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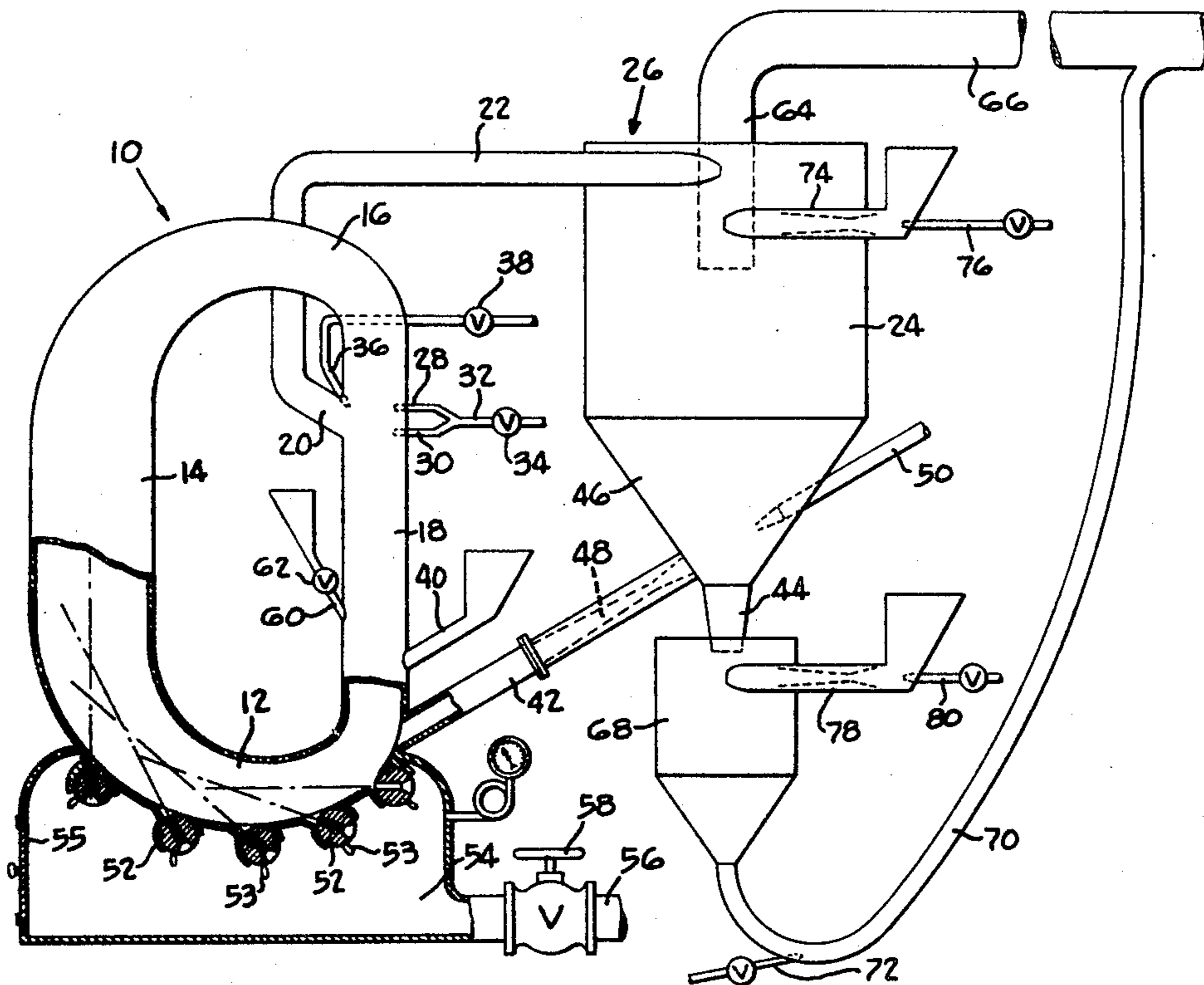
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[54] **TREATMENT OF GRANULAR SOLIDS BY FLUID ENERGY MILLS**

