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## Method of Creating Ultra-Fine Particles of Materials Using a High-Pressure Mill

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(54) **METHOD OF CREATING ULTRA-FINE PARTICLES OF MATERIALS USING A HIGH-PRESSURE MILL**

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(21) Appl. No.: **09/680,271**

(22) Filed: **Oct. 6, 2000**

#### Related U.S. Application Data

(63) Continuation-in-part of application No. 09/413,489, filed on Oct. 6, 1999, now Pat. No. 6,318,649.

(51) **Int. Cl.**<sup>7</sup> ..... **B02C 19/00**

(52) **U.S. Cl.** ..... **241/5; 241/16; 241/29; 241/39; 241/40; 241/152.2**

(58) **Field of Search** ..... 241/1, 16, 5, 29, 241/39, 40, 152.2

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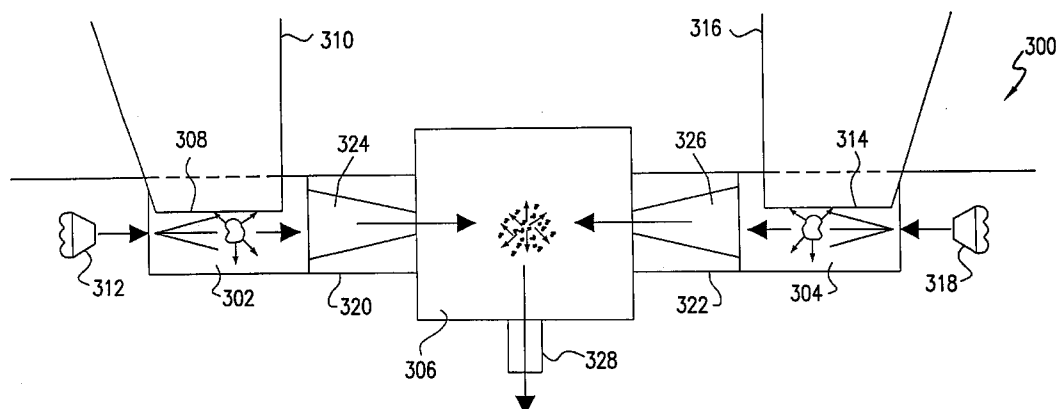
*Primary Examiner*—William Hong

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(57) **ABSTRACT**

A method for creating ultra-fine particles of material using a high-pressure mill is described. The method includes placing a material in a first chamber and subjecting the material to a high-pressure fluid jet to divide it into particles. These particles are then transferred to a second chamber in which they are subjected to cavitation to further divide the particles into relatively smaller particles. These relatively smaller particles are then transferred to a third chamber, in which the particles collide with a collider to still further divide them into ultra-fine particles of the material. The mill of the present invention includes a first chamber having an high-pressure liquid jet nozzle, first and second slurry nozzles, a second cavitation chamber and a third chamber which houses a collider. In one embodiment, the slurry nozzle has an inner surface and sharp edges that project slightly out from the inner surface. Sensors may be located throughout the mill to collect data on the comminution process and to use the data to control the resultant particle size. The product size of the ultra-fine particles made according to the mill of the present invention are preferably less than 15 microns. Further, the particles produced using the mill of the present invention are formed as flakes or platelets which have been broken along nature planes in the material.

**11 Claims, 14 Drawing Sheets**



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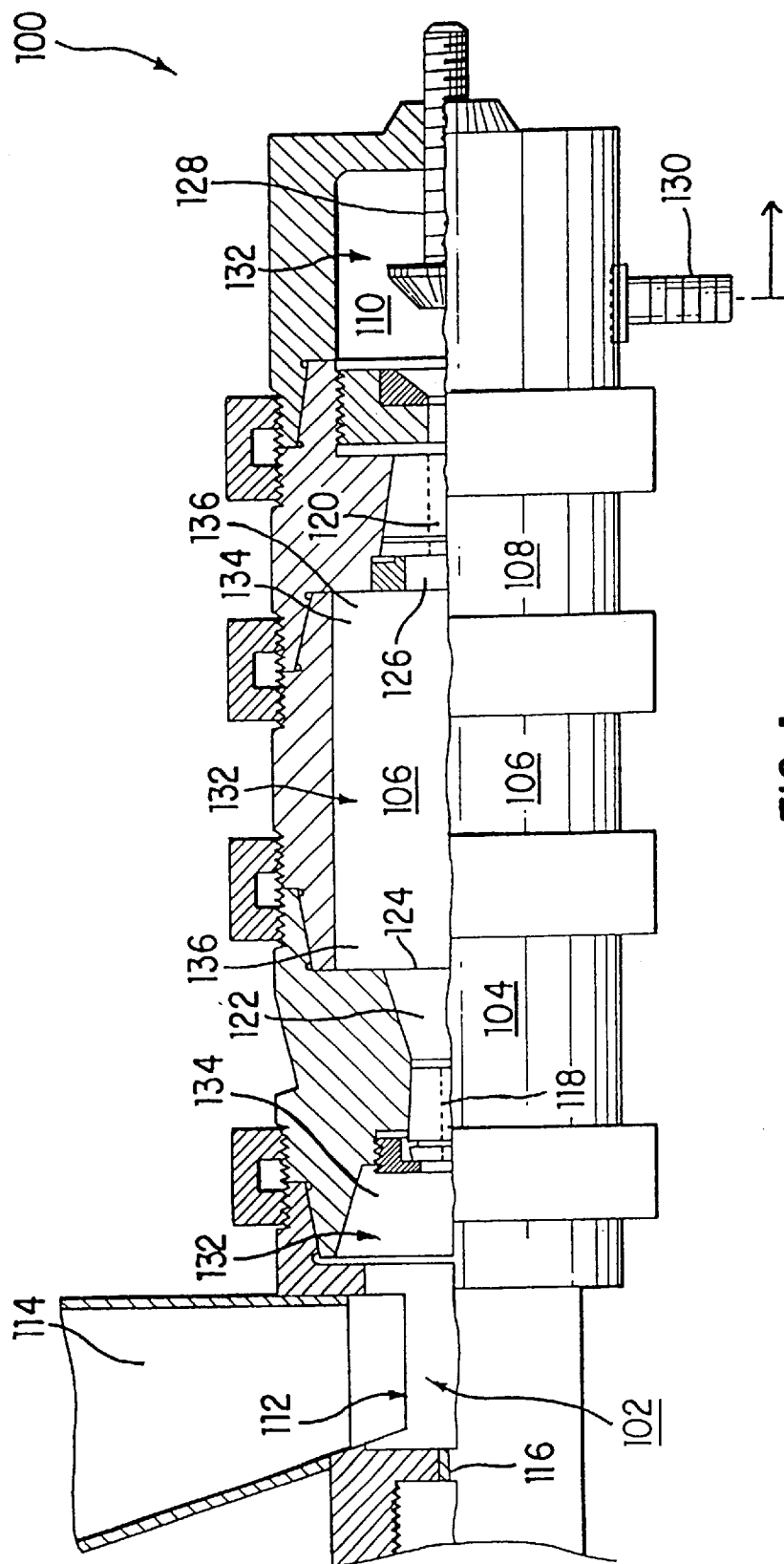


FIG. 1

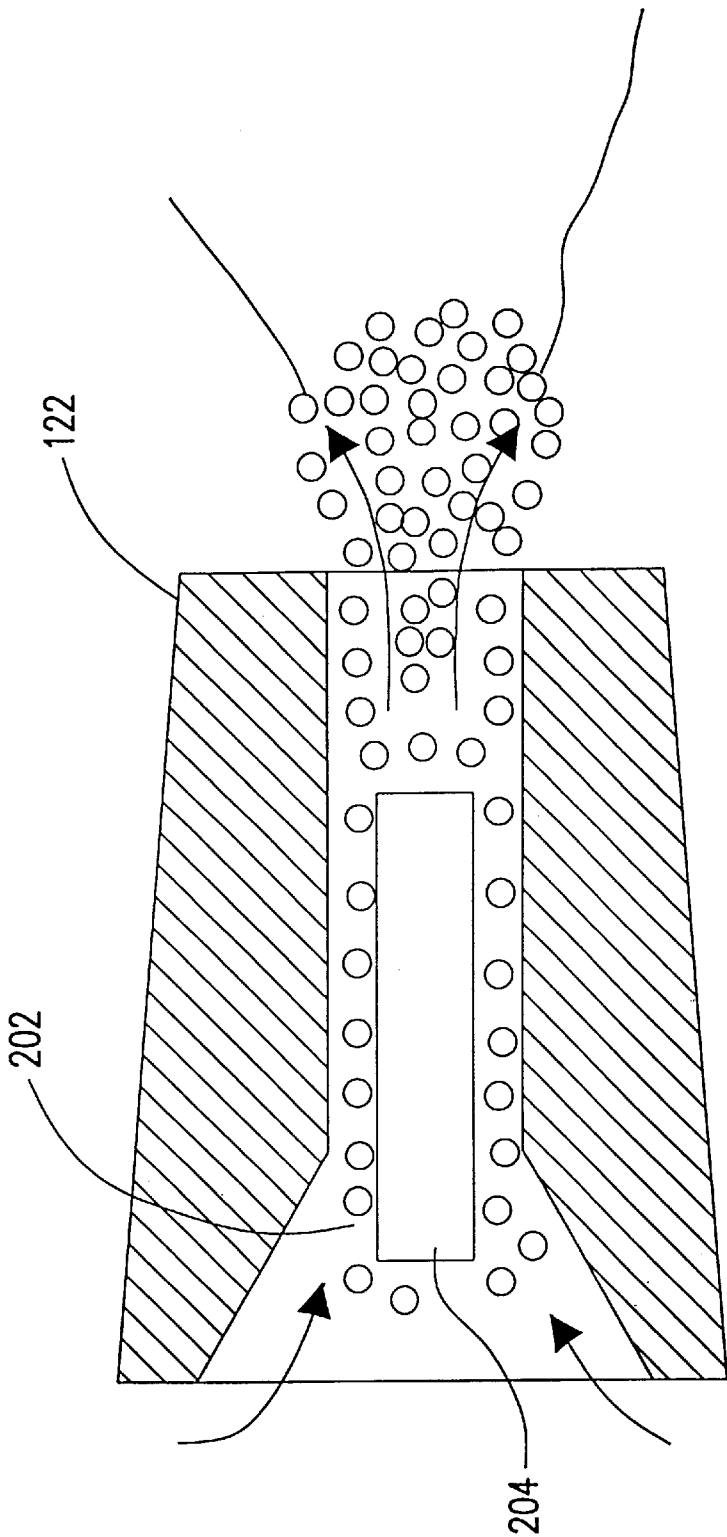


FIG. 2

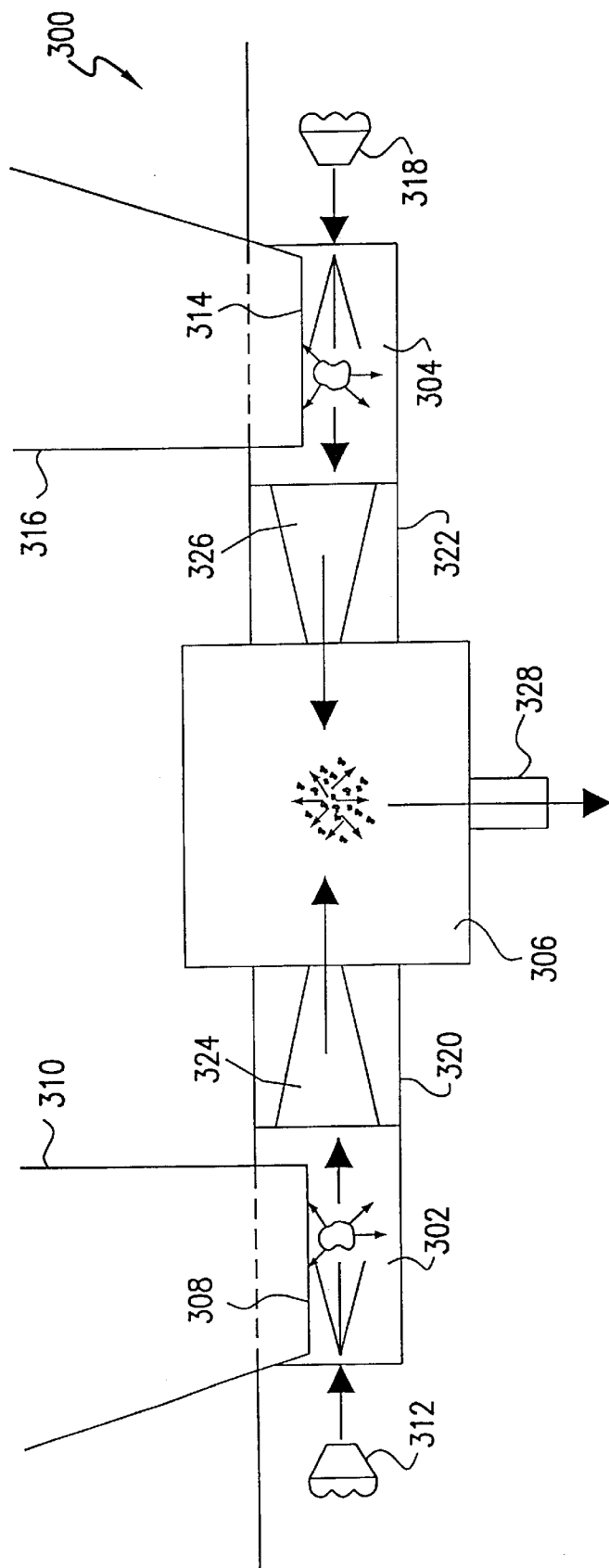
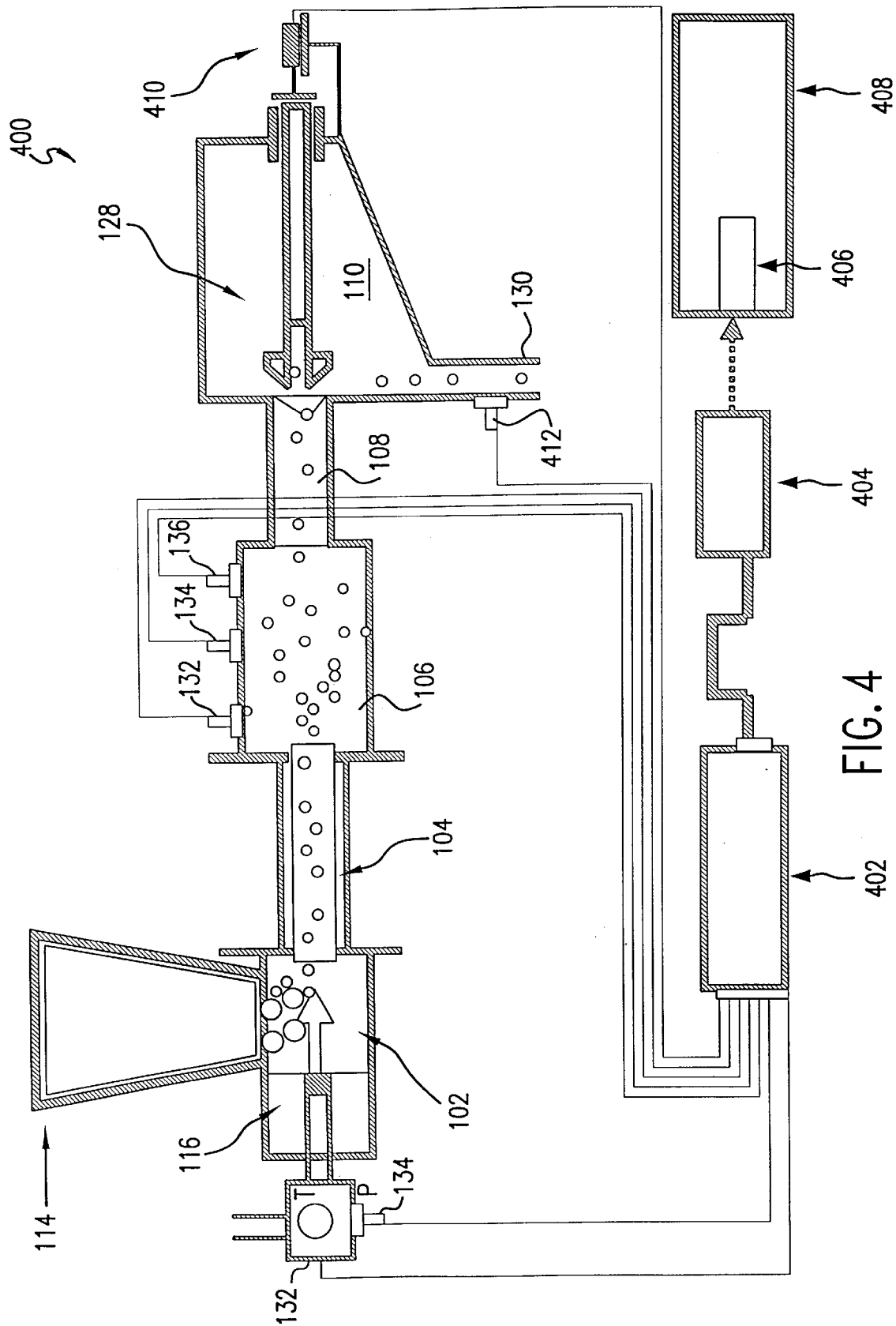


