

- [54] **PROCESS AND APPARATUS FOR THE FINE COMMUNUTION OF SOLIDS**

- [76] Inventor: **Hans Beike, 15 Danzigen Weg,  
Kronberg/Taunus, Germany**

- [22] Filed: **Oct. 1, 1970**

- [21] Appl. No.: 77,074

- [63]
- Related U.S. Application Data**

Continuation-in-part of Ser. No. 786,179, Dec. 23,  
1968, Pat. No. 3,614,001.

- [52] U.S. Cl.....241/17, 241/65  
[51] Int. Cl.....B02c 23/00  
[58] Field of Search.....241/17, 18, 38, 42, 65

- [56]
- References Cited**

## UNITED STATES PATENTS

- |           |        |                  |          |
|-----------|--------|------------------|----------|
| 2,919,862 | 1/1960 | Beike et al..... | 241/65 X |
| 2,609,150 | 9/1952 | Bludeau.....     | 241/65 X |

- |           |        |             |          |
|-----------|--------|-------------|----------|
| 2,836,368 | 5/1958 | McCoy.....  | 241/17   |
| 2,879,005 | 3/1959 | Jarvis..... | 241/65 X |
| 2,974,883 | 3/1961 | Engel ..... | 241/17   |

*Primary Examiner*—Granville Y. Custer, Jr.

*Attorney—Connolly and Hutz*

- [57]
- ABSTRACT**

A process for the fine comminution of solids made brittle by their immersion into a cold liquefied gas is characterized in that all of the cold material comminuted after its cooling down to the temperature of the liquefied cooling gas is subjected to at least one further pass of comminution relaying mainly on the pressure effects for causing the average particle size of the materials to be considerably reduced.