5 Claims, 1 Drawing Fig.

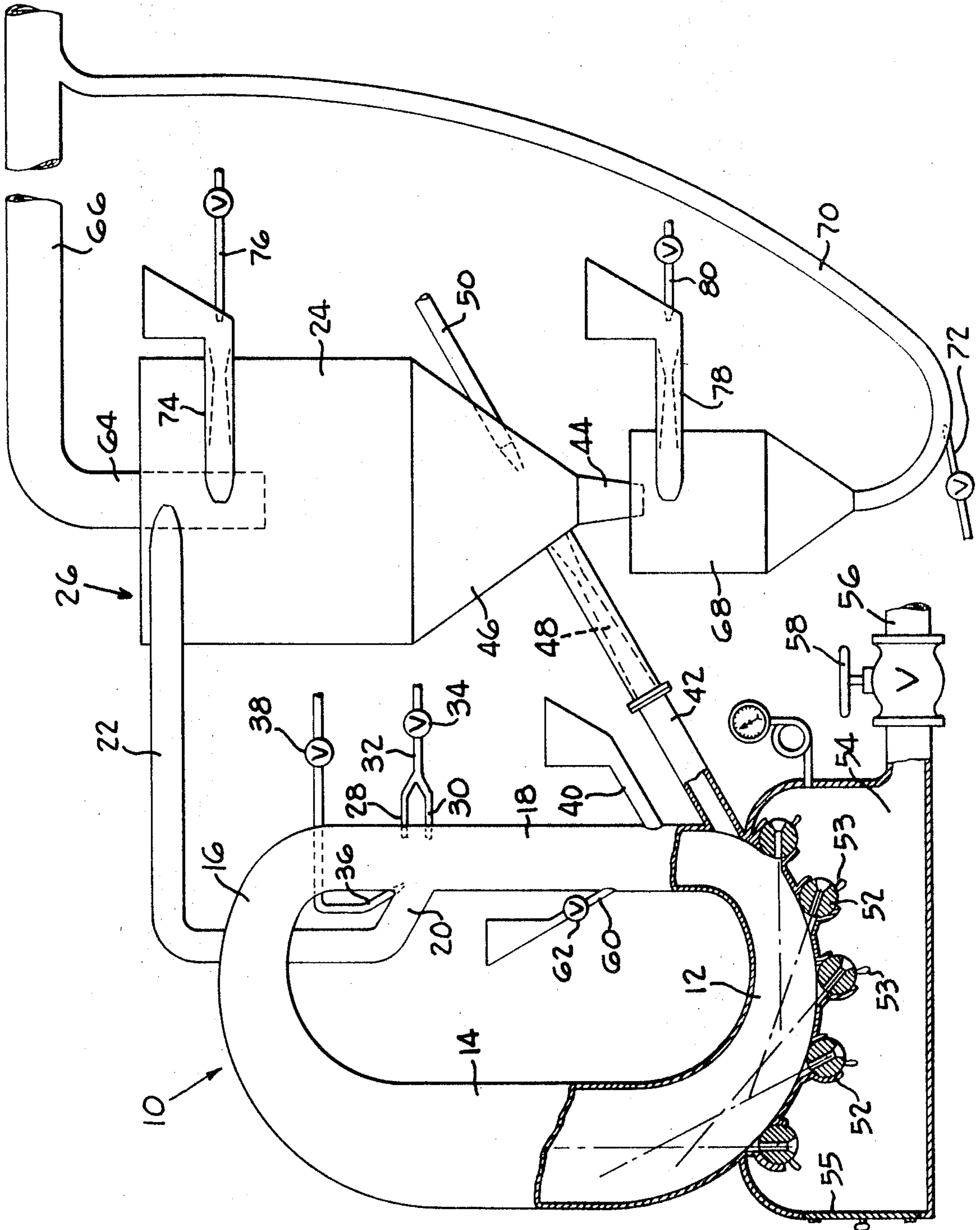
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ABSTRACT: A method of treating solid particles by propelling jets of elastic fluid, such as gas or vapor, in selected lateral directions against a circulating vortex of particles entrained in similar elastic fluid to selectively propel a larger proportion of the particles toward or away from an outlet positioned adjacent the inner peripheral portion of the vortex.



