United States Patent

[11] 3,608,835

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[54] ULTRADISINTEGRATION AND AGGLOMERATION OF MINERALS SUCH AS MICA, PRODUCTS THEREFROM AND APPARATUS THEREFOR
11 Claims, 19 Drawing Figs.

[52] U.S. Cl. 241/4, 241/5, 241/24, 241/39, 241/46.06
[51] Int. Cl. B02c 19/06
[50] Field of Search 241/4, 46, 46.02, 46.04, 46.06, 46.13, 79.1, 97, 266, 38, 20, 27, 5, 39, 24

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Primary Examiner—Donald G. Kelly
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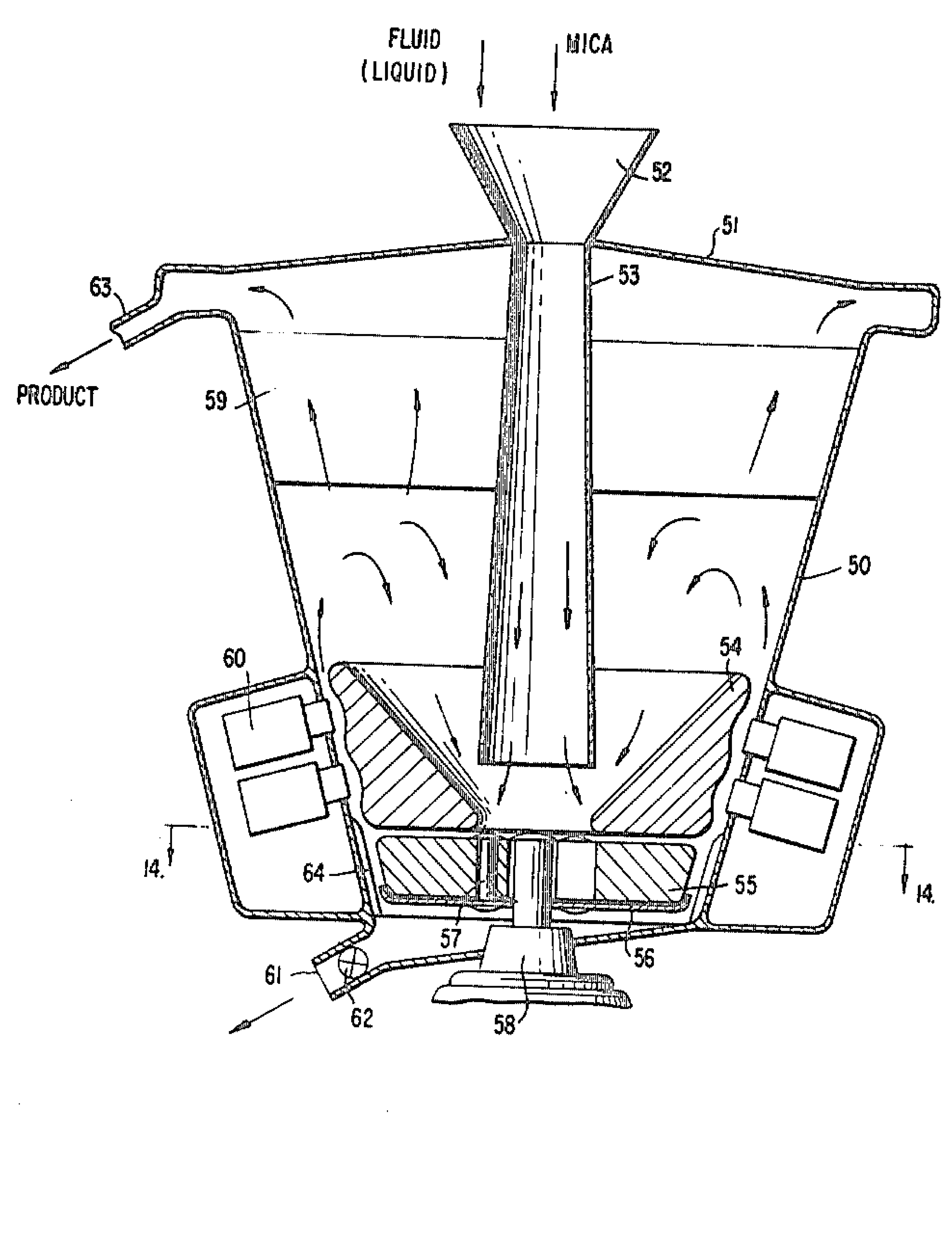
ABSTRACT: Frangible or cleavable solids, such as mica, are disintegrated in oriented, high-velocity streams of a fluid medium so as to produce thin smooth-surfaced particles or flakes having a high specific surface area and a high ratio of length to thickness. The resulting particles or flakes are useful as agglomerants, fillers or pigments or can be agglomerated to form paperlike webs or solid discs or articles of other predetermined configurations, with or without added binder, either in self-supporting form or adhered to a substrate. Various methods and apparatus for such disintegration and agglomeration are also disclosed.



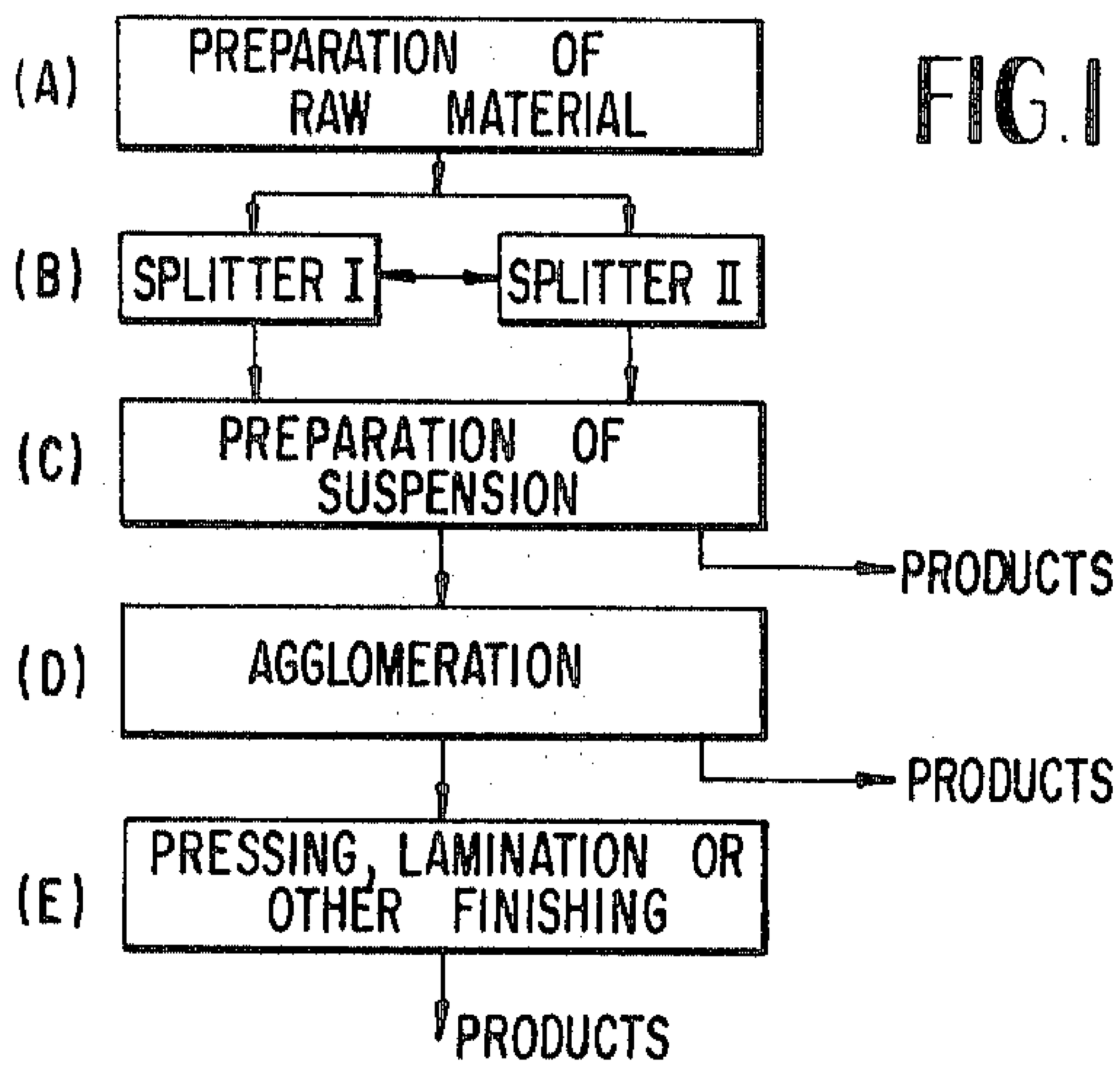
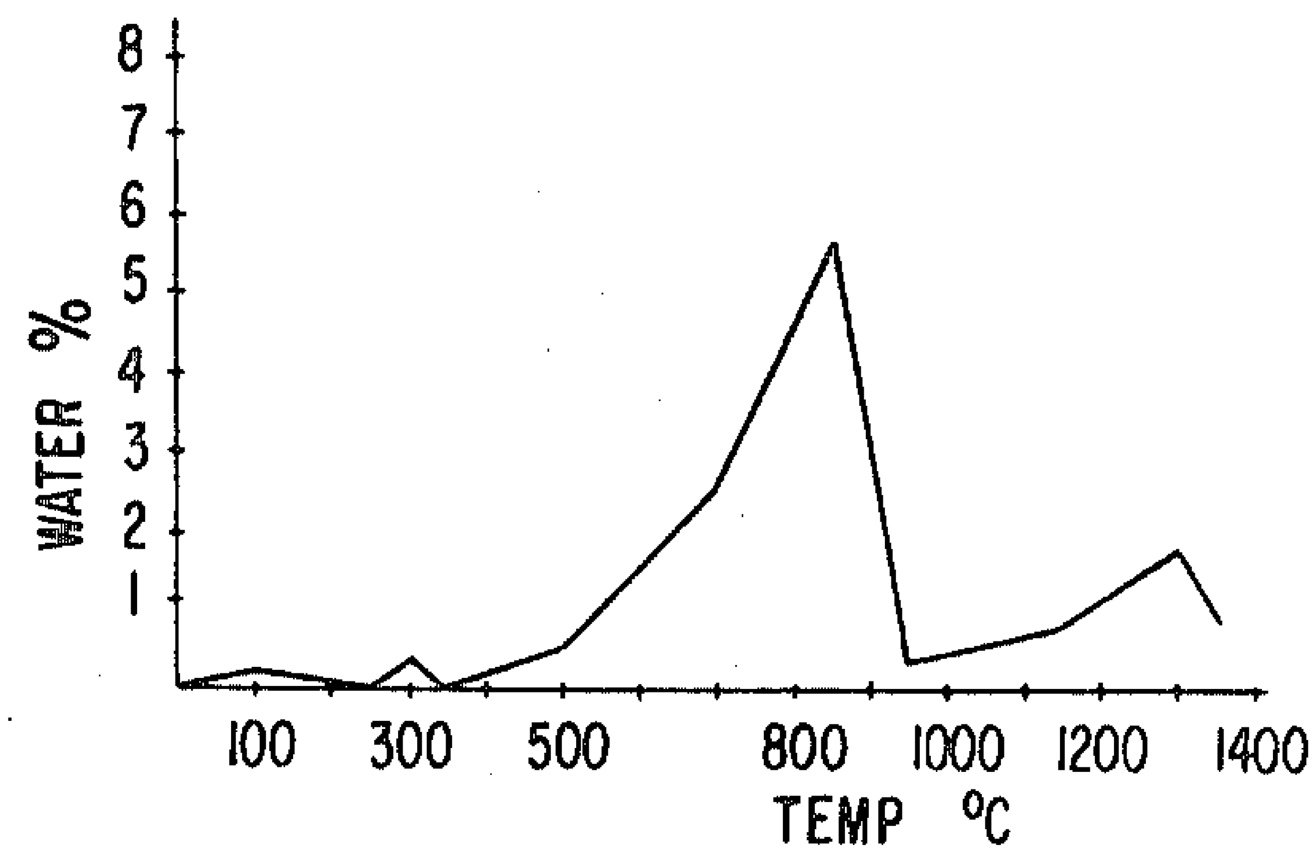