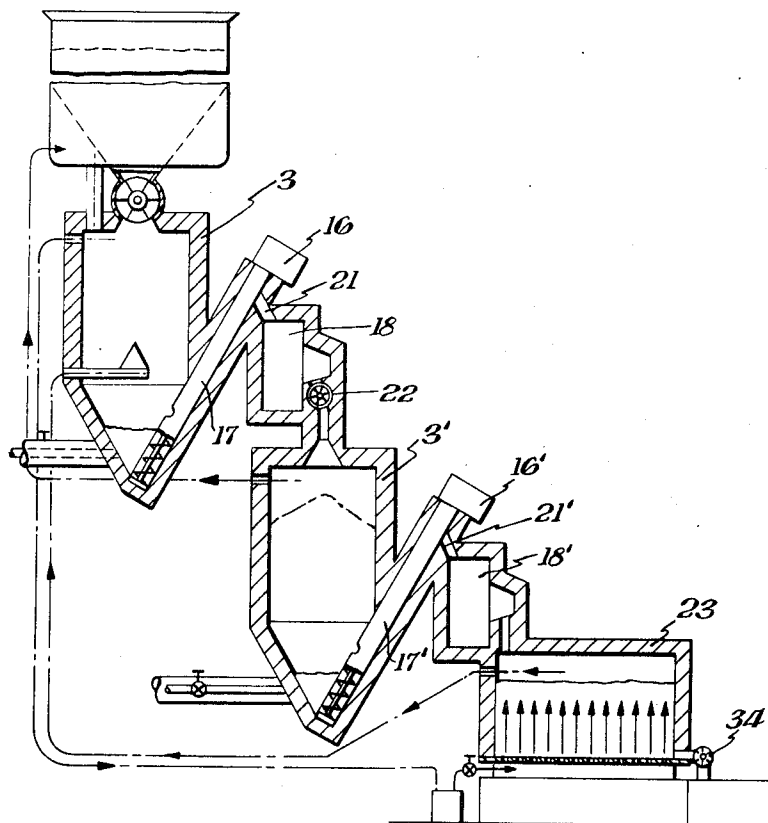


*Fig. 1.*

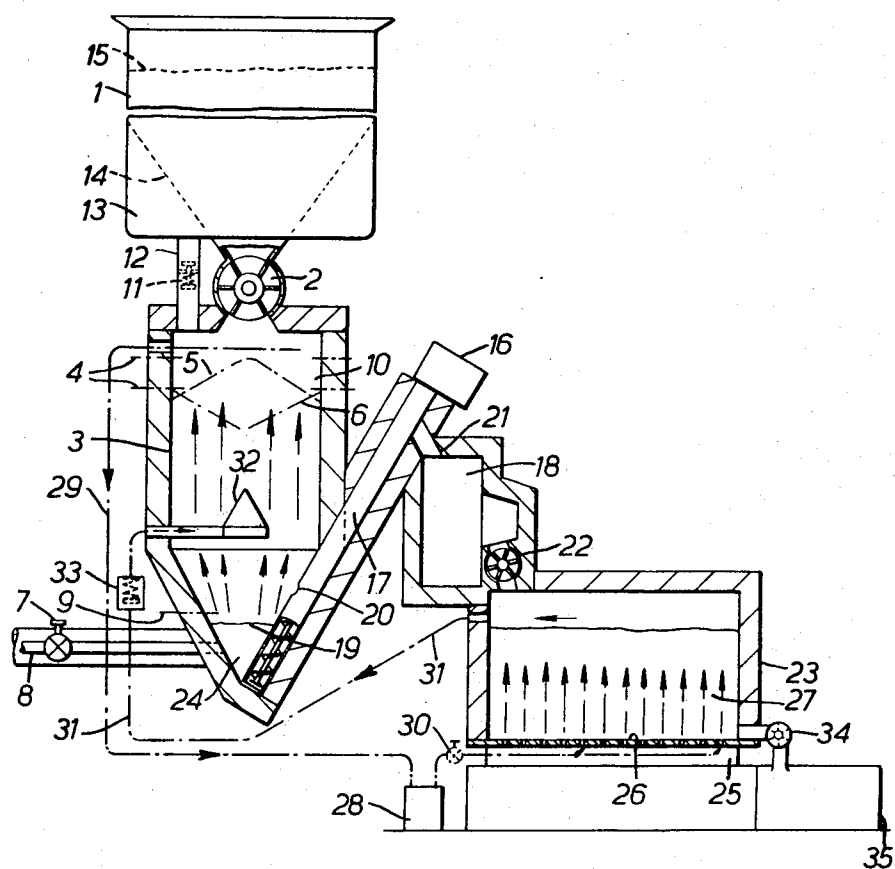
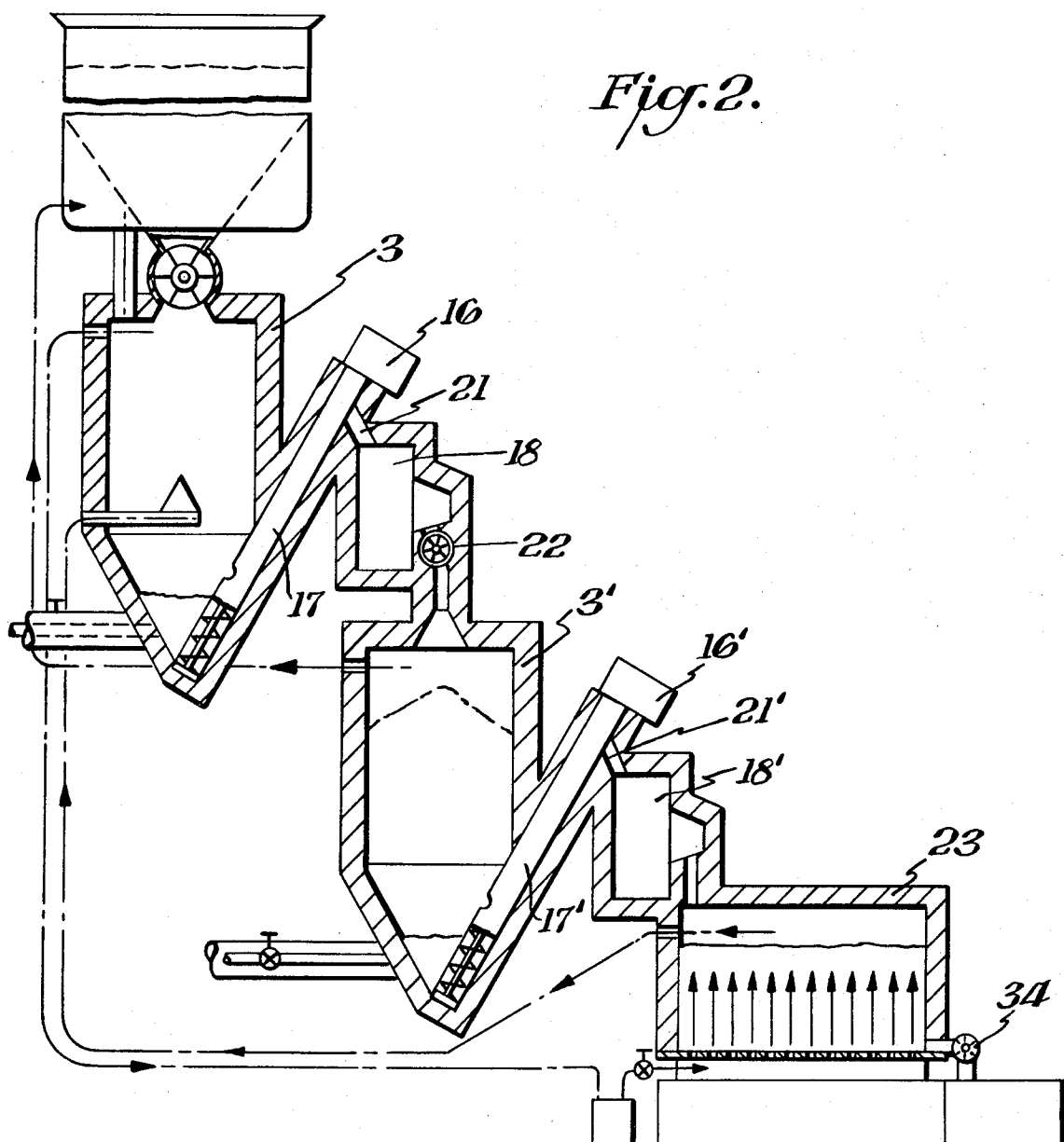


