AN INVESTIGATION OF
FINE COAL GRINDING KINETICS

by

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In recent years, a great deal of interest has been shown in developing methods for preparing super-clean coal containing less than 2% ash and 0.5% sulfur. New techniques for recovering fine coal, such as micro-bubble flotation, can achieve the desired result provided mineral matter is sufficiently liberated. To achieve sufficient liberation, however, it is often necessary to grind to a mean particle size finer than 10 microns. Since conventional ball mills are highly inefficient in this fine size range, the stirred ball mill has been proposed as a more suitable means for ultrafine grinding.

A five inch diameter mill has been designed and constructed to investigate the milling characteristics of stirred ball mill grinding using an Elkhorn seam coal. A study of particle size distributions produced under various operating conditions and constant energy input revealed that slow shaft speeds and small ball sizes improved grinding efficiency. Feed percent
solids in the range of 20% to 60% had no effect on grinding efficiency below 100 kwh/ton of energy input. Breakage parameters were also determined in order to model the grinding process using a population balance technique. The grinding model was found to accurately predict particle size distribution for short grind times but to deviate from actual data over longer grinding periods.
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CHAPTER 1.
Introduction

1.1 Need for Fine Coal Grinding

The primary purpose of grinding coal is the liberation of finely dispersed shale particles which cannot be separated by conventional cleaning techniques. It appears that nearly complete liberation of coal and ash occurs when the ground product has a mean size less than ten microns. The needed mean size may, however, be as little as one or two microns. The liberation provided by grinding to these small sizes can assist in producing super-clean coals which contain less than 1% ash and 1% sulfur. New separation techniques, such as the microbubble flotation process, are now being developed at Virginia Tech to produce these high quality coals at a high recovery, provided the feed coal is sufficiently liberated. The recent economic pressures for maximizing coal recovery, and current concerns about limiting SO₂ emissions through government acid rain legislation, make super-clean coals, and coal grinding, particularly important.

Fine coal is also needed in many newly developed processes such as the preparation and burning of coal-water fuels and other liquid coal mixtures. Liquid-coal
technology has advantages over current solid coal processing and burning techniques since a liquid fuel can be pumped, stored in tanks, transported via pipes, and injected into furnaces and boilers. Furthermore, less capital is required to convert a furnace from oil to a pumpable coal-liquid fuel than is required to convert to a solid fuel. Finally, the use of coal-liquid fuels makes it possible for utilities to become less dependent on foreign raw materials, particularly oil.

It is important in transportation and storage of liquid-coal fuels, that the mixture is both pumpable and stable. Ultrafine coal enhances the stability of a liquid coal mixture. Depending on the requirements of a particular application, varying the degree of coal grinding may be a useful technique for controlling the stability of a mixture. Department of Energy research has demonstrated that a median particle size of about 15 microns will give good slurry stability without the need for stabilizing additives.

In processes in which coal is mixed with liquids and burned in engines, a primary concern is wear on valves and other internal machine parts. It appears that wear could be reduced by grinding to ultrafine sizes (Atlantic Research Corp., 1984). Fine coal may be less abrasive on pumps and piping than larger sized coal. Provided it can
be ground inexpensively, ultrafine coal may prove economically beneficial in reducing wear and extending the life of coal burning machinery.

It is apparent that fine coal grinding will be important in producing superclean coals and coal mixtures. Economic conditions in today's coal industry require that size reduction occur at the lowest possible cost. For this reason, it is important to examine the coal grinding process and to determine the operating characteristics which influence its energy efficiency. It is equally important to be able to describe mathematically the breakage process in terms of the rate of size reduction and the size distribution of the final product in order that these fine grinding devices may be properly designed.

1.2 Grinding Techniques

There are several grinding techniques which may be employed to produce fine and ultrafine products. Ball milling is the most commonly used technique in the mineral processing industry today. Although it is the most common, it is not necessarily the most energy efficient method of grinding. Conventional ball milling cannot achieve a minus ten micron product without requiring extremely long retention times and very large energy inputs. Other
approaches to ultrafine grinding include vibratory, impact, ultrasonic, and attrition milling. Past studies conducted by the United States Bureau of Mines indicate that of these methods, attrition milling may be the most promising for producing micronized material in a fast and energy efficient manner (Davis, et al., 1980).

The Bureau-developed attrition process grinds through intense agitation of a mixture consisting of the material to be ground, a grinding media, and a suspending fluid. This is also true of the stirred ball mill process described by Herbst and Sepulveda (1978). Breakage in this process is thought to occur by a shearing action as particles are captured between balls, between balls and the impeller, and between balls and the vessel wall.

The USBM data, shown in Figure 1.1, indicates that the attrition process is far superior to other grinding methods for reducing coarse kaolin to a product size below 2 microns. Studies done by Herbst and Sepulveda (1978) have further demonstrated that a stirred ball mill, similar to the USBM attrition mill, is also more efficient in particle reduction than the conventional ball mill and the vibratory mill. Their study demonstrates that particle size reduction continues in a stirred ball mill after large energy inputs, while conventional ball milling appears to slow and eventually reach a limiting product size at which
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MATERIAL LESS THAN 2 MICROMETERS E.S.D., pct

**Figure 1.1 - Comparison of different types of grinding machines (Davis et al., 1980).**
additional energy input has little effect on size reduction. This is shown graphically in Figure 1.2.

With the reported success of these systems, it is apparent that the stirred ball mill process should be further quantified in terms of its energy requirements and rate of size reduction for grinding of coal.

1.3 Related Studies

1.3.1 Attrition and Stirred Ball Mill Grinding

The first attrition grinder was adapted from a device developed by the United States Bureau of Mines for scrubbing glass sand to remove iron oxide stain (Dasher and Ralston, 1941). In the original scrubbing device, designed by J.E. Norman and O.C. Ralston (1938), the slurried material was scrubbed without grinding media. By simply adding a granular grinding media, along with a suspending liquid, the scrubber was modified to a grinding device.

Full introduction of the attrition grinder was presented several years later by I.L Feld, T.N McVay, H.L. Gilmore and B.H. Clemmons (1960). This work presented diagrams showing the design characteristics of the mill and data for grinding of coarse kaolin. All testing was done in a batch mode using a 5 and a 10-inch diameter mill.
Figure 1.2 - Comparison of the effectiveness of various grinding devices for the ultrafine grinding of a chalcopyrite concentrate (Herbst and Sepulveda, 1978).
M.H. Stanczyck and I.L. Feld further studied the operation of the attrition mill in an open circuit, a closed circuit and a batch mode (1963;1965). Using a 5 inch diameter mill, they determined that the most important operating characteristics in the attrition grinding of kaolin were type, size and shape of grinding media, grinding media to clay weight ratio, rotor speed, pulp density, degree of pulp dispersion, and the angular arrangement of rotor and stator bars. The reported operating characteristics which had little effect on grinding efficiency were pulp temperature, design of rotor cage, rotor clearance, rotor-stator bar interval, addition agents and chamber liners. Their report also indicated that spherical media is preferable to sharp angular or long bladed media with respect to media degradation, machine abrasion and grinding efficiency (1968). The first attempts to quantify the milling process were done while studying the grinding of ceramic oxides in a 5 inch mill (Stanley,et.al.,1974). Milling rate constants calculated for 45x50 mesh dolomite were obtained under a variety of operating conditions and reported to follow first-order kinetics.

Continuing the initial mathematical study of the attrition process, Sadler and Stanley (1975) further verified the assumption of first-order kinetics for coarse
sizes using Ottawa sand as a grinding media. They determined that for the size range studied, grinding efficiency was constant regardless of power input to the mill.

Another more recent study by the Bureau of Mines (Davis, et al., 1980) examines the attrition grinding process for several minerals. An initial qualitative presentation shows attrition milling to be superior to other methods of grinding for producing -2 micron material. Preliminary results are also reported on autogenous grinding of olivine and mica. The majority of data presented in the literature has concentrated on the basic ability of the attrition mill to reduce material to fine sizes. Grinding performance, therefore, is often stated in terms of the ability of the mill to rapidly reduce a coarse material to extremely fine sizes. Very little has been published which presents specific data describing energy requirements and breakage rate values particularly for grinding of coal. Sepulveda and Herbst (1982) have presented data for grinding of several types of materials in a stirred ball mill similar to that used in this research. Aside from their verification of the high efficiency levels of the stirred ball mill in comparison with more conventional grinding methods, they demonstrated that a simplified modeling approach using the Charles Equation produced good results.
in predicting the median diameter size of products for different energy inputs to the mill. Mill operating conditions were shown to have little effect on the energy efficiency of the grinding process leading to the conclusion that product size distributions are generally a function of energy input alone. A generalized grinding model based on the concept of distributed strength for the material being ground was developed and an analytical solution was obtained for batch grinding conditions.

