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The Spiral Jet Mill Cut Size Equation

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ABSTRACT

Micronisation, or fine grinding, is a key unit operation for many industries, with the spiral jet mill being popular as it is robust and reliable. Within this paper an analytical derivation for spiral jet mill cut size as a function of micronisation settings, gas thermodynamic properties and empirically derived constants for the material and mill is presented for the first time. This has been corroborated by experimental evidence and previously reported data in the academic literature and provides an insight into the interaction between aerodynamic particle classification and fine grinding. A scale up methodology is proposed for a high value material by using a small scale mill to determine the material specific constants of the high value material and a cheaper surrogate material to determine mill specific constants at increased scale.

1. INTRODUCTION

1.1. Context

Micronisation, or fine grinding, is a key unit operation for a number of industries with the spiral jet mill being popular as it is robust and reliable. Despite the spiral jet mill being a well established and widely applied technology, there are no proven scale up methodologies. Scale up of the spiral jet mill is still possible by empirical iteration to achieve a desired particle size at increased throughput, however this can be wasteful and is particularly undesirable for high value materials. Additionally, the interaction between aerodynamic particle classification and fine grinding in a spiral jet mill is not fully understood within the academic or industrial community, making informed optimisation of mill design challenging.

Within this paper an analytical derivation for spiral jet mill cut size as a function of micronisation settings, gas thermodynamic properties and empirically derived constants for the material and mill is presented for the first time. The derivation is corroborated by experimental evidence and previously reported data in the academic literature and provides an insight into the interaction between aerodynamic particle classification and fine grinding. The constants within the equation can be determined empirically for a given material and mill, leading to a better prediction across a design space than standard empirical models. A scale up methodology is proposed for a high value material by using a small scale mill to determine the material specific constants of the high value material and a cheaper surrogate material to determine mill specific parameters at increased scale.

1.2. Process Description

Grinding in a spiral jet mill is achieved as a result of particle collisions caused by high velocity gas exiting a series of nozzles situated around a grind chamber as per Fig. (1) which shows the plan view and process description of a spiral jet mill and Fig. (2) which shows a side view. The grind chamber is typically cylindrical but may also be elliptical in shape. The nozzles are angled such that gas and particles circulate at high velocity around a central exit, resulting in a centrifugal force which retains particles in the grind chamber until micronised. The spiral jet mill is generally operated at steady state as a semi-continuous process with a controlled solids feed rate and gas mass flow rate. When the solids feed rate and gas mass flow rate are controlled, the spiral jet mill delivers a consistent output Particle Size Distribution (PSD). Micronised material can be collected by a combined vane-less axial entry reverse flow cyclonic separator (bottom discharge system) or other means such as a filter sock or bag (top discharge system).

Fig. (1) **Spiral Jet Mill Grind Chamber (Plan View)**

Fig. (2) **Spiral Jet Mill Grind Chamber (Side View)**

1.3. Current Literature for Estimation of Cut Size

1.3.1. Underlying Physics & Numerical Simulation

The underlying physics governing particle size obtained from a spiral jet mill were discussed in 1969 by Dobson and Rothwell [1] who noted that output particle size could be estimated by opposing centrifugal and radial drag forces on a particle at the grind chamber exit. Theoretically, the spiral jet mill has a size of particle (d_{cut}) that will remain balanced at the grind chamber exit with equal drag and centrifugal force. Fig. (3) illustrates the forces balance for a particle at the grind chamber exit.

Fig. (3) **Particle Forces Balance**

The forces balance can be solved for cut size (d_{cut}):

$$d_{cut} = k_1 \frac{v_r^2}{v_t^2} \tag{1}$$

where $k_1 = 3C_D\rho_g r/4\rho_p$, C_D is the drag coefficient of the particle, ρ_g is the gas density, ρ_p is the particle true density, r is the radial position of the particle, v_r is the gas radial velocity and v_t is the tangential velocity of the particle. The drag coefficient, C_D, is known to vary with both gas velocity and particle diameter and is considered in detail in Section 2.3. Assuming relatively low mass fluxes of powder and high gas tangential velocity, it is possible to assume that the particle tangential velocity equals the gas tangential velocity [2]. In most cases this assumption is valid as the mass flux of powder through the grind chamber exit is generally less than 10 kg/m^{2}s and the gas tangential velocity is much greater than 14 m/s. The Cunningham slip correction factor may need to be considered for very fine particles, however with respect to defining the largest particle that can escape the grind chamber it has not been taken into consideration.

Despite Dobson and Rothwell [1] showing that output particle size is likely to be a function of aerodynamics at the grind chamber exit, their theory offered no explanation for the variation in particle size with solids feed rate or solids mechanical properties. An earlier discussion on milling by Berry in 1946 [3] noted that the solids feed rate could have an impact on the rotational speed in the milling chamber of a spiral jet mill, however this was not investigated further until later.

Proposed explanations for changes in particle size with varying solids feed rates in the loop jet mill by Dotson [4] form the basis of subsequent research papers on jet milling [5, 6, 7]. It is observed that at very low solids feed rates, the mill is in a starved condition where there are not enough particle-particle collisions for efficient size reduction. In this starved state, increases in the solids feed rate result in a reduction in output particle size. Following on from the starved condition, further increases in the solids feed rate result in an increase in output particle size. The proposed explanation for increases in output particle size with increasing solids feed rate, in these papers, is a reduction in collision velocity and energy with increased particle population. A reduction in average particle-particle collision velocity was observed in a 2-D combined Computational Fluid Dynamics and Discrete Element

Method (CFD-DEM) simulation by Han et al. [8] and as such this may be a contributing factor to changes in particle size with solids feed rate.

However, changes alone to the collision kinetics do not provide a full explanation for the observed response of particle size to varying gas mass flow rate and solids feed rate. Although the interaction between grinding and aerodynamic classification were further investigated [9], no advancement was made beyond Berry [3] and Dobson and Rothwell [1] until Müller et al. [10] discussed the impact of material held up in the grind chamber (hold up) on cut size. Solids entering the spiral jet mill are retained and continue to accumulate as aerosolised hold up until the number of collisions increases and the output rate of micronised powder is equal to the solids feed rate. As the spiral jet mill operates as a semi-continuous process at steady state, there will be a fixed amount of hold up for a given solids feed rate and gas mass flow rate combination. Müller et al. [10] noted that cut size varied with solids feed rate and solids physical properties as a result of the effect of powder hold up on tangential velocity and centrifugal force retaining particles in the grind chamber.

Müller et al. [10] further discussed the concept of the grinding limit, which is the smallest mean output particle size that can be obtained for a given material and mill. The grinding limit of a spiral jet mill for a given material can be approached by reducing the solids feed rate, repassing ground material or increasing the gas mass flow rate. All approaches result in the hold up tending towards zero. As the grinding limit or cut size for zero hold up is essentially a gas only system, it logically follows that if information about the particle shape and density is known, it is possible for the grinding limit to be estimated by CFD.

Rodnianksi et al. [11] undertook a CFD investigation into the flow fields of a spiral jet mill and their dependence on geometry and gas throughput. It was shown that the spin ratio (v_t/v_r) does not vary significantly across a range of gas mass flow rates. However, the grinding limit determined by CFD cannot be used to predict particle size at different combinations of solids feed rate and gas mass flow rate as the level of hold up and its impact on tangential velocity is unknown.