*Fig. 2.*



## PROCESS AND APPARATUS FOR THE FINE COMMUNITION OF SOLIDS

### CROSS-REFERENCE TO RELATED APPLICATION

This application is a continuation-in-part of copending application Ser. No. 786,179, filed Dec. 23, 1968, is now U.S. Pat. No. 3,614,001 issued, Oct. 19, 1971.

### BACKGROUND OF INVENTION

Parent application Ser. No. 786,179, filed Dec. 23, 1968 relates to a process wherein the material to be pulverized is passed into a countercurrent heat exchanging relation with cold vapors produced by evaporation of a low temperature liquefied gas. The material is then immersed into the liquefied gas and is pulverized in a mill.

It has been found, however, that especially thermoplastic materials cannot be comminuted to any desired fineness in one pass. This is so because of the necessary balance between the calories contained in the material below the temperature where it loses its brittleness and the calories produced by the comminution, as further explained below.

### SUMMARY OF INVENTION

In the further development of the process according to the parent application, it was found, that practically any desired fineness of grain size can be obtained in an efficient way in one operation by passing all of the obtained cold comminuted material through at least a second pass of comminution relying mainly on pressure effects, after it has been cooled down again to the temperature of the liquefied cooling gas. This following pass will lead to an average grain size substantially smaller than the one obtained by the previous pass of comminution.

By putting the two steps of comminution into the proper relation to each other, one obtains a comminuted material which has the desired corn spectrum, (i.e., screen analysis or distribution of particle size in a given quantity of particles) whereby a classification of the comminuted material for instance by sifting is not necessary.

### THE DRAWINGS

FIG. 1 shows an apparatus constructed in accordance with the invention as described in the parent application; and

FIG. 2 is a schematic showing of a portion of an apparatus modified in accordance with this invention.

