one kind of material used as grinding samples are gypsum, barite, ilmenite, quartz, and ferrosilicon respectively. Some physical and chemical properties and diagnostic features of these samples are shown in Table 1.

2.2 A laboratory jet mill system

In this study, a laboratory jet mill CP-10 (Seishin Enterprise) schematically illustrated in Figure 2 was used. In the pulverization test system, compressed air from compressor is cleaned from its oil and moisture by an air dryer and air filter. It becomes clean air and is then introduced into the SK Jet-O-Mill. The materials, fed through a hopper and accelerated to supersonic speed by a venture nozzle, are pulverized by mutual collision and refraction in the pulverizing area and then with a fluid medium coming out through the grinding nozzles at the lower part of the mill. Discharges particles are collected by a cyclone and received by a vessel. Ultra fine particles which cannot be collected by cyclone are collected by bag filter and received by a vessel.

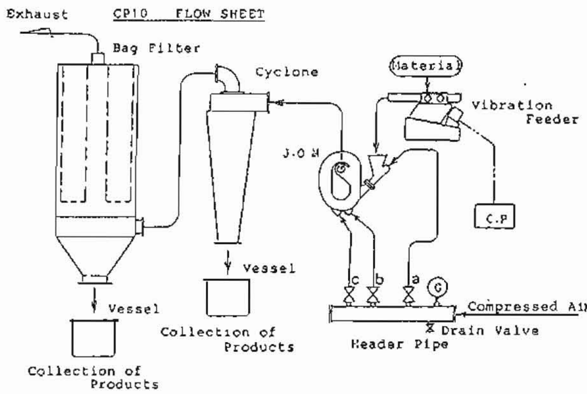


Figure 2. Illustration of a laboratory jet mill system (Seishin Enterprise, 1991).

2.3 Grinding test and materials characterizations

The main variables of grinding test are the feed sizes with -20+35 mesh (595 microns), -35+65 mesh (297 microns), -65+100 mesh (177 microns) and -100+200 mesh (105 microns), the feed rates (0.2-2.0 g/s) and the compressed air pressures (2~7 kg/cm^2 or 0.2~0.7 MPa). Hardness of mineral and material samples was determined by means of Vickers micro hardness tester. After grinding at each designed condition, particle size distribution and morphology of ground samples were determined by using light scattering

Table1. Physical and chemical properties of materials used (Huvbut and Klein, 1977).

Minerals & Material	HardnessMohs (VHN)	S.G.	Crystal structure	Diagnostic features	Chemical	Chemical compositions formula
Gypsum	2 (32.8)	2.32	Monoclinic	Soft and three unequal cleavages	$CaSO_4.2H_2O$	CaO 32.6%, SO_3 46.5%, H_2O 26.9%
Barite	3.35 (173)	4.5	Orthorhombic	Cleavage{001} perfect and high specific gravity	$BaSO_4$	BaO 65.7%, SO_3 34.3%
Ilmenite	5.5-6 (244.5)	4.7	Rhombohedral	Massive, compact; also in grain or as sand	$FeTiO_3$	Fe 36.8% , Ti-31.6% and O 31.6%
Ferrosilicon	6 (381.2)	3.2	-	Porous	-	SiO_2 78.79%, Fe_2O_3 19.7%
Quartz	7 (725.8)	2.65	Hexagonal	Glassy luster, conchoidal fracture and crystal form	SiO_2	Si 46.7% and O 53.3%

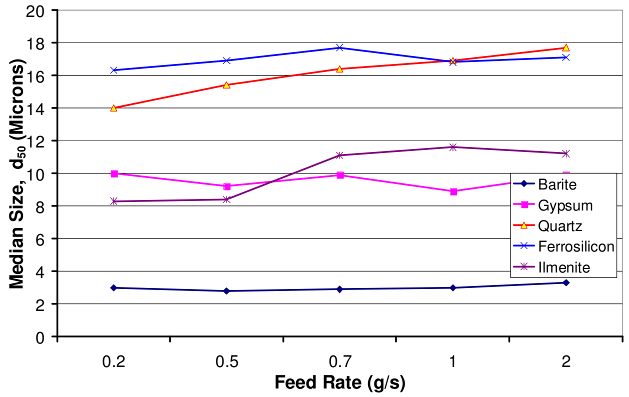


Figure 3. The variation of ground median size (d50) of minerals and material tested at pressure of 2 kg/cm^2 versus the feed rate of 297 microns feed size.

