Publication Number: 03531310 **Section: Claims** **5 of 6 pages** Help pigment was tested for fineness of grinding by the 3M Test, heretofore described. The results of these tests are tabulated in Table I.

TABLE I

	Grind media		3M Fines			
	First pass	Second pass	5 min.	10 min.	15 min.	20 min.
Run:						
1	Steam	None	3.75	4.0	4.5	5.0
2	do	Steam	2.75	3.0	4.0	5.0
3	Nitrogen	None	3.5	4.5	4.5	5.5
4	do	Nitrogen	2.5	4.0	5.5	5.0
5	Steam	do	4.75	5.0	6.0	6.5
6	Nitrogen	Steam	0.5	2.0	3.75	4.0

The data in Table I show that fluid energy milling with steam followed by nitrogen milling (Run 5) produced a product that had a better fineness of grind than products produced by single passes of steam or nitrogen (Runs 1 and 3), double passes with steam or nitrogen (Runs 2 and 4), than other combinations. Portions of this data are plotted in the attached figure, which graphically illustrates that the combination of steam milling followed by nitrogen milling yields a product of increased fineness of grind.

Example II

Example I is repeated except that air and carbon dioxide are substituted in turn for nitrogen as the fluid energy milling fluid in Runs 3, 4, 5, and 6. Fineness of grind measurements by the 3M Test show that steam-carbon dioxide and steam-air milling combinations yield a product of improved fineness of grind than sequential carbon dioxide-steam and air-steam millings, double stage milling with carbon dioxide or air, and single stage milling with steam, carbon dioxide, or air.

Example III

Titanium dioxide prepared and coated with hydrous titania and alumina in a manner analogous to Example I was micronized with steam having a temperature of about 430° F. and a pressure of about 150 p.s.i.g. A portion of the pigment was incorporated into a paint vehicle; the paint fines were measured by means of a Hegman gage and found to be 3. The tint efficiency of this pigment was measured and found to be 91 percent. Another portion of the steam-milled pigment was fluid energy milled twice with nitrogen in a Trost Mill having an ambient temperature, i.e., about 70° F. Pusher and opposing jet pressures were respectively about 240 and 220 p.s.i.g. This pigment was incorporated into a second equal quantity of the same paint vehicle. The paint fines of the steam-nitrogen milled pigment were measured and found to be 7 Hegman. The tint efficiency was found to be 95 percent.

A repeat of the above-described procedure increased the Hegman paint fines from a value of 2 to 7¾ and the tint efficiency from 89 to 96 percent.

Example IV

Titanium dioxide prepared and coated in a manner analogous to Example I was steam micronized with steam at a temperature of about 430° F. and a pressure of 150 p.s.i.g. A paint prepared with this pigment had a paint fines measurement of 4 on the Hegman gage and a tint efficiency of 95 percent. A portion of the steam milled pigment was double milled with carbon dioxide at ambient temperature in a Trost Mill at a pusher jet pressure of about 240 p.s.i.g. and an opposing jet pressure of about 220 p.s.i.g. This CO_2 milled pigment was incorporated into a second equal quantity of the same paint

Example V

Titanium dioxide pigment prepared and coated in a manner analogous to that described in Example I was fluid energy milled in a 20-inch micronizer with steam having a temperature of 470° F. at a grind pressure of 155 p.s.i.g. The steam rate was about 950 pounds per hour and the titanium dioxide feed rate was from 7 to 12 pounds per minute. The titanium dioxide product from the 20-inch micronizer was tested for dispersion by means of the Cowles dispersion test. After ten minutes in the paint vehicle, the pigment tested from 6 to 7½ on a Hegman gage. The 20-inch steam micronized titanium dioxide was fed to a T–15 Trost mill and fluid energy milled with nitrogen having a temperature of from 125 to 135° F. The nitrogen feed rate was approximately 480 pounds per hour and was introduced at about 210 p.s.i.g. The TiO_2 feed rate was about 120 pounds per hour and was introduced at a feed pressure of about 225–240 p.s.i.g. The Hegman gage results after ten minutes milling in the Cowles dispersion test for the nitrogen milled pigment was 7¾. This data illustrates that the dispersion of titanium dioxide pigment was significantly improved by fluid energy milling the pigment with nitrogen after a preceding fluid energy milling with steam.

Example VI

The fluid energy milling procedure described in the the present specification and as illustrated in the preceding examples is utilized in the fluid energy milling of zinc oxide, antimony oxide, silicon dioxide, and zinc sulfide pigments. Improvements in dispersion of these pigments is realized by the use of the above-described procedure of steam followed by a substantially non-condensable, liquid-free gas fluid energy milling. A blend of 85 weight percent titanium dioxide and 15 weight percent silica is also submitted to the above-described milling procedure and the dispersion property of the blend is similarly improved.

While there are above described a number of specific embodiments of the present invention, it is obviously possible to produce other embodiments and various equivalent modifications thereof without departing from the spirit of the invention.

Having set forth the general nature and specific embodiments of the present invention, the true scope is now particularly pointed out in the appended claims.

What is claimed is:

1. In a process for fluid energy milling substantially dry pigmentary titanium dioxide, the improvement which comprises subjecting the pigment to at least one fluid energy milling with a gaseous fluid comprising steam and thereafter to at least one fluid energy milling with a substantially liquid-free, non-condensible gas chemically inert to the pigment, said liquid-free gas having a temperature of from about 20° C. to about 70° C.

2. A process according to claim **1** wherein the titanium dioxide fluid energy milled has a coating of at least one