United States Patent

[11] 3,595,486

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[21] Appl. No. 879,322
[22] Filed Nov. 24, 1969
Division of Ser. No. 607,974,
Jan. 9, 1967, Patent No. 3,491,953
[45] Patented July 27, 1971
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[54] **TREATMENT OF GRANULAR SOLIDS BY FLUID ENERGY MILLS**
5 Claims, 1 Drawing Fig.

[52] U.S. Cl. 241/5
[51] Int. Cl. B02c 19/06
[50] Field of Search 241/5, 39

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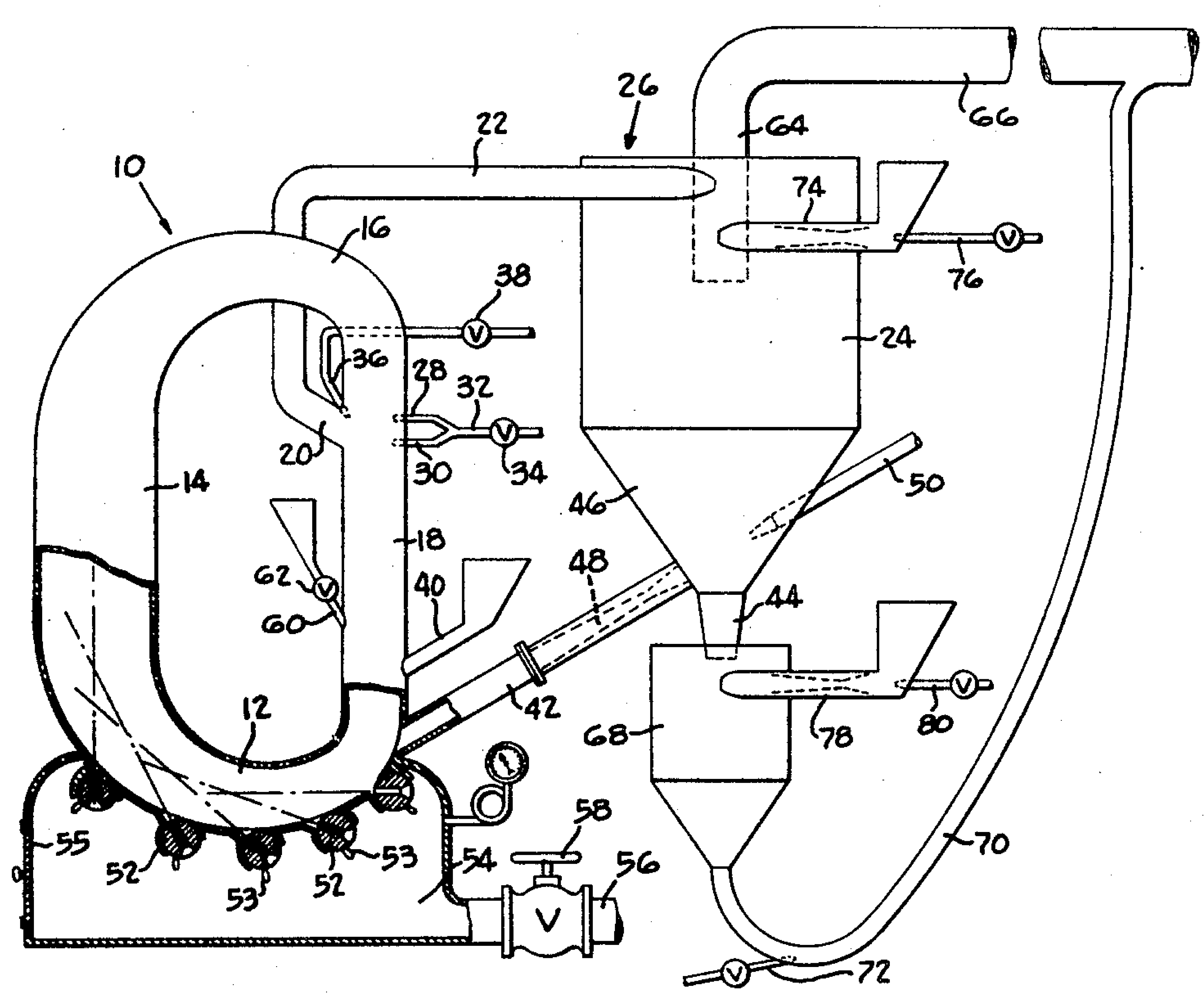
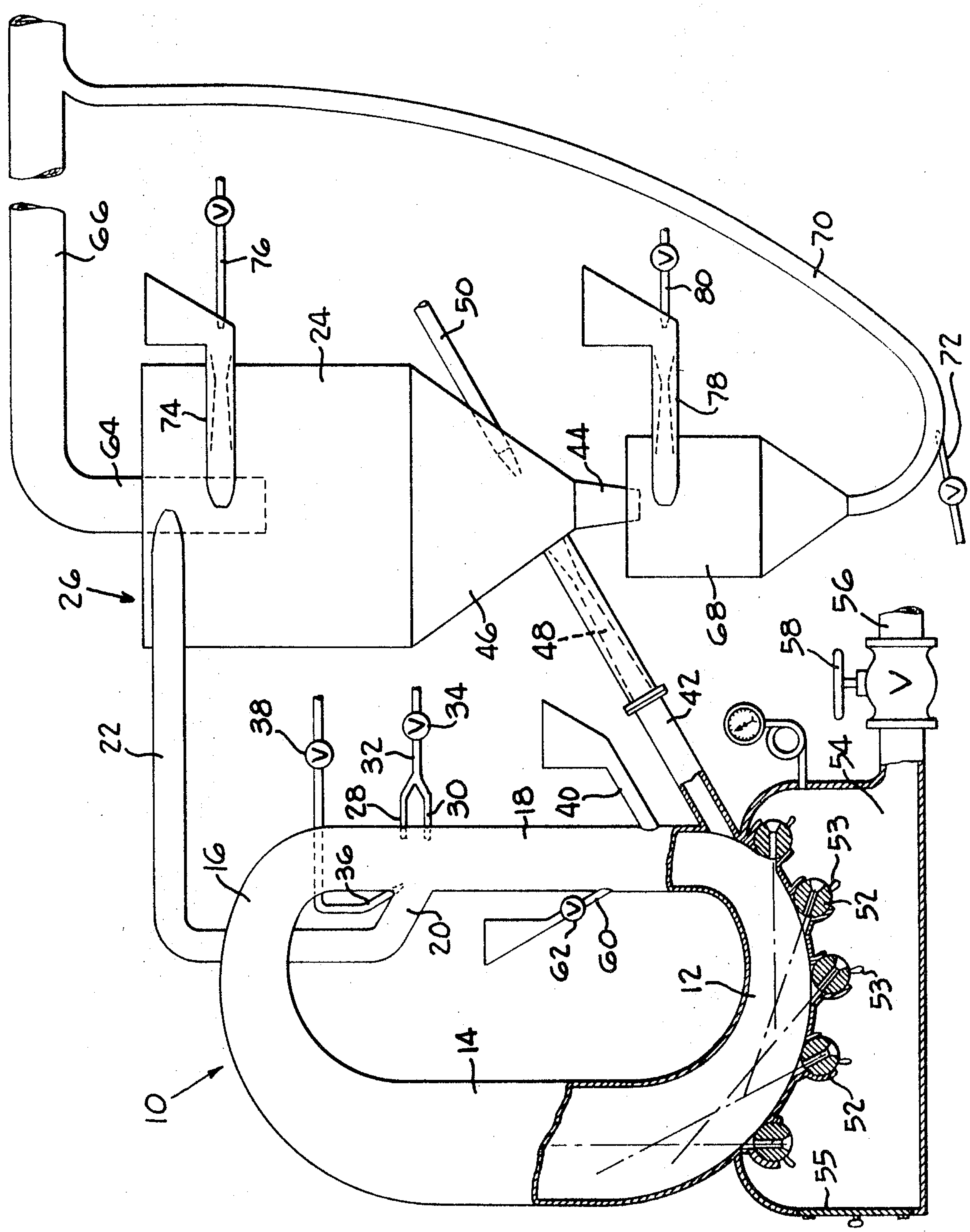
ABSTRACT: A method of treating solid particles by propelling jets of elastic fluid, such as gas or vapor, in selected lateral directions against a circulating vortex of particles entrained in similar elastic fluid to selectively propel a larger proportion of the particles toward or away from an outlet 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 hydroscopic 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 bound 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 desireable 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** leasing form 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 controlled an 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 heave 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 haloginated polyvinylidene, epoxy resins, furane resins, ionomers, isocynates, parylene polymers, polyamides such as nylon, phenolics, phenol-furfural and resorcinal formaldihyde. Also phenoxy resins, polyallomers, polycarbonates, polyimides, polyethylene both low and high molecular weight as well as cross-linked. Also unsaturated polyesters, polyphenylene oxides, 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, therefor, 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.