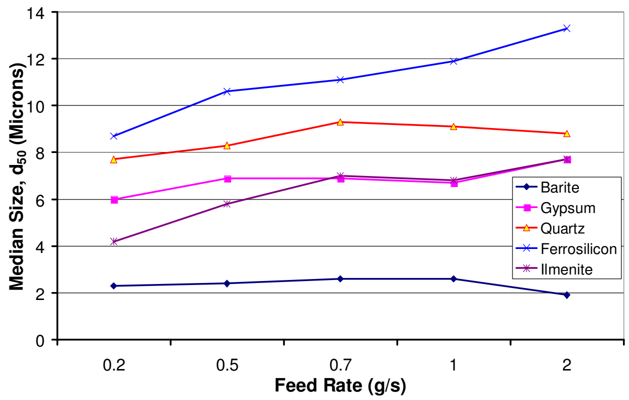


Figure 4. The variation of ground median size (d50) of minerals and material tested at pressure of 7 kg/cm^2 versus the feed rate of 297 microns feed size.

particle size analyzer (LS230, Coulter) and scanning electron microscope (SEM) respectively. The tests were duplicated for each grinding condition.

3. Results and Discussion

3.1 Effect of feed rate

Figure 3 and Figure 4 give the variation of the median sizes (d_{50}) of ground minerals and material versus the feed rate of 297 microns feed size grinding at air pressures of 0.2 and 0.7 MPa. The median size seems slightly to increase with an increase in the feed rate from 0.2 g/s to 2.0 g/s. Because the particle velocity at the nozzle exit decreases when the solid volume fraction (feed rate) increases significantly, due to the particle interaction leading to diminish the kinetic energy (or grinding power supplied) of the gas-solid mixture (Eskin et al., 1999). This trend was found to become more distinctively for harder materials such as quartz, ferrosilicon, and ilmenite. This phenomenon can similarly be seen with other feed size ranges. Thus, the optimum feed rates are suggested to be 0.2~0.5 g/s in order to minimize the median size of the ground products.

3.2 Effect of air pressure and feed size

It was found that the ground product median size (d_{50}) of various grinding conditions related significantly to the applied air pressure. Figure 5 illustrates a proportional decrease in d_{50} with the increasing pressure, as an applied higher pressure induces a higher particle velocity.

The correlation between d_{50} vs. air pressure of all materials concluded in Table 2 can be expressed as a general equation;

$$d_{50} = aP^b \tag{8}$$

Where P is the applied air pressure (kg/cm^2) and the a- and b-values depend upon feed size and d_{50} is an average median size (microns) of the feed rate in the range of 0.2 ~ 0.5 g/s.

Figure 6 and Figure 7 show the relationship between the a-value and b-value of the Equation (8) and feed size, respectively. It can be seen that the a-value increases, but the b-value decreases proportionally with an increase in the feed size.

Thus, the correlation of a-value and b-value with feed size are considered to express the equations listed in Table 3. By substitution of a- and b-values as a function of the feed size into Equation (8), the empirical formula to estimate d_{50} of ground materials at a constant feed rate can be achieved as a function of feed size and air pressure listed in Table 4. This shows that d_{50} increases with an increase in the feed size, but decreases with air pressure. Figure 8 shows the correlation between calculated d_{50} and measured d_{50} for gypsum, barite, quartz, and ferrosilicon, ground with a feed rate of 0.2 g/s. The calculated d_{50} agrees well with the measured one. However, for a hard material, such as quartz and ferrosilicon, there seems to be an over estimation of d_{50} since it is more difficult to grind this kind of material by

