United States Patent

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[54] METHOD FOR GRANULE PULVERIZATION

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[52] **U.S. Cl.** **241/5,** 241/23, 241/48, 241/65

[51] **Int. Cl.** **B02c 19/06**

[58] **Field of Search** 241/5, 17, 18, 23, 48, 65

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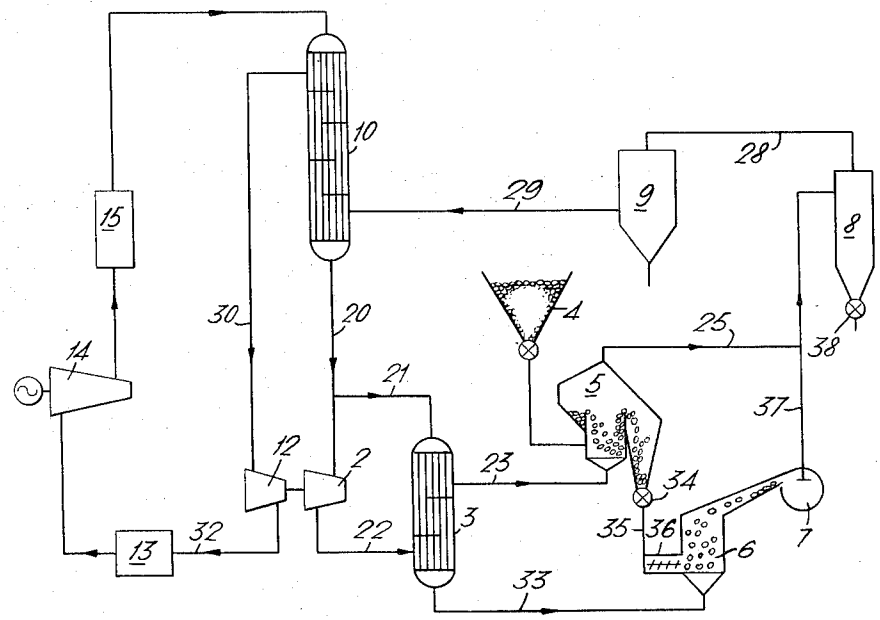
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[57] **ABSTRACT**

Apparatus and process for pulverizing granules at low temperatures in a jet mill is provided wherein two cold-gas streams are used, one for precooling the granules to be pulverized and the other for cooling the granules as they are pulverized.