Population balance models, in particular CFD-DEM, could be used to estimate cut size, however examples in the literature have so far only shown similarity with experimental data when the cut size has been set based on prior knowledge. Examples of cases where the cut size has been predefined include a population balance model [12] which used experimental aerodynamic classification data and a 3-D CFD-DEM simulation [13] which removed particles from the simulation as ground material when below a predetermined size. In the case of the 3-D CFD-DEM [13] study the removal of fine particles is necessary due to current limitations in computational power, suggesting that in the future this technique may be able to estimate cut size if the mechanical properties of the crystals being micronised could be accurately determined and improvements to processing speed are made.

1.3.2. Empirical Correlation with Specific Energy

Prior to this paper, an approach proposed for predicting particle size for varying combinations of solids feed rate and gas mass flow rate was through utilising empirical correlations between Specific Energy Consumption (E_{sp}) and particle size for a given mill and material. The concept of specific energy consumption, E_{sp}, was introduced by Schurr and Zhao [14] as the ratio of kinetic energy delivery rate to solids feed rate, to give the total amount of energy consumed by the microniser per unit weight of powder:

$$E_{sp} = \frac{\dot{E_k}}{\dot{m}_s} \tag{2}$$

where $\dot{m}_s$ is the solids feed rate to the microniser and $\dot{E}_k$ is the kinetic energy delivery rate. The kinetic energy delivery rate is defined as follows:

$$\dot{E}_k = \frac{1}{2}\dot{m}_g v_{sonic}^2 \tag{3}$$

where $\dot{m}_g$ is the gas mass flow rate and v_{sonic} is the gas sonic velocity which is defined as:

$$v_{sonic} = \sqrt{\frac{kRT_{throat}}{M_W}} \tag{4}$$

where k is the ratio of specific heat capacities, R is the specific gas constant, T_{throat} is the temperature at the nozzle throat and M_W is the gas molecular weight. It should be noted that Schurr and Zhao did not explicitly account for temperature reduction at the nozzle throat, and as such the actual grinding energy may be overestimated.

Empirical correlations for a given material between particle size and E_{sp} have been shown to serve as a predictive model for particle size with similar spiral jet mills by Midoux et al. [15]. These correlations result in a relatively accurate prediction of particle size for a given combination of material, solids feed rate and gas mass flow rate. If a small increase in powder throughput is desired for a given material at the same mill scale, it is possible to predict the gas pressure increase required to maintain similarity of particle size.

Midoux et al. [15] subsequently showed that the change in Specific Surface Area (SSA) of a material, as a result of micronisation, correlates with E_{sp} for different gas molecular weights and at different scales. Zhao and Schurr [16] reported similar results but also that the grinding limit varied for gases of differing molecular weights.

Both Midoux et al. [15] and Zhao and Schurr [16] suggested that E_{sp} could be used for scale up without presenting supporting evidence and in contrast to detailed experimental studies on mill geometry by Tuunila and Nyström [17] and Katz and Kalman [18]. These two research papers [17, 18] showed experimentally that varying geometrical parameters leads to differences in particle size for identical combinations of gas flow rate and solids feed rate. Some mill designs are more or less efficient than others, leading to differences in size at the same E_{sp} value. Katz and Kalman [18] for example showed that a grind angle of 45° was optimal compared to larger angles, and Tuunila and Nyström [17] reported that some classifier settings are more efficient than others.

Furthermore, as discussed in Section 3 of this paper, variation in particle size can occur at a constant value of E_{sp} for certain materials when micronised at different gas mass flow rates and solids feed rates on the same spiral jet mill. This issue makes the prediction of particle size with an E_{sp} correlation not viable for certain materials.

Currently there is no mechanistic basis to substantiate the correlation between E_{sp} and cut size obtained for a given material and spiral jet mill, nor is there a relationship to describe how cut size varies with mill geometry. An analytical derivation of the relationship between particle size, mill geometry, gas mass flow rate, gas physical properties, solids feed rate and solids physical properties could potentially lead to a reduction in time and material required for scale up. Within this paper, such an analytical derivation is proposed.

2. DERIVATION OF CUT SIZE AS A FUNCTION OF SPECIFIC ENERGY

The approach adopted for the derivation of the cut size equation is based on the populating of the forces balance with terms that can be determined from the grind chamber geometry and an energy balance. The forces balance is then solved for cut size to produce a general equation. Assumptions are made about the system so that cut size can be described as a function of gas mass flow rate and solids feed rate.

2.1. Geometry and Velocity

It is possible to define the gas kinetic energy delivery rate in terms of gas volumetric flow rate in the grind chamber by combining Eq. (3) and Eq. (4):

$$\dot{E}_k = \frac{1}{2}\dot{m}_g \frac{kRT_{throat}}{M_W} \tag{5}$$

Assuming ideal gas behaviour, $\dot{m}_g$ can be defined in terms of volumetric flow rate through the grind chamber:

$$\dot{m}_g = \dot{V}\frac{PM_W}{RT} \tag{6}$$

where P is the pressure in the grind chamber, T is the gas temperature in the grind chamber and $\dot{V}$ is the volumetric flow rate through the grind chamber. Combining Eq. (5) and Eq. (6) yields:

$$\dot{E}_k = \frac{T_{throat}}{2T}kP\dot{V} \tag{7}$$

From Eq. (7) it can be deduced that for gases with similar ratios of specific heat capacity, the volumetric flow rate will be similar for the same kinetic energy delivery rate, regardless of the gas molecular weight. It should be noted that the gas volumetric flow rate ($\dot{V}$), mass flow rate ($\dot{m}_g$) and kinetic energy delivery rate ($\dot{E}_k$) refer to the sum of the grinding gas, feed gas and entrained gas. Additionally, although the total gas flow rate is being considered in this derivation, the constant spin ratio assumption [11] is only likely to hold when the ratio of grinding gas, feed gas and entrained gas is constant as the CFD simulations it is based on did not address variation in feed gas or entrained gas.

The grind chamber exit geometry can be defined in terms of three parameters (r, h_1 and h_2 which are the classifier radius, exit gap and grind chamber height, respectively). Fig. (4) shows the side view of a grind chamber with a “classifier” at its exit and the various parameters. Many spiral jet mills have a classifier that results in a different gap size for gas flow at the grind chamber exit compared to the rest of the grind chamber. The purpose of the classifier is to prevent the escape of larger particles which can travel along the walls of the grind chamber where the radial velocity is higher and radial drag dominates over centrifugal forces. In this case, a flat grind chamber geometry has been presented where the height of the entire grind chamber is h_2. This derivation should also apply to elliptical plates (both symmetrical and asymmetrical), however it should be noted that changing the plate shape will potentially impact on both the collision kinetics and the spin ratio. Although only a single classifier has been shown in Fig. (4), the dimensions refer to the gap for gas flow rather than classifier height and as such could also be applied to a spiral jet mill with a double classifier.

Fig. (4) Grind Chamber Geometry

The general gas average radial velocity at a point in a cylinder with gas flowing towards the centre, $v_{r\ (general)}$, can be defined in terms of gas volumetric flow rate:

$$v_{r\ (general)} = \frac{\dot{V}}{2\pi r h} \tag{8}$$

where h is the height of the gap through which the gas flows and r is the radial position. The radial velocity will vary across h [11, 19], however to realise the subsequent analysis of the system as a whole, Eq. (8) assumes that the radial velocity is constant across the height of the grind chamber.

