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INVESTIGATION OF OPERATING CONDITIONS IN STIRRED BALL MILLING OF COAL

by

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER I

Introduction

1.1.-General

Grinding is an important step in the production of most minerals. It results in the production of particles of the required size and shape, the liberation of valuable material from the gangue for further concentration and an increase in available surface area. Unfortunately, grinding is also the most energy intensive process in mineral processing. It has been estimated that 1% of the total U.S. steel consumption and 2% of the total U.S. energy consumption can be attributed to comminution (National Materials Advisory Board, 1981). These facts coupled with the depletion of higher grade ore deposits and a need to process more finely disseminated ores have produced a drive towards developing more energy efficient methods of fine grinding.

Aside from mineral liberation, however, ultrafine grinding is used in numerous applications, for example to produce fillers in the plastics industry, to produce fillers and extenders in the paint industry, and to produce fillers and coating grade pigments in the paper industry. More recently, the grinding of coal to micron sizes in order to liberate pyrite and other mineral matter has been extensively studied. Once this material is liberated, advanced

separation techniques, such as microbubble flotation, can be employed to produce a super-clean coal containing less than one or two percent ash and less than one percent sulfur (Luttrell et al, 1985).

The production of coal-water and coal-oil slurries also requires the particle size to be reduced to 70 to 80% minus 200 mesh in order to reduce the errosion of pumps, valves and pipes, as well as, to improve the stability of the slurry. Long term stability requires the use of chemical additives or very fine pulverization of the coal to about 15 microns or less (Freedman, Porter and Jamgochian, 1978). Micronized slurries reduce errosion problems, are more manageable and improve combustion efficiencies (Sepulveda, 1981). The single drawback encountered is a reduction of the maximum solids loading to about 50% by weight (Davis, 1986).

Conventional ball milling techniques require long retention times and tremendous energy input in order to produce micronized coal. For this reason several new techniques have been developed over the past years to reduce the energy consumption when grinding to fine sizes. These include vibratory milling, impact milling and stirred ball milling. Of these techniques, stirred ball milling appears to be the most promising, producing a finer product with a lower specific energy input (Sepulveda, 1981).

1.2.-Literature Review

1.2.1.-Particle Size Analysis

The primary objective of particle size analysis is to determine the size and distribution of particles in a given sample. The particle size distribution is the most fundamental characterization of a mass of granular material (King, 1984). However, a true representation of particle size is often difficult to obtain due to irregularities in the shape of the particle. A spherical particle can be accurately described by its diameter, whereas an irregularly shaped particle may require several dimensions to approximate its shape. For this reason, equivalent diameters are frequently used to report the size of an irregularly shaped particle. An equivalent diameter refers to the diameter of a sphere which behaves similarly to an irregularly shaped particle when subjected to a given influence. For example, the Stokes' diameter corresponds to the diameter of a spherical particle with the same settling velocity as an irregularly shaped particle. The effect of shape on the determination of particle size is one of the most difficult problems encountered with any technique of particle measurement.

Numerous methods have been developed over the past decades for accurately determining the particle size distribution of a powder. These include decantation,

centrifugal sedimentation, elutriation, photo- and X-ray sedimentation, laser diffraction and electrical resistance. Each method is applicable over a certain range of particle size. The range depends upon the physical parameter which is used to measure particle size. For instance, sedimentation techniques are influenced by the density of the particle and the suspending fluid. Photo, x-ray and laser techniques are limited to the sensitivity of the detector.

Most techniques also have a lower detection limit. This limit is usually a function of the physical parameter which is being used to size the material. For instance, centrifugal processes, such as the cyclosizer, are limited to the maximum fluid velocity which can be generated. Sieving has no limit in theory, however construction of screens smaller than 5 microns is extremely impractical. Optical microscopy is also limited to about 1 micron due to the resolution of the microscope.

Electrical sensing techniques also have lower detection limits. The limits are usually a function of the signal to noise ratio generated by the device. For instance, the Coulter Counter can easily recognize a 40 micron particle when using a 100 micron orifice. However, the resistance generated by a 0.1 micron particle while passing through the same orifice may be indistinguishable from the background noise.

The current attention focused on grinding to ultrafine sizes makes it necessary to have an understanding of the various fine sizing techniques, including the capabilities and limits of these techniques. The following is a review of the various techniques which have been used for determining particle size distributions of fine powders.

Sieving

Laboratory sieving is one of the oldest methods of particle size determination. The use of punched plate sieves is recorded in early Egyptian drawings. Agricola had illustrated woven wire sieves in 1556. Sieving is carried out by placing a known quantity of material through a series of screens having successively smaller apertures. The material retained on each screen is weighed to determine the percent of the original mass in each size interval. Although used extensively, several problems can be encountered when applying sieving techniques to irregularly shaped particles. For instance, the presence of near size material can cause blinding of the screen and result in a reduction of the available screen surface. Additional considerations affecting screen performance are wire diameter, aperture shape, screen motion, depth of particle bed and moisture.

Sieving can be performed either wet or dry. Wet screening is considered to be more accurate due to a tendency to remove fines which adhere to the coarser particles. The

smallest particle size which can be effectively measured using conventional laboratory sieves is 25 microns. This lower limit can be extended to 5 microns with the use of micromesh sieves.

Sedimentation

Sedimentation techniques are based on the principle of Stokes' settling. This method is quick and cheap, but can be far too time consuming for ultrafine particles. One of the simplest devices available is the Andreason pipette. This apparatus consists of a calibrated, graduated cylinder and a pipette connected to a 10 ml reservoir. A dilute suspension is placed into the cylinder and allowed to settle. Samples are periodically withdrawn through the pipette. The particle size corresponding to the appropriate time and settling distance is calculated using Stokes' equation. A practical lower limit for the Andreason pipette is about 5 microns. Below this, the effect of Brownian motion becomes highly significant compared with the systematic motion due to gravity settling (Chung and Hogg, 1985).

Elutriation, also governed by Stokes' equation, utilizes the upward current of a fluid for sizing particles. Particles which have settling velocities less than that of the fluid velocity will report to the overflow, while particles with higher velocities sink to the underflow. The Blythe elutriator operates according to this principal. It

consists of several beakers having gradually larger diameters. The increase in diameter provides a corresponding decrease in the fluid velocity. As the feed material passes through the system, different size fractions are captured in each beaker. This process becomes impractical for particle sizes below 10 microns.

Time constraints involved with sedimentation techniques can be overcome by taking advantage of centrifugal forces generated by a rotating fluid. The Warman cyclosizer is one of the best examples of this type of particle sizer. It uses a series of inverted cyclones having a successive decrease in inlet area and vortex outlet diameter. This results in an increase in fluid velocity which creates a larger centrifugal force and produces a finer particle separation in each cyclone. A typical range of application for material with a specific gravity of 2.7 is 8 to 50 microns.

Several of the most advanced particle size analyzers utilize the principle of sedimentation in combination with light extinction and x-ray detection. The light extinction process is used to determine the concentration of sedimenting particles. It directly plots the projected area of a particle versus Stokes' equivalent spherical diameter. Results are obtained in minutes with better than $\pm$ 2% reproducibility. The measureable particle size range is 0.1 to 100 microns. This procedure is applicable to organics and low density powders, such as coal (Micromeritics, 1980).

The second type of advanced sedimentation technique utilizes a finely collimated beam of low energy x-rays to measure the concentration of particles. It measures the sedimentation rates, according to Stokes' law, and automatically plots these data as cumulative mass percent versus equivalent spherical diameter. Analysis can be performed on any material which is capable of absorbing x-rays. The effective particle size range for this type of particle size analyzer is also 0.1 to 100 microns.

Three general criteria must be satisfied in order to analyze a powder with the x-ray sedimentation techniques: 1) the particles must be more dense than the liquid in which they are dispersed so they will settle, 2) the particles must absorb more light or x-rays than the liquid in which they are dispersed so that there is enough contrast to detect them, and 3) the particles should disperse in the sedimentation liquid (Micromeritics, 1980).

Centrifugal particle size analyzers have been developed to increase the measureable size range of sedimentation techniques. These analyzers are capable of detecting particles as fine as 0.01 microns. Centrifugal analyzers use a combination of high-speed centrifugation and photo- or x-ray sedimentation to determine particle size. They have been used for measurement of paint pigments, carbon black, metallic powders, ceramics and various other agricultural,

chemical and medical materials (Horiba Instruments, 1983).

Microscopy

Microscopy is the only method in which individual particles are observed and measured. It permits the examination of shape and composition with a sensitivity far greater than other techniques. Optical microscopy is capable of accurately determining particle sizes from 0.8 to 150 microns. This range can be extended down to 0.001 microns with the addition of an electron microscope.

Difficulties encountered in microscopy are (1) preparation of a uniformly dispersed sample which is representative of the powder and (2) counting enough particles to be statistically representative of the bulk material. The latter problem can be overcome by the use of automated image analysis. This procedure removes the tedious operator dependency of particle sizing with microscopy.

Image analysis uses a television camera to scan a particle bed and display the image on a console. Electrical information describing the sample is passed to a detector. Measureable parameters include count, size distribution by area or statistical diameter, cord size distribution, etc. (Allen, 1981).

Light Detection

Perhaps the most widely used and versatile particle size analyzers are based on the principle of light diffraction.

These techniques have been applied to a variety of materials including cement, ceramics, minerals, coal, pharmaceuticals, pigments and toners, as well as, various chemical precipitates and catalysts. The principle of operation is based on the phenomenon of low-angle forward-scattering of light. Quantities of light scatter through precise angles as a function of particle size. The scattered light is focused onto a photocell detector. The detector output can be directly correlated to particle size. By combining two particle size analyzers a total size range of 0.12 to 1000 microns can be measured (Leeds & Northrup, 1984; Malvern,1985).

Several new particle size analyzers have been developed which extend the range of measurement down to 0.001 microns. These analyzers detect the amount of light which passes through a suspension of colloid particles. The amount of light is a function of the varying patterns generated as the particles undergo random thermal or Brownian movement. This information can be related to a number of parameters including molecular weight, particle size and diffusion coefficients (Malvern, 1985; Hiac/Royco, 1984; Coulter Electronics, 1983).

Light detection analyzers have also been developed which are capable of sizing individual particles over a range of 0.25 to 9000 microns. This is achieved by passing single

particles through a detection zone which is perpendicular to a collimated light source. Particles interrupting this beam cause a reduction in the amount of light reaching a photodetector. The resulting voltage pulse is directly proportional to the maximum projected area of each individual particle (Hiac/Royco, 1981).

Electrical Sensing

The last type of particle sizing technique to be discussed is the electrical sensing zone method or Coulter principle. In the Coulter principle, particles are suspended in an electolytic solution having a higher conductivity than the material to be measured. The particles are allowed to pass through a small orifice which has electrodes located on either side. As particles pass through the orifice an equal volume of fluid is displaced. This results in a change in the electrical current which is proportional to the volume of the particle (Coulter Electronics, 1983). The technique was originally developed to count blood cells (Coulter, 1956; Morgan, 1957).

It was quickly pointed out, however, that this principle could be applied to the measurement of cell volume distributions as well as number counting (Kubitschek, 1958; 1960). This led to the development of modified machines which are capable of measuring the number and size of particles. A number of different machines are available,

which are capable of measuring particle diameters in the size range of 0.3 to almost 1000 microns (Coulter, 1983; Particle Data, Inc., 1980).

1.2.2.-Fine Grinding Techniques

Several techniques are currently available for producing ultrafine particles. These include tumbling ball mills, vibratory mills, fluid energy mills, the Szego mill and stirred ball mills. Tumbling ball mills are most commonly used for achieving products in the size range of 1/4 inch to a few microns. However, long retention times and excessively high energy inputs are required in order to obtain products below 2 microns. Throughputs in the range of several tons per hour have been produced by vibratory milling, but its applicability to grinding below 10-20 microns has not been demonstrated (Vogeno, 1969).

Fluid energy milling was first developed for producing particles less than 100 microns in diameter and having a high degree of purity (Andrews, 1936). Its main initial use was for milling sulphur, although it has since been used for dyes, pigments, cosmetics, antibiotics and other pharmaceutical products (Skelton, Khayyat and Temple, 1980). The typical fluid energy mill design incorporates a horizontal, cylindrical grinding chamber having fluid jets which enter tangentially. Feed material enters by way of a venturi arrangement. Comminution is achieved by multiple particle/particle interaction (Dobson and Rothwell, 1970). This process has the distinct advantage of having removed the use of media in the mill. The problem of contamination of

the material to be ground is therefore eliminated. One major disadvantage of this process is the low capacity.

The Szego mill consists of a cylindrical vessel with a series of helically grooved rollers connected to a central shaft with flexible rope supports. The feed material is repeatedly crushed between the rotors and the cylinder (Trass, 1980). The mill has several design variables including number, size and shape of rotors. It has a high capacity, modest power consumption and can be operated either wet or dry.

Compared to the above mentioned techniques, stirred ball milling is capable of producing a finer product with a smaller energy input. It has been shown that, for energy inputs exceeding 200 kwh/t, the stirred ball mill continues to grind into the submicron range when other mills have almost stopped (Sepulveda, 1981). In addition, the throughput of the stirred ball mill is relatively high as compared to other devices. This is due to exceptionally high power input per unit volume of the grinding chamber.

1.2.3.-The Development of Stirred Ball Milling

Past studies have indicated that stirred ball milling is capable of producing micronized material. This technology has been used quite successfully in the ceramic and paint industries. A stirred ball mill consists of a water cooled grinding chamber, charged with media and agitated with a central impeller. The material to be ground is usually in a slurry form and fills the void volume of the media. Impact and attrition grinding is thought to occur by collision of the media with itself and the mill wall.

One of the earliest types of stirred ball mill was the shot mill. This mill utilized 0.5-2.0 mm media, set into motion by rotating discs mounted on a central shaft. The United States Bureau of Mines (USBM) followed in the late 1930's with the development of the Szegvari attritor system (Szegvari, 1956). Originally developed for the removal of surface contaminents prior to flotation, this technique proved to be an efficient grinding device (Norman and Ralston, 1939).

The USBM attritor consisted of a cage-like rotor-stator arrangement located inside of a water cooled vessel. The Szegvari attritor, simpler in design, was eventually developed from the original USBM attritor. This system closely resembles the mill used in this study. It consists of a branched central shaft for fluidizing the media and a water

cooled grinding chamber.

The tower mill, developed in Japan, is similar to the Szegvari mill. This design uses a screw shaped impeller and is lined with abrasion-proof rubber (Koppers Company, Inc.). This mill has been used for both wet and dry grinding.

More recently, attrition mills developed and marketed by Draiswerk and Union Process dominate in the area of industrial fine grinding. The Drais mill is similar to the Szegvari mill in construction, with the addition of pins mounted perpendicularly to the inside of the mill wall. The mill is equipped with a self-contained feed pump for operating on a continous basis. The Drais mill has been used extensively for the production of coal water slurries (Draiswerke, Inc., 1983).

The attritor type mills use various types of media including steel and ceramic balls as well as sand and gravel. The media usually varies in size from 1/2 mm to 1/2 inch.

1.2.4.-Past Studies on Stirred Ball Milling

A great deal of literature concerning the stirred ball mill has been published by the USBM. The first study involving the use of the USBM attritor as a grinding device was reported in 1960 (Feld, McVay, Gilmore and Clemmons, 1960). This study demonstrated that coarse kaolin (13% passing 2 microns) could be successfully ground to a product which is 88% passing 2 microns. The work was carried out in 5- and 10-inch batch mills.

Later work defined the influence of operating parameters on a 5-inch USBM attritor (Stanczyk and Feld, 1968). It was concluded that several parameters were important in governing comminution efficiency and energy consumption requirements. These were type, size and shape of grinding media, grinding media to clay weight ratio, rotor speed, pulp density, degree of pulp dispersion and arrangement of rotor and stator bars. Variables having the least influence on efficiency were pulp temperature, rotor cage design, rotor clearance, rotor-stator bar interval and chamber liners. Grinding media was found to have the greatest affect on abrasion of mill parts. It was determined that spherical shaped media is most efficient in consumption, mill wear and grinding efficiency. This study was further extended to involve industrial minerals such as mica, pyrophyllite, talc, marble, barite and flourite (Stanczyk and Feld, 1974).

A study involving large scale continuous grinding of kaolin

(d_{50}=15 microns) was conducted in 1973 (Davis, Collins and Feld, 1973). A 20-inch USBM attritor was used to perform single and multi-stage open circuit grinding, as well as, closed circuit grinding with a 6-inch solid bowl centrifuge. It was concluded that multi-stage grinding was more efficient than single-stage grinding for equivalent feed rates.

Early attempts at modeling a stirred ball mill were made while studying attrition milling of ceramic oxides in a 5-inch USBM attritor (Stanley, Sadler, Brooks and Schwartz, 1974). The effects of operating variables such as rotor speed, grinding media and ultrafine grinding performance between different mills were studied. It was determined that the milling rate constant, for a 45 x 50 mesh dolomite, is proportional to the cube of the rotational speed up to speeds of 1600 RPM. A linear relationship was found to exist between the mill power requirement and the cube of the impeller speed, which increases with slurry density and is independent of viscosity in the turbulent range.

A later study using the USBM attritor found that the breakage rate function is first order and that it increases linearly with power input to the mill (Sadler, Stanley and Brooks, 1975). More recently, a summary of the different solid materials which have been studied using the USBM attritor has been presented (Davis, Hansen and Sullivan, 1980).

Few results are available on the operating characteristics of the Szegvari attritor, other than what is obtainable through the patents. Three examples of typical applications were presented in the first patent (Szegvari, 1956). The first study reports the grinding of paint pigments from 60 microns to 4 microns over a period of 2 to 4 hours. A 130 gallon attritor was used with 900 pounds of 5/16-inch rounded flint media. The material was ground at 50% solids by weight using a stirring speed of 80 RPM.

The second test involved grinding a zinc oxide slurry in a 75 gallon attritor with 600 pounds of 1/2-inch flint pebbles. The material was reduced to less than 0.5 microns in less than 5 hours. It was claimed that a conventional pebble mill would require 90 hours to achieve the same size.

In the last test a laboratory size attritor, with 3/16-inch stainless steel balls, was used to grind a 50% titanium dioxide paste to 6.25 microns. A grinding time of 20 to 30 minutes was necessary as compared to 24 to 48 hours in a conventional pebble mill.

Since the development of the Szegvari attritor, many improvements have been suggested by its inventor, Andrew Szegvari. These include a multiple agitator design, tear drop shaped impellers and a continuous grinding process. The use of multiple agitators was suggested in order to create zones of increased turbulence, which would result in a higher degree of grinding activity in the mill (Szegvari,

1961). Tear drop shaped impellers were developed to increase the acceleration of the media toward the rear of the impeller blade, thus resulting in better agitation of the media (Szegvari, 1964).

A continuous version of the Szegvari attritor was developed for coarse and fine grinding of feed sizes ranging from 45 microns to 1/4 inch and product sizes ranging from 0.5 to 5 microns (Szegvari, 1964). Contrary to the present findings, the author reported that feed and product particle size is not critical to the performance of the mill.

Union Process, founded by Andrew Szegvari in 1946, currently supplies a number of stirred ball mills, both batch and continuous, ranging from small scale laboratory size attritors to full scale industrial mills. The most recent development is a circulation attritor in which the material to be ground is recycled through the media. It is claimed that the fines pass quickly through the grinding zone while the coarse material is retained for further grinding. Slurry is repeatedly passed through the grinding chamber until the desired particle size is reached, resulting in a narrow particle size distribution (Union Process, 1984).

A detailed study of stirred ball milling has been done at the University of Utah (Sepulveda, 1981). Three Union Process attritors were used to evaluate the mill performance in grinding chalcopyrite, limestone, quartz, pyrite,

sphalerite, coal, a synthetic cupric oxide and nickel matte.

It was found that the grinding rate of the stirred ball mill has no dependence on the operating condition for a fixed specific energy input, although slight advantages were seen with a reduction of speed and ball size. A change from 30% to 70% solids by weight had no effect on the efficiency of the mill. It was further shown that a form of Charles' equation was able to predict the median particle diameter for different energy inputs to the mill with an accuracy of $\pm$ 5%. An increase in viscosity resulted in a corresponding increase in the slope and intercept of the Charles' plot.

