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METHOD FOR MILLING A POWDER

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(57) Claim

1. A method for producing a milled powder including:

(1) providing a starting powder;

(2) imparting an electrostatic charge to said starting powder to form a charged powder; and

(3) milling said charged powder to produce a milled powder.

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METHOD FOR MILLING A POWDER

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The following statement is a full description of this invention, including the best method of performing it known to applicant(s):

METHOD FOR MILLING A POWDER

Background of the Invention

The invention relates to a method for milling a powder.

Commercially useful powders including pigments, fillers and waxes typically undergo micronizing as a final processing step before collection and packaging of the powders. Micronizing refers to comminution of a starting powder to produce a product powder having a particle size in the submicron range, usually in the range of from about 0.01μ to about 1.0μ. Fluid energy mills are commonly used to perform this micronizing step. Fluid energy mills use a high pressure fluid such as a high pressure gas stream to impart shear forces to the starting powder to be micronized, thereby reducing particle size to the desired submicron particle size range.

Milling aids may be introduced into the fluid energy mill to improve the efficiency of the micronizing process so that less power needs to be expended to achieve a particular degree of grind. While milling aids and/or their residues have the desirable effect of improving milling efficiency, these milling aids become incorporated in the product powder and, depending upon the end use of the product powder, can lead to undesirable side reactions. For example, when a product powder, including residual milling aids is used as a pigment in an acid-catalyzed paint system, unwanted chemical reactions such as premature initiation of the epoxy-curing reaction in epoxy-based coating systems can occur. Also, when the product powder is used as a pigment or filler in a plastic, milling aid residue can be volatilised in high temperature plastic processing to generate undesirable vapors.

Collection of micronized product powders for packaging is made especially difficult because of the small particle size of the micronized powders. Micronized powders are conventionally collected using a bag filter

or cyclone collector or, rarely, using an electrostatic precipitator. Currently, the extent to which a starting powder can be micronized is limited by collection efficiency for the micronized powder.

Thus, there exists a need for a method for efficiently milling a starting powder without the use of additives whose residues can cause undesirable reactions when the powder is used in a particular application. It is also required that the milling method not further complicate collection of the milled product powder. It is preferred that the milling method also improves collection efficiency of the product powder.

It would be advantageous to provide a method for milling a powder reasonably efficiently so that the amount of power required to produce a specified particle size or degree of grind is minimized without the use of additives and so that ease and efficiency of collection of the milled powder is improved.

Summary of the Invention

According to an aspect of this invention there is provided a method for producing a milled powder including:

(1) providing a starting powder;

(2) imparting an electrostatic charge to said starting powder to form a charged powder; and

(3) milling said charged powder to produce a milled powder.

Preferably the method further includes:

(4) providing a further starting powder;

(5) imparting a further electrostatic charge, opposite to said one electrostatic charge, to said further starting powder to form a further charged powder;

(6) milling said further charged powder to produce a further milled powder;

(7) combining said further milled powder with a substantially equal quantity of said one milled powder to form a product powder; and

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(8) collecting said product powder.

Description of Preferred Embodiments

A method for producing a milled powder in accordance with the invention manifest itself in a variety of forms. A detailed description of several preferred embodiments are described below for the purposes of providing an enabling disclosure. It is to be understood however that the specific nature of this description does not supersede the generality of the preceding statements.

The invention provides a method for efficient milling of a starting powder without the use of additives by imparting an electrostatic charge to the starting powder before milling.

According to a preferred embodiment of the invention, a second starting powder can be provided, electrostatically charged to form a second charged powder having an electrostatic charge opposite to that of the charged powder, milled to produce a second milled powder and combined with the milled powder to form a product powder which is finally collected. The product powder can be collected using a product powder collector such as a cyclone, a bag filter or a settling chamber.

The starting powder can be non-electrically conductive and can be made up of insulating particles. As used herein in the specification and claims, the term "electrically conductive" is used to refer to a powder having an electrical resistivity in the range of from about 0μohms-cm to about 200,000μohms-cm, while the terms "electrically non-conductive" and "insulating" are used to refer to powders and individual particles of a powder having an electrical resistivity greater than about 200,000μohms-cm. The starting powder particles can also be characterised by a dielectric constant greater than about 1.5 as measured at $1.0X10^6$Hz at room temperature. Particles having a dielectric constant in the range of from about 4 to about 100 are particularly suited for use in the

method of the invention; however, any material which holds a charge well can be used in the method. The insulating particles can be titanium dioxide, zinc oxide, zirconia, alumina, silica, wax, polymer, inorganic oxide or organic material particles or mixtures thereof.