### DETAILED DESCRIPTION

FIG. 1 illustrates an apparatus as illustrated and described in parent application Ser. No. 786,179, filed Dec. 23, 1968, the details of which are incorporated herein by reference thereto. As indicated therein, the hopper 1 is charged with the granular or lumpy material (e.g. different types of nylon, polyvinyl chloride, cellulose acetate, polyester, rubber, material for brake shoes for automobiles, different types of spices and raisins for baby food, etc.) to be crushed. When a motor-powered cell-type charging valve 2 is switched on the container of a counter-current heat exchanger 3 is charged. A sensing device 4 disconnects the motor of the charging valve 2 when the upper charging limit 5

has been reached and switches it on again as soon as the lower charging limit 6 has been reached during the operation of the crushing plant. When the heat exchanger 3 has been fully charged a valve 7 in a pipe 8 conducting the liquefied low temperature cooling gas slowly opens. The valve 7 may be motor-driven or may be a solenoid valve and controlled by a temperature-sensing device 9. The evaporated gas rises through the charge to be pulverized and, during its passage, cools the charge, emerging at the surface 5, or 6, nearly at the temperature of the non-cooled charge. The volume of the counter-current heat exchanger 3 is dimensioned such that this result is guaranteed. When a slight excess pressure has been attained in the space 10, the non-return valve 11 opens and the gas can escape through the connecting duct 12 into the space 13 of the hopper. The wall 14 separating the space 13 from the hopper 1 may consist of perforated sheet metal covered by a fine-mesh tissue. The gas passes through the wall 14, rises through the charge up to the surface 15 and escapes to the atmosphere. While it rises the gas surrenders any residual low temperature to the charge.

As soon as the desired low temperature has been attained in the vicinity of the sensing device 9 (approximately the temperature of the liquid gas), the motor 16 of a worm conveyor 17 is activated. The speed of the worm conveyor 17 or the inlet opening to said worm conveyor is variable so as to adapt the amounts conveyed by the worm to the output of a crushing device 18 (such as a mill using suitable serrated discs, cones or cylinders) in relation to the desired grain size. Above the highest level 19 of the liquefied gas 24 in the container the worm conveyor housing 17 has a pressure compensation orifice 20 preventing the liquid from being urged upwards in the worm conveyor housing, due to overpressure. The cooled brittle charge is conveyed through the connection duct 21 to the crushing device 18. The drive motor of the latter is switched on simultaneously with the worm conveyor motor, and so is the motor driving the cell-type charging valve 22 which takes the pulverized material to the counter-current heat exchanger 23 which operates, for example, according to the fluidized-bed principle, as illustrated in the drawing. The grain size of the pulverized material leaving the crushing device can be controlled, if desired. If the counter-current heat exchanger 23 operates according to the fluidized-bed principle it consists of a gas chamber 25, a porous plate 26 and a container 27 for the pulverized material. When the charge has reached a suitable level in the container 27 the blower 28 is switched on, this sucking in the warm vapors from the space 10 in the first heat exchanger through the pipe 29 and forcing it into the gas chamber 25. The warm vapor penetrates the porous plate 26 and rises finely distributed through the cold pulverized material in the container 27. The valve 30 is set such that the pulverized material assumes the state of a fluidized bed with a clearly defined surface, and without a lot of duct developing. The gas, after cooling during its passage through the cold, pulverized material, is forced through a pipe 31 towards a distributor which, if necessary, is provided in the shape of a hood 32 from which it issues into the charge to be crushed. The gas is led to the distributor 32 in similar manner also where a heat exchanger of different type is em-

ployed. Incorporated in the connecting pipe 31 is a non-return valve 33 which prevents the gas from flowing in the reverse direction while the plant is started up. The cooled gas rises through the charge to be comminuted into the space 10 and surrenders its low temperature to the charge.

As a result of the gas being circulated, the pulverized material assumes something like the temperature of the induced warm gas directly above the porous plate 26, whereas the material is kept cold at the surface of the fluidized bed owing to the crushed cold material continuously introduced from the crushing device 18. By means of the motor-driven cell-type discharge valve 34 the heated pulverized material is continuously transferred to the storage container 35 or filled in bags in amounts per unit of time substantially corresponding to the amounts fed in from the crushing device.