FIG.1

FIG.2



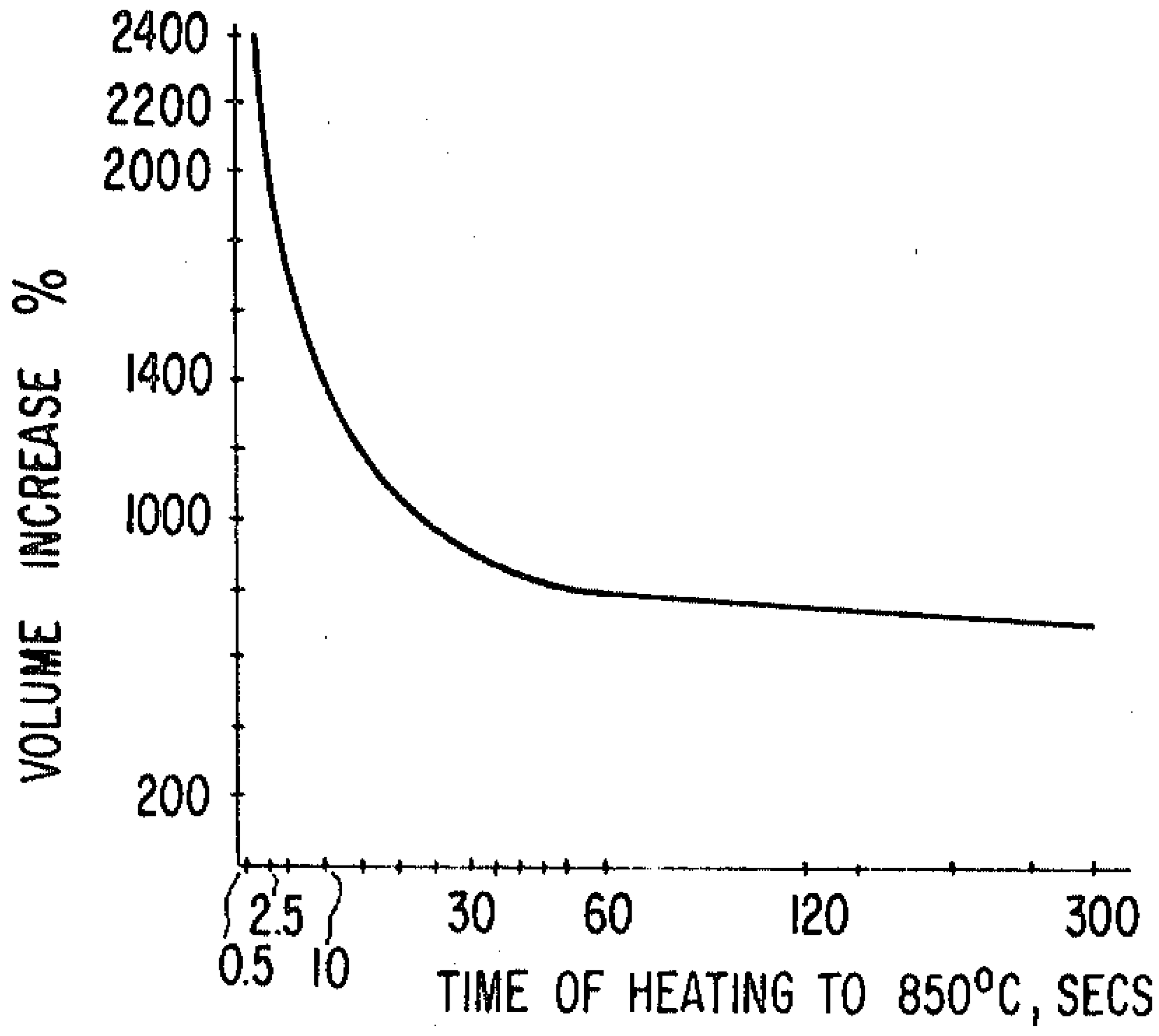


FIG.3

FIG.4

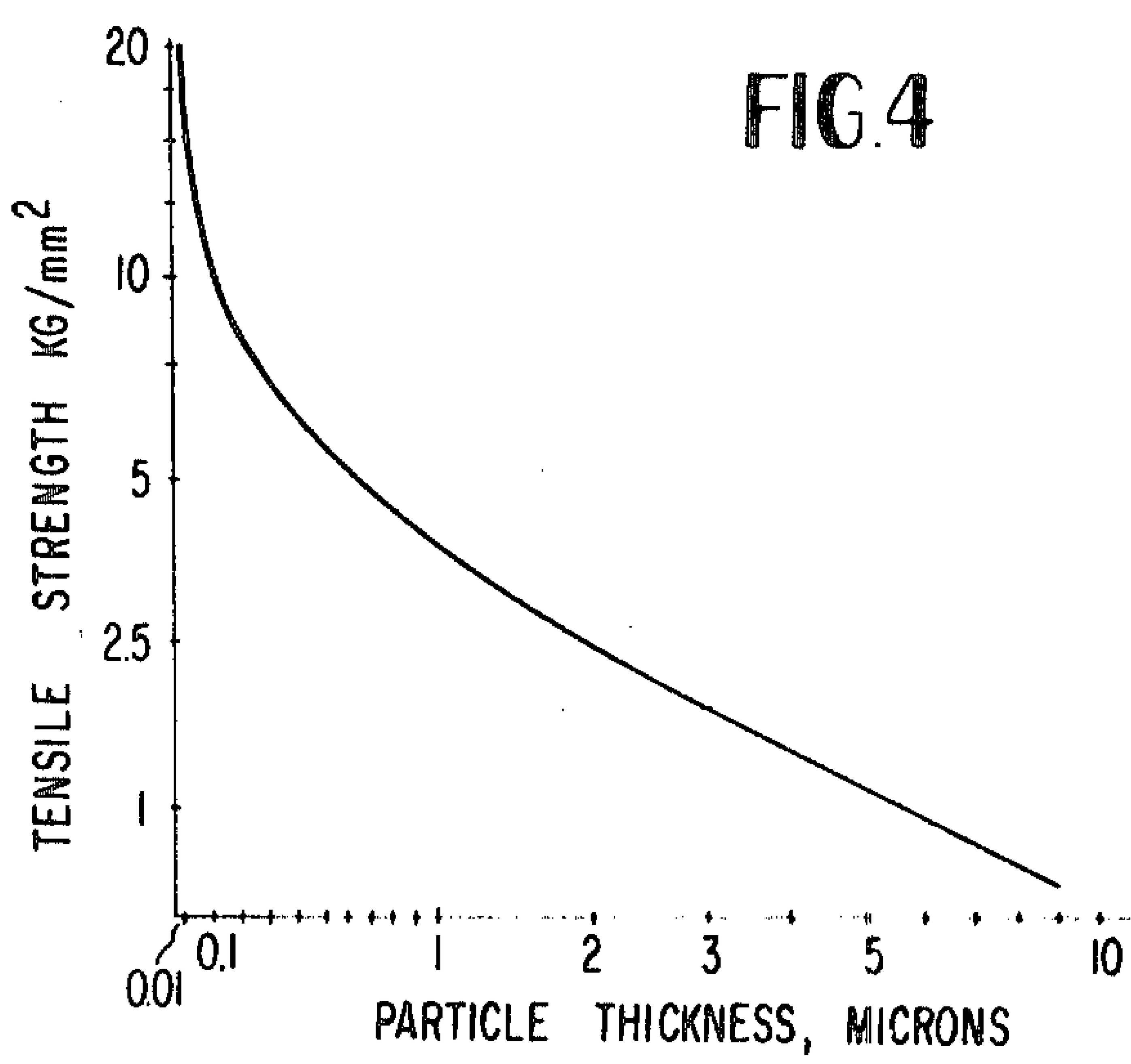
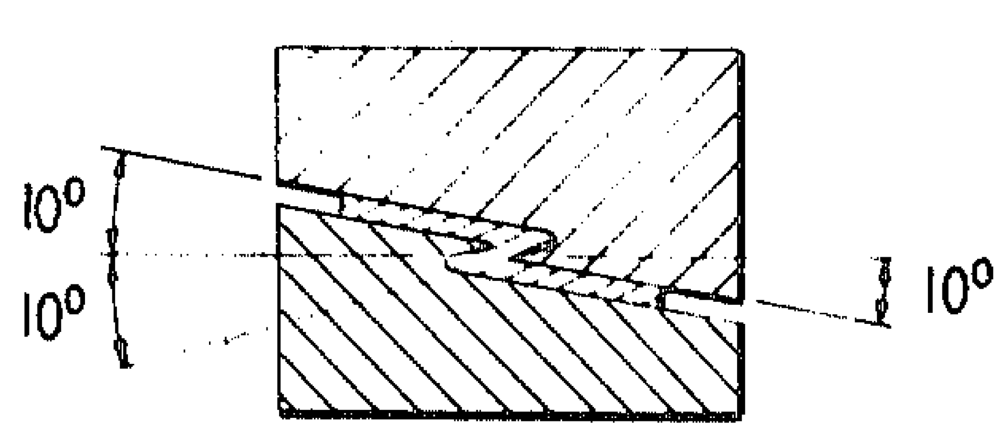


