

Feb. 3, 1970

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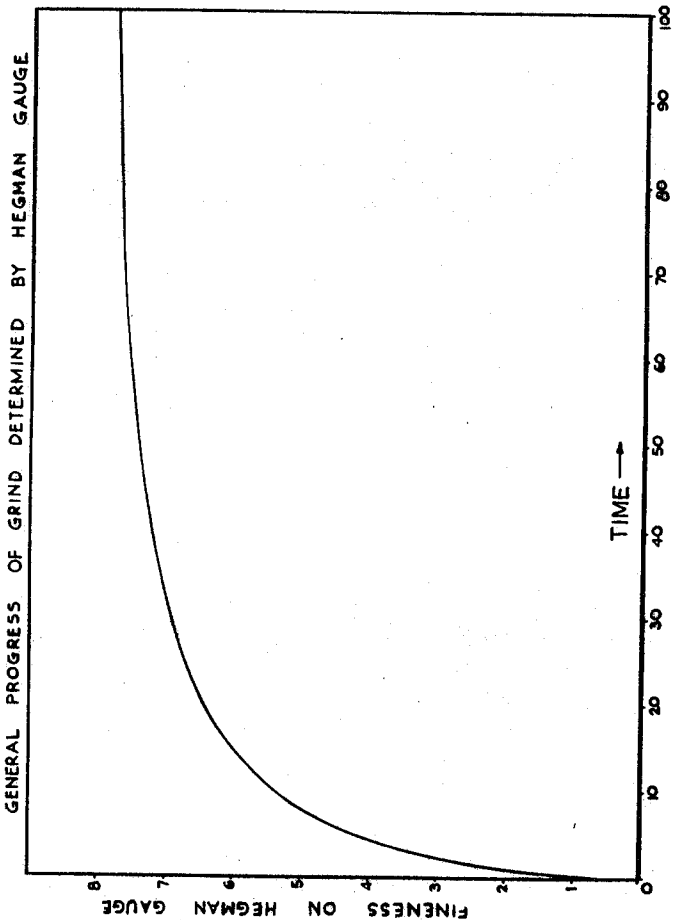
3,493,182

GRINDING AND APPARATUS THEREFOR

Filed Nov. 2, 1966

6 Sheets-Sheet 1

FIG. 1



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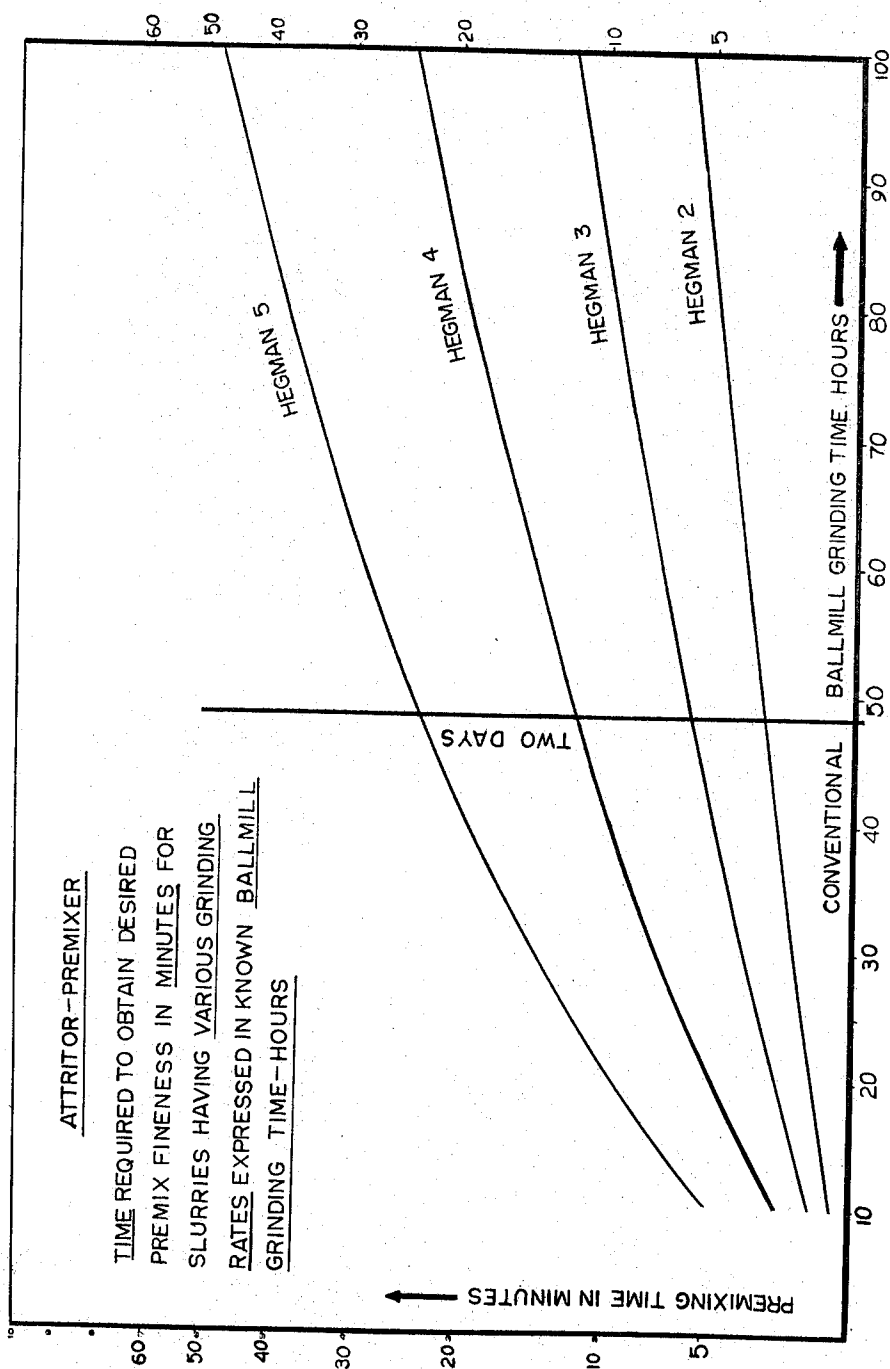
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FIG. 2