All the cold parts of the plant are preferably externally insulated against heat (this insulation being represented by shaded portions in the drawing).

The gas may be circulated in the described manner several times, if necessary. An amount of gas corresponding to the amount continuously evaporating from the liquefied gas escapes through the non-return valve 11 into the space 13, permeates the charge in the hopper 1 and then issues into the atmosphere or is used for other purposes after having surrendered any residual low temperature to the charge. To replace the amount of hot gas leaving the closed system, an equivalent amount of liquefied low-temperature gas is introduced therein.

To effect the exchange of heat between the cold pulverized material and the warm gas in the heat exchanger 23, a gas other than the gaseous medium represented by the evaporated liquefied gas may be employed, for example dry air, which may be drawn by the blower 28 and fed through the counter-current heat exchanger 23 and through the connecting pipe 31 within the circuit to the space 10 in the described manner or, through a separate pipe, to the space 13. In the latter case it would be an advantage to insulate also the container 1 against heat losses.

As used in the claims the term vapor is intended to mean a gas or a vapor.

The process according to the present invention is carried out in an apparatus such as described above but modified as shown in FIG. 2 (accordingly like numerals have been applied to like parts) whereby between the mill 18 and the second heat exchanger 23 a further heat exchanger 3' with worm conveyor 17', motor 16', connection duct 21' and mill 18' are placed. The heat exchanger 3' contains in its lower part the liquefied cooling gas and is fed by the material leaving the mill 18 and the second mill 18' uses mainly pressure effects for further comminution.

It is advantageous to connect the upper part of the heat exchanger 3' with a duct to room 13 in the hopper 1 to make use of the cold energy in the gas evaporated in the heat exchanger 3' to precool the granulated material to be ground.

In the following the underlying principals of the process for the fine comminution of solids are explained.

When grinding tough materials which were made brittle by a cold liquefied gas only a part of the cold

energy transferred to the material to be ground can be utilized, that is the part, which lies below the temperature, where the material loses its brittleness or the ability to splinter under pressure.

The preferred cooling agent is liquefied nitrogen. It is chemically inert, odorless, has a very low boiling point (minus 196° C. at 760 mm Hg) and a large cold volume in calories. Nevertheless it is relatively inexpensive in large quantities.

The granulated material to be comminuted is flooded by liquefied nitrogen. Within a short time, which does not exceed 20 seconds for normal granulated materials it acquires the temperature of minus 196° C. (the temperature of the liquefied nitrogen), while the liquid evaporates. The cold contained in the vapor or gas is transmitted to the granulated material flowing towards the cooling bath.

The cold granulated material to be ground is transported to a mill using suitable serrated discs, cones or cylinders, which according to the invention uses mainly pressure effects. The energy needed to splinter or fracture the material to be comminuted is lowest at minus 196° C. and rises for a while proportional to the increasing temperature. By the comminution heat energy is produced which first arises at the breaking areas and edges. It takes a short time before the heat is evenly distributed all over the fragments. The temperature at the edges of the fragments is considerably higher than the mean temperature of the fragments after the heat has been distributed. The further comminution by the pressure of the grinding means (discs, cones, cylinders) leads to smaller and smaller fragments with higher and higher temperature till the material becomes tough again. At this point the energy for the further comminution and the temperature of the fragments rise rapidly. The material especially in the case of thermoplastic resins becomes plastic, sticky and finally liquid. The mill gums up and may come to a standstill. Even before this condition arises the edges of the fragments become sticky whereby the fragments glue together. When tearing them apart threads are produced, which makes the material unusable.

From the above it is evident that after a single cooling of the materials especially the thermoplastic ones, they cannot be comminuted to any fineness.

The attainable fineness of the material to be comminuted depends on the energy which is necessary to fracture the different materials after they were rendered brittle. This energy differs greatly, depending on the materials. Certain types of high density polyethylene need ten times more energy at minus 130° C. than PVC at minus 55° C. Furthermore, the corn fineness attainable in one pass depends on the efficiency of the grinding means. As little friction as possible shall arise during the breaking or fracturing by pressure. Therefore the material to be comminuted has to be conducted over the shortest attainable distance through the grinding means.