FIG.5



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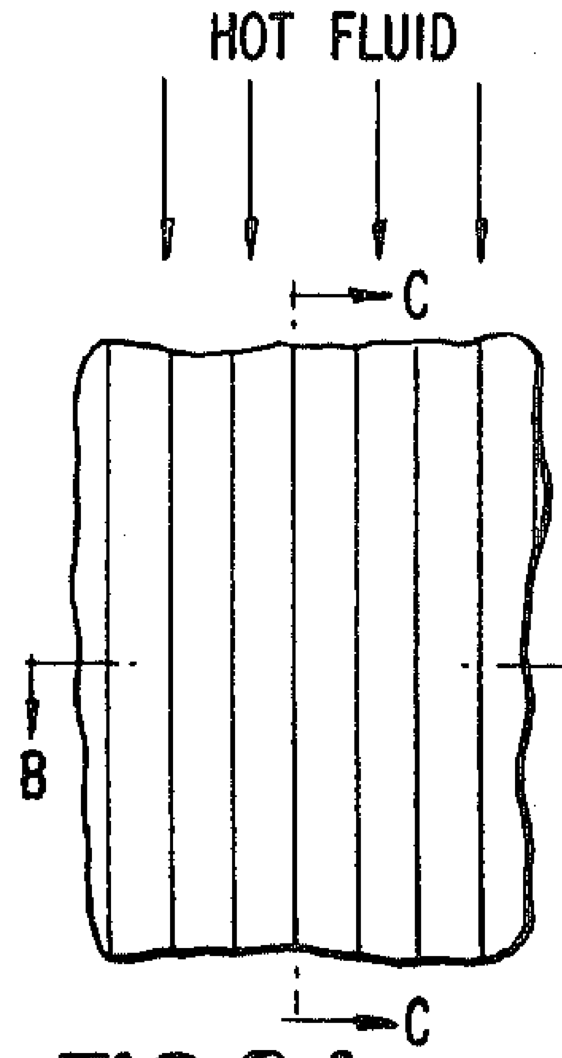


FIG.6A

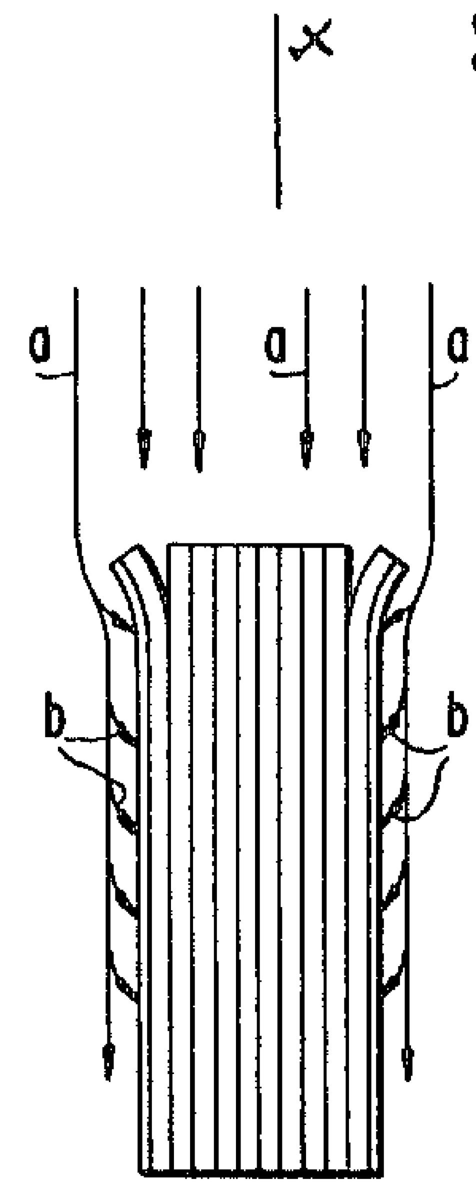


FIG.6C

TOO THICK

FIG.8

(PRIOR ART)

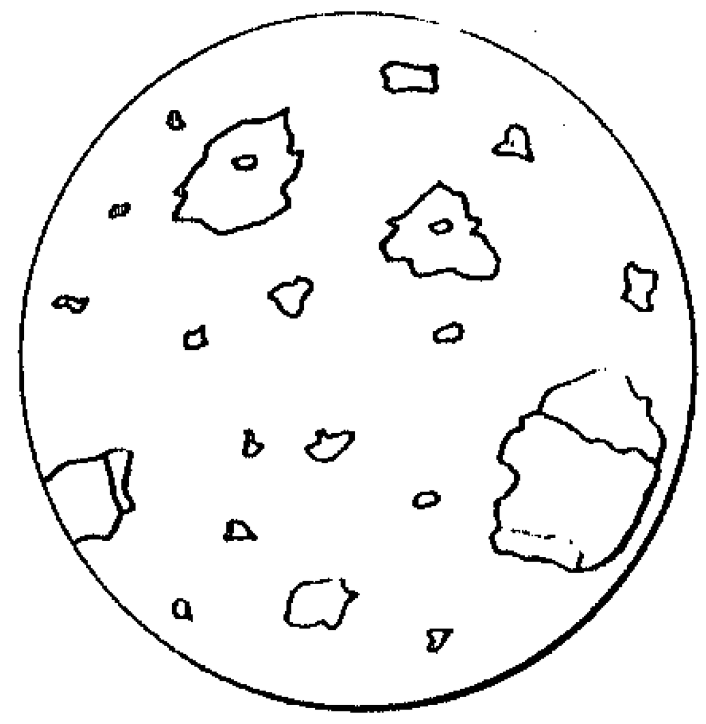


FIG.7

(PRIOR ART)

FIG.6B

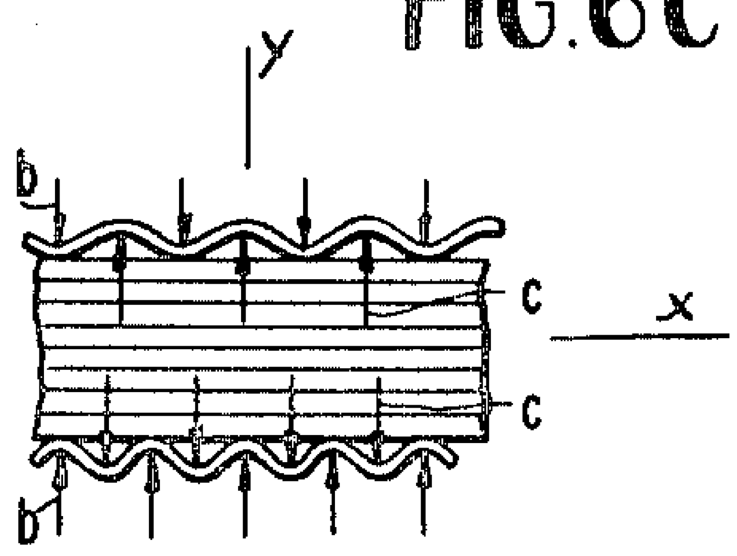
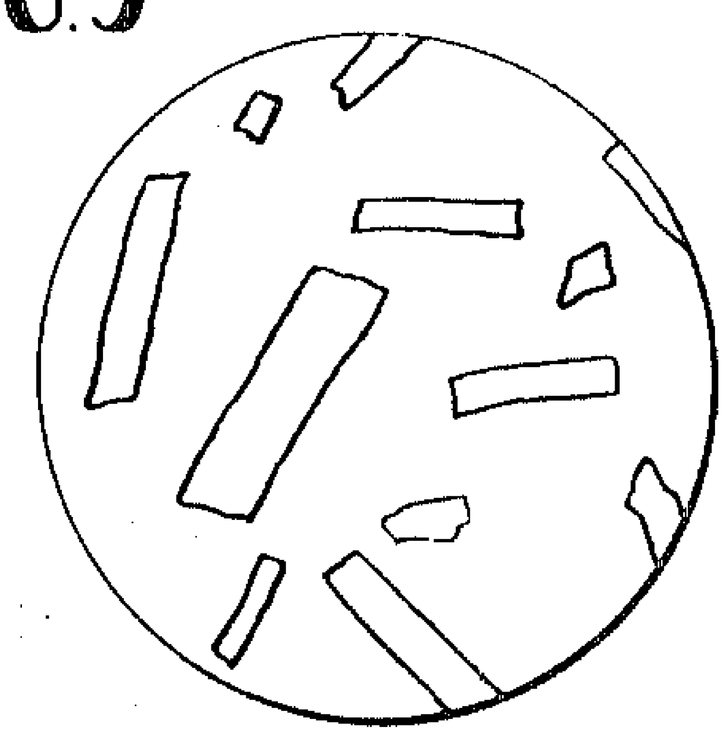