1.3.2 Grinding Kinetics

The first attempts to relate breakage energy to the performance of grinding devices were formulated as empirical energy-size reduction relationships. These "laws of comminution" proposed by Rittinger (1867), Kick (1885), and Bond (1952) have been used for many years to estimate mill capacity and power requirements. The laws are based on work indices which are related to surface area, particle volume or crack length. Since the relationships are empirically based, they only fit experimental data for a limited range of variables and in specific circumstances (Hukki, 1962). None of the laws is capable of defining the ground product in terms of its particle size distribution. In contrast to the empirically developed relationships of
Bond, Kick, and Rittinger, the population balance model is capable of predicting a product size distribution when the initial feed distribution is known. Epstein (1947) established the probabilistic approach to modeling the comminution process. His major contribution to the development of a population balance model was describing the comminution process in terms of a selection function and a breakage function. The selection function, $S_n(y)$, was defined as the probability that a particle of size $y$ would break during the $n$th step of the breakage process. The breakage function $B(x,y)$ was defined as the cumulative weight distribution of particles appearing in or below size class $x$ as a result of breakage from a unit mass of size $y$.

Broadbent and Callcott (1956) used Epstein's selection and breakage functions to introduce a matrix approach for representation of the breakage process. The terms of the feed matrix were obtained from either simple relationships with particle size or from experimental data. The selection and breakage approach was further modified by Gardner and Austin (1962) who derived a size-mass balance equation in differential terms to construct a breakage model for batch grinding. A radioactive tracer technique was employed to determine the breakage parameters and computer analysis provided an iterative solution to their equation.
Using the techniques introduced by the previously mentioned researchers, several investigators have been able to present convincing cases for the use of population balance models as an alternate to the empirical laws of comminution (Herbst, et al., 1971, 1972, 1973; Austin, 1973).

1.3.3 Population Balance Model

Several authors have discussed the formulation of population balance models (Herbst, 1971; Kim, 1974; Reid, 1965). The most useful form of these models is generally agreed to be the size-discretized, time continuous description. This model completely describes the breakage process through size-discretized selection and breakage functions which may be obtained experimentally. The derivation of the model is briefly described below.

The distribution of material in a batch mill can be described in terms of incremental size intervals with a maximum size, $x_i$, and a minimum size $x_{n+1}$. The mass in the size fraction, $m_i(i)$ will therefore be defined as the fraction bounded above by $x_i$ and below by $x_{i+1}$. Each size increment can be defined through screening by establishing the $x_i$ to $x_{i+1}$ size ranges such that they correspond to the geometric progression of a standard sieve series.

To describe the time changes in the original
distribution of fractions m(i) through m(n) it is necessary to track the material which is broken out of and into each individual mass fraction. Most size-mass balance models use the "first-order hypothesis" which states that the rate of breakage of any size is proportional to the amount of that size present in the mill. In this instance, the rate of breakage is represented by the equation

\[
\text{Rate of Breakage} = S_i \cdot m_i(t) \cdot H
\]  

(1)

where \( S_i \) is the fractional rate of breakage of size \( i \) \((\text{time}^{-1})\) and \( H \) is the total mass of material being ground.

The material which enters into a size fraction as a result of the breakage of larger particles must be counted to complete the mass balance. The breakage function, \( b_{ij} \), is defined as the fraction of primary breakage products of material in the \( j \)th size interval which appears in the \( i \)th size interval. Using this value, the rate of appearance of material in the \( i \)th interval from the \( j \)th interval can be expressed as

\[
b_{i,j} \cdot S_j \cdot m_j(t) \cdot H
\]  

(2)

To describe the full breakage of particles from the \( j \) size into smaller intervals, a cumulative breakage function can be defined as follows:
By final mass balance of particles leaving and entering a size interval, the basic form of the batch grinding equation is achieved.

\[
B(i,j) = \sum_{k=\eta}^{i} b_{k,j}
\]  

(3)

\[
\frac{d[m_i(t)H]}{dt} = \sum_{j=1}^{i-1} b_{ij} S_j m_j(t)H - S_i m_i(t)H
\]  

(4)

The selection function may be dependent on the operating characteristics of the mill at time \( t \) and is thus said to be environment-dependent. The breakage function, on the other hand, is thought to be a function of the characteristics of the material being ground and therefore environment-independent (Herbst and Fuerstenau, 1968).

The above model has successfully been applied to dry ball milling systems but it may not be strictly applicable to other systems. Since it is desirable to take advantage of the mathematical simplicity of a linear model for engineering applications, it is useful to study the possible application of the population balance model to other grinding systems.
1.4 Scope of the Present Investigation

Previous studies on the stirred ball mill process have focused on the grinding of many different types of materials. Little work has been done which specifically concentrates on coal. In this investigation, the stirred ball mill grinding process is examined as it relates to producing ultrafine coal products with a mean particle size less than 10 microns. A primary interest in this investigation is to determine the effect of the controllable variables in the milling process on grinding rate and energy consumption. Controllable variables include media size, weight percent solids of the slurry, rotational speed of the central shaft, and grind time. The population balance technique for grinding simulation is also applied for the stirred milling process to determine its ability to represent product distributions over short and long grind periods.
CHAPTER 2
Experimental Techniques

The experimental portion of this research consisted mainly of grinding experiments performed on one coal, using one stirred ball mill, operated in a batch mode. Standard sizing techniques were used on coarse material while sub-sieve material was measured using a particle size analysis system similar to the familiar Coulter Counter. Power input to the mill was measured using a torque sensor mounted on the central shaft of the stirred ball mill.

2.1 Coal Sample

The coal used during this investigation was obtained from United Coal Company's Wellmore operation in Southwestern Virginia. The raw product was a bituminous coal, typical of the Appalachian region, which was mined from the Elkhorn seam. Approximately two hundred pounds were removed from a raw coal stockpile and stored in barrels at the lab.

The large coal was reduced below 20 mesh by passing it through laboratory roll crushers. This product was then cleaned in a heavy media suspension at a specific gravity
of 1.30 to reduce the ash level to approximately 6.5%. A large portion of the cleaned coal was hammer milled and wet screened at various sizes to produce monosized feeds for determining grinding rates. The sized products were split into 350 gram lots and stored for grinding experiments. Other portions of the cleaned coal were passed through a small hammer mill and stored for subsequent testing.

2.2 Grinding Equipment

All experiments conducted during this investigation were performed in a batch mode using a stirred ball mill designed and constructed specifically for this research. The mill, shown in Figure 2.1, was designed for effective swirling action of the media and material to be ground. Rounded shafts, chambers, and pins were used to minimize the amount of internal wear and to provide for easy construction.

In order to eliminate rust problems, the mill shell, impeller pins, and impeller shaft were all constructed from stainless steel. The mill shell consisted of a 1/8-inch thick stainless steel tube having inside dimensions of 5 inches in diameter and 6 inches in length.
Figure 2.1 - Three dimensional schematic of batch stirred ball mill.
as shown in Figures 2.2 and 2.3. The shell was surrounded by a larger tube to create a cooling jacket for the mill. The sealed jacket was fitted with two small steel tubes which were used to circulate cooling water across the inner vessel as tests were conducted.

The central shaft consisted of a 3/4-inch diameter stainless steel rod with six, 3/8-inch diameter pins to provide stirring. The pins were 4 inches long and were offset by 90 degrees at 1-inch increments along the length of the shaft. The mill lid was allowed to rest on top of the mill in most cases; however, for very high stirring speeds the lid was strapped in place. A teflon bearing was used to provide a seal between the lid and the central shaft.

The entire mill assembly was mounted on a variable speed drill press with a 1 horsepower drive motor as shown schematically in Figure 2.4. A series of belts and pulleys enabled the stirring speed to be varied from 200 to 3500 rpm. A movable support platform allowed the mill to be raised or lowered for easy loading and unloading of the sample and media.

A Teledyne Model A-05 torque transducer with a rotational speed pick-up was used to provide a measurement of power input and rpm. The signal from the transducer was amplified on a Brewer Engineering Model DDJ-335A/2 signal
Figure 2.2 — Front view of 5-inch diameter stirred ball mill.

NOTE: ALL DIMENSIONS GIVEN IN INCHES
Figure 2.3 - Top view of 5-inch diameter stirred ball mill.
Figure 2.4 - Schematic of mill mounted to drill press (Mankosa, 1986).
amplifier to provide a digital readout of torque in inch-pounds, rotational speed in rpm, and energy input in horsepower. The torque transducer was mounted above the mill on the central shaft. Grinding media consisted of 1/4-inch, 1/8-inch, 3/32-inch, and 1/16-inch steel burnishing media obtained from Pioneer Steel Ball Company. The media were formed from a hard carbon steel and were thus prone to rusting. Stainless steel media are more desirable if one can afford the added expense. The carbon steel balls required a great deal of care to prevent rusting. Especially in the small sizes, these balls retained a significant amount of water and had to be dried immediately after use.