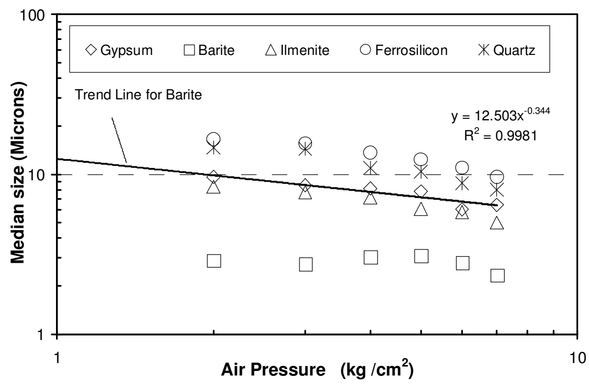


Figure 5. Correlations between the median sizes with air pressure of the ground materials at feed rates of 0.2~0.5 g/s and a feed size of 297 microns for different materials.

Table 2. Correlation between d_{50} of ground materials with air pressure for various feed sizes.

Minerals and Material	Feed Size (Microns)	R^2-values	Equations
Gypsum	595	1.00	$d_{50} = 14.26P^{-0.40}$
	297	1.00	$d_{50} = 12.50P^{-0.34}$
	177	1.00	$d_{50} = 11.34P^{-0.30}$
	105	1.00	$d_{50} = 10.78P^{-0.25}$
Barite	595	1.00	$d_{50} = 7.10P^{-0.48}$
	297	0.99	$d_{50} = 5.25P^{-0.43}$
	177	1.00	$d_{50} = 4.53P^{-0.40}$
	105	1.00	$d_{50} = 4.06P^{-0.35}$
Ilmenite	297	1.00	$d_{50} = 10.96P^{-0.36}$
Ferrosilicon	595	1.00	$d_{50} = 21.88P^{-0.36}$
	297	1.00	$d_{50} = 19.65P^{-0.30}$
	177	0.99	$d_{50} = 17.08P^{-0.26}$
	105	0.99	$d_{50} = 14.95P^{-0.21}$
Quartz	595	0.99	$d_{50} = 18.58P^{-0.38}$
	297	0.99	$d_{50} = 16.99P^{-0.31}$
	177	0.99	$d_{50} = 15.91P^{-0.26}$
	105	1.00	$d_{50} = 13.81P^{-0.22}$

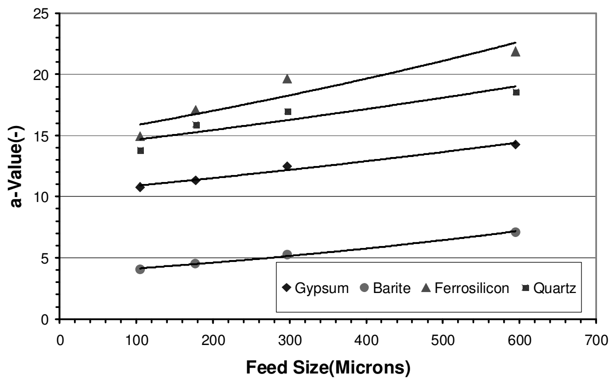


Figure 6. Relationship between a-value of an Equation (8) and feed size.

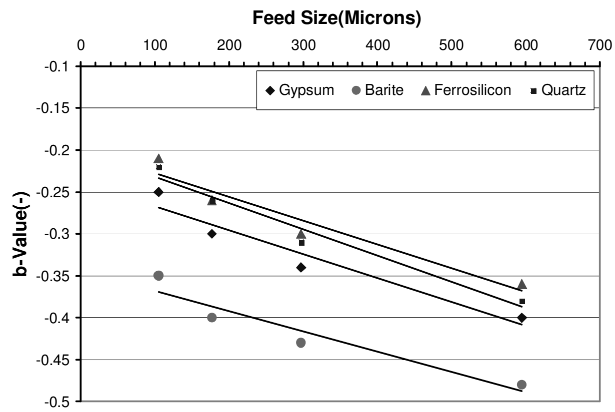


Figure 7. Relationship between b-value of an Equation (8) and feed size.