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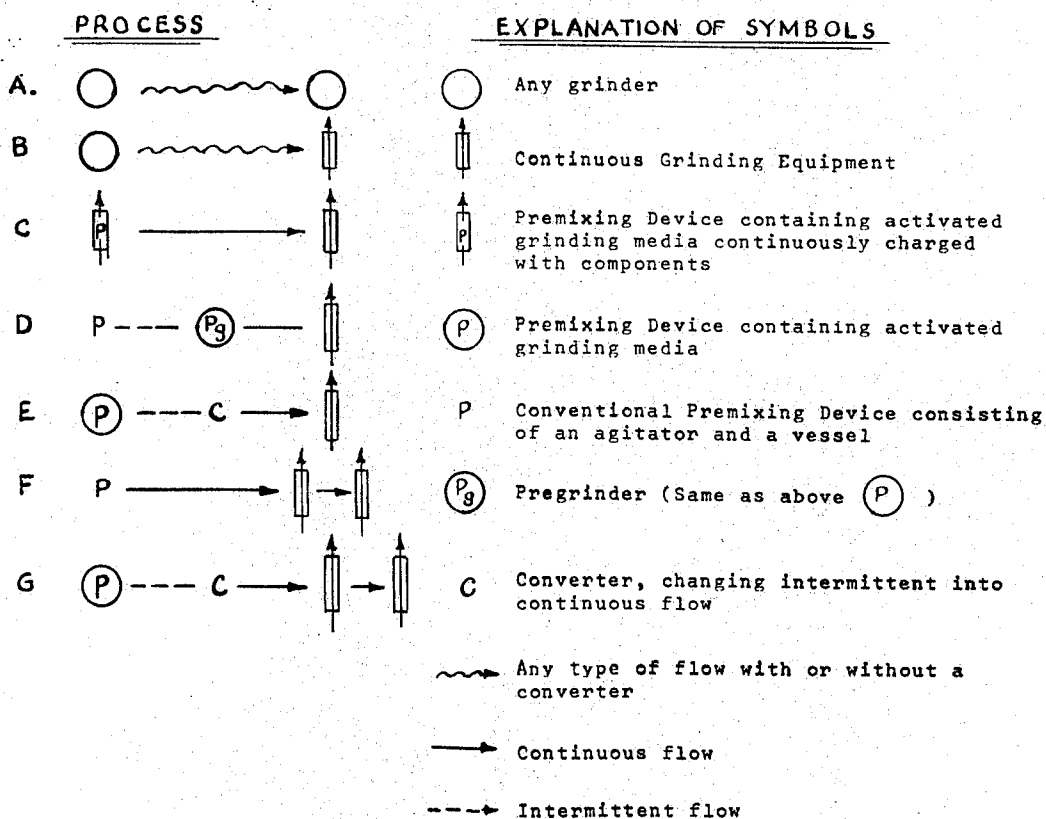
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FIG. 3