FIG. 3



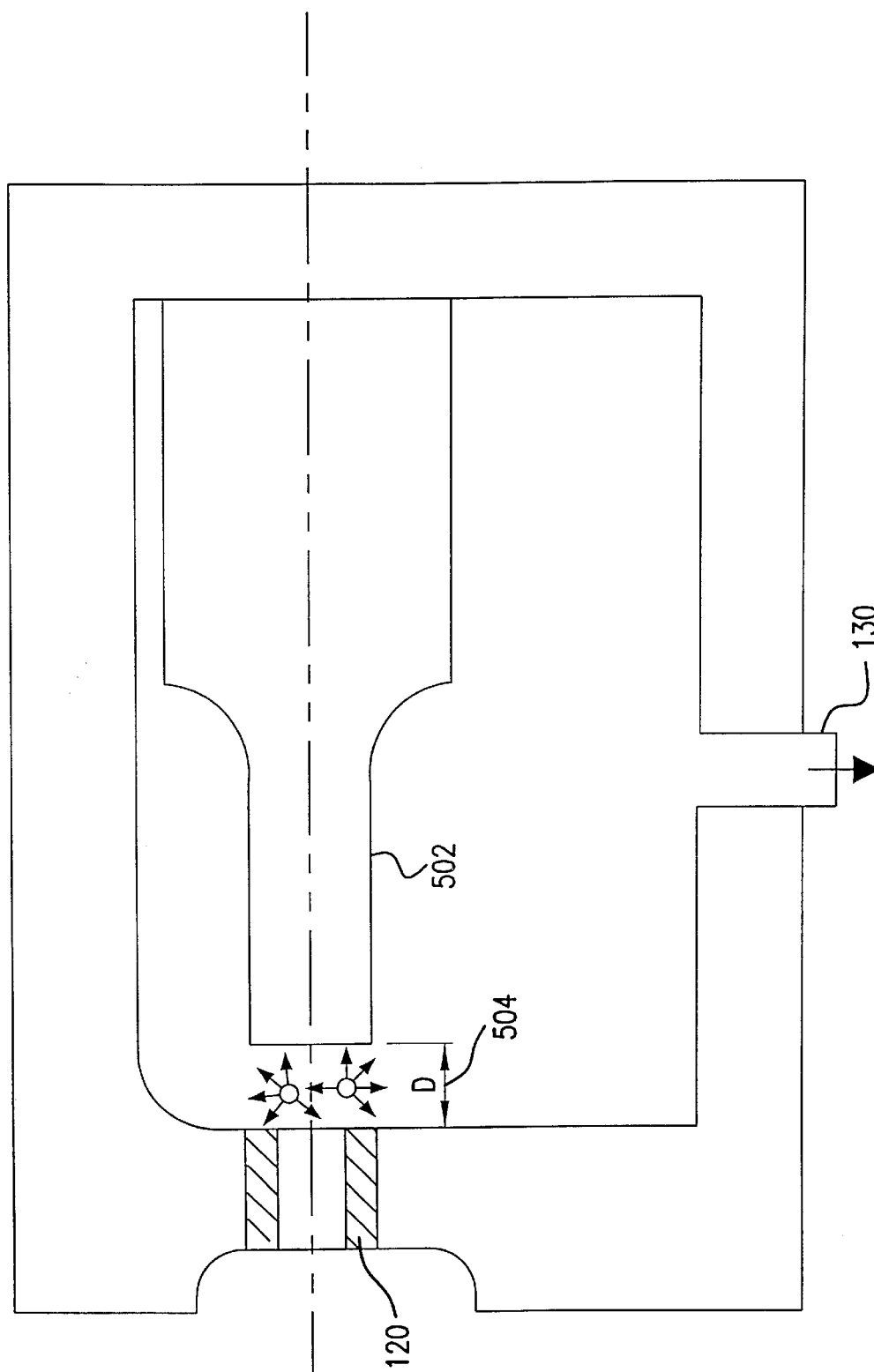


FIG. 5



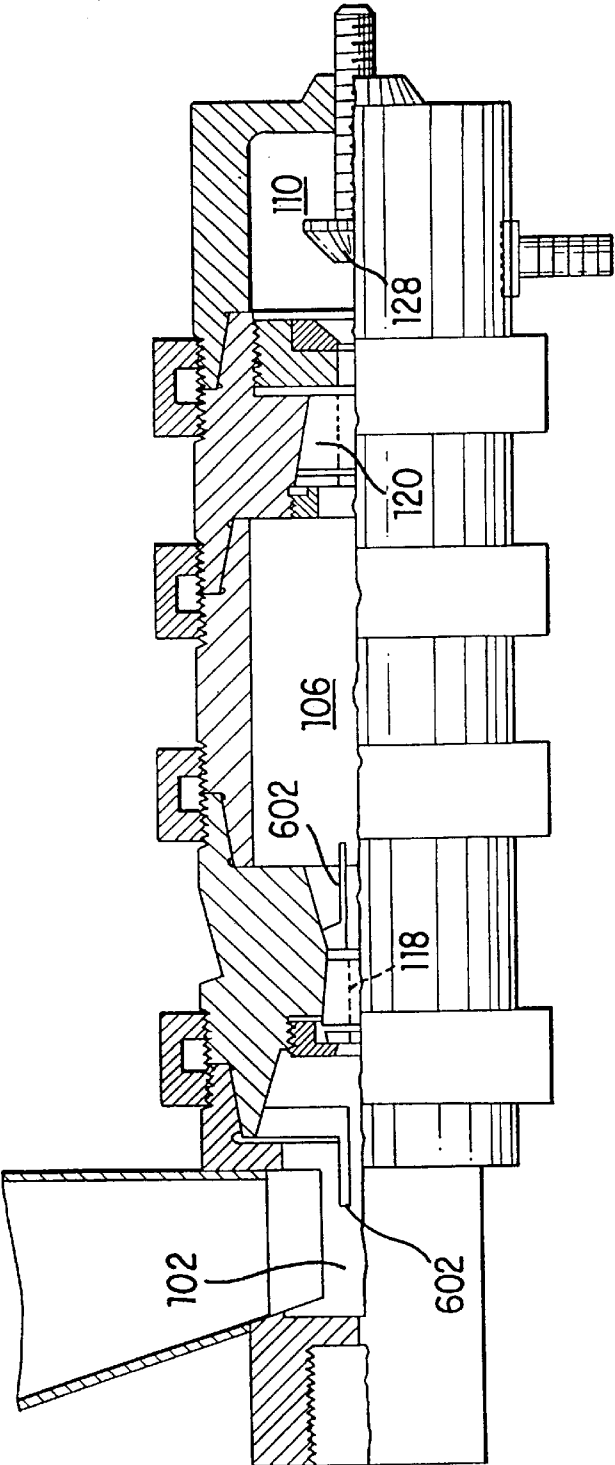


FIG. 6

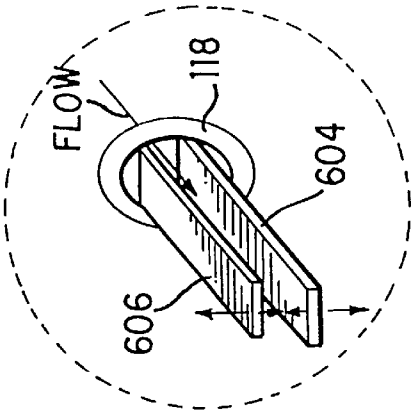


FIG. 6A

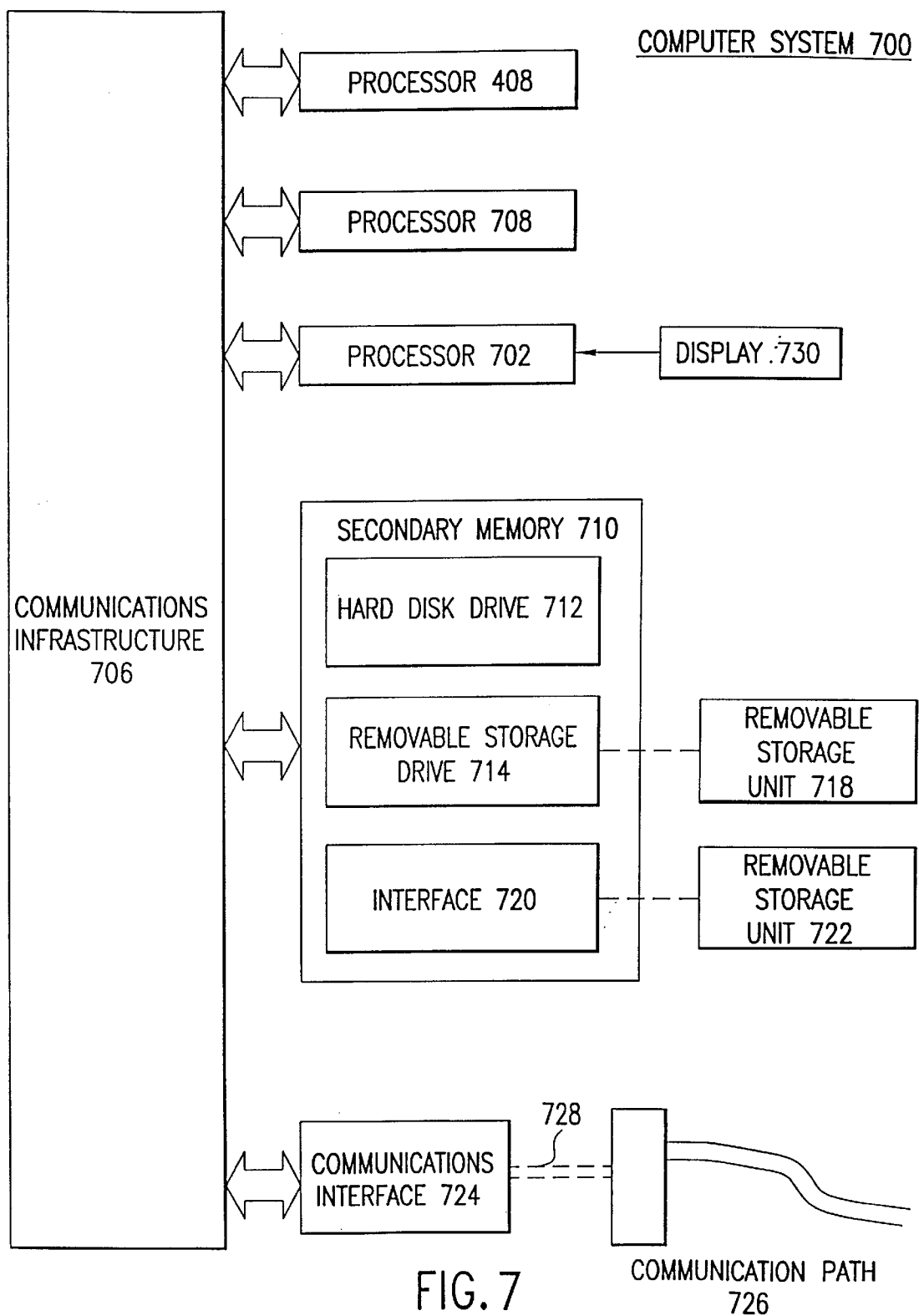


FIG. 7