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TREATMENT OF GRANULAR SOLIDS BY FLUID ENERGY MILLS

This is a division of copending application Ser. No. 607,974, filed Jan. 9, 1967 now issued as U.S. Pat. No. 3,491,953, dated Jan. 27, 1970.

This invention relates to the treatment of solid granular materials by subjecting them to the action of moving gases or vapors in an enclosed mill, referred to generally as a fluid energy mill, and it particularly relates to the control of such gases or vapors to selectively vary the size, shape and other characteristics of the particles of such materials.

The so-called fluid energy mills have various inherent characteristics. For example, they have no moving parts in the actual treating zones. Therefore, when they are used for grinding or pulverizing, only the material is in motion and the grinding is effected by impacts between the particles themselves and between the particles and the walls of the mill. There is, therefore, no overheating due to the friction of moving parts. In fact, the finished product is often cooler than the material fed into the mill, or than the gas or vapor (hereafter referred to as the elastic fluid) used as the energizing medium. One reason is that the elastic fluid expands as it is expelled from the expanding gas nozzle, there is an exothermic Joule-Thompson effect, and the impacts between the particles generate less heat than the cooling due to the fluid expansion. This is the usual effect with most elastic fluids such as air, oxygen, nitrogen, steam and water vapor, carbon dioxide, etc. Hydrogen appears to be an exception to the rule because the inversion of the Joule-Thompson effect occurs at -112° F., and any flow through the nozzle at temperatures above this point produces heating. The treatment, whether it be grinding, chemical reaction, coating or merely dispersing of adherent particles, can be carefully controlled by using different fluids, or combinations of fluids, to effect a desired degree of cooling or heating. Inert gases are preferably used to prevent chemical reactions, coating deposits, etc., during grinding or dispersing of the particles. Selected fluids are, on the other hand, utilized at controlled temperatures to obtain hydration, dehydration, oxidation, mixing, blending, coating, or any other desired chemical or physical reaction.

The expansion effect of the elastic fluids in fluid energy mills also makes them extremely well adapted for injection mold processes. In this respect, many natural and synthetic plastics and resins are extremely hygroscopic even at room temperature. In the usual injection molding process, the materials must, therefore, be preheated to expel the moisture prior to introducing them into the injection feeder. This not only necessitates an extra step involving additional apparatus, time and labor, but may sometimes deleteriously affect the materials themselves. However, when fluid energy mills are used to prepare the materials for injection molding, most of the moisture is expelled due to the low relative humidity of the expanded gases and to the fact that the finely ground particles have so much of their surfaces exposed that the adherent moisture usually found in the interstices between the particles is open to the action of the expanding gases. For example, if air is compressed on a warm summer day at 80° F., and 90 percent relative humidity, a pound of this air will contain about 140 grains of water. But if this air is compressed to 100 p.s.i.g. pressure, and cooled to 70° F., then one pound of the air can hold only 14 grains of water, while 126 grains, or 90 percent of the moisture, appears as water and can be removed before entering the mill. Now, when the air is expanded in the mill to approximately atmospheric pressure, it will still have only 14 grains of water per pound, but at atmospheric pressure it will have only 13.5 percent relative humidity. This extremely dry air will not deposit any moisture unless it is cooled below 20° F. As a result, the resin or other material will be quite dry and will not produce any blistering such as so often occurs in injection molding processes where too much moisture is present in the material. It is to be noted that since the mill is an enclosed system, no outside moisture or dirt can contaminate the finished product.