GRINDING OF SUSPENSION OF SOLID FEEDS USING FIRST LARGER THAN SMALLER GRINDING MEDIA



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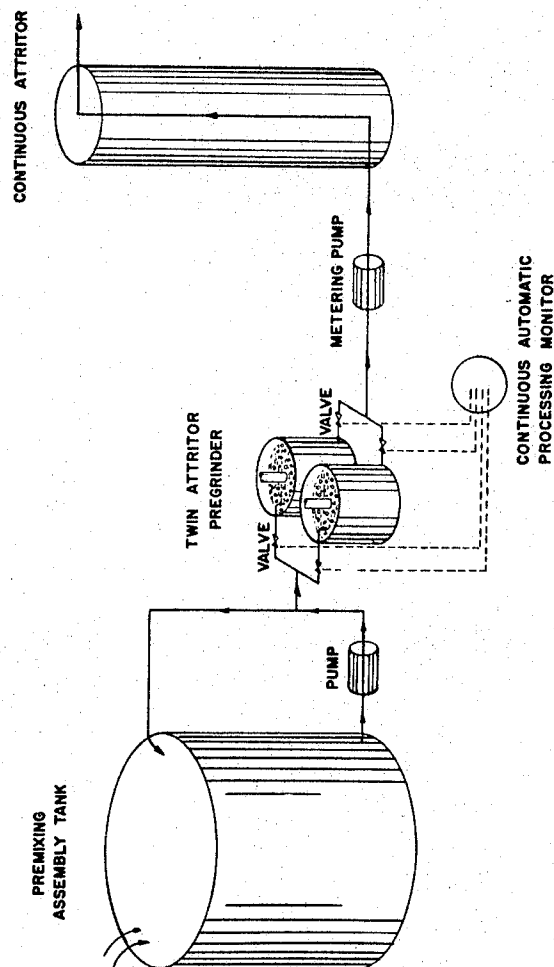
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FIG. 4

ATTRITOR PREMIXER INSTALLATION
AUTOMATIC CONTROL



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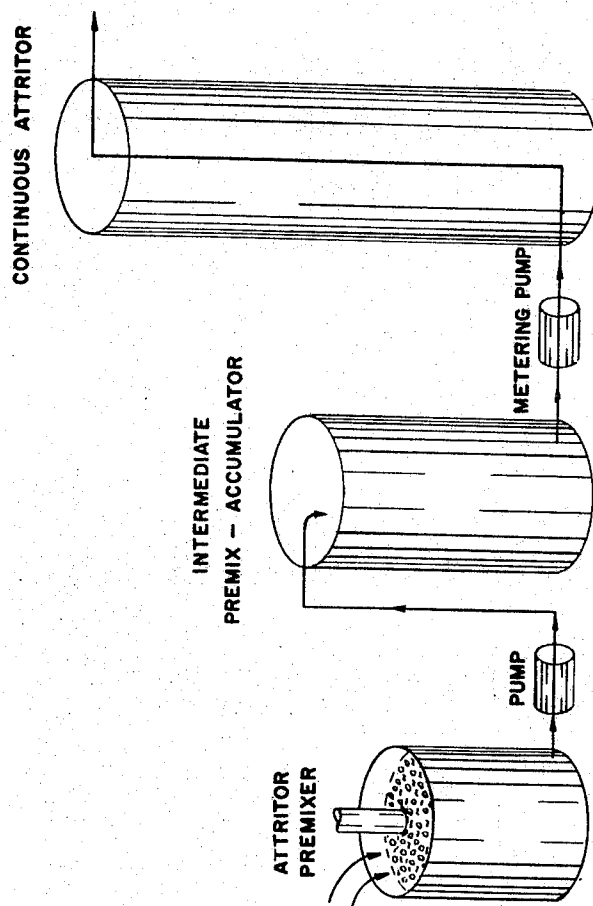
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FIG. 5