7 Claims, 2 Drawing Figures



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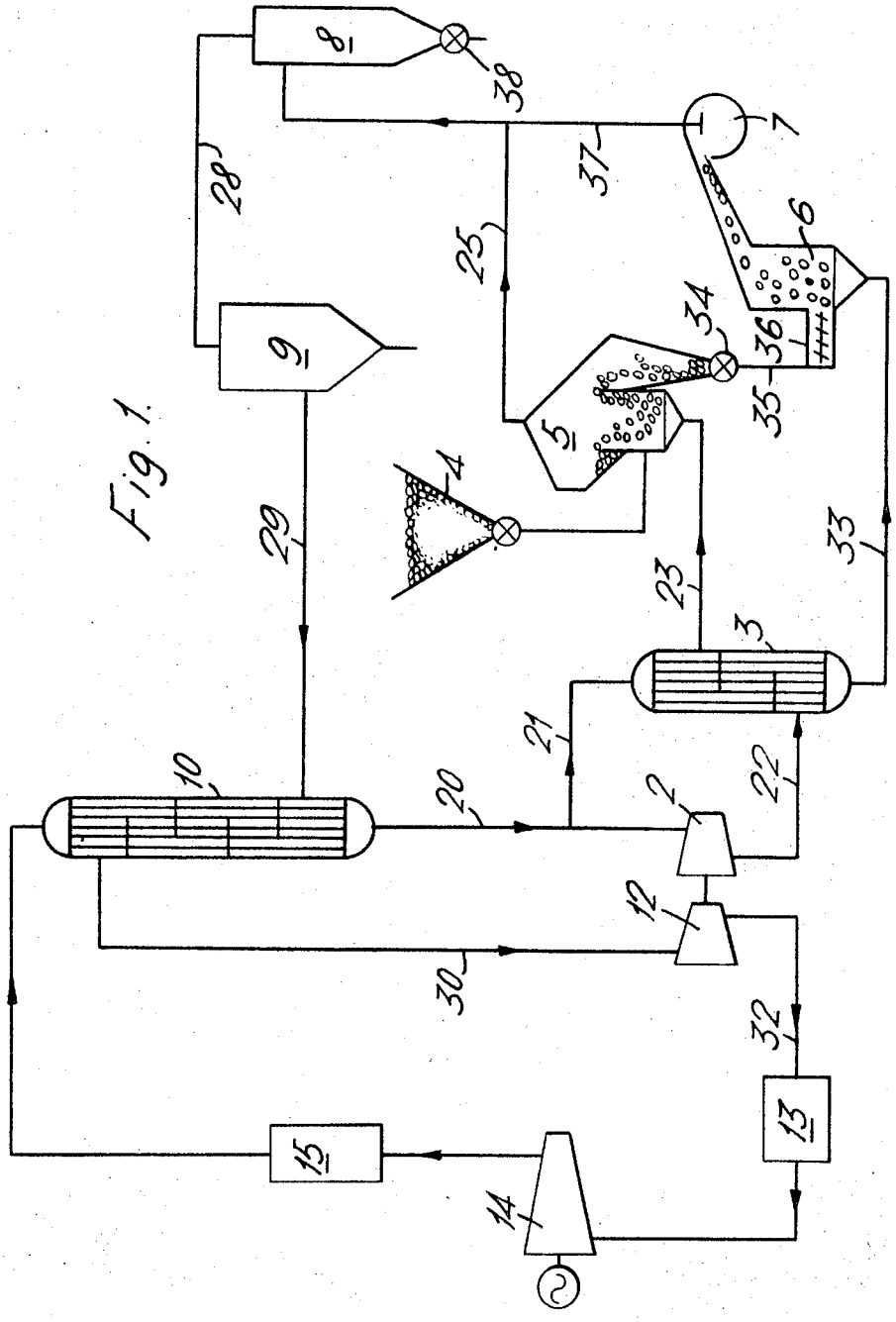


Fig. 1.

