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METHOD OF PREPARING FERROMAGNETIC POWDER

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This invention relates to a method of preparing ferromagnetic powder. More particularly, this invention relates to the preparation of a ferromagnetic powder having a permanently adherent coating or film of black iron oxide, FeO.

The ferromagnetic powder made by my present invention has been found particularly adapted for use in the magnetic particle inspection method of testing metal articles for surface discontinuities or other defects. Conventional ferromagnetic powders used for inspection by the magnetic particle method comprise finely divided iron coated with a pigment of a color contrasting with that of the surface of the metal article to be tested. However, the pigment particles attached to the particles of iron powder tend to rub off and to form a fine dust, which obscures the testing results and makes the testing procedure more difficult and unpleasant to the operator.

I have now found that iron or steel powder may be colored jet black and rendered water repellent and rust resistant by treating the iron or steel powder at a suitable elevated temperature with an oxidizable fatty oil, such as a drying or semi-drying oil. While all of such fatty oils are operative to produce the black surface film, or coating, desired, the temperatures required for forming a jet black surface of FeO vary with the particular oil used, as will hereinafter be more specifically pointed out. By the method of my invention, a black surface film is formed that does not tend to rub off or to dust, and that is both water repellent and rust resistant.

It is therefore an important object of the present invention to produce an improved ferromagnetic powder for use in the magnetic particle inspection method and characterized by a deep black color and by the absence of any tendency of the surface to rub off or dust.

Another important object of this invention is to provide a method of preparing a ferromagnetic powder having a permanently adherent film thereover of black FeO, which method includes the treating of iron or steel powders with an oxidizable oil at elevated temperatures, the optimum temperature being selected for the particular oil used to give a jet black color to the powder as the result of the treatment.

A further important object of this invention is to produce a ferromagnetic testing powder having a coating or film of black FeO thereover, which is water repellent and rust resistant and which will not rub off or dust.

Other and further important objects of this invention will become apparent from the following description and appended claims.

In the preparation of ferromagnetic powder embodying the principles of my present invention, iron or steel powder is used as the ferromagnetic material. If a steel powder is used, a low carbon

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steel is employed. In general, the powder used should be of such particle size as to pass through a 60-mesh screen and may suitably be of a fineness such that 50% of the powder will pass through a 325-mesh screen. The starting powder is mixed with a minor proportion of an oxidizable oil selected from the group consisting of drying and semi-drying oils. The proportion of oil used will vary depending upon the degree of fineness of the ferromagnetic powder, but will be within the range of from one-half to 3%, by weight, more oil being necessary with the finer particle size of powder. The preferred range is between 1 and 2½% of oil by weight of the ferromagnetic powder.

The readily oxidizable oils that are operative for the purposes of the present invention include the drying oils and the semi-drying oils, in particular linseed oil, corn oil, soya bean oil, fish oil, oiticica oil, perilla oil, tung oil, cotton-seed oil, and the like. Enough oil is used so that all of the oil will not be volatilized during the early stages of the heat treatment. If less than 2% of oil by weight of the ferromagnetic powder is employed, the heat treatment should be carried out slowly and carefully so as not to volatilize all of the oil before the heat treatment is completed.

The oil and ferromagnetic powder are first mixed, for instance, in a tumbling barrel, until the oil is distributed as a uniform film over the powder particles. The resulting mixture may then be charged into an inclined tube, heated by suitable means to bring the mixture to the desired temperature. Air, or other oxygen furnishing gas, is supplied to the reaction mass, since without oxygen the reaction does not occur as desired.

The mass is heated at a moderate rate until reaction occurs, as indicated by a color change and by fuming of the oil. For the different oils, the temperature will vary between 475 and 675° F., but within that range there will be an optimum temperature for each particular oil used. The following table indicates the optimum temperatures for various oils of the drying and semi-drying types:

Oil	Temp.
	° F.
linseed.....	550
corn oil.....	483
soya bean oil.....	610
Oiticica oil.....	527
China-wood oil.....	500
perilla oil.....	545
fish oil.....	590
castor oil.....	662

With an iron powder of such particle size as to pass completely through a 60-mesh screen with 50% retention on a 325-mesh screen, the pre-

ferred portion of the various oils by weight of the iron powder, using the above specified temperatures, was between $1\frac{1}{2}$ and 2%. In each case, the temperature was maintained at the optimum point, as indicated, for a sufficient length of time to form a jet black film over the iron particles. The time required for the completion of the reaction is of the order of 20 seconds, but longer heating is not deleterious provided the temperature is maintained within the limits specified. Care should be taken that the temperature is not raised too rapidly in the initial heating stage, since otherwise the oxidizable oil is likely to be driven off before the reaction is complete.