Table 3. Equations of a- and b-values derived from Figure 6 and 7(x = feed size in microns)

Minerals and Materials	Equations	Remarks
Gypsum	a = 0.0071x+10.1450	$R^2 = 0.9329$
	b = -0.0003x-0.2393	$R^2 = 0.9876$
Barite	a = 0.0062x+3.4198	$R^2 = 0.9999$
	b = -0.0002x-0.3445	$R^2 = 0.9086$
Quartz	a = 0.0086x+13.7970	$R^2 = 0.8612$
	b = -0.0003x-0.2005	$R^2 = 0.9640$
Ferrosilicon	a = 0.0133x+14.4960	$R^2 = 0.9022$
	b = -0.0003x-0.1993	$R^2 = 0.9329$

Table 4. Empirical formula proposed for d_{50} estimation (x = feed size in microns and P = applied air pressure in kg/cm^2)

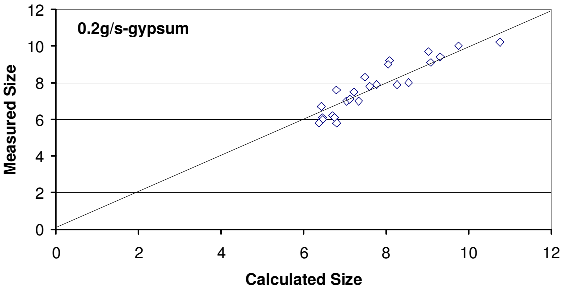
Minerals and Materials	Empirical formula for d_{50} estimation
Gypsum	$d_{50} = (0.0071x+10.1450).P^{(-0.0003x-0.2393)}$
Barite	$d_{50} = (0.0062x+3.4198).P^{(-0.0002x-0.3445)}$
Quartz	$d_{50} = (0.0086x+13.7970).P^{(-0.0003x-0.2005)}$
Ferrosilicon	$d_{50} = (0.0133x+14.4960).P^{(-0.0003x-0.1993)}$

applying low air pressures.

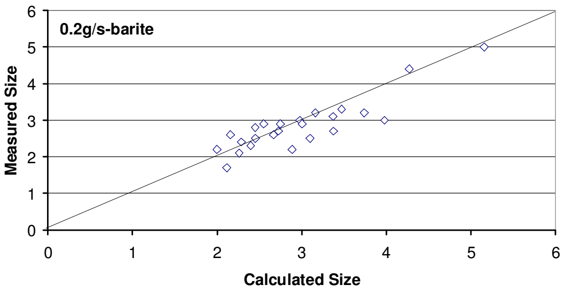
3.3 Effect of materials hardness and density

The material hardness plays an important role on the grindability and wear of the grinding mill. Usually, it is easy to grind materials having a low hardness However, gypsum, having a hardness of 32.8 VHN, seems to be more difficult to grind than barite, having hardness of 173 VHN, because gypsum is very soft and has plate like cleavages. When grinding with lower air pressures this will lead to a lesser degree of destructive grinding, and therefore, the ground products include blocks and platelets. With higher air pressures, grinding would lead to a larger degree of destructive breakage and gives a finer product. Agglomeration was also found to be involved in grinding of gypsum. In addition, barite has a higher density and a more perfect cleavage than gypsum, thus this will lead to higher particle-particle and particle-wall collision forces as shown in Equation (7) (Berthiaux and Dodds, 1999). This will lead to the breakage of the barite particles into very fine ground sizes with a narrow distribution. Comparing to gypsum, ilmenite has the hardness of 244.5VHN but exhibits a finer ground size than gypsum. This is due to ilmenite having a high-density effect similar to barite as discussed above. However, when compared to barite, ilmenite has a lower grindability, because it has a higher hardness than barite. Similar results were also found in the grinding of ferrosilicon and quartz as illustrated in Figure 9.