ATTRITOR PREMIXER INSTALLATION
MANUAL CONTROL



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FIG. 6a

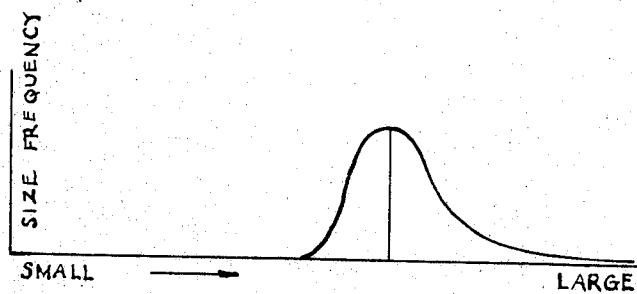


FIG. 6b

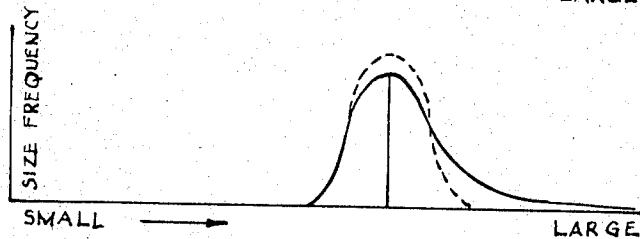


FIG. 6c

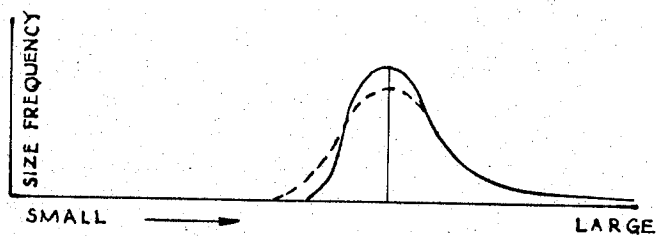
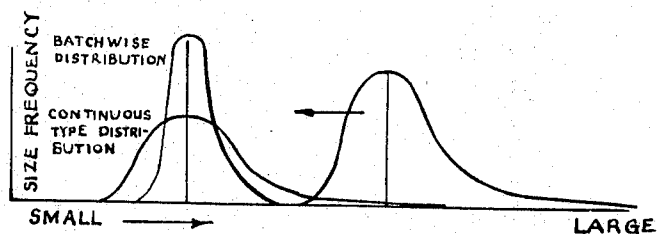


FIG. 6d



3,493,182

GRINDING AND APPARATUS THEREFOR

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Continuation-in-part of application Ser. No. 407,716,
Oct. 30, 1964. This application Nov. 2, 1966, Ser.
No. 591,459

Int. Cl. B02c 17/16

U.S. Cl. 241—15

9 Claims

ABSTRACT OF THE DISCLOSURE

A process for grinding in machines in which the grinding is effected by grinding media maintained in motion by agitating means. The grinding may be effected by (1) using the different effects of grinding on a batch basis and grinding on a continuous basis, or (2) by using grinding media of different sizes in a multi-step operation, or (3) both. When using grinding media of different sizes, larger grinding media are first used, and then smaller grinding media, in separate vessels. In a preferred operation larger grinding media are used in a batch operation, followed by smaller grinding media in a continuous operation.

This application is a continuation-in-part of my abandoned application Ser. No. 407,716 filed Oct. 30, 1964. The invention as there disclosed is also disclosed in my pending application Ser. No. 534,934 filed Jan. 19, 1966.

The invention relates to the grinding of solids suspended in a pumpable dispersion by contact with grinding media which are kinetically activated by agitating means. The grinding media may be balls of different sizes as is known in the art, or pebbles of French flint, as disclosed in Szegvari U.S. 2,764,359 and 3,149,789 which describe apparatus known in the industry as Attritors in which a plurality of grinding media are activated by an internal agitator; or the grinding media may be smaller particles such as the sand or the like employed in the so-called "sand mills," such as disclosed in Hochberg U.S. 2,581,414 and 2,855,156, for example.

The advantages of the process of this invention are best understood against a background of the following facts:

(1) The operation involving the diminution starts with the combining of solids to be diminished with liquids. This initial processing step is usually referred to as pre-mixing and takes place in vessels equipped with a mechanically driven agitator in the absence of grinding media. The acting forces are derived from liquid shear. The nature of liquid shear is such that it is mechanically limited, that is, in a given apparatus its action is limited to a certain amount of dynes per square centimeters of acting area which cannot be exceeded. As a result, the action or liquid shear is limited regardless of duration.