An important consideration in ground or pulverized particles is the shape and size of the particles. Both the shape and size of the particles depend on the inherent characteristics of the fluid energy mills and are a function of the type of material used, the type of elastic fluid used, the directional velocity of the fluid, the accelerative motion of the fluid, the temperature in the mill, etc.

When the direction of the elastic fluid stream from the nozzle is at an angle of about 40° relative to the direction of flow in the mill, there is a balance between the highest induced flow of the circulating stream in the mill and the most efficient grind, thereby obtaining the most satisfactory separation at the outlet from the mill, which generally extends in a reverse direction from the direction of flow in the mill. Under these conditions, the particle-loaded nozzle streams impact the circulating load at a fairly sharp angle, and the resulting cracked particles appear to roll in the manner of pebbles in a fast-flowing stream. This results in relatively round, smooth, discrete particles. This is an important characteristic for such polymeric materials as "Teflon" (tetrafluoroethylene), "Nylon" (polyamides), polystyrene, etc. The smooth, somewhat flattened particles, without an excess of extreme fines, gives the smoothest finish after molding.

In grinding very tough, resilient plastics such as "Teflon" and "Nylon," which do not fracture easily, it has, in accordance with the present invention, now been found preferable to use a separator or secondary classifier in stream with the primary mill, whereby the secondary classifier is selectively controlled to reject particles over a predetermined size but to pass those under such size to a collector. The oversized particles are automatically returned to the primary mill for further grinding. Those particles that do pass into the secondary classifier are whirled in stratified layers, causing them to be further rounded and polished. This has been found to be particularly effective with such materials as Ceylon flake graphite and artificial graphite.

If it is desired to produce products having sharper edges and less roundness, the nozzle angles are increased accordingly, as, for example, to 50° or 60° . With such angles of impact, the collisions are more direct and the particles shatter rather than abrade. It is, furthermore, possible to obtain a combination of particle types, i.e., some shattered and some abraded, by using a plurality of fluid nozzles and reversing one or more of them up to 180° against the main circulatory stream in the mill. This may also be utilized to vary the size of the particles from close to 0 microns to about 100–150 microns or more. This is desirable in some cases, as, for example, in the grinding of cement or fillers, because the smaller particles fill the voids between the larger particles.

It is, therefore, one object of the present invention to provide a method which is highly effective in treating solid particles to obtain any desired shape and size of finished particle size and any desired chemical or physical reaction or interaction.

Another object of the present invention is to provide a method which is adapted to selectively obtain any desired shape and size of particles and any desired chemical or physical reaction or interaction.

Other objects and many of the attendant advantages of this invention will be readily appreciated as the same becomes better understood by reference to the following description when read in conjunction with the accompanying drawing wherein the single FIGURE of drawing is a somewhat diagrammatic side elevational view of an apparatus embodying the present invention.

In accordance with the present method, jets of elastic fluid, such as gas or vapor, are propelled against a circulating vortex of particles entrained in similar elastic fluid, to selectively propel a larger proportion of the particles toward or away from an outlet positioned adjacent the inner peripheral portion of the vortex.

Referring in greater detail to the drawing wherein similar reference characters refer to similar parts, there is shown a fluid energy mill, generally designated 10, having a curved

lower inlet or treating section 12 integral with an upstack 14 that is, in turn, integral with a curved upper classifier section 16. The classifier section 16 is integral with a return duct or downstack 18 that leads back into the inlet section 12. An outlet duct or primary classifier 20 leads from the lower end of the classifier section to a conduit 22 which is, in turn, tangentially connected to a chamber 24 comprising the upper section of a secondary separator or classifier, generally designated 26.