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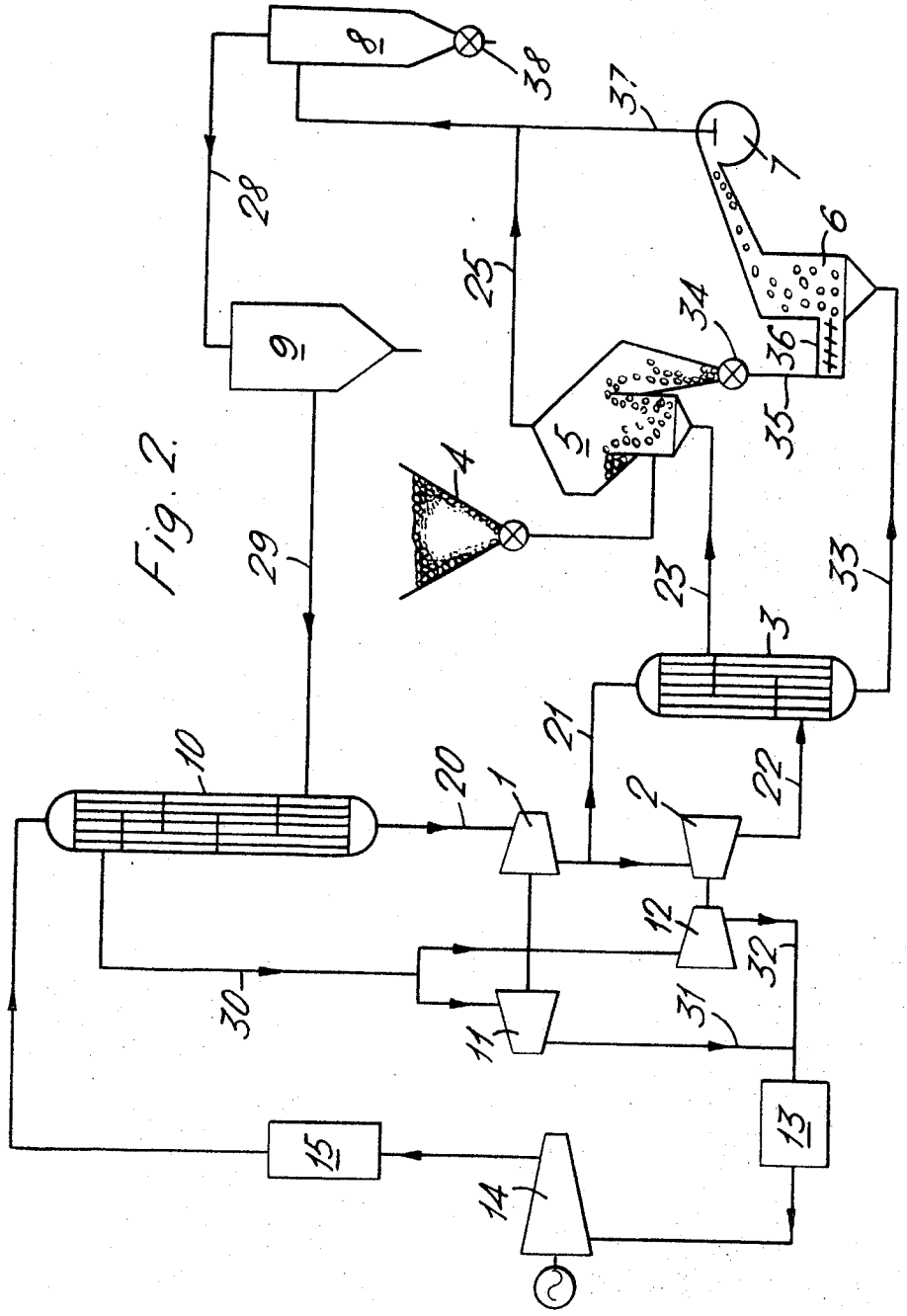


Fig. 2.

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METHOD FOR GRANULE PULVERIZATION

For grinding granular materials and plastics granules into powder of medium fineness (for example, in the range of 50 – 500μ) mechanical mills operating on the impact principle are mostly employed. Polyamides of the 6, 6,6 and 12 types and copolymers of these types have a much greater toughness than the materials which are normally pulverized with these mills. These polyamides therefore cannot be pulverized at normal temperatures or can be pulverized only with a very great expenditure of energy, which is fed into the mill as mechanical energy and is converted into heat therein. If this heat cannot be removed rapidly, temperatures that are too high occur. As a result, the material may be partly heated to melting point and the grinding effect is thereby reduced and the output of the mill is considerably decreased. By reducing the temperature of the charging product before it enters the mill and by removing heat from the mill itself, the grinding conditions can be considerably improved, since the brittleness and consequently the destructibility of the grains fed in are increased. In this manner, the expenditure of energy for grinding is considerably reduced as is the temperature increase within the mill.

A known method of precooling the granules and removing the heat of grinding consists in that pre-ground solid carbon dioxide is admixed with the charging product. In spite of the relatively good grinding results achieved in this way in an impact mill, it has been found that the energy required to carry out pulverization and for turbulence losses is about 20 to 30 times greater than in the grinding of mineral substances in mills of similar type in order to obtain the same fineness or specific area of the ground product. This energy converted into heat is generally used for vaporizing the solid carbon dioxide that is introduced and it is also used for reheating the precooled charging product, so that there is a considerable increase in temperature in the mill. This method has the disadvantage that uniform metering of the cooling medium is difficult to attain so that the maintenance of a precise temperature cannot be readily achieved. The minimum grinding temperature is limited by the vaporization temperature of the solid carbon dioxide. Moreover, the handling of this cooling medium is complicated and, in consequence of the high cost of procuring solid carbon dioxide, operation is very uneconomical.

Another known method of achieving the desired low grinding temperatures consists in the use of liquid nitrogen as a cooling medium. However, liquid nitrogen is extremely expensive, and the controllability of the system is limited. Moreover, if there is direct contact of the liquid nitrogen with the product, a deterioration in the structure of the material and in the comminution properties of the material may be caused by the extremely low temperatures.