Sepulveda suggested that different grinding mechanisms operate in the mill as grinding proceeds. It was proposed that the kinetics, which are initially first order, become non-linear due to a build-up of fines in the mill. This build-up results in an increase in the slurry viscosity which impedes grinding.

Finally, several mathematical models were developed by Sepulveda which predict the product size distribution obtained from a stirred ball mill. Models were developed based on three different types of breakage mechanisms which are thought to occur in the mill. These include models for the combined effect of attrition and impact breakage, attrition breakage exclusively and fragmentation breakage. This study indicated that attrition did not play an important role in the description of stirred ball mill grinding.

1.3.-Objectives

The objectives of this investigation will be 1) to optimize the operating conditions associated with stirred ball milling of coal, and 2) to develop a reliable method of analyzing the product size distributions. To meet these objectives, the investigation will be conducted in three different stages.

The first stage will be concerned with the development of a procedure to more accurately determine the product particle size distribution using an Elzone 80XY particle size analyzer. Based on this procedure a computer program will be developed in order to expedite the size analysis.

The second phase will involve determining the effect which the ball and particle diameter has on the kinetics of the grinding process in the stirred ball mill. To meet this goal, feed disappearance plots will be generated for a wide range of monosize feeds and ball diameters. An attempt will be made to develop a relationship between the ball and particle diameter based on the energy specific breakage rates determined from these tests.

In the final stage, other fundamental operating parameters involved with stirred ball milling will be examined. This will include determining the effect which stirring speed, percent solids and grinding aids has on the mill energy consumption.

CHAPTER II

A Procedure For Blending and Mass Balancing of Fine Particle Size Distributions Using the Elzone Particle Size Analyzer

2.1.-Introduction

In recent years, a great deal of interest and effort has been devoted to the development of new comminution techniques for producing micron-sized particles. Devices such as stirred ball mills, vibratory mills and ultrasonic mills have been employed for this purpose with mixed success (Sepulveda, 1981; Grimshaw and Albus, 1983; Herbst and Sepulveda, 1979). Typically the results from tests conducted in these various grinding devices are reported on the basis of product size distribution and energy consumption. In theory, this should make it possible to compare any of the grinding devices and determine which is the most efficient. Unfortunately, many of the size distributions being presented are suspect because they have not been properly mass balanced.

It is well known that many particle size analyzers have a lower detection limit below which particles are simply ignored by the analyzer. Thus, the mass of the missed particles is not included when the instrument computes the size distribution. Depending on the fineness of the sample and the value of the lower detection limit, failure to

include this missed material can result in a difference of several microns in the mean particle size of the distribution. Although this difference may not seem significant, it can be very important when dealing with the production of micron-sized particles. For example, it has been shown that for the stirred ball mill grinding of Elkhorn seam coal, a change in the mean product size from 5 to 4 microns may require as much as 25 additional kwh/ton of energy input (Mankosa, Adel and Yoon, 1986). This clearly illustrates the importance of having mass balanced size distributions when comparing various fine grinding techniques on an equal basis.

The electrical sensing zone method (Coulter principle) has long been recognized as a rapid and reproducible method for determining accurate particle size distributions (Lines, 1973). The Coulter principle determines the number and size of particles which are suspended in an electrolyte. This is accomplished by forcing the individual particles through a small orifice located between two electrodes. The change in resistance as a particle passes through the orifice is directly proportional to the volume of the particle. The size distribution for the suspended particles is derived from these pulses (Allen, 1981). This method has been shown to agree quite well with other techniques, such as sieves and cyclosizers (Napier-Munn, 1985).

As with most size analysis techniques, the Coulter principle also suffers from its inability to recognize fine particles. The practical lower limit for this technique is about 0.5 microns. Particles in a given sample which are smaller than this size will pass through the orifice tube undetected. This will bias the resulting distribution towards the larger particle sizes.

The purpose of this chapter is to present a procedure and computer program developed for mass balancing and blending size distribution data obtained with an Elzone 80XY particle size analyzer. Emphasis has been placed on developing a simple, standardized procedure which is reproducible and compares favorably with other size analysis techniques.

2.2.-Theory

2.2.1.-Data Blending

In the electrical sensing zone sizing technique, each orifice tube is valid over a range of 0.5% to 40% of the orifice diameter. Particles larger than the upper limit may block the orifice and particles smaller than the lower limit may not be detected by the analyzer. As a result, a series of orifice tubes may be required to cover the entire size range for a given sample.

Each orifice tube in the series is used to analyze a portion of the original sample from which all particles larger than the upper limit for a given tube have been removed. Only the largest orifice tube in the series is exposed to the entire sample. The remaining tubes analyze size fractions which may be prepared by a variety of techniques including sieving, centrifuging and sedimentation. Furthermore, the orifice tubes in the series are usually selected so that a slight overlap in the size analysis data will exist between two sequential tubes.

Now consider an ideal situation. If the original sample presented to the largest orifice tube could be cut such that only those particles larger than the upper limit of the next orifice tube were removed, than the number of particles counted in the overlap region for each tube would be the same. This would then make reconstruction of the complete

size distribution a simple matter. Unfortunately, laboratory separation processes do not provide a perfect cut. Furthermore, it is virtually impossible to present the same concentration of particles to each orifice tube. As a result, the particle counts for two tubes in the overlap region are not identical. It is possible, however, to make them identical by normalizing one to the other. This is the basis for the blending of size distribution data.

An example of the data blending procedure is illustrated in Table 2.1. Here, particle count data from a 76 micron orifice tube are being blended with particle count data from a 380 micron orifice tube by using a normalization factor determined from the overlap region. The normalization factor is calculated for a particular size class in the overlap region by dividing the number of counts for the larger orifice by the number of counts for the smaller orifice. Care must be taken, however, in selecting the size class to be used for normalizing the data. As mentioned earlier, sieving and centrifuging are not perfect separation techniques, and the size classes immediately surrounding the cut size will reflect a certain amount of error due to misplaced material. For this reason, the normalization factor is calculated for a size class which is far enough below the cut size to have an unbiased representation in the smaller orifice tube, but large enough to remain above the lower detection limit of the

larger orifice tube.

Finally, the combined size distribution is determined by using the data from the larger orifice tube down to the size class selected for the normalization, and then using the data from the smaller orifice tube multiplied by the normalization factor. This procedure is illustrated in Table 2.1. The process is then repeated for each orifice tube used in the analysis.

Table 2.1

Data for Blending of Two Orifice Tubes

Size Class (microns)	Count Data		
	380 um orifice	76 um orifice	Blended
140 x 150	5		5
130 x 140	12		12
120 x 130	11		11
110 x 120	30		30
100 x 110	47		47
90 x 100	75		75
80 x 90	125		125
70 x 80	198		198
60 x 70	331		331
50 x 60	603	7	603
40 x 50	1226	11	1226
30 x 40	2314	14	2314
20 x 30	5616	48	5616
15 x 20	9835	84	9828
10 x 15	28051	240	28080
9 x 10	12894	113	13221
8 x 9		105	12285
7 x 8		202	23634
6 x 7		334	39078
5 x 6		532	62244
4 x 5		917	107289

$$\text{Conversion Factor} = \frac{\text{\# counts 20 x 30 size class (380)}}{\text{\# counts 20 x 30 size class (76)}}$$

2.2.2.-Mass Balancing

Having blended all of the data, a certain amount of material will still exist which has passed the smallest measureable size. This material must be accounted for in order to determine an accurate representation of the particle size distribution. The distribution can then be corrected for the mass fraction of material missed.

The mass of material missed by the largest orifice is simply the difference between the mass presented to the orifice for analysis, M_p, and the mass counted by the analyser, M_C. The mass counted by the analyzer is determined by

$$M_C = \frac{\pi}{6} \rho \sum_{i=1}^{N} d_i n_i \qquad (2.1)$$

where d_i = the equivalent spherical diameter for each size class,

n_i = the number of particles in each size class,

ρ = the specific gravity of the material being sized, and

N = the total number of size classes.

The mass presented to the analyzer is calculated from

$$M_p = CQt \qquad (2.2)$$

where C = the concentration of solids in the electrolyte being analyzed,

Q = the volumetric flow rate through the orifice, and

t = the analysis time.

In Equation (2.2), Q and t are known parameters which are fixed by the equipment and the operator. The concentration of solids in the electrolyte, C, however, must be determined experimentally.

Once M_P and M_C have been determined, the mass fraction of material in any size class, given by

$$f_i = \frac{\pi}{6} d_i n_i, \tag{2.3}$$

can be corrected for the missed material by using the following relationship:

$$f_i(c) = \frac{f_i M_C}{M_P} \tag{2.4}$$

where $f_i(c)$ is the corrected mass fraction in a given size class.

2.3.-Experimental

2.3.1.-General Sizing and Analysis Procedure

A general procedure for sizing, blending and mass balancing an entire size distribution has been developed for the Elzone particle size analyzer manufactured by Particle Data, Inc. The procedure described here, however, should be valid for any electrical sensing zone technique.

All samples are initially prepared as a suspension in water to facilitate handling and allow for more accurate sampling in subsequent steps. The largest particle size in the distribution is then determined by finding the sieve size for which 100% of the sample will pass. The first orifice used in the analysis is chosen so that this sieve size corresponds to 40% of its diameter. A sub-sample is removed using a pipette and diluted with doubly distilled water. This sub-sample, S_1 in Figure 2.1, is dispersed with the proper reagents and placed on a magnetic stirrer until needed for analysis.

The samples for the remaining orifice tubes are then screened at the appropriate sieve size. For example, a 100 mesh sieve is used for preparing samples for the 380 micron orifice tube. This provides a top size of 150 microns, which closely corresponds to the 152 micron upper limit for this orifice. The undersized material is sampled and the oversized material is discarded. The series of orifice tubes

is chosen so that an overlap range occurs which meets the previously mentioned requirements for blending.

For the extremely small aperture orifice tubes, screening becomes impractical. Therefore a centrifuge is employed to make the appropriate separation for these samples. The cut size is calculated using Stokes' law, based on the experimentally determined gravitational force generated in the centrifuge. In this case, the decant is removed and collected as sample. The settled solids are repeatedly dispersed and centrifuged until all fines are removed.

Following the sample preparation procedure, a size analysis is carried out on each of the samples. Before conducting the size analysis, however, two sub-samples are removed from S_1, as shown in Figure 2.1. The solid/liquid ratio in S_1, is determined from sub-sample S_r, while the analysis sample, S_a, is added to a known weight of electrolyte, M_e. This final sample, S_f, is then analyzed with the appropriate orifice tube and the resulting size and count data are stored on diskette.

Following the analysis of all samples, the size and count data are retrieved from the diskette and entered into an interactive computer program which blends the results and mass balances the distribution based on the series of weights recorded for the first analysis. The corrected particle size distribution is then stored and either displayed on screen or

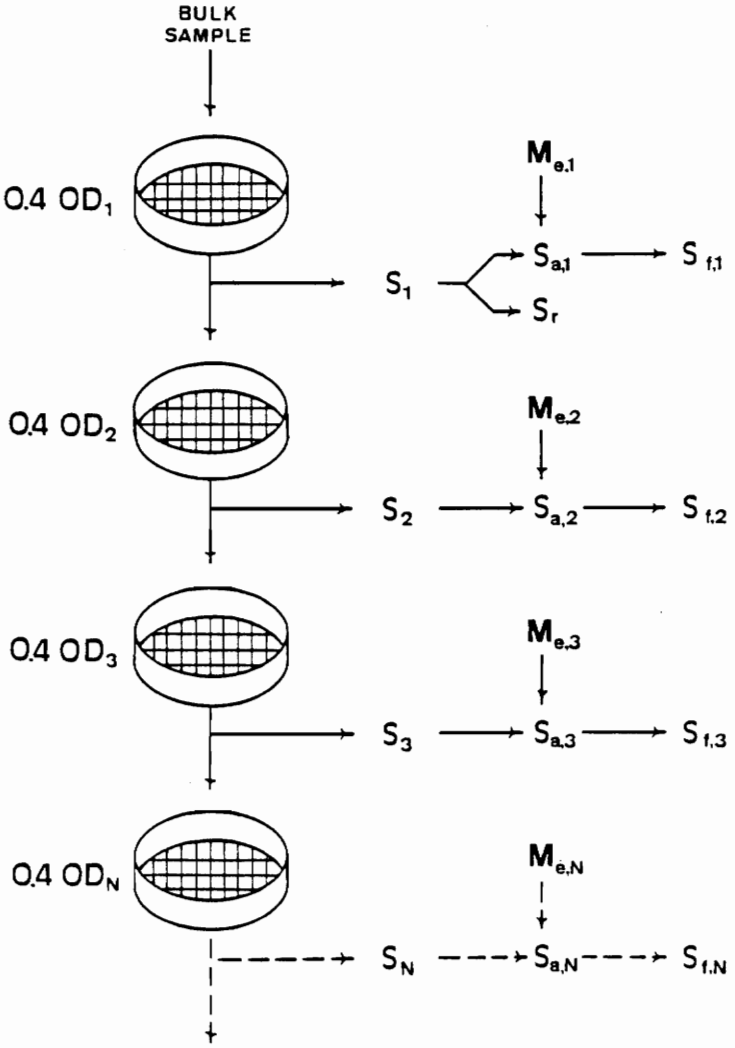

Figure 2.1. Flow diagram of sample preparation procedure for particle size analysis.

printed as a tabular listing or a plot of cumulative percent finer versus particle size.

2.3.2.-Sizing a Coal Sample

To demonstrate the blending and mass balancing technique, a coal sample was prepared, sized, blended and mass balanced according to the procedure described above. The coal was ground in a hammer mill to produce a sample which was approximately 100% passing 100 mesh.

It was determined that three orifice tubes, with diameters of 380, 76 and 24 microns would be necessary to cover the entire size range of the sample, as well as to provide the necessary overlap region for blending. The sample for the 380 micron orifice tube was screened at 100 mesh (150 microns), thus assuring a top size below the 40% upper limit. If any +100 mesh material remained, a larger initial orifice tube was used. Using a pipette, a sample of the -100 mesh material was removed and diluted with doubly distilled water. The remainder of the sample was then screened at 400 mesh (37 microns) to prepare a sample for the 76 micron orifice tube. A sample was again removed and diluted and the +400 mesh material was discarded.

The last stage of the preparation was to obtain a sample suitable for the 24 micron orifice. A centrifuge was employed for this task in order to make a size separation at approximately 8 microns. The decant was repeatedly removed

and collected as sample for the 24 micron orifice. All samples were kept continuously mixed until the sizing was completed.

As described earlier, two sub-samples were removed from the sample prepared for the 380 micron orifice tube and the proper weights recorded. Electrolyte was added to the analysis sample which was then analyzed using the 380 micron orifice tube. The resulting size and count data were stored on an IBM PC. The two remaining samples were then analyzed with their respective orifice tubes and the size and count data also stored.

Having completed the analysis for each orifice, the size and count data, along with the previously recorded weights, were entered into a program called BLENDBAL which was developed for this work to blend and mass balance particle size distributions. The BLENDBAL program is part of a software package developed at Virginia Tech to provide an interface between an IBM-PC and the Elzone 80XY particle size analyzer. This package allows all sizing operations, data logging and data manipulation to be controlled from the IBM-PC. An example of the input display for BLENDBAL is given in Figure 2.2.

Enter Size of First Orifice Tube in Analysis	?
Enter Analysis Time (seconds)	?
Enter Beaker Weight (grams)	?
Enter Beaker Plus Sample Weight (grams)	?
Enter Beaker Plus Solid Weight (grams)	?
Enter weight of Electrolyte Used (grams)	?
Enter Specific Gravity of Solid	?
Enter Specific Gravity of Liquid	?
Enter Weight of Electrolyte Used for Specific Gravity Determination (grams)	?
Enter Weight of Analysis Sample (grams)	?
Are These Values Correct (Y/N)	?

Figure 2.2 Sample Display of Input Data Required for Mass Balancing a Particle Size Distribution.

2.4.-Results

2.4.1.-Effect of Blending and Mass Balancing

Figure 2.3 shows the effect of blending and mass balancing on the size distribution of a finely ground coal sample prepared and analyzed as discussed in section 2.3. The drop in the tail of the distribution sized with the 380 micron orifice tube is a clear indication that fine material has been missed in the analysis. As more material is included in the analysis by blending the data from the 76 micron orifice and the 24 micron orifice, the drop in the tail becomes less pronounced; however, material is still missed. Finally, when the fraction missed by the 24 micron orifice tube is included in the analysis, the tail is raised further and the distribution is mass balanced.

2.4.2.-Reproducibility

One of the problems often cited in using mass balancing and blending procedures is the difficulty in obtaining reproducible size distributions (Sepulveda, 1983). This is often the case when the amount of sample slurry, electrolyte, etc. is measured on the basis of volume. In this procedure, however, all measurements were made on the basis of mass which is usually more accurate.

To demonstrate the reproducibility of this technique, two separate size analyses were conducted on an Elkhorn seam

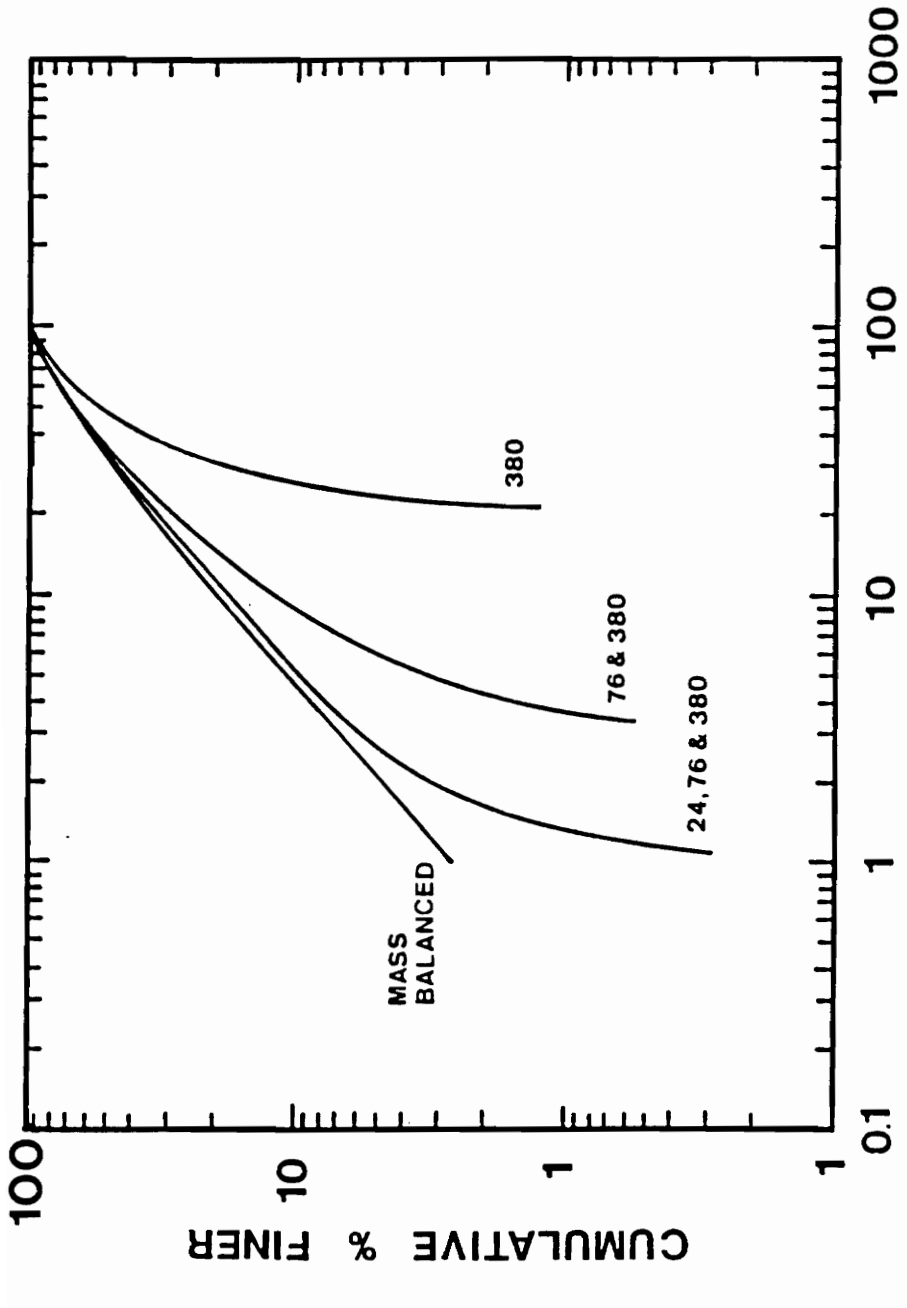

Figure 2.3. Effect of blending and mass balancing on the particle size distribution.

coal hammer milled to produce a -400 mesh product. Both analyses were performed using the 76 and 24 micron orifice tubes, and the resulting size distributions were blended and mass balanced. As shown in Figure 2.4, the two distributions are virtually identical. The slight deviation at very fine sizes is well within normal experimental error. The reproducibility of this procedure has been checked quite frequently with similar results.