Eq. (8) can be combined with Eq. (7) and solved for the radial velocity at the grind chamber exit, v_r:

$$v_r = \frac{k_2 \dot{E_k}}{h_1} \tag{9}$$

where $k_2 = \frac{T}{T_{throat}\pi r k P}$.

The constant spin ratio assumption [11] can be combined with Eq. (8) to define the tangential velocity at the grind chamber exit for the zero hold up system, $v_{t\ (gas\ only)}$:

$$v_{t\ (gas\ only)} = \frac{k_2 k_3 \dot{E_k}}{h_2} \tag{10}$$

where k_3 is the spin ratio (v_t/v_r) for the zero hold up system. The height h_2 is used in Eq. (1) as opposed to h_1 as Rodnianski et al. [11] showed that the constant spin ratio assumption only holds for changes in grind chamber height, and not for variations in the classifier height.

The forces balance can be defined for a particle at the interface between the classifier and the grind chamber where it is subjected to the tangential velocity of the grind chamber and the radial exit velocity of the classifier. Fig. (5) shows the forces balance for a particle at the interface between the grind chamber and classifier.

Fig. (5) Classifier to Grind Chamber Interface

Although Fig. (5) suggests that for increasing classifier height (reduction in h_1) there would be an increase in radial drag force and cut size and, for a reduction in classifier height (increase in h_1) there would be a decrease in radial drag force and cut size, this does not apply to all classifier heights [19]. Where there is no classifier, the output is expected to be coarser as there is a shortcut route for large particles to escape along the grind chamber wall, and therefore the initial introduction of the classifier will result in a finer output.

2.2. Force and Energy Balance

Prior to performing a balance between the gas only and gas and powder systems with respect to the rate of kinetic energy consumption at the grind chamber exit, the effect of powder hold up on tangential velocity must be first considered. Once the system has reached steady state, there will be a constant mass of solids held up (m_h) in the grind chamber volume (V_{grind}):

$$\frac{m_h}{V_{grind}} = constant \tag{11}$$

Assuming a constant solids concentration and no slip between the solids and gas, there will be an equivalent steady state solids hold up rate associated with the gas volumetric flow rate to maintain the solids concentration:

$$\frac{m_h}{V_{grind}} = \frac{\dot{m}_h}{\dot{V}} \tag{12}$$

where $\dot{m}_h$ is the equivalent rate of powder hold up. It should be noted that $\dot{m}_h$ refers to the sum of the retained particles and particles flowing through the system ($\dot{m}_s$). The equivalent rate of powder hold up is an important concept with respect to energy consumption, as maintaining hold up in rotation will consume energy at a given rate.

A balance can subsequently be performed on the rate of kinetic energy consumption at the grind chamber exit based on the assumption that the solids concentration is uniform and that the radial and tangential components of velocity do not change with position. Although it is known that the radial and tangential components will change with position and that the solids concentration is not uniform, they have been assumed to be constant as this simplification allows the behaviour of the system to be approximated analytically. It is also assumed that axial movement of the gas and powder is negligible. The energy balance is performed by considering a unit volume (dV) in the grind chamber for the gas only and also the gas and powder systems. Fig. (6) illustrates the energy balance by considering a unit volume for the gas only system on the left, and the gas and powder system on the right. The rate of consumption of kinetic energy from the gas only system is a function of the gas radial and tangential velocity at the grind chamber exit, whereas the rate of consumption of kinetic energy for the gas and powder system is a function of the gas and powder radial and tangential velocities and additional frictional energy losses associated with particle collisions. As the gas radial velocity is fixed by geometry and volumetric flow rate this does not change when moving to the gas and powder system. However, the gas tangential velocity reduces with powder hold up as maintaining this hold up in circulation consumes energy.

Fig. (6) Energy Rate Balance on a Unit Volume at the Grind Chamber Exit

The kinetic energy consumption rates can be integrated across the volume for the gas only system, Eq. (13) and Eq. (14), and for the gas and powder system, Eq. (15) and Eq. (16):

$$\dot{E}_{exit\,(gas\,only)} = \int \left(\frac{1}{2}\frac{d\dot{m}_g}{dV} v^2_{t\,(gas\,only)} + \frac{1}{2}\frac{d\dot{m}_g}{dV} v_r^2\right) dV \tag{13}$$

$$\dot{E}_{exit\,(gas\,only)} = \frac{1}{2}\dot{m}_g v^2_{t\,(gas\,only)} + \frac{1}{2}\dot{m}_g v_r^2 \tag{14}$$

$$\dot{E}_{exit} = \int (\frac{1}{2}\left(\frac{d\dot{m}_g}{dV} + \frac{d\dot{m}_h}{dV}\right) v_t^2 + \frac{1}{2}\frac{d\dot{m}_g}{dV} v_r^2 + \frac{1}{2}\frac{d\dot{m}_s}{dV} v^2_{p(radial)} + \frac{d\dot{E}_{loss}}{dV}) dV \tag{15}$$

$$\dot{E}_{exit} = \frac{1}{2}\left(\dot{m}_g + \dot{m}_h\right) v_t^2 + \frac{1}{2}\dot{m}_g v_r^2 + \frac{1}{2}\dot{m}_s v^2_{p(radial)} + \dot{E}_{loss} \tag{16}$$

where $\dot{E}_{loss}$ is the rate of energy loss associated with friction, particle acceleration and collisions and $v_{p(radial)}$ is the particle radial velocity on exit from the grind chamber. The kinetic energy consumption rates at the grind chamber exit may be balanced for the gas only and gas and powder systems and solved for v_t^2 as follows:

$$v_t^2 = \frac{\dot{m}_g v^2_{t\,(gas\,only)} - \dot{m}_s v^2_{p(radial)} - 2\dot{E}_{loss}}{\dot{m}_g + \dot{m}_h} \tag{17}$$

By combining Eq. (1), Eq. (9) and Eq. (17), a general equation for spiral jet mill cut size can be derived:

$$d_{cut} = \frac{k_1(h_2 k_2 \dot{E_k})^2(\dot{m_g} + \dot{m_h})}{\dot{m_g}(h_1 k_2 k_3 \dot{E_k})^2 - 2\dot{E}_{loss} - \dot{m}_s v_{p(radial)}^2} \quad (18)$$

Eq. (18) cannot be applied in its current form to determine cut size as a number of the parameters are unknown for the system, including the drag coefficient and the response of solids hold up to solids feed rate and gas mass flow rate. To render Eq. (18) into a useable format it is necessary to obtain a relationship for the solids hold up in terms of solids feed rate and gas mass flow rate and also to understand the variation in drag coefficient with gas mass flow rate.

2.3. Variation in Drag Coefficient

The drag coefficient reduces with increasing Reynolds number as per Haider and Levenspiel [20] towards a constant value, C_1 depending on the particle shape. To approximate the drag coefficient, the following relationship is utilised:

$$C_D = \frac{24}{Re} + C_1 \quad (19)$$

where C_1 is the drag coefficient as the Reynolds number, Re, tends towards infinity and is defined for a given sphericity by Haider and Levenspiel [20]. Eq. (19) can then be combined with the definition of Reynolds number and Eq. (9) to obtain Eq. (20) for the drag coefficient of a particle at the grind chamber exit:

$$C_D = \frac{24\mu k_4 h_1}{\rho_g k_2 d_{Reynolds} \dot{m}_g} + C_1 \quad (20)$$

where μ is the gas viscosity, $d_{Reynolds}$ is the Reynolds length scale and $k_4 = 2/v_{sonic}^2$. The Reynolds length scale, although equivalent to cut size, will be assumed to be constant in the subsequent analysis as variation in cut size is generally small when compared to variation in solids feed rate or gas mass flow rate. This can be observed in Fig. (11), a plot of variation in cut size for Product A discussed in Section 3.2, where doubling the gas mass flow rate reduced the cut size by less than 25%.