The starting powder can also be a non-electrically conductive aerosol made up of particles which are themselves electrically conductive, but present at such a dilution that no continuous path for conduction of electricity exists within the aerosol. As used herein in the specification and claims, the term aerosol refers to a finely divided powder in a continuous phase gaseous medium and conductive particle dilutions which result in a non-electrically conductive aerosol are in the range of from about 0.0001 lb/ft^3 to about 0.1 lb/ft^3. Electrically conductive particles appropriate for use in such a non-electrically conductive aerosol are carbon black or metallic particles or mixtures thereof. Metallic particles can include particles of metals and their alloys such as aluminum, copper, brass and the like and mixtures thereof.

The method of the invention is useful in grinding powders to particle sizes in the micron range and especially in the submicron range, i.e., in the range of from about 0.01μ to about 1.0μ under fairly dilute conditions such as particle concentrations in the range of from about 100:1 to about 4:1 fluid to solids ratio wt/wt where conventional grinding processes often fuse individual particles together rather than comminuting them into smaller particles.

The starting powder can be characterized by a starting powder particle size less than about 1cm. The closer the starting powder particle size is to the milled powder particle

size, the more efficient the milling process will be. The first and second milled powders can be characterized by first and second milled powder particle sizes in the range of from about 0.01μ to about 1000μ, more preferably in the range of from about 0.05μ to about 100μ and most preferably in the range of from about 0.1μ to about 10μ.

The starting powder can be electrostatically charged by providing a milling fluid and charge generator and flowing the milling fluid through the charge generator so that the milling fluid becomes electrostatically charged. The milling fluid imparts kinetic energy to the starting powder to result in comminution of the starting powder and can be selected from among nearly any fluid depending upon the particular characteristics of the powder being milled, such as powder reactivity, flammability and explosion hazard as considered in conjunction with requirements for milling efficiency of the milling fluid. The milling fluid can be compressed air or superheated steam. Hydrogen gas exhibits a high potential velocity upon expansion and is a highly efficient milling fluid for powders where explosion hazard is not a consideration. If explosion hazard, particle reactivity or flammability are important considerations, fairly chemically non-reactive gases such as nitrogen, xenon or argon can be used as the milling fluid. Depending upon the particular requirements for the milling fluid, mixtures of the already-described gases can also be used.

The charge generator can be a high voltage source capable of providing a DC voltage in the range of from about 10,000volts to about 1,000,000volts, more preferably in the range of from about 10,000volts to about 500,000volts and most preferably in the range of from about 10,000volts to about

100,000volts. The charge generator can also be an AC high voltage source providing an AC voltage in the range of from about 10,000volts rms to about 100,000volts rms at a frequency of from about 1Hz to about 10,000Hz.

Alternatively, a charge generator, such as those already described, can be used to impart charge directly to the starting powder.

The method of the invention is compatible with any milling apparatus or method as known to one skilled in the art including a jet mill or autogenous grinding process.

In order to further illustrate the present invention, the following example is provided. The particular compounds, processes and conditions utilized in the example are meant to be illustrative of the present invention and are not limited thereto.

Example 1

The following example is provided to demonstrate how a pigment powder milled according to the method of the invention displays superior chemical inertness when used in a paint and superior recovery efficiency after milling by comparison with a pigment powder milled conventionally using an amine additive.

A standard enamel grade TiO_2 pigment was prepared according to the following steps from a raw pigment obtained from oxidation of $TiCl_4$ and $AlCl_3$. The raw pigment was dispersed in water with addition of 0.25% wt/wt of an inorganic dispersant, and the pH was adjusted to 10 using sodium hydroxide. The dispersed pigment was sandmilled for eight minutes with 10-40 mesh silica sand to deagglomerate the pigment. An amount of sodium aluminate equivalent to 1.4% wt/wt Al_2O_3 was added to the pigment. Sulfuric acid was then added in an amount sufficient to neutralize twice the alumina added. Then additional sodium aluminate was added to bring the total Al_2O_3 added to 2.8% wt. with respect to wt. dispersed solids. Finally, the pH was adjusted to 7.0 by addition of 1 normal acid and 1 normal caustic.

The resulting aqueous slurry was filtered, repulped with additional water and dewatered with an additional filtration step to remove soluble salts from the pigment. The washed filtercake thus obtained was dried with a conventional gas fired dryer and the dried product was divided into two equal samples.

The two equal samples were then jet milled. One sample was jet milled according to conventional practice using an amine additive and the other sample was electrostatically charged and then jet milled.

The first sample was treated with 0.25% wt. with respect to wt. dispersed solids triethanol amine and jet milled with super heated steam.

The second sample was further subdivided into two equal parts. The first part was charged to 15,000 KV/inch using a hand-held voltage source, and jet milled. The second part was simultaneously equally and oppositely charged and milled in a separate jet mill. The oppositely charged first and second parts were then collected together in a single cyclone collector yielding a collected second sample.