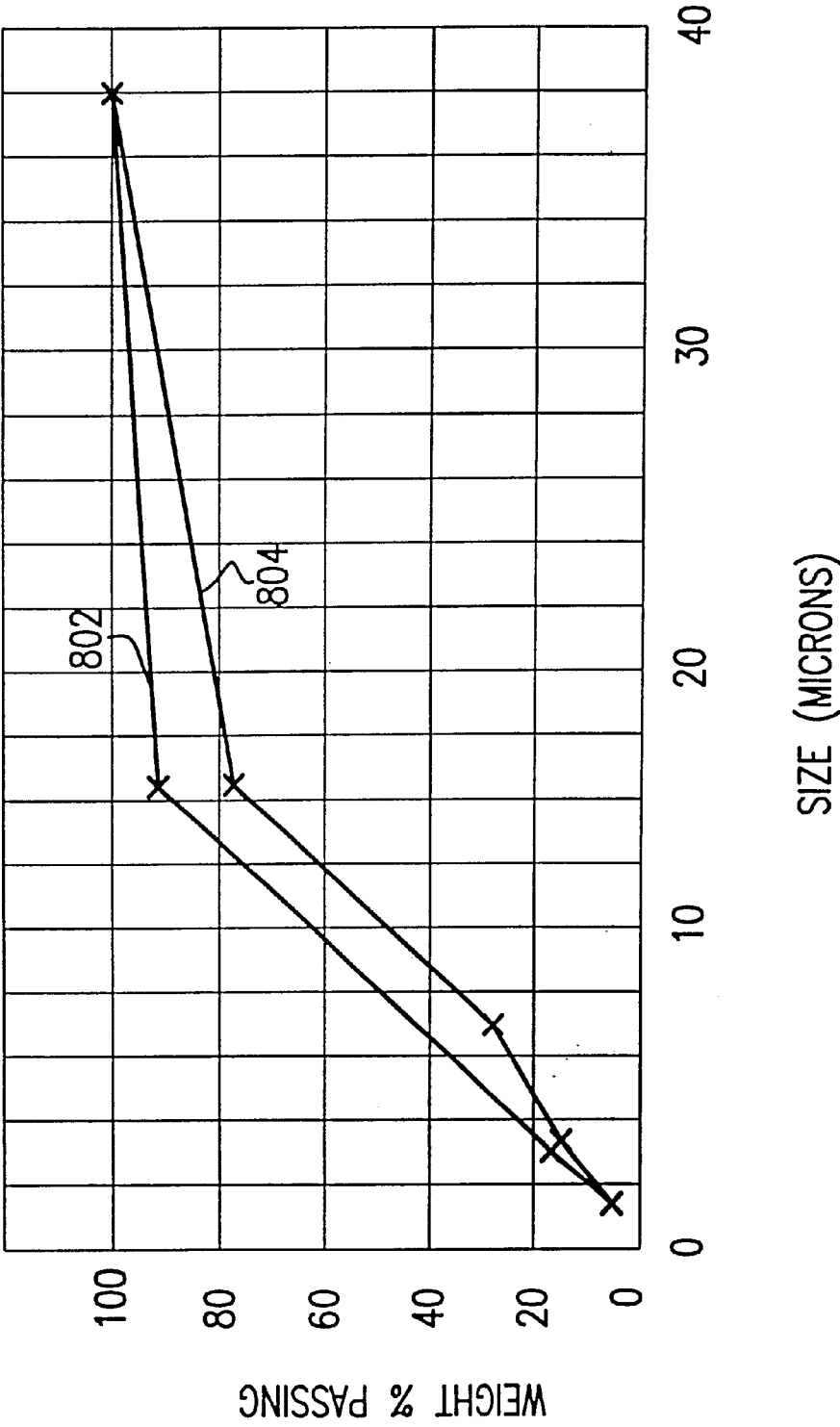
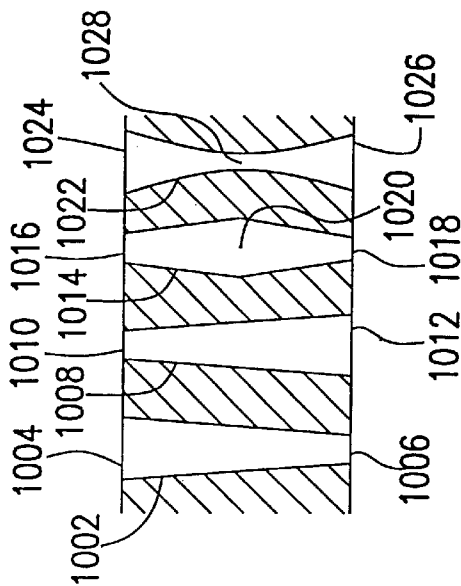
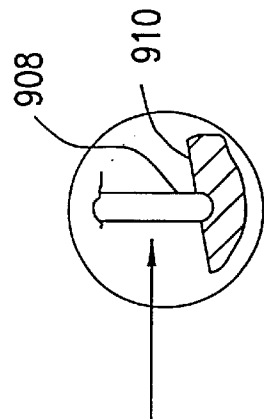
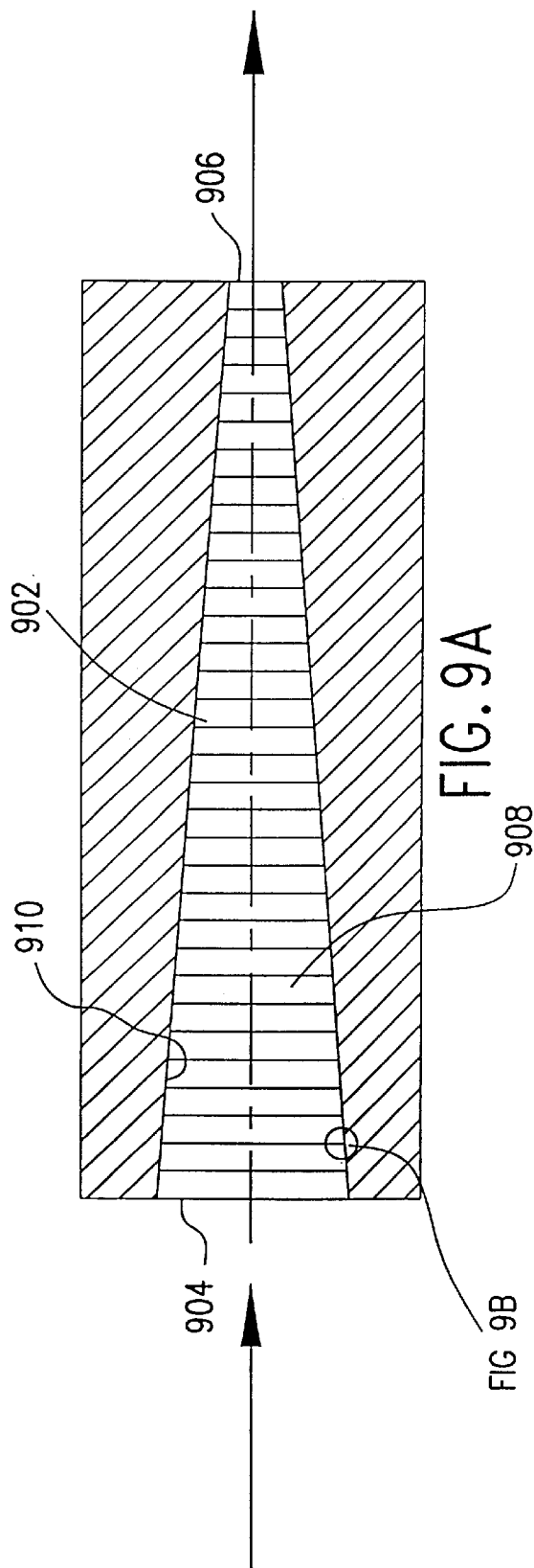
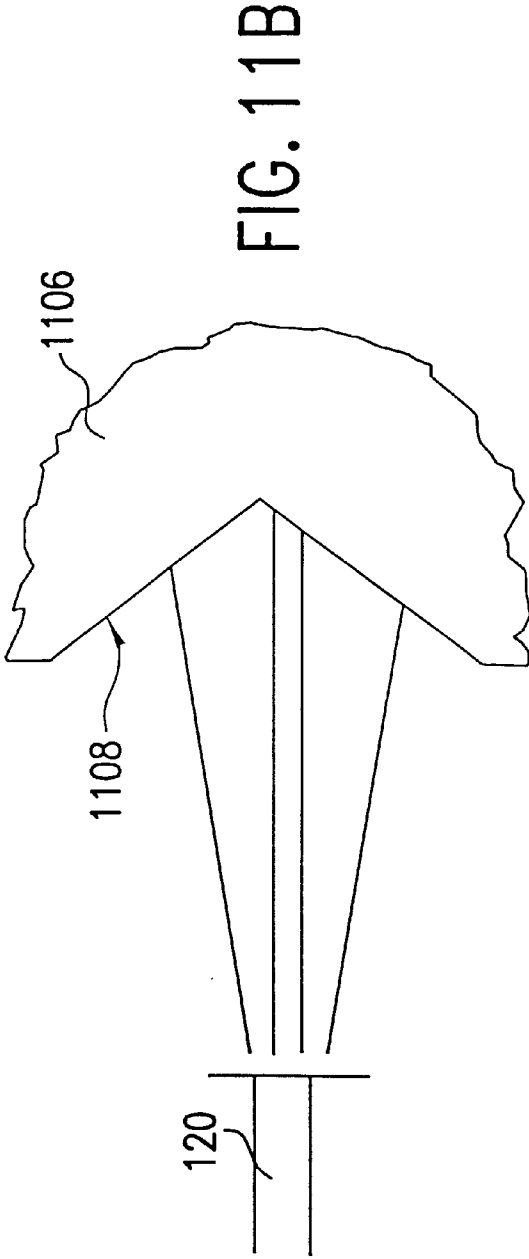
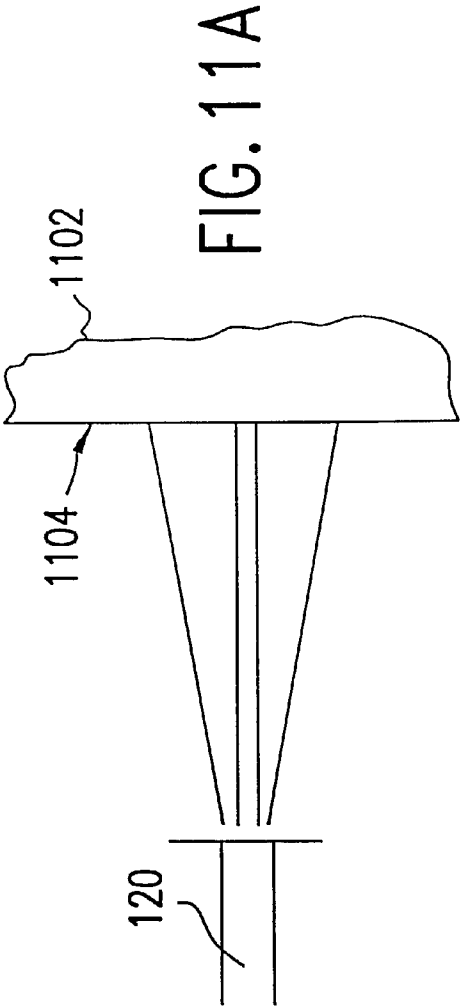