(2) The mechanical action of a system of contacting balls is such that the forces available at the contact points could exceed practically all limitations reigning in an apparatus of conventional size. Therefore, the action of such a system on diminution is controlled primarily and only by statistical fact as a function of time.

(3) The statistical effect of interaction between a system of activated grinding media and the feed to be diminished depends first on the size of the interacting grinding media, and second, on the size relationship of these grinding media and the material to be diminished, and third, on whether the interaction is carried out on a batch or continuous basis.

(4) The end result in an industrial diminution process such as described is that the resulting particles have a size range of qualitative characteristics which is usually re-

ferred to as a distribution curve. In most instances the industrially required property of this distribution curve is that it should be "narrow"—that is, the occurring particle size range should be within the narrowest possible limits.

The control of the distribution curve is described in the following:

The "width" or range of the distribution curve is narrower in batch type operations (FIGURE 6d) and much wider, or flatter, in continuous operations because the probability of something happening, which is different from the average, is much smaller in the closed circuit batch operation, where there is chance processwise of each cubic inch being like every other cubic inch, which is nothing else but the condition to have a "narrow" or sharp distribution curve. The processing draw-back of this statistical procedure is that it takes time; much longer than necessary to obtain the flatter distribution curves of a continuous statistical process.

Producing the largest amount of fine particles, or in statistical terms, the maximum area under the fine end of the distribution curve (FIGURE 6c): This is obtained with small or smallest grinding media, high agitator shaft speeds and continuous grinding equipment. The cause is the statistical probability of contacts between small grinding media being much larger than between large grinding media, and obviously in order to produce a large number of fine particles we have to have the largest number of contacts.

Elimination of the largest or coarsest particles: That is reduction of the area of the distribution curve under the large end and in addition terminating it (FIGURE 2), or bringing it to practically zero at the required size. This can be most effectively done with large or larger grinding media and in a batch type operation easier than in a continuous operation.

Other factors influencing the distribution curve: The most important is the relationship of the size of the grinding media to the size of the suspension to be processed, with the following results:

(A) The action of large grinding medium on a coarse feed would eliminate the large particles without producing fines.

(B) A large grinding medium on a fine feed would have no required effect on the absent large particles; on the other hand, it does not have any statistical chance to produce fineness effectively.

(C) A small grinding medium on a coarse feed has very little statistical chance to eliminate the large particles but it will effectively make small particles from the small fraction, thus resulting in a very wide distribution curve.

(D) A small grinding medium on a fine feed will effectively produce the largest amount of small particles.

(5) It is impractical to combine large and small grinding media in the same grinding tank. There are two causes for this: One cause is that the over-riding momentum of the larger grinding media prevents the activity of the smaller grinding media; and the second cause is that larger grinding media segregate from the smaller grinding media in a mechanically undesired way.

(6) It is a significant fact that because of statistical reasons the rate of diminution obtained as a result of the interaction between a system of activated grinding media and the feed is such that it proceeds first much faster—in fact, at a surprisingly fast rate—and this rate slows down more and more during the subsequent processing.

(7) There is a significant difference in the statistical procedure of the diminution between (1) the case where diminution is effected in a closed circuit—that is, in a batch operation and (2) a continuously progressing "through flow" or "block flow" type of continuous opera-

tion. In the first case the condition obtained, i.e. the diminution of the particles, is continuously equalized so that the size of the particles is the same throughout the vessel at any given time, while in the second case the diminution progresses from one end of the vessel or operation to the other, and the size of the particles is continuously being diminished without any diminished condition being equalized with a previous state.

(8) The consequence of the facts set forth in No. 3 above, and part of No. 7 above, is that the best statistical progress in diminution by a system of activated grinding media is obtained by using small grinding media interacting with a feed which is ground to a size as small or fine as possible. Particularly, this is done in continuous processing machines requiring very fine feed. The above results from the use of apparatus which accomplishes two things: (a) a very good "pre-mix" is provided, and (b) the process is carried out in series fashion—that is, the diminution result initiated in a first unit is continued in a second unit containing smaller grinding media, resulting in a "series" type of processing. By "pre-mix" is meant the mixing of the solids and liquid previous to feeding them into the continuous diminishing apparatus. Some diminution may occur during the pre-mixing. While pre-mixing is essential for continuous diminution units, this is not the case with batch operations where the solids and liquid can be fed without "pre-mixing."