If the total of the ground material is led a second time through the same grinding means it will become only insignificantly finer. When using the known grinding processes the usable powder is separated from the too coarse and the too fine powder. The too coarse part is cooled down and ground a second time. With the types of mills used so far the same percentage of too coarse

powder is produced at the second, third or further pass as has arisen at the first pass. It is the advantage of mills working with pressure effects that the grinding means can easily be exchanged or altered.

The process according to the parent application can be applied in such a way that the usable powder is separated from the too coarse material, for instance by sifting. Then the too coarse material is ground again after cooling it down and after the grinding means have been exchanged or altered so that the ground material, after the second pass, has the corn spectrum desired.

It was found, however, that surprisingly it is more efficient if the whole quantity of the material ground in the first pass according to the main patent is continuously in one operation subjected to a second pass after it has been cooled down again to minus 196° C. Further passes can be added as necessary.

The reasons for the efficiency of this method are the following: Using the process according to the invention in question the comminuted material leaves the mill normally with a temperature below minus 110° C.

The process has to be led in such a way that the material comes out of the mill with such a low temperature so that the fragments do not sinter together at the edges and that a sharply defined easily flowing corn is obtained.

As furthermore the specific heat of materials becomes lower in proportion to the decreasing temperature one needs only one quarter of the liquid nitrogen for cooling the material from minus 110° C. to 196° C. than for cooling it from room temperature to 196° C. Consequently, less liquid nitrogen is needed and a separating or sifting operation is saved.

As pointed out in the parent application, the crushing device suitably used in accordance with the invention is a mill operating generally with shearing and frictional effects, but predominately with a compression effect, for example a mill with grinding or crushing disks, cones, wheels or cylinders the surface of which has been serrated suitably and the distance of which can be altered; in which the charge substantially does not follow long, accelerated random paths of movement as is the case, for example with pin, hammer, rebound crusher mills, etc., which operate either predominately or exclusively on the impact or rebound principle.

What is claimed is:

1. A method of pulverizing material in a crushing mill comprising the first step of passing the material to be pulverized into a countercurrent heat-exchanging relation with cold vapors produced by evaporation of a low-temperature liquefied gas, the second step of immersing the material in the liquefied gas from which the vapors have evaporated, the third step of conveying the material from the liquefied gas to the mill, and the fourth step of pulverizing the material in the mill, the fourth step taking place in the mill predominantly operating with crushing elements, the grain size of the pulverized material being determined in accordance with dimensions of the crushing elements of the mill, and the material being conveyed through the mill solely by mechanical force, and all of this cold material, pulverized in the fourth step after its cooling down to the temperature of the liquefied cooling gas, being subjected to at least one further pass of comminution taking place in a mill of the type used in the fourth step with serrated crushing elements having its serrated surfaces arranged to substantially reduce the average particle size in comparison to the preceding pass by predominantly compression effects.

2. The process of claim 1 wherein liquefied nitrogen is the cooling agent.

3. In an apparatus for pulverizing materials comprising a hopper for the material to be crushed, a first countercurrent heat exchanger communicating with the hopper whereby the material may be fed from the hopper, inlet means in the first heat exchanger for controllably allowing a low-temperature liquefied gas to form an immersion region of a controlled depth at the bottom of the heat exchanger, a mill communicating with the heat exchanger for pulverizing the material after immersion in the liquefied gas, including a second countercurrent heat exchanger for cooling a vapor, means for leading the resultant cooled vapor back to the first heat exchanger to assist in precooling of the material before its immersion in the liquefied gas, the second heat exchanger and the first mill communicating with each other by means of a further heat exchanger containing a cold liquefied gas at its lower part and being fed with the material leaving the first mill, a further mill between the second and further heat exchangers, and the further mill having serrated crushing elements using shearing and frictional, but predominantly compression effects for comminution.

\* \* \* \* \*

50

55

60

65