FIG. 8





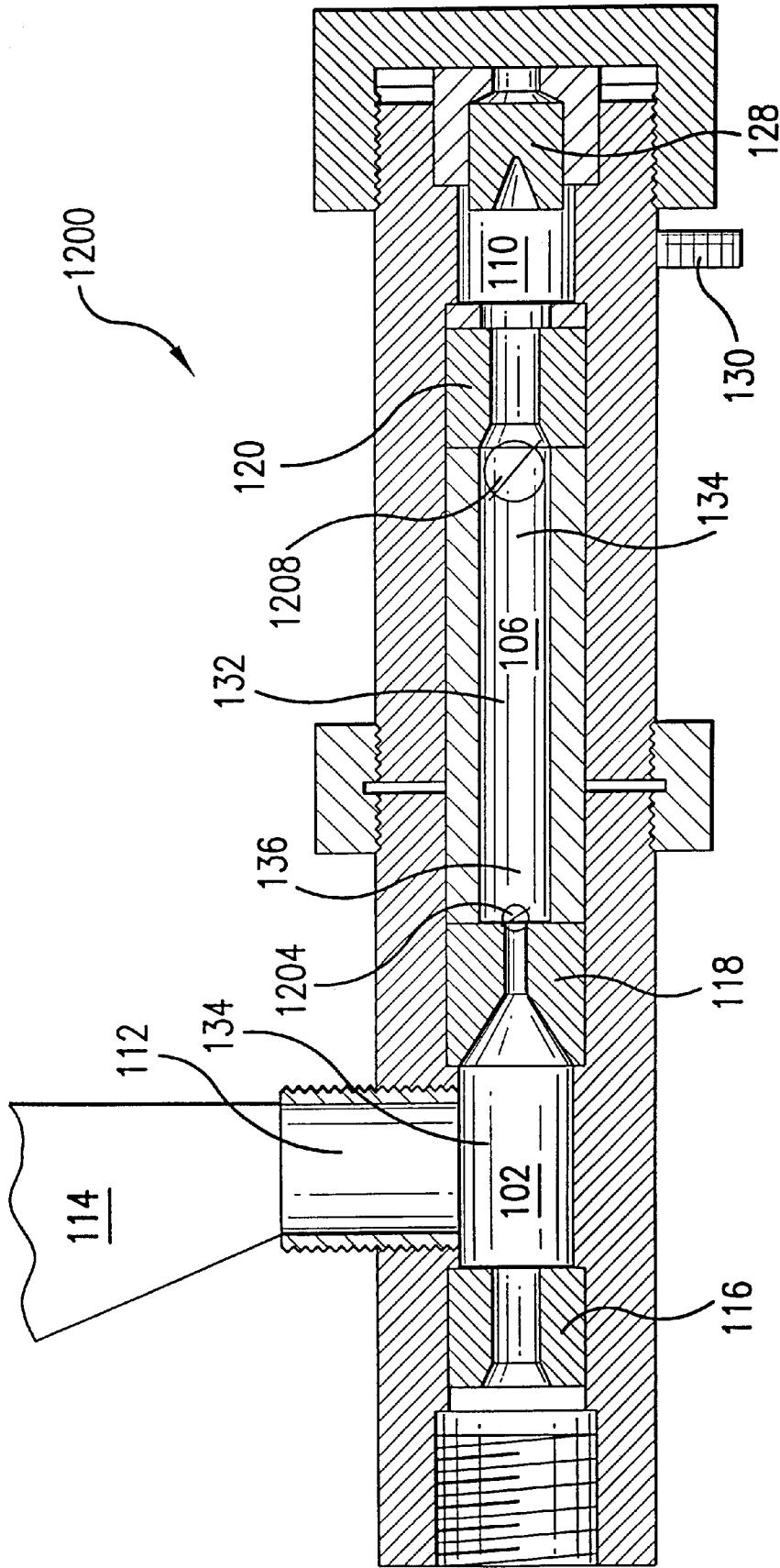


FIG.12

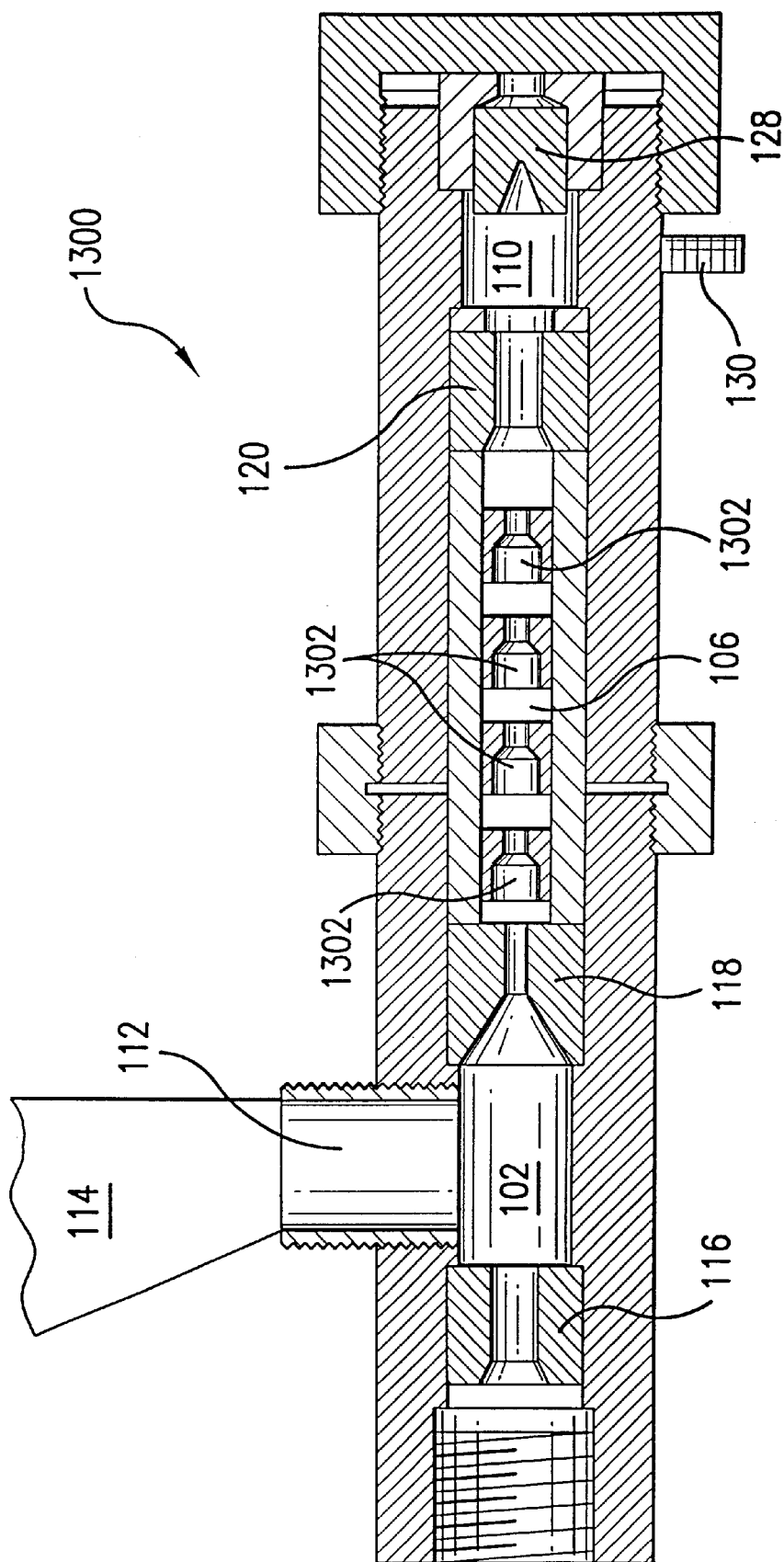


FIG. 13

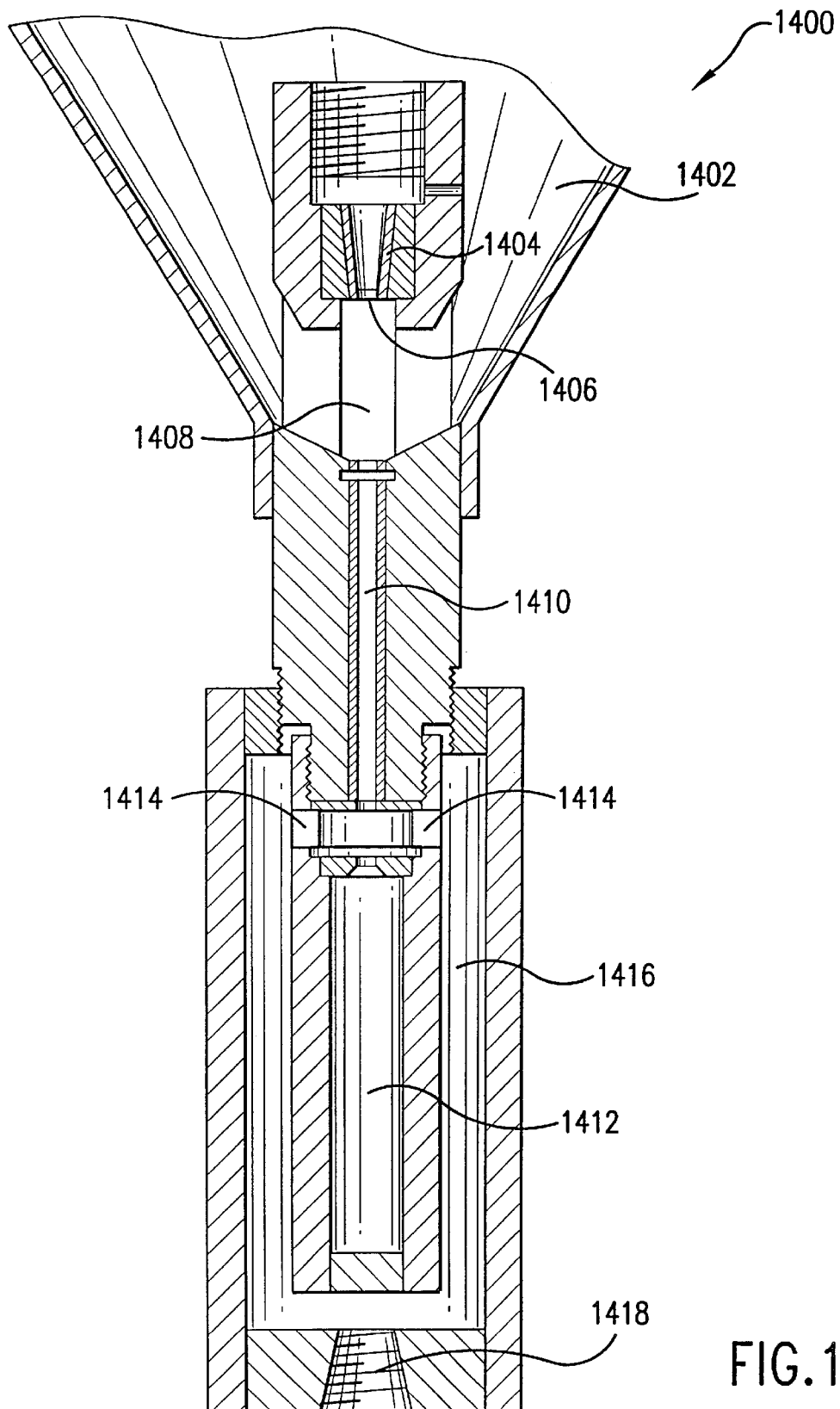
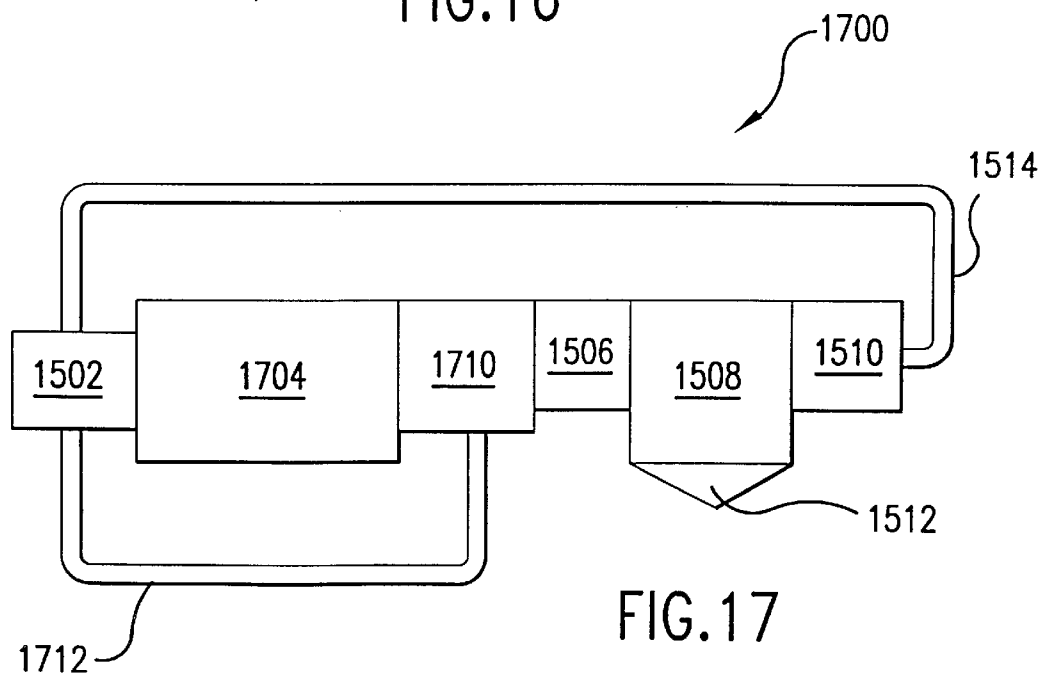
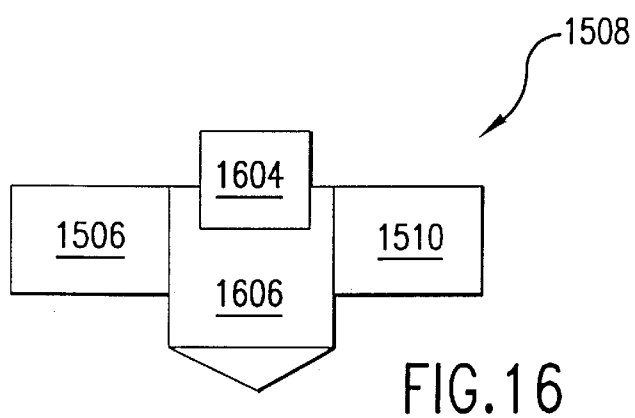
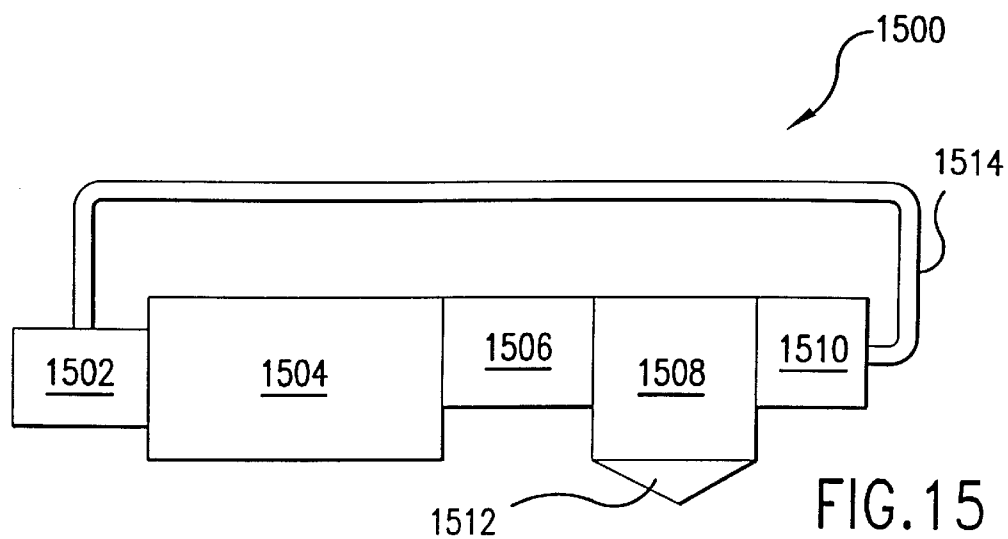


FIG. 14





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# METHOD OF CREATING ULTRA-FINE PARTICLES OF MATERIALS USING A HIGH-PRESSURE MILL

## CROSS-REFERENCE TO RELATED APPLICATIONS

The present application is a continuation-in-part of application Ser. No. 09/413,489; filed Oct. 6, 1999 now U.S. Pat. No. 6,318,649, pending, the disclosure of which is incorporated herein by reference in its entirety.