Adjacent to, but in opposed relation to the entrance of the duct 20 are a pair of flow-diverting nozzles 28 and 30 extending from a common manifold 32 having a manually operable valve 34. The nozzles 28 and 30 direct fluid toward the top and bottom of the entrance to duct 20. The valve 34 is connected to a source of elastic fluid under pressure (not shown). Another flow-diverting nozzle 36 is provided at the upper bend between the section 16 and duct 20. This nozzle 36 is positioned to direct fluid to bypass the duct 20 and direct it toward the downstack 18. The nozzle 36 is connected to a manual valve 38 connected to the source of elastic fluid (not shown). A two-way valve (not shown) is provided at the source to direct the flow either toward nozzles 28 and 30 or toward nozzle 36. There may also be provided a valve means of standard design (not shown) to vary the pressure or velocity of the fluid from the source, or the pressure or velocity may be varied in any other manner known to the art.

By selectively directing jets of fluid through either nozzles 28 and 30 or nozzle 36, and by selectively varying the pressure or velocity of these jets, more of the larger particles on the outer periphery of the circulating flow can be directed toward the outlet duct 20 by nozzles 28 and 30 or more of the finer lighter particles on the inner periphery of the circulating flow can be bypassed away from the outlet duct into the downstack 18 by nozzle 36. The nozzles 28, 30 and 36 can also be made inoperative merely by closing off their respective valves 34 and 38 to permit normal circulatory flow. If the nozzles 28 and 30 are in use, there will be a larger proportion of larger particles removed from the mill through duct 20, whereas if the nozzle 36 is in use, there will be a larger proportion of smaller particles since substantially all the larger particles will be recycled through the downstack 18 for further grinding. An increased velocity of the jets from the diverting nozzle 36 also increases the velocity of the circulating particles through the downstack and adds to the kinetic energy of these particles as they impact each other and entering new particles in the inlet or grinding chamber 12, thereby causing a greater grinding effect. The nozzles 28 and 30, on the other hand, when the velocity of their jets is increased, increase the velocity of the stream flowing into the chamber 24 of the secondary classifier 26.

A feed inlet 40 (illustrated as a simple chute, but which may be any desired form of feed means) is provided between the lower end of the downstack 18 and the inlet chamber 12. Just below the feed inlet 40 is a bridging conduit 42 leading from the upper end of the inlet chamber 12 to the lower or nozzle portion 44 of a cone-shaped lower chamber 46 of the secondary separator-classifier 26. This conduit 42 is provided with a Venturi passage 48 and is positioned in opposed relation to a nozzle 50 connected to a source of elastic fluid under pressure (not shown).

At the lower end of the mill 10 is provided a plurality of fluid nozzles 52. These nozzles 52 are each in the form of an individual ball positioned in a ball socket. The ball nozzles 52 each have a handle 53 that can be used to rotate the ball through angles of about 120° to direct the nozzles individually in all increments of angular direction from about 30° in the direction of flow through the chamber 12 to about 30° in the opposite direction. The nozzles 52 receive their fluid from a manifold 54 that has an access door 55 to permit entry to manipulate the ball nozzles. The manifold 54 is provided with high-pressure elastic fluid from a conduit 56 that is controlled by a valve 58. The conduit 56 is connected to a source of fluid (not shown). The valve 58 as well as the valves 34 and 38, although shown as being manually operable, may be con-

trolled in any other feasible manner, as by remotely controlled solenoid systems, hydraulic or pneumatic pressure systems, or the like. This may also be true of the ball nozzles 52.

The nozzles 52 are illustrated as being of the standard convergent-divergent type to obtain high fluid velocities, this being preferred for grinding processes and the like. However, abrupt-type nozzles or any other desired nozzle types may be used in accordance with the treatment being practiced and the materials being processed. Furthermore, although high-pressure fluid is illustrated as being passed through the nozzles 52, it may sometimes be preferred to use low-pressure fluid in accordance with the particular process and materials.

A separate feed means 60 is positioned just above the inlet section 12 and is controlled by a valve 62. This inlet 60 is provided as an optional feature for the insertion of a liquid spray when such is desired for the coating of particles or for chemical reaction therewith.

