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# Study of High Velocity Attrition Nozzles in a Fluidized Bed

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## STUDY OF HIGH VELOCITY ATTRITION NOZZLES IN FLUIDIZED BEDS

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#### ABSTRACT

The objective of this study was to test different high velocity attrition nozzles and operating conditions in order to determine the effects of various parameters on the grinding efficiency. Nozzle geometry, as well as gas properties such as speed of sound and density had a significant impact on the grinding efficiency. In addition, a model was developed in order to understand the particle breakage mechanisms during particle attrition. The model showed that the primary attrition mechanism was the splitting of particles rather than their erosion.

#### INTRODUCTION

Fluidized bed jet attritors use high velocity gas jets to grind fluidized particles. Jet mills are used to grind materials such as toners, high purity ceramics, foodstuffs, ultrafine metal oxides, pharmaceutical powders, pigments, polymer powders and ultrafine particles for powder coating (<u>1</u>). Jet attritors are also applied in fluidized bed processes such as fluidized bed coal combustion and fluid coking, where they are used to control the size of the fluidized coke particles by steam injection. Currently attrition nozzles require a large quantity of steam and improving the efficiency of these nozzles would reduce steam consumption.

There are two main modes of attrition: abrasion and fragmentation. Abrasion occurs when very fine pieces are removed from the surface of the particle, thus producing many fines, while the particle size distribution of the mother particles changes slowly. Alternatively, fragmentation occurs when a particle breaks into pieces of similar size (2). There are two main variables that affect the attrition process in a fluidized bed. The first is the particle properties, such as shape, surface roughness and strength. The second is the properties of the environment, such as the fluidization velocity, and the jet operating pressure of the attrition gas (3)

An important aspect of particle attrition is the understanding and modeling of the particle dreakage mechanisms. Austin (4) developed a model introduced by Epstein

 $(\underline{5})$  and Reid (<u>6</u>) to estimate the particle size distribution of ground particles after a known grinding time, given the initial size distribution. A cumulative breakage distribution function gave the size distribution of the new fragments created, and a selection function was defined as the fraction of particles broken per unit time. These breakage and selection functions became the standard parameters used to describe the grinding process.

Most recently, Baddour and Briens ( $\underline{5}$ ) developed a simple, two-parameter model to describe jet milling of carbon nanotubes. However, as most other models, it applies only to small fluidized beds where there is intense solids mixing. It does not apply to processes such as fluid coking, where solids mixing may be a limiting factor and it impacts the attrition rate and product size distribution.

The objective of this study was to test different types of attrition nozzles in order to determine the effect of nozzle geometry on the grinding efficiency and attrition gas consumption. Various exit diameters were tested in order to determine the effect of nozzle scale on the grinding efficiency and attrition gas consumption. The nozzle operating pressure and the bed fluidization velocities were also varied in order to determine their effects on the grinding efficiency, as well as the gas properties and the scale of the fluid bed. In addition, a simple model was developed, with a minimum of empirical parameters, to describe and identify the attrition mechanism when a sonic velocity gas jet is used to grind particles in a fluidized bed and to account for the effect of solids mixing.

#### EXPERIMENTAL

Experiments were conducted in a fluidized bed column with a height of 3.2 m and a rectangular cross section of 1 m by 0.3 m. The particles, coke with a Sauter mean diameter of 135  $\mu$ m, filled the column to a height of approximately 0.3 m and were fluidized with air at various fluidization velocities. Entrained particles were separated from the gas stream by two cyclones in series. Particles from the primary cyclone were continuously returned to the fluidized bed by a dipleg. Particles from the secondary cyclone were collected after each run: under most of the studied conditions, their contribution to the new particle surface was negligible. In order to determine the effects of scale, a smaller fluidized bed column with a height of 0.84 m and rectangular cross section of 0.5 m by 0.1 m was also used. This bed was filled with 13 kg of coke to a height of 0.23 m. This bed geometry was used as dimensions smaller than this would have resulted in the jet interacting with the vessel walls.

In both systems, the attrition nozzle was placed inside the bed at a distance of 0.16 m from the gas distributor and injected gas horizontally into the fluidized particles. A constant gas mass flowrate from a high-pressure cylinder was supplied to the injection nozzle during the grinding process. Adjusting the pressure of the cylinder controlled the gas flowrate to the nozzle.