## STATEMENT REGARDING FEDERALLY-SPONSORED RESEARCH AND DEVELOPMENT

The U.S. Government has a paid-up license in this invention and the right in limited circumstances to require the patent owner to license others on reasonable terms as provided for by the terms of one or more of the following contracts: Contract No. N00014-99-C-0277, Contract No. N00014-01-C-0245, Contract No. N00014-01-C-0448 and Contract No. N00014-02-C-0329 awarded by the Office of Naval Research and Subcontract number 4500011933 awarded by Oak Ridge National Laboratory.

## BACKGROUND OF THE INVENTION

### 1. Field of the Invention

The present invention relates to a method for creating ultra-fine particles of a material using high-pressure fluid. More particularly, the present invention relates to a method for subjecting particles to a high-pressure fluid jet, high turbulence condition, cavitation and collision to comminute the particles.

### 2. Related Art

Comminution may be defined as either a single or multistage process by which material particles are reduced from random sizes by crushing and grinding to the size required for the intended purpose.

Size reduction in comminution machines relies on three different fragmentation mechanisms: cleavage, shatter, and abrasion. It is commonly stated that only three percent of the energy used in fragmenting solid particles goes into the creation of new surfaces. Thus, current comminution technology is both energy-intensive and inefficient.

During milling of material, to create a fracture in the particles of material, a stress must be induced which exceeds the fracture strength of the material. The mode of fracture and the path that it follows depends on the material, the shape and structure of the particle, and on the way and rate at which the load is applied. The way in which the load is applied will control the stresses that induce fracture extension or growth within the particle. The force used to induce this growth can be one of simple compression, which causes the particle to fracture in tension, whether at a slow or fast rate. Alternatively, the applied load may be in shear, such as is exerted when two particles rub against each other, or the load may be applied as a direct tensile force on the particle.

For optimum comminution of hard materials such as minerals a shattering fracture is most beneficial. This occurs when the energy applied to the particle is well in excess of that required for fracture. Under these conditions, very rapid crack growth is induced and will cause crack bifurcation. Thus, the multiplicity of areas in the particle that are simultaneously overstressed will combine to generate a comparatively large number of particles with a wide spectrum of sizes.

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Shattering usually occurs under conditions of rapid loading (e.g., a high velocity impact) with maximum size reduction occurring around the impact points.

According to existing theory, the finest product sizes are generated in the zone around the impact point, when insufficient energy is applied to cause total fracture of the particle. The localized nature of the applied stress and the high energy required for this ultra-fine grinding make this process relatively inefficient.

Conventional milling machines use mechanical crushing or crushing and attriting to break mineral particles into smaller particles. The low efficiency of existing reduction processes is frequently due to the application of stress where there are no particles. The result is that much of the energy input is wasted in non-productive contact between, for example, crushing mechanisms or between a crushing mechanism and the mill wall, both of which lower the overall energy efficiency of the process.

Further, for brittle materials, there is a considerable difference between the values of uniaxial compressive strength and tensile strength of the material. Thus, the amount of energy which must be consumed in breaking the mineral into small particles under compressive loading is substantially higher than that required if the material can be induced to fail under a tensile stress. To induce simple tensile failure, high pressure liquid jets or different liquid jets have been used in comminution processes.

Size reduction involves rupturing the chemical bonds within the material in order to generate new surfaces. Thus, the chemical processes associated with fracture will significantly affect the energy required to induce this fracture. This influence extends beyond the bonds themselves to include the surrounding environment. For example, the presence of liquid at the crack tip will lower the forces required to expand the crack and improve efficiency, especially where the liquid contains inorganic ions and organic surfactant. One explanation for this effect is that the additives penetrate into microcracks ahead of the major crack front and thus take part in the highly reactive events that occur during fracture. Because the capillary flow of these liquids into the material ahead of the main front runs at the velocity of crack propagation it provides a means of transmitting energy more easily within the crack tip zone. A high-pressure liquid jet containing chemical additives creates extremely dynamic conditions in which microcracks grow ahead of the main failure plane and become pressurized, thereby enhancing any chemical changes which might occur.

For use in liquid-fueled power plants, it is necessary to produce a homogeneous, pumpable suspension of coal that will not settle in delivery lines and which burns at the required rate. Therefore, the coal must be ground from the "standard plant size" to a diameter below 40 microns. Among the many milling methods used for this process the finest product is achieved by the use of autogenous attriting machines. The distinguishing feature of these machines is that size reduction is effected by particles impacting upon each other, after being given the necessary energy to induce fragmentation through a solid or liquid impeller. Included in this class are the following systems: (1) Buhrstones—which cause comminution through an abrasion action; (2) Colloid Mills—in which comminution occurs by collision between particles; (3) Fluid Energy Mills—in which particles interact upon one another; and (4) Sand Grinder—in which particles are reduced by contact with sand particles.

The advantage of the conventional equipment is that the product is reduced to very small sizes (below 40 microns)

and distributed within a narrow size range. The equipment, however, can only operate, at any one time, with small quantities of feed, and the initial feed size of particles lies in the range between 0.5 inches and 50 microns, depending on the type of unit. For the sand grinder, for example, the feed stock should already be crushed to below 70 microns. A much greater disadvantage for this type of machine is the very high power consumption required to achieve the required crushing.

The energy required to achieve a given size reduction increases as the product size decreases. This increase is due to many factors and is a consequence not only of the type of mill or the microscopic condition of the material, but also relates to the mechanism of failure at the individual particle level. This is obvious because fragmentation in a chamber is partly brought about by an interaction between the particles and the chamber wall.

In such situations, the treatment of individual particles requires special attention. For example, a coal particle is anisotropic, heterogeneous, and extensively pre-cracked. Physical properties of coal vary as a function of the degree of metamorphism of the coal particle. Because of the organic nature of the material, this means that different properties may be encountered, even within a single particle. Under such a situation an analytical approach to coal fragmentation is very complex.

The efficiency of coal comminution depends on the ability to take advantage of the anisotropy of coal particles which is, in turn, a function of the internal structure. However, with liquid jet comminution, failure occurs on the basis of differential coal porosity and permeability, as these properties control the specific rates of liquid absorption, which directly influence the rate of disintegration.

Experiments conducted with shaped explosive charges to investigate fracture formation in coal showed that there is intense fracturing of coal near the jet path, with this zone of fracture usually bounded by joints, bedding planes and cleat planes. The coal breaks into large and small pieces, usually parallel following natural cleavage planes. Beyond this intensely crushed zone, some large fractures were observed. These crossed joints and traveled long distances, while fractures originating at the base of the jet penetration also crossed bedding planes and extended the zone of influence deeper into the target material.

A Comminution technology can also be used to comminute organic materials. One example of such a material is wood. These organic materials are generally softer than the inorganic materials discussed above. In the case of organic materials, the impact of the waterjet causes a shearing force to occur to break apart the material, rather than the crack propagation discussed above.

Conventional comminution technology is both energy intensive and inefficient. Up to 97% of the energy consumed during the operation of conventional size reduction devices can go into non-productive work, with only 3% of the energy input then being used to create new surfaces. Comminution is thus an appropriate target for significant energy savings, since the tonnages of materials involved in the size reduction operations are so great that even small improvements in comminution efficiency would provide considerable savings in energy and mineral resources.

Further, conventional comminution devices are very expensive and wearing process of the friction parts are very significant and costly.

Through study it has been found that a high-pressure liquid jet has an excellent, and in some ways a unique,

ability to improve material disruption. Such a capability is due to the following features:

A liquidjet of 10,000 psi pressure moves at approximately 1,332 ft/sec, with a narrow jet diameter providing a concentrated energy flux input to the target.

The high energy density of the liquid jet is concentrated in a very small impact zone, while the intense differential pressure across the jet enhances microcrack generation and growth.

Subsequent to the initial impact, the jet stagnation pressure forces liquid into the cracks and microcracks. It develops a hydromechanical jet action in these cracks and creates an increasingly dense network of cracks in the walls of the cavity created.

Rapid jet penetration into pre-cracked minerals can be enhanced by the use of surface active agents, which will also work to further comminute the coal and retreat any mineral matter in the coal.

In those circumstances where a coal/oil mixture (COM) is required, the liquid jet can be changed to an oil jet, for example, to eliminate the intermediate drying process.

The separation of mineral matter from coal is improved by use of pressurized liquid jets. On occasion, this separation is enhanced by the differential response of the constituent materials to the jet attack which can facilitate separation of the resulting particles on the basis of the size differential in the grain or crystal sizes of these materials.

There is a reduced expectation of mechanical wear or process contamination of the product.

Conventional jet energy mills have a size reduction factor of approximately 50. This means that conventional mills can reduce the size of a particle so that the product size of the final, resultant particles is 50 times smaller than the original feed size of the particles. What is needed is a mill that makes efficient use of high-pressure liquid jets in the comminution of materials into ultra-fine particles.

#### SUMMARY OF THE INVENTION

The present invention relates to a method of creating ultra-fine particles of materials using a high pressure jet energy mill. The method is designed to achieve a size reduction factor of approximately 500 and that has relatively lower energy consumption than conventional jet energy mills. The mill of the present invention includes a first chamber in which a material is subjected to a high-pressure liquid jet attack to achieve comminution of the material. The comminuted particles are then transferred via a primary slurry nozzle to a second chamber, in which the particles undergo cavitation in a cavitation chamber. The particles are then transferred via a secondary slurry nozzle to a third chamber, in which the particles are caused to collide with a stable collider or an ultrasonically vibrating collider to cause further comminution of the particles. The position of this collider, with respect to the secondary slurry nozzle can be adjusted to affect the comminution process. Further, in one embodiment, self-resonating elements can be placed in various chambers in the mill to cause further comminution of the particles. The product size of the resultant particles is preferably less than 15 microns.

In another embodiment of the invention, the mill includes a first chamber in which a material is subjected to a high-pressure liquid jet to achieve comminution of the material. A similar, second chamber is disposed exactly opposite the first chamber. The slurry from each of the first

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and second chambers is transferred to a third central chamber, located between the first and second chambers, via nozzles, such that the jets from each nozzle undergo a high velocity collision to cause further comminution of the particles. A further embodiment of the mill discloses a vertical configuration. The mill may also be used in conjunction with a hydrocyclone and/or a spray dryer.

A mill and data control system can also be used to implement the present invention. In such a system, temperature, pressure and/or sound sensors can be located throughout the mill to measure characteristics of the system during particle processing. This data can be transferred to a processor for storage and/or used for feedback to different portions of the mill to control the comminution process. Other sensors used in the control system include a particle size sensor at the outlet of the mill to measure the size of the resultant particles, and a linear variable differential transducer to measure the position of the collider in the third chamber of the mill.

As such, one object of invention is to comminute a material into an ultra-fine particle size in a consistent and energy efficient manner.

#### BRIEF DESCRIPTION OF THE FIGURES

The foregoing and other features and advantages of the invention will be apparent from the following, more particular description of a preferred embodiment of the invention, as illustrated in the accompanying drawings.

FIG. 1 shows a first embodiment of a mill of the present invention for the comminution of materials.

FIG. 2 shows a cross-sectional view of a cavitating nozzle of the mill of FIG. 1.

FIG. 3 shows a second embodiment of a mill of the present invention for the comminution of materials.

FIG. 4 shows a mill and data control system of the present invention for the comminution of materials.

FIG. 5 shows an alternate embodiment of a third chamber of the mill of the present invention in which an ultrasonically vibrating horn is used.

FIG. 6 shows an alternate embodiment of the mill of the present invention in which one or more self-resonating elements are used.

FIG. 6A shows a detailed view of the self-resonating elements of FIG. 6.

FIG. 7 shows an exemplary computer system used to implement the mill and data control system of the present invention.

FIG. 8 shows a graph of the product size distribution resulting from use of the mill of the present invention for processing anthracite.

FIG. 9 shows an alternate embodiment of a slurry nozzle of the present invention.

FIG. 10 shows alternate embodiments of slurry nozzles of the present invention.

FIGS. 11A and 11B show alternate embodiments of a collider of the present invention.

FIG. 12 shows an alternate embodiment of the mill wherein cavitation is created by electronically controlled valves.

FIG. 13 shows an alternate embodiment of the mill wherein cavitation is created by a series of nozzles.

FIG. 14 shows an alternate embodiment of the mill in a vertical configuration.

FIG. 15 shows an alternate embodiment of the mill including a spray dryer.

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FIG. 16 shows an embodiment of a spray dryer equipped with a collector and condenser.

FIG. 17 shows another embodiment of FIG. 15, including a hydrocyclone.

#### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

A preferred embodiment of the present invention is now described with reference to the figures where like reference numbers indicate identical or functionally similar elements. Also in the figures, the left most digit or digits of each reference number corresponds to the figure in which the reference number is first used. While specific configurations and arrangements are discussed, it should be understood that this is done for illustrative purposes only. A person skilled in the relevant art will recognize that other configurations and arrangements can be used without departing from the spirit and scope of the invention.

FIG. 1 shows a first embodiment of a high-pressure mill 100 for processing materials into ultra-fine particles. Mill 100 includes a first chamber 102, nozzle chambers 104 and 108, a second chamber 106, and a third chamber 110. In one embodiment, chambers 102, 106 and 110 each have a length (measured from inlet to outlet) in the range of 1–20 inches and a diameter in the range of 0.25–10 inches. However, it would be apparent to one skilled in the relevant art that various other sizes and configurations of chambers 102, 106 and 110 could be used to implement mill 100 of the present invention.

First chamber 102 includes an inlet 112. The material to be processed is fed into first chamber 102 via inlet 112. In this embodiment, a funnel 114 is disposed above inlet 112 to facilitate loading of the material to be processed into first chamber 102. In an alternate embodiment, inlet 112 could be connected via a port to an outlet of another similar mill, so that the particles exiting a first mill could be pumped into a second stage mill to achieve further comminution of the particles. The second stage mill could be designed with the same chambers and features as the first mill, however, the nozzle sizes would be smaller than the first mill to accommodate the reduced size of the particles.