The coating, or film, that forms on the ferromagnetic powder as a result of the above described treatment has been identified as black FeO. In the case of a coarse powder subjected to the treatment described, the colored film on the surface of the ferromagnetic material can be dissolved away in hydrochloric acid and the dissolving action stopped before dissolution of the iron or steel base is dissolved. Such treatment with hydrochloric acid demonstrates the absence of any elemental carbon in the coating, or film, and confirms my observation that the black coloration is due to FeO, itself.

The treatment above described does not affect the magnetic properties of the ferromagnetic powder, but leaves on the powder particles a jet black film of FeO which does not tend to rub off and which does not dust. Ferromagnetic powder thus prepared is practically rustproof, for it will show no color change after forty-eight hours' exposure to water vapor. Although apparently the reaction oil is completely driven off during the preparation of the black ferromagnetic powder of my invention, a water repellent film remains on the powder, for it has been found extremely difficult to wet the finished powder with water. The finished powder may, however, be readily dispersed in a light mineral oil, of the kerosene type, and is thus suitable for use in testing by the magnetic particle inspection method wherein a suspension or dispersion of the ferromagnetic particles in a light oil is flooded onto the surface of the metal article to be tested while the latter is in a magnetized condition, or is subjected to magnetic lines of force.

Many details of procedure may be varied within a wide range without departing from the principles of this invention and it is therefore not my purpose to limit the patent gained from this invention otherwise than necessitated by the scope of the appended claims.

I claim as my invention:

1. The method of preparing a ferromagnetic powder having a permanently adherent film thereover of black FeO, which comprises mixing a ferromagnetic powder with from $\frac{1}{2}$ to 3% by weight of a material selected from the group consisting of drying and semi-drying oils and heating the resulting mixture in the presence of air to that temperature within the range of 475° to 675° F. at which for the particular material selected reaction occurs to produce a film of black FeO on the surfaces of said powder, the heating being carried out at such a rate as to insure that the oil selected is not driven off before the reaction is complete.

2. The method of preparing a ferromagnetic powder having a permanently adherent film thereover of black FeO, which comprises mixing an iron powder with from 1 to $2\frac{1}{2}$ % by weight of a material selected from the group consisting of

drying and semi-drying oils, heating the resulting mixture in the presence of air to that temperature within the range of from 475° to 675° F. at which for the particular material selected reaction occurs to produce a film of black FeO on the surfaces of said powder, and maintaining such temperature until the reaction is complete, the heating being carried out at such a rate as to insure that the oil selected is not driven off before the reaction is complete.

3. The method of preparing a ferromagnetic powder of jet black color, which comprises mixing iron powder with from 1 to $2\frac{1}{2}$ % by weight of linseed oil, heating the resulting mixture in the presence of air to a temperature of about 550° F. until said powder acquires a jet black color and cooling said powder, the heating being carried out at such rate as to insure that the oil selected is not driven off before the reaction is complete.

4. The method of preparing a ferromagnetic powder of jet black color, which comprises mixing iron powder with from 1 to $2\frac{1}{2}$ % by weight of corn oil, heating the resulting mixture in the presence of air to a temperature of about 483° F. until said powder acquires a jet black color and cooling said powder, the heating being carried out at such a rate as to insure that the oil selected is not driven off before the reaction is complete.

5. The method of preparing a ferromagnetic powder of jet black color, which comprises mixing iron powder with from 1 to $2\frac{1}{2}$ % by weight of soya bean oil, heating the resulting mixture in the presence of air to a temperature of about 610° F. until said powder acquires a jet black color and cooling said powder, the heating being carried out at such a rate as to insure that the oil selected is not driven off before the reaction is complete.

6. The method of preparing a ferromagnetic powder having a permanently adherent film thereover of black FeO, which comprises mixing an iron powder, of a size such that all of the powder passes through a 60-mesh screen with from 1 to $2\frac{1}{2}$ % by weight of a material selected from the group consisting of drying and semi-drying oils, heating the resulting mixture in the presence of air to that temperature within the range of from 475° to 675° F. at which for the particular material selected reaction occurs to produce a film of black FeO on the surfaces of said powder and maintaining said temperature for a period of time sufficient to produce a jet black film on said surfaces that is water repellent, the heating being carried out at such a rate as to insure that the oil selected is not driven off before the reaction is complete.

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