FIG.9



VERY THIN (INVENTION)

FIG.10

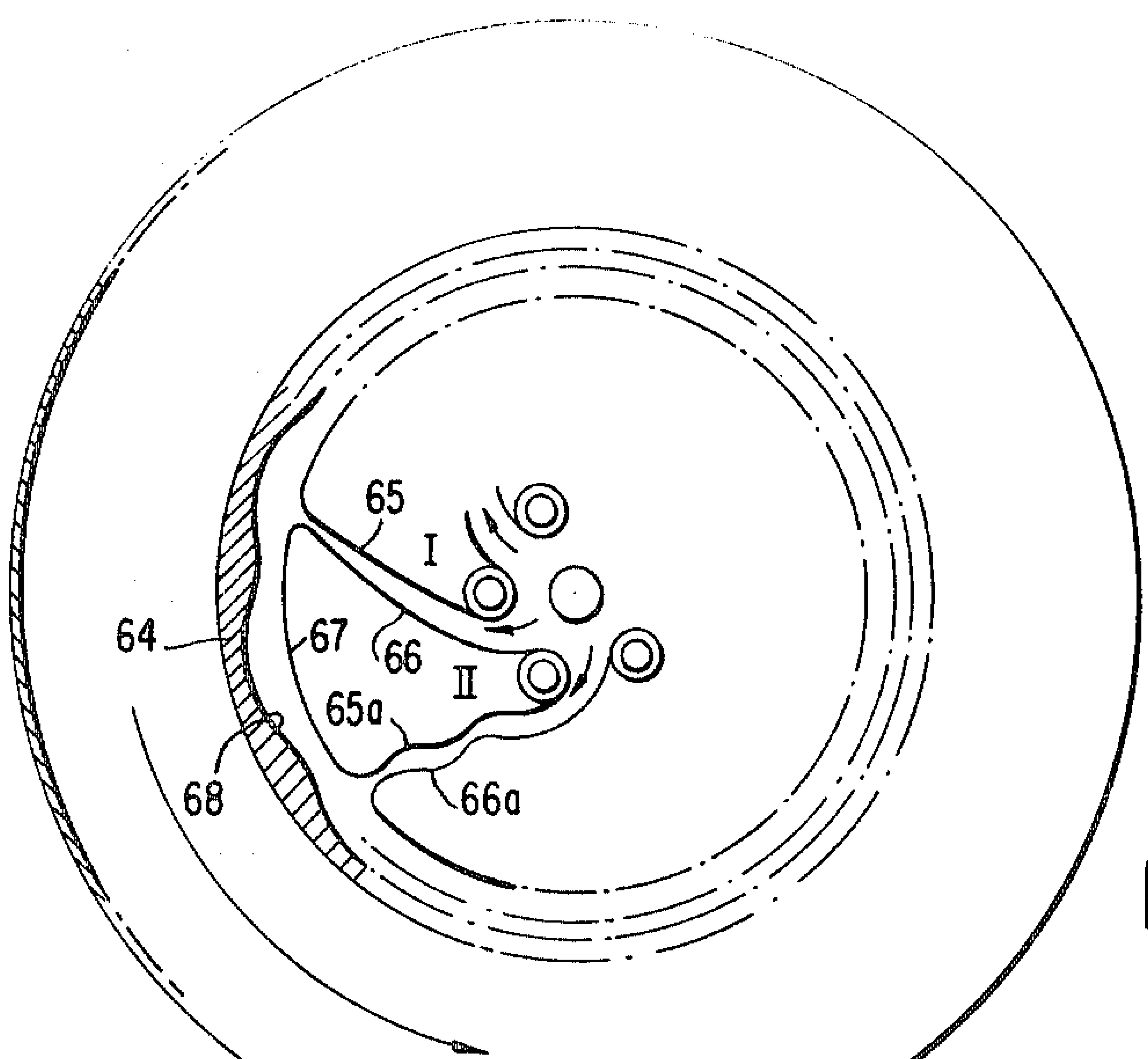


FIG.14

FIG.9A

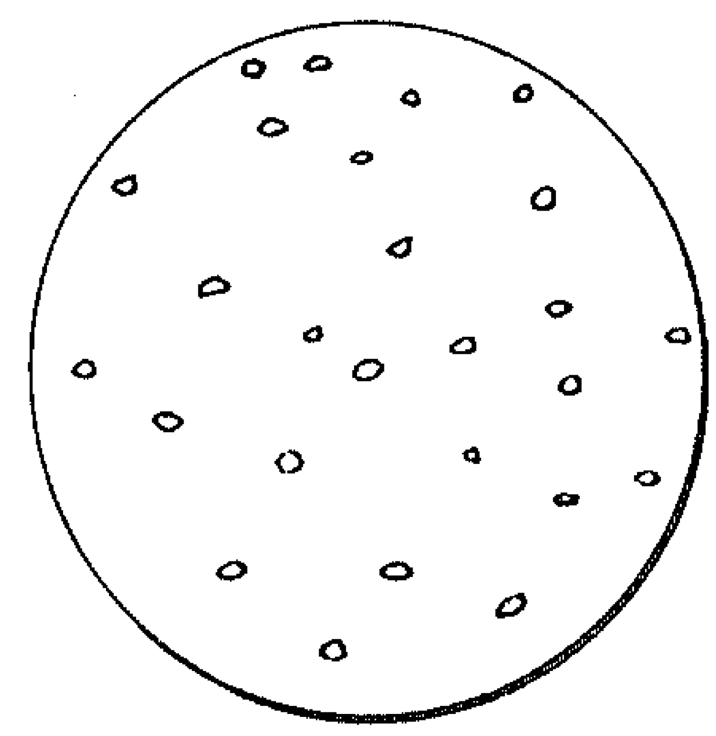
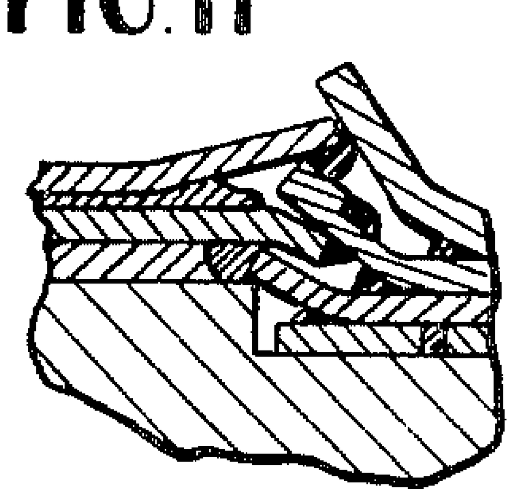
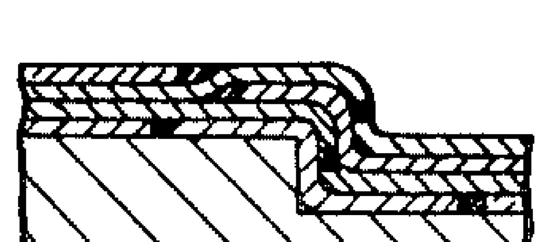