This estimate of the drag coefficient may then be combined with the definition of k_1 giving;

$$k_1 = \frac{x_1 k_4}{d_{Reynolds} \dot{m}_g} + C_2 \quad (21)$$

where $x_1 = 18\mu h_1 r/\rho_p k_2$ and $C_2 = 3C_1\rho_g r/4\rho_p$. It should be noted that k_1 is now a function of the Reynolds length scale and gas mass flow rate. As the cut size (Reynolds length scale) is reduced, the dependence of k_1 on gas mass flow rate will increase. Similarly as cut size increases $k_1$will tend towards C_2.

2.4. Collision Kinetics

As the rate of generation of small fragments is dependent on collisions occurring between particles, it may be possible to analyse the particle size reduction process in a similar manner to collision kinetics and reaction chemistry. Similar to reaction kinetics, two or more particles must collide with sufficient activation energy to result in fragmentation such that particles below the cut size are generated. If a parallel were to be drawn between collision kinetics and reaction chemistry, a second order relationship for collision rate with respect to powder concentration would be expected:

$$\dot{m}_{collide} \propto \left(\frac{m_h}{V_{grind}}\right)^2 \tag{22}$$

However, the collision process in a spiral jet mill differs in principle to typical bimolecular reactions. In the case of a spiral jet mill, highly energetic collisions primarily occur at the intersection of the nozzle jets and the rest of the grind chamber. Prior to collision, particles must be transported from the grind chamber to the grind nozzles, leading to a two stage process for particle collisions. Transport of particles to the nozzles can be defined as:

$$\dot{m}_{nozzles} = D\left(\frac{m_h}{V_{grind}}\right) \tag{23}$$

where D is the mass transfer coefficient in $m^3.s^{-1}$ which will be specific to the transport properties of the particles being micronised, the gas physical properties and the flow conditions in the grind chamber. The mass transfer coefficient relates to the volume of gas-solids mixture intersecting with the nozzles per second. The rate of collisions is thus expected to be second order:

$$\dot{m}_{collide} = K\left(\frac{\dot{m}_{nozzles}}{\dot{V}}\right)^2 \tag{24}$$

where K is a rate constant in $m^6.kg^{-1}.s^{-1}$for a collision between two particles that will vary depending on the solids concentration, particle velocities and particle cross sectional area.

The angled configuration of grind nozzles is designed to result in particle collisions with enough energy to cause them to fragment. The most likely collision scenario, and the scenario resulting in the greatest momentum exchange [8], is where a particle (1) accelerated by the nozzle jets collides with another particle (2) and whose path coincides with the nozzle jet as illustrated in Fig. (7). Although subdivided into the particles accelerated by the nozzle jets (1) and particles colliding with them (2), this is one collision scenario. A second collision scenario (3) exists where two particles, both accelerated by different nozzle jets, collide with each other. This would be more likely to occur for either closely spaced nozzles or low solids concentrations.

The third collision scenario (4) involves collisions between a particle and the mill surfaces. These could involve a particle being pulled by centrifugal force to the edge of the grind chamber and colliding with the periphery of the chamber, particles colliding with the classifier or particles hitting the top or bottom of the chamber. This third scenario may account for a significant amount of attrition in mill designs with fewer nozzles such that the nozzle jets do not intersect. Teng et al. [21] reported a numerical simulation utilising CFD-DEM for a spiral jet mill design consisting of two nozzles which did not intersect and were angled approximately tangentially in the direction of the mill chamber walls. This particular mill design [21] led to an increased prevalence of the third collision scenario compared to the first two. Particle-mill collisions may also be more likely for particularly large input particles where centrifugal forces are very high or for low solids concentrations where particle-particle collisions are less likely than particle-mill collisions. As most spiral jet mills have nozzle jets which intersect with each other, the first two scenarios as per Fig. (7) are considered to be the most frequent. As the third scenario also requires mass transport to the nozzles for the most energetic wall collisions, the derivation in this paper also applies to this attrition mechanism.

Fig. (7) Particle-Particle Collision Scenarios

Drawing on an analogy with reaction kinetics, the overall reaction order will be defined by the rate limiting step. It is assumed that mass transport to the nozzles is the rate limiting step in the particle fragmentation process. This assumption has been made as the angled configuration of grind nozzles is such that once a particle intersects with a nozzle jet it is likely to undergo a subsequent collision, making this process faster than mass transport to the nozzles. Therefore, it is possible to deduce Eq. (25) for the rate of collisions:

$$\dot{m}_{collide} = D\left(\frac{m_h}{V_{grind}}\right) = D\left(\frac{\dot{m}_h}{\dot{V}}\right) \tag{25}$$

By drawing a parallel with reaction chemistry it should be noted that as the nozzle jet velocity is limited to sonic velocity for choked flow, it can be assumed that the maximum collision energy will be similar for a fixed grind angle across a wide range of grind pressures. Similarity in maximum collision velocity will be maintained for grind pressures in the choked flow regime where the ratio of pressure in the mill chamber to the grind pressure is less than $(2/k+1)^{k/k-1}$. Therefore, the collision energy remains approximately constant for a given nozzle jet velocity and collision angle. By applying the similarity of fracturing law [22] and assuming that variation in cut size is small with respect to overall size reduction, it is possible to assume that the mass fraction of collision fragments below the cut size is constant for a given spiral jet mill across a range of gas pressures and flow rates. This then allows the rate of generation of particles below the cut size ($\dot{m}_{below\ cut\ size}$) to be defined by the time averaged mass fraction of collision fragments below the cut size (b):

$$\dot{m}_{below\ cut\ size} = b\dot{m}_{collide} \tag{26}$$

Kürten et al. [23] showed for opposed jets with a nozzle pressure difference of 4 bar, that increasing the solids feed rate from 6 g/min to 800 g/min resulted in a reduction in collision velocity from 142 m/s to 83 m/s as the time available for acceleration in the nozzle jet reduces with increases in powder concentration and collision probability. It would therefore be expected that for changes in powder concentration greater than an order of magnitude the mass fraction of collision fragments, b, will decrease with a reduction in acceleration time and collision energy. The assumption of b remaining constant is expected to be valid for changes of powder concentration within an order of magnitude as the collision velocities and energies will be approximately similar.

The collision energy may vary with particle size as large primary particles will be able to retain their velocity following exit from a nozzle jet, whereas smaller particles will quickly lose their velocity due to fluid forces. The velocity retention and subsequent higher collision energies may result in a greater value for b when hold up has a greater mass fraction of large particles, and a lower b value for when hold up has a greater mass fraction of fine particles. For a given material, output PSD can vary with feed material PSD, potentially as a consequence of differing values of b. Determining the effect of feed material PSD and mechanical properties on b analytically is not considered in this derivation, however experimentation with different sizes of particles with contrasting mechanical properties could provide an empirical relationship.

