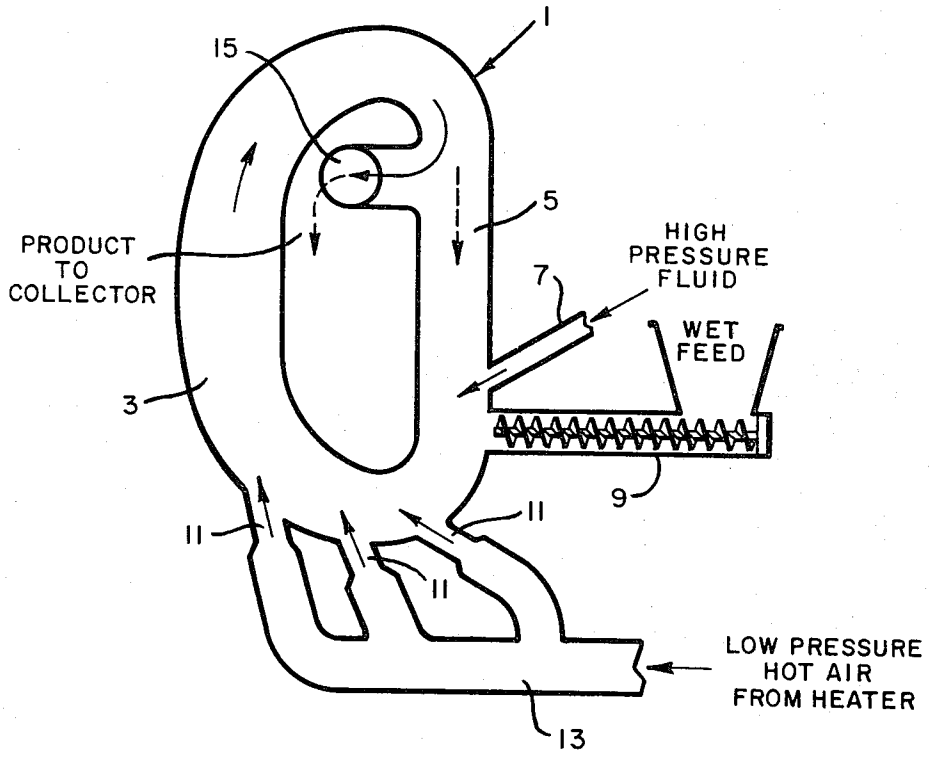
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LAMINAR GAMMA FERRIC OXIDE

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LAMINAR GAMMA FERRIC OXIDE

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ABSTRACT OF THE DISCLOSURE

Gamma ferric oxide having high acicularity is produced by subjecting a slurry of yellow iron oxide to a jet fluid energy mill which serves to break up the particles along their long axes, thus improving the acicularity of the particles. The particles can then be converted to gamma ferric oxide in the usual manner, and the acicularity carries over to the gamma form. Such particles of acicular gamma ferric oxide produce magnetic recording media of improved performance.

BACKGROUND OF THE INVENTION

Field of the invention

Acicular gamma ferric oxide.

Description of the prior art

Previously, expensive chemical conversion processes were necessary to produce yellow iron oxide, and ultimately gamma ferric oxide, of high acicularity. The present invention provides a simple mechanical method of converting the inexpensive yellow iron oxide of commerce to an acicular form.

SUMMARY OF THE INVENTION

It has been long recognized in the manufacture of magnetic recording media that the acicularity of the magnetic particles, such as gamma ferric oxide, is highly important both from the standpoint of output and noise, particularly at short wavelengths.

Gamma ferric oxide particles inherit their shape from the precursor alpha ferric oxide monohydrate and heretofore it has been necessary to employ expensive chemical conversion steps to secure alpha ferric oxide monohydrate having the desired acicularity. Alpha ferric oxide monohydrate is available commercially at low cost as yellow iron oxide, a pigment utilized in the paint industry.

The present invention provides an inexpensive, purely mechanical breaking up of the particles in such a manner that the acicularity of the particles is greatly improved. When the alpha ferric oxide monohydrate is then converted to gamma ferric oxide, the acicularity is retained and magnetic recording media of improved characteristics can be produced therefrom.