2.3 Sizing Equipment

Sizing of the raw feed and the product was carried out using 8" diameter Tyler series sieves ranging from 20 to 500 mesh. A mechanical sieve shaker, manufactured by Ro-Tap, was used for preliminary separation of dry coal, followed by manual wet screening to insure proper size segregation. An Electrozone 80XY particle size analyzer manufactured by Particle Data Inc. was used to determine the size distribution of sub-sieve particles appearing in both the product and feed samples.
The Electrozone analyzer uses a measurement principle in which particles suspended in an electrolyte flow between two electrodes through a small orifice contained in a glass tube. As particles pass through the orifice, electrical pulses are generated at rates from a few thousand to a few hundred per second, depending on flow velocity, orifice size, and particle concentration. When the particle concentration is kept low, individual particles can be counted and sized. The amplitude of each pulse is directly proportional to the volume of the particle. By assuming the particle to be roughly spherical, the volume can be converted to an "equivalent spherical diameter" as is often used in sedimentation measurements. Size distribution data obtained by measuring particle pulses is digitized and stored in computer memory. A schematic drawing of the particle measuring circuit is shown in Figure 2.5.

Ground products from the stirred ball mill often had size distributions which spanned the range from screenable sizes to sub-sieve sizes. In all cases, the products from the mill were carefully wet screened to capture any material larger than 500 mesh. Two chambers, each containing a different sized measuring orifice, were available for sub-sieve size analysis. Each orifice allowed acquisition of distribution data within a specific
Figure 2.5 - Schematic of size measuring technique employed by Elzone 80-XY particle size analyzer (Davis, 1986).
size range. Because the ground product size distributions often occurred across a broader size range than either orifice was capable of counting, both orifice tubes were required for complete analysis. The distribution data from each tube was collected and stored through an on-line link up with a Perkin Elmer mini-computer. A program was written which combined the distributions from each tube along with any size data which may have been obtained through wet screening. The resulting solution was a table showing the complete product size distribution by weight over a chosen range of size classes. The program also calculated a cumulative weight percent passing for each size fraction. The complete program along with an example of the computer generated size distribution curves appears in Appendix A.

2.4 Torque Measurement

The torque applied to the central shaft of the batch mill was measured to accurately define the power drawn by the mill during grinding. The Teledyne Model A-05 torque transducer, attached directly to the shaft, contained strain gauges which measured torque deflection and transmitted an output signal to an amplifier manufactured by Brewer Engineering. The amplifier converted the analog
signal into a digital display of torque in inch-pounds. The transducer also monitored rotational speed of the shaft which was similarly displayed by the amplifier.

The displayed torque and shaft speed were used to calculate the power applied to the shaft using the following formula:

\[ P = (2 \pi n) T. \]  

(5)

where

- \( P \) = power input by the shaft (in-lb/min)
- \( T \) = torque (in-lb.)
- \( n \) = rotational velocity (rpm).

By converting power into Kilowatts, the equation becomes

\[ P = 1.182 \times 10^{-5} Tn. \]  

(6)

If the specific energy of grinding, \( E \), is defined as the amount of energy consumed during grinding in Kwh/Ton, an equation for specific energy can be derived from the power-torque-relationship:
where

\[ t = \text{time (sec)} \]

\[ W = \text{weight of material ground (tons).} \]

Applying the conversions

\[ 1 \text{ ton} = 9.0703 \times 10^5 \text{ grams} \]

and

\[ 1 \text{ hour} = 3600 \text{ sec} \]

and combining equation (6) with equation (7)

\[
E = \frac{(1.182 \times 10)(9.0703 \times 10)}{3600} Ttn
\]

\[
E = 2.978 \times 10^5 \frac{Ttn}{w}
\]

where

\[ E = \text{specific energy of grinding (kwh/ton)} \]
\[ t = \text{time (sec)} \]
\[ T = \text{torque (in-lb)} \]
\[ n = \text{rotational velocity (rpm)} \]
\[ w = \text{weight of material being ground (grams)}. \]

The digital display of torque was generally constant across the time period used for each test. Initial readings on torque appeared slightly higher than readings occurring later during the testing. These higher readings can most likely be attributed to the torque sensing equipment since they occur in the absence as well as the presence of grinding media and material regardless of the test conditions. Values displayed by the amplifier were observed regularly in 15 second intervals and recorded. The values were numerically averaged to determine the torque applied over the test period. This procedure is similar to that used by Sepulveda(1982) after he determined that the minor variation in torque readings could accurately be approximated by assuming a linear relationship in torque change throughout the duration of the test.
2.5 Experimental Procedure

Initial tests on the 5-inch stirred ball mill were conducted by charging the mill with media such that the vessel was 50% filled by volume. An amount of coal slurry which corresponded to 100% filling of void spaces was blended with the media as it was loaded in the mill. In the tests involving very short grind times, it was particularly critical to load the media and coal slurry so as to obtain good blending and filling. This was accomplished by carefully measuring the quantities of coal, water, and media necessary for the test and then layering the inner vessel alternately with the media, coal, and water.

The original ball charge at 50% mill filling was weighed to establish the general weight of media which would be used during all subsequent tests. The ball charge consisted of an equal mixture of 1/4-inch, 1/8-inch, and 3/32-inch balls and weighed 3610 grams. Rather than constantly changing the amount of the monosized media to correspond to 50% mill filling, the media weight for each test was held constant for all ball sizes to allow accurate comparison of torque and energy requirements.
Similarly, the original calculations on void filling at 43% solids indicated that 150 grams of coal would be an appropriate sample size for grinding. All subsequent tests were conducted on the same weight of material, unless otherwise noted, regardless of particle size or size distribution. The amount of water used during the testing was adjusted to achieve the desired percent solids of the feed. No compensation was made in the weight of media or coal to account for the increased or decreased volume of water in the grinding chamber.

2.6 Product Sampling

The first test results were obtained by charging the mill, grinding, and then discharging the entire mill contents for media separation and sizing. The ground product was then discarded and the entire process repeated to obtain product sizing at the next time increment. This process required extensive sample preparation and a great deal of lab time.

To reduce the time requirements, a product sampling technique was developed. The technique involved halting the grinding process at various time increments and withdrawing a sample of the product. A scoop sampler was used to withdraw approximately 20 grams of material from
the mill. The withdrawn material consisted of both ground coal slurry and media with the slurry fraction weighing 4 to 5 grams. An equal weight of clean dry media was placed back into the mill prior to additional grinding. This process was repeated over the entire testing period; thus, several data points could be obtained without discharging the mill. Great care was taken to obtain a representative sample by withdrawing material from several areas of the mill at each time increment.

A comparison of size analysis data obtained by totally discharging the mill and by using the sampling technique shows good agreement between the product distributions. Figure 2.6 shows a comparison of cumulative percent passing versus size for a hammer mill feed ground for one minute. Comparison of the actual point values of these distributions, shown in Table 1, demonstrates that there is no more than 5 percent variance in the calculated passing values at each size increment. After only one minute of grinding, the potential for variance in the two product distributions should be greater than most tests done in this research. After longer periods of time, the mill material becomes more fluid and homogeneous and the sample is thus more likely to be representative of the entire mill contents. It is important to remove both coal and media in order to fully account for material which may
Figure 2.6 - Comparison of sampling techniques for 1-minute grind.
### Table 1 - Comparison of Sample Technique

<table>
<thead>
<tr>
<th>Size Class</th>
<th>Particle Size Microns</th>
<th>Cumulative Full Mill</th>
<th>Percent Passing Scoop Sample</th>
<th>Variance</th>
<th>% Variance</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>725.0</td>
<td>100.00</td>
<td>100.00</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>513.0</td>
<td>100.00</td>
<td>100.00</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>363.0</td>
<td>100.00</td>
<td>100.00</td>
<td>-</td>
<td>-</td>
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<tr>
<td>4</td>
<td>256.0</td>
<td>100.00</td>
<td>100.00</td>
<td>-</td>
<td>-</td>
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<tr>
<td>5</td>
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<td>99.46</td>
<td>0.14</td>
<td>0.14</td>
</tr>
<tr>
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<td>98.75</td>
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<td>-0.13</td>
</tr>
<tr>
<td>7</td>
<td>90.5</td>
<td>93.28</td>
<td>94.26</td>
<td>-0.98</td>
<td>-1.05</td>
</tr>
<tr>
<td>8</td>
<td>64.0</td>
<td>78.38</td>
<td>79.42</td>
<td>-1.04</td>
<td>-1.33</td>
</tr>
<tr>
<td>9</td>
<td>46.0</td>
<td>58.28</td>
<td>59.25</td>
<td>-0.97</td>
<td>-1.66</td>
</tr>
<tr>
<td>10</td>
<td>32.5</td>
<td>41.90</td>
<td>43.30</td>
<td>-1.40</td>
<td>-3.34</td>
</tr>
<tr>
<td>11</td>
<td>23.0</td>
<td>32.50</td>
<td>32.10</td>
<td>0.40</td>
<td>1.23</td>
</tr>
<tr>
<td>12</td>
<td>16.3</td>
<td>24.16</td>
<td>24.52</td>
<td>-0.36</td>
<td>-1.49</td>
</tr>
<tr>
<td>13</td>
<td>11.5</td>
<td>16.25</td>
<td>15.60</td>
<td>0.65</td>
<td>4.00</td>
</tr>
<tr>
<td>14</td>
<td>8.1</td>
<td>9.90</td>
<td>9.48</td>
<td>0.42</td>
<td>4.24</td>
</tr>
<tr>
<td>15</td>
<td>5.8</td>
<td>4.87</td>
<td>4.77</td>
<td>0.10</td>
<td>2.05</td>
</tr>
<tr>
<td>16</td>
<td>4.1</td>
<td>1.16</td>
<td>1.21</td>
<td>-0.05</td>
<td>-4.31</td>
</tr>
<tr>
<td>17</td>
<td>2.9</td>
<td>0.00</td>
<td>0.00</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>18</td>
<td>2.1</td>
<td>0.00</td>
<td>0.00</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>19</td>
<td>1.5</td>
<td>0.00</td>
<td>0.00</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>20</td>
<td>1.0</td>
<td>0.00</td>
<td>0.00</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>
be coated around the balls or trapped in void spaces. At very high percent solids concentration, or during dry grinding, it may be necessary to evaluate product size by discharging the entire mill contents since there appears to be severe coating of material which would be difficult to sample using the scoop technique.
CHAPTER 3
Experimental Results

Several operating variables have been examined in terms of their effect on energy requirements and rate of size reduction. These include media size, rotor speed, weight percent solids, feed size and grinding time. Previous investigations concentrated on extended grinding times and very large energy inputs. The data presented in this section represents coal grinding at grind times generally less than 50 minutes and energy inputs below 100 kilowatt-hours per ton. For those tests conducted under conditions of constant energy input, the time of grinding was varied depending upon the measured torque values.

3.1 General Operating Characteristics

To establish a preliminary understanding of the energy requirements for operating the stirred ball mill, torque measurements were recorded under a variety of operating conditions. In all cases, the feed coal had a natural size distribution produced after hammer milling 1/4-inch material. Typically, this coal was 60% passing 100 mesh and contained 14% mineral matter. The torque relationships are important since shaft torque is
proportional to energy input during grinding. This data is shown in Figures 3.1 through 3.3.

Contrary to conventional ball mill grinding, no maximum is observed in the plot of torque vs. media load shown in Figure 3.1. Instead, a continuous increase in torque can be seen as media load is increased up to 100% filling (7.6 kg) at constant stirring speed. Since there is no cascading or centrifuging of material in a stirred ball mill, the torque is always seen to increase as the mill load increases regardless of how that increased load is achieved.

Figure 3.2 demonstrates the effect of increased shaft speed on shaft torque using a constant weight of media. It is observed that as the stirring speed increases the torque value also increases. This relationship is true for all ball sizes. The addition of coal to the mill has little additional effect on the measured torque since this extra load is only a fraction of the load created by the grinding media. In fact, tests were conducted with up to 300 grams of coal at 43% solids with little effect on the torque readings.

In examining the effect of ball size on measured torque, the overall weight of material in the mill was held constant. Figure 3.3 indicates that as the ball size decreased the measured shaft torque also decreased. It
Figure 3.1 – Effect of media load on torque requirement.
Figure 3.2 - Effect of stirring speed on torque requirement (Mankosa, 1986).
Figure 3.3 - Effect of ball diameter on torque requirement.

- 3610 grams media
- 150 grams coal
- 43% solids
- 335 RPM
appears that it is easier for the impeller to move through the smaller balls than through the larger balls. This is indicated by the observed fluctuations which occur on the torque sensing equipment as well as by the noise created during mill operation. During torque measurements using the 1/4-inch balls, the displayed values of torque changed rapidly within 0.5 in-lbs of the mean. As the smaller balls were tested, the degree of fluctuation about the mean decreased. At the smallest size the fluctuation was below the measurable limits of the sensing equipment. The amount of noise produced from interaction between the media, chamber walls and impeller followed a similar pattern with the noise decreasing as the balls became smaller.

3.2 Equal Energy Testing

3.2.1 Feed Percent Solids

To examine the effects of variations in the feed percent solids, the stirred ball mill was operated under the conditions given in Table 2. During this series of tests the initial weight of the coal sample remained the same, but the amount of water was varied for each test to achieve the desired feed slurry.
### Table 2 - Standard Conditions for comparison of Feed Percent Solids

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample Weight</td>
<td>150 grams</td>
</tr>
<tr>
<td>Feed Size</td>
<td>60% -100 mesh</td>
</tr>
<tr>
<td>Rotor Speed</td>
<td>335 rpm</td>
</tr>
<tr>
<td>Media Weight</td>
<td>3800 grams</td>
</tr>
<tr>
<td>Media Size</td>
<td>1/4, 1/8, 3/34 mix</td>
</tr>
<tr>
<td>Coal Type</td>
<td>Elkhorn Seam, 13.7% Ash</td>
</tr>
</tbody>
</table>
The relationship between feed percent solids and product size was investigated by grinding at solids concentrations of 20, 43, and 60 percent by weight while holding the energy input to the mill constant. This was accomplished by adjusting the time of grinding based on the measured torque on the central shaft during the milling process. Within the range of solids concentrations investigated, the 20% mixture required the least amount of torque. The torque requirement increased with an increase in solids concentration.

Figure 3.4 demonstrates the results of grinding at an energy input of 94 kwh/ton. At this level of energy input, the product distributions are identical with a median size of 5.2 microns. This indicates that at low energy levels, the grinding efficiency is unaffected by solids concentration. The time requirements, on the other hand, favor a higher solids concentration since it will allow equal energy efficiency at higher mill throughput. For example, the 60% solids mixture was ground for only 10 minutes while the 43% and 20% mixtures were ground for 14.2 and 20.5 minutes respectively.

To further examine the effect of solids concentration, grinding was continued with a larger energy input. Figure 3.5 shows the resulting product distributions obtained after 280 kwh/ton of energy was input to the mill. After
Figure 3.4 - Effect of percent solids on product size distribution at 94 kwh/ton.
Figure 3.5 - Effect of percent solids on product size distribution at 280 kWh/ton.
this considerably larger energy input, the 60% solids mixture produced a product with a median particle diameter of 7 microns and the 20% and 43% solids mixtures produced a product with a median particle diameter of 3.8 microns. This indicates that after an extended grinding period, the grinding efficiency is influenced by solids concentration. A higher solids concentration seems to adversely affect the ability of the stirred ball mill to efficiently reduce the feed to extremely fine sizes. Again, the time of grinding was significantly shorter for the higher percent solids feeds.

It is interesting to note that an additional 186 kwh/ton energy input was required to further reduce the 5.2 micron product to 3.8 microns. This large amount of additional energy which was necessary to reduce the end product by only 1.4 microns in diameter demonstrates the significance of a small change in the median particle size of a ground product in the ultrafine size ranges.

3.2.2 Dry Grinding

As an extension of the investigation of feed percent solids, the mill was charged with 150 grams of dry coal and run using conditions similar to those used for wet grinding. The feed consisted of the same Elkhorn seam
coal at 6.5% ash. The mill water jacket was used to cool the inner vessel during the dry test since there was considerable heating of the inner chamber. The mill was again operated in a batch mode. There was no air sweeping of the inner chamber during the dry grind to attempt to remove ultrafine material.

Figure 3.6 demonstrates the product size distributions attained for wet and dry grinding after an equal energy input of 90 kwh/ton. The product distributions indicate that the addition of water enhances the grinding efficiency of the mill. A median product diameter of 8.5 microns was measured for the dry grind which compares to a median product diameter of 5.2 microns in the wet grind.

It was apparent during this test that there may be some error in the measured particle sizes of the dry distribution. This is suspected since there was a heavy coating of fine coal on the balls and central shaft. This indicates that there may have been particle breakage followed by melting and agglomeration of fine particles. Since a particle agglomerate would be counted as a large diameter particle in the size analyzer, the true particle distribution may be misrepresented. Testing at higher energy inputs seemed inadvisable after the results of the initial testing.
Figure 3.6 - Effect of dry grinding on product size distribution.
3.2.3 Shaft Speed

By varying the belts and pullies on the power drive, it was possible to investigate the effects of rotor speed on grinding efficiency. The lowest attainable speed was 216 rpm and the highest speed investigated was 830 rpm.

All mill conditions were held constant during the tests and a ball mixture was used to minimize any potential effects of ball size. The feed consisted of Elkhorn seam coal containing 13.8% ash and having a natural size distribution resulting from one pass through a hammer mill.

In Figure 3.7 the product size distributions for grinding at 216, 521, and 830 rpm are plotted. Each product was attained after grinding at a fixed energy input of 63.5 kwh/ton. The test results indicate that for low energy inputs, slower shaft speeds provide more energy efficient grinding than faster shaft speeds. In this particular instance, the 216 rpm test reduced the 65 micron feed to a median product diameter of 6 microns while the products produced at 521 and 830 rpm had median diameters of 7.4 and 9.0 microns respectively.

The relationship between shaft speed and median particle diameter shown in Figure 3.8 indicates that there is a linear relationship between the size produced and the
Figure 3.7 - Effect of shaft speed on product size distribution.

E = 63.5 kwh/ton
43% solids
media: 3800 g mix
Figure 3.8 - Effect of shaft speed on median product diameter at 63.5 kwh/ton.
shaft speed between 200 and 900 rpm at low energy inputs. Although it appears beneficial in terms of energy efficiency, the longer time requirements for grinding at slower shaft speeds may be prohibitive. As can be seen in Table 3, the grind time required at 216 rpm was over 10 times that of the 830 rpm test at equal energy input. By extending the energy input over a longer period of time, however, the product size was reduced by an additional 3 microns.

3.2.4 Ball Size

Product size comparisons were done using balls of 1/4-inch, 1/8-inch, 3/32-inch, and 1/16-inch diameter at 216 rpm and 43% solids concentration. The energy input was again held constant by adjusting the time of grinding based on the torque values measured on the central shaft. Since the smaller balls required less shaft torque, the time of grinding during the tests with the small balls was longer at equal energy input.

Figure 3.9 demonstrates the relationship between ball size and product size distribution for grinding a coal feed at an energy input of 90 kilowatt-hours per ton. It is apparent from the diagram that at equal energy input the smallest particle sizes are produced with the
Table 3 - Products produced at 64 kwh/ton and varied shaft speeds

<table>
<thead>
<tr>
<th>RPM</th>
<th>Time (min)</th>
<th>Median Product Diameter (microns)</th>
</tr>
</thead>
<tbody>
<tr>
<td>216</td>
<td>23.4</td>
<td>6.0</td>
</tr>
<tr>
<td>521</td>
<td>5.0</td>
<td>7.4</td>
</tr>
<tr>
<td>830</td>
<td>2.14</td>
<td>9.0</td>
</tr>
</tbody>
</table>
Figure 3.9 - Effect of ball size on product size distribution.
1/16-inch balls. It was also found that as the media size increased, the product size also increased. A ball mixture consisting of equal weights of 1/8-inch, 3/32-inch, and 1/16-inch balls produced a product distribution falling very near the average of the products produced from the monosized media tests. Table 3 lists the median particle sizes of the products corresponding to each ball size after 90 kilowatt-hours per ton energy input as well as the grinding time required to input 90 kwh/ton.

Figure 3.10 demonstrates the product size distributions attained when different ball sizes are used to grind a 100x150 mesh monosized feed. In this test, the time of grinding was held constant at 10 minutes while the media size was varied. The size distributions of the ground products appeared to be nearly identical with a median particle diameter of 8 microns. The energy input required for grinding to this size varied with media diameter. Again it was found that the most efficient grinding occurred when the smallest balls were used. This test indicates that with 1/16-inch media it is possible to reduce a feed with a median particle size of 128 microns to a median size of 8 microns with an energy input of only 35.1 kwh/ton.

Grind times of 1 minute and 5 minutes were also
Figure 3.10 - Effect of ball size on product size distribution after a 10-minute grind (100 x 150 mesh feed).
examined using each ball charge. In the case of these short grind times the product size distributions produced with the different sized media were not the same. In Figure 3.11 a 100x150 mesh monosized feed is shown after grinding for 1 minute. This figure shows that the finest product distribution is produced with the larger 1/8-inch balls. A slightly larger product is produced as the balls become smaller. As grinding progresses to 5 minutes, the product distributions begin to become closer together as can be seen in Figure 3.12. This seems particularly true in the coarser ends of the product distributions. At this stage of grinding it is apparent that the smallest balls produced a slightly finer product than the larger 3/32-inch and 1/8-inch balls. The 1/8-inch balls have nearly the same product size as the 1/16-inch balls, but the mid sized 3/32-inch balls did not grind the coal as fine as either of the other two sizes.

At the 10 minute stage of grinding, all distributions fall together, as was previously indicated, with a median size of 8 microns. This series of tests, conducted on a 100x150 mesh feed, presents evidence that there is a relationship between the rate of particle size reduction and ball size. The tests also indicate that particle size of the feed influences the rate of grinding.

To examine the effect of ball size on the rate of size
Figure 3.11 - Effect of ball size on product size distribution after a 1-minute grind (100 x 150 mesh feed).
Figure 3.12 - Effect of ball size on product size distribution after a 5-minute grind (100 x 150 mesh feed).
reduction, the median product diameter was traced as it changed through a 30 minute grind time. This plot appears in Figure 3.13. As indicated by the product distributions, the smaller balls do not grind the 100×150 mesh monosized feed as quickly as the larger balls over the first 3 to 4 minutes of grinding. After 3.5 minutes of grinding, the median particle diameter produced using the 1/16-inch balls is the same as that produced by the 3/32-inch balls. The median diameter produced by each ball becomes equal after 10 minutes of grinding.

3.3 Analysis of Breakage Parameters

To quantify the process of particle size reduction in greater detail it is necessary to determine the breakage parameters which were discussed in Chapter 2. There are several techniques which may be employed to determine the selection and breakage functions for use in the population balance model. Two experimental techniques involve: 1) grinding a series of monosized feeds, and 2) using radioactive tracers. In this investigation, monosize feeds were prepared and tested for those size ranges which could be prepared through screening. The values for sizes which were below 500 mesh could not be tested and thus were projected through techniques developed by Herbst.
Figure 3.13 - Change in median product diameter over 30-minute grind time for 1/16-, 3/32- and 1/8-inch balls.
3.3.1 Selection Function

The selection functions for the screenable size fractions were determined by plotting the feed disappearance curves of each monosize as it was ground at 335 rpm and 43% solids using 1/4-inch media. A typical disappearance plot obtained for a series of monosized feeds is shown in Figure 3.14. Over the time period plotted, first order breakage kinetics were observed. This finding has also been reported elsewhere (Sadler, Stanley and Brooks, 1975; Herbst and Sepulveda, 1978). In this case it was necessary to use very short grinding times since the feed disappeared so quickly in the coarse size range.

A steeper slope on the feed disappearance plot indicates a higher value for $S_i$ and thus a higher rate of size reduction. Contrary to the typical disappearance plots presented by prior investigators, the slopes do not continually decrease with particle size. In this case, the rate of breakage of the largest fraction, 28x35 mesh, does not appear to be as fast as that for the next smaller size fraction. This observation lead to the preparation of Figure 3.15 which demonstrates the relationship between
Figure 3.14 - Feed disappearance plots for various size fractions using 1/4-inch media.
Figure 3.15 - Breakage rate function versus feed particle diameter using 1/4-inch media.

335 rpm
43% solids
1/4" balls
feed particle size and rate of size reduction using 1/4-inch balls. This figure shows that the rate of grinding increases as the particle size increases to a maximum at the 35x48 mesh size class. After this maximum, there is a decrease in grinding rate as the particle size becomes smaller.

The selection functions for sub-sieve size fractions were determined by plotting the known values of $S$ against the normalized size function as has been previously done by Herbst and Fuerstenau (1968). Figure 3.16 shows that the experimentally determined selection values fall along a straight line on a log-log plot. The sub-sieve selection values were extrapolated from the curve under the assumption of first order breakage kinetics. The resulting values are presented in matrix form in Appendix B.

3.3.2 The Breakage Function

The cumulative breakage distribution functions were estimated from a relationship between the breakage function and the selection function presented by Herbst and Fuerstenau (1968). The relationship is presented as follows:

$$S_j B_{ij} = F_i$$
Figure 3.16 - Selection function versus geometric mean size using 1/4-inch media.
where \( F_i \) is the fines production rate which can be determined from the slope of a plot of the cumulative fraction finer than \( X_i \) versus grinding time. Several investigators have reported that the breakage distribution function is normalizable for many different materials (Herbst and Fuerstenau, 1968; Klimpel 1964; Austin et al., 1971). This means that particles of all sizes for a particular material will break down to the same fractional distribution. Thus, a log-log plot of \( F_{ij} \) against the relative size \( X_i/X_{j+1} \) should result in a straight line. This type of plot, shown in Figure 3.17 did not result in a single straight line for the data prepared in this investigation. Instead, a series of lines can be drawn through the data points with each successively smaller size fraction following a line which is closer to the previous line. It is suspected that the relationship demonstrated in Figure 3.17 can be related to the possible inaccurate measurement of the finest sizes by the particle size measurement technique. Nevertheless, an assumption was made that the breakage functions for the sub-sieve sizes were normalizable along the line produced by the lowest experimentally determined breakage values. The complete breakage function is given in Appendix B.
Figure 3.17 - Cumulative breakage function versus normalized size variable.
4.1 Percent Solids

Tests conducted in this research indicate that there is no change in coal grinding efficiency between 20 and 60% solids for energy inputs of 94 kwh/ton. This tends to agree with the findings of Sepulveda (1980) for grinding of several different materials between 20 and 50% solids. For a higher specific energy input (280 kwh/ton), however, a coarser size distribution is produced at 60% solids while the distributions at 20% and 43% are, once again, identical.

As the coal becomes finer, the surface area of the entire mass increases. The increased surface area will possibly result in increased adsorption of water and thus create a highly viscous mass. Sepulveda showed, through grinding with heavy oils, that increasing the viscosity of the mill material is detrimental to efficient grinding. This effect seems to apply in the case of high percent solids as well. During high solids testing the inner vessel walls become heavily coated with a pasty layer of media and fine coal. This action renders the balls motionless and thus they are unable to make contact with
fine particles and other media.

Provided the energy input is not excessive, it is possible to reduce the time required for size reduction and increase mill throughput by grinding at higher solids concentrations. This is true at 94 kWh/ton between 20 and 60% solids where the higher concentration results in a 50% reduction in residence time.

It may not be valid to assume that grinding efficiency is independent of solids concentrations below 94 kWh/ton. For example, below 20% solids, it is expected that fine coal particles would be affected by hydrodynamics in the grinding chamber. Since the fine coal has a specific gravity very near to that of water, particles would seem to travel with the grinding fluid and thus have a lower chance of impacting directly with the balls or between the balls and the grinding chamber.

If the size or type of media changes, the conclusion of equal energy efficiency at varying percent solids may become invalid. Very small or light media is more likely to be influenced by the amount of water which is added to the mill. At very low solids concentrations, for example, the light media may flow too easily with the mill material.
4.2 Rotor Speed

The chief observation made during the examination of various stirring speeds was that lower speeds provided finer grinding than higher speeds at equal energy input. The tradeoff, however, is that a significantly longer period of grinding is necessary to achieve an equal energy input as the shaft speed decreases. Since the minimum allowable shaft speed in this research was 216 rpm, it is not known to what minimum speed the ball mixture will continue to grind with increasing efficiency. At some point the rotor speed will become too slow to provide enough kinetic energy for the media to effectively shear or crush the material being ground.

From observations of the grinding process during numerous tests it was observed that the increased efficiency at lower speeds may be the result of a more fluid swirling action of the media in a lower breakage zone. The slower speeds ground the product with much less noise and less fluctuation in torque readings. More noise indicates that there is increased media interaction, increased turbulence in the rotating mass, and accelerated mill and media wear. None of these actions contribute to efficient grinding.

Further observation of the shaft pins indicated that there was a parabolically-shaped grinding zone which
appeared in the mill at high speeds. Upon emptying the mill after one experiment it was observed that the coating of coal on the top stirring pin existed only on the outside edges. For each succeeding pin below, the coating extended closer toward the central shaft. At slower speeds, the coating may not even appear on the uppermost pin and the angle of the cone becomes smaller. The majority of media and coal during slow speed tests seems to reside in the lower portion of the mill. As the shaft speed increases, media is thrown to a higher region in which less contact can be made with coal particles. This is shown schematically in Figure 26.

Other investigators (Sadler, et al., 1974; Nguyen, 1981) have demonstrated an advantage for high shaft speed in terms of energy efficiency. In these cases however, the rotor speeds were varied at speeds far greater than those tested during this research. The increase in grinding efficiency with slower rotor speed as found in this investigation, may indicate that there is a different breakage process occurring between very low and very high shaft speeds. It may be possible that when the shaft speed increases to a particular level there is turbulent action in the mill. Increasing the rotation to a higher speed may cause centrifugal forces which lead to uniform
Figure 4.1 - Diagram of parabolic depression occurring in the batch mill at higher shaft speeds.
circulation of media around the inner wall of the grinding chamber. This action may change the mechanism of breakage to one similar to low speed ranges and thus increase the grinding efficiency.

4.3 Ball Size

Based on equal energy input, small balls produced a finer product distribution than larger balls. This observation is consistent with the findings of other researchers who have found a similar relationship for grinding of coal in attrition and stirred ball mills (Stanczyk and Feld, 1972; Herbst and Sepulveda, 1978).

From simple observation of the grinding tests, one might conclude that the energy input to the mill is being used more effectively with smaller balls. The mill operates with much less noise and much smaller variance in the measured torque values when grinding with the 1/16-inch balls. This observation indicates that there is more uniform swirling of mill material in the grinding chamber. For larger sized balls, there is less uniform flow and a more noise. Energy is apparently lost to ball motion in regions outside of the breakage zone.

Since there is a larger number of small balls than large balls at equal weights, it is possible that
grinding efficiency is improved because of the increased number of points of media contact. In addition, the smaller interstitial spaces between the smaller diameter balls may provide less opportunity for very fine particles to escape contact. The probability of contact may be related to the rate of particle breakage and is likely to be a function of both the diameter of the media and the average particle diameter of the feed.

It was shown during the determination of the grinding rate constants that a 35x48 mesh feed had an initial grinding rate which was faster than the size fractions both immediately above and immediately below when it was ground using 1/4-inch balls. It is not proper to assume from this data that 1/4-inch balls are optimum for the 35x48 mesh feed since a different ball size might result in a higher breakage rate. In order to determine optimum ball size for various feed sizes, breakage rates can be measured using several ball sizes on different monosized feeds. Figure 4.2 shows the feed disappearance plots for a 100x150 mesh feed ground with varying media size. Closer evaluation of Figure 4.2 reveals that the 1/8-inch balls provide a faster rate of size reduction than 1/16-inch, and 1/4-inch balls. This relationship is more easily seen in the plot of the breakage rate function vs. ball diameter shown in Figure 4.3. Apparently, ball size is
Figure 4.2 - Feed disappearance plots for 100 x 150 mesh feed using various ball sizes.
Figure 4.3 - Breakage rate function versus ball diameter.
important to the initial rate of particle size reduction. Particles which are larger than a specific size (35x48 mesh in the case of 1/4 inch balls) may not be easily broken because they are not being contacted in the same manner in which a smaller particle is being contacted.

Tracking the median particle size as it changes over time, as was done in Figure 3.13, further demonstrates that there is a relationship between the rate of grinding of a product and the size of the media. Although the initial rates of size reduction using the 1/16 and 3/32-inch balls was less than that of the 1/8-inch balls, the products eventually became equal in size indicating that the long term grinding rate becomes equal after 10 minutes. This indication however, may not be valid since subsequent testing revealed that some fine material may not have been accounted for in the sizing distributions after long periods of grinding.

Careful examination of Figure 3.13 shows that size reduction begins immediately for the 1/8-inch and 3/32 inch media and continues at a constant rate until roughly 5 minutes of grinding. The 1/16-inch balls do not begin rapid size reduction until nearly 45 seconds after beginning the milling process. After that time, size reduction occurs at a very rapid pace until it slows at 5 minutes with the other balls. The rate of size reduction
of the median particle size appears to be greater for the smaller balls than for the larger balls in the range of 100 to 10 microns. This is likely to result from the ability of the smaller balls to contact coal particles when larger balls cannot as was previously discussed.

4.4 Energy-Size Relationship

The results of most of the grinding tests presented in the preceding chapters can be represented on a single log-log plot in terms of specific energy input vs. median particle diameter of the product. Herbst and Sepulveda (1978) have previously shown that stirred ball mill grinding can be represented by Charles Equation. By assuming that the feed size is much larger than the resulting product size, this equation reduces to the following form

\[ E = A \left( d_p \right)^{-\alpha} \]  

(11)

where \( E \) is the specific energy input, \( A \) is the Charles Constant, \( \alpha \) is the slope of the equation when plotted on log-log paper, and \( d_p \) is the median product size.

A plot of Charles Equation for the data collected in this research appears in Figure 4.4. Through linear
Figure 4.4 - Charles Law plot for stirred ball milling of Elkhorn seam coal.
regression analysis, the Charles constant was determined to be 5896 and the slope was 2.33. These values are near those reported for stirred ball mill grinding of Lower Freeport and Montana Rosebud coal by Herbst and Sepulveda (1978). As was previously reported, it appears to be more difficult to grind coal in the stirred ball milling process than it is to grind other industrial materials. It may be possible that the tendency for coal to plastically deform makes it more difficult to grind than harder, more brittle materials.

Sepulveda (1980) concluded that although there is some minor variation in grinding efficiency which can be attributed to the operating characteristics in the mill, stirred ball mill behavior is to a good first approximation only dependent on the energy input per unit mass and not the manner in which the energy is input. The data presented in Figure 4.4 seems to indicate that the effect of mill operating conditions may in fact be more pronounced than was previously reported by Sepulveda. The three darkened data points which tend away from the straight line plot result from the tests of the influence of shaft speed at equal energy input. As the shaft speed increased, the product particle size also increased. This relationship seems to indicate that if all the plotted points on the Charles plot were run at a higher speed the
line would be shifted to the right effectively raising the value of the Charles constant. Coincidentally, all of the data points available for plotting the energy-size relationship were obtained while milling at 335 rpm.

On the lower portion of the graph, three points are plotted which lie in a vertical progression. These points demonstrate the difference in energy required to produce a 12 micron product by varying the media size. Again, there may be a slight change in the movement of the Charles plot depending on the size of the media used in grinding.
The population balance model, described in Chapter 1, can be symbolically expressed as follows:

\[
\frac{dm_i(t)H}{dt} = \sum_{j=1}^{i-1} b_{ij}S_i m_i(t)H - S_i m_i(t)H
\]

where \( S_i \) and \( B_{ij} \) are experimentally determined parameters defined as the selection function and breakage function respectively. Although the above equation has no analytical solution, it can be simplified to one with an exact solution if \( S_i \) and \( B_{ij} \) are assumed to be independent of changes in size distribution in the mill. Under these circumstances, the size distribution occurring after one breakage event can be viewed as the feed distribution for the following breakage event and so-forth. The differential mass balance equation can thus be solved numerically through matrix multiplication and computer iteration.

The Crude Euler technique has been used to solve the population balance equation for stirred ball mill grinding of coal. The numerical expression for this technique is as follows:

\[
y_i - y_{i-1} = \Delta x f(x_{i-1} , y_{i-1})
\]
A computer program was written to compute the particle size distribution resulting from stirred ball mill grinding of coal when given an initial feed size distribution and a grinding time. Input data consisted of experimentally determined selection and breakage functions as described in Chapter 3.

Over very short grinding periods, the computer model was found to accurately predict the product particle size distribution of the coarse size classes. In the fine sizes however, there is a slight deviation from the experimentally determined product distribution. The model predicts that a larger quantity of material will be broken out of the finer size range than was actually measured by the particle size analyzer. This is demonstrated in Figure 5.1 after only 1-minute of grinding. Figure 5.2 shows that after a longer grinding time, 5- minutes, the difference in computed and experimentally determined size distributions becomes more pronounced.

The increase in product fineness predicted by the computer model after 1-minute may in fact be more representative of the true product distribution than the experimentally determined distribution. The computer generated distribution indicates that there is a possibility that fine particles are not being counted in the product measuring process. This error is likely since
it was not possible to obtain a single orifice tube for use in the size analyzer which was capable of detecting the entire particle distribution in the product, particularly in the finest size ranges.

Because the feed particles disappear so quickly in the stirred milling process it was expected that the computer model would begin to deviate from experimentally determined size data after short grinding times. As has been observed in the modeling of ball mill grinding, the first order assumption for breakage kinetics seems to apply over short grind times but to become invalid after longer grind times. In the case of stirred ball milling, the rate of initial size reduction is very rapid and thus non-linear breakage seems to occur very early in the grinding process. Non-linearity can be associated with the fact that the size-discretized selection functions are not time independent. In addition, overestimation of product fineness resulted from prediction of the particle breakage rates for the very small size classes based on the measured values for large particles.

A complete list of the computer program, written in basic, is included in Appendix C.
Figure 5.1 - Comparison of experimental and predicted size distributions after a 1 minute grind.
Figure 5.2 - Comparison of experimental and predicted size distributions after a 5 minute grind.
6.1 Conclusions

1. Stirred ball milling was shown to be effective in producing fine coal products with a median size between 3.8 and 10 microns.

2. A scoop sampling technique was shown to accurately represent the product size distribution when compared with fully discharging the mill contents.

3. Variations in slurry percent solids between 20 and 43% were found to have no effect on grinding efficiency for energy inputs up to 280 kwh/ton. Below 94 kwh/ton, there was no effect on grinding efficiency between 20% and 60% solids.

4. Between 840 and 216 rpm, the grinding efficiency of the stirred milling process was found to increase with decreasing shaft speed.

5. Tests using monosized ball feeds of 1/4", 1/8", 3/32" and 1/16" diameter showed increasing grinding efficiency as ball size decreased.

6. The energy required for particle size reduction was found to be closely approximated by Charles Equation but dependent on operating characteristics of the
mill for particle grinding to 5 microns.

7. Initial rate of particle breakage was shown to be dependent on the relative size of the feed particles and the media diameter.

8. A computer model of the breakage process, using the population balance approach, showed good predictive capability at very short grind times but deviated from experimental data after 5 minutes.

6.2 Recommendations

Based on the observations and data obtained in this investigation, the following are areas recommended for future research:

1. Since it is desirable to achieve ultrafine product sizes at minimal cost, additional research should be conducted to determine the optimum combination of operating conditions for minimizing energy consumption.

2. Grinding test should be conducted using particle dispersion agents to determine if it is feasible to reduce the viscosity effect seen at high percent solids. Such studies could allow lower mill residence times and higher throughput levels.

3. Studies should be conducted to determine the amount
of media wear which occurs in the milling process.

4. The effect of different ball materials should be investigated as a possible means for reducing mill energy requirements while achieving fine particle distributions.

5. The observed relationship between ball size and particle size in determining the initial rate of coal breakage should be further quantified through additional grinding of monosized material. The effect of ball mixtures should also be investigated using monosized feeds.

6. Additional studies should be conducted to describe the mechanism of particle breakage at very high and very low shaft speeds. This work could include the effects of variations in the design of the impeller and the chamber walls.

7. Continuous studies, and large scale studies should be conducted to verify the observations made using the small batch mill constructed for this research. The focus of this additional work should be to determine the potential for practical application on an industrial scale.
REFERENCES


APPENDIX A
PROGRAM FOR BLENDING SIZE DISTRIBUTION DATA
MICHEAL D. BROWN
APRIL 1984

DIMENSION P(30),RETAN(30),PASS(31)
OPEN (1,FILE='INPUT')
N=0
DO 17 J=1,20
   P(J)=0
17 CONTINUE
READ(1,*)(P(I),I=1,5)
14 CONTINUE
READ(1,*)WGHT,TSIZE,NCLASS,NUMBR
WRITE(*,11)NCLASS,NUMBR
11 FORMAT(5X,'DISTRIBUTION FROM CLASS ',I2,' TO CLASS ',I2
   I=NCLASS
   OLDY=1.0
15 CONTINUE
READ(1,*,END=50)X,Y
IF(X.GE.TSIZE) GOTO 100
IF(I-NUMBR) 202,203,204
203 PRINT*,'THERE IS SOME AMOUNT OF MATERIAL PASSING THE
 & SMALLEST SCREEN SIZE IN THE PREVIOUS DISTRIBUTION.
 & YOU SHOULD DEFINE A WIDER RANGE OF SIZES.'
204 CONTINUE
202 CONTINUE
WRITE(*,201)I,TSIZE
201 FORMAT(2X,'CLASS ',I2,' =+',F6.2,' MICRONS')
TSIZE=TSIZE/1.4142
I=I+1
100 CONTINUE
DR.Y=1 SOB
Y=Y/100.0
PCT=OLDY-Y
P(I)=PCT*WGHT+P(I)
OLDY=Y
IF(I.LE.NUMBR) GOTO 15
50 CONTINUE
N=N+1
IF(N.EQ.2) GOTO 300
CLOSE (1)
OPEN(1,FILE='PUTIN')
GOTO 14
300 CONTINUE
TOTAL=0
DO 110 I=1,NUMBR
   TOTAL=TOTAL+P(I)
110 CONTINUE
TRETAN=0
DO 120 I=1,NUMBR
    RETAN(I)=P(I)/TOTAL*100
    TRETAN=TRETAN+TRETAN(I)
    PASS(I)=100.0-TRETAN
120 CONTINUE
WRITE(*,130)
130 FORMAT(/3X,'SIZE CLASS',3X,'WEIGHT',3X,'% RETAINED',
     &3X,'CUM % PASSING')
DO 200 M=1,NUMBR
    WRITE(*,140)M,P(M),RETAN(M),PASS(M)
200 CONTINUE
<table>
<thead>
<tr>
<th>X MAX</th>
<th>SIZE</th>
</tr>
</thead>
<tbody>
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</tr>
<tr>
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<td>2.04</td>
</tr>
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</tr>
<tr>
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<td>2.32</td>
</tr>
<tr>
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<tr>
<td>88.4</td>
<td>2.88</td>
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<td>83.8</td>
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APPENDIX B
The selection function values for each size class as discussed in Chapter 3 are as follows:

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<td>35 x 48</td>
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</tr>
<tr>
<td>3</td>
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<td>5</td>
<td>100 x 150</td>
<td>1.15</td>
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<table>
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</table>
Breakage Function

The breakage function described in Chapter 3 can be represented as a lower triangular matrix with values as follows:

\[
\begin{align*}
    b(1,1) &= 0.000 \\
    b(2,1) &= 0.268 \\
    b(3,1) &= 0.200 \\
    b(4,1) &= 0.150 \\
    b(5,1) &= 0.112 \\
    b(6,1) &= 0.082 \\
    b(7,1) &= 0.058 \\
    b(8,1) &= 0.041 \\
    b(9,1) &= 0.028 \\
    b(10,1) &= 0.020 \\
    b(11,1) &= 0.013 \\
    b(12,1) &= 0.009 \\
    b(13,1) &= 0.006 \\
    b(14,1) &= 0.004 \\
    b(15,1) &= 0.003 \\
    b(16,1) &= 0.002 \\
    b(17,1) &= 0.002 \\
    b(18,1) &= 0.002 \\
    b(19,1) &= 0.000
\end{align*}
\]

Class 19 = 1 - SUM \( b(1,j) \) to \( b(18,j) \)
CLS: KEY OFF
1010 PRINT "MODELING OF GRINDING KINETICS"
1020 PRINT: PRINT "VIRGINIA POLYTECHNIC INSTITUTE & STATE UNIVERSITY"
1030 PRINT "MICHAEL D. BROWN"
1040 PRINT "APRIL 19, 1985"
1050 PRINT
1060 REM *****************EULER METHOD - GRINDING KINETICS *****************
1070 REM
1080 REM ******************ARRAYS**************
1090 DEFDBL A-Z
1100 DEFINT I, J
1110 REM
1120 DIM S(50, 50)
1130 DIM B(50, 50)
1140 DIM N(50)
1150 DIM L(50)
1160 DIM N(50)
1170 REM
1180 REM *****INITIAL CONDITIONS - ALL ZERO****
1190 REM
1200 FOR I = 1 TO 25: M(I) = 0: NEXT I
1210 T = 0: Z = 0: !TOT = 0
1220 REM
1230 REM ********INPUT DATA***********
1240 REM
1250 INPUT "DO YOU WISH TO INPUT AN INITIAL SIZE DISTRIBUTION (Y/N)"; BS$  
1260 PRINT
1270 IF BS$ = "N" THEN GOTO 1380: ELSE GOTO 1290
1280 PRINT
1290 INPUT "NUMBER OF SIZE CLASSES"; SC
1300 PRINT
1310 FOR Ji = 1 TO SC
1320 PRINT "WEIGHT IN SIZE CLASS "; Ji; " = ";
1330 INPUT "; Ji;" = ";
1340 !TOT = !TOT + M(Ji)
1350 NEXT JI
1360 PRINT
1370 GOTO 1400
1380 INPUT "NUMBER OF SIZE CLASSES";SC
1390 INPUT "INITIAL WEIGHT OF TOP SIZE";MTOT;N(1)=MTOT
1400 INPUT "STEP SIZE";DT
1410 INPUT "GRINDING TIME (MIN)";GT
1420 REM
1430 REM ********READ IN SELECTION FUNCTION*************
1440 REM
1450 FOR I=1 TO SC
1460 FOR J=1 TO SC
1470 READ S(I,J)
1480 NEXT J
1490 NEXT I
1500 REM
1510 REM ***** READ IN BREAKAGE FUNCTION *************
1520 REM
1530 FOR I=1 TO SC
1540 FOR J=1 TO SC
1550 READ B(I,J)
1560 NEXT J
1570 NEXT I
1580 CLS
1590 REM
1600 REM *************** CALCULATIONS ****************
1610 REM
1620 LOCATE 1,1:PRINT " TIME (MIN) = ";:PRINT USING "#####.#";T
1630 LOCATE 2,26:PRINT " CUMULATIVE"
1640 LOCATE 3,26:PRINT " MASS     % RETAINED   % PASSING"
1650 FOR J=1 TO SC
1660 N(J)=N(J)*100/MTOT
1670 LOCATE J+4,1:PRINT " SIZE CLASS ";J;" = ";
1680 PRINT USING "#####$####.####";N(J),N(J),100-N(J)-TOT
1690 TOT=N(J)+TOT
1700 TOTAL=I1(J)+TOTAL
1710 NEXT J
1720 LOCATE 2,1:PRINT " TOTAL = ";
1730 PRINT USING "###########.###";TOTAL
1740 TOT=0
1750 TOTAL=0
1760 REM
1770 REM *****EULER TECHNIQUE & MATRIX MULTIPLICATION*****
1780 REM
1790 FOR J=1 TO SC
1800 FOR I=1 TO SC
1810 Z=Z+B(J,I)*S(I,I)*M(I)
1820 NEXT I
1830 L(J)=N(J)+DT*(-S(J,J)*M(J)+Z)
1840 Z=0
1850 NEXT J
1860 FOR I=1 TO SC
1870 H(I)=L(I)
1880 NEXT I
1890 IF T>GT THEN GOTO 1920
1900 T=T+DT
1910 GOTO 1620
1920 INPUT "CONTINUE GRINDING (Y/N)";A$
1930 IF A$="Y" THEN GOTO 1960:ELSE GOTO 1940
1940 IF A$="N" THEN END:ELSE GOTO 1950
1950 INPUT "ENTER Y OR N";A$:GOTO 1930
1960 INPUT "FOR HOW MUCH LONGER (MIN)";X
1970 CLS
1980 GT=X+GT:GOTO 1890
1990 REM *****DATA ***********SELECTION FUNCTION***************
2000 REM
2010 DATA 2.83,0,0,0,0,0,0,0,0,0,0,0,0,0,0,0,0,0,0
2020 DATA 0,2.26,0,0,0,0,0,0,0,0,0,0,0,0,0,0,0,0,0
2030 DATA 0,0,1.79,0,0,0,0,0,0,0,0,0,0,0,0,0,0,0,0
2040 DATA 0,0,0,1.45,0,0,0,0,0,0,0,0,0,0,0,0,0,0,0
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<th>Data Points</th>
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<td>2160</td>
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<td>2180</td>
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<td>2190</td>
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<tr>
<td>2200</td>
<td>REN **DATA <strong><strong><strong><strong><strong>BREAKAGE FUNCTION</strong></strong></strong></strong></strong></td>
</tr>
<tr>
<td>2210</td>
<td>REN</td>
</tr>
<tr>
<td>2220</td>
<td>DATA 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0</td>
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<td>2230</td>
<td>DATA 0.268, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0</td>
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<td>2240</td>
<td>DATA 0.200, 0.268, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0</td>
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<td>2250</td>
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<td>2260</td>
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<td>2270</td>
<td>DATA 0.082, 0.112, 0.150, 0.200, 0.268, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0</td>
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<td>2280</td>
<td>DATA 0.058, 0.082, 0.112, 0.150, 0.200, 0.268, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0</td>
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<td>2290</td>
<td>DATA 0.041, 0.058, 0.082, 0.112, 0.150, 0.200, 0.268, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0</td>
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<td>2300</td>
<td>DATA 0.028, 0.041, 0.058, 0.082, 0.112, 0.150, 0.200, 0.268, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0</td>
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<tr>
<td>2310</td>
<td>DATA 0.020, 0.028, 0.041, 0.058, 0.082, 0.112, 0.150, 0.200, 0.268, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0</td>
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<td>2320</td>
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<td>2330</td>
<td>DATA 0.009, 0.013, 0.020, 0.028, 0.041, 0.058, 0.082, 0.112, 0.150, 0.200, 0.268, 0, 0, 0, 0, 0, 0, 0, 0, 0</td>
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<td>2340</td>
<td>DATA 0.006, 0.009, 0.013, 0.020, 0.028, 0.041, 0.058, 0.082, 0.112, 0.150, 0.200, 0.268, 0, 0, 0, 0, 0, 0, 0</td>
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<tr>
<td>2350</td>
<td>DATA 0.004, 0.006, 0.009, 0.013, 0.020, 0.028, 0.041, 0.058, 0.082, 0.112, 0.150, 0.200, 0.268, 0, 0, 0, 0, 0</td>
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<td>2360</td>
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2400 DATA .000,.002,.004,.006,.009,.013,.019,.028,.041,.061,.089,.130,.188,.270,.382,.532,.732,1,0
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