Modeling of Continuous Self-Classifying Spiral Jet Mills Part 2: Powder-Dependent Parameters from Characterization Experiments

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The milling model described in Part 1 has been expanded to a three-level model with the addition of powder-dependent parameter function models with simple material characterization measurements as inputs. This allows the determination of these parameters with minimal consumption of powder. Specifically, the powder-dependent parameters are related to material hardness from microindentation or to a breakage measure from single-impact milling. Three crystalline powders, sodium bicarbonate, lactose monohydrate, and sucrose, have been used to test the described material characterization techniques and expanded milling model. © 2014 American Institute of Chemical Engineers *AIChE J*, 60: 4096–4103, 2014
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Introduction

In Part 1, a population balance model was developed to describe and predict the continuous milling of a spiral jet mill. The breakage functions of this population balance model include parameters that can be subdivided into two categories: (1) mill-dependent and (2) powder-dependent. For a specific mill, the mill-dependent parameter values can be determined through experiments conducted with inexpensive powders. Powder-dependent parameters can be determined with small quantities of high-value powders using characterization experiments.

In previous literature, several material characterization techniques have been used to measure material properties and powder characteristics, which relate to particle size reduction. The most common techniques include: indentation (both micro- and nano-), single-impact milling, flaw analysis, compression tests, and solution theory. Indentation is the most common method, and can be used to obtain several material properties: hardness, elastic modulus, and fracture toughness. Oliver and Pharr[1] developed a technique to determine hardness and elastic modulus from nanoindentation. The Oliver and Pharr technique has been applied to determine hardness measurements of several pharmaceutical solids using atomic force microscopy nanoindentation.[2,3] Meier et al.[4] used nanoindentation not only to determine hardness and elastic modulus but also to measure fracture toughness from cracks resulting from overloaded indentations. Microindentation techniques were used by Singh et al.[5] and Marshall et al.[6] to obtain hardness measurements from indentation with a Vickers tip and elastic modulus values from Knoop indentations. Others have simply related breakage parameters to the Moh's hardness values for well-defined materials.[7] In this study, the microindentation technique was used to measure hardness. A descriptive procedure was developed and used for creating suitable specimens for microindentation of soft, brittle powders.

Single-impact micromilling has been studied extensively by Meier et al. and Vogel and Peukert.[4,8–12] They defined two material parameters using a single impact mill: the resistance against fracture in impact comminution, f_{mat}, and the specific energy a particle can take up without comminution, $w_{m,min}$. These parameters are determined by inverting the population balance of the single-impact mill. Vogel and Peukert[11] have shown that these parameters can be used to build a master curve to model the breakage probability for multiple powders and sizes. Meier et al. have even related the material parameters to material properties from nanoindentation. Specifically, they show a relation between the f_{mat} and $w_{m,min}$ parameters and the brittleness index defined as hardness (H) divided by the fracture toughness (K_c)[4]

This contribution was identified by Priscilla J. Hill (Mississippi State University) as the best presentation in the session "Particle Breakage and Comminution Processes" of the 2013 AIChE Annual Meeting in San Francisco, CA.

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$$\text{Brittleness index} = \frac{H}{K_c} \quad (1)$$

In this study, a micromill was designed to prevent multiple impacts and reduce the required powder consumption. Also, a new material characterization measurement, the breakage measure (BM), has been defined from single impact milling. This measurement is easy to obtain and can be used to predict the powder-dependent parameters in the breakage functions.

De Vegt et al.[13,14] have developed a breakage probability function, which includes many powder properties: fracture energy, crack propagation stress and velocity, flaw size analysis, hardness, stress intensity factor, and so forth. Many of these properties are not simple to measure and were approximated using a solubility parameter. Therefore, these approaches and properties are not used in this study. However, it is noteworthy that the BM from single-impact micromilling theoretically includes all material properties.

Materials and Methods

Three common excipient powders were used in this study: sodium bicarbonate obtained from Arm and Hammer®, α-lactose monohydrate from Foremost Farms® (Product Code 310), and sucrose provided by Michigan Sugar Company. Two material characterization methods were tested in this study: microindentation and single-impact milling. EpoThin® epoxy was purchased from Buehler®.

Hardness: Microindentation

Most previous studies involving indentation use supersized single crystals that are not encountered normally, compressed "bricks" of powder, or much larger and harder blocks of material. As these techniques may alter the measured mechanical properties, a process was developed to successfully create specimens for the microindentation of soft, brittle materials.

Typically, polishing is done with a lubricant to enhance sliding between the specimen and polishing pad and thus prevent excessive scratching. Because all three powders used in this study are water-soluble, the most common lubricant used in polishing, water, could not be used. Many different alcohols, oils, and polymers were tested as possible lubricants. However, all lubricants tested actually held the specimen closer to the polishing pads. This phenomenon caused deep scratches, which led to particle fracture and eventual pull out. It was determined that dry polishing, with no lubricant, was a more suitable technique for soft brittle particles embedded in epoxy, because the specimen could be held slightly above the polishing pad to minimize or prevent excessive scratching.