The entire interior of each chamber is coated with a thin layer of a material. Preferably, the material used for the coating is made from a material with the same chemical composition as the material that is being processed. For example, when treating anthracite, the interior surfaces of each chamber can be coated by thin diamond layer, which creates a very thin, durable coating that is very hard and has the same chemical composition as anthracite. The coating may be applied by a process called chemical vapor deposition, which is well known in the art of coatings, or any other coating process that would be apparent to one skilled in the relevant art. The purpose of the coating is to reduce potential contamination by the material of the mill construction. When the high-pressure slurry jets contact the interior surfaces of the mill, any material that is dislodged from the mill will have the same composition as the material being processed.

As the particles are passed through the mill, the volume of fluid in the slurry increases, thereby decreasing the comminution effect of the fluid jets. As such, in another embodiment, the slurry exiting mill 100 could be processed in a centrifuge to eliminate the excess fluid and make the slurry more concentrated before it is fed into the second stage mill, as described above. Alternatively, the particles could be completely dried and introduced again into the mill 100 in a dry state.

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In one embodiment, the material to be processed is anthracite, commonly known as coal, having a starting size, also referred to as a feed size, of 600–1,200 microns. Although this is a preferable range for the feed size, the feed size could be less than 600 microns and could be as high as 0.5 inches.

It would be apparent to one skilled in the relevant art that the present mill **100** could be used to process a variety of other materials, both organic and inorganic, having various feed sizes. For example, the mill of the present invention could be used to process any of the following: silica carbides for abrasive use; various silica compounds for high density ceramics; garnet for abrasive and cutting uses; alumina for abrasive and structural ceramic uses; coke and coke by-products; metal powders such as magnetite, zinc, copper, brass and nickel; mica; vermiculite; silicon dioxide; carbon black; and any other brittle material that needs to be finely ground. Further, the mill of the present invention could be used to process a variety of organic materials, including, for example: wood, food products and products for use as pharmaceuticals.

In one embodiment, the material particles are dry as they are fed into first chamber **102**. In another embodiment, the material particles could be fed into first chamber **102** as part of a slurry, e.g., a mixture of material particles and a fluid.

It would be apparent to one skilled in the relevant art that the present mill **100** could be used with a variety of fluids, such as water or oil. Preferably, a fluid used in the mill will be able to penetrate the microcracks in the material being treated. The ideal fluid for use in the mill has the following properties: low viscosity for penetrating the crack of the material to be processed; high density for better impaction; low boiling point (50° C. or 106° F.) for easier separation of the fluid and solid; non-toxic; and not harmful to the environment. An example of fluids meeting these requirements are certain perfluoro carbons, available from Minnesota Mining and Manufacturing Company (3M) of Maplewood, Minn. Other fluids that could be used in the mill include: water; oil; cryogenic liquids including cryogenic carbon dioxide; liquified gases including liquid carbon dioxide and liquid nitrogen; alcohol; silicone-based fluids including perfluoro carbon fluids; supercritical fluids including carbon dioxide or inert gas such as xenon or argon in a supercritical state; or organic solvents.

First chamber **102** further includes a high-pressure fluid jet nozzle **116** that creates a fluid jet using a pump (not shown). Fluid jet nozzle **116** preferably creates a water jet, however, it would be apparent to one skilled in the relevant art that other fluids could also be used. The fluid jet generated by nozzle **116** is configured in first chamber **102** such that the jet of fluid exiting from fluid jet nozzle **116** impacts or collides with the material particles after they enter inlet **112** to effect comminution of the material. The pump is designed for a particular volume discharge and a particular pressure. In the example of processing coal, the nozzle diameter is preferably in a range between 0.005 to 1 inches, and more preferably in the range of 0.005 to 0.060 inches. The nozzle diameter is directly related to the pressure of the fluid and the volume discharge generated by the pump. As such, the range of nozzle diameters described above is suitable for a pressure range of fluid of 100, 000–150,000 psi, respectively.

It would be apparent to one skilled in the art that the nozzle diameter could be larger than the above-mentioned range, depending on the size of the pump used to create the available pressure range for the fluid jet. As such, as the

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amount of pump pressure capable of being achieved increases, the diameter of the nozzle can be increased, in relation thereto, when the volume of the fluid supply is sufficient.

In this embodiment, the nozzle of high-pressure fluid jet nozzle **116** is configured to emit a jet of fluid in the general direction of nozzle chamber **104**. One or more fluid jet nozzles **116** can be disposed in first chamber **102**. If more than one fluid jet nozzle **116** is used, the plurality of fluid jet nozzles can be arranged in a straight line through first chamber **102**, thereby directing each jet of fluid toward nozzle chamber **104**. In one embodiment, the fluid jets from the multiple nozzles are arranged so that the jets are emitted substantially in parallel to each other. In an alternate embodiment, the fluid jets are designed to converge with each other. As the jet(s) of fluid impact the material, the particles are broken into smaller particles, and the slurry, i.e., the combination of the smaller particles and fluid, is forced into nozzle chamber **104**.

Nozzle chamber **104** includes a primary slurry nozzle **118**. Primary slurry nozzle **118** creates a jet of the slurry, and delivers the slurry jet into second chamber **106**. Primary slurry nozzle **118** further creates turbulence in second chamber **106**, which causes the smaller particles of the material to interact with each other and comminute further. In one embodiment, primary slurry nozzle **118** has a diameter in a range of 0.010–1 inch, and preferably within a range of 0.010–0.250 inches. The size of nozzle **118** is directly related to the size of fluid jet nozzle **116**. As such, as the size of fluid jet nozzle **116** increases, so does the resultant size of slurry nozzle **118**.

In one embodiment, nozzle chamber **104** further includes a cavitation nozzle **122**. Cavitation nozzle **122** is shown in further detail in FIG. 2. As shown in FIG. 2, cavitation nozzle **122** has a channel **202** through which high velocity fluid flows. Cavitation nozzle **122** further includes an inner pin **204**. In use, a hydrodynamic shadow is created in front of inner pin **204** that creates a pocket in which the flow is not continuous. Evaporation occurs in this pocket which creates cavitation bubbles in the fluid as it exits cavitation nozzle **122**.

Cavitation nozzle **122**, as shown in FIG. 1, is disposed adjacent second chamber **106**. As such, as the slurry is passed through primary slurry nozzle **118** and into second chamber **106**, the cavitation bubbles from the fluid exiting cavitation nozzle **122** implode and generate a local shock wave initiated from the center of each collapsing bubble in the whole volume of second chamber **106**. The shock wave acts on the particles in the slurry and causes them to comminute further. As such, the particle size of the material entering second chamber **106** via an inlet **124** is larger than the particle size as the particles exit second chamber **106** via an outlet **126**.

A secondary slurry nozzle **120** is disposed adjacent outlet **126** of second chamber **106**. Secondary slurry nozzle **120** creates a second jet of slurry as it passes through the nozzle. In one embodiment, the diameter of secondary slurry nozzle **120** is within a range of 0.010–1 inch, and preferably within a range of 0.010–0.250 inches. Again, as discussed above with respect to primary slurry nozzle **118**, the size of secondary slurry nozzle **120** is also related directly to the size of the high-pressure fluid jet nozzle **116**.

Various embodiments of slurry nozzles are shown in FIGS. 9 and 10. In particular, FIG. 9 shows an embodiment of a slurry nozzle **902** that has an inlet **904** and an outlet **906**, where the diameter of inlet **904** is larger than the diameter

of outlet **906**. Further, an inner surface **910** of slurry nozzle **902** has sharp edges **908** that project slightly out from the inner surface. In this embodiment, sharp edges **908** are formed as rings and are disposed at intervals around inner surface **910** of slurry nozzle **902**. As the particles travel through slurry nozzle **902**, they hit one or more of the sharp edges **908**, which causes further comminution of the particles.

FIG. **10** shows various possible embodiments of channel design for the slurry nozzles used in the present invention. In a first slurry nozzle **1002**, an inlet **1004** has a diameter larger than an outlet **1006**, similar to nozzle **902** of FIG. **9**. In a second design, slurry nozzle **1008** has an inlet **1010** with a diameter which is smaller than the diameter of its outlet **1012**. A third slurry nozzle **1014** has an inlet **1016** and an outlet **1018** of approximately the same diameter, however, the inner surface of nozzle **1014** gradually tapers out from inlet **1016** toward a center point **1020** and then gradually tapers back in from center point **1020** toward outlet **1018**. A fourth slurry nozzle **1022** also has an inlet **1024** and an outlet **1026** of approximately the same diameter. In this embodiment, the inner surface of nozzle **1022** gradually curves inwardly from inlet **1024** toward a center point **1028**, and then gradually curves back outwardly from center point **1028** to outlet **1026**. It would be apparent to one skilled in the relevant art that various other nozzle designs could also be used to implement the present invention.

The slurry jet emitted from secondary slurry nozzle **120** is directed toward third chamber **110**. A collider **128**, which also could be referred to as a “stopper” or “energy absorber,” is disposed in third chamber **110** directly in the path of the slurry jet. Collider **128** can be a stable collider, such as the screw mechanism shown in FIG. **1**. Alternatively, collider **128** could be an ultrasonically vibrating collider **502**, as shown in FIG. **5**. Ultrasonically vibrating collider **502** can be configured to have a vibration within a range of up to 20,000 Hz or higher. In one embodiment, ultrasonic vibrating collider **502** is the XL2020 Generator, available from Misonix Incorporated, Farmingdale, N.Y. In either embodiment, the position of collider **128** within third chamber **110** is preferably adjustable so that the collider can function to restrict the flow out of secondary slurry nozzle **120** and into third chamber **110**. This flow restriction causes increased turbulence to occur in second chamber **106**, which further aids in the comminution of the particles.

Two embodiments of colliders are shown in FIGS. **11A** and **11B**. In the embodiment of FIG. **11A**, collider **1102** has a front surface **1104** which is the surface that the slurry impacts. In this first embodiment, front surface **1104** is flat. In this embodiment, the slurry exits nozzle **120** and collides with flat front surface **1104**. In a second embodiment shown in FIG. **11B**, collider **1106** has a front surface **1108** that is concave in the shape of an inverted cone. In this embodiment, as the slurry exits nozzle **120** and collides with front surface **1108**, the concave shape causes the particles to bounce off and collide with each other and/or collide with other areas of front surface **1108** to thereby cause further comminution of the particles. It would be apparent to one skilled in the art that the front surface **1108** could be formed in a variety of concave-like shapes to cause the same effect. For example, a hole could be formed in front surface **1108** to cause the particles to further comminute.

In either embodiment, the slurry jet from secondary slurry nozzle **120** directly collides with collider **128** to effect additional comminution of the particles of material in the slurry. As discussed above, the position of collider **128** is preferably positionable at various distances away from sec-

ondary slurry nozzle **120**. This distance,  $D$ , is shown in FIG. **5** and marked with reference number **504**. As collider **128** is moved closer to the flow of slurry exiting from slurry nozzle **120**, i.e., as  $D$  decreases, the flow becomes more restricted. This restricted flow causes turbulence in second chamber **106**, which assists with comminution of the particles in that chamber.

Although mill **100** is described with respect to FIG. **1** as an example, mill **100** could be used to achieve the desired particle size without the use of cavitation nozzle **122**. An alternate embodiment of a mill **1200** is shown in FIG. **12**. In this embodiment, electronically controlled valves are used instead of a nozzle to create cavitation inside second chamber **106**. In particular, a first valve **1204** is disposed at an inlet to second chamber **106** and a second valve **1208** is disposed at an outlet to second chamber **106**. Cavitation can be induced in second chamber **106** by creating a pressure differential between the pressure in primary nozzle **118** and the pressure in second chamber **106** of approximately 100:1. Depending on the distance  $D$  between collider **128** and secondary slurry nozzle **120**, the flow restriction may cause such a pressure differential, which will in turn cause cavitation to be induced in second **106**. Electronically controlled valves **1204** and **1208** on the inlet and outlet of second chamber **106** are connected to pressure sensors **134**. These valves can be used to change the size of the valve orifice to maintain the pressure differential in second chamber **106**.

Third chamber **110** further has an outlet port **130** disposed at the bottom of the chamber. After the collision between the slurry and collider **128**, the slurry flows to the bottom of third chamber **110** and exits via outlet port **130**. The mill **100** of the present invention is designed to achieve ultra-fine particles having a resultant size, also referred to as a product size, of less than 15 microns. Preferably, the ultra-fine particles have a product size within a range of 1–5 microns. More preferably, the ultra-fine particles have a product size within a range of 150 nanometers to 1 micron.

In an alternate embodiment, comminution of the material can be achieved using different combinations of the nozzles and chambers discussed above. For example, in one embodiment, comminution can be achieved using only first chamber **102**, primary slurry nozzle **118** and third chamber **110**. In an alternate embodiment, comminution can be achieved using only first chamber **102**, secondary slurry nozzle **120** and third chamber **110**. In another embodiment, multiple nozzles can be used in lieu of primary slurry nozzle **118**. The use of multiple nozzles in any portion of mill **100** will create more turbulence in the chambers of the mill thereby further increasing the size reduction factor, i.e., the ratio of the feed size of the particles to the product size of the resultant particles, of the mill. In a further embodiment, a self-resonating device **602**, as shown in FIG. **6**, can be placed throughout mill **100**. In the embodiment shown in FIG. **6**, beams **604** and **606** of self-resonating device **602**, shown in FIG. **6A**, are disposed at a certain distance apart from one another and configured to have a self-resonating frequency, such that the amplitude of the movement of beams **604** and **606** will contribute to the comminution process. It would be apparent to one skilled in the relevant art that two or more such beams could be positioned around a center line to create self-resonating device **602**.

In the example shown in FIG. **6**, self-resonating devices **602** are disposed in first chamber **102** and in front of primary slurry nozzle **118**. However, it would be apparent to one skilled in the relevant art that these devices could be placed in a variety of locations in mill **100** to aid in comminution.