FIG.11



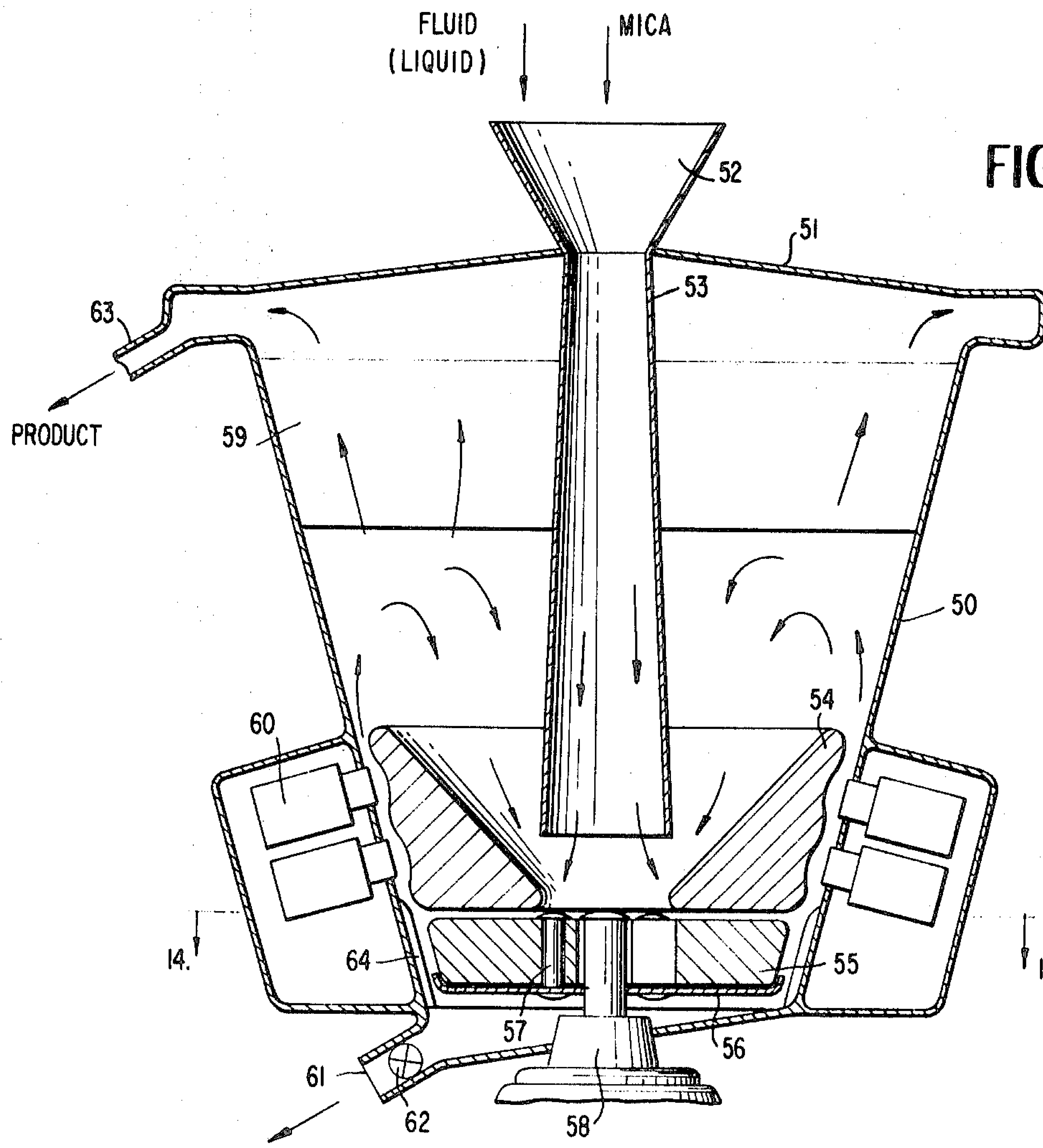
TOO THICK
(PRIOR ART)

FIG.12



THIN
(INVENTION)

FIG. 13



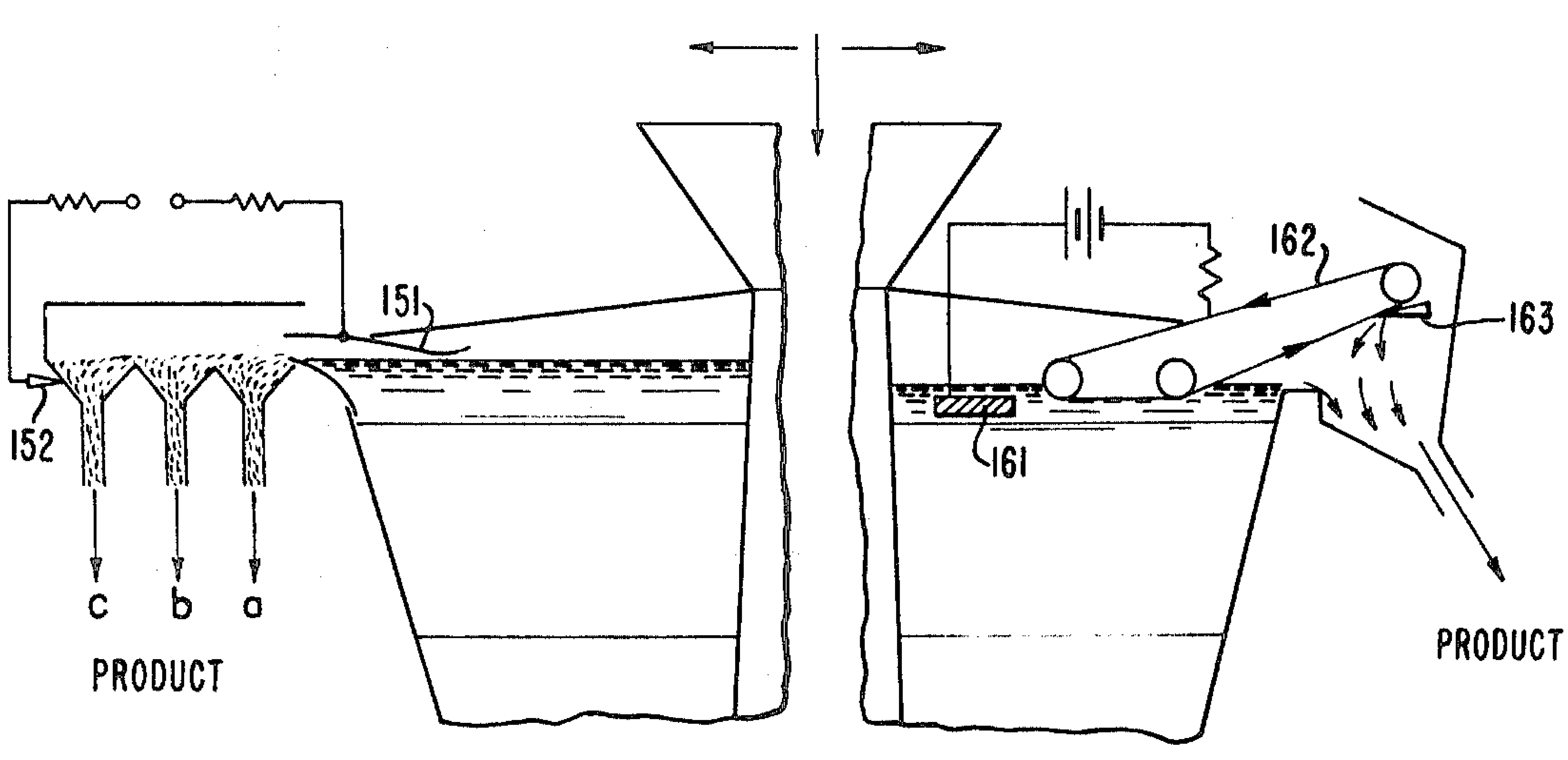


FIG. 15

FIG. 16

ULTRADISINTEGRATION AND AGGLOMERATION OF MINERALS SUCH AS MICA, PRODUCTS THEREFROM AND APPARATUS THEREFOR

SUMMARY OF INVENTION

This invention relates to the disintegration of frangible solids into ultrafine particles and the reagglomeration of such particles into desired products, to apparatus for carrying out such operations, and to various products obtained thereby. More particularly the invention relates to the oriented disintegration of a mineral such as mica into a multiplicity of fluidly suspended ultrathin particles or flakes and the production of new or improved products such as mica paper therefrom.

This invention generally is concerned with the splitting of easily splittable or cleavable materials to form fine particles, and especially the cleavage of minerals such as mica into small, thin flakes or scales which have active surfaces and which fall predominantly within a relatively narrow size range. The resulting products include mica particles characterized by an unusually high ratio of surface to thickness.

The disintegrating or splitting equipment is intended primarily for the splitting of mica but is also highly effective in splitting other materials, especially minerals, which liberate molecularly bound water or water of crystallization upon heating.

The aforementioned materials can be used in many different ways. For instance, the free particles or unoriented, easily redispersible agglomerates of such particles are useful as pigments and fillers for paints or other coating compositions, for resinous plastics, for elastomeric compositions; or as adsorbents or carriers for other materials, etc. In the form of oriented agglomerates they are useful as insulators or coatings in electrical equipment, as construction materials, etc.

BACKGROUND OF INVENTION

Mica forms a group of silicates, which are minerals characterized by their highly pronounced ability of being cleaved along their basic crystalline plane while being substantially less susceptible to cleavage along the crystalline plane which is substantially perpendicular to the first plane, and being still less susceptible to cleavage along any other plane. Consequently, this type of mineral has crystalographically a platelike structure which is highly flexible, resilient and strong and can be divided and subdivided into very thin flakes or scales.

Mica as a mineral is found in nature in various crystalline sizes, large sizes being quite rare, and in various chemical compositions such as muscovite, phlogopite, biotite, etc. Because of its excellent dielectric and mechanical properties, chemical stability and resistance to high temperature, mica is used for various industrial purposes, the highest grades of mica being used principally in the electrical industry as an insulating material. Its properties and usefulness, however, differ substantially not only depending on its basic type but even in a given type the properties depend on the exact chemical composition. The chemical composition of natural mica differs substantially, sometimes even within a single crystal. Yet the exact chemical composition determines the thermal resistance of individual mica crystals and when the critical dehydration temperature of a given piece of mica is exceeded, usually above 500° C., the mica becomes dehydrated and swells up and disintegrates depending on temperature and duration of heating. Synthetic mica has similar characteristics and properties.

PRIOR ART

Natural and synthetic mica crystals are relatively small while modern industrial requirements point increasingly toward large surfaces. For this reason the efforts in the art have increasingly been toward splitting mica into flakes of ever smaller thickness and reintegrating these thin flakes with or without the aid of binders into coherent sheets or leaves of large surface area. However, prior methods for making products of large surface area from mica particles having a thickness on the order of a few hundredths of a millimeter, e.g., 0.010 to 0.030 mm., have proved to be very laborious, the utilization of the mica is relatively low and the resulting products are quite nonuniform as well as expensive. Moreover, they lack adhesive surface forces.