On the other hand, it is known that granular materials, in particular color pigments, pharmaceutical products, pest-control agents and other materials, can be ground in jet mills to very great degrees of fineness (1 to 10μ). It has also been possible to grind plastics with rather high melting points successfully with medium degrees of fineness. These jet mills are operated partly with steam jets and partly with compressed air. Through the repeated introduction of the coarse grains into the pulverization zone with successive removal of the fine grains at the same time, a very uniform grain size can consequently be obtained. When air is employed as a propellant, a considerably lower temperature is obtained in the working jet after expansion. The temperature of the air increases to the original temperature again only through the turbulence in the pulverization chamber. Mechanical energy does not have to be supplied to the mill, in contrast to mechanical mills which operate on the impact or impingement principle. In the jet mill, the required mechanical energy is introduced in the air compressor and the heat generated thereby can consequently be removed in the conventional way by cooling water at normal temperature before the air enters the mill.

If, however, lower grinding temperatures than are obtained in normal operation with a jet mill are required, then an admixture of solid carbon dioxide or of liquid nitrogen is also possible in principle in this instance, but in such case it is also necessary to put up with the disadvantages mentioned in connection with mechanical mills. Moreover, jet mills have the great drawback that the consumption of energy for producing a certain product area is several times greater than in mechanical mills, so that jet mills are not normally used for the production of cheap bulk goods, but only for high-grade products or where a high degree of fineness is required.

Surprisingly, it has now been found that the disadvantages of the grinding processes which have been described above can be avoided even with the use of a jet mill if a new process is employed, in accordance with the instant invention.

In accordance with the present invention a jet mill is employed for fine grinding and a cold-gas circuit is employed for refrigeration, the required low temperatures being obtained by the expansion of a precooled stream of gas. In this way, the advantages obtained through use of a jet mill are retained while achieving greater economy than that achievable with a mechanical mill.

The expansion is carried out in conventional expansion machines for which purpose expansion turbines are used with advantage. These turbines can either be braked electrically with the interposition of conventional gearing to obtain mechanical power to run other portions of the cold-gas circuit or directly with a blower, the second case also having the advantage, apart from simplicity, that a pre-compression of the recycle gas can be effected.

The process according to the invention is characterized by the grinding being carried out in a jet mill with the aid of a working gas stream which is taken from a cold-gas circuit which achieves the low temperature required for the working gas stream and for the precooling of the charging product by approximately isentropic expansion of a precooled and compressed recycle gas, this precooling being effected by heat exchange with expanded recycle gas obtained after its use for cooling the charging product and the removal of the generated heat of grinding.

The process can be carried out in various ways. Thus, for example, the compression of the recycle gas may be effected only to the working pressure of the jet mill, following which the recycle gas is divided after precooling into two partial streams, the first of which is further cooled by expansion in a turbine. The second partial stream is reduced to approximately the temperature of the first partial stream by heat exchange, whereupon the compressed second partial stream serves as the working gas stream for the jet mill, while the expanded first partial stream is employed for precooling the charging product.

Another way of carrying the process into effect consists in compressing the recycle gas to a pressure which is two to 30 atmospheres higher than the working pressure of the jet mill. In this case, the entire amount of recycle gas is expanded to the pressure of the jet mill, whereupon the total stream is divided into two partial streams, one of which is used for precooling the charging product and the other as working gas stream for the jet mill.

In this method of carrying the process into effect, which is suitable when somewhat higher temperatures can be tolerated, the partial stream which is not carried through the jet mill only has to be compressed to the compression pressure from the working pressure of the mill, thereby reducing the energy input necessary to carry out the process.

For very low temperatures, however, the preferred method of carrying the process into effect is that in which the partial stream which is not carried through the jet mill is expanded in another expansion stage to the outlet pressure of the jet mill, as a result of which its temperature is lowered further so that by means of heat exchange the working gas stream for the jet mill can also be cooled still further.