After injection, the fluidization gas was stopped in order to slump the bed. The fluidization gas was turned on again at a velocity just above the minimum bubbling velocity for approximately 5 minutes in order to mix the particles. Separate experiments indicated that background particle attrition in the fluidized bed, in the absentee of anti-attrition agent, was negligible when compared to the attrition observed

with the nozzles. A sample of solids was taken from the bed before and after each run and analyzed using a Malvern laser diffraction apparatus in order to obtain the size distribution of the particles before and after the grinding process.

Three different types of attrition nozzles were tested in order to determine the effect of nozzle geometry on the grinding efficiency and attrition gas consumption. The first nozzle, designated as Type A, was a convergent-divergent, Laval-type nozzle, similar to the nozzles used by Benz et al. (7). The second nozzle, Type B, was a uniquely shaped nozzle which was similar to Type A, but contained a 10 mm straight section at the tip of the nozzle. The third nozzle type tested, Type C, was a simple straight tube nozzle. Type C nozzles with various exit diameters were tested in order to determine the effect of nozzle scale on the grinding efficiency and attrition gas consumption. The nozzles that were tested had tip diameters, d<sub>N</sub>, of 4.3 mm, 2.4 mm, 1.7 mm, and 1.2 mm.

Different gas flowrates and various types of attrition gases (carbon dioxide, argon, air, helium, and different combinations of air/helium mixtures) were tested in order to determine the effect of gas properties on the grinding efficiency, such as density, viscosity, and the equivalent speed of sound. The equivalent speed of sound was calculated using Equation 1, which takes into account the fact that the temperature of the gas changes ( $\underline{8}$ ).

$$U_{sound \ equivalent} = \sqrt{\frac{RT\kappa}{M}} \left(\frac{2}{\kappa+1}\right)^{\left(\frac{\kappa+1}{\kappa-1}\right)}$$
(1)

Different fluidization velocities and grinding times were also used to determine their effects on the particle attrition rate.

In order to compare the results from all of the tests, a grinding efficiency was calculated, which was defined as the amount of new particle surface created per mass of attrition gas used

$$E = \frac{new \ particle \ surface \ created \ by \ attrition}{mass \ flowrate \ of \ required \ attrition \ gas} = \frac{\frac{m^2}{s}}{\frac{kg}{s}} = \frac{m^2}{kg}$$
(2)

#### **Model Development**

The model presented in this study predicts the particle breakage frequency, the proportion of particles which have not been ground, and the size distribution of the product, after grinding with a sonic velocity attrition jet in a fluidized bed for a given period of time.

The proposed model assumes that one mother particle breaks into two daughter particles of different size. A symmetry coefficient,  $\gamma$ , is introduced which represents the ratio of the volume of the small daughter particle to the volume of the large daughter particle. For example, the two daughter particles will be equally sized if  $\gamma = 1$ . The diameter of the small and large daughter particles can be defined by Equations 3 and 4, respectively.

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 $d_{p,small} = \frac{1}{\sqrt[3]{\frac{1}{\gamma} + 1}}$ 

$$d_{p,large} = \frac{d_{p,o}}{\sqrt[3]{\gamma + 1}}$$
(4)

It was found that the grinding symmetry coefficient,  $\gamma$ , remained approximately constant for coke particles and equal to 0.8 throughout the grinding process, as well as between runs. Therefore, the value of  $\gamma$  was kept constant at 0.8.

Since the grinding symmetry coefficient is fairly high, the mother particle is splitting into two particles of fairly equal size and the primary attrition mechanism occurring in the bed is fragmentation, as opposed to abrasion.

Each split of a mother particle into two daughter particles increases the number of particles in the bed by a value of one, and the total number of breaks during the grinding process is defined as N. The particle breakage frequency, F, is determined from the total number of breaks and the grinding time, t. The particle breakage frequency, F, is a result of both the solids entrainment rate into the jet cavity and the extent of micro-mixing within this jet. The majority of the particle attrition occurs at the end of the jet cavity where entrained particles traveling at high speed, impact particles in the dense phase of the bed. Entrained particles start near the jet boundary, where the gas velocity is small and migrate through micro-mixing to the high velocity central region. As both the entrainment rate and the impact velocity of the entrained particles at the tip of the jet increase, the particle breakage frequency, F, also increases.

$$F = \frac{N}{t}$$
(5)

Some particles are never exposed to the attrition jet during the grinding process, even if the mixing in the fluidized bed is perfect. Therefore, a parameter  $\beta$ , was introduced to represent the proportion of original mother particles that have not been ground.