Each of the three materials used in this study were sampled in the same way. First, a thin layer of EpoThin® epoxy was painted onto a glass slide with a foam brush. Then, particles were dispersed onto the slide using vacuum dispersion. The largest particles available were used for indentation. Typically, the particles used were on the order of 200 μm in diameter. The specimen was allowed to sit for 24 h to allow the epoxy to harden. Ideally, the thickness of the epoxy layer is thinner than the particle diameter so that the tips of the particles will stick out of the epoxy. If this is the case, only the tips of the particles need to be removed by polishing. Once the epoxy was completely hardened, the specimen was dry polished using a sequence of polishing pads from 30 to 1 μm grit size. Polishing was done by hand with no lubricant, as described previously. To polish uniformly, a figure eight motion was used with specimens held just above the polishing pad. Optical microscopy was used to determine the success of each polishing step and when to move to the next grit size. If the majority of surface scratches were on the order of the most recent grit size, the next polishing pad would be used. If not, more polishing would be done with the current polishing pad. If larger scratches were found, a larger grit polishing pad would be reused depending on the scratch sizes. Typically, use of a previous grit size failed to recreate a polished surface from one that was excessively scratched. This was especially true if particles had begun to pull out. More often than not, trying to recreate a surface from an excessively scratched specimen failed. For this reason, foreign material was removed by frequently cleaning the specimen surface and polishing pad with a low gas flow.

Microindentation procedure

Samples of each of the powders were indented with a Tukon 2100™ microindenter. Suitable particles were identified using the built in microscope on the microindenter. Once a particle was located, the low-load indenter tip was positioned and the indent was made at the load desired. Approximately, 50 total indents were made for each powder using a Vickers tip at 10, 15, 20, and 50 g loads. After the indentations were made, the specimen was taken to a high-power optical microscope to be imaged. Digital photographs of each indent were taken, labeled by material and load, and saved for analysis. An example indent is shown in Figure 1.

Microindentation analysis: Hardness

Each indentation photo was used to measure the hardness of the imaged particle. Using ImageJ software, both diagonals of each indent were measured. Then, the hardness (H) was obtained from

$$\text{Hardness}(H) = 1.8544 + \frac{P}{d^2} \quad (2)$$

where P is the load and d is the average diagonal length.

Breakage measurement: Single-impact micromilling

A single-impact micromill was designed, constructed, and used to determine the BM of each material. The micromill

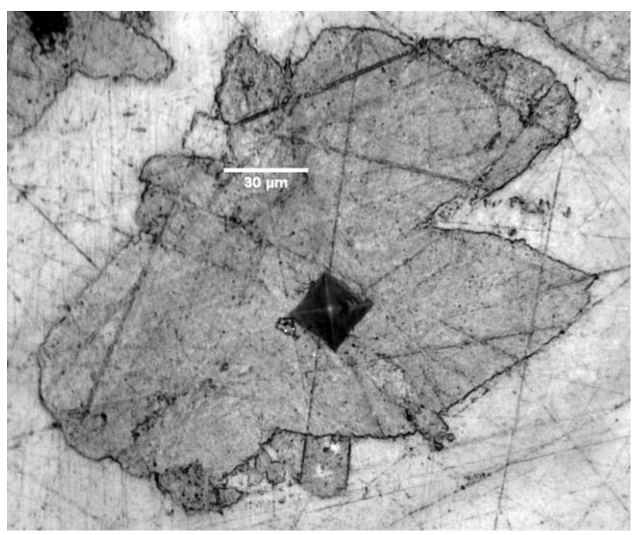

Figure 1. Optical image of 50-g load Vickers indent of sodium bicarbonate.

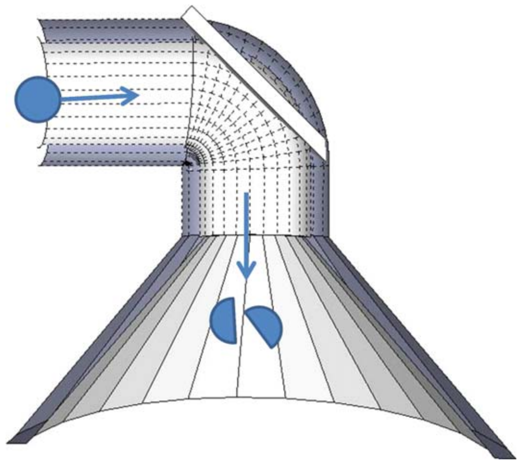

Figure 2. Single-impact micromill design.

[Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

design is shown in Figure 2. The micromill used the same feed funnel and eductor that was feeding the jet mill. Particles were fed into the feed funnel and accelerated at a fixed 45° target. The 45° angle was chosen to minimize reacceleration for multiple impacts and impose both tangential and normal forces. With both forces occurring at impact, multiple breakage mechanisms can be tested. If multiple powders are tested in the micromill at the same energy, any difference in breakage would derive from material differences.

Micromilling procedure

For the energy to be constant in each micromill experiment, the feed pressure, feed size distribution, and feed rate must be identical for each experiment. Each micromill experiment was operated with a feed pressure of 100 psig. An Accurate 102M volumetric screw feeder was used to control the feed rate to the micromill. The feed rate was set to 0.010 g/s, the lowest achievable level, to ensure minimal particle–particle interactions. Feed material was created by sieving powder stocks. Powder between 106 and 150 μm (100–140 mesh) was used as feed. The micromill was fed for a total of 5 min so that approximately 3 g of material would be used. A filter bag was attached over the exit of the micromill to collect product powder. After the experiment was complete, the filter was removed and the product powder was collected. A sample of unmilled material from the feeder was also set aside. After the experiments were completed, a representative sample of each (feed and product) was sized using the Coulter LS13320 laser diffraction particle sizer. Powders were analyzed with the Coulter in duplicate or triplicate.

Micromilling analysis: Breakage measure

For each feed and product particle size distribution, the volume weighted arithmetic mean diameter was determined. Then, a BM was defined by

$$\text{Breakage measure(BM)} = \frac{\bar{x}_\text{f} - \bar{x}_\text{p}}{\bar{x}_\text{f}} \tag{3}$$

Here $\bar{x}_\text{f}$ and $\bar{x}_\text{p}$ are the volume weighted arithmetic mean diameters of the feed and product distributions, respectively. The harder the material the smaller the difference between the feed and product mean diameters will be, as illustrated in Figure 3. Thus the BM varies inversely with the hardness of the material. However, other powder characteristics such as elastic modulus and fracture toughness will affect the BM.

Expanded Model Structure

Expanding on the milling model described in Part 1, the model presented in Figure 4 takes material properties into account so that it can be applied to multiple powders. This model can determine the product size distribution produced by a self classifying air jet mill with only small quantities of powder being consumed. As inputs, the model requires the feed size distribution entering the mill, the mill operating conditions (grinding pressure, pusher pressure, and feed rate), and the hardness of the particle (measured by microindentation) or BM (measured with a single-impact micromill). The model can be subdivided into three parts: a population balance model (Level 1), breakage function models (Level 2) described in Part 1, and powder-dependent parameter function models (Level 3). The powder-dependent parameter function models take the material properties measured by microindentation or single-impact milling: hardness and BM, respectively, and relate them to the powder-dependent parameters in the breakage function models.

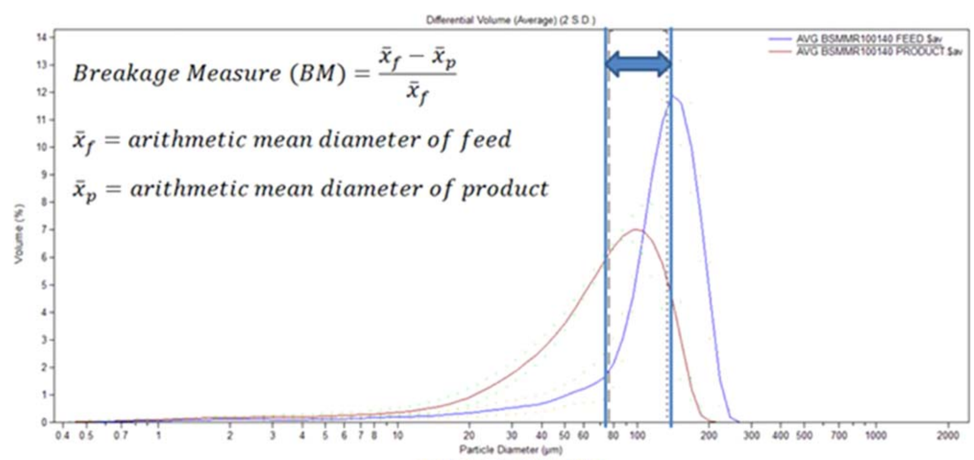

Figure 3. Visualization of breakage measure with sodium bicarbonate micromill results.

[Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

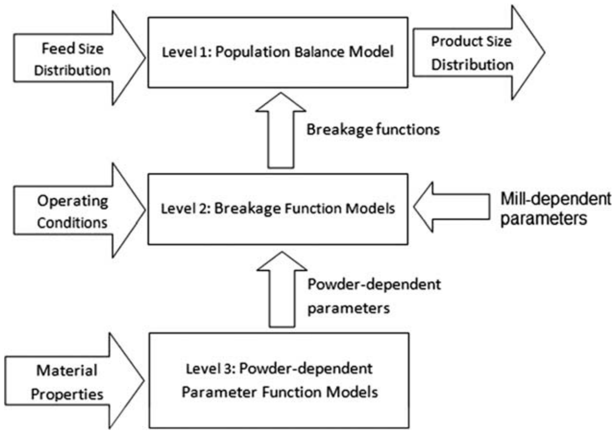

Figure 4. Three-level model architecture.

Although model details can be found in Part 1, a brief description of the model is presented below:

The probability of breakage function (sigma function) is given by

$$\sigma_i=\left(\frac{1}{1+\exp(-n)}\right) \tag{4}$$

where

$$n=w_0+w_{GP}\cdot GP+W_{PP}\cdot PP+W_{FR}\cdot FR+w_{size}\cdot D_i + W_{GPPP}\cdot GP\cdot PP \tag{5}$$

where all parameters are mill-dependent except for w_{size}. The probability a particle of size j breaks into size i is given by

$$\beta_{ij}=\begin{cases}(1-k)*\sigma_j & \text{if } i=j+1 \text{(next in size bin)}\\ k*\sigma_j & \text{if } i=\text{smallest bin}\\ 0 & \text{any other } i\end{cases} \tag{6}$$

where k is conditional probability of chipping function

$$k=\frac{1}{2}\cos(a_0+a_{GP}\cdot GP+a_{PP}\cdot PP+a_{FR}\cdot FR)+\frac{1}{2} \tag{7}$$

where a_0 is the only material-dependent parameter.

In Part 1, w_{size} and a_0 were determined for the three powders tested by fitting experimental results from jet mill experiments. Based on the literature, it is expected that the hardness is inversely proportional to the probability of breakage.[14] Furthermore, since the BM can be thought of as the probability of breakage in a single impact it would be logical that the BM was proportional to the probability of breakage in the multiple impact jet mill. This assumption will be tested for the crystalline powders used in this study. These ideas can be implemented into the model with linear correlations with both the hardness and BM

$$w_{size}=a*x+b \tag{8}$$

$$a_0=c*x+b \tag{9}$$

where x can be either the BM from micromilling or the hardness from microindentation.

All powder-independent parameters of Eqs. 5 and 7 can be determined from experiments on the mill with an inexpensive primary base powder. These experiments will also determine w_{size} and a_0 for the primary base powder. A second set of a_0 and w_{size} can be obtained by running a small number of mill experiments (a single experiment could suffice) with a secondary base powder. Measuring the hardness or BM of the base powders allows determination of the parameters of Eqs. 8 and 9. To predict the performance of the mill for any other powder (given the feed size distribution and operating conditions) all that is needed is measurement of hardness and/or the BM. Equations 8 and 9 provide the powder-dependent parameters, which can be used together with the mill-dependent parameters and mill operating conditions to calculate the breakage functions σ_i and β_{ij}, through which the population balance model will predict the milled powder size distribution for any initial feed composition.

Table 1. Powder-Dependent Parameters of All Test Powders

	Sodium Bicarbonate	Lactose Monohydrate	Sucrose
w_{size} (μm^{-1})	1.81 E −02	4.95 E −03	3.54 E −02
a_0 (–)	1.45 E +00	1.17 E +00	2.06 E +00

Table 2. Hardness and Breakage Measure of All Test Powders

	Sodium Bicarbonate	Lactose Monohydrate	Sucrose
H (GPa)	0.89	1.06	0.76
BM (–)	0.39	0.28	0.55

Powder-Dependent Parameter Function Models

The powder-dependent parameters for sodium bicarbonate, lactose monohydrate, and sucrose, determined by fitting milling data to the breakage function models, are shown in Table 1. All three powders were tested with the microindenter and the micromill to obtain the hardness (H), in GPa, and dimensionless BM. These values are displayed in Table 2.

When the BM was plotted vs. the powder-dependent parameters, w_{size} and a_0, linear relations with positive slope were observed as seen in Figures 5 and 6, respectively. Similarly, when hardness was plotted vs. w_{size} and a_0, linear trends with negative slope were found, as shown in Figures 7 and 8. The BM correlations imply that as the micromill BM increases, the breakage probability in the jet mill will increase. In other words, the more a powder breaks in a single-impact mill the more likely it will break in a multi-impact jet mill, as

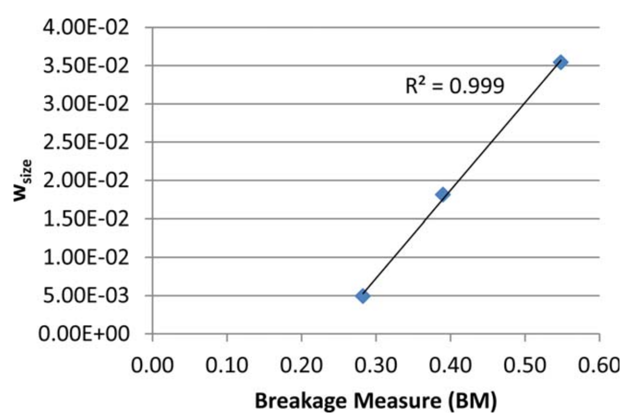

Figure 5. Breakage measure vs. w_{size} powder-dependent parameter.

[Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

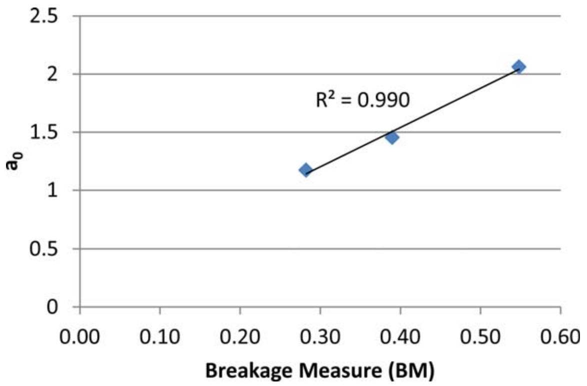

Figure 6. Breakage measure vs. a_0 powder-dependent parameter.

[Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

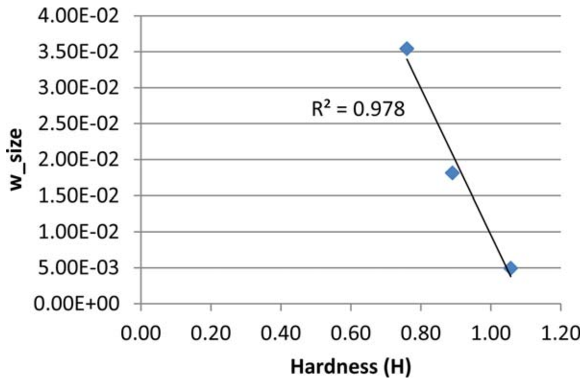

Figure 7. Hardness vs. w_{size} powder-dependent parameter.

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expected. Likewise, the more a powder breaks in a single impact the higher the conditional probability of chipping in the jet mill. Inversely, the hardness correlations say as the hardness increases, the breakage probability in the jet mill will decrease as will the conditional probability of chipping. These correlations align with expectations based on theory.

The linear fits are better for the BM correlations (R^2= 0.999 and R^2= 0.990) compared to the hardness correlations (R^2 = 0.978 and R^2= 0.923). This is expected as the hardness of a powder is just one material property, whereas the BM is

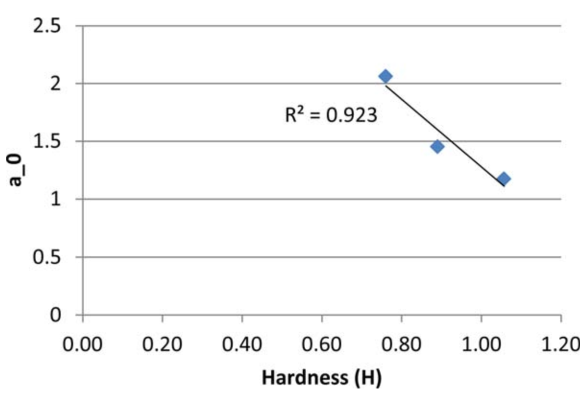

Figure 8. Hardness vs. a_0 powder-dependent parameter.

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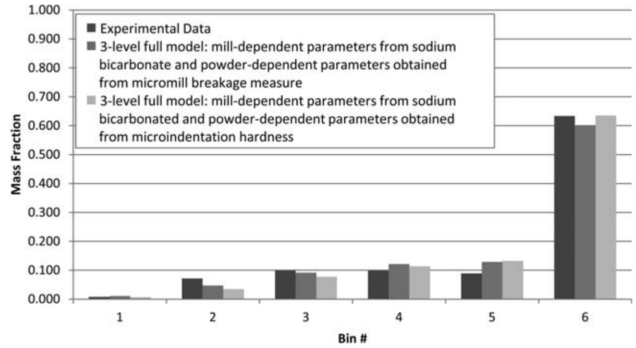

Figure 9. Three-level model: sodium bicarbonate product from jet mill operated at: grinding pressure of 30 psig, pusher pressure of 30 psig, feed rate of 0.100 g/s, and feed size of 106–150 μm.