In one embodiment, mill **100** may be fitted with sensors to monitor the comminution process, as will be discussed in

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further detail below with respect to FIG. 4. For example, temperature sensors 132, pressure sensors 134, and sound sensors 136 may be disposed in various areas of each chamber of mill 100. By way of example, these sensors are shown placed in various positions within mill 100 in FIG. 1. For example, temperature sensors 132 are shown disposed in front of nozzle 116, in front of primary slurry nozzle 118, in second chamber 106, and in third chamber 110. Similarly, pressure sensors 134 are disposed in front of nozzle 116, in front of primary slurry nozzle 118 and in second chamber 106, and sound sensors 136 are disposed adjacent the inlet 124 and outlet 126 of second chamber 106. The pressure sensors 134 controlling the cavitation action in the chamber can be linked to a centralized data control system 400. An embodiment of this data control system for the mill of the present invention will be discussed in further detail with respect to FIG. 4.

Temperature and pressure can be measured merely to collect data to keep track of the temperature ranges that occur during the comminution process and to ensure that the pressure created by the various nozzles is sufficient to result in the ultra-fine particles. The sound is measured in second chamber 106 to obtain a reading of how intense the comminution process is in the cavitation chamber. In particular, the frequency of the sound that occurs in this chamber is measured. Typically, the frequency emitted depends on the conditions when cavitation is induced. Frequencies are generally within the range of 10–1000 KHz. In an alternate embodiment, mill 100 can be used in a production line to comminute the material in mass volume. In such a case, the data from the sensors can be fed back to a computer-controlled mill to control the comminution process.

Another embodiment of a mill 1300 is shown in FIG. 13. In this embodiment, cavitation is created in a second chamber by a series of nozzles. Second chamber 106 made up of multiple nozzles 1302 arranged in a series. The nozzles 1302 may be all the same size and shape or may be a variety of diameters and shapes. In a preferred embodiment, the nozzles are made of carbide. As the fluid flows through the nozzles 1302, a pressure drop occurs in the larger diameter portion of the nozzles 1302. The sudden reduction in pressure causes cavitation bubbles to form, introducing cavitation into the comminution process.

Another embodiment of a mill 300 is shown in FIG. 3. Mill 300 has a first chamber 302 and a second chamber 304 disposed on opposite ends of a third chamber 306. First chamber 302, similar to first chamber 102, has an inlet 308, a funnel 310, and a high-pressure fluid jet nozzle 312. As described previously in FIG. 1 as the particles of the material travel down funnel 310 and enter first chamber 302 via inlet 308, the fluid jet from nozzle 312 collides or impacts with the particles, thereby breaking them apart. The fluid jet nozzle 312 is oriented in first chamber 302 such that the slurry passes through first chamber 302 and into a nozzle chamber 320.

Nozzle chamber 320 contains a first slurry nozzle 324. First slurry nozzle 324 creates a fluid jet of the slurry created in first chamber 302. Similarly, second chamber 304 includes an inlet 314, a funnel 316, and a fluid jet nozzle 318. The same process occurs in second chamber 304 in which the particles travel down funnel 316 through inlet 314 and are impacted by a jet of fluid from nozzle 318. The slurry from second chamber 304 passes through to a nozzle chamber 322. Nozzle chamber 322 includes a second slurry nozzle 326, which creates a jet from the slurry produced in second chamber 304. The jets from first and second slurry nozzles 324 and 326 are disposed such that they collide with

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each other in a high velocity collision within third chamber 306. This collision causes further comminution of the particles. The slurry then falls to the bottom of third chamber 306 and exits via an outlet 328. Temperature, pressure and sound sensors, similar to those discussed with respect to mill 100 in FIG. 1, can also be used in mill 300 to acquire data and control the comminution process.

FIG. 4 shows a mill and data control system 400 of the present invention. The mill of system 400 is similar to mill 100 in that it includes a first chamber 102 in which particles are impacted by a high-pressure fluid jet generated by nozzle 116, a nozzle chamber 104, a second chamber 106 in which cavitation occurs, a second nozzle chamber 108, and a third chamber 110 in which the particles impact a collider for further comminution.

Another embodiment of a mill 1400 is shown in FIG. 14. Mill 1400 is vertically configured and includes a primary nozzle 1404, a first chamber 1408, a secondary nozzle 1410, a catcher 1412, an overflow nozzle 1414, and an overflow channel 1416. Secondary nozzle 1410 could be a single nozzle, as shown, or could be multiple nozzles arranged in series as described and shown with reference to FIG. 13. The material to be processed is fed into first chamber 1408. In this embodiment, a funnel 1402 facilitates loading of the material to be processed into first chamber 1408 and into the mill. As in a previous embodiment, the particles may be fed into the mill dry or as part of a slurry. Primary nozzle 1404 is a high-pressure fluid jet nozzle. The fluid from primary nozzle 1404 collides with the particles fed into first chamber 1408 from funnel 1402.

Primary nozzle 1404 is configured to emit a stream of fluid through the first chamber 1408 and through the secondary nozzle 1410. The secondary nozzle 1410 has a significantly larger diameter than primary nozzle outlet 1406 to allow the stream to flow through it. After the slurry flows through secondary nozzle 1410, it flows into the catcher 1412 through overflow nozzle 1414, where the churning action created by the fluid jet comminutes the particles.

The use of the catcher 1412 in this embodiment rather than the collider 128 in the earlier discussed embodiment helps to prevent contamination by the material of the collider. The jet formed by secondary nozzle 1410 and directed toward catcher 1412 allows the slurry from the catcher 1412 to exit back up through overflow nozzle 1414 as catcher 1412 fills and overflows. The slurry escapes through a space in the periphery of nozzle 1410. The amount and rate of outflow from the catcher 1412 can be controlled by adjusting the size of overflow nozzle 1414. As a result, the amount of comminution of the particles can be increased or decreased by adjusting the amount of time the particles are held in catcher 1412.

After the slurry backflows through overflow outlet 1414, it flows through the periphery of nozzle 1410 and into an overflow channel 1416 where it exits mill 1400 through outlet port 1418.

Other embodiments of the mills described include a hydrocyclone and/or a spray dryer. A specific embodiment is shown in FIG. 15, where system 1500 includes a high pressure slurry pump 1502, connected to a high pressure mill 1504. Mill 1504 has attached a feed pump 1506 for introducing particles into a spray dryer 1508. Connected to spray dryer 1508 is a condenser 1510 and a collector 1512. A recycling circuit 1514 connects condenser 1510 to high pressure slurry pump 1502. However, it would be apparent to one skilled in the relevant art that various configurations of these elements could be used to implement system 1500.

of the present invention. High pressure mill **1504** outputs a slurry containing comminuted particles of a material and the energy transfer fluid. If an additive was introduced into the high pressure mill, the output will include the comminuted material, the energy transfer fluid and the additive. As would be apparent to one skilled in the relevant art, the material and the additive could be comprised of more than one material or additive.

As shown in FIG. **16**, spray dryer **1508** is attached to feed pump **1506**, and is comprised of atomizing components, such as a nozzle **1604** and a heating chamber **1606**. Typically, a spray dryer mixes a spray and a drying medium, such as air, to efficiently separate the particles from the fluid as the particles fall through the air.

There are four general stages to spray drying: atomizing, mixing, drying, and separation. First, the feed or slurry is atomized into a spray. This is accomplished by introducing the slurry to feed pump **1506**, which forces the slurry through atomizing nozzle **1604**. The energy required to overcome the pressure drop across the nozzle orifice is supplied by feed pump **1506**.

Second, the spray is mixed with a drying medium, such as air. Air can be added through a blower via nozzle **1604**, via an additional nozzle, or can be merely present in chamber **1606**. As would be apparent to one skilled in the relevant art, other drying mediums could be introduced in spray dryer **1508**. For instance, when the fluid, additive, or material is oxygen sensitive, inert gases such as nitrogen can be introduced as the drying medium. If a gas is added through a blower, the gas can be injected into chamber **1606** simultaneously with the atomized slurry. A conventional method of introducing gas and slurry simultaneously uses concentric nozzles, where one nozzle introduces gas and the other nozzle introduces slurry.

Third, the spray is dried. Drying occurs as the atomized spray is subjected to a heat zone in chamber **1606** or, alternatively, a hot gas, such as air or an inert gas as described above, is injected into chamber **1606**. Flash drying quickly evaporates the fluid from the slurry, leaving only the dry particles. The small size of droplets allows quick drying, requiring a residence time in the heat zone ranging from 1–60 seconds, depending on the application. This short residence time permits drying without thermal degradation of the solid material.

Fourth, the product is separated from the gas. As the particles continue to fall, they exit chamber **1606**, accumulating in particle collector **1512**, located at the bottom of chamber **1606**. The now vaporized fluid is exhausted, or alternatively, collected in condenser **1510**. The spray dryer by-products are vaporized fluid and dry particles.

Using a spray dryer in connection with a high pressure mill provides several advantages over conventional drying techniques. For instance, spray drying produces an extremely homogeneous product from multi-component solids/slurries. A spray dryer can evaporate the energy transfer fluid from the slurry, leaving an additive, if used, and material. If the additive is a fluid, drying temperatures are held below the degradation temperature of the binder. As the energy transfer fluid evaporates, a very thin coating of binder polymerizes on each particle. After being dried in the spray dryer, the particles are sufficiently coated for molding into compacts for sintering. Additional processing is not necessary.

Furthermore, the resulting collected particles are fine, dry and fluffy. Conventional techniques, such as boiling the vapor off the particles, leave clumpy conglomerates of

particles and result in less thorough blending of additives. The spray dryer also dries particles much faster than drying by conventional techniques. A spray dryer quickly dries a product because atomization exposes all sides of the particles to drying heat. The particles are subjected to a flash dry, and depending on the application, can be dried anywhere between 3 and 40 seconds. Thus, heat sensitive particles can be quickly dried without overheating the particles. As drying begins, the vaporized fluid forms around the particle. This “protective envelope” keeps the solid particle at or below the boiling temperature of the fluid being evaporated. As long as the evaporation process is occurring, the temperature of the solids will not approach the dryer temperature, even though the dryer temperature is greater than the fluid evaporation temperature.

An additional advantage is that the spray dryer can operate as part of a continuous process providing dry particles as they are collected, rather than having to collect particles and then dry them. This also allows for fast turnaround times and product changes because there is no product hold up in the drying equipment.

The volume of an acceptable chamber **1606** can be determined by the equation, (residence time) \* (volume flow rate) = volume of chamber, where volume flow rate is the throughput. Additionally, because of a larger surface area per unit mass, finer particles normally require longer residence time to dry than larger particles. Therefore, residence time may be longer for the finer materials. Also, materials having hygroscopic properties will require a longer residence time in chamber **1606**. Increased temperature may also be used to accelerate drying of such materials.

The spray dryer can be used for drying any slurry, whether the slurry is comprised of particles of a material, an additive, and an energy transfer fluid or comprised of only particles of a material and an energy transfer fluid. Further, the spray dryer can be a standard spray dryer, known in the art of spray drying. Spray dryer manufacturers and vendors include companies such as U.S. Dryer Ltd. of Migdal Ha'emek, Israel, Niro, Inc. of Columbia, Md., APV of Rosemont Ill., and Spray Drying Systems, Inc. of Randallstown, Md.

A conventional spray dryer can be outfitted with condenser **1510**. Because all drying takes place in an enclosed chamber **1606**, capture and condensation of the vapors is easily performed. Condenser **1510** collects the vaporized fluid from chamber **1606** and allows the spent fluid to be recovered. Thus, spray drying offers a simple way to contain the vapors from the evaporated fluid. Fluid recycling circuit **1514**, as shown in FIG. **15**, can connect condenser **1510** to high pressure slurry pump **1502** located at the first chamber of the high pressure mill. This allows condensed fluid to be recycled by returning the used fluid from the spray dryer to the high pressure mill. This reduces waste and contains the fluid, which is especially important when the fluid is a regulated product, such as isopropanol. Isopropanol can be used as the fluid in the high pressure mill, introduced into the spray dryer where it is vaporized, recondensed in the condenser and returned to the high pressure mill for reuse. In this way, the fluid vapors are contained without risk of releasing harmful vapors into the atmosphere.

If the fluid is water, the water can be released from the spray dryer as vapor, can be condensed to be discarded, or can be recycled through the fluid recycling circuit. As described above, a variety of fluids could be used as the energy transfer fluid in the mill.

In another embodiment, the slurry is introduced from the high pressure mill directly into the spray dryer. This embodi-



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ment does not use a feed pump connected to the nozzle for atomizing. Instead, fluid restrictors are used at the high pressure mill outlet port to maintain the high pressures in mill 100. The slurry bypasses feed pump 1506 and is injected directly from the outlet of mill 100 into spray dryer 1508. In order to achieve proper separation of particles and fluid in spray dryer 1508, the slurry jet at the outlet of mill 100 must have sufficient speed to enter dryer 1508 to achieve complete atomization of the slurry. By eliminating the need for a feed pump to introduce the slurry to the spray dryer, the system operates more economically.

FIG. 17 shows another embodiment of system 1700 for comminution, blending and processing materials into particles. This embodiment includes a hydrocyclone 1710 located between mill 1704 and feed pump 1506. Hydrocyclone 1710 can be located either before or after feed pump 1506, but is preferably located before it. A second feed (not shown) can be used to introduce slurry from mill 1700 to hydrocyclone 1710, or, the slurry can be introduced into hydrocyclone 1710 directly from mill 1704, as shown in FIG. 17.

Hydrocyclone 1710 aids in classifying solid particles exiting high pressure mill 1704 by separating very fine particles from coarser particles. The coarser particles are fed through a recycling line 1514 back into high pressure slurry pump 1502, to be reintroduced into mill 1704 for further comminution and processing. As the particles are still under pressure from hydrocyclone 1710, recycling line 1514 is a tube or enclosed circuit, which transfers the particles to mill 1704.

The slurry from mill 1704 enters the hydrocyclone 1710 at high velocity through an inlet opening and flows into a conical separation chamber. As the slurry swirls downward in the chamber, its velocity increases. Larger particles are forced against the walls, dropped to the bottom, and discharged through a restricted discharge nozzle into recycle line 1514. The spinning forms an inner vortex which lifts and carries the finer particles up the hydrocyclone 1710, before they exit the discharge nozzle, and propel them through a forward outlet to feed pump 1506 or, alternatively, directly to spray dryer 1508.