It is proposed that the mass fraction of collision fragments below the cut size, b, could be determined experimentally at laboratory scale with a 4" or 2" diameter spiral jet mill for a material and used to model behaviour at pilot or industrial scale. It may also be possible to estimate b by following a similar approach to that proposed by Vogel and Peukert [24, 25], using a single particle impact device and then estimating the collision conditions in the spiral jet mill.

Upon delivery of a given solids feed rate to the grind chamber ($\dot{m}_s$), the equivalent rate of powder hold up ($\dot{m}_h$) will continue to increase until the rate of generation of collision fragments below the cut size ($\dot{m}_{below\ cut\ size}$) equals the rate of powder delivery ($\dot{m}_s$). This allows the rate of powder hold up ($\dot{m}_h$) to be defined in terms of the solids feed rate ($\dot{m}_s$) and gas mass flow rate ($\dot{m}_g$) for steady state:

$$\dot{m}_h = \frac{\dot{m}_g \dot{m}_s}{x_2} \tag{27}$$

where $x_2 = DbPM_w/RT$ assuming the gas in the grind chamber is ideal.

2.5. Equation Solution

Due to the unknown rate of energy loss, Eq. (18) is difficult to solve. However, if it is assumed that the rate of unknown energy loss ($\dot{E}_{loss}$) and particle radial velocity energy loss ($\frac{1}{2}\dot{m}_s v_{p(radial)}^2$) are negligible with respect to the energy losses associated with maintaining powder hold up in circulation, Eq. (18) can be simplified:

$$d_{cut} = \left(\frac{h_2}{h_1}\right)^2 \left(\frac{k_1}{k_3^2} + \frac{k_1}{k_3^2}\frac{\dot{m}_h}{\dot{m}_g}\right) \tag{28}$$

It is known that energy losses associated with particle collisions are not completely negligible, as the size reduction process requires energy and must be a result of energy transfer from the grind nozzles to the particles and then from the particles to other particles. However it is known that collision energy losses will be less than those associated with maintaining hold up in circulation as long as the rate of particles intersecting with the grind nozzles ($\dot{m}_{nozzles}$) is less than the rate of powder hold up ($\dot{m}_h$). The rate of particles intersecting with the nozzles is a small fraction of the hold up rate due to the low nozzle intersection volume compared to total grind chamber volume. As the rate of particles intersecting the nozzles is by definition a small fraction of the rate of hold up, the hold up will always consume more energy, making this assumption valid.

Additionally, the rate of particles intersecting the grind nozzles should be directly proportional to the solids concentration, hence the associated energy loss will be directly proportional to that for hold up. If the constants in the cut size equation were to be determined empirically, the energy losses associated with particle acceleration for collisions would be accounted for by an increase in the magnitude of constants associated with hold up. It is thus possible to combine Eq. (21), Eq. (27) and Eq. (28):

$$d_{cut} = \left(\frac{h_2}{h_1}\right)^2 \left(\frac{C_2}{{k_3}^2} + \frac{x_1 k_4}{{k_3}^2 d_{Reynolds}\dot{m}_g} + \frac{C_2 \dot{m}_s}{{k_3}^2 x_2} + \frac{x_1}{{k_3}^2 d_{Reynolds} x_2}\frac{1}{E_{sp}}\right) \tag{29}$$

This equation not only shows that cut size can be defined with specific energy (E_{sp}) as a variable with constants describing the system, but also that for different combinations of $\dot{m}_s$

and $\dot{m}_g$, differences in cut size could potentially be observed for the same specific energy. This derivation also indicates that different spiral jet mill geometries will result in different particle sizes for the same value of E_{sp}.

The grinding limit, d_{limit}, or cut size for zero hold up, is of particular interest as it can be determined experimentally. The grinding limit can be approached by allowing the gas mass flow rate to tend towards infinity, or letting the solids feed rate tend towards zero. According to Eq. (29) the grinding limit should differ depending on how it is approached. If the solids feed rate tends towards zero for a constant gas mass flow rate, the grinding limit is given by:

$$d_{limit} = \left(\frac{h_2}{h_1}\right)^2 \left(\frac{C_2}{{k_3}^2} + \frac{x_1 k_4}{{k_3}^2 d_{Reynolds} \dot{m}_g}\right) \tag{30}$$

However, if the grinding limit is approached by increasing the gas mass flow rate to infinity for a constant feed rate the grinding limit is:

$$d_{limit} = \left(\frac{h_2}{h_1}\right)^2 \left(\frac{C_2}{{k_3}^2} + \frac{C_2 \dot{m}_S}{{k_3}^2 x_2}\right) \tag{31}$$

Assuming that changes in d_{cut} and $d_{Reynolds}$ are less than an order of magnitude, it would be expected that a plot of cut size against $1/E_{sp}$ would produce a straight line if either solids feed rate or gas mass flow rate is kept constant. The gradient and intercepts of the line for the constant solids feed rate and the constant gas mass flow rate will differ as indicated in Eq. (29) with the intercepts for a constant gas mass flow rate and the solids feed rate defined by Eq. (30) and Eq. (31) respectively. This behaviour is observed for Product A as shown in Section 3.

For studies involving difficult to micronise materials where x_2 is small, or studies at very high solids feed rates such that E_{sp} is small, it is expected that the response of cut size to varying gas mass flow rate and solids feed rate could be approximated by Eq. (32) as the final term in Eq. (29) becomes large compared to the other terms:

$$d_{cut} = \left(\frac{h_2}{h_1}\right)^2 \left(\frac{C_2}{{k_3}^2} + \frac{x_1}{{k_3}^2 d_{Reynolds} x_2} \frac{1}{E_{sp}}\right) \tag{32}$$

It is important to note if x_2 is assumed constant, Eq. (32) is solely a function of specific energy for a fixed geometry. This may explain why the relationship between particle size and specific energy has been a valid observation in previously reported analysis.

3. EXPERIMENTAL

3.1. Materials and Methods

Product A is a relatively coarse Active Pharmaceutical Ingredient (API) with a high Specific Surface Area (SSA) (>10 m^2/g) that is particularly friable and is used in this study as an example material that does not fit well with standard correlations between cut size and E_{sp}. The response of Product A to varying grind pressures and feed rates was investigated for a fixed geometry. Additionally, h_2/h_1 was also varied at a fixed feed rate and grind pressure.

A single input batch of Product A was micronised with nitrogen on an industrial 8” spiral jet mill with tangential powder entry and eight grinding nozzles. The precise dimensions of the mill cannot be disclosed for confidentiality reasons. The feed and grind gas pressures were controlled to a set point and the gas mass flow rate was measured by a coriolis flow meter. The feed and grind gas absolute pressures were maintained at a constant ratio (5:4) so that the ratio of feed and grind gas mass flow rates is approximately constant throughout the experiment. The screw speed was set for a volumetric feeder and the average solids feed rate was determined by measurement of the input mass and the time taken for the feeder to fully discharge.

Powder was collected by a vaneless axial entry reverse flow cyclonic separator that is attached directly below the grind chamber exit. As it was not possible to sample from a moving stream during micronisation with the available equipment, the total collected powder was tumbled within its container prior to sampling so as to minimise the impact of segregation and variation in solids feed rate. Two 1 g samples were taken for each experiment from six different locations within the container to ensure that the sub-sample was representative of the bulk material. An initially clean container was used for each experiment.