Accordingly, it is an object of the present invention to provide an inexpensive conversion means for improving the acicularity of the common yellow iron oxide of commerce.

Another object of the present invention is to produce gamma ferric oxide having a high acicularity ratio.

A further object of the invention is to provide a method of making magnetic recording media of improved output and signal to noise ratio, particularly at short wavelengths.

Other objects will be apparent from the balance of the specification.

BRIEF DESCRIPTION OF THE DRAWING

The sole figure is a side view of an apparatus for carrying out the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

In carrying out the present invention a fluid energy mill is employed. Such mills are described in the text "Unit Operations of Chemical Engineering" (McGraw-Hill, 1956), page 233. A typical mill is shown in the drawing. Such a mill comprises a torus **1**, arranged in a generally vertical configuration having an ascending section **3** and a descending section **5**. An opening **7** is provided for the introduction of a high pressure fluid tangentially to the flow of material within the torus while a screw feeder **9** is provided immediately below the opening **7** for the introduction of the wet feed. It is clear from the drawing that as the material in wet form is introduced into the mill, the jet of high pressure fluid from the opening **7** impinges upon it. Near the bottom of the mill there are a plurality of relatively large openings **11** leading to a source of low pressure hot air **13**. Near the top of the descending column **5** is provided an outlet **15**. Exhaust air and product are taken from this outlet **15**. Because of the centrifugal action of the circulating fluid with the torus, relatively large particles will be recirculated for further fracture while relatively small particles will be removed through the opening **15**. Eventually, of course, large particles will be fractured by the attrition produced within the mil and will eventually be removed as small particles through the opening **15**.

In operation, a slurry is pumped into the drier and is dispersed and deagglomerated by the stream of hot pressure fluid which can be either superheated steam or compressed air. It is believed that the laminar fracture of the yellow oxide particles occurs at this moment. The dispersed particles are instantly dried by a current of hot air fed through the openings **11**. It is obvious that in order to dry the slurry, an adequate volume of hot gas at a reasonably high temperature should be employed. Low mill temperatures require a large volume of air and high mill temperatures make for a poor utilization of heat. However, the volume and temperature should be adjusted so that drying is substantially instantaneous and that no wet particles move into the ascending column **3**.

It has been found that the mill pressure is an important factor. High mill pressure will bring about a violent agitation of the particles within the grinding chamber wherein the particles may break along their short axes to produce particles of poor acicularity. On the other hand, a positive pressure must be maintained within the mill to assure adequate circulation and product removal. Thus the pressure within the mill must be maintained within the range of 0.1 to 10 p.s.i.g. and preferably from 0.5 to 2 p.s.i.g.

Similarly, since the laminar breakage occurs during the dispersion of the slurry with the high pressure fluid, it is obviously necessary to maintain a minimum pressure in line **7** to cause a proper fracture. In practice it has been found that this pressure must be at least 50 p.s.i.g.

It has been found essential for conducting the process of the present invention to feed the material into the mill in the form of a slurry. When it was attempted to repeat the process of the examples with a dry powder, no increase in acicularity could be found. At low pressures with dry feed there is no change in particle size and as the pressure goes up, the particles break along their short axes, defeating the purposes of the present invention.

In each of the following examples, the starting material was ordinary yellow iron oxide of commerce as is used by the paint industry. This is alpha ferric oxide monohydrate and the particles had an average acicularity, i.e., the ratio of the length to breadth, of 4.7 to 1. The particles had an average Stokes' equivalent diameter of 0.55 micron.

EXAMPLE I

Seven hundred pounds of the yellow oxide described above was added to thirteen hundred pounds of water and the mixture was homogenized in a mixer to obtain a smooth slurry. The slurry was fed into an 8 inch diameter jet fluid energy mill, manufactured by the Fluid Energy Processing and Equipment Company at a rate of 600 pounds per hour. High pressure steam at 150 p.s.i.g. fed at a rate of 850 pounds per hour was used to atomize the slurry. Action of the mill was such that laminar fractures were produced in the particles, i.e., the particles were broken on their long axes to produce long slender particles. The slurry was dried in a current of hot air at 590° F. at thirteen hundred s.c.f.m. while maintaining the temperature of the mill between 240° and 280° F. The mill pressure was maintained below 2 p.s.i.g. The dried product had an average Stokes' equivalent diameter of 0.41 micron and an acicularity ratio of 6. The product was then dehydrated to alpha ferric oxide at 350° C., reduced to magnetite at 350° C. in an atmosphere of hydrogen; it was then oxidized to gamma ferric oxide in air at a temperature of about 250° C.