In another embodiment, hydrocyclone 1710 is a dry-type cyclone, located after spray dryer 1508. In this embodiment, particles are dried in spray dryer 1508 and gathered in collector 1512. The dry particles are introduced from collector 1512 into cyclone 1710, where the particles are sorted according to size. Cyclone 1710 operates substantially similar to the hydrocyclone described above, using a gas as the fluid. Again, oversized particles are reintroduced into high pressure mill 1704 or high pressure slurry pump 1502 via recycling line 1514. Because gases normally have less surface tension than fluids, dry separation normally results in finer and more accurate size distribution.

Hydrocyclone 1710 can be a commercially available hydrocyclone used for classification, clarification, counter-current washing, concentration, etc., of particles. Examples of hydrocyclone and cyclone manufactures are Warman International, Inc. of Madison, WI (CAVEX® Hydrocyclone Technology), Polytech Filtration Systems, Inc., of Sudbury, MA (POLYCLON® Hydrocyclone Technology), and Dorr-Oliver, Inc., of Milford, CT (DORRCLONE® HYDROCLONES).

Because hydrocyclone 1710 recycles the larger or more coarse fraction of material back to mill 1704 for further size reduction, hydrocyclone 1710 assists in achieving a narrow size distribution of finished particles. Furthermore, hydro-

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cyclone 1710 offers more intimate mixing of the particles and additives. Residence time in hydrocyclone 1710 is typically short, and is a function of the processing rate, and the equipment size (volume). Thus, residence time = equipment volume/processing rate (volume/time). Typically, the residence time in hydrocyclone 1710 is less than 60 seconds, and is preferably from 2–50 seconds. Thus, use of hydrocyclone 1710 does not restrict the processing rate achievable in mill 1704 and subsequent spray dryer 1508.

Depending on the size and capability of the hydrocyclone, residence time will vary for a given processing rate. Therefore, a properly sized hydrocyclone must be used to efficiently comminute, blend and process particles. An improperly sized hydrocyclone could impose limits on the residence times in other components of system 1700.

Referring back to FIG. 4, temperature sensor 132, pressure sensor 134 and sound sensor 136 are shown disposed in second chamber 106 of mill 100. In one embodiment, sensors 132, 134 and 136 are implemented using various transducers, thermocouples and user input, as would be apparent to one skilled in the relevant art.

Data collected by each of these sensors are fed into a signal conditioning module 402. In one embodiment, signal conditioning module 402 is a signal conditioner/isolator available from Omega Engineering, Stamford, Conn. Signal conditioning module 402 converts the signals transmitted from the sensors 132, 134 and 136 into a computer-readable format and passes them to data acquisition (DAQ) card 404. In one embodiment, DAQ card 404 is a data acquisition card available from National Instruments Corporation, Austin, Tex. The DAQ card 404 can be inserted or disposed in a PCMCIA slot 406 of a processor 408. Processor 408 processes the signals to acquire data regarding the comminution process. In one embodiment, processor 408 is running LabView software that enables the user to view, store and/or manipulate the data received from the sensors to be used as control parameters in the control system.

It would be apparent to one skilled in the relevant art that the present invention may be implemented using hardware, software or a combination thereof and may be implemented in a computer system or other processing system. In fact, in one embodiment, the invention is directed toward one or more computer systems capable of carrying out the functionality described herein. An example of a computer system 700 is shown in FIG. 7. The computer system 700 includes one or more processors, such as processor 408. Processor 408 is connected to a communication infrastructure 706 (e.g., a communications bus, cross-over bar, or network). Various software embodiments are described in terms of this exemplary computer system. After reading this description, it will become apparent to a person skilled in the relevant art how to implement the invention using other computer systems and/or computer architectures.

Computer system 700 can include a display interface 702 that forwards graphics, text, and other data from the communication infrastructure 706 (or from a frame buffer not shown) for display on the display unit 730.

Computer system 700 also includes a main memory 708, preferably random access memory (RAM), and may also include a secondary memory 710. The secondary memory 710 may include, for example, a hard disk drive 712 and/or a removable storage drive 714, representing a floppy disk drive, a magnetic tape drive, an optical disk drive, etc. The removable storage drive 714 reads from and/or writes to a removable storage unit 718 in a well-known manner. Removable storage unit 718, represents a floppy disk, mag-

netic tape, optical disk, etc. which is read by and written to by removable storage drive 714. As will be appreciated, the removable storage unit 718 includes a computer usable storage medium having stored therein computer software and/or data.

In alternative embodiments, secondary memory 710 may include other similar means for allowing computer programs or other instructions to be loaded into computer system 700. Such means may include, for example, a removable storage unit 722 and an interface 720. Examples of such may include a program cartridge and cartridge interface (such as that found in video game devices), a removable memory chip (such as an EPROM, or PROM) and associated socket, and other removable storage units 722 and interfaces 720 which allow software and data to be transferred from the removable storage unit 722 to computer system 700.

Computer system 700 may also include a communications interface 724. Communications interface 724 allows software and data to be transferred between computer system 700 and external devices. Examples of communications interface 724 may include a modem, a network interface (such as an Ethernet card), a communications port, a PCMCIA slot and card, etc. Software and data transferred via communications interface 724 are in the form of signals 728 which may be electronic, electromagnetic, optical or other signals capable of being received by communications interface 724. These signals 728 are provided to communications interface 724 via a communications path (i.e., channel) 726. This channel 726 carries signals 728 and may be implemented using wire or cable, fiber optics, a phone line, a cellular phone link, an RF link and other communications channels.

In this document, the terms "computer program medium" and "computer usable medium" are used to generally refer to media such as removable storage drive 714, a hard disk installed in hard disk drive 712, and signals 728. These computer program products are means for providing software to computer system 700. The invention is directed to such computer program products.

Computer programs (also called computer control logic) are stored in main memory 708 and/or secondary memory 710. Computer programs may also be received via communications interface 724. Such computer programs, when executed, enable the computer system 700 to perform the features of the present invention as discussed herein. In particular, the computer programs, when executed, enable the processor 704 to perform the features of the present invention. Accordingly, such computer programs represent controllers of the computer system 700.

In an embodiment where the invention is implemented using software, the software may be stored in a computer program product and loaded into computer system 700 using removable storage drive 714, hard drive 712 or communications interface 724. The control logic (software), when executed by the processor 704, causes the processor 704 to perform the functions of the invention as described herein.

In another embodiment, the invention is implemented primarily in hardware using, for example, hardware components such as application specific integrated circuits (ASICs). Implementation of the hardware state machine so as to perform the functions described herein will be apparent to persons skilled in the relevant art(s). In yet another embodiment, the invention is implemented using a combination of both hardware and software.

As shown in FIG. 4, a second temperature sensor 132 and pressure sensor 134 are disposed on fluid jet 116 to measure

the temperature and pressure of the fluid as it exits fluid jet 116 and enters first chamber 102. The data from these sensors is also fed into signal conditioning module 402 and processor 408.

A linear variable differential transducer (LVDT) 410 is disposed on one end of collider 128 of third chamber 128. LVDT 410 measures the linear position of collider 128 with respect to the slurry flow as it enters third chamber 110. The data from LVDT 410 are also fed into signal conditioning module 402 and processor 408.

Finally, a particle size sensor 412 is disposed in outlet port 130 of third chamber 110 to measure the final size of the particles after mill processing is complete. The data from particle size sensor 412 are also fed into signal conditioning module 402 and processor 408.

Although system 400 of FIG. 4 is shown as only a data acquisition system, it would be apparent to one skilled in the relevant art, that processor 408 could use the data acquired to control mill processing of the mineral particles. In such an embodiment, a feedback loop would be created between processor 408 and each of the chambers 102, 104, 106, 108 and 110 to control the flow and comminution at each stage of the processing.

For example, the user could select the final particle size to be achieved via computer interface, and the data acquired by processor 408 could be used to vary the pressure of the fluid streams through the nozzles and/or to adjust the position of the flow restrictor with respect to the secondary slurry nozzle. In this way, the data acquired can be used to control and accurately maintain the desired product size of the materials being processed.

#### EXAMPLE

FIG. 8 shows a graph of particle size distribution resulting from use of the mill of the present invention for processing anthracite. In this example, the distribution marked as 802 is based on a feed size of 0.25–0.5 inches. For the distribution marked as 804, the feed size was 0.02–0.05 inches. The test mill included first chamber 102, nozzle chamber 104, second chamber 106 and third chamber 110, as described above with respect to mill 100. The nozzle for high pressure fluid jet 116 had a diameter of 0.012 inches and the primary slurry nozzle of nozzle chamber 104 was 0.045 inches. The fluid pressure for jet 116 used for the particles shown in distribution 802 was 40,000 psi, and the fluid pressure for jet 116 used for the particles shown in distribution 804 was 30,000 psi. Collider 128 in third chamber 110 was in a fully open position.

As shown in the graphs of FIG. 8, for a feed size of 0.25–0.5 inches, the mill of the present invention comminuted approximately 90% of these starting particles to a product size within a range of submicron to 15 microns. For a feed size of 0.02–0.05 inches, the mill of the present invention comminuted approximately 90% of these particles to a product size within a range of submicron to 28 microns.

The mill of the present invention is intended to be used for the comminution of both organic and inorganic materials, including comminution of minerals. In the comminution of certain minerals, such as mica, the resulting particles achieved using the mill of the present invention are in the shape of flakes of minerals. In particular, the mill of the present invention creates flakes or platelets of ultra-fine particles of minerals. The fluid jets cause the fluid to enter the tip of cracks in the minerals, which create tension at the tip. This tension causes the cracks to propagate along the natural plane in the mineral so that small particles of the

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minerals separate into flakes. As such, the present invention provides a unique shape to these particles, viz, the natural smallest particle of the mineral available. Particles generated using other methods which do not incorporate the comminution techniques of the present invention do not result in flakes because they do not take advantage of the natural cracks in the minerals.

The ultra-fine anthracite particles resulting from processing using the mill of the present invention can be utilized in a variety of applications. For example, the resulting anthracite particles can be used in the following applications: electrodes of metallurgical furnaces; graphite and graphite-based products; carbon black; carbon-based hydrogen storage systems; molds and dies for casting; water trays for chemical vapor deposition processing; electrodes for plasma etching; brushes for electric motors; fuel cells plates, catalysts and electrodes; electrodes for EDM; aerospace and naval structural components; meso-phase carbons for lithium-ion batteries; carbon fibers, whiskers, filaments, tapes and composite materials; molecular sieving carbons; carbon fiber reinforced plastics; activated carbons; activated carbon fibers; fullerenes and carbon nanotubes; diamond-like films; organic chemicals including ethylene, propylene, butadiene, benzene, toluene, xylene and methanol; and engineering polymers and engineering plastics including general engineering plastics such as PET, PBT, PAR, high-temperature heat-resistant plastics, fluid crystalline polymers, functional polymers, condensed polynuclear aromatic resins and inorganic/organic polymers.

While a number of embodiments of the present invention have been described above, it should be understood that they have been presented by way of example, and not limitation. It will be apparent to persons skilled in the relevant art that various changes in form and detail can be made therein without departing from the spirit and scope of the invention. Thus the present invention should not be limited by any of the above-described exemplary embodiments, but should be defined only in accordance with the following claims and their equivalents.

What is claimed is:

1. A method for creating ultra-fine particles of a material using high-pressure fluid, comprising:

- (a) placing a material in a first chamber;
- (b) subjecting the material in the first chamber to a high-pressure fluid jet generated by at least one high pressure fluid jet nozzle, whereby at least a portion of the material is divided into particles;
- (c) transferring the particles of the material to a second chamber;
- (d) subjecting the particles of the material to cavitation in the second chamber, whereby at least a portion of the particles are further divided into relatively smaller particles;
- (e) transferring the relatively smaller particles to a third chamber; and
- (f) causing the relatively smaller particles to collide with a collider in said third chamber, whereby at least a

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portion of said relatively smaller particles are divided into ultra-fine particles of the material, wherein said fluid is selected from the group consisting of: water; oil; cryogenic liquids including cryogenic carbon dioxide; liquified gases including liquid carbon dioxide and liquid nitrogen; alcohol; silicone-based fluids including perfluoro carbon fluids; supercritical fluids including carbon dioxide in supercritical state; or organic solvents.

2. The method of claim 1, wherein said ultra-fine particles of the material have a product size less than 15 microns.

3. The method of claim 1, wherein said ultra-fine particles of the material have a product size less than 5 microns.

4. The method of claim 1, wherein said fluid jet is delivered at a pressure within a range of 5,000–150,000 psi.

5. The method of claim 1, wherein a primary slurry nozzle is used in said step (c) to transfer the particles of the mineral to the second chamber.

6. The method of claim 1, wherein a cavitation nozzle is used in said step (d) to attack the particles exiting said primary slurry nozzle.

7. The method of claim 1, wherein a secondary slurry nozzle is used in said step (e) to transfer the relatively smaller particles of the mineral to the third chamber.

8. The method of claim 7, wherein the secondary slurry nozzle directs the output of the relatively smaller particles of the mineral at the collider in the third chamber.

9. The method of claim 1, wherein the material is selected from the group consisting of: solid phase organic and inorganic materials.

10. The method of claim 1, wherein the material is a mineral selected from the group consisting of: anthracite, silica carbides, silica compounds, garnet, alumina, coke, coke by-products, magnetite, zinc, copper, brass, nickel, mica, vermiculite, silicon dioxide, carbon black, zirconia, silica, barium titanate, wollastonite, and titania.

11. A method for creating ultra-fine particles of material using a high-pressure fluid jet, comprising:

- (a) placing a first material in a first chamber;
- (b) subjecting the first material in the first chamber to a high-pressure fluid jet generated by at least one high pressure fluid jet nozzle, whereby at least a portion of the first material is divided into particles;
- (c) placing a second material in a second chamber;
- (d) subjecting the second material in the second chamber to a high-pressure fluid jet generated by at least one other high pressure fluid jet nozzle, whereby at least a portion of the second material is divided into particles;
- (e) creating a first jet of slurry of said particles from said first chamber and creating a second jet of slurry of said particles from said second chamber, such that said first and second slurry jets impact each other in a third chamber, whereby at least a portion of said particles are divided into ultra-fine particles of the material.

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