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pressed in terms of pounds of steam to pounds of pigment fed to the mill. Such intensity typically varies from about 0.25:1 to about 100:1, preferably between about 0.5:1 and about 10:1, and usually from about 1:1 to about 3:1. Economically, the lowest possible ratio of steam to pigment that will accomplish the desired milling in the shortest time is used. Pigment feed rates for production sized steam fluid energy mills can range from about 400 pounds per hour up to about 5000 pounds per hour. The exact feed rate will depend on the article size desired and the size capability of the equipment. Laboratory mills will, of course, have smaller capabilities. Steam feed rates will vary in accordance with the above-recited ratios and typically range from about 250 pounds per hour to about 8000 pounds per hour.

The substantially liquid-free gas employed as the energy supplying fluid for milling the pigment in the second stage after the initial milling with the steam comprising fluid is a gas that is chemically inert with respect to the pigment and is non-condensable. By non-condensable is meant that the boiling point of any liquid component of the gas is less than the lowest temperature to which the pigment is likely to encounter during processing and handling subsequent to and during milling. Typically, such temperatures will not be lower than 0° C. Preferably, the gas is substantially anhydrous. By substantially anhydrous is meant that the amount of water vapor present in the gas is such so that the milled pigment does not contain greater than 0.5 weight percent water. Suitable examples of substantially liquid-free, non-condensable gases that can be employed in the second stage milling include: nitrogen, air, carbon dioxide, the noble gases, i.e., helium, argon, krypton, neon, xenon, etc. Typically, unprocessed air from the atmosphere can be used. However, if the humidity of the air is excessive, it should be dried before being used.

The liquid-free, non-condensable gas, such as nitrogen or air, used in the second stage milling step is generally supplied at pressures of from about 50 to about 300 pounds per square inch gage, usually about 100 pounds per square inch gage, and at temeratures of from about 20° C. to about 70° C. or higher. Usually, room temperature is employed. The intensity of milling with the liquid-free gas, as expressed in pounds of non-condensable gas per pound of metal oxide pigment fed to the mill, is in the range of from about 0.25:1 to about 100:1, preferably between about 0.5:1 and about 10:1, and usually from about 1:1 to about 4:1. Here again, economic considerations will dictate the exact ratio of milling fluid to pigment feed. Pigment feed rates in the second stage of anhydrous gas milling will likewise vary depending on the particle size desired and the size of the unit. Feed rates of from about 50 pounds/hour to about 3500 pounds/hour are typical. Non-condensable gas feed rates vary in accordance with the above-recited ratios and typically vary from about 60 cubic feet per minute to about 1000 cubic feet per minute. Pressures for the milling fluid can vary from about 100 p.s.i.g. to about 1000 p.s.i.g. or higher, usually about 350 p.s.i.g.

Additives can be incorporated into pigment subjected to fluid energy milling in order to improve the milling of the pigment. Such additives can be incorporated in either or both milling stages. Particularly useful are organic materials, such as polyols, e.g., glycerol, pentaerythritol, and trimethylolpropane; fatty acids, e.g., oleic, and stearic acid; trialkanolamines, e.g., triethanolamine; etc. The amount of such additives incorporated during milling or added to the pigment before milling can vary

verizing. Dry-fluid energy milling is to be distinguished from wet milling wherein fluid in a liquid form as distinguished from a gaseous or vaporous form, is utilized with a particulate pigment.

The ease and degree of dispersion of titanium dioixde pigment can be measured by the Cowles dispersion test which is described in A.S.T.M. Test Method D–1210–54.

Another method for evaluating dispersion can be performed by the use of a conventional malted milk mixer (3M Test). In this test, and as performed in the subsequently described examples, a vehicle such as linseed oil and mineral spirits is placed in a metal canister affixed to the mixer and the test pigment, such as titanium dioxide, added thereto. The mixer is then operated at low speed for five minutes. A sample is removed and a Hegman fineness measurement made. The mixer is started again and samples taken at three more five-minute intervals so that Hegman fineness measurements are obtained at 5, 10, 15, and 20 minutes of mixing.

The tint efficiency of a pigment can be determined by the reflectometry method disclosed on pages 704 to 715, volume 34, of the Journal of Paint Technology and Engineering, (Official Digest), July 1962.

The fluid energy milling process of the present invention is applicable to the production of inorganic pigments. Particularly, this process is applicable to pigmentary metal oxides such as titanium dioxide (anatase or rutile), as well as other metal oxides, such as the oxides of aluminum, arsenic, iron, chromium, silicon, strontium, tin, zinc, zirconium, antimony, lead, and mercury. In addition, the present process is applicable to other inorganic pigments such as zinc sulfide, cadmium sulfide, iron sulfide, magnesium silicate, chromates, and carbon blacks. Moreover, the present invention is applicable to blends of inorganic pigments. Examples of typical blends or composites include: titanium dioxide-zinc oxide and titanium dioxide-silica. Blends of two or more pigments are also contemplated.

The pigments subjected to fluid energy milling by the above-described process can be utilized as pigments in the preparation of surface coating compositions in combination with conventional vehicle systems comprising a film former or binder and dispersion medium. Of particular utility for titanium dioxide is its use as a pigment in paints, paper, plastics, and printing inks.

The present process is more particularly described in the following examples which are intended as illustrative only since numerous modifications and variations therein will be apparent to those skilled in the art.

Example I

Titanium tetrachloride, in an amount of about 34 gram moles per minute, was oxidized in the vapor phase in a suitable reactor with about 45 gram moles per minute of oxygen. The vapor phase reaction was carried out in the presence of silicon tetrachloride and aluminum chloride in amounts that provided about 0.5 weight percent silica and about 1.8 weight percent alumina on the titanium dioxide produced.

The titanium dioxide thus produced was coated in a procedure similar to that described in U.S. Pat. 3,146,119 so as to provide a coating of about 0.7 weight percent hydrous titania (TiO_2) and about 1.2 weight percent hydrous alumina (Al_2O_3) on the pigment. The coated pigment was dried and portions of the dried pigment were fluid energy milled in a Fay 12-inch Micronizer utilizing steam, nitrogen, and combinations thereof as the milling fluid. The pigment feed rate to the micronizer was about