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oxidation of titanium tetrahalide is described in U.S. Pat. 3,214,284. This patent is incorporated herein by reference in toto to eliminate the need to substantially reproduce a detailed description of such oxidation reaction. The titanium dioxide produced by vapor phase reaction, sometimes called "chloride" pigment, does not generally contain residual soluble salts, such as found in sulfate pigment, and, therefore, does not require an additional washing step to eliminate such salts. In addition, the rutile crystal structure to "chloride" pigment is highly developed during the oxidation or hydrolysis step when rutilizing agents, such as aluminum or zirconium are present, and, therefore, rutilization by calcination is not a required operative step for such pigment.

Titanium dioxide pigment, as well as other pigments, that have not had their surface chemically modified, for example, the deposition of hydrous metal oxides or organic coatings thereon, are commonly referred to as "raw pigment." Examples of raw TiO_2 pigment include: calcined sulfate-based titanium dioxide pigment, calcined wet milled sulfate-based titanium dioxide pigment, chloride-based TiO_2 pigment, as received from the vapor phase reaction, and degassed chloride-based TiO_2 pigment. Such "raw" pigment can, and often is, subjected to a dry milling before further treatment, such as hydroclassification and coating with hydrous metal oxides. This initial dry-fluid energy milling pulverizes gritty or aggregative material existing in the titanium oxide product and provides pigment particles having a more even particle size distribution for coating procedures which typically follow. A hydroclassification typically follows such grinding step in order to eliminate large aggregates and over-sized particles before further treatment. Coating procedures are employed to optimize the essential pigmentary properties of the titanium dioxide pigment and typically involves coating the titanium oxide particles with hydrous oxides of titanium, aluminum, silicon, and other metals such as magnesium, zirconium, tin, zinc, and cerium. A typical coating procedure is described in U.S. Pat. 3,146,119. Such patent is incorporated herein by reference in toto, to eliminate the need to substantially reproduce a detailed description of such coating procedures.

In the case of titanium dioxide pigment, the amount of titania, silica, and alumina hydrates deposited on the surface of the pigment can vary respectively from 0.05 to 10 percent, 0.01 to 10 percent, and 0.05 to 15 percent. Other metal hydrates can be deposited in amounts of from about 0.01 to about 5 percent. The total amount of hydrous metal oxide coating placed on the pigment typically varies from about 2 to about 20 weight percent, based on the weight of the pigment.

One of the steps suggested for developing the pigmentary properties of, for example, uncoated titanium dioxide or hydrous metal oxide coated titanium dioxide particles, has been to dry-fluid energy mill the pigment with air or steam. Such fluid energy milling or dry grinding, as it is commonly known, typically reduces the oil absorption and improves the tinting strength of the pigment. Dry grinding of the coated pigment is usually performed by passing the pigment into a fluid energy mill employing super-heated steam or air as the gaseous fluid supplying the grinding energy. While dry grinding of the coated pigment in accordance with such procedures enhances certain pigmentary properties, it has been found that the development of other properties, such as ease of dispersion in paint vericles, is not fully accom-

fail to properly incorporate into the paint vehicle and can cause commercial rejection of an otherwise acceptable pigment. The percentage of coarse agglomerates causing such a dispersion problem is typically less than 0.1 percent of the pigment.

It has now been found that the dispersion of inorganic pigments, particularly metal oxide pigments and especially pigmentary titanium dioxide, can be improved by subjecting the pigment to a particular sequence of fluid energy milling. In partciular, it has been found that the dispersability of inorganic pigments, such as titanium dioxide pigment, and particularly hydrous metal oxide coated titanium dioxide pigment, in paint vehicles, is improved by subjecting such pigment to at least one fluid energy milling with a steam comprising gas followed by at least one fluid energy milling with a substantially liquid-free gas chemically inert to the pigment. While it is economically preferred that the two different fluid energy millings are conducted in sequence and without intervening process steps, they need not be performed in sequence. Intervening processing steps which alter the subdivision of the pigment should be avoided since such a step may cancel the benefits derived from the first fluid energy milling stages.

A fluid energy type mill is an apparatus in which pigment particles are conveyed by one or more streams produced by jets of milling fluid, such as air or steam, in such a manner to provide particle to particle collisions. For example, in a Micronizer, the jets of milling fluid are placed in a manner which will maintain an inwardly spiraling vortex at a high rotative speed and relatively small inward speed. As a result, the pigment particles conveyed by such milling fluid rub or strike against each other within the apparatus. The milling fluid supplying the grinding energy is withdrawn at an inward point tending to cause the fluid to travel spirally. Smaller particles are carried out with the gaseous fluid, and coarser particles thrown to the periphery where they are subjected to further reduction. Thus, the grinding chamber also serves as an internal classifier.

Examples of fluid energy type mills include: the Micronizer mill, which is described in U.S. Pats. 2,032,827 and 2,219,011, Trost jet mills, Jet-O-Mizers, Reductionizers, Jet Pulverizers, etc. A detailed description of fluid energy type mills including the micronizer, the reductionizer, and the Eagle mill appears in Industrial Engineering Chemistry, volume 38, page 672, et seq., (1946). Fluid energy or jet type mills are also described in Perry's Chemical Engineers Handbook, Third Edition, John H. Perry, Editor, pages 1145–47, McGraw-Hill Book Company, 1950, New York, and Chemical Processing, July, 1966, pages 50–64.

In accordance with the present invention, inorganic pigments such as pigmentary metal oxides, particularly titanium dioxide pigment, are subjected to at least one fluid energy milling with a gaseous fluid comprising steam followed by at least one fluid energy milling with a substantially liquid-free gaseous fluid chemically inert to the pigment. Milling procedures such as wet or dry milling, pulverizing, grinding, fluid energy milling, etc., can be performed prior to the aforementioned sequence of dry-fluid energy milling. Preferably, the pigmentary metal oxide after milling in accordance with this procedure has a moisture content of not more than about 0.5 weight percent, and preferably less than about 0.5 weight percent.

The gaseous fluid comprising steam employed as the energy supplying fluid in the first milling stage is usually