Air is preferably employed as recycle gas, since in this way the cost of replenishing the system in the event of leakage losses would probably be lowest. If required, other gases which are not liquified in the operating range of the turbines may also be employed. Of course, if air is employed, it is also possible to use a so-called open circuit and in this case precautions must be taken for eliminating foreign gases (for example, water vapor and CO_2) which crystallize out at low temperatures from the circuit.

The process according to the invention allows the temperature during the grinding operation to be adjusted within wide limits and with simple means conventionally used in control engineering to any desired level and to be kept exactly constant at this level.

The adjustment may be effected by a variation of the pressure gradient in the refrigerating circuit, a variation of the amount of gas in circulation or of the amount of the partial stream passing through the mill, as well as by varying the effective heat-exchanger area.

The cost of the refrigeration by the new process is considerably lower than in the other processes that are known and handling is extremely simple. Due to the use of a refrigerating circuit for cooling the gas used in the grinding operation, a separate compressor for the jet mill can be saved, as a result of which the cost to construct the mill and the cost of its operation is greatly reduced.

FIG. 1 illustrates an embodiment of the process according to the invention.

FIG. 2 illustrates a second embodiment of the process according to the invention.

The recycle gas is compressed by the compressor **14** to the pressure of the jet mill **7** and is freed from the heat of compression in the after-cooler **15** by means of cooling water. The recycle gas is further precooled in the heat exchanger **10** to a temperature determined by the existing gas pressure and the desired grinding temperature. The recycle stream coming from the line **20** is branched into two partial streams, one partial stream being expanded in the turbine **2** and being cooled further in the process. Through the line **22** this partial stream enters the heat exchanger **3**, where it absorbs heat by cooling the second partial stream flowing in through the line **21**. The first partial stream enters the fluidized bed cooler **5**, where the plastics granules fed from the supply vessel **4** are precooled and are brought by way of the bucket lock **34**, the line **35** and the conveyor worm **36** into the fluidized bed cooler **6**, where the low-temperature cooling is effected by the second partial stream flowing in through the line **33**. The resulting mixture of granules and air passes into the jet mill **7**, where the grinding into powder takes place. The powder-charged second partial stream enters a cyclone separator **8** through the line **37** after being combined with the first partial stream from the line **25**, in which separator the powder can be separated from the air and drawn off by way of the lock **38**. The recycled air, charged with fine residues of powder, passes through the line **28** into the filter **9** and is finely purified therein. The clean air is supplied through the line **29** to the heat exchanger **10**, where it absorbs heat from the compressed air. The purified preheated air is fed through the line **30** to the blower **12**, is precompressed therein in accordance with the output available and, after cooling in the intermediate cooler **13**, is supplied to the compressor **14** again, where the cycle begins afresh.

Another embodiment of the process will now be described with reference to FIG. 2.

5,000 kg of air at **15** atmospheres absolute are expanded in the expansion turbine one to six atmospheres absolute, the air being cooled in the process from −90° C. to −122° C. A partial stream of 2,500 kg of air is expanded in the turbine **2** from six atmospheres absolute to 1.8 atmospheres absolute. In the process, the air is cooled to −157° C.