A formulation was then made up of the following (all parts are by weight):

	Parts
Gamma ferric oxide	1251
Carbon black	114
Dihydrogen phosphate	15
Lecithin	55
Melamine-formaldehyde resin	25
Silicone oil	140
Phenyl mercuric oleate	9
Methylisobutyl ketone	217
Methyl ethyl ketone	302
Toluene	201

This mixture was ground in a pebble mill for 72 hours and the following was added:

	Parts
Polyurethane resin	190
Polyvinylidiene chloride resin	169
Methylisobutyl ketone	244
Methyl ethyl ketone	1194

Grinding was continued for another 24 hours to produce a well dispersed slurry.

The slurry was coated on a 1 mil thickness polyester film to produce a dried coating having a thickness of 0.4 mil. The particles were oriented 30° to the longitudinal direction with a thousand gauss magnet. The finished film was tested as a magnetic tape as is hereinafter described.

EXAMPLE II

A second sample of the yellow oxide was treated as is described in Example I except that the steam pressure was lowered to 100 p.s.i.g. The resulting yellow oxide had an average Stokes' equivalent diameter of 0.45 micron and an average length to breadth ratio of 5:7. This material was converted to gamma ferric oxide and made into a magnetic tape as is described in Example I.

EXAMPLE III

A sample of the original yellow oxide previously described was directly converted to gamma ferric oxide and made into a magnetic tape as described in Example I.

Three tapes were tested in an Ampex VR–2000 video recorder at 15 inches per second. The following results were obtained:

	Output, db			Noise, db		
Frequency	Untreated oxide tape	Treated oxide tape No. 1	Treated oxide tape No. 2	Untreated oxide tape	Treated oxide tape No. 1	Treated oxide tape No. 2
4 mHz	−6.7	−6.5	−6.7	−36.0	−37.0	−37.0
5	−7.0	−6.8	−7.0	−39.7	−40.2	−40.2
6	−7.8	−7.5	−8.0	−43.7	−44.0	−43.9
7	−9.0	−8.7	−8.8	−46.3	−46.5	−46.3
8	−9.0	−9.5	−9.7	−46.5	−46.9	−46.7
9	−10.6	−10.4	−10.5	−45.6	−45.8	−45.9
10	−11.8	−11.3	−11.5	−43.5	−43.7	−44.0
11	−13.3	−12.8	−12.9	−40.7	−41.1	−41.3
12	−16.2	−15.6	−15.8	−37.5	−38.2	−38.6

The jet mill produces an unusual type of grinding action. A high pressure fluid is fed into the grinding chamber tangentially, causing violent agitation of a slurry of particles wtihin the grinding chamber and causing their immediate fracture. This produces the unique laminar fracture desired resulting in the increase in acicularity. When yellow oxide was ground in an ordinary attrition mill, no increase in acicularity was found.

I claim:

1. The process of making acicular alpha ferric oxide monohydrate comprising forming an aqueous slurry of alpha ferric oxide monohydrate and subjecting the slurry to the action of a fluid energy mill operated at an internal pressure of from 0.1 to 10.0 p.s.i.g. and at a gas feed pressure of at least 50 p.s.i.g. and drying the particles within said mill to recover alpha ferric oxide monohydrate having improved acicularity.

2. The process of claim 1 wherein the mill is operated at a pressure of from 0.5 to 2.0 p.s.i.g.

3. The process of claim 1 wherein the resulting alpha ferric oxide monohydrate is dehydrated to alpha ferric oxide, reduced to magnetite by heating the same with a reducing gas and then oxidized to gamma ferric oxide wherein the gamma ferric oxide preserves the increased acicularity of the treated alpha ferric oxide monohydrate.

References Cited

UNITED STATES PATENTS

2,954,303 9/1960 Westcott ..... 23—200 X

OTHER REFERENCES

Perry: Chemical Engineers' Handbook, 3rd ed., McGraw-Hill Book Co., Inc., 1950, p. 1145.

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