An exhaust duct 64 leads upwardly from the chamber 24 of separator-classifier 26 to a manifold 66 connected to a collector (not shown). The lower nozzle portion 44 leads into the top of a conical after-blender chamber 68 from the lower end of which extends a conduit 70. The conduit 70 curves upwardly to connect with the manifold 66, and is provided with a nozzle 72 at its lower bend. The nozzle 72 is connected to a source of elastic fluid under pressure (not shown) to provide a propellant for the flow from the chamber 68 upwardly to the manifold 66.

A feed means 74 extends tangentially into the chamber 24 and is provided with a valve-controlled nozzle 76 connected to a source of fluid pressure (not shown) to act as a propellant means. A similar feed means 78 provided with a similar nozzle 80 tangentially extends into the after-blender chamber 68. Both of these feed means are optionally used to feed additives of any desired type into the respective chambers.

In the operation of the above-described apparatus, when used as a grinding mill, the solid particles are fed through inlet 40 and are ground or pulverized by impacts between themselves and the chamber walls as they pass through the inlet chamber 12 where high-pressure fluid from the nozzles 52 acts as the energy medium in the standard manner of fluid energy mills. The degree of pulverization and the size and shape of the pulverized particles are controlled by the angles of the various nozzles 52 and the velocity of the fluid. In this respect, if the nozzles are directed in the direction of flow through the chamber 12, there will be a minimum of violent impacts and the result will be more of a separation of particles from each other rather than a pulverization. When the nozzles are directed against the flow, there will be a maximum amount of impacts and maximum pulverization. These effects can be varied between extremes by selectively varying one or more of the nozzle angles and by varying the velocities of the fluids passing through the different nozzles.

As the particles pass around the mill to the classifier section 16, in the normal operation, the finer, lighter particles on the inner periphery of the circulating vortex would pass out through the outlet duct 20, while the larger, heavier particles on the outer periphery would pass through the downstack 18 for further grinding. However, if it is desired that the distribution of the particles in the final product include more intermediate or larger size particles, the diverting nozzles 28 and 30 are actuated. The jetstreams from these nozzles propel more of the larger particles toward the entrance into duct 20, the size of such particles being determined by the velocity of the jets, whereby higher velocities, with greater kinetic energy, will propel larger, heavier particles toward the duct 20. In this manner, only such particles as are too large and heavy to be sufficiently propelled by the jets will continue to fall through the downstack 18 for further grinding. On the other hand, when it is desired to obtain a particle distribution in the final product that includes only relatively small, light particles, the nozzle 36 is actuated to propel the heavier particles in a direction to bypass the entrance to duct 20.

The particles that pass out of the mill through duct 20 flow through conduit 22 and pass into the upper portion of the secondary classifier chamber 24 in a tangential direction. The tangency of the conduit 22 creates a horizontal, downwardly moving vortex in chamber 24. From this vortex, the finer particles, on the inner periphery of the vortex, pass upwardly through duct 64 and manifold 66 to the collector (not shown) while the heavier particles on the outer periphery pass down with the circulating vortex into the conical chamber 46 from where they pass, by gravity, through the nozzle 44 into the chamber 68 from where they are propelled through conduit 70, by the fluid jet from nozzle 72, into the manifold 66.

If it is desired to return some, or all of the particles falling through chamber 46 to the mill chamber 12 for further grinding, the nozzle 50 is actuated to propel such particles into the bridging conduit 42, the amount and size of the returned particles being determined by the velocity or force of the fluid jet from the nozzle 50.

If it is desired to coat the particles with a particular material or to obtain a chemical or physical reaction therewith, such material may be inserted through the inlet 60. If additional reaction with the same or other material is desired, such additional material may be inserted through either or both of the inlets 74 and 78, depending upon what stage of the processing and which particles are to be reacted. Alternatively, the inlet 60 may be deactivated and only one or both of the inlets 74 and 78 may be used.