The value of  $\beta$  can be calculated from a probability model, as shown in the next section. The proportion of original particles that have not been ground,  $\beta$ , depends on the amount of macro-mixing in the fluidized bed. If excellent macro-mixing exists, fewer particles will be left intact, and the value of  $\beta$  will be decreased.

#### **Model Calculation Procedure**

The model assumes that  $n_o$  original particles are present in the bed before grinding. These particles can be divided into two groups: attritable particles, and non-attritable particles. The proportion of non-attritable particles,  $\beta$ , corresponds to the particles that never enter the grinding region of the fluidized bed and, therefore, remain intact. The attritable particles represent the particles in the grinding region of the bed. The total number of non-attritable particles in the bed is equal to  $n_o\beta$ , and the total number of <u>attritable particles</u> in the bed is equal to  $n_o(1-\beta)$ .

McMillan et al.: High Velocity Attrition Nozzles The first step of the calculation procedure is to calculate, from the known experimental size distributions, the arithmetic mean diameters, based on number fractions, of the particles before and after grinding;  $d_{Pam,o}$  and  $d_{Pam,e_exp}$ , respectively. The Sauter mean diameter of the experimentally ground particles, d<sub>Psm.e exp</sub>, can also be calculated from the given size distribution.

Next,  $\beta$ , the proportion of non-attritable particles, is determined using a probability model which assumes that the fluidized bed is perfectly mixed, and therefore all of the particles in the bed are equally likely to be ground. This probability model takes into account the fact that either a fresh unground particle, or a particle that has already been ground, can split into two daughter particles, and then  $\beta$  is calculated for a given breakage frequency, F, and grinding time, t. Particles from the attritable region of the bed are selected using a random number obtained from a uniform distribution in the range from zero to unity. In a previous study by McMillan et al. (9), it was found that the smallest particle that could be ground was  $18\mu m$ . If a random particle with a size of 18 µm or smaller is selected, it is assumed that this particle will not be ground. If the particle chosen is larger than 18  $\mu$ m, it is split into two daughter particles, and their diameters are determined using the grinding symmetry coefficient,  $\gamma$ . The total number of particles in the bed is then increased by a value of one and, as a result the final number of particles in the bed after grinding is equal to n<sub>o</sub> + N. Particles are randomly selected to be broken until the arithmetic mean diameter of the calculated ground particles, d<sub>Pam,e cal</sub>, become equal to the arithmetic mean diameter of the experimentally ground particles, d<sub>pam,e exp</sub>. The corresponding values of N, the number of particle breaks, and F, the particle breakage frequency, were determined. The size distribution of the final product is also calculated.

At the end of the calculation procedure, the proportion of unground particles,  $\beta$ , particle breakage frequency, F, and the final size distribution of the ground particles are, therefore, known.

#### **RESULTS AND DISCUSSION**

The model was used to predict the size distribution of the ground particles, as well as the particle breakage frequency, F, and the proportion of non-attritable particles,  $\beta$ , for various experimental conditions. A typical example of the comparison between the experimental size distribution and the calculated size distribution of the ground particles is shown in Figure 1. There is excellent agreement between the cumulative volume percent of the experimentally ground particles and the cumulative volume percent of the ground particles predicted from the model.

McMillan et al. (9) found that the particle grinding efficiency, E, was proportional to the particle breakage frequency, F<sub>m</sub>. Therefore, the particle breakage frequency, F<sub>m</sub>, calculated from the model, can be used to characterize particle attrition. The effects of various operating conditions and nozzle designs on the value of F<sub>m</sub> was determined and the following conclusions were made:

- The mass of particles broken per second remained approximately constant as the grinding time increased.
- As the fluidization velocity was increased, a greater mass of particles were broken per unit time. Published by ECI Digital Archives, 2007

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- The profiled shape of the Type A nozzle increased the particle breakage frequency.
- The particle breakage frequency, F<sub>m</sub>, remained approximately constant as the amount of solids in the bed was changed.



Figure 1. Comparison of experimental and calculated size distribution of ground particles

The particle breakage frequency,  $F_m$ , depends primarily only on the nozzle geometry, its operating conditions and the fluidization velocity. This means that if  $F_m$  is determined for a given nozzle and solids in a small bed, it can be used to predict the results of attrition in a larger bed.