(9) The mechanical or, more precisely, the kinetic behavior of agitating grinding media and their interaction with the feed to be diminished is different with large media from what it is with small media. Because of this difference in behavior, the large media perform best in batch type operations and cannot be used satisfactorily in continuous apparatus, while small media perform best in continuous apparatus, which applies particularly to operations in which the suspension of the feed rises vertically.

(10) The invention relates to fine grinding—the grinding of pumpable dispersions which is quite different from coarse grinding. Fine grinding is applied to pigments for paints and inks, drugs, ferrites, etc. The industrial purposes and controlling factors of fine grinding are entirely different from those of coarse grinding. The only purpose of coarse grinding is diminution. The purpose of fine grinding is to obtain certain definite and sensitive properties of the end product not otherwise obtainable. These are reflected in the distribution curve.

The invention in its various adaptations utilizes one or more of the following novel principles:

(A) Advantages are gained by the superimposing of consecutively applied diminution process based on activated grinding media yielding different size distribution statistics, such as the following:

- (1) The statistics of a batch-type diminution operation;
- (2) The statistics of a continuous diminution operation;
- (3) The statistics prevailing when using large grinding media in relationship to the feed.
- (4) The statistics prevailing when using small grinding media in relationship to the feed.
- (5) The interrelated statistics when using consecutive units in case of continuous operations.

(B) Advantages of extending the use of activated grinding media to the pre-mixing operations and thereby superimposing the distribution obtained on the distribution of subsequent grinding operations.

(C) Advantages of combining the operation involving batchwise processing with subsequent continuous operation by means of a converter; by converter is meant apparatus to convert pulsating flow into continuous flow.

The accompanying drawings explain and illustrate the invention. In these drawings:

FIGURE 1 is a distribution curve in which fineness measured in Hegman gauge is plotted on the vertical axis and time is plotted on the horizontal axis. (The

Hegman gauge is used commercially in the paint and other industries, and ranges from Hegman 0 to Hegman 8. The larger numbers indicate greater fineness.)

FIGURE 2 is a family of graphs, with the vertical axis measuring pre-mixing time in minutes required to arrive at a certain Hegman fineness (using activated grinding media), and the horizontal axis indicating the "grindability" of the particular slurry in some practical units thus indicating the "grindability" of the particular dispersion in a practical yardstick. For such a yardstick we use the ordinarily well-known grinding time in conventional ball mills, expressed in hours.

FIGURE 3 is a flow sheet of various processing operations which are within the scope of the invention;

FIGURE 4 is a representation, largely schematic, of apparatus for carrying out Process D of FIGURE 3;

FIGURE 5 is a representation, largely schematic, of apparatus for carrying out Process E of FIGURE 3; and

FIGURES 6a to 6d are distribution curves. FIGURE 6a is a representative distribution curve of the particle sizes of a solid powder. FIGURE 6b shows in dotted lines the effect of diminution using relatively large grinding media (the solid line showing is the original FIGURE 6a). FIGURE 6c shows in solid lines the effect of diminution with relatively small grinding media, the solid line showing is the original curve of FIGURE 6a. FIGURE 6d shows the curve of FIGURE 6a to the right, and to the left shows the effect of a batch and a continuous operation carried out to the same particle size.

FIGURE 1 shows the fineness expressed in Hegman gauge grades, as a grinding progresses, which applies generally to the effect of activated grinding media on solids in liquids. The horizontal axis is scaled so as to indicate full grinding time of "100," regardless of how long the grinding continues. This applies to all materials and equipment. The percentage means simply the percentage of the full grinding time expressed in the same time scale. The graph shows the extremely rapid rise in the amount of fine particles obtained during the first few percent of the elapsed total grinding time.

FIGURE 2 indicates that when pre-mixing in a batch type pre-mixer, containing activated grinding media such as described for use in Process D below, one can obtain 3 to 5 Hegman grade fineness in a relatively few minutes, mostly less than 10 minutes, with any slurry with usual average "grindability," such as finished in a conventional ball mill within two days.

FIGURE 3 shows a series of operations schematically in each of which a slurry of solid material in a liquid is subjected to two or more grinding steps by activated grinding media, in the first of which larger grinding media are employed and in the second of which smaller grinding media are employed.

All of the materials mentioned in the following examples are solids the physical properties of which remain the same throughout the respective grinding operations except for the change in size.