Methods of making sheets of large surface area from mica particles having a thickness on the order of less than 0.01 mm. e.g., about 0.002 to 0.008 mm. have been known for more than 50 years. However, poor physical and especially mechanical properties of the resulting products have prevented them from becoming commercially important. More recent methods such as those described by Heyman in U.S. Pat. No. 2,405,576 or by Bardet in U.S. Pat. No. 2,549,880 have achieved a certain degree of commercial significance particularly because the mechanical properties of the resulting products are better than those of earlier products. However, though these processes are now more than 20 years old they have never achieved wide use. They have only partially succeeded in replacing the older methods which resulted in particles having a thickness greater than 0.01 mm., because their physical and especially their mechanical and dielectric properties still leave much to be desired, their processing is difficult, the utilization of the mica raw material is incomplete, and the operating costs are high.

Even when mica particles are to be used as pigments or fillers the trend in the art is to require particles of ever smaller thickness, that is, particles having the greatest possible surface area per unit weight. However, in requiring this there is also often the further requirement that the particles should not exceed a specified maximum dimension and should fall within a rather narrow particle size range. On the other hand, especially in the case of particles having a small diameter, such as 1 micron or less, the art has heretofore been unable to obtain high yields of particles falling within a predetermined narrow size range. The previously known mica particles at best had only very weak adhesive surface forces.

OBJECTS

It is accordingly an object of this invention to prepare fine solid particles such as mica flakes having a high specific surface area and other new or improved properties which make such particles particularly valuable as agglomerants or pigments and also in the production of aggregated products. Another object is to prepare improved products by agglomeration of fine particles.

A still further object is to provide new or improved methods and apparatus for oriented cleavage of mica principally along its main plane of crystallization and secondly along one further plane of crystallization while limiting the cleavage or splitting along any other planes, so as to facilitate the production of particles or flakes having a large specific surface area and a geometrically elongated configuration with predominantly submicron thickness, on the order of a few tenths or even thousandths of one micron or less, permitting the segregation of flakes having specified geometric dimensions, wherein the invention permits recycling of insufficiently disintegrated mica pieces to be split further until particles or flakes having the specified dimensions are produced.

A further object is to provide methods and apparatus for preparing and maintaining a fluid suspension of the fine mica flakes, to be subsequently converted either into an agglomerate or into free flowing particles to be used as a pigment or the like.

A still further and particular object is to provide apparatus and methods for producing improved mica paper or other structures either solely from the fine mica flakes or from a mixture of such flakes with other conventionally used auxiliary materials such as binders, fillers and so forth, particularly mica papers less than 20 microns thick.

THE DRAWINGS

In the drawings

FIG. 1 is a diagram of a process beginning with the preparation of raw material feed and leading through a splitting step and production of a fluid particle suspension to a final product molding step, with or without a separate intervening agglomeration step.

FIG. 2 illustrates the amount of water which is lost from a typical sample of mica by heating it to progressively higher temperatures until a constant anhydrous weight is reached.

FIG. 3 shows the volume increase of mica in relation to the time employed in heating it from 18° C. to 885° C., i.e., the effect of rate of heating on the degree of swelling achieved.

FIG. 4 is a diagram showing the relationship between the tensile strength (and also dielectric strength) and the thickness of mica after it has been bent between two complementary surfaces having a Z-shaped profile under a load of 10 kg./cm.^{2}.

FIG. 5 is a representation in vertical section of the Z-shaped device used for bending the mica as referred to in FIG. 4.

FIGS. 6A, 6B and 6C are three views showing a piece of mica being split along its "x" and "y" axes into thin flakes or plates by the simultaneous action of heat and a high velocity stream of a fluid medium.

FIG. 7 is a microscopic illustration of a group of typical mica particles obtained according to a process such as that of Bardet, showing both the irregular shape and wide range of prevailing particle sizes.

FIG. 8 is a greatly enlarged view showing one of the typical particles from FIG. 7 in vertical section.

FIG. 9 is a microscopic illustration of a group of typical mica particles made according to the present invention, showing their predominantly rectangular shape and relatively narrow size range.

FIG. 9A is a microscopic illustration of a group of mica particles of ultrafine size useful, for instance, in making pigments or fillers.

FIG. 10 is a greatly enlarged view in vertical section of a typical particle or flake of this invention, showing its essential flat surface free of irregularities.

FIG. 11 is a view in vertical section showing a deposit of relatively thick, inflexible mica particles of the prior art on a solid substrate, with cavities between some of the adjacent particles as well as between the particles and the substrate.

FIG. 12 is a view in vertical section showing a dense deposit of the thin, flat, flexible particles of this invention on and closely conforming to the surface of a solid substrate comparable to that shown in FIG. 11.

FIG. 13 is a view in vertical section of one embodiment of the apparatus for disintegrating materials such as mica in accordance with the present invention, preferred for use with a liquid suspension medium.

FIG. 14 is a plan view of the apparatus shown in FIG. 13, taken along line 14—14.

FIG. 15 is a partial view in vertical section of a variation of the apparatus shown in FIG. 13, wherein product particles are removed from the disintegration chamber via a wide spout by electrostatic means, liquid overflow is absent or very small, and the particles are classified into different fractions according to size.

FIG. 16 is a partial view in vertical section of still another variation of the apparatus shown in FIG. 13, wherein product particles are removed from the disintegration chamber by electrophoresis employing a moving belt which serves as an electrode to which the product particles adhere and from which they are removed by scraping.

PIGMENTS, FILLERS AND ACTIVE AGGLOMERANTS

The term "pigment" refers here to finely divided solids intended for addition to paints, other liquid coating compositions, glazes and the like while the term "filler" refers to finely divided solids intended for addition to molding resins, powders, pastes, elastomeric mixtures, graphite compositions, insulating compositions, papers as well as layers of free flowing solids such as layers intended for use as thermal or acoustic insulators. The term "agglomerant" refers here to fine mica particles with active surfaces or adsorptive capacities which make them suitable as carriers for active substances such as insecticides or herbicides, or as components of filtration media, or as carriers for pigments or other colorants or for materials such as silver or titanium dioxide powder or the like to make semiconductive products therefrom. From FIGS. 9 and 10 it is apparent that mica particles of this invention have the required geometric configuration, that is, small thickness and a relatively large and flat surface, narrow particle size range and large specific surface area. Depending on requirements, the new flakes have a very much higher specific surface area than similar products made previously, i.e., a surface area in excess of 7 m.^{2}/g., e.g., from above 7 to 700 or even 2,500 m.^{2}/g. with certain kinds of mica. The maximum dimension of the new thin mica flakes or particles can be predetermined in accordance with requirements and depending on the desired specific surface area may be of the order of 1 or more millimeters, tenths or hundreds of a millimeter and for special purposes may be of the order of 1 or more microns, tenths, hundreds or even thousands of microns, especially in preselected narrow size ranges falling within the overall range between 30 millimeters down to 2 millimicrons. For instance, the product illustrated in FIGS. 9 and 10 desirably will consist predominantly of particles having a high ratio of length to thickness, of the order of from 1,000/1 to as much as 5 million/1.

Pigments, fillers and agglomerants made in accordance with this invention make possible new applications and new methods of utilization which were not previously possible, because the characteristics of the new particles are of a fundamentally new kind in the physical sense such that, for instance, the finely divided particles when dispersed in an appropriate fluid behave like colloids, have a surprising ability to adsorb particles of other materials on their surfaces, conform tightly to substrates of various configurations without breaking, etc.

PROCESS OF INVENTION

Step A - Preparation of Raw Material

All available forms of mica, natural or synthetic, may be used in the present invention. The raw mica is cleaned in any conventional manner to remove organic matter, dirt and foreign mineral, preferably to obtain a feed of at least 90 percent purity. One of the important advantages of the present invention is that it permits simultaneous processing of mixtures of mica crystals differing from each other in chemical composition and having a wide particle size range, i.e., mixtures of large and small pieces.

Step B - Cleavage or Delamination

The method of effecting selectively oriented cleavage of mica in accordance with the present invention is illustrated in FIGS. 6A, 6B and 6C. Sudden local temperature effects are indicated by arrows *c* in FIG. 6B while the effects of the high velocity and high frequency fluid stream are indicated by arrows *a* and *b* in FIGS. 6B and 6C, and these bring about perfect cleavage of the mica predominantly in two directions, i.e., primarily along the plane of lowest cohesion (the basic plane) and further along the plane having the next lowest cohesion which substantially is perpendicular to the first plane. The effects in other directions are not greatly developed and are suppressed by the elasticity of the mica and are therefore so weak that predominantly they do not reach values necessary for disrupting the mechanical cohesion of the mica in any further, less easily splittable directions.

According to this method the continually fed pieces of mica (FIG. 6A) are exposed to the necessary mechanical, delamination forces, or combination of mechanical and thermal forces, in one or more splitting chambers which are arranged in series or in parallel. The forces, at temperatures between as low as about 100° C. and up to about 1,350° C., act on the large pieces of feed material for periods which depending on individual particle size may range from a fraction of a second to a few minutes within a fluid, and preferably inert, medium. The forces cause splitting of the mica predominantly in the direction of two planes, by the pulsating, vibrating and accelerating or decelerating streams of the medium which whirl in a distinctly oriented manner and which cause delamination predominantly progressively from the surface of the mica inward as indicated in FIGS. 6B and 6C until the original pieces are delaminated to the desired extent. For some kinds of mica and some kinds of end use the method may be performed in a single chamber whereas in other cases the splitting may be effected in a plurality of like or different splitters, e.g., first at ambient temperature in a liquid medium and then at elevated temperature in a gaseous medium. This method may of course be modified in that, for instance, the pieces of mica being fed to the splitter may be preheated or thermally pretreated prior to introduction into the splitter chamber, preferably in an inert or protective fluid such as argon or hydrogen.