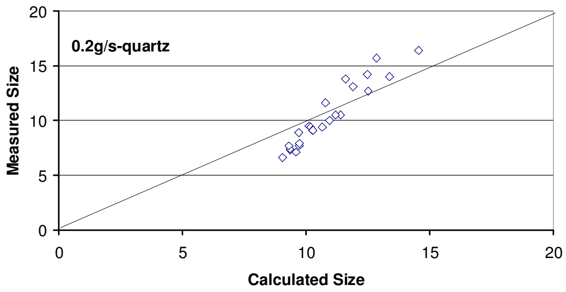
It was apparent that quartz having a higher hardness



(a)



(b)



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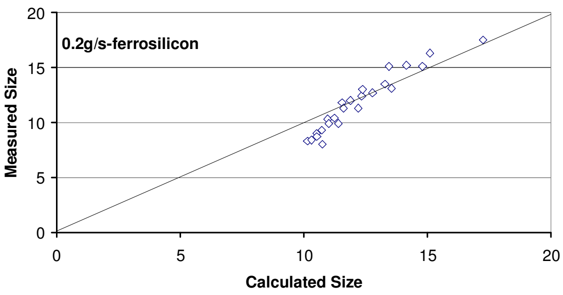

(d)

Figure 8. Correlation between calculated d_{50} and measured d_{50}

seems to be ground easier than ferrosilicon. This is due to the fact that ferrosilicon has a porous matrix that can absorb the kinetic energy of gas jets resulting in the deceleration of particle velocities in the milling zone.

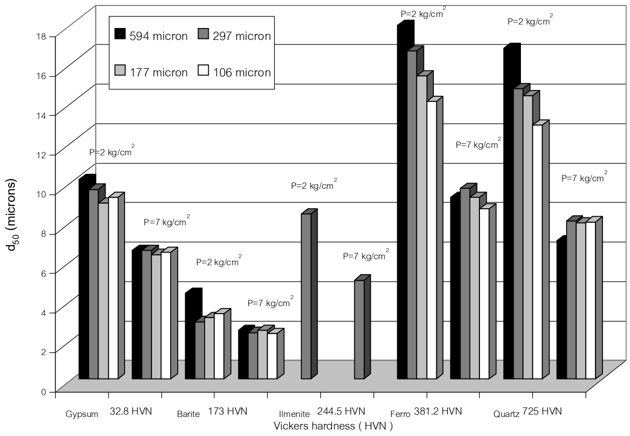

Figure 9. Relationship between d_{50} of ground products and the hardness of tested materials ground at various feed sizes and air pressures.

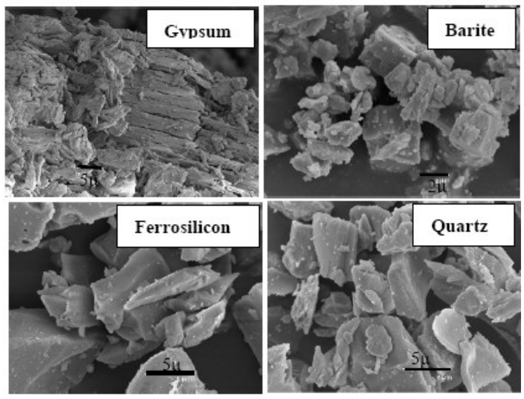

Figure 10. SEM images of ground gypsum, barite, ferrosilicon and quartz particles.

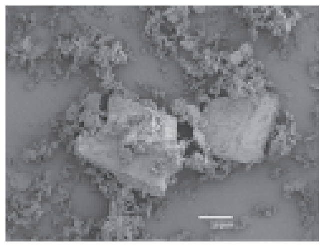

Figure 11. SEM image of gypsum ground product including blocks and platelets after milling at low pressure.

3.4 Breakage behavior and morphology

Gypsum and barite have shown to break along their cleavages as illustrated in Figure 10. The breakage behavior of gypsum is similar to hydrargillite (Berthiaux and Dodds, 1999). Grinding with a lower air pressure will lead to a lesser degree of breakage, so that abrasion mechanism would be encountered and the ground products include blocks and platelets (Figure 11), whereas grinding with a higher pressure will give a higher degree of breakage and finer sizes of