Each 1 g sample was then tumbled prior to taking two smaller 35.0 mg aliquots. Each aliquot was then subject to duplicate particle size analysis with a Malvern Mastersizer 2000 following sonication in a Hydro 2000S sample handling unit. Each individual data point in Figures 9-13 is the mean of four PSD measurements with the error bars reflecting the minimum and maximum values. Error bars on the x-axis in subsequent figures reflect the variability in the measured process parameters.

3.2. Results

3.2.1. Particle Size Distribution (PSD) Analysis

By convention x50 (particle diameter for which 50% of the volume of assumed spherical particles are smaller) has used as the basis of the discussion for milled materials and has been compared to E_{sp}. Additionally, when discussing the cut size of a cyclone x50 is again typically reported. However, some materials produce a significant quantity of very fine particles during micronisation regardless of the solids feed rate or gas pressure as the collision energy is fixed by sonic velocity. This is observed in the Particle Size Distribution (PSD) plots in Fig. (8) for Product A (x50 = 191 µm, x90 = 358 µm) where it can be seen that, at a low specific energy (1140 kJ/kg), there is a significant “fine shoulder” of particles of less than a micron in diameter. The impact of this fine shoulder on the distribution is that the x50 only changes from 2.0 µm to 1.7 µm with increasing energy in Fig. (8) whereas the x90 decreases from 5.6 µm to 3.8 µm.

Fig. (8) Product A PSD Plots

Due to the presence of a persistent portion of fine particles, analysis of x50 will not give an accurate reflection of what is happening to the cut size for Product A. x90 is the assumed spherical particle diameter for which 90% (by volume of assumed spherical particles) of the PSD is of a smaller diameter. x90 will be assumed to be equivalent to the cut size for the subsequent analysis as it provides a better reflection of changes in cut size than x50 for product A.

The x90 measured for Product A micronised at a range of solids feed rates and gas mass flow rates with a fixed geometry is shown in Fig. (9) for solids feed rate versus x90, and Fig. (10) for specific energy versus x90, with the data grouped according to gas mass flow rate. As can be observed from Fig. (10) there is a significant level of variability in the x90 obtained for a given specific energy depending on the gas mass flow rate.

Fig. (9) **Solids Feed Rate against x90 with Lines of Constant Gas Mass Flow Rate**

Fig. (10) **E_{sp} against x90 with Lines of Constant Solids Feed Rate**

3.2.2. Variation in Solids Feed Rate and Gas Mass Flow Rate

According to Eq. (29) it is expected that a plot of $1/E_{sp}$ versus x90 will give a relationship of the form "y = mx + c" depending on whether the solids feed rate or gas mass flow rate is held constant while the other varies. Fig. (11) and Fig. (12) are plots of $1/E_{sp}$ against x90 for the set of data shown in Fig. (9) and Fig. (10), with the data grouped by gas mass flow rate and solids feed rate in Fig. (11) and Fig. (12) respectively. As can be seen from Fig. (11) and Fig. (12), straight lines are obtained which differ depending on whether the solids feed rate or gas mass flow rate is held constant. For ease of data presentation and analysis, outliers have been removed from Fig. (11).

Fig. (11) **1/ E_{sp} against x90 with Lines of Constant Gas Mass Flow Rate**

Fig. (12) **1/ E_{sp} against x90 with Lines of Constant Solids Feed Rate**

3.2.3. Variation in Classifier Geometry

Fig. (13) shows $(h_2/h_1)^2$ against x90 for Product A for a fixed solids feed rate of 3.5 kg/hr and gas mass flow rate of 177 kg/hr.

Fig. (13) **$(h_2/h_1)^2$ against x90**

3.3. Analysis

The grinding limits, or y-intercepts from the linear regression analysis, are of particular interest and are expected to take the form of either Eq. (30) or Eq. (31). Linear regression models with an R^2 value of less than 0.9 and derived from less than 10 data points are included in subsequent analysis as the data points are the means of four or more values and the linear regression models are, in most cases, within the error bars associated with measurement and process variability. According to Eq. (30) the grinding limit obtained by solids feed rate reduction (cut size for zero solids feed rate) should be directly proportional to the reciprocal of the gas mass flow rate. This can be seen from Fig. (14) where the reciprocal of the gas mass flow rate is plotted against the grinding limits obtained from the linear regression model reported in Fig. (11).

Fig. (14) **1/ Gas Mass Flow Rate against Feed Rate Reduction Grinding Limit**

The grinding limit approached by increasing gas mass flow rate (cut size for infinite gas mass flow rate) does not however behave as predicted by Eq. (29) and Eq. (31) as observed in Fig. (15) where the solids feed rate is plotted against the grinding limits obtained from the linear regression model reported in Fig. (12).

Fig. (15) **Solids Feed Rate against Gas Mass Flow Rate Increase Grinding Limit**

Although the reduction in grinding limit with increasing solids feed rate in Fig. (15) is small, it is likely to be a real effect. Both the y intercepts from the linear regressions in Fig. (14) and Fig. (15) should by definition be equivalent as they are both the cut size for zero solids feed rate and infinite gas mass flow rate as per Eq. (29). As can be seen from Fig. (14) and Fig.

(15), the y intercepts are similar despite Fig. (15) not matching the expected behaviour of Eq. (31).

Eq. (29) and Eq. (31) have assumed that the mass fraction of collision fragments below the cut size, b, remains constant for varying solids feed rates. Collisions between large primary particles, which are likely to be better at retaining momentum following acceleration by grind nozzles, should be more energetic than collisions between smaller particles which will quickly lose their momentum as a result of fluid forces following exit from a nozzle jet. It would therefore be expected that larger input particles, although requiring more energy for surface area generation, may provide more energetic collisions as a result of their ability to retain momentum. It would therefore be expected that for increased solids feed rates the proportion of collision fragments below the cut size, b, may increase as a result of the average collision energy increasing. For input materials that are particularly sensitive to this effect or where the solids concentration is particularly low (mill starvation), it could be possible to observe a reduction in particle size for increasing solids feed rates at a constant grind pressure. The similarity of the y intercepts in Fig. (14) and Fig. (15) is because the grinding limits in Fig. (14) are both by definition for zero solids feed rate.

The converse of the phenomenon of a reduction in grinding limit, and potentially particle size, with increasing solids feed rate is that the particle size will not reduce as expected with feed rate reduction. Some of the atypical results excluded from Fig. (11) but included in Fig. (12) are thought to be a result of the solids feed rate being so low such that the spiral jet mill was in a state of starvation whereby the grinding process becomes less effective due to the reduced particle concentration.

Although the grinding limit for infinite gas mass flow rate changes with solids feed rate in Fig. (15), is minimal when compared to changes observed in the grinding limit for zero solids feed rate with varying gas mass flow rate per Fig. (14). If variation in the grinding limit, as a result of differences in feed rate, is considered insignificant it is possible to determine a number of the constants specific to Product A and its spiral jet mill. Fig. (16) shows $1/E_{sp}$ against x90 minus the grinding limit obtained by a reduction in feed rate. By rearranging Eq. (29) and assuming $C_2\dot{m}_s/{k_3}^2x_2$ to be insignificant, Eq. (33) can be developed to describe the expected behaviour of Fig. (14):

$$d_{cut} - \left(\frac{h_2}{h_1}\right)^2 \left(\frac{C_2}{{k_3}^2} + \frac{x_1k_4}{{k_3}^2 d_{Reynolds}\dot{m}_g}\right) = \left(\frac{h_2}{h_1}\right)^2 \frac{x_1}{{k_3}^2 d_{Reynolds}x_2} \frac{1}{E_{sp}} \qquad (33)$$

As the y intercept is expected to be zero as per Eq. (33), the y intercept of the linear regression in Fig. (16) has been set as zero.

Fig. (16) 1/ E_{sp} against x90 – Reduction in Feed Rate Grinding Limit

The slopes and intercepts obtained in Fig. (14) and Fig. (16) can be used to determine the milling constants for Product A and the industrial 8" spiral jet mill used during the trial as per Table 1. Calculating the empirical constants by linear regression as demonstrated in this paper is not the most practical method for all data sets. For sets of data that are not appropriately grouped by solids feed rate or gas mass flow rate, it is possible to use a numerical solver to iteratively fit the constants rather than performing linear regression.

$\frac{C_2}{k_3{}^2}$ (µm)	$\frac{k_4 x_1}{k_3{}^2 d_{Reynolds}}$ (µm.kg.hr^{-1})	x_2 (kg^2.kJ^{-1}.hr^{-1})
0.375	28.9	0.121

Table 1 Milling Constants for Product A

Based on the calculated value of x_2 and the magnitude of $d_{Reynolds}$, it is possible to see that the term $C_2\dot{m}_s/k_3{}^2x_2$ is expected to be very small compared to the other terms, and have a negligible impact on cut size. This explains why there is almost no variation in the grinding limit observed in Fig. (15).

The relative magnitude of x_2 to other terms in Eq. (29) means that the resultant cut size is more sensitive to gas mass flow rate than solids feed rate. Product A, and similar materials may therefore be approximated by:

$$d_{cut} = \left(\frac{h_2}{h_1}\right)^2 \left(\frac{C_2}{k_3{}^2} + \frac{x_1 k_4}{k_3{}^2 d_{Reynolds}\dot{m}_g} + \frac{x_1}{k_3{}^2 d_{Reynolds} x_2}\frac{1}{E_{sp}}\right) \tag{34}$$

Eq. (34) with empirically derived constants may be used to form a predictive model for x90. Fig. (17) shows the predicted x90 against actual x90 for the Product A data set including all outliers previously excluded from Fig. (9).

Fig. (17) Predicted x90 against Actual x90 for Product A using Eq. (34)

The cut size equation has also been verified by Fig. (13) as the observed change in cut size with classifier height fits with the prediction as per Eq. (29). It is expected that at either extreme of classifier height, this relationship will breakdown. For a zero classifier height such that $h_2 = h_1$and $(h_2/h_1)^2$ is at its minimum possible value of 1, a coarser output is expected to be seen as the classifier no longer prevents larger particles escaping via a short cut route out of the grind chamber along the wall closest to the gas exit [19].

4. CUT SIZE EQUATION COMPARED TO PREVIOUSLY REPORTED DATA

Zhao and Schurr [16] performed micronisation with gases of differing molecular weight and showed that at high values of specific energy consumption, gases with a lower molecular weight give a smaller average particle size. An even more important observation is that at lower values of specific energy consumption, gases with a lower molecular weight give a larger average particle size. Fig. (18) shows the log-log plot of average particle size against specific energy presented by Zhao and Schurr [16].

Fig. (18) Average particle size against E_{sp} for various motive gases (Zhao and Schurr, 2002)

Based on the assumption of similarity of grinding and similarity of spin ratio, Eq. (29) may be used to investigate the impact of changing gas physical properties such as molecular weight, viscosity and ratio of specific heat capacities on cut size. Fig. (19), which has used Eq. (29) at a fixed solids feed rate (1.5 kg/hr) to model the behaviour of a material to different motive gases, shows a close alignment to previously reported data from Zhao and Schurr [16].

Fig. (19) Predicted cut size against E_{sp} for various motive gases

The cut size equation predicts that at higher values of E_{sp}, gases of a lower molecular weight give smaller cut sizes whilst at lower values of E_{sp} gives larger cut sizes. The smaller cut size at high values of E_{sp} is a result of the grinding limit being smaller for gases of a lower molecular weight, whereas the larger cut size at lower values of E_{sp} is a consequence of there being a lower gas mass flow rate for a given volumetric flow rate and hence greater deceleration of gas is required to maintain a given quantity of hold up in circulation around the grind chamber.

In addition to the cut size equation aligning with the experimental observations by Zhao and Schurr [16], Eq. (32) replicates the "transition" observed by Midoux et al. [8] when plotting E_{sp} against changes in Specific Surface Area (SSA). Fig. (20) shows a typical log-log plot of specific energy against SSA presented by Midoux et al. [15]. Although the cut size equation cannot be used to predict changes in SSA, the cut size can be inferred across a range of values and the spherical SSA can be calculated based on the assumption that all particles are of the cut size diameter. Such a case is shown in Fig. (21), which replicates the typical shape of data obtained by Midoux et al. [15].

Fig. (20) E_{sp} against Change in Specific Surface Area (Midoux et al., 1999)

Fig. (21) E_{sp} against Spherical Specific Surface Area of Cut Size

Fig. (21) shows that the observed "transition" is not a result of a change in the grinding process from fragmentation to attrition, but is a result of the cut size beginning to tend asymptotically towards the grinding limit as a result of the aerodynamic limitations of the system.

Importantly, the location of the asymptote will vary depending on the grind chamber flow conditions and the aerodynamic properties of the material being micronised. Operating within the asymptotic region is generally preferred from a process robustness perspective as the output PSD is insensitive to changes in solids feed rate. It would therefore be recommended to understand how the grinding limit varies with grind pressure and grind chamber geometry and adjust these such that the target particle size can be attained while operating within this asymptotic region.

In addition to the cut size equation aligning with previously reported data for variation in solids feed rate and gas mass flow rate, the variation in classifier geometry and grind chamber height also matches previously reported data in the academic literature [17, 18]. Tuunila and Nyström [17] showed that for a fixed grind chamber height, a reduction in the gap h_1 results in a coarsening of the output PSD, matching the prediction of Eq. (29). Katz and Kalman [18] showed that variation in h_2 with no classifier resulted in no notable change in PSD, also concurring the prediction of Eq. (29).

5. DISCUSSION

Eq. (29), the cut size equation:

$$d_{cut} = \left(\frac{h_2}{h_1}\right)^2 \left(\frac{C_2}{{k_3}^2} + \frac{x_1 k_4}{{k_3}^2 d_{Reynolds} \dot{m}_g} + \frac{C_2 \dot{m}_s}{{k_3}^2 x_2} + \frac{x_1}{{k_3}^2 d_{Reynolds} x_2} \frac{1}{E_{sp}}\right) \tag{29}$$

may have applicability with respect to scale up as it provides a mechanistic explanation for the variation observed in particle size with respect to changes in gas mass flow rate, gas physical properties, solids feed rate, solids aerodynamic properties and classifier geometry. Good agreement is observed with data reported in the academic literature and robust explanations are provided for the observed phenomena of the asymptotic approach to the grinding limit and variation in particle size with different motive gases.

Many of the parameters in Eq. (29) are by definition mill specific, and could be determined by data generated from several materials micronised on a number of mills. The parameter C_1 is a function of particle shape, and therefore should remain similar for a given material micronised by different spiral jet mills so long as the fragmentation process results in similarly shaped particles. It may be possible to consider x_2 as material specific if the mass transport parameter, D, and mass fraction of collision fragments below the cut size, b, remain similar during scale up. The parameter D is likely to be mill specific as it relates to the mass transfer of particles to the grind nozzles, whereas b is likely to be both material and mill specific as it relates to both the collision energy and the fragmentation/attrition process for a given material. Micronisation of a number of materials across several spiral jet mills should be able to show whether D and/or b may be assumed constant or not. The collision energy varies with the size of particles and the momentum they can retain, the angle at which collisions occur and the distance particles can accelerate prior to collision. If the grind angle is kept constant, and the nozzle separation distance maintained by increasing the number of nozzles during scale up, it may be possible to assume that b is material specific. By maintaining similarity of grind chamber shape and overall nozzle intersection volume to total volume ratio (by increasing nozzle diameter with increasing scale) it may be possible to assume D as constant during scale up

A proposed scale up methodology for a high value product between geometrically similar spiral jet mills such that b remains similar with scale (from 4” to 16” or larger for the internal diameter of the grind chamber) is as follows:

1. Micronise at least two inexpensive surrogate materials at small scale and at least one at large scale across a wide range of solids feed rates and grind pressures so as to fully characterise the performance of both spiral jet mills and identify the material specific parameters C_1 and b for both materials. This will allow mill specific parameters to be determined for both the small and large scale spiral jet mill.
2. Micronise the high value product at the small scale to determine its material specific parameters.
3. Combine the material specific constants for the high value product determined at small scale with the mill specific constants for the large scale mill so that Eq. (29) can be used to describe the response of particle size to variation in solids feed rate and gas mass flow rate.
4. Identify which combination of solids feed rate and gas mass flow rate is necessary to robustly achieve a desired output particle size at increased scale.

The cut size equation can be used for more than just process modelling and scale up, it also provides insight into the limitations of the process from an energy efficiency perspective. Four key observations can be made from the cut size equation with regards to possible improvements in energy efficiency:

i. Some geometries are more efficient than others with regards to particle classification as shown in Fig. (13). It could be possible to optimise the classifier height and radius such that C_2 and $\left(\frac{h_2}{h_1}\right)^2$ are minimised and optimise the grind angle and grind chamber shape such that $k_3$is maximised.
ii. Higher gas mass flow rates and reduced gas molecular weight will tend to be more efficient and for a given specific energy will give a finer powder as a result of improved particle classification.
iii. The collision angle and nozzle separation distance will influence the mass fraction of collision fragments below the cut size, b, and could be optimised to increase the momentum exchanged during collisions.
iv. The greatest consumption of energy within a spiral jet mill is keeping hold up in circulation around the grind chamber. Any changes that increase the mass transport of particles to the grind nozzles, D, should lead to increased energy efficiency.

Some of the changes to increase efficiency may contradict each other, for example the collision angle to maximise momentum exchange during collisions may result in a reduced spin ratio, k_3. As such experimentation may be required to find the optimal balance between milling and classification. The energy delivered to a spiral jet mill is primarily consumed by maintaining hold up in circulation, and as such any change to reduce the hold up to solids feed rate ratio without detriment to the aerodynamic particle classification characteristics could lead to increased efficiency

6. CONCLUSIONS

The derivation of the cut size equation brings a significant advancement to the understanding of the spiral jet mill. It provides a reliable explanation for the observed response of cut size to changes in solids feed rate, gas mass flow rate, mill geometry, gas physical properties and material properties. Although the equation cannot be used in its raw form, and requires empirical determination of material and mill specific constants, some of these constants may be scale and gas independent. It would be recommended to further investigate the applicability of the cut size equation to scale up of the spiral jet mill.

7. ACKNOWLEDGEMENTS

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8. NOMENCLATURE

Symbol	Description	Units
b	Time Averaged Mass Fraction of Collision Fragments Below the Cut Size	N/A
C_1	Drag Coefficient as the Reynolds Number Tends Towards Infinity	N/A
C_2	$3C_1\rho_g r/4\rho_p$	m
C_D	Drag Coefficient of Particle	N/A
d_{cut}	Diameter of Particle Balanced by Radial Drag and Centrifugal Force at Classifier	m
$d_{Reynolds}$	Length Scale of Particles at the Grind Chamber Exit	m
d_{limit}	Diameter of Particle Obtained as the Specific Energy Consumption Tends Towards Infinity	m
D	Mass Transfer Coefficient	$m^3.s^{-1}$
E_{sp}	Specific Energy Consumption	J/kg
$\dot{E}_k$	Kinetic Energy Delivery Rate	W
$\dot{E}_{exit\ (gas\ only)}$	Kinetic Energy of Gas Exiting Grind Chamber for Gas Only System	W
$\dot{E}_{exit}$	Kinetic Energy of Gas and Powder Exiting Grind Chamber for Gas and Powder System	W
$\dot{E}_{loss}$	Rate of Energy Loss due to Particle Collisions and Friction	W
h_1	Height of Grind Chamber Exit	M
h_2	Height of Grind Chamber	M
K	Rate Constant for a Bi-Particular Collisions	m^6.kg-1.s^{-1}
k	Ratio of Specific Heat Capacities	N/A
k_1	$3C_D\rho_g r/4\rho_p$	m
k_2	$T/T_{throat}\pi rhkP$	$m.J^{-1}$
k_3	Gas Spin Ratio, v_t/v_r	N/A
k_4	$2/v_{sonic}^2$	$m^{-2}.s^2$
$\dot{m}_g$	Gas Mass Flow Rate	$kg.s^{-1}$
$\dot{m}_s$	Solids Feed Rate	$kg.s^{-1}$
m_h	Total Solids Hold Up in Grind Chamber	kg
$\dot{m}_h$	Theoretical Rate of Powder Hold Up, $\dot{V}m_h/V_{grind}$	$kg.s^{-1}$
$\dot{m}_{collide}$	Rate of Particle Collisions	$kg.s^{-1}$
$\dot{m}_{nozzles}$	Rate of Mass Transfer of Solids to Grind Nozzles	$kg.s^{-1}$
$\dot{m}_{below\ cut\ size}$	Rate of Generation of Particles Below the Cut Size	$kg.s^{-1}$
M_W	Gas Molecular Weight	$kg.mol^{-1}$
P	Grind Chamber Pressure	Pa
r	Radial Position	m
R	Specific Gas Constant	$J.K^{-1}.mol^{-1}$
T	Gas Temperature	K
v_r	Gas Radial Velocity	$m.s^{-1}$
v_t	Particle Tangential Velocity	$m.s^{-1}$

$v_{t\ (gas\ only)}$	Gas Tangential Velocity for Gas Only System	$m.s^{-1}$
$v_{p(radial)}$	Radial Velocity of Particles Exiting Grind Chamber	$m.s^{-1}$
v_{sonic}	Gas Sonic Velocity	$m.s^{-1}$
$\dot{V}$	Gas Volumetric Flow Rate	$m^3.s^{-1}$
x_1	$18\mu h_1 r/\rho_p k_2$	$m.kg.s^{-1}$
x_2	$DbPM_w/RT$	$kg.s^{-1}$
ρ_g	Gas Density	$kg.m^{-3}$
ρ_p	True Particle Density	$kg.m^{-3}$
μ	Gas Viscosity	Pa.s

9. REFERENCES

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