The resulting flaked or disintegrated products having active surfaces (i.e., an adsorptive surface), which they obtain by virtue of their predetermined geometric dimensions, are immediately and continuously separated and transferred to the next step. In some cases one may add binders or other additives such as organic or inorganic fibers, platelets and the like in order to distribute them uniformly in the eventual product.

In making pigments, fillers and agglomerants of the kind illustrated in FIG. 9A, rather than the predominantly two-dimensional flakes illustrated in FIGS. 9 and 10, it is necessary to split the mica as much as possible not only along the first and the second splitting or fissioning planes in order to obtain the greatest possible specific surface area, but also to further split the mica to form an ultrafine particle size.

The ultimate size may be specified in terms of the maximum permissible dimension or diameter or better in terms of the permissible particle size range, e.g., 10 to 30 microns, or 0.1 to 1 micron, etc. Consequently, the splitting method is oriented for splitting according to all planes of fission and for producing the smallest particle size possible it can utilize further effects of the high velocity of the splitting medium, 100 meters per second or more, and high frequency waves (20 kilocycles per second or more) and the acceleration and deceleration of the particles and the consequent cavitations. The method can be still more effective when the splitting medium enters into the reaction chamber intermittently and thus produces pulsations. The apparatus illustrated in FIGS. 13 to 19 are equipped with devices for the production of the aforementioned effects, such that pigments, fillers or agglomerants of various sizes and ratios of length or particle size to thickness may be produced by adjustment of the appropriate variable or variables, e.g., by increasing the velocity of the fluid medium, by increasing the number of operating jets, etc.

Step C - Preparation of Fluid Suspension

The mica particles having active surfaces are kept in or conducted to and maintained in a fluid suspension in the previously present or in a different protective medium. Various combinations of gaseous or fluid media are possible depending principally on the requirements of subsequent utilization. It is possible to make intermediate products in a continuous manner and to concentrate the suspension and only adjust the consistency or concentration of the suspension prior to the next processing step and depending on the requirements of the latter. The maintenance of these particles as a suspension is advantageously effected with the stream of the aforesaid medium, and only by mechanical means, but in some cases it may be useful to employ additionally the effect of an electrical field.

The suspension of particles of the proper concentration can then be continuously or intermittently added to an appropriate agglomerating step or it can be added directly to some other finishing step.

Product I - Fillers, Pigments and agglomerants,

Fillers, pigments and agglomerants, i.e., mica particles which can be added to coating compositions, electrical putty, synthetic resins, rubber compositions, etc. can have two different forms, that is, either as a loose conglomerate of various shapes such as a block from which a desired amount of pigment can be easily broken off for use, or as a free flowing pulverulent mass. An agglomerant made as outlined above in Step C can be a final product as such. If it was made from a gaseous suspension it does not require any finishing operation or it may be classified into fractions of different size in the dry state in any otherwise known manner. If it was made from a liquid suspension, finishing operations can comprise concentration or compacting and drying, or if necessary, the particles can be classified while wet prior to compacting in any otherwise known manner. The production of particles in a free flowing state from a gaseous suspension requires essentially only contacting them with the ordinary atmosphere while being circulated, whereby the adhesive surface properties are destroyed, whereas in the case of a liquid suspension a drying step will usually be required.

APPARATUS

Splitter

Referring to FIGS. 13 and 14, the apparatus is an axially symmetrical splitting chamber in the shape of an inverted truncated cone 50 of circular cross section. However, chambers of other axially symmetrical configurations, e.g., chambers having a horizontal cross section which is quadrangular, octagonal, etc. or which has parallel rather than diverging sidewalls are also usable. Incompletely split pieces of mica are recirculated back into the splitting zone in the direction of the main vertical axis. This device is particularly intended for splitting mica in a liquid medium such as water or ethyl alcohol though it is possible to operate it with a gaseous medium. It can be operated either at ambient temperature, or the operating temperature may be lowered below the freezing point of water if a suitable nonaqueous fluid medium is used or it can be increased, e.g., above the temperature at which mica splits out bound water.

Vessel 50 is provided with a cover 51 which in its central portion contains a funnel 52 for feeding the pieces of mica which are to be split. Tubular element 53, which preferably has the shape of a flared cylinder or cone, is spaced from orienting element 54 which is attached to and spaced from the sidewall of vessel 50. A rotating assembly of hinged splitting elements or paddles 55 is spaced above a rotatable supporting plate 56. The paddles 55 are hingedly and movably supported on pivots 57 which are attached to plate 56. The entire splitting assembly is rotated by motor 58. Vertically arranged baffles or ribs 59 are arranged in the upper part of chamber 50 and are attached both to the outer wall of chamber 50 and to tubular element 53. Sonic or ultrasonic vibrators 60 are attached in the outer wall of chamber 50 facing the orienting element 54. At the bottom of vessel 50 is a drain 61 with valve 62 which may be used for periodic cleaning of the vessel or otherwise as needed.

In the upper part of chamber 50 there is a collector and overflow spout for the finished product. In the bottom circumference of vessel 50, facing the exterior faces of revolving elements 55, there is arranged a directional element or ring 64. The pivoted members 55 are easily removable such that they may be replaced with members of different configurations, and the total number of the members can also be varied. The shape and quality of the working vertical surfaces 65 and 66 are such that they form a resilient system of channels of outwardly narrowing horizontal cross section. When rotating the members form a system of essentially vertically oriented planar streams of the splitting medium flowing radially outward between adjacent members. It is possible to regulate the mutual relationship of the lower portion of the walls of filler tube 53, the directional element 54 and the working surfaces of members 55 by displacing these elements relative to each other and thereby vary the flow pattern in the unit. Individual parts of the equipment are made from appropriate structural materials such as stainless steel or other metal, synthetic resins, etc. The unit may be virtually of any size. For instance, it is possible to build small laboratory type units with splitter chambers having a bottom diameter of about 35 cm. or less and a height of, for instance, 50 cm.; or large commercial units with splitter chambers having a bottom diameter as large as 1 or 2 meters or more and a height of 3 to 10 meters or more.

In operation, cleaned chips or pieces of mica of whatever kind and thickness and size are continuously fed into funnel **52**. The required amount of liquid, for instance 500 parts by weight per part of mica feed, is also preferably added through funnel **52**. The mica feed and cleavage medium are then aspirated by the rotating effect of elements **55**, partially from supply tube **53** and partially from the space between the inner wall of directional element **54** and the lower portion of supply tube **53** where insufficiently disintegrated particles are recirculated into the splitting zone until they are comminuted to the desired size.

The intensity of the cleavage action can be controlled by the speed of rotation of the rotor assembly **56**, the width and configuration of the channels formed between adjacent elements **55** and between the outer faces of these elements and the outer ring **64**, by the viscosity of the splitting medium, etc. For instance, the assembly carried on plate **56** may rotate at about 20 to 500 r.p.m. or more and the contracting width of the channels between individual elements **55** may range from as much as 50 mm. near the center of the vessel to 1 mm. or less near the periphery. It will be understood of course that the width of individual channels varies in operation and that the narrower the exit of the channel the more effective will be its splitting action as the vertically oriented mica particles are carried therethrough in the circulating fluid.

This apparatus has the important advantage that as the mica raw material is sucked in between the splitting elements **55** it becomes oriented in accordance with the principal surface of each piece of feed, essentially parallel to the direction of the fluid flow through the aforementioned channels and the laminar surfaces of the resulting fluid streams. Essentially the pieces retain this orientation throughout the entire splitting process both between the elements **55** and after tangential discharge from the channels and passage along directional ring **64**, and up into the upper part of the vessel, and even when vibrators **60** are operating. This orientation prevails both in the case of freshly added pieces of mica and in the case of pieces which are recirculated.

The splitting elements **55** form in the course of a process a resilient assembly with highly effective working surfaces which are self-cleaning and therefore cannot become obstructed and which can be easily regulated and simply increased by increasing the diameter and/or height of the elements, by increasing their surface (as shown at **65*a*** and **66*a***) and by increasing their total number.

The total effective working surface can also be increased by increasing the surface of the directional elements **54** and **64**. The effectiveness of the cleavage operation may be further increased by placing vibrators **60** in operation.

The cleavage effects are based principally on the effect of the high velocity laminar plane streams of the fluid medium acting essentially parallel to the major surfaces of the oriented mica particles. The velocity of the stream in this process increased in the centrifugal direction while the thickness of the channels and hence the laminar fluid streams flowing therethrough decrease in the radially outward direction. Being resilient, individual channels become narrower or wider during operation depending on the thickness of the pieces of mica passing through them.

A shape of the splitting elements such as that illustrated at **65*a*** and **66*a*** additionally provides a sort of delaying chambers and increases the number of the narrowest channels or passages through which the mica is dragged in the process. This produces a pulsating flow and further enhances the cleavage action.

The velocity of the fluid stream changes according to channel cross section and is therefore usually lowest in the central part of the vessel and conversely highest in the peripheral portions. Mica is thus split by the effects of a fast laminar stream of fluid and the changes in its velocity, i.e., its acceleration and decelerating, by the rapid increase in velocity of the fluid stream in the radially outward direction, the resulting cavitation and vibration or pulsation along sinusoidal faces **65*a*** and **66*a***, the slowing down along the effective surface, etc. When these effects are insufficient to achieve the desired degree of comminution in any particular case the cleavage action may be further intensified by the use of devices **60** which may produce sonic or even ultrasonic vibrations, etc., vibrations in the range from about 10 kc./sec. up to about 1 mc./sec.

