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[54] **PROCESS FOR PRODUCING IRON POWDER FROM HEMATITE ORES**
8 Claims, No Drawings

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[50] Field of Search 75/0.5, 1

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ABSTRACT: A process for producing iron powder in which a hematite ore concentrate is ground and passed through a high-intensity wet magnetic separator to remove silica and other acid insoluble impurities, after which the purified hematite is reduced in the presence of hydrogen to form iron powder.

PROCESS FOR PRODUCING IRON POWDER FROM HEMATITE ORES

BACKGROUND OF THE INVENTION

1. Field of the Invention

The invention relates to a process for producing iron powder.

2. Description of the Prior Art

There has been a rapidly expanding industrial interest in iron powder, particularly for use in powder metallurgy. Powdered metal parts have the advantages of requiring no high-temperature melting furnaces and little or no machining. Moreover, powder formulations can be closely controlled to meet specific needs. One of the newest uses of iron powder is in the molding of gears for automobile transmissions.

For best results in powder-metallurgy processes, the particular size distribution, uniformity and apparent density of the powder must be appropriately controlled within well defined limits. Accordingly, powders produced by the known methods are usually pulverized, screened and blended to assure that they have the desired properties before use. For example, an iron powder to be acceptable for most purposes must conform to the following requirements:

1. It must have an apparent specific gravity between 2.2 and 2.5

2. It must have a flow rate of 40 seconds or less, as determined by the time required for 50 grams to pass through a Hall Flowmeter.

3. The material should be less than 100 mesh in particle size, and a maximum of 60 percent may be less than 325 mesh. Within these limits, the particle size distribution or gradient may vary somewhat, but should be approximately of the order of one-third greater than 200 mesh, one-third greater than 325 mesh, and one third less than 325 mesh.

It is known to produce iron powder by direct reduction of iron oxide and typical sources of this iron oxide include chemically produced oxide, mill scale and natural ore concentrates. The chemically produced oxide is of very high purity but is prohibitively expensive for pressing powders. Mill scale has the obvious disadvantage of only being available in very limited quantities and subject to considerable variations in grade. In addition, powder produced from mill scale contains undesirable alloy elements such as manganese, which were in the steel. Moreover, mill scale must be collected from a large number of sources, which cannot be uniform, and dirt and contamination are picked up during collection in the mill and shipment. This means that extensive cleaning and blending is required before it may be used and, on occasion, as much as 30 percent must be discarded as being unsuitable.

Natural ore concentrates are available in unlimited quantities but these contain as much as 6 percent silica. Magnetites may be processed using conventional magnetic techniques but acceptable separation of the silica from most magnetite ores is almost impossible and few are suitable. Canadian Pat. No. 519,725, issued Dec. 20, 1955 describes a process for producing iron powder from magnetite ore in which the silica is removed by chemical treatment with hydrofluoric acid.

Another process for producing iron powder from magnetite is described in Canadian Pat. No. 527,302, issued July 3, 1956. This process employed magnetic separation to purify the magnetite ore but in order to reduce the silica content to below 0.2 percent by magnetic separation it was necessary to grind the ore such that over 90 percent was −325 mesh. Since this produced particles too small for iron powder it was necessary to sinter and pregrind the purified material to produce particles which after reduction would meet the requirements of iron powder.

Moreover, it is suggested in Canadian Pat. No. 496,781, issued Oct. 13, 1953 that ores such as hematite should first be converted into magnetites and then concentrated by wet magnetic separation.

Iron powder can also be produced by processes other than direct reduction. One such process involves atomizing molten low-carbon steel by spraying. The steel becomes partly oxidized during spraying but depending on the carbon content, more oxide, usually mill scale, is mixed with it. This mixture is then reduced in a furnace, at which time oxygen combines to remove the carbon. This is obviously a relatively complex and expensive process but again the main problem is the source of raw materials. Only scrap is sufficiently cheap to be considered and not all scrap is suitable. Here again the raw material supply is governed independently of the demand for powder. In addition, the powder contains the undesirable alloying elements present in the steel.

Some iron powder is also produced electrolytically but sells at three times the price of reduced powders so that it can obviously be considered only for very special applications.

Thus, it will be seen that up until now iron powder has not been available in unlimited supplies at a low cost because of the short supply of the raw material from which it could be produced and/or because of the cost of production.

SUMMARY OF INVENTION

According to our invention, an iron powder meeting the above requirements can be simply and inexpensively obtained by passing a finely divided natural hematite ore through a high-intensity wet magnetic separator to reduce silica and other acid insoluble impurities therein to preferably less than 0.2 percent and treating the purified hematite obtained with a reducing agent at reducing temperatures below the melting point of iron to produce a substantially pure iron product. The iron product is obtained in the form of an agglomerate of lightly adhering particles which is easily broken down into discrete particles.

The use of hematite for the production of iron powder has the great advantages of low cost and unlimited availability. As stated above, it is already known to purify magnetite by magnetic separation but it has been found to be very difficult to obtain a clean separation of magnetite during magnetic separation. Thus, since hematite is considered to be a non-magnetic material, it could not have been expected that it could be purified magnetically and it is particularly surprising that hematite can be purified in this manner, to the very high degree necessary to permit direct reduction to substantially pure iron. It has also surprisingly been found that the hematite can be initially ground to a particle size range such that after magnetic separation and reduction, iron powder is obtained without further grinding or classifying which is within the commercially acceptable particle size distribution.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The reduction can be conducted by means of any of the usual reducing agents, such as solid carbon, gaseous reducing agents such as hydrogen or a mixture of both the solid and gaseous reducing agents. A wide range of temperatures are possible during reduction with higher temperatures providing a faster reaction rate. Temperatures between 1,600° F. and the melting point of iron are preferred and a temperature between 2,000° –2,200° F. has been found to be particularly desirable.

The reduction can be conducted in any suitable furnace and as indirect gas fired belt furnace has been found to be very satisfactory. According to a preferred procedure both solid carbon and hydrogen gas are used during reduction with the carbon being deposited in finely divided form on the belt under the purified hematite particles. It has also been found to be advantageous to conduct the reduction in a two state operation.

The iron product obtained from the reduction furnace was found to be in the form of an agglomerate of loosely adhering particles. This agglomerate was easily broken down into its component particles.

The particles of iron powder obtained from the reduction stage are advantageously subjected to densification to adjust density requirements. This can be conveniently done by im-

pact densification, e.g. in a hammer mill. It has also been found that the agglomerate obtained from the reduction furnace can be fed directly to the hammer mill where the agglomerate is broken down to particle size and the particles are densified in a single operation. When a two stage reduction is used, densification is carried out after each stage.

The iron powder obtained by the preferred procedure of this invention, which includes the steps of forming the agglomerate of loosely adhering particles and impact densification, has the very important advantage that the particles have sharp edges. Thus, sharp-edged particles not only produce a superior sintered product but also provide better green strength. The green strength is a measure of the strength of a compact of iron powder before sintering.

The iron powder obtained according to the invention also has a very low silica content, which results in the very desirable property of the powder being soft. A soft powder is highly desirable because it facilitates removal of a molded part from a mold and also decreases the amount of wear on the mold face.

The invention will now be illustrated by the following examples:

EXAMPLE I

As starting material a commercial specular hematite ore concentrate was used containing 66.2 percent iron, 5.1 percent silica and 0.61 percent alumina. A screen analysis of this ore concentrate is given below in table I:

TABLE I

Mesh	% by weight
+6	
−6+10	3.7
−10+20	23.2
−2+35	38.1
−35+65	22.8
−65+100	5.6
−100+150	4.2
−150+200	1.3
−200+325	0.6
−325	0.1

If necessary, the ore concentrate can be ground to a desired particle size in a ball mill or rod mill.

The ore concentrates was passed through a Jones high-intensity wet magnetic separator to obtain an iron oxide powder containing less than 0.2 percent by weight of acid insolubles. A screen analysis of the purified iron oxide powder is given in table II below:

TABLE II

Mesh	% by weight
+65	
−65+80	16.1
−80+100	9.5
−100+150	19.4
−150+200	16.0
−200+270	15.4
−270+325	6.6
−325	17.0

The purified iron oxide was subjected to hydrogen reduction in two stage operation. The reduction was carried out in a belt furnace with indirect gas heating. For the reduction approximately two times the theoretically required amount of hydrogen is used i.e. about 25,000 cubic feet per ton of ore.

The purified iron oxide was contacted with the hydrogen at a furnace temperature of 2,220° F. and remained in contact with the hydrogen for approximately 4 hours. The partly reduced powder emerging from the furnace was then subjected to densification in a hammer mill and the densified, partially reduced product had the screen analysis given in table III below:

TABLE III

Mesh	% by weight
+65	
−65+80	
−80+100	2.1
−100+150	23.2
−150+200	22.7
−200+270	22.6
−270+325	8.4
−325	21.0

The partly reduced product contained 0.4 percent by weight of acid insolubles. The hydrogen loss on the product was 4 percent (which indicated that 4 percent of oxygen yet remained). The flow rating of the product was 41 seconds and its apparent density was 2.1 gram per cc.

The partially reduced product was then passed through the furnace a second time in contact with hydrogen at a furnace temperature of 2,100° F. for a period of 4 hours.

A screen analysis of the final product emerging from the furnace is given in table IV below:

TABLE IV

Mesh	% by weight
+65	
−65+80	
−80+100	0.6
−100+150	22.9
−150+200	26.6
−200+270	25.1
−270+325	8.6
−325	16.2

The final product contained approximately 0.62 percent by weight of acid insolubles and showed a hydrogen loss of 0.15 percent. Its flow rating was 35 seconds and its apparent density 2.2 grams per cc.

The iron powder produced in accordance with this invention was subjected to a series of tests by an independent testing organization. In these the standard used for comparison was an iron powder available from Hoeganaes Sponge Iron Corporation under the trademark Hoeganaes Ancor MH100, which is generally accepted in the industry as the standard.

Compaction tests were conducted on the two samples with each being compacted at 30 and 50 tons per square inch with 0.75 percent stearate lubricant. The samples were sintered in dissociated ammonia at 2,050° F. for 45 minutes.

The results of physical tests on these samples were as follows:

TABLE V

	Product of Invention		Hoeganaes	
Compacting Pressure, tsi	30	50	30	50
Green Density, g./cc.	6.43	6.86	6.46	6.95
Green Strength, p.s.i.	900	2200	1820	3800
Sintered Density, g./cc.	6.40	6.82	6.43	6.93
Change in Length on Sintering	% −0.33	−0.23	−0.13	−0.15
Tensile Strength,	24,500	30,300	19,500	25,500

p.s.i.				
Elongation, % in 1 inch	9.7	14.1	7.1	9.8
Rockwell Hardness	R_H74	R_H85	R_H76	R_H88

It will be seen from the above data that the product of this invention compared extremely favorably with the accepted industrial standard, and, in fact, is considerably superior in two of the most important strength characteristics i.e. tensile strength and elongation.

Although the green strength in the above data for the product of the invention does not compare with the industrial standard, this was not found to represent a difficult problem and minor adjustments have succeeded in substantially raising this value.

EXAMPLE 2

A sample or iron powder was prepared using the same process as Example 1 with the following properties.

Apparent Density, g./cc.	2.41
Flow, sec.	25.0
Screen analysis %	
+100 mesh	1.4
−100+150	14.8
−150+200	25.9
−200+270	15.5
−270+325	17.6
−325	24.8
Average Particle Size, microns	23.5
Weight Loss in Hydrogen, % (1,150° C.—1 hour)	0.18

Comparative physical tests were conducted on the above sample and a corresponding sample of Hoeganaes Ancor MH–100. The samples were each compacted at 30 tons per square inch with 0.75 percent zinc stearate lubricant and were sintered in dissociated ammonia at 2,050° F. for 45 minutes. The results of tests on the sintered samples are as follows:

	Product of Invention	Hoeganaes Ancor MH–100
Green Density, g./cc.	6.13	6.26
Green Strength, p.s.i.	2150	1850
Sintered Density, g./cc.	6.12	6.25
Change in Length on Sintering, %	−0.18	−0.13
Tensile Strength, p.s.i.	18,800	16,800
Elongation, % in 1 inch	10.0	8.2
Rockwell Hardness	R_H65	R_H66

This test shows an improvement over the standard iron powder not only in tensile strength and elongation but also in green strength.

EXAMPLE 3

The softness or lack of abrasiveness of iron powder is an important characteristic since it affects the ease with which a molded part can be removed from a mold and also the amount of wear on the mold face. Accordingly, comparative tests were conducted comparing the ejection pressures for iron compacts made with the powder of this invention and Hoeganaes Ancor MH–100 iron powder.

The powders were compacted in 1 inch cylindrical dies with a compacting pressure of 50 tons per square inch. The results were as follows:

Weight, gm.	Diam., in.	Length, in.	Density, g./cc.	Ejection load pressure, Lbs.	Ejection load pressure, P.s.i.	Wall contact pressure, p.s.i.
Iron powder of invention plus ¾% zinc stearate						
90.85	1.005	1.008	6.93	8,225	10,360	2,580
90.75	1.005	1.006	6.92	9,000	11,330	2,830
90.41	1.005	1.003	6.92	12,000	15,110	3,790
					[1] 12,300	[1] 3,070
Hoeganaes Ancor MH–100 plus ¾% zinc stearate						
90.78	1.005	0.991	7.04	13,500	17,000	4,260
91.43	1.005	1.000	7.02	14,500	18,760	4,590
91.48	1.005	0.999	7.04	18,000	22,670	5,690
					[1] 19,480	[1] 4,850

[1] Average.

From the above results it will be seen that the iron powder of this invention has appreciably lower ejection and wall contact pressures than the standard iron powder, pressed under the same conditions. This means that the iron powder of the invention ejects more readily and less friction heat is created and this represents a major improvement in iron powder.

What we claim as our invention is:

1. A process for producing iron powder which comprises passing a finely divided natural hematite ore through a high intensity wet magnetic separator to remove silica and other acid insoluble impurities, treating the purified finely divided hematite with a reducing agent at reducing temperatures below the melting point of iron and collecting the substantially pure iron produce obtained.

2. A process according to claim 1 wherein the purified hematite contains less than about 0.2 percent by weight of silica and other acid insoluble impurities.

3. A process according to claim 1 wherein the reducing agent is a reducing gas containing free hydrogen.

4. A process according to claim 3 wherein a carbonaceous reducing agent is also used.

5. A process according to claim 3 wherein the reduction is conducted in two stages.

6. A process according to claim 3 wherein the reduction is conducted at temperatures within the range of 1,600° F. to the melting point of iron.

7. A process according to claim 6 wherein the reduction is conducted at a temperature of 2,000° – 2,200° F.

8. A process according to claim 3 wherein the iron is obtained from the reduction zone in the form of a mass of loosely adhering particles which are separated into discrete particles and densified by means of impact densification.