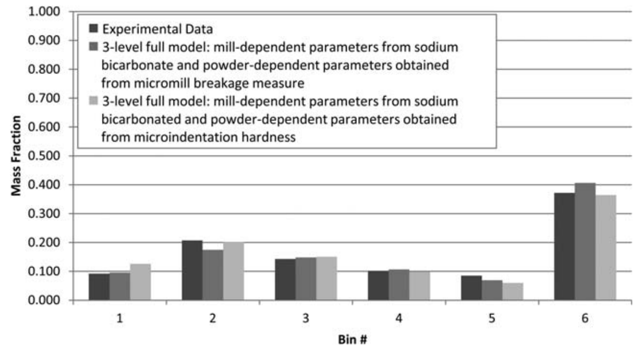

Figure 10. Three-level model: lactose monohydrate product from jet mill operated at: grinding pressure of 30 psig, pusher pressure of 30 psig, feed rate of 0.100 g/s, and feed size of 106–150 μm.

influenced by many material properties. The linear equations determined from the above correlations are for BM

$$w_{\text{size}} = 0.114 * \text{BM} - 0.027 \tag{10}$$

$$a_0 = 3.38 * \text{BM} + 0.189 \tag{11}$$

and for microindentation hardness (H)

$$w_{\text{size}} = -0.102 * \text{H} + 0.111 \tag{12}$$

$$a_0 = -2.93 * \text{H} + 4.21 \tag{13}$$

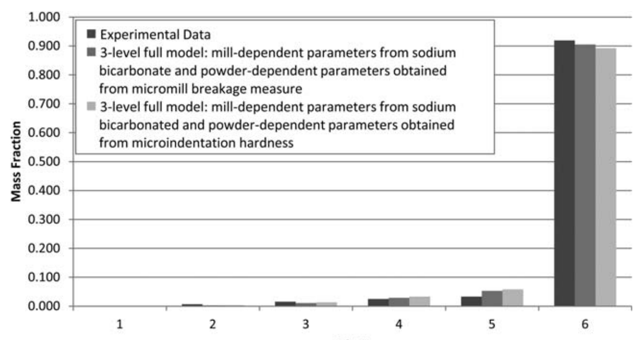

Figure 11. Three-level model: sucrose product from jet mill operated at: grinding pressure of 30 psig, pusher pressure of 30 psig, feed rate of 0.050 g/s, and feed size of 106–150 μm.

Table 3. Sodium Bicarbonate Experimental and Modeled Volume Weighted Geometric Mean Diameter of the Product Distributions for All Jet Mill Runs, Reported in Microns, Using Micromill Breakage Measure (Mod-BM), and Microindentation Hardness (Mod-H)

Operating Conditions															
GP		30			100			30			100			65	
PP		30			100			100			30			65	
FR		0.100			0.100			0.100			0.100			0.100	
Feed	Exp	Mod-BM	Mod-H	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*
1	32.3	33.1	32.8	30.0	30.0	30.0	30.3	30.4	30.3	30.0	30.0	30.0	30.1	30.2	30.1
2	34.6	35.4	34.8	30.0	30.0	30.0	30.8	30.6	30.5	30.0	30.0	30.0	30.1	30.2	30.2
3	35.3	36.5	35.7	30.0	30.0	30.0	30.3	30.7	30.6	30.0	30.0	30.0	30.2	30.3	30.2
4	37.0	36.8	35.9	30.0	30.0	30.0	30.6	30.7	30.6	30.0	30.0	30.0	30.2	30.3	30.2
5a	39.7	40.0	38.6	30.0	30.0	30.0	31.4	30.9	30.7	30.0	30.0	30.0	30.0	30.3	30.2
5b	38.2	40.0	38.6	30.0	30.0	30.0	31.5	30.9	30.7	30.0	30.0	30.0	30.0	30.3	30.2
6	42.6	42.4	40.5	30.0	30.0	30.0	31.4	31.1	30.8	30.0	30.0	30.0	30.0	30.3	30.2
Operating Conditions															
GP		65			65			30			100			65	
PP		30			100			65			65			65	
FR		0.200			0.200			0.200			0.200			0.200	
Feed	Exp	Mod-BM	Mod-H	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*
1	30.7	30.7	30.6	30.3	30.3	30.2	32.7	32.5	32.2	30.2	30.0	30.0	30.7	30.5	30.4
2	30.8	30.9	30.7	30.2	30.4	30.3	32.9	33.1	32.8	30.1	30.1	30.0	30.7	30.6	30.5
3	31.3	31.0	30.9	30.4	30.4	30.3	33.6	34.2	33.6	30.3	30.1	30.0	30.2	30.6	30.5
4	31.5	31.1	30.9	30.6	30.4	30.3	35.1	35.7	34.9	30.1	30.1	30.0	30.3	30.7	30.6
5a	32.5	31.5	31.2	30.8	30.5	30.4	37.7	38.1	36.9	30.0	30.1	30.0	31.1	30.9	30.7
5b	32.5	31.3	31.1	30.4	30.5	30.4	37.0	38.4	37.1	30.1	30.1	30.0	31.3	31.0	30.7
6	34.5	31.8	31.4	30.7	30.5	30.4	41.1	39.7	38.1	30.1	30.0	30.0	31.0	31.0	30.7

Grinding pressure (GP) and pusher pressure (PP) are reported in psig and feed rate (FR) is given in g/s.