The performances of the amine-treated first sample and the electrostatically processed second sample in a paint were compared. The first and second samples were each separately incorporated into a two part epoxy paint. The paint containing the amine-treated first sample exhibited undesirable gellation after one week caused by the amine additive to the pigment. The paint containing the electrostatically processed second sample exhibited no gellation or other undesirable side effects of pigment addition.

Collection efficiency after jet milling was also compared for the amine-treated first sample and the electrostatically processed second sample. About 90% of the amine-treated first sample was recovered from the jet milling step, while about 99% of the electrostatically processed second sample was recovered from the jet milling step.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A method for producing a milled powder including:

(1) providing a starting powder;

(2) imparting an electrostatic charge to said starting powder to form a charged powder; and

(3) milling said charged powder to produce a milled powder.

2. A method according to claim 1 further including:

(4) providing a further starting powder;

(5) imparting a further electrostatic charge, opposite to said one electrostatic charge, to said further starting powder to form a further charged powder;

(6) milling said further charged powder to produce a further milled powder;

(7) combining said further milled powder with a substantially equal quantity of said one milled powder to form a product powder; and

(8) collecting said product powder.

3. A method according to claim 1 or claim 2 wherein said one and further starting powders are non-electrically conductive.

4. A method according to claim 3 wherein said one and further starting powders further include starting powder particles and wherein individual starting powder particles are insulating particles.

5. A method according to claim 4 wherein said insulating particles have a dielectric constant greater than about 1.5.

6. A method according to claim 5 wherein said insulating particles are selected from the group consisting of titanium dioxide, zinc oxide, zirconia, alumina, silica, wax, polymer, inorganic oxide and organic particles and mixtures thereof.

7. A method according to claim 3 wherein said starting powder is a non-electrically conductive aerosol including individual electrically conductive particles.

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8. A method according to claim 7 wherein said electrically conductive particles are selected from the group consisting of carbon black and metallic particles and mixtures thereof.

9. A method according to any one of claims 1 to 8 wherein said starting powder has a starting powder particle size less than about 1cm.

10. A method according to any one of claims 1 to 9 wherein said step (2) of imparting an electrostatic charge to said starting powder further includes:

(a) providing a charge generator and a milling fluid; and

(b) flowing said milling fluid through said charge generator so that said milling fluid becomes electrostatically charged.

11. A method according to claim 10 wherein said milling fluid is selected from the group consisting of superheated steam, air, hydrogen, nitrogen, xenon and argon and mixtures thereof.

12. A method according to claim 10 or claim 11 wherein said charge generator is a high voltage source.

13. A method according to claim 12 wherein said high voltage source provides a DC voltage in the range of from about 10,000 volts to about 1,000,000 volts.

14. A method according to claim 13 wherein said high voltage source provides a DC voltage in the range of from about 10,000 volts to about 500,000 volts.

15. A method according to claim 14 wherein said high voltage source provides a DC voltage in the range of from about 10,000 volts to about 100,000 volts.

16. A method according to claim 12 wherein said high voltage source provides an AC voltage in the range of from about 10,000 volts rms to about 100,000 volts rms at a frequency of from about 1Hz to about 10,000 Hz.

17. A method according to any one of claims 1 to 9 wherein said step (2) of imparting an electrostatic charge to said starting powder further includes:

(a) providing a charge generator; and

(b) directly charging said starting powder.

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18. A method according to any one of claims 1 to 17 wherein said step (3) of milling said charged powder further includes providing a mill and a milling fluid to impart kinetic energy to said starting powder to result in comminution of said starting powder.

19. A method according to claim 18 wherein said mill is a jet mill.

20. A method according to any one of claims 1 to 19 wherein said step (3) of milling is performed using autogenous grinding.

21. A method according to claim 1 wherein said milled powder has a milled powder particle size in the range of from about 0.01µ to about 1000µ.

22. A method according to claim 21 wherein said milled powder has a milled powder particle size in the range of from about 0.01µ to about 5µ.

23. A method according to claim 22 wherein said milled powder has a milled powder particle size in the range of from about 0.05µ to about 0.5µ.

24. A method according to claim 2 wherein said further milled powder has a further milled powder particle size in the range of from about 0.01µ to about 1,000µ.

25. A method according to claim 24 wherein said further milled powder has a further milled powder particle size in the range of from about 0.01µ to about 5µ.

26. A method according to claim 25 wherein said further milled powder has a further milled powder particle size in the range of from about 0.05µ to about 0.5µ.

27. A method according to claim 2 wherein said step (8) of collecting said product powder further includes steps of providing a product powder collector selected from the group consisting of a cyclone, a bag filter and a settling chamber collector.

28. A method for producing a milled powder substantially as herein described with reference to any one of the examples.

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Abstract of the Disclosure

A method for producing a milled powder including imparting an electrostatic charge to the starting powder and milling the electrostatically charged starting powder is provided.