This partial stream passes through the line **22** into the heat exchanger **3**, where it is conveyed in counter-current to the second partial stream, which has been separated from the first partial stream via the line **21** at the intermediate pressure of six atmospheres absolute. In the heat exchanger **3**, the first partial stream is heated from −157° to −122° C. and passes through the line **23** into the fluidized bed cooler **5**. Plastics granules are fed to this cooler from the supply vessel **4** and are cooled from +20° to −110° C. by the first partial air stream. The precooled granules pass through a bucket lock **34** into the line **35** and are conveyed by means of the conveyor worm **36** into the fluidized bed cooler **6**, where they are cooled to −140° C. by the second partial air stream. The second partial air stream leaves the heat exchanger **3** at a temperature of −150° C. and is conveyed through the line **33** to the fluidized bed cooler **6**, where the air becomes heated to −142° C. The cold mixture of air and granules now enters the jet mill **7**, where the air expands to a pressure of about 1.5 atmospheres absolute. Immediately after the expansion, the temperature of the air is reduced to −130° C. and the air is then heated to −120° C. through turbulence and absorption of heat which is generated during the pulverization of the granules. The plastic is ground in the mill into powder with a particle size of 100 to 200μ. The mixture of powder and air leaves the mill **7** by way of the line **37** and combines with the first partial air stream coming from the fluidized bed cooler **5** at a temperature of −113° C. The powder is separated from the air in the cyclone separator **8** and can be drawn off via the lock **38**. The air charged with residues of fine powder passes to the filter **9** where the powder is retained and the pure air is drawn off through the line **29** and enters the heat exchanger **10** at a temperature of −116° C., being heated therein to −1° C. in counter-current to the compressed air. Due to the pressure losses of the various interposed pieces of apparatus, the pressure of the recycle air in the line **30** is about one atmosphere absolute. The air stream is distributed between the two blowers **11** and **12** in accordance with the output available and is compressed therein to about 1.5 atmospheres absolute. Via the lines **31** and **32**, respectively, the recycle air arrives at the intermediate cooler **13** and from here at the compressor **14**, where compression to 15 atmospheres absolute is effected. In the after-cooler **15**, the heat of compression is transferred to cooling water and the air finally passes at a temperature of about 20° C. to the heat exchanger **10**, where it is precooled to −90° in counter-current to the low-pressure air, and in the end is again fed to the turbine **1**, whereby the cycle recommences.

The process is not limited to the values mentioned in the example, but the pressures may vary within wide limits. Thus, the pressure in advance of the turbine may be, for example, between five and 40 atmospheres absolute, the intermediate pressure between the turbines **1** and **2** may be three to 10 atmospheres absolute and the pressure in the low-pressure system may vary, for example, from 0.5 to two atmospheres absolute.

The temperatures may also range within wide limits, depending on the nature of the materials to be ground and the pressures employed.

While only a limited number of embodiments of the foregoing invention have been expressly described, it is nonetheless to be broadly construed and not to be limited except by the character of the claims appended hereto.

I claim:

1. A process for grinding granular material at low temperatures in a jet mill comprising the steps of contacting the granular material with a first stream of cold gas before the granular material is fed to the jet mill to precool said granular material, feeding the precooled granular material to the jet mill, pulverizing said granular material in said jet mill, by means of introducing an expanding second cold gas stream into said jet mill effecting the granular material being pulverized and to reduce the temperature of the granular material as it is being pulverized.

2. The process specified in claim **1** further including the steps of admixing the first and second gases downstream of the jet mill, separating the pulverized material from the admixed gases, and recycling the admixed gases to a cold gas circuit for reuse.

3. The process specified in claim 1 further including the steps of cooling said first stream of gas by substantially isentropic expansion to a predetermined low temperature, said second stream of gas being substantially at the working pressure of said jet mill, passing said first and second stream of gas in proximity to each other to effect a cooling of said second gas stream to a predetermined low pressure, and then cooling said granular material with said first stream of gas and feeding said second stream of gas to said jet mill.

4. The process specified in claim 3 wherein said first and second streams of gas are admixed downstream of said jet mill, feeding said admixed gas to a heat exchanger in which said admixed has is heated, compressing said heated admixed gas to approximately the working pressure of said jet mill, and separating said compressed gas into said first and second streams of gas.

5. The process specified in claim 4 wherein said first and second streams of gas are at different temperatures and further including the step of bringing the temperatures of said first and second streams of gas closer to each other by passing the said streams of gas in close proximity to each other before feeding the said streams of gas to the granular material and the jet mill.

6. The process specified in claim 1 further comprising the steps of admixing said first and second streams of gas, recycling and compressing said admixed gas, cooling said admixed gas by substantially isentropic expansion to a predetermined low temperature, separating said expanded gas into said first and second streams of gas, further expanding said first stream of gas to a still lower pressure and temperature, passing said first and second streams of gas in proximity to each other to effect a heat exchange between them, and then feeding said first stream of gas to said granules and said second stream of gas to said jet mill.

7. The process specified in claim 6 wherein the admixed gas is compressed to a pressure which is between two and 30 atm higher than the working pressure of the jet mill, expanding said compressed gas to the working pressure of the jet mill, and then separating said expended gas into said first and second gas streams.

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