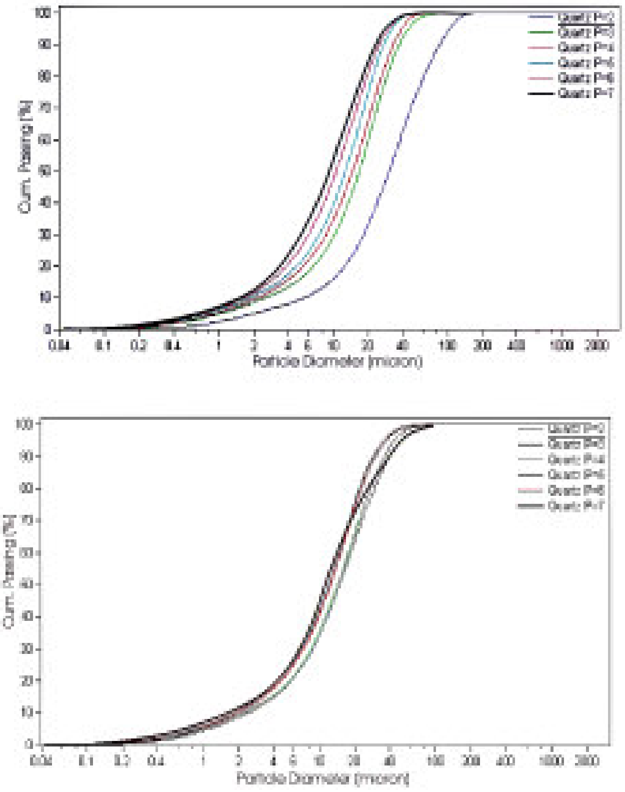

Figure 12. Particle size distributions of quartz ground products at a feed rate of 2 g/s, feed sizes of 595 (above) and 297 microns (bottom).

platelets are obtained. Barite seems to have shattering breakage along its cleavages, thus the ground products exhibit a cleavage shape and narrow size distribution. Ferrosilicon and quartz, both exhibit shattering breakage mechanisms, thus their product shapes look angular and irregular and the size distributions are in a wider size ranges (Figure 10). For quartz, in particular, particle size distributions of the ground products are significantly affected by the feed size and air pressure. Air pressure seems to have the larger effect on the size distributions at a feed size of 595 microns and a minor effect at a smaller feed size as illustrated in Figure 12. It is due to the decrease in forces acting on smaller particle sizes. It was also revealed for other materials that at a higher feed rate, air pressure has a slight effect on the size distributions of the ground products. According to the suggestion of Eskin and Voropayev (2001) for an opposed jet milling efficiency large particles of diameters more than 200 microns cannot be efficiently accelerated, because they are rapidly decelerated by collisions with smaller particles without fragmentation, as occurring in a viscous media (i.e. at high feed rate).

4. Conclusions

The purpose of this research was to study the fine grinding by a jet mill in terms of various variables such as feed size, feed rate, air pressure, hardness, and density, and their effect on grinding of gypsum, barite, ilmenite, quartz, and ferrosilicon. The grinding characteristics were also investigated by means of particle fineness, size distribution and morphology of the ground products. It was found that air pressure was the most vital variable, which affects grinding by jet mill. The d_{50} was apparently related to the air pressure in terms of power law equations, in which the parameters, a- and b- value, seemed closely relate to the feed size has when grinding at a lower feed rate in the range of 0.2~0.5 g/s. For soft minerals such as gypsum and barite, the feed size has slightly affected the ground product's d_{50}. Whereas for hard materials, such as quartz and ferrosilicon, the feed size has an effect on the fineness of the ground product; i.e. a larger size fed, a larger ground d_{50} obtained. It was also revealed that at a higher feed rate of 2 g/s, air pressure has a slight effect on the size distributions of the ground products. Diagnostic features, such as cleavages and physical properties, for example hardness and density of minerals, and materials are found to have an effect on the particle size distribution and the morphology of the ground products. Gypsum and barite have shown to break along their cleavages, resulting in a narrow size distribution. Ilmenite, ferrosilicon and quartz, having a higher hardness, exhibit a shattering breakage mechanism. Which is resulting results in a wider size distribution. Agglomeration was also found to be involved in the grinding of gypsum. Barite has a higher density than gypsum and a nearly perfect cleavage, which result in higher particle-particle and particle-wall collision forces, and thus affecting the breakage of the barite particles into very fine and narrow distributed ground sizes.

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