

UNITED STATES PATENT OFFICE

2,687,349

PRODUCTION OF IRON POWDER

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No Drawing. Application February 20, 1951,
Serial No. 211,978

Claims priority, application Germany
February 24, 1950

1 Claim. (Cl. 75-0.5)

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This invention relates to improvements in the manufacture of iron powder having valuable magnetic properties.

In the production of mass cores, for example such cores as are used for pupin coils, special stress is laid on a high permeability (μ) and a low hysteresis-loss (h) of the iron powder employed for the production of said cores. The value h :
 $\sqrt{\mu}$ should therefore be as low as possible.

We have found that iron powder which has been obtained by thermal decomposition of iron carbonyl in the free space of a heated vessel, preferably in the presence of ammonia, can be considerably improved in the above respect by subjecting the iron powder to a grinding and then at least partly freeing it from its carbon and oxygen content. This is preferably effected by treating the powder with hydrogen or hydrogen containing gases while heating, preferably at about 400° C.

It has been found that the pretreatment of the iron powder by grinding exerts a surprising improving influence on the hysteresis-loss of the powder. The preliminary grinding also has the advantage that the subsequent grinding of the powder which is usually carried out after the heat treatment, is facilitated. The subsequent grinding can be considerably shortened due to the preliminary grinding so that the calcined powder particles, which are considerably softer than the uncalcined particles, are deformed less and retain their original globular form in a higher degree. Moreover, the process renders it possible to employ such carbonyl iron powder as, for example, by reason of defects in the iron carbonyl decomposition, such as too high or too low a temperature, contains particles the size of which is unsuitable for magnetic purposes. Thus in the carbonyl decomposition spongy or tuftlike structures are readily formed which in the hitherto usual treatment with hydrogen following immediately after the decomposition had an unfavorable influence on the magnetic properties, in particular the hysteresis-loss. Such non-uniformly composed carbonyl iron powders could hitherto usually not be employed for the production of highly permeable types of iron powder by treatment with hydrogen, so that allowance had to be made for considerable losses in yield. All these difficulties are considerably reduced by the preliminary grinding of the raw carbonyl iron powder.

It is preferable to use a ball mill, but other comminution devices, such as disintegrators and the like, are also suitable. The duration of grinding depends on the nature of the raw carbonyl

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iron powder, in particular on the size and number of the coarser particles grown to form agglomerates.

The powder prepared by grinding yields by a conventional treatment with hydrogen at elevated temperature, whereby the powder is substantially decarbonized and deoxidized, a product with high permeability. When stress is laid on magnetic stability at the same time, it is preferable to treat the powder with nitrogen as well as with hydrogen.

The following example will further illustrate this invention but the invention is not restricted to this example.

Example

Iron powder, which has been obtained by heating iron carbonyl vapor with an addition of ammonia in a free space, is treated for 30 hours at 400° C. with flowing hydrogen and then ground for 24 hours in a ball mill. The value h : $\sqrt{\mu}$ of this powder is 5.9. If, on the contrary, the iron powder is ground for 10 hours in a ball mill before the treatment with hydrogen, and then further worked up as above described, the value h : $\sqrt{\mu}$ is 4.7. If the preliminary grinding is for 30 hours instead of 10 hours, the value h : $\sqrt{\mu}$ falls even to 3.8. This powder is therefore especially well suited for example for the production of pupin coils.

What we claim is:

A process for the production of iron powder having low oxygen and carbon content and especially suitable for use in mass cores which comprises ball milling for 10 to 30 hours an iron powder obtained by the thermal decomposition of iron carbonyl in the free space of a heated vessel, heating said ground powder with hydrogen at a temperature of about 400° C. to remove oxygen and carbon, and subsequently grinding said heated powder to small particle size.

References Cited in the file of this patent

"Powder Metallurgy" by Schwarzkopf, published by the MacMillan Co., 1947, page 325.

"Symposium on Powder Metallurgy," second ed., published by the Iron and Steel Institute, London, December 1947, page 49.

Buckley, S. E., et al., "Carbonyl Nickel and Carbonyl Iron Powders, Their Production and Properties," B. I. O. S. Final Report No. 1575 (Interrogation Report No. 590) Item No. 21. Pages 20-29, specifically page 21, page 28, pages 34 and 35.

Heyer, "Engineering Physical Metallurgy," page 291, D. Van Nostrand Co., Inc., New York N. Y.