The split particles having the desired dimensions are continuously sorted out from the process as soon as they reach the desired size, being floated up and removed in the overflow of the liquid through spout **63** and then transferred to whatever further operation may be desired. The principal variable by which circulation and recirculation of the particles within the splitting chamber is controlled, is the speed of rotation of the revolving assembly in the bottom, but rate of circulation of liquid through the unit can also be adjusted to cause the desired range of particle sizes to be floated out of the unit.

Instead of removing the comminuted particles in an overflowing stream of the liquid medium as shown in FIG. **13**, it is possible to remove them without virtually any liquid by using an arrangement as shown in FIGS. **15** or **16**. As shown in FIG. **15**, the liquid medium is maintained in the splitting chamber **50** with no or virtually no liquid addition or overflow while two-spaced electrodes are arranged at the overflow spout **63** such that the particles of mica product become charged near the liquid surface in the splitting chamber and then jump out from the liquid by being attracted to the other electrode whence they are finally removed. An inert gas such as argon or neon may be maintained in such a system to avoid any degradation of the product particles by contact with air or oxygen. By arranging a series of collecting bins beneath the two-spaced electrodes, the mica particles being ejected from the splitter can simultaneously be classified into several fractions according to their weight, the heaviest particles dropping out first and the progressively finer ones dropping down at more distant points from the spout.

In an alternative arrangement, illustrated in FIG. **16**, a moving belt may be arranged in the upper portion of the splitter and an electric field set up such that the product particles become charged near the top of the liquid medium in chamber **50** and then are carried by electrophoresis to the oppositely charged moving belt to which they remain attached until they are scraped off and recovered. Example 1

A mixture containing about equal proportions of clean pieces of muscovite and phlogopite about 3 to 100 mm. in diameter is introduced into the funnel **52** of the apparatus in FIG. **13**. 800 parts of clean water per part of mica is simultaneously introduced and the mixture of mica particles and water is aspired between the jaws of the rotary elements at the bottom of the splitter and the mica is thus split into small particles in accordance with its laminar structure by the action of the water stream and the auxiliary high frequency means **60** vibrating at 100 kc./sec. The rotor revolves at 100 r.p.m. When a particle reaches a specific surface area of 5 $m.^2/g.$ it is immediately sorted out from the process by floating up in the water whereas coarser particles settle out from the water stream and return downwardly toward the rotating elements for further disintegration.

The resulting suspension of mica flakes of proper size overflows into a storage vessel (not shown in FIG. **13**) and then, after adjustment of proper solids concentration, into the chamber of a paper making machine.

The new mica products described herein are substantially better in terms of their mechanical, electrical and other physical properties, than similar mica products heretofore available, generally several times better, such that in effect new classes of mica products having new types of utility are now made available. The advantages are particularly apparent in fabricated products made from the new basic material, i.e., the ultrafine mica flakes. For instance, because of the extremely small thickness of the new mica flakes, it now becomes possible to make self-supporting coherent mica webs, coatings and laminates only a few microns thick.

The key pieces of equipment designed in accordance with this invention have large capacity and relatively small dimensions, such that as much as ten times more production can be obtained from a given plant area than heretofore. Moreover, the disintegration or cleavage technique of this invention is unusually advantageous in that it permits the simultaneous utilization of different kinds of mica such as muscovite and phlogopite, it also permits the use of a mica feed containing a wide range of particle sizes, and in all of this it makes possible essentially 100 percent conversion of the feed material into desired products.

The methods and apparatus of the invention further make it possible to make combination products from mica and various other materials such as glass fibers or platelets, fibers of asbestos, silica, cellulose or synthetic fiber forming resins, glass cloth, binders, foils of synthetic resins or metals, etc. Because of their high surface area, the novel mica particles themselves offer unusual advantages as pigments, fillers and also as carriers for other pigments and for physiologically active substances and catalytic substances. Because of the extremely small thickness, coatings or compositions made from these new mica particles have far superior barrier effects, novel decorative effects, etc.

As compared with similar products known previously, the ultradelaminated flakes made in accordance with this invention are such that they fall into a quite different and new physical field and are therefore governed by different physical laws than the earlier products. One of the predominant characteristics of the new particles is that they behave like colloids in a suitable liquid medium. Their sedimentation times are extremely long, such that they can be used in processes where the coarser, previously available particles were useless or gave poor results.

In addition to the aforementioned product properties, important advantages are obtained in that certain features of the present invention permit a very high degree of flexibility of the process, permitting the economical use of different types of splitters in parallel or in series depending on types of products required, and high production capacity per unit area.

It should be understood that the foregoing general description and specific examples have been given primarily for purposes of illustration and that numerous variations and modifications thereof are possible without departing from the scope or spirit of the disclosed invention. It should also be understood that, in the absence of indications to the contrary, all percentages and proportions of materials are expressed in the disclosure on a weight basis.

The scope of the invention is particularly pointed out in the appended claims.

1. A process for ultradelaminating a crystalline laminar solid into thin flakes which comprises:

a. forcing a fluid medium through apertures having narrow elongated cross section in a bottom portion of a conversion zone, said apertures being oriented substantially parallel to the height of said zone, and thereby forming in said bottom portion a system of whirling fluid streams having an orientation approximately parallel to the apertures,

b. introducing relatively coarse pieces of said solid into said conversion zone whereby said pieces become suspended in said whirling streams and disintegrated principally in planes corresponding to the principal two axes of the solid crystal lattice,

c. circulating the resulting fluid suspension comprising comminuted solid from said bottom portion upwardly in the peripheral portion of said zone into an upper portion thereof,

d. settling insufficiently comminuted solid particles from said upwardly circulating suspension back into said bottom portion for further disintegration in said whirling streams,

e. removing solid particles of the required fineness from the upper portion of said conversion zone.

2. Process for disintegrating mica into thin flat flakes which comprises suspending relatively coarse pieces of mica in an essentially inert liquid, in a proportion of 1 part of mica per 5 to 5,000 parts of liquid, in a bottom portion of an upwardly expanding conversion zone having substantially the shape of a truncated cone, propelling the mixture near the bottom of said conversion zone in a centrifugal direction through constricted substantially vertical channels having a varying width such that the mixture becomes more constricted as it passes therethrough, circulating the mixture upwardly along the periphery of the conversion chamber such that mica particles having the desired fineness rise to the top and overflow from said conversion zone to be recovered while insufficiently fine particles settle downward toward the central portion of the conversion zone for further passage through said narrow channels for further disintegration.

3. A process according to claim 2 wherein said channels have alternating areas of larger and narrower width.

4. A process for ultradelaminating a crystalline laminar solid into thin flakes which comprises:

a. forcing a liquid medium through apertures in a bottom portion of a generally circular conversion zone, said apertures being normal to the height of said zone whereby said liquid forced through said apertures in said bottom portion forms a system of whirling streams having an orientation approximating the circular path of said conversion zone,

b. introducing relatively coarse pieces of said solid into said system of whirling liquid streams and thereby disintegrating said pieces principally in planes corresponding to the principal two axes of the solid crystal lattice,

c. circulating the resulting liquid suspension comprising comminuted solid from said bottom portion upwardly in the peripheral portion of said zone into an upper portion thereof,

d. recirculating insufficiently comminuted solid particles from said upwardly circulating suspension back into said bottom portion for further disintegration in said whirling streams,

e. removing solid particles of the required fineness from said conversion zone.

5. A process for disintegrating mica into thin flat flakes which comprises feeding a suspension of insufficiently fine pieces of mica in an essentially inert liquid to a bottom portion of an upwardly extending conversion zone having a substantially circular cross section, propelling the mixture near the bottom of said conversion zone in a centrifugal direction through constricted channels having substantially vertical walls and having a resiliently varying width such that the mixture becomes more constricted as it passes therethrough, circulating the mixture upwardly in the conversion zone such that mica particles having the desired fineness rise to the top and are recovered from said conversion zone while insufficiently fine particles settle downward toward the central portion of the conversion zone for further passage through said narrow channels for further disintegration.

6. A process according to claim 5 wherein said channels have alternating areas of larger and narrower width.

7. A process according to claim 5 wherein said mica particles are recovered from said conversion zone by electrostatic means.

8. A process according to claim 5 wherein said mica particles are recovered by flotation.

9. An apparatus for disintegrating solids suspended in a liquid medium comprising a vessel having a sidewall of generally circular cross section and providing a disintegration chamber in the lower portion thereof and an elutriation zone

in the upper portion thereof, disintegration means in said chamber comprising a plurality of channel-forming members having substantially vertical walls extending generally radially outward from the central area of said chamber, said channel-forming members forming channels of resiliently varying width generally narrowing from said central area toward the periphery of said chamber and terminating short of said periphery, means mounting said channel-forming members for rotation in said chamber and means operatively connected therewith for rotating said members, said vessel including means for introducing solids in liquid medium into the central area of said chamber and means of withdrawing product from said elutriating zone.

10. An apparatus according to claim **9** wherein said vessel includes baffle means above said channel-forming means and separating said disintegration chamber and elutriation zone from one another.

11. An apparatus according to claim **10** wherein said baffle means includes a wall surface adjacent and parallel to the sidewall of said vessel for guiding disintegrated particles upwardly along the periphery of said sidewall as the particles are circulated from said chamber to said elutriating zone.