Although the process described above relates to grinding or pulverization, the apparatus may be used for drying, mixing, chemical or physical reaction, without grinding, merely by passing low-pressure fluid through the nozzles 52 or by using high-temperature, low-pressure fluid. Drying and grinding is effected by using high temperature, high pressure fluid.

Fluid energy mills are adapted to grind, mix, blend, combine or chemically or physically react any type of solid particles including foods, cosmetics, pharmaceuticals, metals, minerals and natural and synthetic plastics. Among such materials are polyesters, polyethers including polymers of acrylonitrile, butadiene, styrene, acrylates and methacrylates, acetal resins and fibers, allyl resins, amino resins such as melamine and urea formaldehyde. Also cellulose and cellulose/resin fibers such as cellulose triacetate, cellulose acetate butyrate, cellulose propionate, cellulose nitrate, ethyl cellulose, etc. Also haloplastics such as paraffinic hydrocarbon polymers in which all or part of the hydrogen atoms are replaced with a halogen such as chlorine, bromine and fluorine. Particularly important among these are polytetrafluoroethylene, fluorinated ethylene propylene, chlorotrifluoroethylene, etc. Also halogenated polyvinylidene, epoxy resins, furane resins, ionomers, isocyanates, parylene polymers, polyamides such as nylon, phenolics, phenol-furfural and resorcinol formaldehyde. Also phenoxy resins, polyallomers, polycarbonates, polyimides, polyethylene both low and high molecular weight as well as cross-linked. Also unsaturated polyesters, polyphenylene ox-

ides, silicones and silicon. Also polypropylene, propylene copolymer and polysulfane. Vinyl polymers and copolymers such as polyvinylchloride and polyvinyl acetate, are effectively processed by these mills. They are also effective in the treatment of inorganic polymers such as glass, fiberglass and Wollastonite, as well as asbestos, carbon black, silica gel, etc. They are also very effective in the treatment of polyparaxylenes which are formed of linear, highly crystalline molecules, as well as the polybenzimidazoles, polyphenylene oxides and fiber glass reinforced thermoplastics.

Fluid energy mills may also be effectively used to provide a so-called "matt finish" or "satinizing" of the particle surfaces. This type of finish is nothing more than a slight roughness on the surface and has, heretofore, generally been obtained by chemical treatment.

This apparatus can also be used to accomplish chemical reaction under increased pressure by merely restricting the exhaust from the mill, thereby building up pressure in the treating chamber 12. For example, coarse silicon metal may be abraded and reacted with high-pressure methyl chloride at about 500° to 800° F., or above, while the pressure in the mill is increased to between about 30 to 80 p.s.i.g.

The above-listed materials are merely illustrative of the type of materials effectively processed by fluid energy mills and are not intended as a limitation since any solid or semisolid material of whatever kind or type can be treated. Any of these materials can be made of any desired size and shape by merely regulating the functioning of the mill and are, therefore, to be considered inherent in the functioning of the mill.

The invention I claim is:

1. In a method of treating solid particles whereby the particles are subjected to the turbulent action of elastic fluid under pressure and are thereafter propelled in a circulating vortex through a classification area where the lighter particles in the inner peripheral portion of the vortex are separated from the heavier particles in the outer peripheral portion of the vortex and pass through an outlet adjacent said inner peripheral portion, the step of propelling jets of elastic fluid in selected lateral directions against said vortex to selectively propel a larger proportion of said particles toward and away from said outlet.

2. In the method of claim 1, the step of secondarily separating lighter from heavier particles in the product passing through said outlet.

3. In the method of claim 2, the step of recycling the secondarily separated heavier particles back to be resubjected to the turbulent action of said elastic fluid.

4. In the method of claim 2, the step of applying an additional material for selective chemical and physical interaction with the secondarily separated particles.

5. In the method of claim 1, the step of applying an additional material for selective chemical and physical interaction with said particles prior to their initial subjection to the turbulent action of said elastic field.

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