2.4.3.-Sensitivity Analysis

The sensitivity of the blending and mass balancing procedure to small changes in the particle size distribution is illustrated in Figure 2.5. A slight variation in the size distribution was achieved by attrition grinding a coal sample for periods of 15, 30 and 45 minutes. Although the mean particle size of the distrbutions changes by no more than a few microns, the procedure is clearly capable of showing the decrease in product size distribution as grinding time increases.

2.4.4.-Comparison with Other Sizing Techniques

In order to compare the results obtained using the Elzone to other particle size analysis methods, a -20 micron coal sample was analyzed with a Micromeritics Sedigraph L particle size analyzer. This process uses photosedimentation to determine the particle size distribution. The results

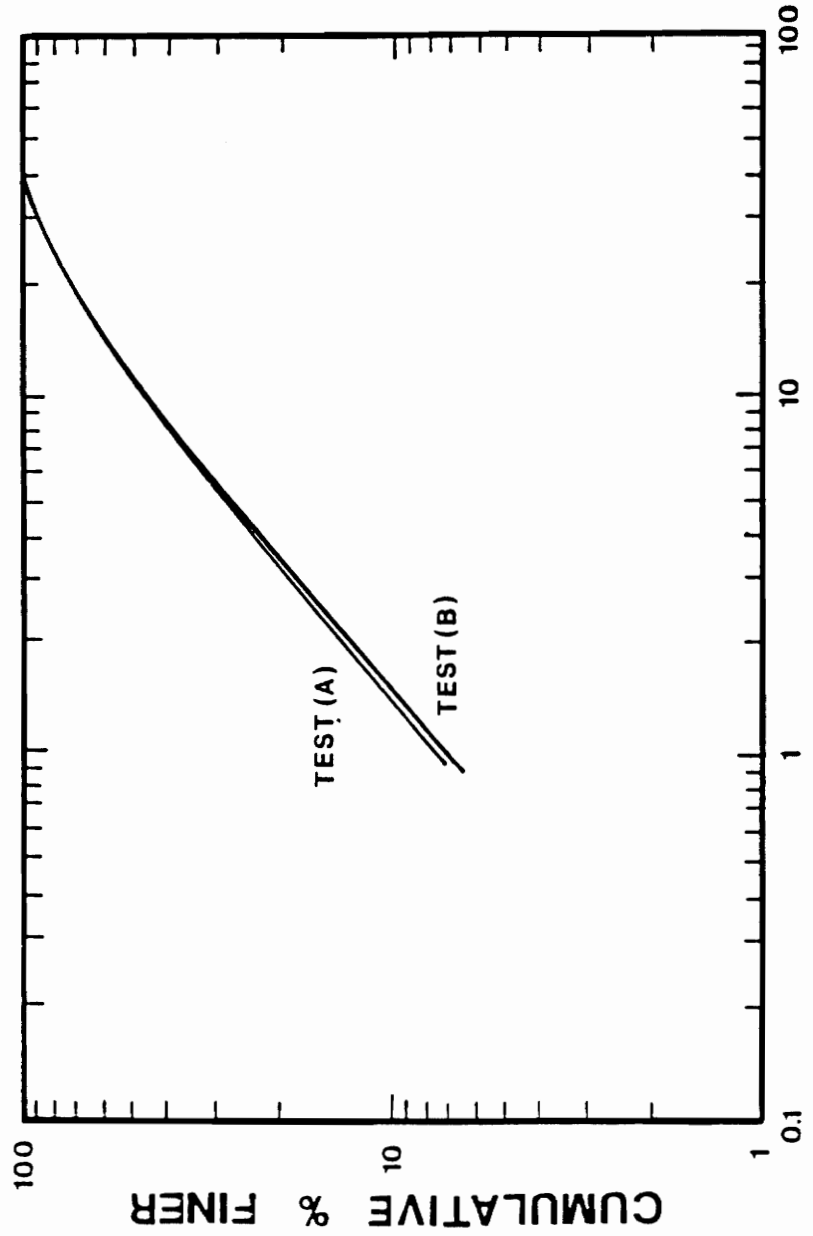

Figure 2.4. Demonstration of Reproducibility using the 380, 76 and 24 micron orifice tubes and mass balancing.

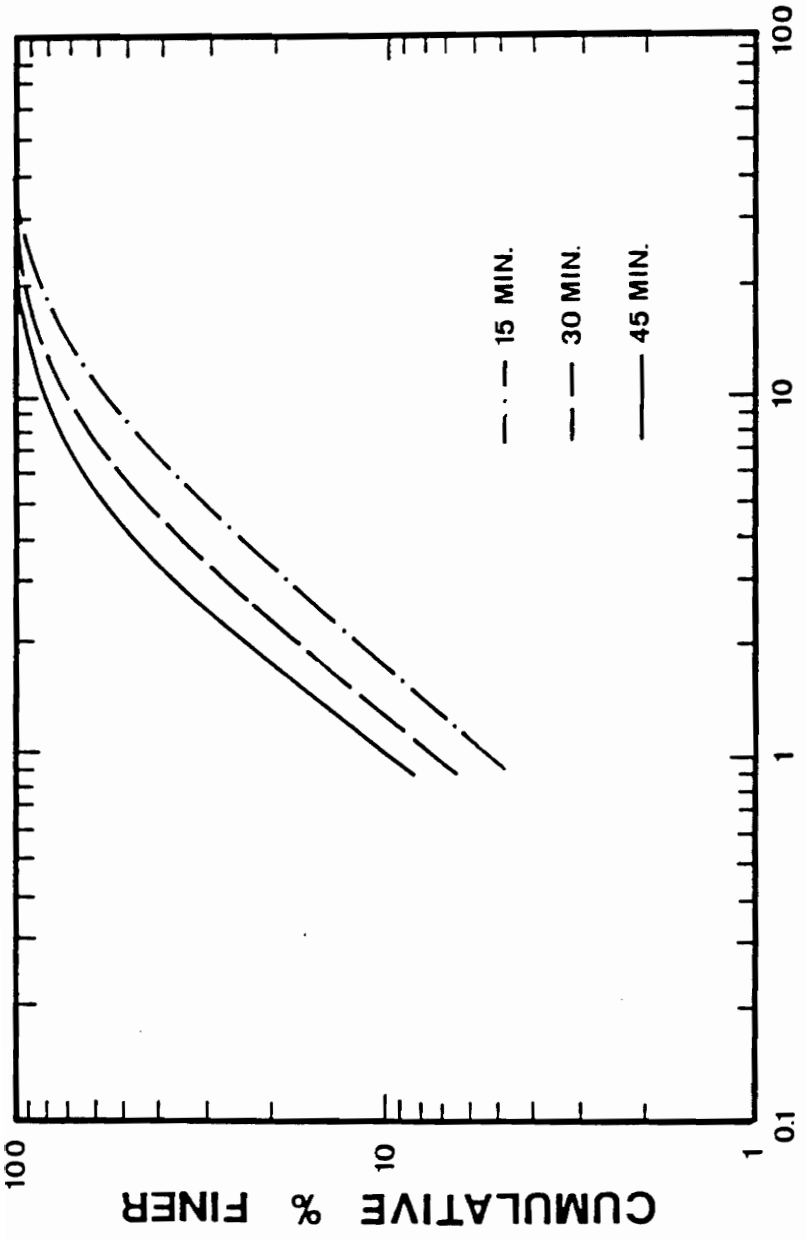

Figure 2.5. Particle size distributions for an Elkhorn seam coal sample attrition ground for 15, 30, and 45 minutes.

shown in Figure 2.6 compare favorably with the Sedigraph L; however, it can be seen from the drop in the tail of the distribution that this technique also has a lower detection limit.

A similar comparison was made between an Andreason pipette and the Elzone 80XY using a -150 mesh silica sample. The results, shown in Figure 2.7, also indicate a close agreement between these techniques. It should be noted that the distribution obtained using the Andreason pipette does not drop off for the fine sizes since this technique mass balances itself.

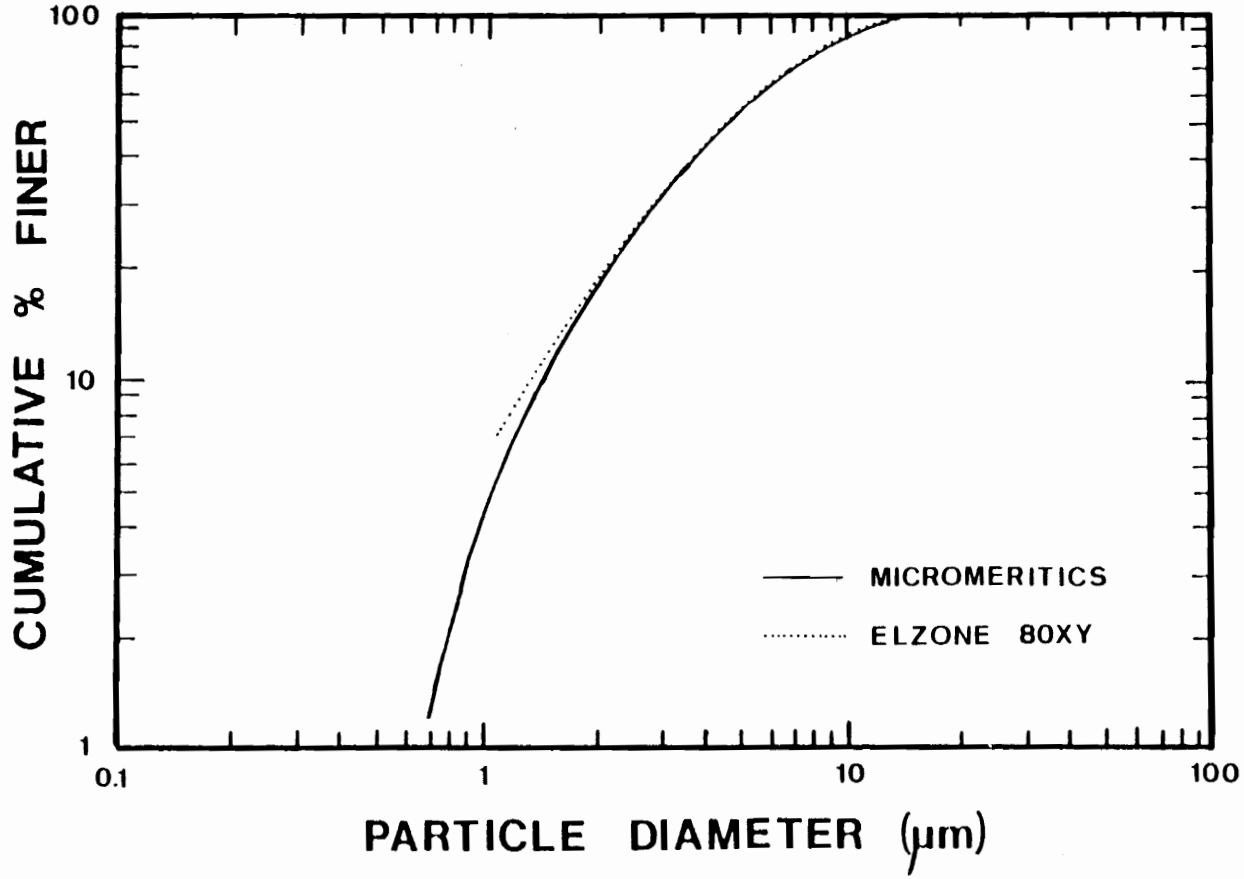

Figure 2.6. Comparison of the size distribution for a minus 20 micron Elkhorn seam coal sample using the Elzone 80XY and Micromeritics Sedigraph particle size analyzers.

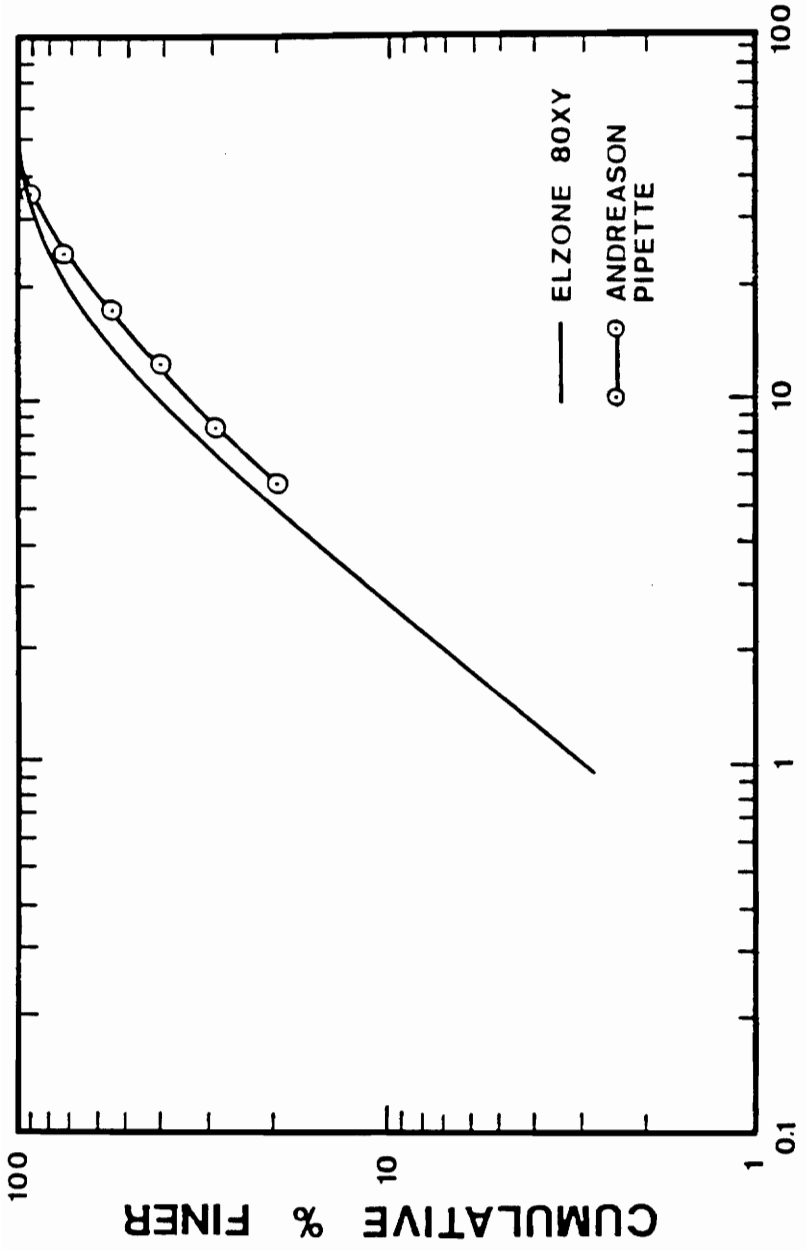

Figure 2.7. Comparison of the size distribution for a minus 150 mesh silica sample using the Elzone 80XY and the Andreason Pipette.

2.5.-Discussion

2.5.1.-Error due to Particle Characteristics

The results presented in section 2.4 indicate that the blending and mass balancing procedure provides an accurate and reproducible means of measuring fine particle size distributions. It is important to remember, however, that most sizing techniques possess several sources of error in addition to the error caused by missed material. These sources include errors due to the nature of the material being analyzed, errors due to sample size, and errors due to sample preparation. Methods of minimizing the errors due to sample preparation, caused by the non-ideal splitting, screening or centrifuging of samples, have already been discussed.

Consider now the errors due to the nature of the material being analyzed. Many materials exhibit compositions or porosities which vary widely with particle size. As a result, the specific gravity of these materials will vary with particle size. Equation (2.1), however, shows that the mass balancing procedure requires a single value for specific gravity. This may be considered a mean specific gravity for the sample. If there is any tendency for particles in a given size range to deviate significantly from the mean specific gravity, error will be introduced in the mass balancing procedure. Thus, one would expect the likelihood

of this error to increase for more heterogeneous materials.

Particle shape is another material characteristic which can have a bearing on the size distribution. Most particle size analyzers report particle size on the basis of an equivalent spherical diameter. For example, the Elzone measures the true particle volume and reports size on the basis of the diameter of a sphere having the same volume. If a material is sized which has a shape widely differing from spherical, than the size analyzer may produce misleading results. Fortunately, since the Elzone measures the true particle volume, a shape correction can be made provided the particle shape can be properly quantified.

2.5.2.-Statistical Error Analysis

The other source of error mentioned previously, i.e. errors due to sample size, refers to the number of particles which are counted and sized to produce statistcally meaningful data. Since the concentration of particles presented to the analyzer is usually controlled by the requirements of the equipment (e.g. to keep particle coincidence errors low), the number of particles to be sized determines the length of time for the analysis.

An examination of Gy's theory for sampling can be used to determine the error involved in sampling, as well as, the sample size necessary to minimize the standard deviation

(Gy, 1982). The variance on the cumulative size distribution can be calculated as follows:

$$\text{Var}\ (Y_i) = \left\{ \frac{Y_i^2}{M_S} \left[\sum_{j=1}^{N} X_j \rho V_j + \frac{1 - 2Y_i}{Y_i^2} \sum_{j=1}^{i} X_j \rho V_j \right] \right\}^{1/2} \quad (2.5)$$

where X_j is the weight fraction of material in a particular size class Y_i is the cumulative weight fraction finer than size i, ρ is the specific gravity of the sample, M_s is the sample mass and V_j is the mean volume of material in a particular size class, represented as:

$$V_j = 0.5 * \frac{d_i^3 + d^3_{i+1}}{2}. \quad (2.6)$$

Using equation (2.5), the sample mass required to maintain the relative error under 2%, for the cumulative distributions shown in Figure 2.3, was determined to be 7.0 x 10^{-4} grams. The actual mass presented to the orifice for analysis was 8.35 x 10^{-4} grams, which produced a total relative error of 1.89% on the cumulative distribution. This error analysis procedure has been included as part of the software package discussed earlier.

2.6.-Summary and Conclusions

The purpose of this study was to develop a procedure and computer software for providing an accurate representation of a particle size distribution determined through the use of an Elzone 80XY particle size analyzer. The following summarizes the results of this work.

1) A computer software package was developed to aid in blending the data from any number of consecutively smaller orifice tubes used with an Elzone 80XY particle size analyzer. A variety of subroutines were included in this package to assist in the manipulation of particle size distribution data.
2) A mass balancing procedure was developed to determine the amount of material passing the lower detection limit of the Elzone 80XY particle size analyzer. This procedure was incorporated into the software package to correct the original particle size distribution.
3) An error analysis procedure was included in the software package to determine the sample size necessary to maintain the relative error less than a specified value.

The results from this work indicate that an accurate and reproducible representation of fine particle size distributions can be obtained using the procedure described here. In addition, it is expected that the software package

could be extended to size analyzers other than the Elzone 80XY with only slight modifications.

2.7.-Nomenclature

C - concentration of solids in electrolyte being analyzed

d_i - particle diameter in size class i

f_i - mass fraction in size class i

$f_{i,c}$ - corrected mass fraction in size class i

M_C - mass of solids counted by a particular orifice tube; calculated from size and count data obtained from particle size analyser

M_e - mass of electrolyte added to S_a

M_P - mass of solids presented to a particular orifice tube for analysis

M_S - sample mass for error analysis

ρ - specific gravity of solids to be analyzed

N - total number of size classes

n_i - number of particles in size class i

Q - orifice volumetric flow rate

S_1 - original sample to be sized by first orifice tube

S_a - sub-sample for size analysis

S_f - final sample for analysis consisting of S_a and M_e

S_r - sub-sample for determining solid/liquid ratio in S_1

t - sample analysis time (sec)

V_j - volume of size class i

X_i - discrete weight fraction in size class i

Y_i - cumulative weight percent finer in size class i

CHAPTER III

Effect of Media Size in Stirred Ball Mill Grinding of Coal

3.1.-Introduction

In producing super-clean coal by any physical cleaning process, it is necessary to liberate the mineral matter from the coal prior to separation. Recent investigations, for example, have shown that microbubble flotation can be an effective method for producing coals containing less than 1% ash and very little sulfur provided that the feed coal has been micronized (Yoon and Miller, 1982; Yoon, 1984; Luttrell et al, 1985; Yoon and Luttrell, 1986) Microscopic examination of many of the United States' coals, however, reveals that sufficient liberation may not occur until the coal is ground finer than ten microns, and sometimes finer than a few microns. Although conventional ball milling is the most widely used technique for producing fines, it is difficult to grind coal to below-10 μm without requiring excessively long retention times and correspondingly high energy inputs. Other techniques such as fluid energy milling and vibratory milling are capable of micronizing coals, but they also tend to be energy intensive and are of low throughput capacity as well (Grimshaw and Albus, 1983; Herbst and Sepulveda, 1978).

Previous studies on mineral systems have indicated that stirred ball milling may be a promising method of producing micronized material (Herbst and Sepulveda, 1978; Stanczyk and Feld, 1968,1972; Sadler, Stanley and Brooks, 1975; Davis, Hansen and Sullivan, 1980; Sepulveda, 1981. In this technique, a large number of small grinding media (1/16- to 1/4-inch) are agitated by a pin-shaped impeller in a cylindrical vessel. Breakage occurs mainly by attrition and shearing as particles are captured between balls. The system is water-cooled to remove the excess heat generated by the attrition. Recently, Sepulveda (1981) has shown that when grinding a Lower Freeport coal to a median size of 8-m, an energy savings of approximately 60% can be achieved by using a stirred ball mill over a conventional ball mill. Greater energy savings should be expected for finer grinding.

Because of the importance of fine grinding in the production of super-clean coal, improvments in the efficiency of the grinding operation are of primary concern. This investigation deals specifically with the production of micronized coal using the stirred ball mill. The objective of this study has been to investigate the effect of various operating conditions, particularly that of media size, on the breakage rates and energy requirements of the process.

3.2.-Experimental

3.2.1.-Samples

The sample used in this investigation was prepared from a run-of-mine coal, Elkhorn seam, Virginia, containing 14% ash. The coal was passed through a roll crusher to produce a -1/4 inch product, which was then screened at 20 mesh. The -1/4-inch x 20 mesh fraction was cleaned in a heavy media bath (magnetite suspension) at a specific gravity of 1.3 to produce a clean coal product containing 6.5% ash. The clean coal was then hammer-milled to reduce the size to a suitable level for stirred ball milling. Size analysis showed that the hammer mill product had a 70-μm mean size. A portion of the hammer mill product was screened to obtain monosized feeds.

3.2.2.-Experimental Apparatus

Batch grinding experiments were conducted using a homemade 5-inch diameter mill with a 6-inch height, as shown in Figure 3.1. The mill included a double wall of 1/8-inch thick stainless steel, which acted as a water jacket. A five-level impeller made up of 4-inch pins was held in place by a sealed bearing. The rotation of the impeller was provided by a variable-speed drill press (Figure 3.2) equipped with a one-horsepower drive motor. A series of

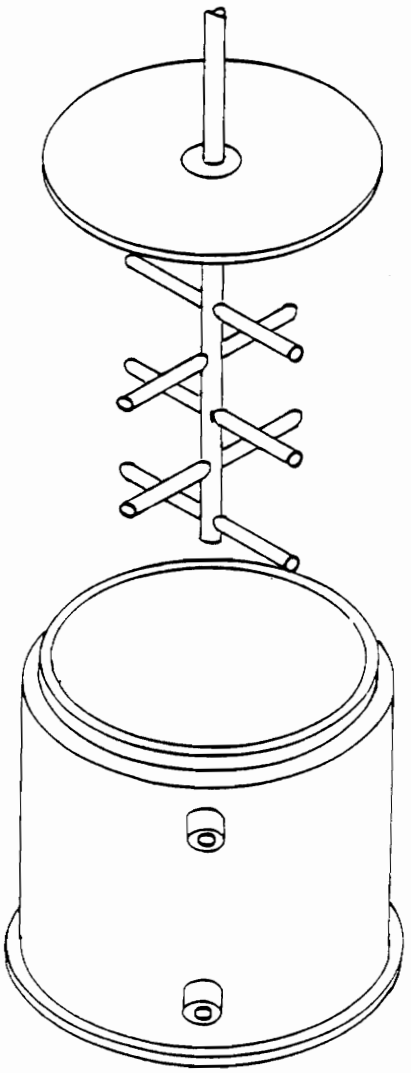

Figure 3.1. Schematic diagram of the batch attrition mill (Brown, 1986).

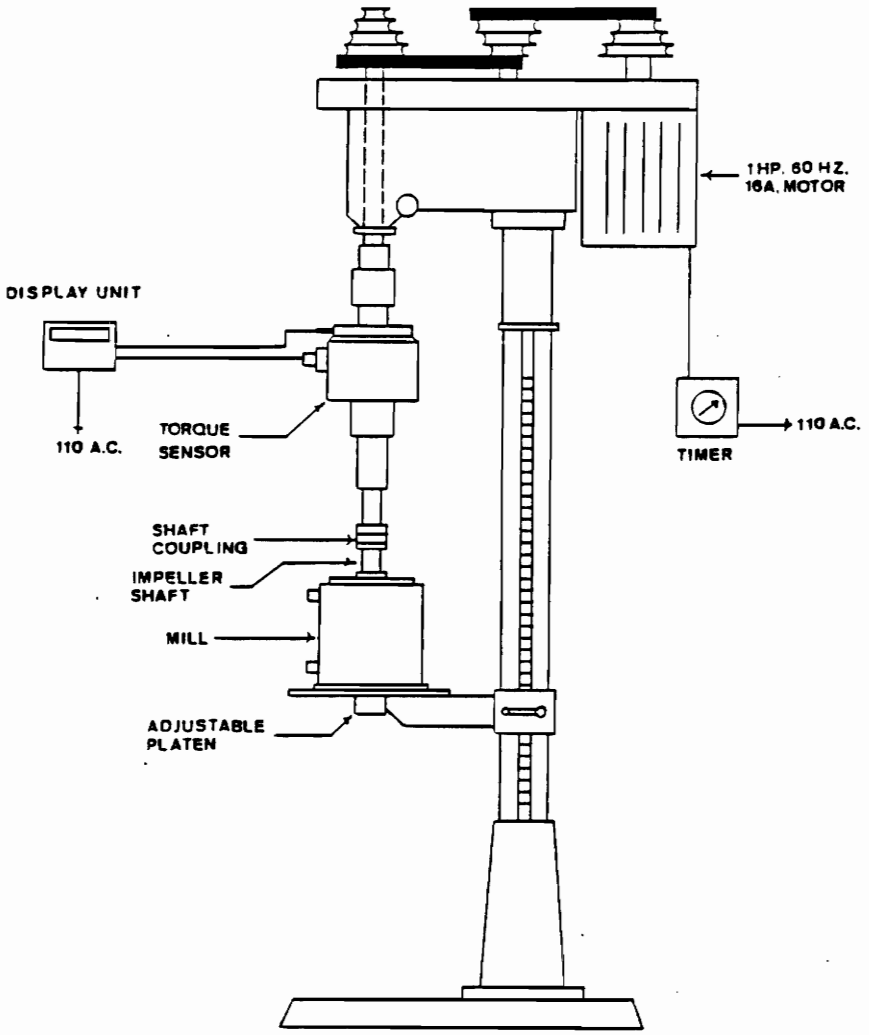

Figure 3.2. Schematic diagram of the mill assembly.

belts and pulleys enabled stirring speeds to be varied from 200 to 3500 rpm, while a moveable support platform allowed the mill to be raised or lowered for ease in loading and unloading of the mill charge. A Teledyne Model A-05 torque sensor, mounted on the drive shaft between the motor and the mill, provided an indication of the power input to the mill.

The grinding media used in this investigation included carbon steel and stainless steel balls of 1/4-, 1/8-, 3/32-, and 1/16-inch diameters, and glass beads of various sizes. Both monosized balls and ball mixtures were studied in this investigation.

Particle size distributions were determined using an Elzone 80XY particle size analyzer manufactured by Particle Data, Incorporated. This device uses the electrical sensing zone method for particle sizing in a manner similar to the Coulter Counter. With a given probe, the largest measureable size is limited to approximately 40% of the orifice diameter, while the smallest detectable size is about 1% of the orifice diameter. Therefore, several probes having different orifice diameters may be required for a complete size analysis.

3.2.3.-Experimental Procedure

Three series of tests were conducted to determine i) the effect of ball size on grinding kinetics, ii) the

effect of ball size on energy consumption, and iii) the effect of media type on stirred ball mill performance. The media loading for each test was held constant at 50% of the total mill volume. For those tests involving a ball mixture, the media charge was made from equal weights of 1/8-, 3/32-, and 1/16-inch balls. Slurry was added to the mill in a quantity sufficient to fill the void volume of the ball charge. Operating conditions for the tests varied from a solids loading of 30% to 43% by weight and from a stirring speed of 200 to 330 rpm. Torque readings were recorded at regular intervals during each test, and the area under the torque vs. time curve was used to give a measure of the total energy input to the mill.

Particle size analysis was conducted on all products generated in this investigation. However, since the size analysis technique used in this investigation had an inherent problem of missing particles finer than some finite limit, it was necessary to mass balance the size analysis data in order to get accurate results. It was also necessary to blend the size analysis data from several orifice tubes in order to get a complete size distribution. As a result, a computer program was developed to assist in both the blending and the mass balancing of size distribution data. An interface and the appropriate software were also developed to connect the Elzone 80XY to an IBM personal computer. Thus, all size analysis, blending

and mass balancing procedures were controlled from the computer.

3.3.-Results

3.3.1.-Rate of Breakage

The effect of ball size on the breakage kinetics in a stirred ball mill was studied by following the disappearance of monosized feed with time. These tests were carried out at 43% solids and 330 rpm. A typical set of disappearance plots for the grinding of a series of monosized feeds with 1/4-inch stainless steel balls is shown in Figure 3.3. As indicated by this figure, grinding rates were found to be very high. In fact, the grinding rates observed in this study were, in some cases, ten times higher than those reported for conventional ball mill grinding of similar coals (Klimpel, 1982). This necessitated the use of very short grinding times for conducting the tests. Note that first order breakage behavior was observed over the time period studied. A similar finding was also reported by others (Herbst and Sepulveda, 1978; Stanczyk and Feld, 1968, 1972; Sadler, Stanley and Brooks, 1975).

The breakage rates determined from Figure 3.3 are plotted in Figure 3.4 as a function of feed size. This figure shows that as the feed size increases, the grinding rate increases to a peak value at approximately 350-μm, and then decreases rapidly. The decrease in grinding rate above 350-μm indicates that the 1/4-inch balls are too small to nip the particles and break them.

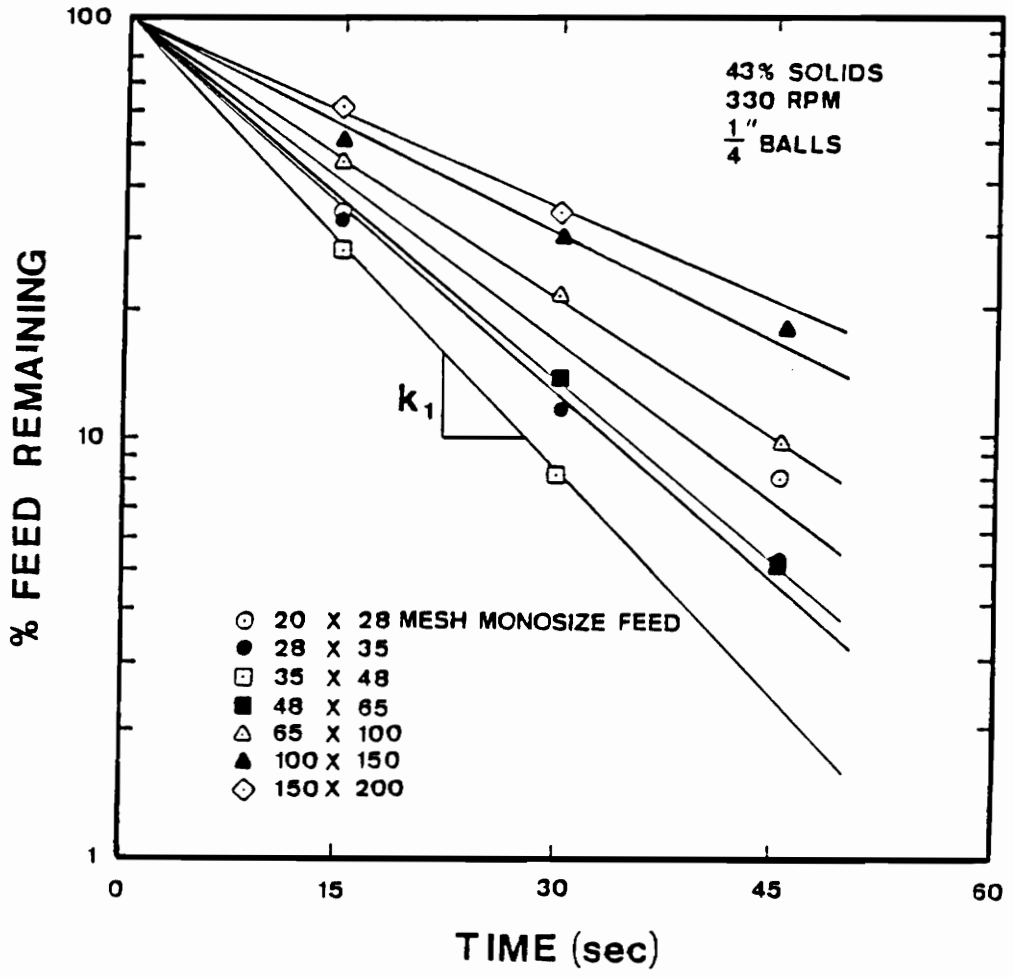

Figure 3.3. Feed disappearance plots for attrition milling of Elkhorn seam coal.

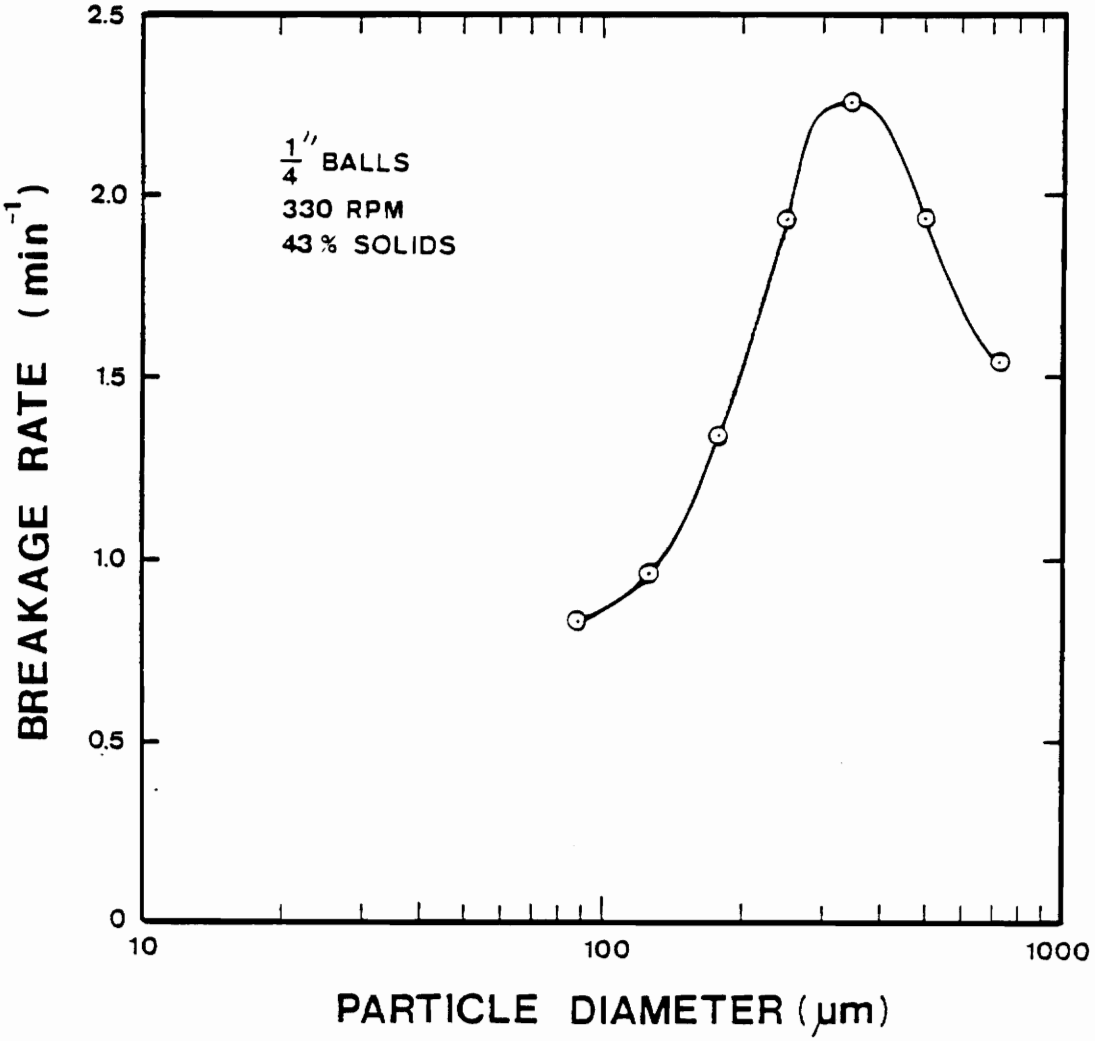

Figure 3.4. Grinding rate vs. mean feed size for attrition milling of Elkhorn seam coal.

Thus, there appears to be an optimum ball size/feed size ratio for stirred ball mill grinding. Figure 3.5 shows the results obtained using stainless steel grinding media of different sizes for four different sizes of feeds. It shows that the breakage rate decreases with decreasing particle size and, more importantly, that there is an optimum ball size for each feed size which decreases with decreasing feed size.

3.3.2.-Energy Consumption

Several tests were conducted at a constant specific energy input of 94 kwh/ton to determine the effect of ball size on the final product size distribution. These tests were carried out using the hammer mill product (70-μm mean size) as the feed at a pulp density of 43% solids and a stirring speed of 200 rpm. The results given in Figure 3.6 show that the product size distribution becomes finer as the media size is reduced. For example, 1/4-inch balls produced a mean size of 6-μm, while 1/16-inch balls produced a mean size of 3-μm. It should be noted that at these fine sizes, even a small change in product size is significant. Also shown in Figure 3.6 is the product size distribution obtained using a mixture of 1/8-, 3/32-, and 1/16-inch balls prepared using equal weights. It is interesting to note that this curve lies where one would expect based on the

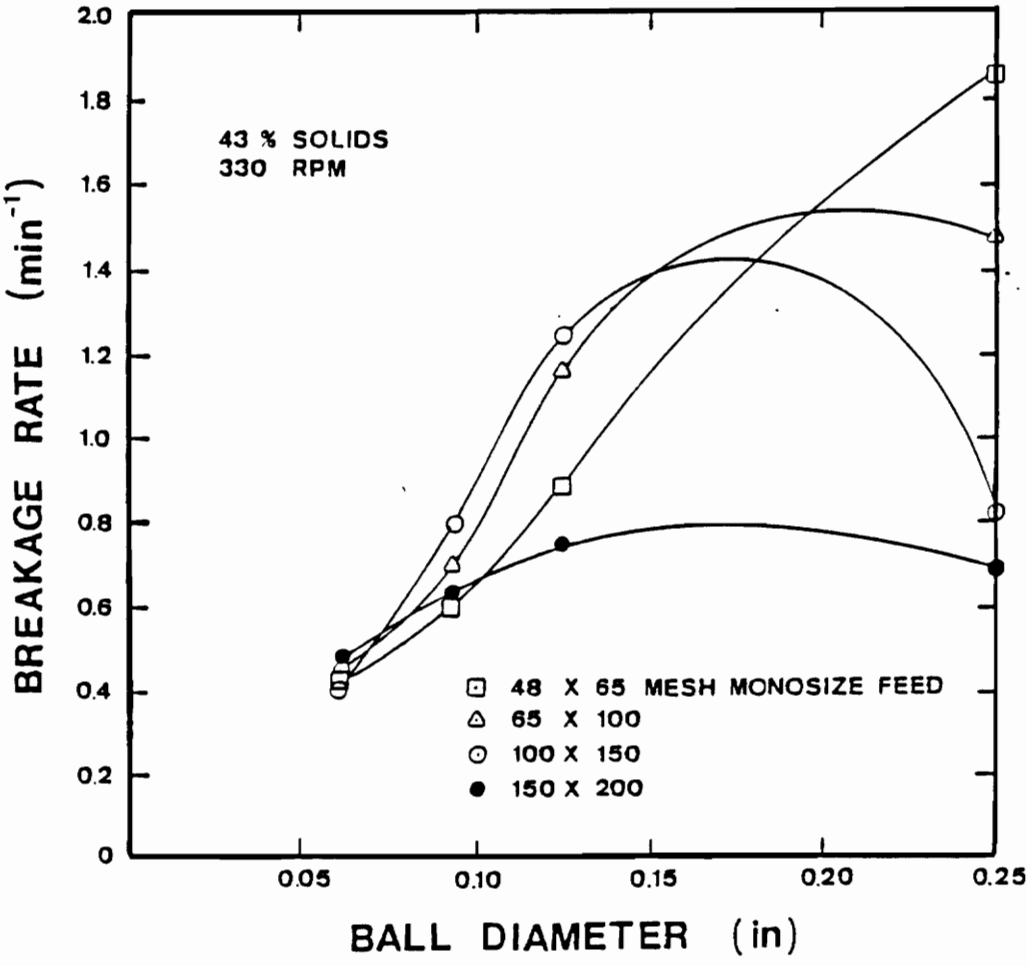

Figure 3.5. Grinding rate vs. ball size for attrition milling of Elkhorn seam coal.

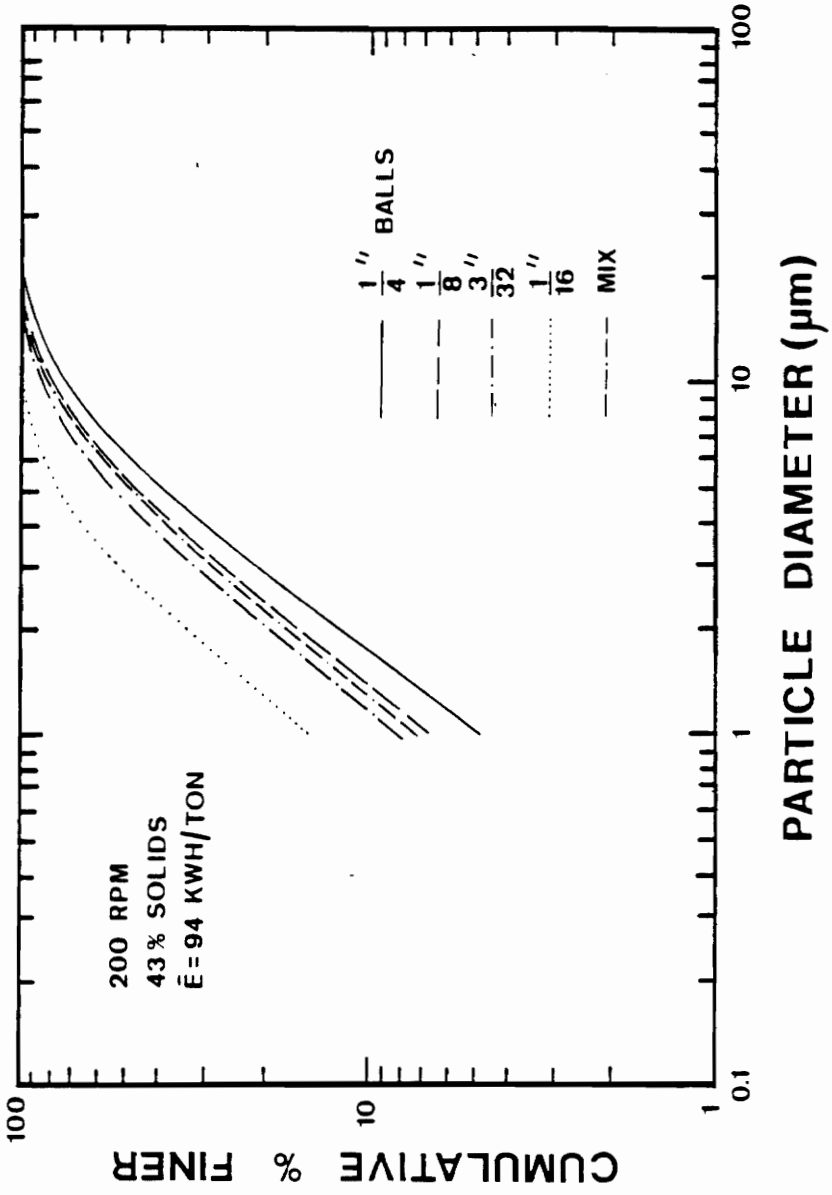

Figure 3.6. Effect of ball diameter on product size distribution at constant energy input for attrition milling of Elkhorn seam coal.

number mean diameter (.07 inches) of the ball mixture.

Utilizing the same operating conditions as previously described, i.e., 43% solids and 200 rpm, a similar series of tests was conducted over a range of energy inputs. These results are shown in Figure 3.7 in which the mass mean diameter of each product distribution is plotted as a function of specific energy input. Previously, Herbst and Sepulveda (1978) have shown that these types of plots can be described by Charles' equation which, for the case where feed size is much larger than product size, is given by

$$\bar{E} = A(d_{m,p}^{-\alpha}), \tag{3.1}$$

in which $\bar{E}$ represents the specific energy input and $d_{m,p}$ is the mean product size. According to Herbst and Sepulveda, there exists a single energy-product relationship for a given material regardless of operating conditions and mill sizes. The results obtained in the present study show, however, that separate energy-product relationships exist for different media sizes. It can be seen that, as ball size decreases, significantly less energy is required to produce a given product size. For example, an energy input of approximately 100 kwh/ton is required to produce a 5- m mean product size with 1/8-inch grinding media, while the same product can be achieved at 45 kwh/ton with 1/16-inch media. This decrease in energy requirement is manifested in a shift in the intercept, A. The slope, α, however, appears

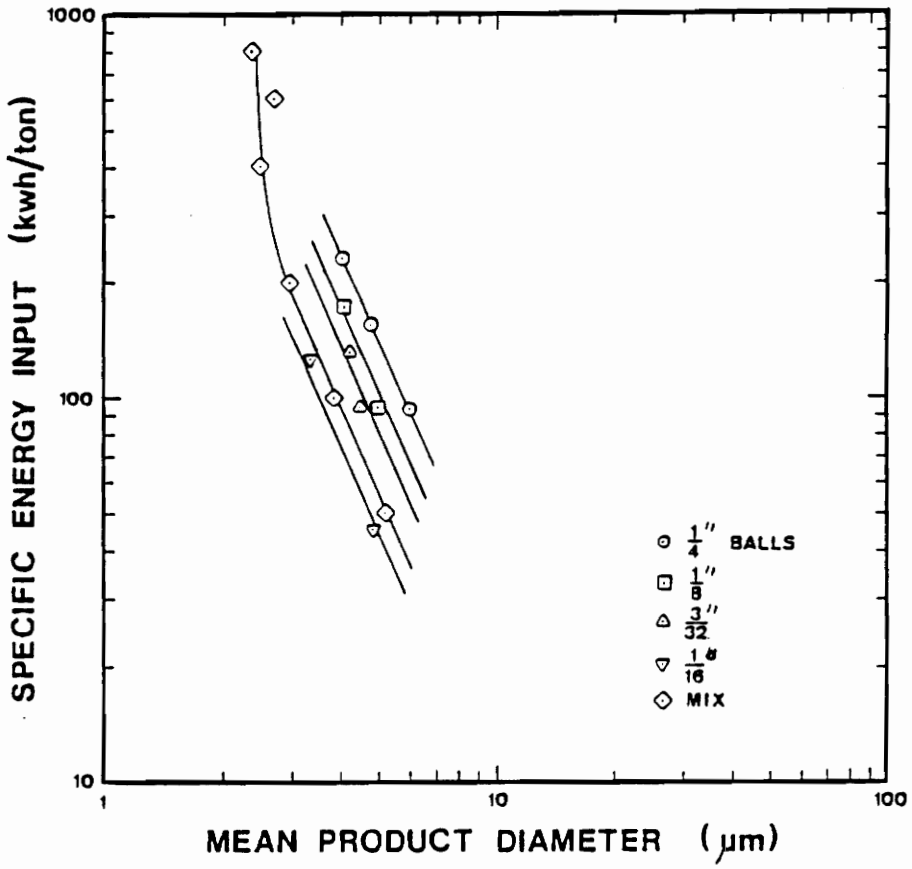

Figure 3.7. Effect of ball diameter on Charles' law for attrition milling of Elkhorn seam coal.

to remain constant at a value of 3 for all ball sizes for relatively short grinding times.

Figure 3.7 also shows that when grinding proceeds for a long time (i.e., high energy inputs), the $\bar{E}$ vs. d_m plot deviates significantly from the linear relationship predicted by Charles' equation. In the tests conducted under the present conditions, it does not appear possible to generate a product having a mean size much finer than 2 μm. Thus, care must be taken when using the data presented in Figure 3.7 to estimate the energy requirements for product sizes finer than those shown.

3.3.3.-**Media Type**

A major part of the energy requirement for stirred ball milling involves stirring and lifting of the ball charge itself. Therefore, if the density of the grinding media can be reduced without significantly affecting the grinding performance, one might expect a lower energy consumption.

Figure 3.8 shows a comparison between the performance of steel and glass media over a range of media sizes. In this comparison, tests were conducted using a constant energy input of 8.5 kwh/ton at a pulp density of 30% and a stirring speed of 330 rpm. The grinding performances of each media type are compared by plotting the percent of monosized feed remaining in the mill as a function of ball

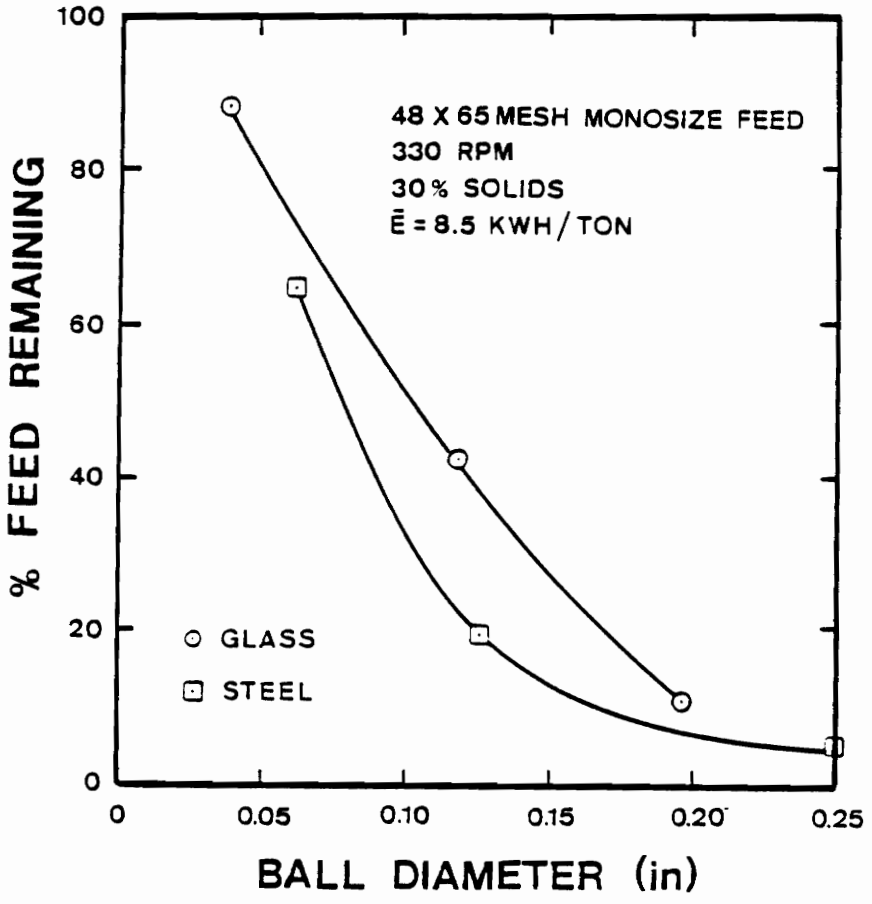

Figure 3.8. Effect of media type on grinding rate for attrition milling of Elkhorn seam coal.

diameter. Since the torque required to turn the glass grinding media was much lower than with steel media, the glass media required a longer grinding time to achieve the same energy input as the steel media. The results show that steel balls were slightly more efficient than the glass beads, indicating that the mass of the individual grinding medium is important in determining the attrition force operating in the mill. Similar results have been obtained using other monosized feeds.

3.4.-Discussion

3.4.1.-Effect of Particle Size on Breakage Rate

From the results presented in Figure 3.4, it is clear that for a given ball size, there exists an optimum feed size for maximum breakage rate. It seems that at small feed sizes, the large number of particles present in the mill results in a low probability that an individual particle will encounter the grinding media. At large feed sizes, on the other hand, particles become too big to be caught between the grinding media and broken effectively. Therefore, the maximum breakage rate occurs when particles are large enough to have a high probability of encountering the grinding media, but are small enough to be effectively caught and broken by the media.

Looking more closely at the grinding behavior at fine sizes, it can be shown that during the course of a typical grinding test, the mass mean particle diameter is decreased by approximately one order of magnitude. For example, the results given in Figure 3.6 show a feed distribution with a mean size of 70-μm reduced to 6-μm using the 1/4-inch balls. This produces an increase in the actual number of particles in the mill by four orders of magnitude. As a result, the probability that a given particle will be captured between two or more balls decreases, resulting in a lower breakage rate as grinding proceeds. In order to increase the

probability of particle capture, it is necessary to increase the number of balls in the mill as the particle size decreases. For a given mill volume, this can be accomplished by decreasing the media size. For example, by decreasing the ball diameter from 1/4-inch to 1/16-inch, the number of balls in the mill is increased from approximately 3300 to 240,000, resulting in a corresponding increase in the breakage rate and the energy efficiency, as shown in Figure 3.6.

3.4.2.-Angle of Nip Analysis

To understand the breakage behavior of coarse particles, it is necessary to consider the grinding action which takes place inside a low-speed stirred ball mill. During the course of the batch grinding tests conducted for this investigation, it was observed that the balls in the mill tended to roll around each other and against the wall like thousands of tiny roll crushers. If breakage between balls in a stirred ball mill can in fact be compared to breakage between the rolls of a roll crusher, one important criterion for breakage will be the angle of nip.

In a roll crusher, the angle of nip (2α) is defined as the critical angle at which a particle can be pulled down between two rolls rotating in opposite directions at equal speed. As illustrated in Figure 3.9, it is a function of

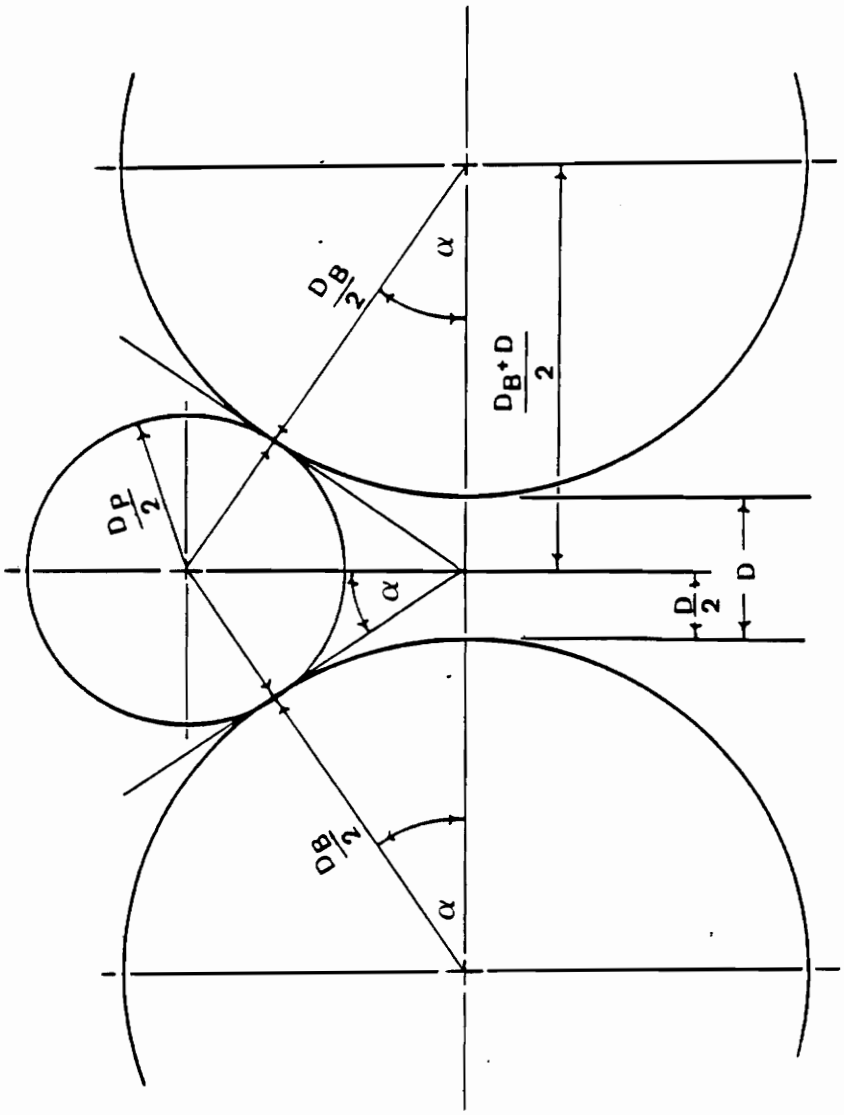

Figure 3.9. Schematic representation of the angle of nip between grinding media in a stirred ball mill.

roll diameter (or ball diameter, D_b, for the case of stirred ball milling), particle diameter (D_p), and the distance (D) between the rolls (or balls). By considering the geometrical relationships and the force balance between the balls and the particle, α can be expressed as a function of D_b, D_p, and D,

$$\alpha = \cos^{-1} \frac{D_b + D}{D_b + D_p} \tag{3.2}$$

and also of the coefficient of friction (μ) between the particle and the balls,

$$\alpha = \tan^{-1} \mu \tag{3.3}$$

as has been shown by Gaudin (1939).

If μ is known, α can be calculated from Equation (3.3), which can then be substituted in Equation (3.2) to obtain a useful parameter such as the critical ball size to particle size ratio $(D_b/D_p)_c$. In stirred ball mill grinding using grinding media of uniform size, the particles that have a D_b/D_p value less than the critical value will not be broken. Unfortunately, the value of μ is not known for the present system. However, it has been shown in Figure 3.4 that when using 1/4-inch balls, the breakage rate passes through a maximum at a D_b/D_p value of approximately 20. As has already been discussed, the breakage rate increases with increasing particle size until the particles become too

large to be nipped. Therefore, the D_b/D_p value at the maximum breakage rate may be regarded as the critical value. If this value is substituted in Equations (3.2) and (3.3), a value of 0.32 is obtained for μ. In this calculation, the separation distance (D) is assumed to be negligibly small compared to D_b and D_p. This value appears to be quite reasonable considering that, typically, the coefficient of friction between steel and most ore particles ranges from 0.2 to 0.3 (Wills, 1985).

3.4.3.-Normalization of Breakage Rate

If the angle of nip is a rate-limiting factor for stirred ball mill grinding, as discussed above, it follows that the optimum D_b/D_p ratio of 20:1 should remain constant regardless of ball size. In order to test this hypothesis, the breakage rates shown in Figures 3.4 and 3.5 have been replotted as a function of the D_b/D_p ratio, as shown in Figure 3.10. This figure continues to show the existence of separate curves for each ball size with the largest breakage rates associated with the largest ball sizes. However, as a result of the normalization of the x-axis, the optimum breakage rate for all ball sizes appears to occur at a D_b/D_p ratio of approximately 20. If the breakage rates plotted on the y-axis in Figure 3.10 are normalized with grinding energy, then all data can be represented by a single curve,

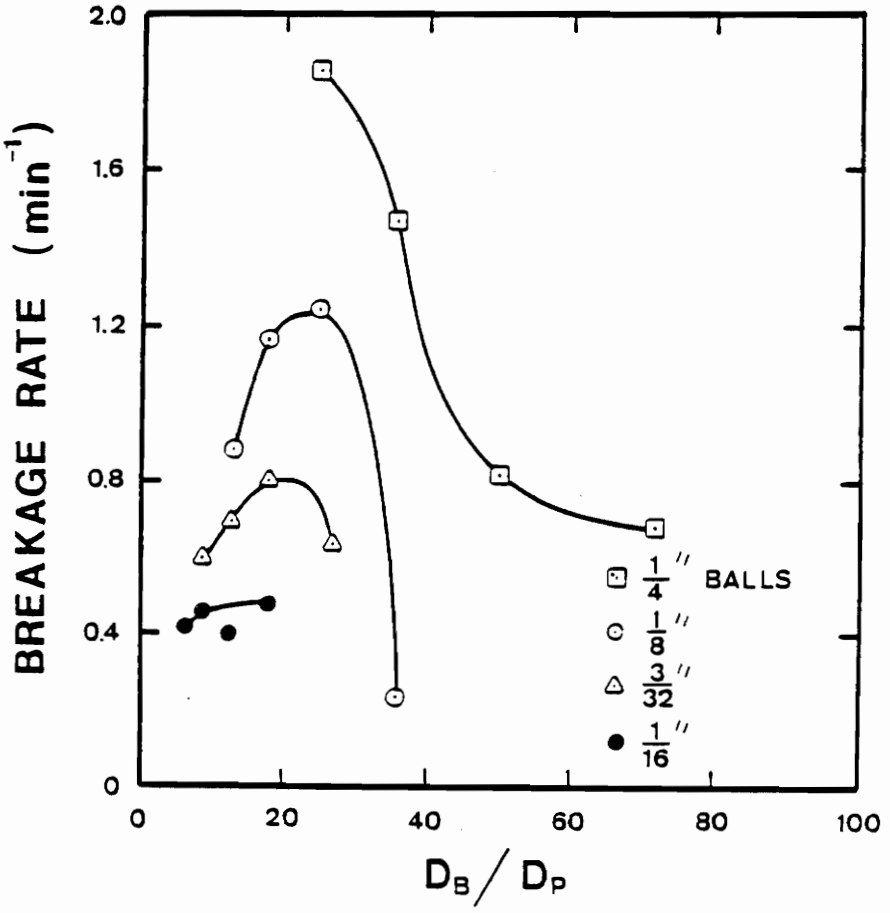

Figure 3.10. Breakage rate vs. normalized ball diameter for attrition milling of Elkhorn seam coal.

as shown in Figure 3.11. This figure shows a well-defined optimum energy-specific breakage rate at a D_b/D_p ratio of 20:1. The steady increase in specific breakage rate with decreasing D_b/D_p is due to the increasing probability that a particle will encounter grinding media. Beyond the optimum ratio of 20:1, however, the probability that a particle will be nipped and broken between churning balls decreases rapidly, resulting in a reduced breakage rate.

A similar analysis can be applied to Figure 3.6 in which the performances of different size media are compared in grinding a feed coal having a 70-μm mean size. This figure shows that 1/16-inch balls gave the best results on the basis of the product size distribution. The reason that this particular ball size gave the best results can now be understood when the D_b/D_p ratios are compared in Table 3.1 As shown, the 1/16-inch balls gave a D_b/D_p value closest to the optimum value of 20:1.

The angle-of-nip theory proposed here is based on the relationship between ball size (D_b) and feed size (D_p). One must realize, however, that once the grinding proceeds, the feed size decreases continuously in a batch operation. This means that as the grinding progresses, smaller grinding media must be used to keep the grinding operation under optimum conditions. Therefore, allowing an excessively long grinding time without changing the media size is an inefficient method of producing fines. An example of this

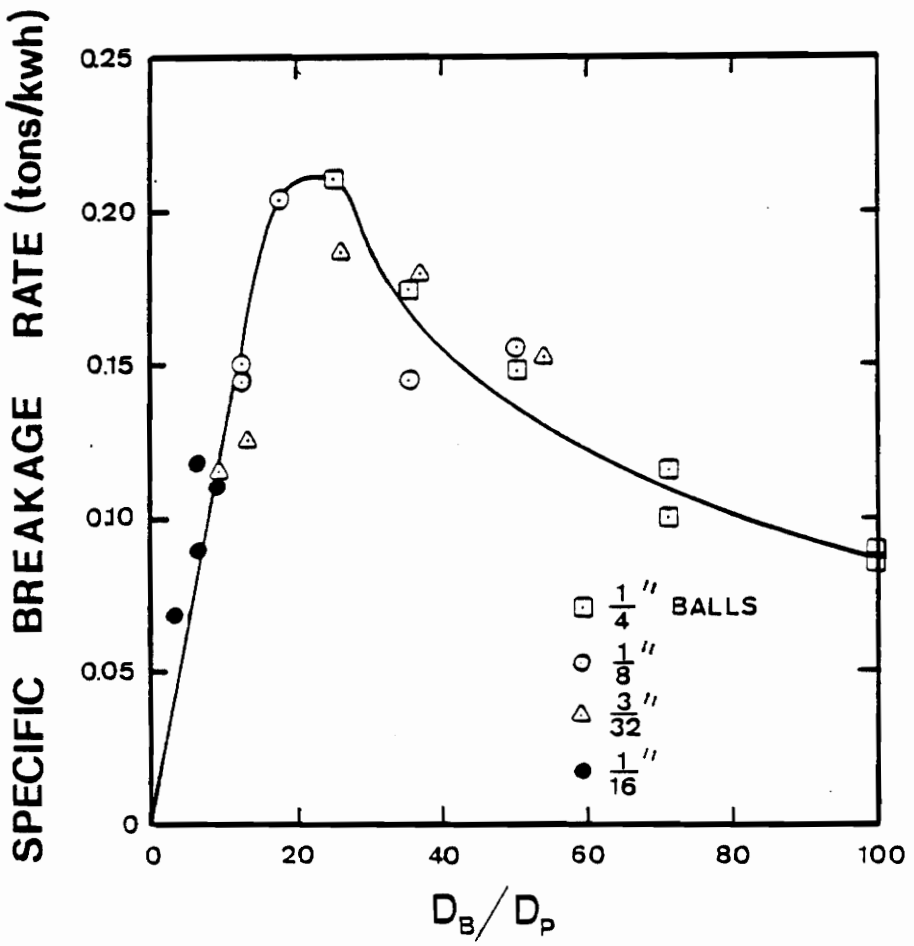

Figure 3.11. Specific breakage rate vs. normalized ball diameter for attrition milling of Elkhorn seam coal.

Table 3.1

Ball/Particle Diameter Ratios

Ball Diameter	Ball/Particle Diameter
1/4-inch	84.7
1/8-inch	42.3
3/32-inch	31.8
1/16-inch	21.2

is given in Figure 3.6, which shows the results obtained after extended grinding time using a mixture of 1/8-, 3/32-, and 1/16-inch balls. Since $(D_b/D_p)_c$ is a function of μ, it is also important to realize that the efficiency of stirred ball milling can be improved by selecting the appropriate type of grinding media.

3.5.-Summary and Conclusions

This investigation has been concerned with the production of micronized coal using the stirred ball mill grinding technique. Specifically, the effects of media type and size have been studied for the purpose of optimizing the energy usage. As a result of this investigation, the following conclusions can be drawn:

1) Grinding kinetics are significantly affected by the ball diameter/particle diameter ratio in the mill.
2) The optimum ball size/feed size ratio can be related to the angle of nip conditions required for roll crushers.
3) For the coal tested in the present work, the specific breakage rate is maximum when the ball size/feed size ratio is 20:1.

The results obtained in the present work show that stirred ball milling is an efficient method of producing micronized coal. For the coal sample used in this investigation, an energy input of approximately 45 kwh/ton was needed to produce a product with a mean size of 5-μm.

CHAPTER IV

Effect of Operating Parameters in Stirred Ball Mill Grinding of Coal

4.1.-Introduction

In addition to the media size used in a stirred ball mill, a number of other operating parameters can affect the grinding behavior in this process. An appropriate change in impeller speed, for example, may improve the breakage kinetics, resulting in a lower required residence time for material in the mill and an increased throughput. If this increase in throughput is larger than the increase in the energy consumption, a net energy savings may be realized. Likewise, if the percent solids can be increased while maintaining the same product size and the same mill residence time, an improvement in the mill throughput per kilowatt hour of energy consumed will also be achieved.

The purpose of this chapter is to present results showing the effect of stirring speed and pulp density on the energy consumed during stirred ball milling. The use of grinding aids to improve the grinding efficiency at high pulp densities is also explored.

4.2.-Experimental

4.2.1.-Samples

The sample used in these tests was Elkhorn seam coal obtained from United Coal Company. The sample was prepared according to the procedure outlined in chapter 3. The bulk of the -1/4-inch x 20 mesh product from the heavy media bath was dried and stored under nitrogen. Sub-samples were periodically removed and frozen in air-tight bags until needed. As previously mentioned, the clean coal was hammer-milled to produce a suitable feed for the stirred ball mill. The natural product size distribution from the hammer mill was used in all of the following tests.

4.2.2.-Reagents

Three different reagents were used in the present work: Sodium meta-Silicate, Lomar D obtained from the Diamond Shamrock Company, and an experimental polymer emulsion (XDR-00057-526-22B) provided by Dow Chemical Company.

4.2.3.-Experimental Procedure

Three series of tests were conducted to determine i) the effect of impeller speed, ii) the effect of feed percent solids, and iii) the effect of grinding aids on the product size distribution from the mill. The media loading was

again held constant at 50% of the mill volume with 100% slurry filling of the media void space. Feed percent solids varied from 20% to 60% solids by weight and impeller speed from 200 to 750 RPM. Torque readings taken at regular intervals for the duration of the test were used to determine the energy input to the mill.

A representative sample was removed from the mill at the conclusion of each test. The samples were prepared and analyzed, using an Elzone 80XY particle size analyzer, as described in Chapter 2.

4.3.-Results

4.3.1.-Effect of Stirring Speed

The effect of impeller speed on the product particle size distribution was determined by conducting a series of tests at equal energy input while varying the speed of the impeller. These tests were conducted at a constant specific energy input of 63.5 kwh/ton using a hammer mill product (70-micron mean size) as the feed at a pulp density of 43% solids by weight. A 3800 gram ball mixture was used which consisted of equal weights of 1/4-, 1/8-, and 3/32-inch balls. The results for stirring speeds of 765, 521 and 200 RPM are shown in Figure 4.1. These results show that as stirring speed is decreased the product size distribution becomes finer. For example, a stirring speed of 765 RPM produced a mean size of 7 microns, while a 200 RPM stirring speed produced a mean size of 5.8 microns. This tends to agree with results reported by Sepulveda (1980) for the stirred ball milling of chalcopyrite which indicates that energy is utilized more efficiently when it is input slowly.

The same results were obtained when repeating these tests at an energy input of 94 kwh/ton, in which the range of stirring speeds was extended to 1200 and 1350 RPM. For example, as shown in Figure 4.2, a stirring speed of 1350 RPM produced a mean product size of 8 microns, while a 765 RPM stirring speed produced a mean product size of 5 microns.

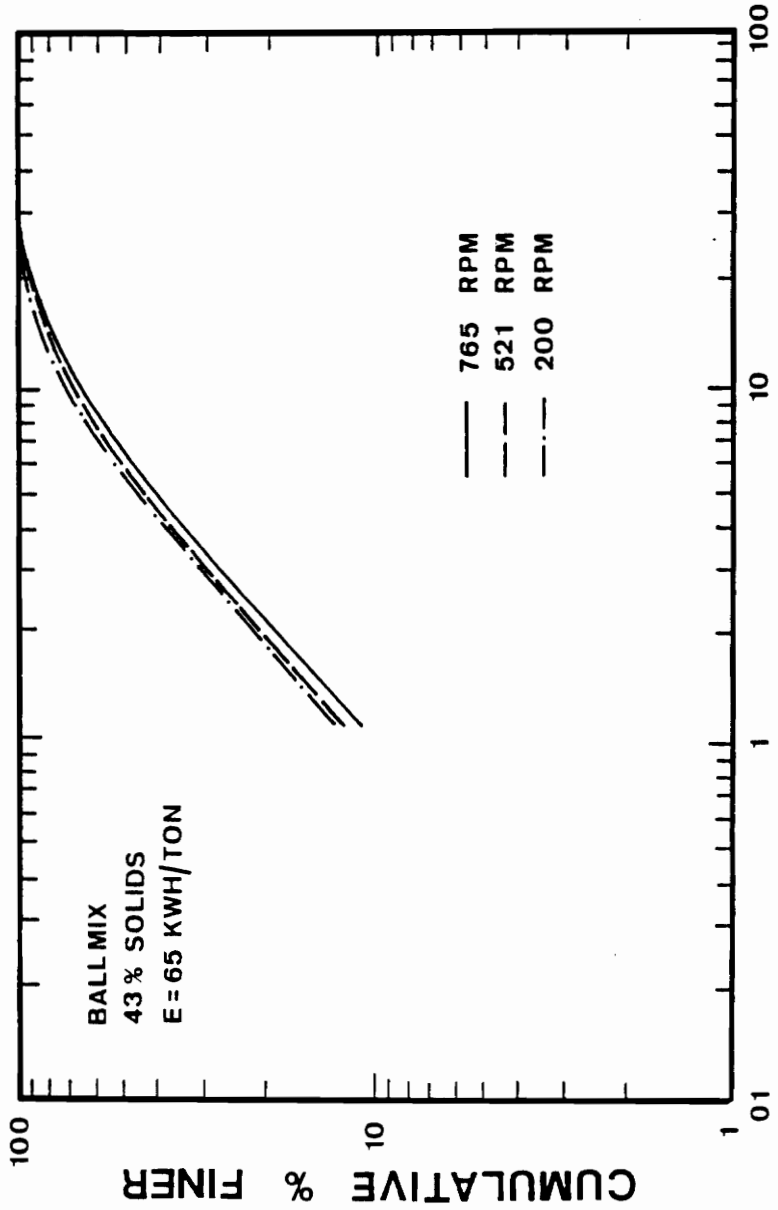

Figure 4.1. Effect of stirring speed on product size distribution at constant energy input of 65 (kwh/ton) for attrition milling of Elkhorn seam coal.

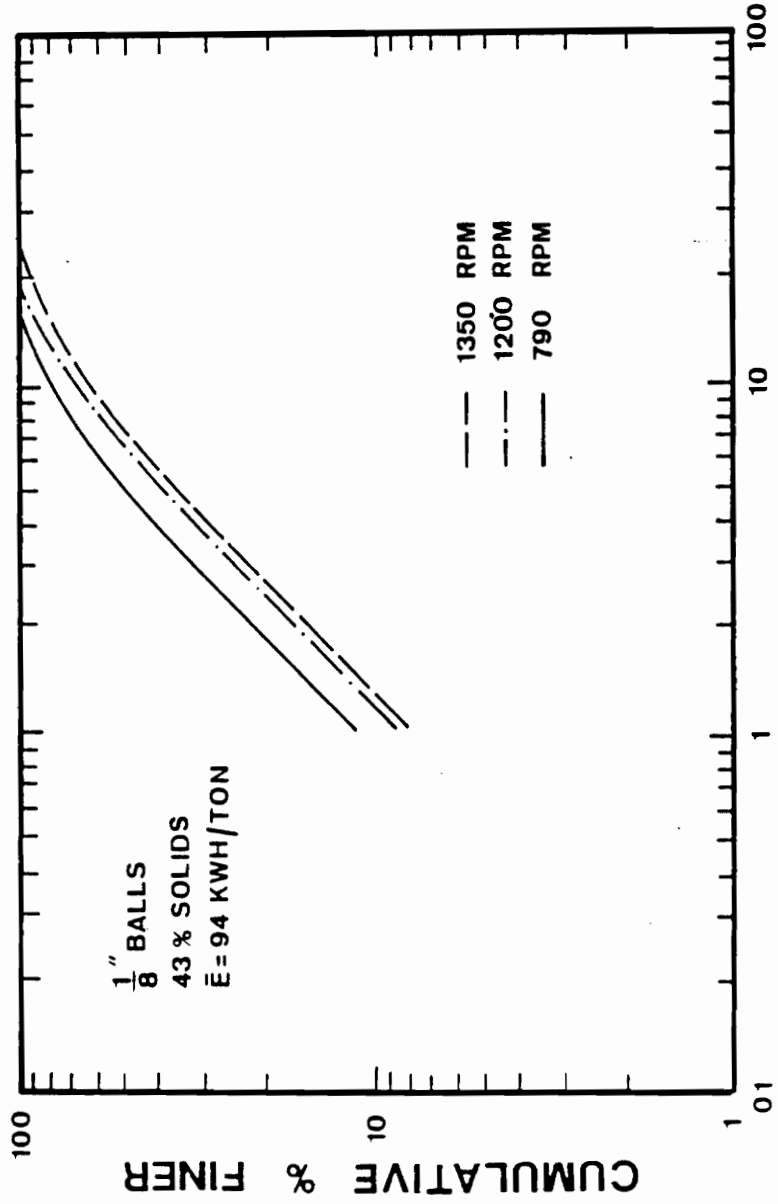

Figure 4.2. Effect of stirring speed on product size distribution at constant energy input of 94 (kwh/ton) for attrition milling of Elkhorn seam coal.

Similar results were reported by Stehr and Herbst (1984) for the grinding of coal water slurries using a Drais stirred ball mill. Their results indicate an energy savings of approximately 25% when grinding at a stirring speed of 562 RPM as opposed to 786 RPM, provided the energy input to the process exceeds 20 kwh/ton. Stirring speeds higher than 1350 RPM were not possible due to equipment limitations.

4.3.2.-Effect of Pulp Density

The effect of percent solids at constant energy input is shown in Figures 4.3 and 4.4. The previously described ball mixture was used for each test. The tests shown in Figure 4.3 were conducted at 94 kwh/ton and a stirring speed of 330 RPM. It was noted that over the range of 20% to 50% solids by weight no significant change in the product size distribution was observed. This agrees with previous work conducted on chalcopyrite (Sepulveda, 1980). The 60% solids test provided only a slightly coarser product size.

For a higher value of specific energy input (280 kwh/ton), as shown in Figure 4.4, the findings were the same for percent solids of 20% to 50%. The product size distributions had a mean size of 3.4 microns. The test involving 60% solids, however, produced a coarser and somewhat broader distribution having a mean product size of 4.5 microns. This could possibly be attributed to a

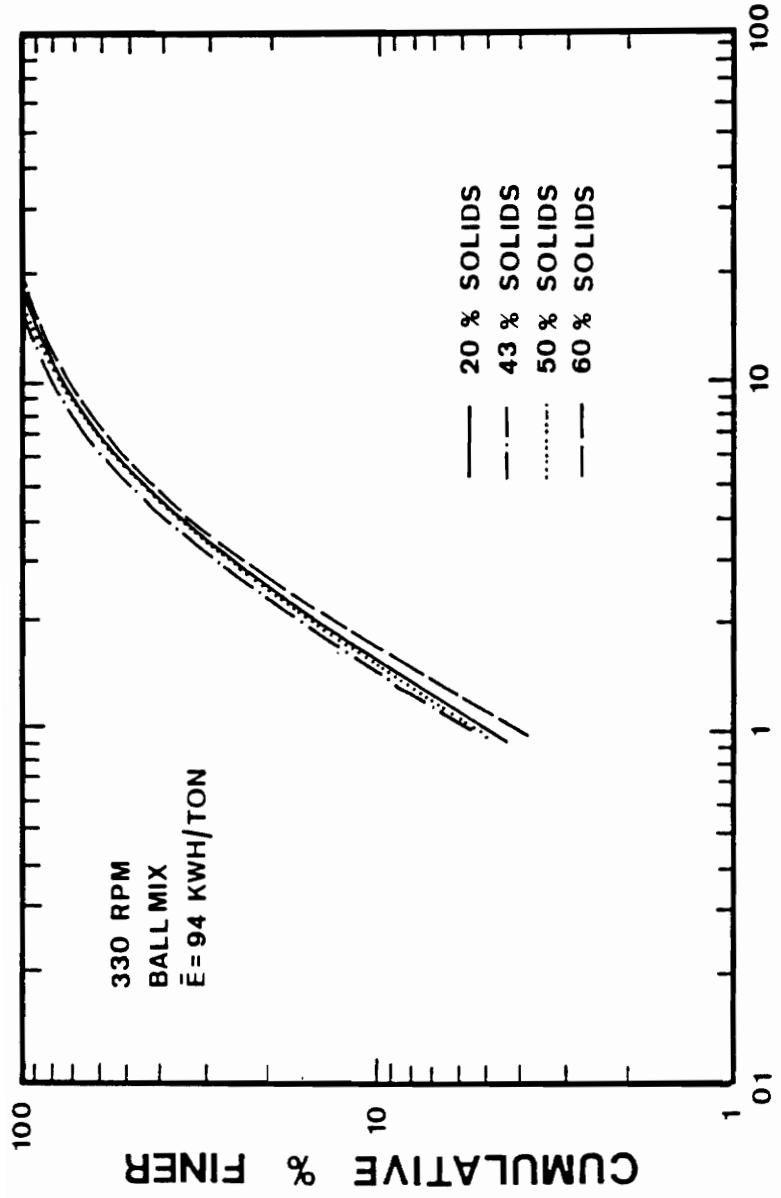

Figure 4.3. Effect of percent solids on product size distribution at constant energy input of 94 (kwh/ton) for attrition milling of Elkhorn seam coal.

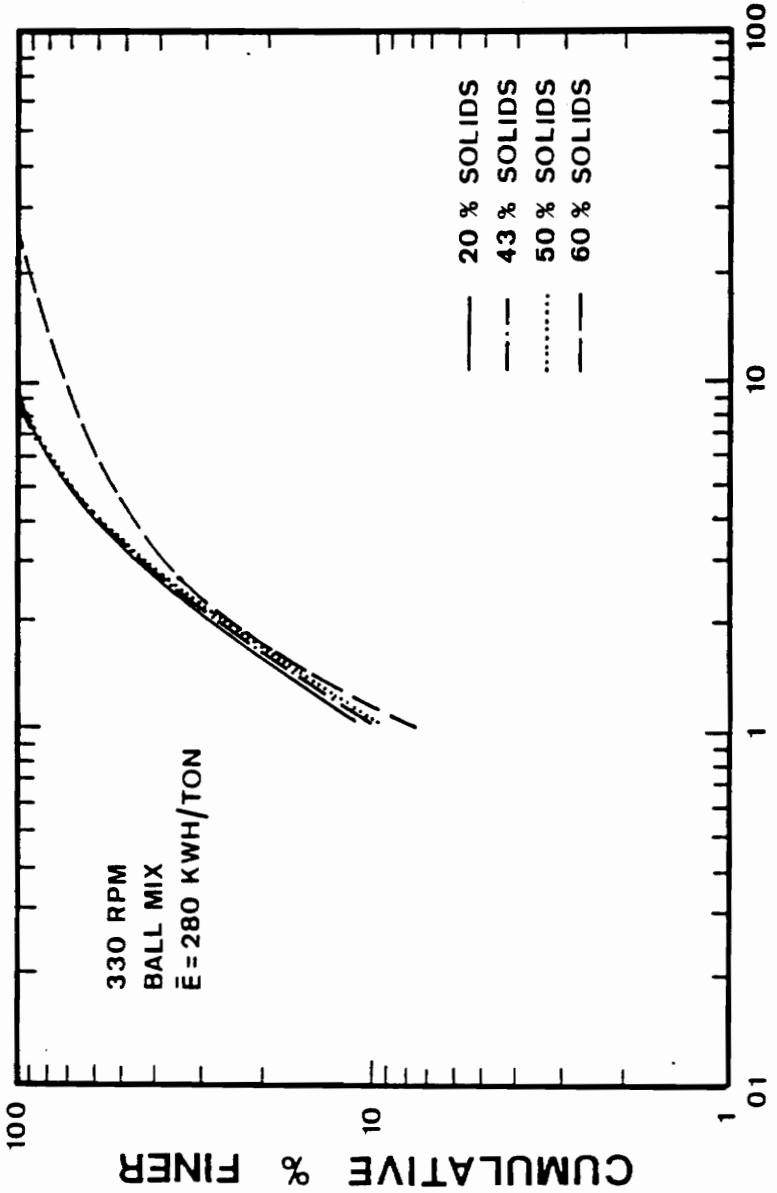

Figure 4.4. Effect of percent solids on product size distribution at constant energy input of 280 (kwh/ton) for attrition milling of Elkhorn seam coal.

viscosity problem indicating that a viscosity modifier might be effective for very fine grinding at high percent solids.

4.3.3.-Effect of Grinding Aids

Several tests were conducted at a constant specific energy input of 94 kwh/ton to determine the effect of grinding aid addition on the final product size distribution. These tests were carried out using a ball mixture, a feed pulp density of 50% and a stirring speed of 330 RPM. The results are plotted in Figure 4.5 as mean product size versus grinding aid addition (lbs/ton of coal) for three different grinding aids; sodium silicate, Dow XDR and Lomar D. It is clear from this figure that the Lomar D was the most effective grinding aid, reducing the mean product size from 7.8 microns with no reagent addition to 2.8 microns at an addition of 16.5 lbs/ton. The Dow XDR reagent produced similar results to Lomar D, although it was not as effective until the addition reached 14 lbs/ton. Sodium silicate appeared to show little beneficial effect even at additions as high as 50 lbs/ton.

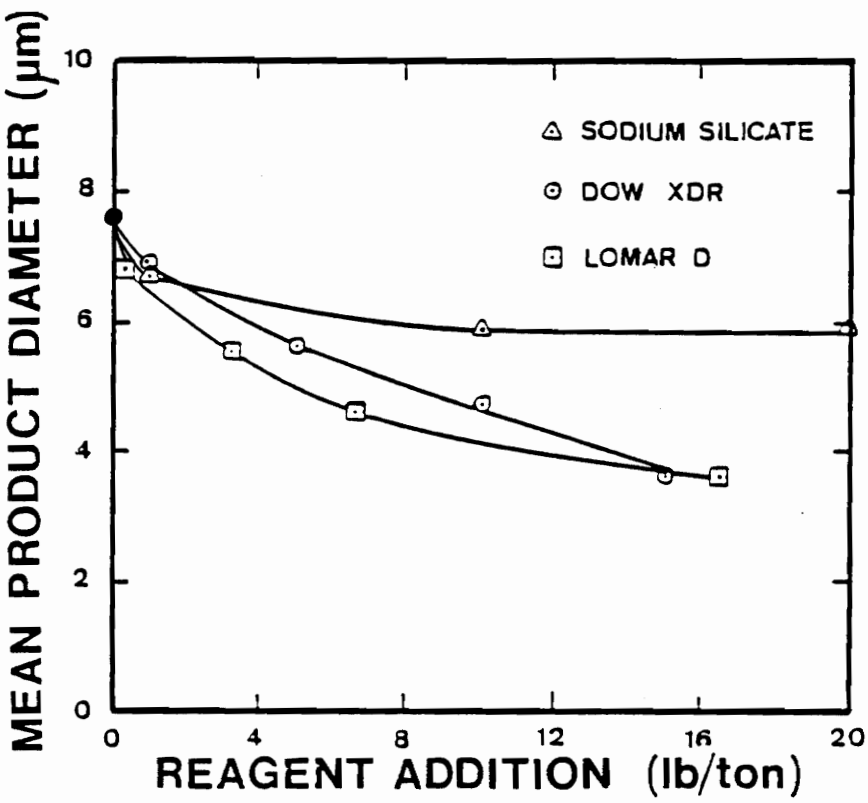

Figure 4.5 Effect of grinding aid addition on the mean product size at constant energy input of 94 (kwh/ton) for attrition milling of Elkhorn seam coal.

4.4.-Discussion

4.4.1.-Stirring Speed

The results presented in Figures 4.1 and 4.2 indicate that slower stirring speeds tend to be more energy efficient. This result may arise from several factors which are affected by a change in stirring speed. The most obvious effect of a change in speed is the change in torque requirements for the mill. The increase in torque with grinding speed is illustrated in Figure 4.6, for several ball sizes. This figure also shows that the addition of slurry to the mill has little effect on the torque requirements. This suggests that the increase in torque with increasing stirring speed is due to interaction between the media. Results of this type are commonly observed when spherical particles are subjected to shear (Bagnold, 1954). Therefore the grinding time must be decreased as the speed is increased in order to achieve an equal energy input to the mill. However, the results shown in Figures 4.1 and 4.2 indicate that lower speeds produce a finer product size distribution. The improved efficiency in utilization of the energy may simply be due to a longer time of exposure of the material to the grinding process. At higher speeds the optimum ball/particle diameter ratio, discussed in Chapter 3, may never be achieved due to the short grinding times. However, the longer grinding times associated with low speed grinding will allow the breakage

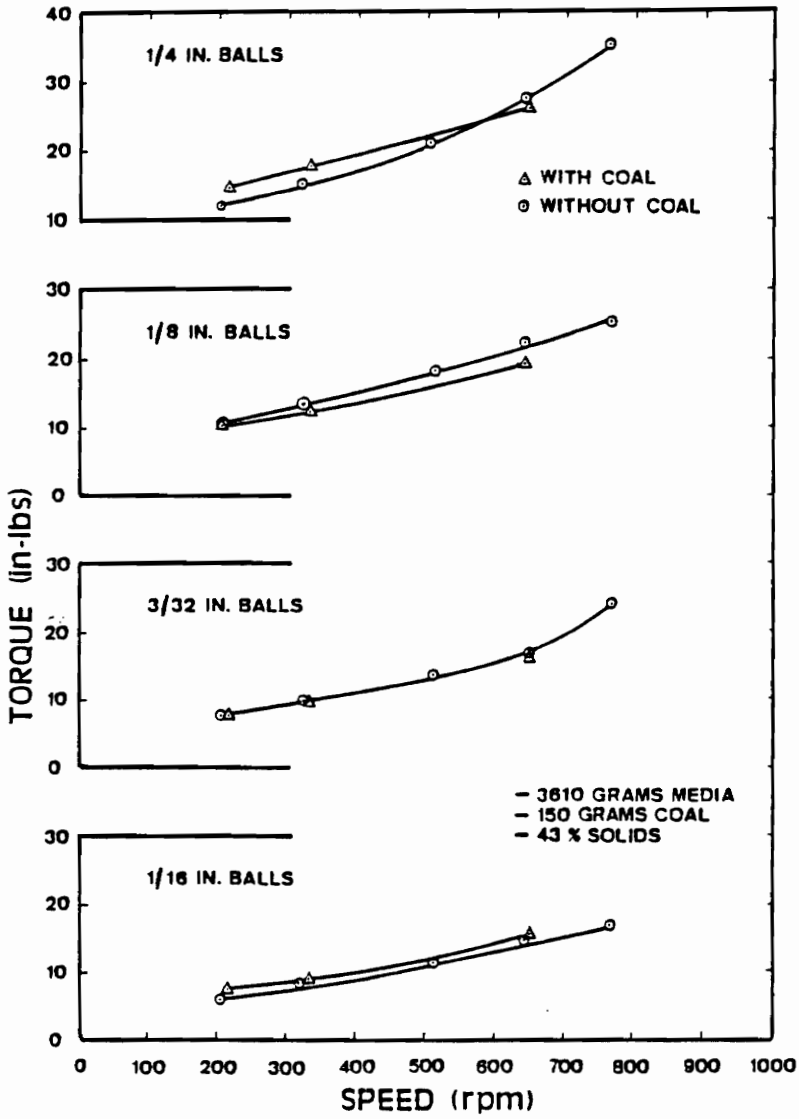

Figure 4.6. Effect of stirring speed on torque draw in attrition milling.

rate function to pass through its optimum value thus providing a more efficient use of energy.

Another explanation may be that a different type of breakage mechanism governs the grinding process as speed increases. As described in chapter 3, a gentle rolling action of the media occurs at lower speeds which appears to resemble thousands of tiny roll crushers. However, as stirring speed is increased this action tends to change into a more violent atmosphere of turbulent mixing in which the impact momentum of the balls may prove to be a more dominating factor than the roll crusher effect. Since impact breakage is less efficient than slow compression breakage, this would result in a more efficient use of energy (Schonert, 1985).

4.4.2.-Percent Solids

As with stirring speed, the torque also varies with a change in the mill percent solids. It can be seen from Figure 4.7 that an increase in percent solids from 20% to 60% raises the torque requirements of the mill nearly 2.5 times. This large increase creates a significant impact on the total energy input to the mill, again drastically decreasing the length of grind at equal energy inputs.

An increase in the percent solids seems to predominantly affect the viscosity of the slurry. As the

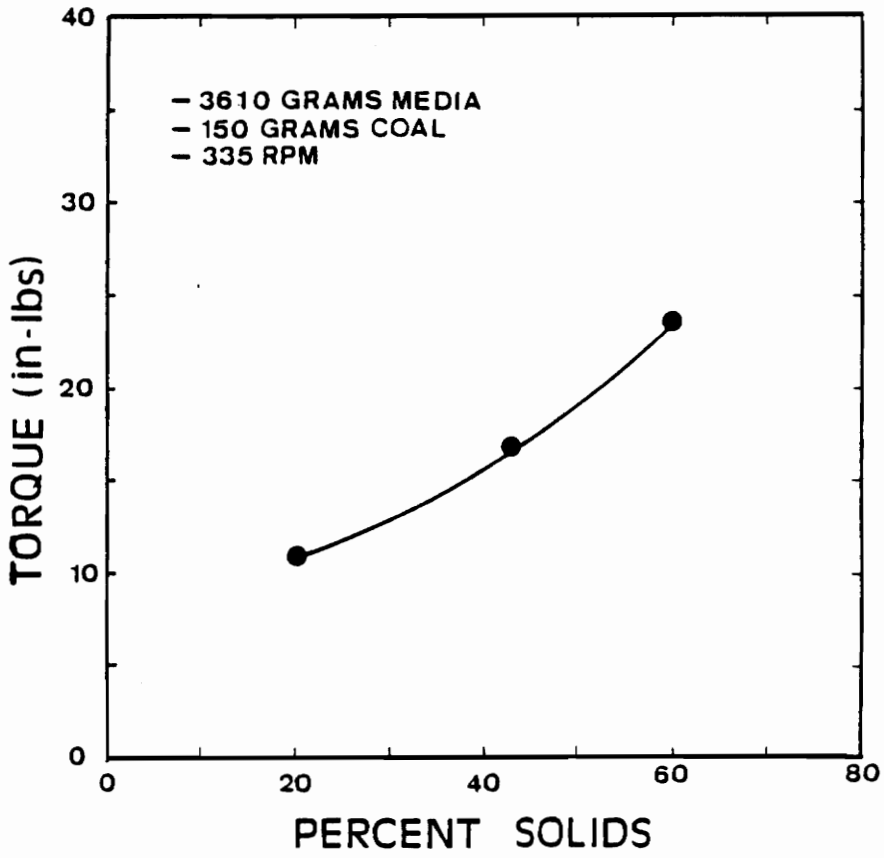

Figure 4.7. Effect of percent solids on torque draw in attrition milling.

slurry weight percent solids was increased from 20% to 50%, in the tests shown in Figures 4.3 and 4.4, the product from the mill remained quite fluid. However, the product from both of the 60% solids tests resembled a paste having no fluidity. Work performed by Davis (1986) indicates that the viscosity of coal water slurries produced with micronized coal increases sharply beyond a loading of 50% solids by weight. The increase in viscosity will tend to reduce the lubricating effect previously mentioned and may, in fact, contribute a resistance to the flow of grinding media. Tangsathitkulchai and Austin (1985) have also reported that an increase beyond 45% solids by volume (51% by weight) significantly reduces the first-order breakage rates for an Upper Kittanning coal.

The difference in the product size distribution for the 60% solids test was amplified at higher energy inputs. This is due to the fact that at higher percent solids the large increase in viscosity causes ball/particle aggregates to form which cling to the mill wall. The lower percent solids samples are unaffected by viscosity problems and will continue to grind. Once the material is attached to the mill wall, it is removed from the grinding process. This reduces the amount of active material in the mill and as a result the torque requirement decreases. This also provides an explanation for the broader product size distribution for

the 60% solids test. The large amount of coarse material in the distribution was a result of material being effectively removed from the grinding process due to adhesion to the mill walls.

Similar results were reported by Fuerstanau (1985) for tumbling ball mills. After a grinding time of 55 minutes the torque drawn by the mill decreased due to the adherance of media to the mill walls. This prevented the cataracting motion of the balls and produced a fly wheel effect on the mill which caused a corresponding reduction in the power draw. The addition of grinding aids eliminated this condition by reducing the viscosity of the slurry.

4.4.3.-Grinding Aids

As can be observed in Figure 4.8, the addition of grinding aids in stirred ball milling of coal can produce a reduction in the torque draw of up to 25%. This appears to be a result of a decrease in slurry viscosity, rather than a reduction of the surface free energy as put forth by the Rebinder theory (Rebinder, 1931; 1940). An interesting observation which can be made from these results is that the torque readings appear to be a crude indicator of reagent consumption. With each increase in reagent addition a region of constant torque input is extended. As grinding procedes the particle size in the mill is continually

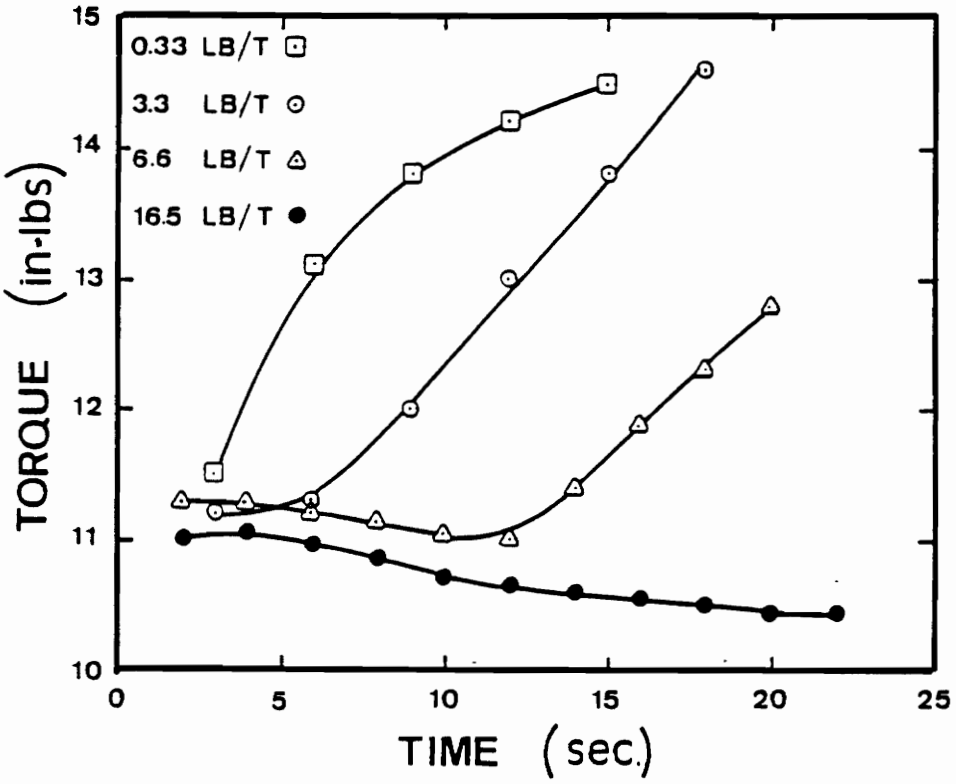

Figure 4.8. Effect of grinding aid addition on torque draw in attrition milling.

reduced requiring higher reagent additions in order to maintain the same viscosity. Beyond a certain critical size the reagents are no longer capable of maintaining this value and the torque readings begin to increase.

The results presented indicate that grinding aids can reduce the mean product size by nearly 50%, but the large reagent additions required may prove to be costly. A comparison should most likely be made between the economic benefits of the increased throughput associated with an increased feed percent solids and the additional cost of reagents.

4.5.-Summary and Conclusions

The results presented in this section were concerned with the optimization of several fundamental operating parameters in attrition grinding. It appears that proper adjustment of the operating conditions can achieve even lower energy requirements. A study of the effect of stirring speed, percent solids and grinding aid addition indicates the following:

1) A more efficient use of the energy consumed in stirred ball milling can be achieved through the use of slower stirring speeds.

2) A change from 20% to 50% solids by weight has no effect on the product size distribution. At higher values, however, adverse viscosity conditions inhibit grinding, producing coarser size distributions.

3) Grinding aids appear useful in maintaining lower viscosity levels which lead to a reduction in the final product size distribution.

The results obtained in this study indicate that mill operating conditions can be optimized to further reduce the energy input to the attrition grinding process. A combination of low stirring speeds, high percent solids and the possible use of grinding aids may provide the most

energy efficient method for producing micronized coal.

CHAPTER V

General Conclusions and Recommendations for Further Work

5.1.-General Conclusions

The results of the present investigation may be summarized as follows:

1. Material unaccounted for in size analysis was found to significantly affect the size distributions reported for fine grinding in a stirred ball mill. An existing procedure has been modified to aid in blending and mass balancing the data obtained from an Elzone 80XY particle size analyzer to account for this missed material.

2. The manipulation of particle size distribution data has been simplified by the development of numerous computer programs. These programs allow for the addition, subtraction, blending and mass balancing of size distribution data, as well as, a statistical error analysis for the distribution.

3. The ball to particle diameter ratio in the stirred ball milling of coal has been shown to significantly affect the grinding kinetics in the mill. The specific breakage rate is at an optimum when this ratio is 20:1. This optimum ratio has been related to the angle of nip concept used for roll crushers.

4. The energy consumption in stirred ball milling was found to be more efficient when using a combination of low stirring speeds and high percent solids. Stirring speed must, however, remain high enough to provide significant input energy to the media for grinding to occur. Percent solids are limited to a maximum of 50 to 55% solids by weight due to adverse viscosity conditions arising at higher values.

5. Grinding aids appear to be useful in providing lower viscosity levels which lead to an increased production of fines. The extremely fine particle sizes, however, dictate high dosages which may not be economical. The addition of dispersant type reagents might also hinder further processing steps such as flotation.

5.2.-Recommendations for Further Work

Based on the experience and information obtained in the present work, further work in defining the following areas are suggested:

1. By maintaining the ball/particle diameter ratio in the mill at 20:1 the specific energy rate is maximized. This suggests that as grinding continues the media size in the mill should be constantly reduced. This can be accomplished by stage grinding. This could also be achieved using a top fed continuous mill with a ball mixture. When stirring begins a stratification will take place forcing the larger balls to the top of the mill, due to a consolidation trickling mechanism. The thickness of each ball zone and the size of the media in that zone can be calculated in order to maximize the breakage rate throughout the duration of the grinding process. This will provide for the most efficient use of the input energy to the mill.

2. An investigation into the possibility of grinding to sub-micron sizes may now be possible through the use of ultrafine media. According to the optimum ratio given above, grinding 5 micron particles will require a media size of 100 microns to maintain an optimum breakage rate. This may be achieved by using fine size fractions

of sand or even coal. Preliminary results indicate that grinding with media finer than 1 millimeter can produce a product size distribution having a mean size less than 1 micron.

3. It was found that as media size is reduced, it tends to behave almost as a slurry itself and that pin type impellers do not provide sufficient agitation. Preliminary tests using blade type impellers similar to that used with Wemco flotation machines proved superior to the pin impeller resulting in a higher degree of mixing in the mill. An investigation into the different types of impellers available for mixing may lead to further reductions in energy.

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APPENDIX I

Sample Calculation of Specific Energy Input

The specific energy input for each grinding test was calculated according to the following equation:

$$E = \frac{2.978 \times 10^{-3} \times \text{Speed(RPM)} \times \text{Torque(in-lbs)} \times \text{Time(sec)}}{\text{Sample Weight (grams)}}$$

Torque readings were recorded at equal time increments and an average torque value calculated for each interval based on the assumption that torque varied linearly with time during that interval. From this value the energy input for each time step was calculated. The energy input was cumulated throughout the duration of the test until the desired value was reached. Calculated values for a test conducted at 330 RPM with 150 grams of sample are given in Table A.1.

The same data was plotted and the area under the curve determined by the use of a planimeter. This result was then substituted into the above equation. The total energy input according to this procedure is 93.6 kwh/ton. This closely agrees with the tabular result of 93.81 indicating that the procedure is valid.

Table A.1

Torque Data

time(min)	Discrete Torque (in-lbs)	Average Torque (in-lbs)	Discrete Energy (kwh/ton)	Cumulative Energy (kwh/ton)
0	10.9	0	0	0
2	11.3	11.1	8.72	8.72
4	11.7	11.5	9.04	17.77
6	12.3	12.0	9.43	27.2
8	12.7	12.5	9.83	37.03
10	13.0	12.85	10.10	47.13
12	13.3	13.15	10.34	57.47
14	13.6	13.45	10.57	68.04
16	13.8	13.7	10.77	78.81
18	13.9	13.85	10.89	89.70
18.75	14.0	13.95	4.11	93.81

APPENDIX II

Example of Charge Calculation

For a Batch Stirred Ball Mill

EXPERIMENT NO. 1

Solids: Elkhorn Seam Coal

Liquid: Water

Weight Percent Solids: 30%

PHYSICAL PROPERTIES

Specific Gravity of Coal: 1.3 g/cm^3

Specific Gravity of Liquid: 1.0 g/ml

Void Volume of Mill: 315 cm^3

MASS BALANCING EQUATIONS:

$$\text{Total Volume} = \frac{X}{1.3} + \frac{Y}{1.0} = 315$$

$$\text{Percent Solids} = \frac{X}{X + Y} = 0.43$$

SOLVING THE SYSTEM OF EQUATIONS YIELDS:

X = 150 Grams of coal

Y = 200 mililiters of water

Therefore a mill charge of 150 grams of coal and 200 milliliters of water will result in a 100% void fraction filling of the ball charge.

APPENDIX III

Application of Angle of Nip Theory to Different Ball Diameters

The concept of angle of nip derived for equal ball (roller) sizes can also be extended to include balls with different diameters. Figure 1 shows the case for two balls having diameters D_1 and D_2. A simplified diagram of the geometry involved for this situation is illustrated in Figure 2. This diagram allows us to develop several expressions based solely on the geometry of the relationship between balls.

From the law of sines we can immediately develop the following equation:

$$\text{SIN } \alpha \ (R_1+r) = \text{SIN } \beta \ (R_2+r) \qquad (1)$$

Based on the trigonometric formulas for an oblique triangle, the following relationships can be derived:

$$s = \frac{1}{2} (a+b+c) = \frac{1}{2} [(R_2+r) + (R_1+r) + (R_1+R_2)] \qquad (2)$$

α is then defined as:

$$\text{SIN } \alpha = \frac{2[s(s-a)(s-b)(s-c)]^{1/2}}{bc} \qquad (3)$$

Substituting into equation (3) and simplifying yields:

$$\text{SIN } \alpha = \frac{2[(r+R_1+R_2)(R_1)(R_2)(r)]^{1/2}}{(R_1+r)(R_1+R_2)} \tag{4}$$

This result can then be substituted into equation (1) and simplified to develop a similar expression for β.

$$\text{SIN } \beta = \frac{2[(r+R_1+R_2)(R_1)(R_2)(r)]^{1/2}}{(R_2+r)(R_1+R_2)} \tag{5}$$

Using equations (4) and (5) theorhetical curves were developed for the calculated coefficient of friction developed between the balls and a particle. These values were obtained by substituting the value for the controlling angle of nip into equation (6).

$$\text{TAN } \alpha = \mu \tag{6}$$

Since two angles are determined for each case, the angle which generates the highest coefficient of friction will govern the process. For this case it is the smallest ball which will require the largest coefficient of friction in order for grinding to occur. For this reason the coefficient calculated for the smallest of the two balls was plotted. Particle sizes from 1 to 1000 microns were plotted as a function of all possible combinations ball diameters ranging from 1 to 10 millimeters.

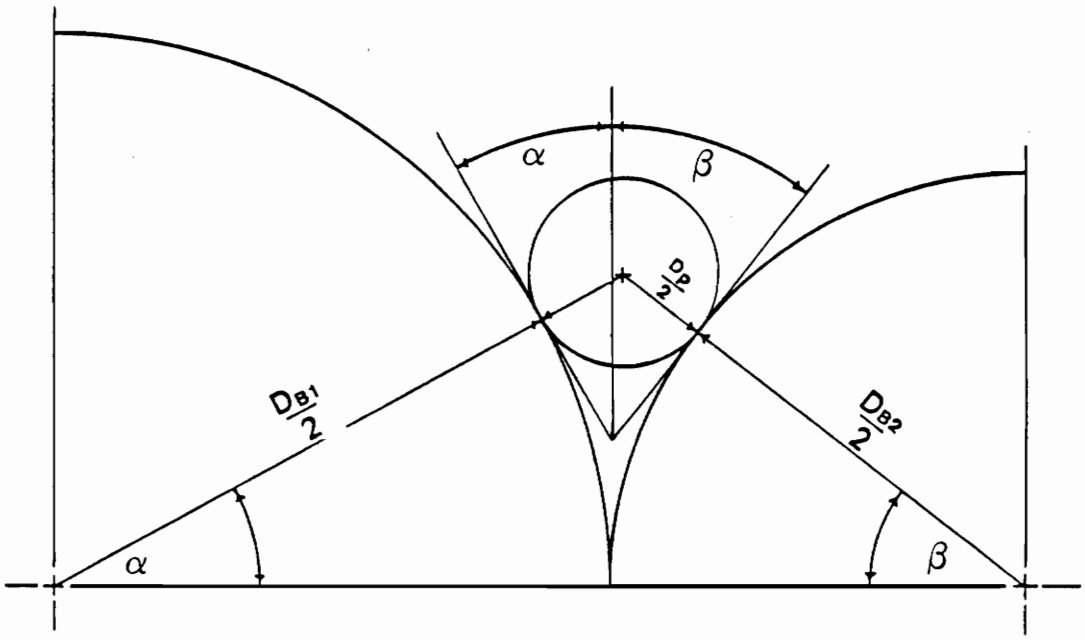

Figure 1. Schematic representation of the angle of nip between different size grinding media in a stirred ball mill.

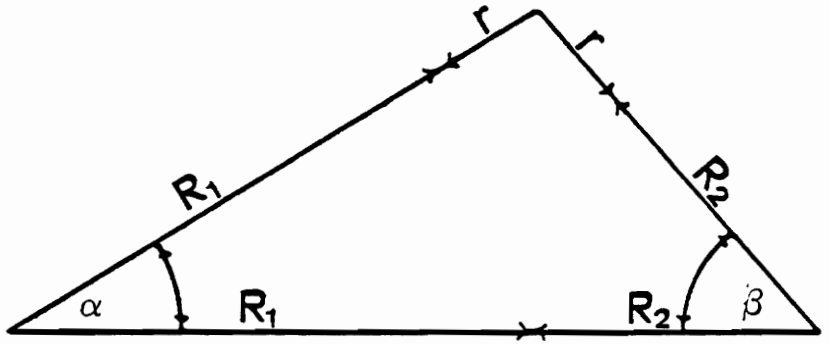

Figure 2. Simplified geometry for balls having different diameters.

As can be seen from Figure 3, the lowest required coefficent of friction occurs when the ball diameters are equal. This proved to be the case for all combinations. This tells us that the use of a ball mix does not provide any significant improvements in grinding. The larger balls in the mix will simply serve to grind the larger particles in the feed distributions which the small media cannot grind, as pointed out in chapter 2.

An interesting observation can be made as to the affect that an increase in the coefficient of friction between the media and particle would have. A plot of ball diameter versus particle diameter (Figure 4) shows us that small balls can be used to grind larger particles if the coefficient of friction can be increased. This would allow the use of smaller balls which are necessary for more energy efficient grinding of fine particles, as already pointed out in Chapter 3.

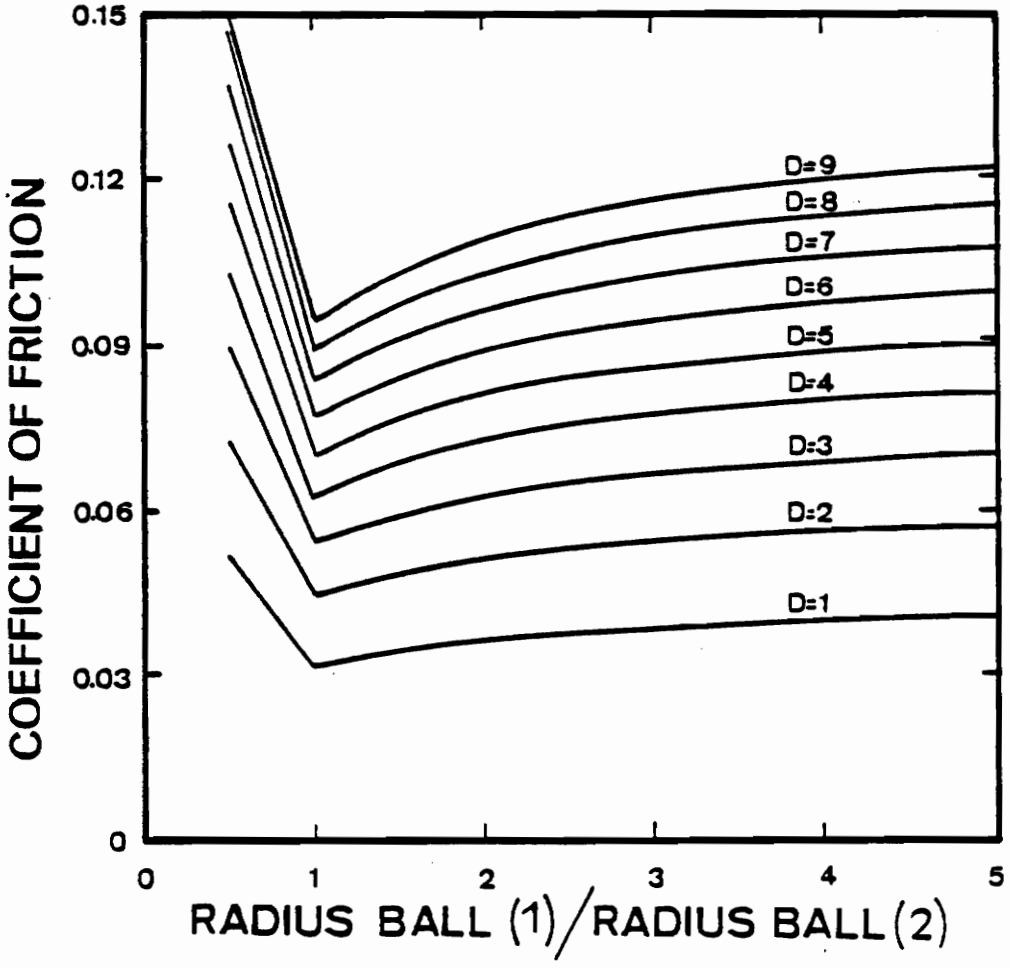

Figure 3. Calculated coefficient of friction required for grinding.

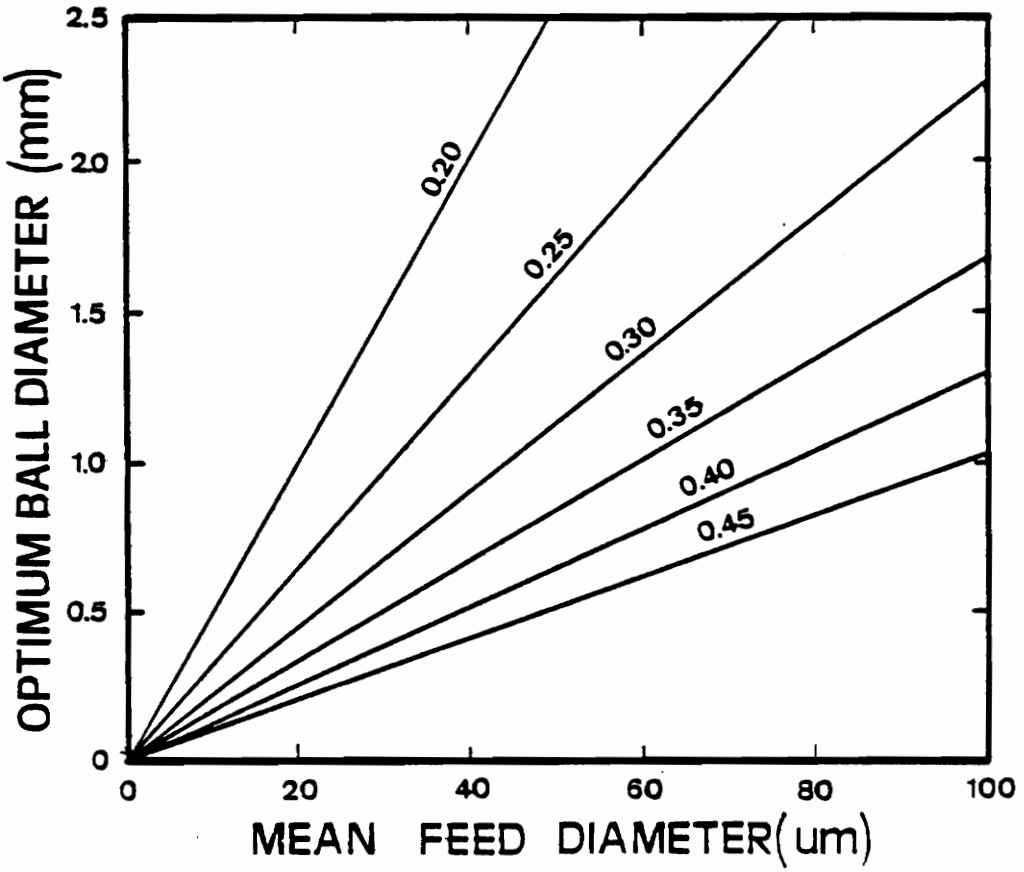

Figure 4. Calculated feed diameter versus optimum ball size for different values of coefficient of friction.

VITA

Michael J. Mankosa was born on December 20, 1960 in Weirton, West Virginia, where he lived until graduating from high school in 1979 in the top twenty of his class. During this time he was actively involved in student and civic affairs and received scoutings highest honor, eagle scout. He declined an appointment to the United States Military Academy in favor of enrolling in the engineering department at Virginia Polytechnic Institute and State University in the fall of 1979.

As a senior, he was active in the service fraternity of Pi Kappa Phi, the Virginia Tech Student Union, the Virginia Tech Jhoon Rhee Martial Arts Club, as well as, president of the Virginia Tech Judo Club and Secretary of the Student Chapter of AIME, The Burkhart Mining Society.

Michael enrolled as a full time graduate student in the summer of 1983 in order to pursue a Master of Science degree in Mining and Minerals Engineering. During this time he was inducted into the honorary society of Phi Kappa Phi. He plans to remain at Virginia Tech for his Ph. D. after completion of his Master of Science degree.

Michael J. Mankosa

OPTIMIZATION OF OPERATING CONDITIONS IN STIRRED BALL MILLING OF COAL

by

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Mining and Minerals Engineering

(ABSTRACT)

As a prerequisite to producing super-clean coal with any physical coal cleaning process, such as microbubble flotation, the feed coal must be micronized to liberate the finely disseminated mineral matter. The stirred ball mill is regarded as one of the most efficient devices for micronizing coal. Using a five-inch batch mill, the optimum operating conditions have been determined in terms of media size, feed size, media type, stirring speed and percent solids. The rates of breakage determined with monosized feeds are compared on the basis of specific energy consumption. It has been found that a 20:1 ball size/particle size ratio gives optimum grinding conditions.

With the fine particle sizes obtained using stirred ball milling, as well as other fine grinding techniques, a growing concern has been generated regarding the accuracy of these size distributions.

An automated technique has been developed in which a complete mass balanced size distribution can be obtained using an Elzone^{R} 80XY particle size analyzer. A computer program is used to blend the data from successively smaller orifice tubes, as well as, to determine the weight percent of material in a particle size distribution finer than the lower detection limit of the analyzer. This result is then used to correct the distribution for the missing fine material.

Experimental results indicate that size distributions obtained using this procedure are reproducible and compare favorably with those obtained using other size analysis techniques.