Results and Discussion

In Part 1, it was shown how well the model describes breakage using the w_{size} and a_0 parameters fitted from mill experiments. Here w_{size} and a_0 are instead calculated either from microindentation hardness or from micromill BM using Eqs. 10 and 11 or Eqs. 12 and 13.

Figures 9–11 show the experimental and modeled product size distribution for sodium bicarbonate, lactose monohydrate,

Table 4. Lactose Monohydrate Experimental and Modeled Volume Weighted Geometric Mean Diameter of the Product Distributions for All Jet Mill Runs, Reported in Microns, Using Micromill Breakage Measure (Mod-BM), and Microindentation Hardness (Mod-*H*)

Operating Conditions															
GP		30			100			30			100			65	
PP		30			100			100			30			65	
FR		0.100			0.100			0.100			0.100			0.100	
Feed	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*
1	34.4	34.3	34.7	30.0	30.0	30.1	30.3	30.3	30.3	30.1	30.0	30.0	30.5	30.3	30.4
2	37.5	37.0	37.6	30.0	30.1	30.1	30.8	30.9	31.0	30.0	30.0	30.0	31.0	30.5	30.6
3	42.5	41.6	42.8	30.0	30.1	30.1	31.1	31.5	31.8	30.0	30.0	30.0	30.7	30.7	30.8
4	47.6	46.0	48.1	30.0	30.1	30.1	31.5	32.0	32.4	30.0	30.0	30.0	30.9	30.9	31.1
5a	57.7	53.5	56.7	30.0	30.2	30.2	33.3	33.1	33.6	30.0	30.1	30.1	31.0	31.2	31.5
5b	53.6	52.8	55.8	30.0	30.2	30.2	32.8	33.0	33.5	30.0	30.1	30.1	31.3	31.2	31.5
6	63.8	61.3	66.9	30.0	30.2	30.2	34.2	34.2	35.1	30.0	30.1	30.1	31.0	31.5	31.9
Operating Conditions															
GP		65			65			30			100			65	
PP		30			100			65			65			65	
FR		0.050			0.050			0.050			0.050			0.050	
Feed	Exp	Mod-BM	Mod-H	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*
1	30.0	30.1	30.1	30.1	30.1	30.1	30.8	30.7	30.8	30.0	30.0	30.0	30.1	30.1	30.1
2	30.0	30.3	30.4	30.4	30.2	30.2	31.5	31.5	31.6	30.0	30.0	30.0	30.0	30.3	30.3
3	30.0	30.5	30.6	30.0	30.2	30.3	32.0	32.8	33.2	30.1	30.0	30.0	30.0	30.3	30.4
4	30.3	30.6	30.7	30.3	30.3	30.4	32.5	33.9	34.6	30.1	30.0	30.0	30.0	30.4	30.5
5	31.3	31.0	31.2	30.0	30.4	30.5	34.0	36.5	37.6	30.0	30.1	30.1	30.0	30.7	30.8
6	30.0	31.3	31.7	30.0	30.5	30.6	33.5	38.9	40.8	30.7	30.1	30.1	30.0	30.8	31.0

Grinding pressure (GP) and pusher pressure (PP) are reported in psig and feed rate (FR) is given in g/s.

Table 5. **Sucrose Experimental and Modeled Volume Weighted Geometric Mean Diameter of the Product Distributions for All Jet Mill Runs, Reported in Microns, Using Micromill Breakage Measure (Mod-BM), and Microindentation Hardness (Mod-*H*)**