Process A: This is a general process illustrated schematically, in which any two types of grinding equipment containing activated grinding media are used with the grinding media in the first vessel or larger size than those in the second vessel. The suspension produced in the first vessel is conveyed continuously or intermittently to the second vessel. There may or may not be some type of converter to which the slurry is subjected between the operations carried out in the two grinding vessels.

The processes following Process A are selected as presently appearing to have the greatest commercial possibilities. Processes C and D are particularly adapted for use in the fine grinding of pigments for paints, inks, etc., and other processes may be particularly adapted for the grinding of other solids including drugs and inorganic materials.

Process B: Process using a pre-mixing device containing activated grinding media which discharges into con-

tinuous grinding equipment, with or without an intermediate converter.

Process C: Process using a pre-mixing device containing activated grinding media which is continuously charged with metered amounts of solid and liquid. The pre-mixer is discharged continuously into continuous grinding equipment.

Process D: Solid and liquid components of a slurry are combined conventionally and fed into a pre-grinding device containing activated grinding media and by way of the instrumentality of a converting mechanism feeding into a continuously grinding unit. An example for this process is the large-scale production of the so-called "white base" of the paint industry consisting essentially of a titanium dioxide pigment suspended in the properly formulated slurry. In this operation the pigment, which may be of a size of about 325 mesh, will, for example, be fed once a day with the suspending medium to a pre-mixer 1 which may be a 600-gallon tank. This will be circulated by the pump 2 through the tank 1 and part of this will be directed through the action of an automatic monitor 3, intermittently, to the first or the second of the twin pre-grinders 4 and 5 containing activated grinding media of a size between $\frac{3}{16}$ to $\frac{3}{8}$ inch. These pre-grinders may each have a capacity of about 40 gallons and will grind the pigment to an average Hegman gauge of 4 to 5. The finished pre-mix is metered automatically and continuously by way of the controlling monitor 3 and pump 6 into the continuous grinding unit 7 which contains grinding media in the size range between $\frac{1}{64}$ to $\frac{1}{8}$ inch. This unit 7 may be of the type illustrated and described in Szegvari U.S. 3,149,789. The solids are ground to an average Hegman fineness greater than 7.

Process E: The solid and liquid components of the slurry are charged into the pre-mixer 10 containing activated grinding media, and pumped intermittently by pump 11 into an intermediate pre-mix accumulator 12. The size of this accumulator is such that it can provide continuous feeding through a metering pump 13 to the continuous grinding device 14, requiring at least two overlapping periods of the pre-mixer 10. The device 14 may be of the type described in Szegvari U.S. 3,149,789. The pre-mixer 10 may be, for example, of 40-gallon capacity, and using balls $\frac{3}{8}$ inch in diameter will produce a pre-mix of a size of about Hegman gauge $2\frac{1}{2}$ to $4\frac{1}{2}$ every 10 minutes. In the continuous grinding device 14, smaller grinding media will be used in the range of $\frac{1}{64}$ inch to $\frac{1}{8}$ inch and the processed slurry will be ground to a Hegman gauge of $7\frac{1}{2}$ to 8.

Process F: Slurry pre-mixed in any type of pre-mixing device is metered into a continuous grinding device from which a product issues into a second continuous grinding device. These continuous grinding devices contain grinding media in the range between $\frac{1}{8}$ inch and $\frac{1}{64}$ inch; the first grinding device having grinding media in the range between $\frac{1}{8}$ inch and $\frac{3}{32}$ inch and the second continuous grinding device having grinding media in the range between $\frac{3}{32}$ inch and $\frac{1}{16}$ inch. The first and second grinding devices may have additional differences, namely, the r.p.m. of the first grinding device may be between the range of 100 and 600 r.p.m., while in the second grinding device, the r.p.m. may be in the range between 150 and 1000 r.p.m. Apparatus such as described in Szegvari U.S. 3,149,789 may be used for for each of these continuous grinding units. This process is adapted for the fine grinding of powders, drugs, etc. to an average particle size in the range of 1 to 2 microns.