In a previous study by McMillan et al. (<u>10</u>) a correlation was developed to estimate the grinding efficiency which took into account the nozzle diameter, attrition gas density, equivalent speed of sound of the gas, and the percent excess fluidization velocity. The particle breakage frequency and  $\beta$  were also correlated using these parameters..  $\beta$  is determined by first calculating N, which is equal to the product of the particle breakage frequency, F, and the grinding time. Then n<sub>o</sub> is calculated by dividing the mass of solids in the bed by the average mass of one particle. Finally,  $\beta$  is calculated. Since  $\beta$  is obtained from F (or F<sub>m</sub>), an equation that requires a solution by trial and error can be obtained, and the following correlation was developed.

$$F_m = 3.33 \times 10^{-7} \,\alpha \beta^{-1.406} d_N^{2.918} U_{sound,eq}^{0.434} (\rho U_{sound,eq}^2)^{0.306} \left(\frac{V_g - U_{mf}}{V_g}\right)^{0.278} \tag{6}$$

where  $\alpha$  is the nozzle geometry coefficient and has a value of 2.52, 1.87 and 1 for the Type A, B and C nozzles, respectively. Each parameter in equation 6 has an impact of the mass of particles that are broken in the bed. A large proportion <sup>6</sup> of

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particles remain unground in the bed if the ß term is large, and therefore, the mass of particles broken will be smaller. If the nozzle diameter is increased, the mass of particles broken will also increase. The equivalent speed of sound takes into account implications for the effect of gas properties and temperature, the gas density term takes into account the effect of upstream pressure and the last term takes into account the effect of fluidization velocity. The experimental mass of particles broken per mass of attrition gas is in good agreement with the calculated values as shown in Figure 2. The particle breakage frequency is closely correlated with the attrition nozzle properties and bed operating conditions.





#### CONCLUSION

A model has been developed which predicts the particle breakage rate, the proportion of original particles that are not ground, and the size distribution of particles in a fluidized bed after they have been ground by the injection of a sonic velocity gas jet. The results have been verified using experimental data obtained using various nozzle designs and different operating conditions. The particle breakage rate increased as the fluidization velocity and nozzle diameter were increased. Nozzle designs which resulted in an increase in particle entrainment and higher grinding efficiencies also resulted in an increase in particle breakage frequency.

#### NOTATION

d\_P,largeDiameter of larger daughter particle (m)d\_P,oDiameter of original mother particle (m)d\_P,smallDiameter of smaller daughter particle (m)d\_Pam,aArithmetic mean diameter of attritable particles based on numbers (m)d\_Pam,oArithmetic mean diameter of original particles based on numbers (m)d\_Pam,oArithmetic mean diameter of original particles based on numbers (m)

- d<sub>Pam,e-gale 1</sub>2th international conference diameter of New Adrizons in Fluid action Engineering, Art. 97 based on numbers (m)
- d<sub>Pam,e-exp</sub> Arithmetic mean diameter of experimentally ground particles based on numbers (m)
- d<sub>Pam,o</sub> Arithmetic mean diameter of original particles based on numbers (m)
- $d_{Psm,e-exp}$  Sauter mean diameter of experimentally ground particles (m)
- d<sub>Psm,e-cal</sub> Sauter mean diameter of calculated ground particles (m)
- d<sub>N</sub> Nozzle diameter (mm)
- E Grinding Efficiency (m<sup>2</sup>/kg)
- F Particle breakage rate (number of particles/s)
- F<sub>m</sub> Particle breakage rate (mass of particles/s)
- M Molecular weight (kg/mole)
- $M_{solids}$  Mass of solids in the bed (kg)
- N Number of particle breaks (-)
- n<sub>o</sub> Number of original particles in the bed
- R Gas constant (N.m / moles / K)
- T Temperature (K)
- t Grinding time (s)
- U<sub>mf</sub> Minimum fluidization velocity (cm/s)
- U<sub>sound, eq</sub> Equivalent speed of sound (m/s)
- V<sub>g</sub> Fluidization velocity (cm/s)

### **Greek Letters**

- α Nozzle geometry coefficient (-)
- β Proportion of original particles that have not been ground (%)
- γ Grinding symmetry coefficient (-)
- κ Isentropic expansion factor (-)
- $\rho$  Density of gas (kg/m<sup>3</sup>)

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