Operating Conditions															
GP	30			100			30			100			65		
PP	30			100			100			30			65		
FR	0.050			0.050			0.050			0.050			0.050		
Feed	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*
1	30.0	30.5	30.5	NA	30.0	30.0	30.0	30.0	30.1	30.0	30.0	30.0	30.0	30.0	30.0
2	30.9	30.8	30.9	NA	30.0	30.0	30.1	30.1	30.1	30.1	30.0	30.0	30.1	30.0	30.0
3	30.6	31.1	31.3	NA	30.0	30.0	30.1	30.1	30.1	30.0	30.0	30.0	30.1	30.0	30.0
4	31.3	31.4	31.6	NA	30.0	30.0	30.4	30.1	30.1	30.0	30.0	30.0	30.7	30.0	30.0
5	30.4	31.8	32.0	NA	30.0	30.0	30.9	30.1	30.1	30.0	30.0	30.0	30.0	30.0	30.0
6	31.9	31.9	32.3	NA	30.0	30.0	30.4	30.1	30.1	30.4	30.0	30.0	30.1	30.0	30.0
Operating Conditions															
GP	65			65			30			100			65		
PP	30			100			65			65			65		
FR	0.100			0.100			0.100			0.100			0.100		
Feed	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*	Exp	Mod-BM	Mod-*H*
1	30.1	30.0	30.0	NA	30.0	30.0	30.3	30.3	30.3	30.1	30.0	30.0	30.0	30.0	30.0
2	30.6	30.1	30.1	NA	30.0	30.0	30.8	30.3	30.3	30.0	30.0	30.0	30.5	30.0	30.0
3	30.5	30.1	30.1	NA	30.0	30.0	30.7	30.3	30.3	30.1	30.0	30.0	30.1	30.0	30.0
4	30.6	30.1	30.1	NA	30.0	30.0	31.1	30.6	30.7	30.4	30.0	30.0	30.6	30.0	30.0
5	30.0	30.1	30.1	NA	30.0	30.0	31.6	30.7	30.8	30.0	30.0	30.0	30.0	30.0	30.0
6	30.5	30.1	30.1	NA	30.0	30.0	34.1	30.8	30.9	30.0	30.0	30.0	30.1	30.0	30.0

Grinding pressure (GP) and pusher pressure (PP) are reported in psig and feed rate (FR) is given in g/s.

and sucrose jet milled with a grinding pressure of 30 psig, pusher pressure of 30 psig, feed size of 106–150 μm, and feed rate of 0.100 or 0.050 g/s (depending on material). Both the hardness measurements and BM measurements lead to reasonably good predictions of powder breakage. For the worst case, between the Level 3 model predictions and the experimental data for sodium bicarbonate, the average difference was 0.022 with a max of 0.043. For lactose monohydrate and sucrose, the average differences were 0.016 and 0.011, respectively, with max differences of 0.034 and 0.027. These differences are lower than the differences of the base case sodium bicarbonate.

Tables 3–5 show the experimental and modeled volume weighted geometric mean diameter obtained from breakage measure (Mod-BM) and microindentation hardness (Mod-*H*) for every experiment described in Part 1. For almost all experiments, the model prediction is in fairly close agreement with the experimental value.

Conclusions

A single-impact micromill was constructed and used to determine the newly defined breakage measure (BM) for sodium bicarbonate, lactose monohydrate, and sucrose. These BMs were then integrated into the developed powder-dependent parameter function models to determine the powder-dependent parameters for each material in the air jet mill. This procedure can be applied to many powders. Small quantities of material can be used in the micromill to determine powder-dependent parameters for high value products (about 3 g were used).

Powder-dependent parameters have also been related to microindentation hardness. The benefit of using indentation is that less material is required compared to micromilling; only a few particles are required. However, specimen preparation is much more difficult. Also, hardness is only one material property that can cause differences in breakage. Therefore, modeling powder-dependent parameters with only microindentation hardness may not work for different materials as well as micromilling where all powder properties affect the resulting BM.

Moving forward, more experimentation is needed to obtain more material properties such as toughness and elastic modulus. Also, more study is needed to understand the relationships between material properties and the powder-dependent parameters, w_{size} and a_0. Finally, more materials with a larger range of powder properties could be tested to extend the development and applicability of the milling model.

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Notation

σ_j = probability of size j breaking in the mill
a_0 = material-dependent constant in k function
a_{GP} = mill-dependent weight of grinding pressure in k function
a_{PP} = mill-dependent weight of pusher pressure in k function
a_{FR} = mill-dependent weight of feed rate in k function
β_{ij} = probability size j breaks into size i
BM = breakage measure from single-impact micromilling
d = average indentation diagonal length
D_i = volume-weighted mean diameter of bin i, μm
f_{mat} = resistance against fracture in impact comminution
FR = feed rate, g/s
GP = grinding pressure, psig
H = microindentation hardness
k = conditional probability of chipping upon breakage
K_c = fracture toughness
P = indentation load applied
PP = pusher pressure, psig
$\bar{x}_f$ = volume weighted arithmetic mean diameter of the feed

$\bar{x}_p$ = volume weighted arithmetic mean diameter of the product
$w_{m,min}$ = specific energy a particle can take up without comminution
w_0 = mill-dependent constant in sigma function
w_{GP} = mill-dependent weight of grinding pressure in sigma function
w_{PP} = mill-dependent weight of pusher pressure in sigma function
w_{FR} = mill-dependent weight of feed rate in sigma function
w_{size} = material-dependent weight of size i in sigma function
w_{GPPP} = mill-dependent weight in sigma function

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