Process G: This process is similar to Process F, with the exception that the components of the liquid slurry are charged into a pre-mixing device containing activated grinding media, which device completes the pre-mix in requested time intervals; and the slurry is passed through a converter "C" by continuous flow into the continuous grinding device. Such apparatus is used in the processing of ferrites, starting with a material between 200 and 325

mesh and finishing at the average particle size of 1 micron. Although various types of pre-mixers may be used in Processes B, C, E and G, a preferred type of pre-mixer contains activated grinding media. It may be such a pre-mixer as is described in the following and known as an Attritor pre-mixer. In many respects this pre-mixer resembles the grinding device disclosed in Szegvari U.S. 2,764,359 but generally it will not contain as many grinding media as used in a commercial grinding operation. The grinding media will usually be of a size of at least between $\frac{1}{4}$ inch and $\frac{3}{8}$ inch. The top of the tank will conveniently be equipped with an apron to facilitate manual loading with solid and liquid feed material and it will be equipped with a discharge conduit suitable for continuous or intermittent discharge of the slurry to a flow converter or a continuous grinder or other equipment as required. Discharge opening is provided for the discharge of the pre-mixed slurry and the grinding media. Exposed parts of the drive are protected from contamination by ingredients floating in the air as a result of the loading process. All apparatus details which serve the purpose of finishing the grinding to ultimate fineness, such as cooling means and specific tank handling equipment are omitted.

In the processes described, a "converter" is used for the purpose of changing intermittent discharge from the pre-mixing devices containing activated grinding media into a continuous flow to feed continuous grinding apparatus.

FIGURE 5 indicates the use of an intermediate pre-mix flow-converter, the regulation of which is manually controlled, following the preferred type of pre-mixer, an Attritor pre-mixer, just described.

FIGURE 4 shows an apparatus where a large quantity of solid and liquid, for instance, the entire supply used in one shift, is combined in a container of sufficient size; the size of the container might be between 200 and 800 gallons. These containers are equipped with a conventional agitator to assist in the distribution of solids in the liquids and to keep the distributed solids from excessive sedimentation. No grinding media are used. The control of the flow which involves charging the twin pre-mixers containing activated grinding media, and the subsequent intermittent discharging of each of these pre-mixers into the continuous grinding equipment, is controlled by an electric monitoring device which acts on the four valves to control the intermittent charging and the discharging of the twin system. The controls of the monitor are effected by conventional timing apparatus to control the fineness to which the particles are ground in the separate grinding operations and thus make the process selective and conventional level control apparatus.

The invention is disclosed in the claims which follow.

I claim:

1. The process of grinding solid particles in a pumpable dispersion in two steps and thereby selectively statistically altering the distribution curve of the resulting particles of the solid, the physical characteristics of the particles remaining the same throughout the grinding operation, except for the change in size, which process comprises first grinding the solid particles in the dispersion by a plurality of larger grinding media kinetically activated in the dispersion by an internal agitator for a preselected time and thus foreshortening the portion of the curve pertaining to larger particles of the solid, and thereafter grinding the resulting solid particles in the resulting dispersion by a plurality of smaller grinding media kinetically activated by an internal agitator and thus increasing the portion of the curve pertaining to the smaller particles of the solid and obtaining a selectively shaped distributing curve.

2. The process of claim 1 in which the first step the solid is ground to an average Hegman gauge fineness of less than 5 and in the second step the solid is ground to an average Hegman gauge fineness of at least 7.

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3. The process of claim 1 which process comprises first grinding the solid by the action of activated grinding media in the dispersion in a batch operation and then grinding the resulting solid particles by the action of activated grinding media in the resulting dispersion in a continuous process.

4. The process of claim 1 in which both diminutions are carried out on a continuous basis.

5. The process of claim 1 in which the primary diminution is carried out on a discontinuous basis and the subsequent diminution is carried out on a continuous basis.

6. The process of claim 5 in which the solids and liquids are separately metered to the apparatus in which the primary diminution takes place.

7. The process of claim 5 in which two primary diminution processes are carried out in parallel and the resulting dispersions are discharged intermittently and combined to a continuous stream and diminished continuously.

8. The process of claim 5 in which the dispersion of the primary diminution is discharged intermittently to a converter where it is accumulated and discharged therefrom continuously for the subsequent diminution which is carried out on a continuous basis.

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9. The process of claim 5 in which the solid resulting from the primary diminution has an average Hegman gauge less than 5 and the product resulting from the subsequent distribution is finer on the average than Hegman gauge 7.

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DONALD G. KELLY, Assistant Examiner

U.S. Cl. X.R.

241—29, 30

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,493,182 Dated February 3, 1970

Inventor(s) Andrew Szegvari

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 6, line 71, "distributing" should read --
distribution --; line 72, after "which" insert -- in --.

Signed and sealed this 2nd day of May 1972.

(SEAL)
Attest:

EDWARD M. FLETCHER, JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents