

Dec. 25, 1945.

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2,391,723

METHOD FOR MAKING IRON OXIDE

Filed Feb. 17, 1941

4 Sheets-Sheet 1

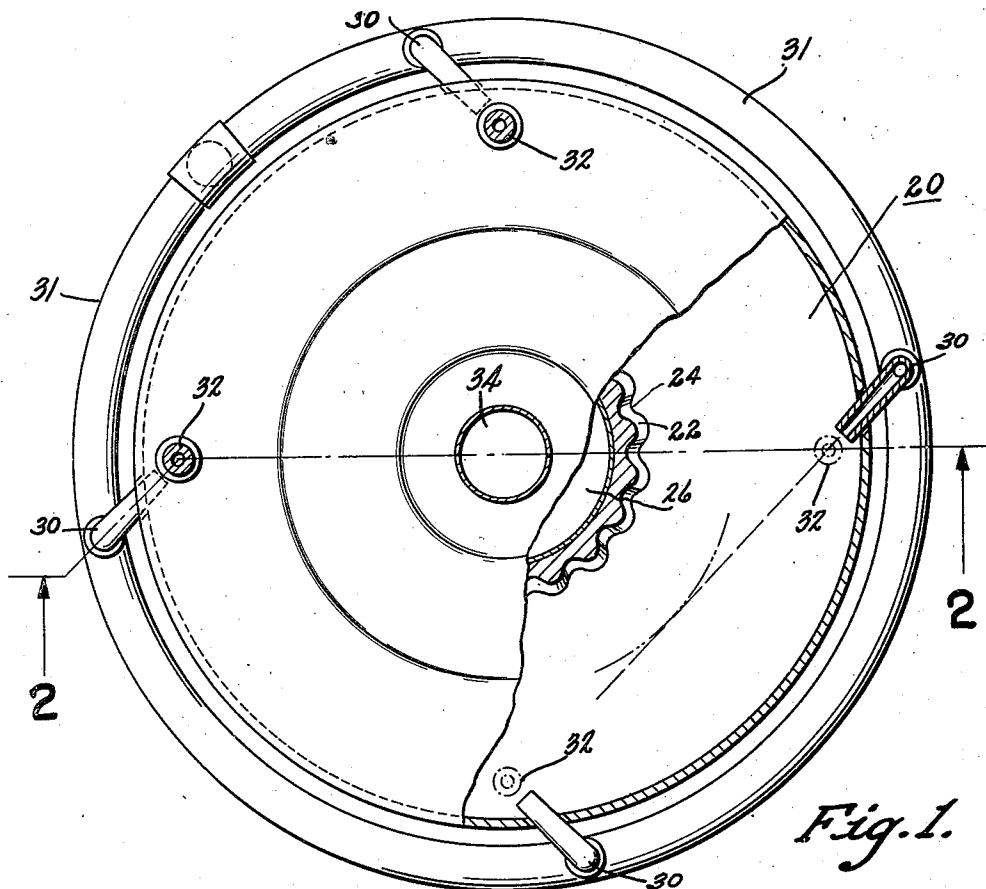


Fig. 1.

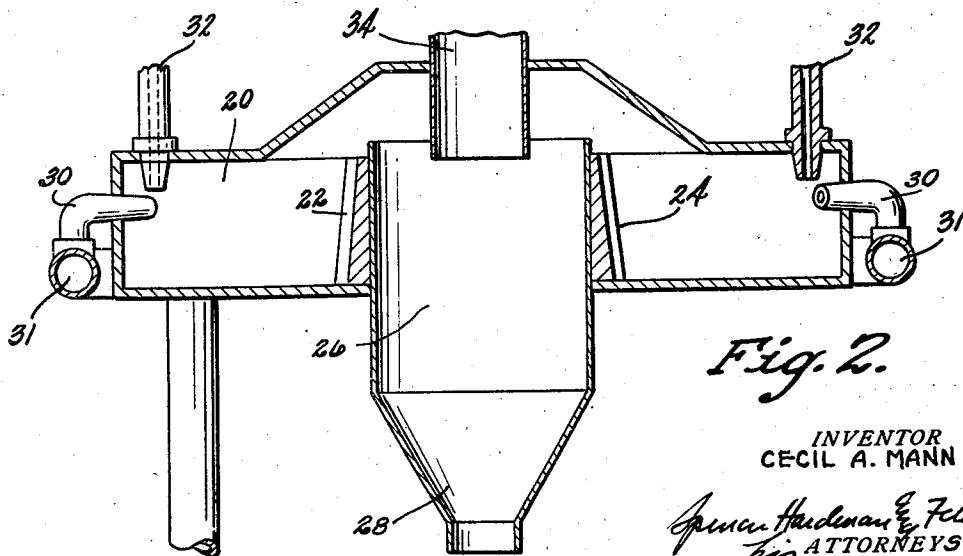


Fig. 2.

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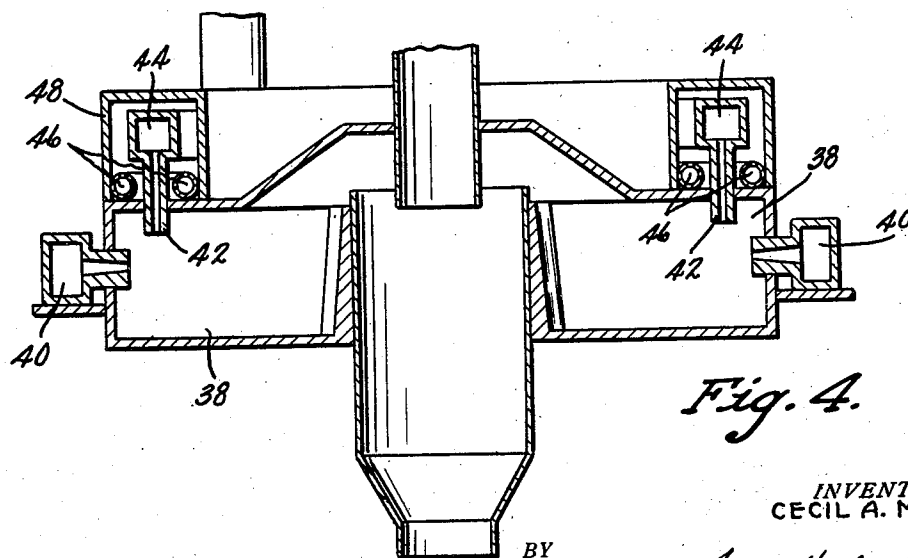
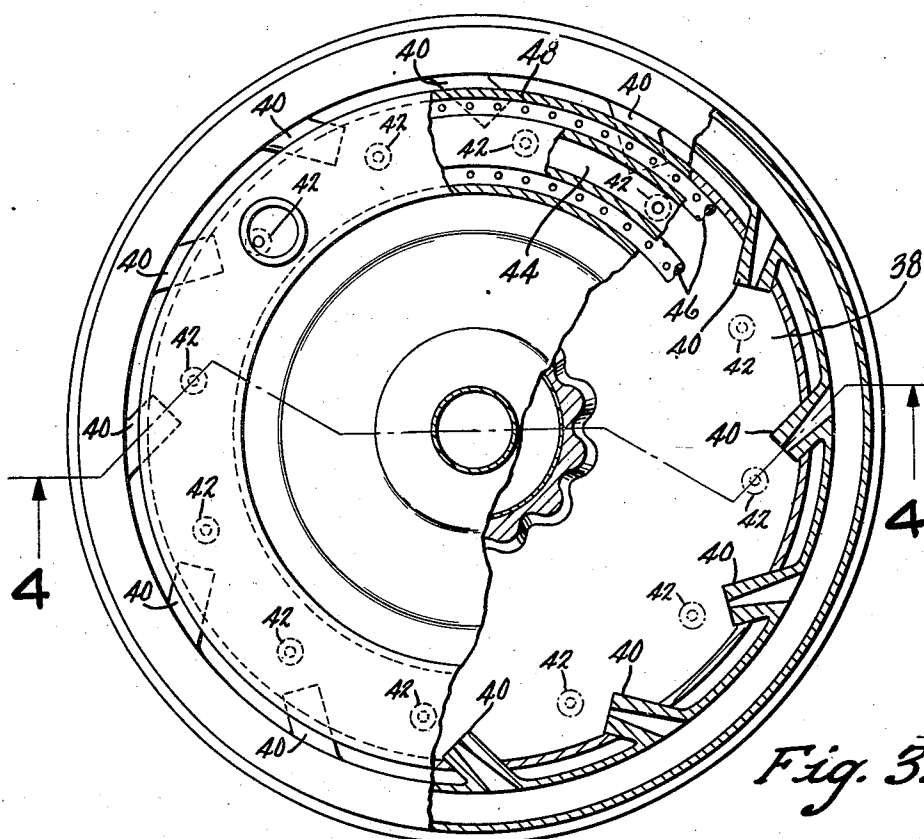
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4 Sheets-Sheet 3

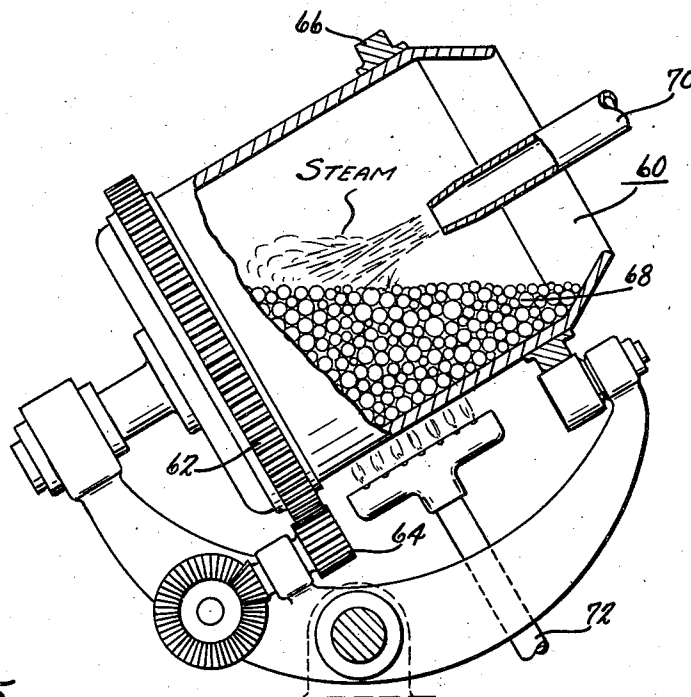


Fig. 5.

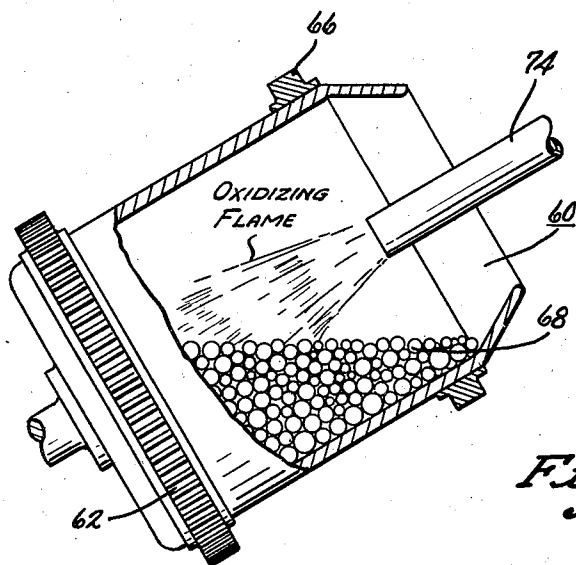


Fig. 6.

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4 Sheets-Sheet 4

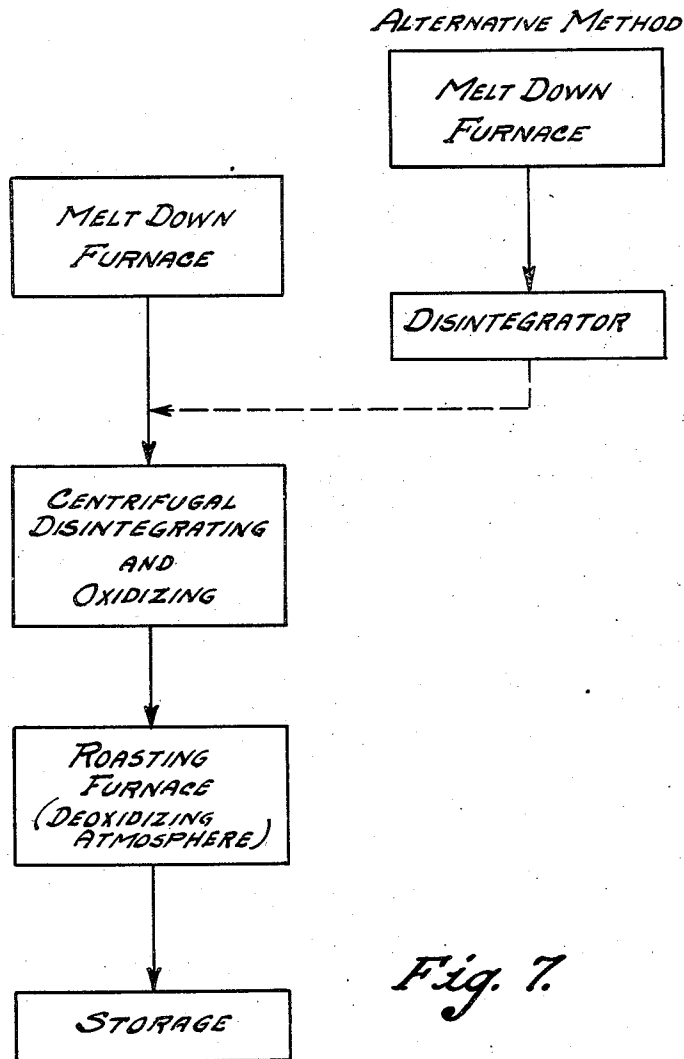


Fig. 7.

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UNITED STATES PATENT OFFICE

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METHOD FOR MAKING IRON OXIDE

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Application February 17, 1941, Serial No. 379,166

6 Claims. (Cl. 23—200)

This invention relates to a method and apparatus for making finely divided iron oxide powder and is particularly concerned with the method and apparatus wherein iron is oxidized and simultaneously comminuted.

The main object of the invention is to provide a method and apparatus for making iron oxide powder of a predetermined particle size in an economical manner, wherein the steps of oxidizing the iron and comminuting the iron are accomplished simultaneously. In carrying out the above object, it is a further object to supply iron either in relatively small particles, or as a molten fluid to a generally-circular axially-confined vortex chamber and causing said iron to rotate at a high rate of speed therein by the injection of high pressure, high temperature steam supplied adjacent the periphery of the chamber, whereby the particles of iron are oxidized by the steam and simultaneously, due to centrifugal force, are impinged at a high rate of speed against the peripheral walls of the chamber whereupon the oxide coating is broken off and wherein the particles of iron are again oxidized while the oxide particles are further comminuted. The rotating fluid within the chamber further causes a centrifugal classifying action of the particles wherein the fine particles which must necessarily be substantially of oxide move inwardly and pass off from the chamber at an inward point thereof.

Another object of the invention is to provide a method and apparatus for producing finely divided iron oxide powder wherein relatively small particles of iron are fed into a suitable apparatus for comminuting the iron which is maintained at a relatively high temperature and which includes an oxidizing fluid therein whereby the surface of the iron is oxidized and progressively is broken off to present new surfaces for oxidation while the oxide broken off is comminuted. The particles may be classified by an air blast or other well-known classifying methods.

In carrying out the above object it is a further object to provide a ball-mill, or a rod-mill as the apparatus is to accomplish the desired result wherein the exterior of the mill is preferably heated and wherein steam or another oxidizing fluid is injected within.

A further object of the invention is to utilize the iron oxide formed in the process as a starting point in the manufacture of sponge iron particles wherein the highly comminuted oxide particles are deoxidized to form the sponge iron.

Further objects and advantages of the present invention will be apparent from the following

description, reference being had to the accompanying drawings wherein preferred embodiments of the present invention are clearly shown.

In the drawings:

Fig. 1 is a partial fragmentary plan view of one type of apparatus which may be used to accomplish oxidation and comminution of iron and iron oxide.

Fig. 2 is a sectional view taken on line 2—2 of Fig. 1.

Fig. 3 is a partial fragmentary plan view of another embodiment of the apparatus which may be used.

Fig. 4 is a sectional view taken on line 4—4 of Fig. 3.

Fig. 5 is a partial fragmentary view of another embodiment of operations to carry out the oxidation and comminution of iron and iron oxide.

Fig. 6 is a modification of the apparatus shown in Fig. 5, and

Fig. 7 is a flow chart showing the steps in the process of making sponge iron powder together with an alternative process as to certain of the steps.

The manufacture of sponge iron powder has heretofore presented numerous difficulties in that from an economic standpoint the oxidation of small particles of iron requires relatively long periods of time. This oxidation is necessary for two reasons. First, it is relatively difficult to comminute iron due to the fact that it is not friable in nature but ductile and therefore usual comminution methods merely change the shapes of particles without breaking them up. Second, iron powder made from anything but iron oxide is not of a spongy nature, which cellular structure of the particles is highly desirable. In the past, methods have been proposed wherein iron is embrittled due to the inclusion of combined carbon whereby the particles are brittle and may be comminuted by conventional disintegrating methods. This embrittled iron in comminuted form is then decarburized. This process however does not produce iron of a spongy character. The process is disclosed in the Clements et al. Patent 2,164,198 assigned to the assignee of the present application.

A method for making sponge iron powder wherein the iron is first decarburized, then oxidized, and then comminuted in separate steps is disclosed in my copending application Serial No. 363,339, filed October 29, 1940, now matured into Patent No. 2,237,867, and assigned to the assignee of the present invention. This application has to do with another method of making a powder sim-

ilar to the powder made by the process disclosed in my aforementioned application, but wherein the steps of the process are reduced due to the simultaneous oxidization and comminution of the iron and iron oxide.

In one embodiment of the invention, I propose to use disintegrating apparatus of the centrifugal pulverizing type wherein a vortex chamber is used in conjunction with an oxidizing fluid to cause centrifugal motion of the particles. Disintegrators of this type are disclosed in the Andrews Patent No. 2,032,827.

Referring particularly to Fig. 1, a generally circular axially confined vortex chamber is shown at 20 which includes an inclined circular central wall 22 which preferably has serrations 24 thereon to aid in the attrition of particles impinging thereon. The outer wall may also be serrated if desired. The annular circular wall 22 forms a discharge port for the chamber as noted at 26 and when material has reached the desired reduced size as predetermined by various calculations, it will pass off from chamber 20 through the discharge port 26 to a collector 28, whence it may be removed, either continuously or intermittently, and passed through a suitable deoxidizing furnace, not shown. The chamber 20 has disposed around the periphery thereof a plurality of injection ports 30 through which steam under high pressure and at a high temperature is admitted to the chamber. The ports 30 are fed from manifold 31 and are all directed so that the stream of injected steam follows a path substantially tangential to an imaginary circle having a radius of approximately one-half the radius of the chamber. In this manner the injected fluid rotates within the chamber so that the path having the greatest rate of rotation is substantially the imaginary circle in the center of the chamber. Above each of said injection ports 30 there is a feeding port 32. The ports 32 are used for introducing the material to be disintegrated into the chamber. The ports 32 are so disposed that material introduced therethrough falls directly into the stream of fluid being injected from ports 30. In this manner material fed into the chamber is immediately whirled therearound whereby particles having the greatest mass are thrown against the outer walls of the chamber and are broken up if they are sufficiently brittle. In the case of iron, if particles of iron are injected through ports 32, said particles being of relatively large size, they are picked up by the stream of steam and due to the high temperature and oxidizing condition of the atmosphere are oxidized at the surface thereof whereby when impinged against the sides of the chamber at high rates of speed the oxide film breaks off in small flakes or particles and the remaining core of iron is again picked up, oxidized, rotated and impinged against the sides of the chamber. Simultaneously the particles of oxide are further impinged. As the particle size of the oxide powder is reduced, there is a tendency for the powder which is of an impalpable nature to be picked up or entrained by the outgoing steam which is drawn off through pipe 34 when the pressure inside the chamber exceeds the pressure in the pipe 34. The entrained particles in the steam passing from the chamber 20 drop by gravity into discharge port 26. Thus only the classified and very fine particles of oxide are taken out of the chamber. In this manner disintegration continues until a desired particle size is arrived at. It is apparent that the velocity of the steam, design of the chamber, etc., may be

varied to accomplish comminution to the desired particle size.

The particles of iron fed into the chamber 20 through ports 32, as heretofore described, are preferably formed by melting iron ingots or scrap, or steel scrap under decarbonizing conditions so that the iron is substantially pure and then pouring this iron into a stream of fluid such as water whereupon it is quickly cooled and simultaneously broken up into relatively small sized particles resembling shot. It is this shot that is fed to the disintegrator. Obviously, the iron may include some alloying ingredients such as manganese, nickel, vanadium, molybdenum, or other conventional ingredients found in steel, if steel happens to be the starting product.

I further propose to use all of the aforementioned steps simultaneously as shown in apparatus illustrated in Figs. 3 and 4. In this instance, the chamber 38 is similar to chamber 20 of Fig. 1 and includes a plurality of injection ports 40 thereon which are preferably directed so that the stream is tangential to a circle having a radius approximately one-half the radius of the chamber. The feeding ports 42 are positioned in a similar manner to those hereinbefore described but are fed from a substantially annular manifold 44. The manifold 44 carries molten iron preferably in a decarbonized condition, thus as the molten iron is fed into the stream of steam 40, it is disintegrated into relatively large size particles and then by the continuous oxidation and attrition within the chamber, it is reduced to small particles of the desired size of iron oxide. In order to prevent premature freezing of the molten iron and subsequent difficulties in the manifold 44 and ports 42 a pair of annular gas burners 46 are provided within a suitably designed chamber 48. The burners 46 heat the manifold 40 to a suitable temperature to maintain the molten condition of the iron. It is apparent that the entire apparatus should be fabricated from a high melting point metal such as high speed steel etc., having characteristics suitable for the proposed use, such metal being well known in the art. In the embodiment shown in Figs. 3 and 4 it is apparent that one of the steps in the process heretofore described may be eliminated since the steam jets break up the molten iron and free it and simultaneously carry out the other functions of disintegrating and oxidizing.

In each instance the steam used should be at an extremely high temperature and pressure so that the chamber and contents are heated to substantially the temperature of the steam. Such temperatures and pressures are best arrived at by trial since under different conditions different control conditions are desirable. I have found, however, that the steam is preferably supplied at temperatures of about 1000° F. and at pressures above 100 pounds per square inch for the most satisfactory results. It is apparent that these temperatures and pressures may be varied and that other oxidizing fluids may be simultaneously injected into the chamber to increase the oxidation, for example, oxygen may be included and it is to be understood in the appended claims that when the term "steam" or "oxidizing fluid" is mentioned that the inclusion of oxygen separately or together with steam, or of other oxidizing fluids separately or together with steam are within the scope of my invention.

Another type of apparatus which may be used to carry out the invention is disclosed in Fig. 5 wherein a conventional type of ball mill 60 is

shown which is driven through a pair of gears 62 and 64, the gear 62 being driven by a suitable motor (not shown). The mill is supported by a plurality of rollers 66, or other suitable supports. In this embodiment iron particles of relatively small size as obtained by passing molten iron into a stream of water or the like, is placed within the mill together with a number of high speed steel balls 68. The mill is then rotated by means of the driving motor whereby the balls mill the powder therebetween and against the sides of the chamber of the mill. In order to obtain an oxide coating on the particles of iron, a pipe 70 is provided in the mouth of the mill and high temperature steam is introduced therein as an oxidizing fluid. The mill is preferably heated to a temperature in the neighborhood of 1,000° F. either by a plurality of gas burners 72 or by electrical heating elements (not shown) incorporated within the walls of the mill.

Another means of obtaining the same results is to eliminate the steam jet 70 and the burner 72 and substitute a burner 74 within the mouth of the mill which burner produces a flame having oxidizing characteristics which plays directly upon the material being milled. Thus the material is heated and simultaneously oxidized.

It will be apparent that these embodiments accomplish the same results as are accomplished in a centrifugal type of disintegrator and in both of the embodiments shown in Figs. 5 or 6, the material may be classified by air flotation methods, screening or other conventional well known classification procedures. Instead of a ball mill, a rod mill, or any other type of attrition apparatus may be used.

The present invention is particularly applicable to the manufacture of sponge iron powder in that it reduces the time period of oxidation usually required. This is occasioned by the fact that as particles of iron are oxidized, the outside coating, in effect, acts as a protecting coating, thereby retarding further oxidation of the article. In the present instance as soon as an oxide coating is obtained, it is removed thereby permitting new iron surfaces to become oxidized. In this manner, the oxide coating does not retard further oxidation of the article. Likewise the broken off particles of oxide may be further disintegrated within the apparatus and drawn off without any possibility of the particles of iron being drawn off unless the iron should happen to be of the desired particle size since the iron itself not being friable is not disintegrated.

Fig. 7 is a flow chart showing the preferred method of making sponge iron powder. In each case a melt down furnace is utilized. However, in one embodiment of the method the molten iron is fed directly to the disintegrator wherein disintegration and oxidation are accomplished simultaneously whereas in the other embodiments the iron is first disintegrated into relatively small size particles which particles were fed to the disintegrator for further disintegration through oxidation and attrition. After the oxide has been classified to the desired particle size, it is then passed through a reducing furnace under deoxidizing conditions and thereby transformed to sponge iron powder which may be stored or used as desired. It is further desirable in many cases to store the iron oxide powder per se and only deoxidize such quantities thereof as are required for immediate use. In this manner iron powder is free from oxides and thereby may be more ad-

vantageously used in the manufacture of porous iron articles which are conveniently prepared by briquetting the iron powder into articles of desired shape and then sintering the briquettes so formed under non-oxidizing conditions to form porous metal articles.

While the embodiments of the present invention as herein disclosed, constitute preferred forms, it is to be understood that other forms might be adopted, all coming within the scope of the claims which follow.

What is claimed is as follows:

1. In a method of making powdered iron oxide, the steps comprising; treating particles of iron with high pressure steam at a sufficiently high temperature to cause an oxide coating to be formed at the surface of the particles of iron, mechanically removing the oxide coating by attrition through agitation by the high pressure steam, oxidizing newly exposed surfaces of the iron and simultaneously disintegrating the removed oxide coating to the size desired.

2. A continuous process of making iron oxide powder, the steps comprising; continuously supplying iron in finely divided form to a disintegrating chamber, continuously agitating said iron with oxidizing fluid at high pressure at a temperature sufficiently high for oxidizing the surface of the iron to iron oxide, continuously removing the iron oxide from the surface of the particles of iron by attrition between the agitated particles as caused by the high pressure fluid, simultaneously disintegrating said iron oxide to a smaller particle size by attrition, oxidizing the particles of iron which have had the iron oxide surface removed therefrom and continuously removing the smaller sized iron oxide particles from said chamber.

3. The process of making iron oxide powder the steps comprising; supplying molten iron to a circular chamber, breaking up the stream of molten iron by means of a stream of an oxidizing fluid at a high temperature and under a high pressure rotating the particles of iron so formed at a sufficiently high rate of speed within the chamber for causing the particles of iron to be thrown against the sides of the chamber, simultaneously oxidizing the surface of the iron by means of said oxidizing fluid whereby a portion of the surface oxide is cracked off of the particles each time they are thrown against the sides of the chamber, simultaneously circulating the particles of iron oxide within the chamber at a speed sufficiently high for causing the particles to impinge against the sides of the chamber for disintegrating the particles to a desired particle size, and then drawing off the excess fluid with the entrained, small particles of disintegrated oxide therein.

4. The method of making comminuted iron oxide comprising the steps of, melting iron, supplying molten iron in small quantities into a zone in proximity to the periphery of a generally-circular, axially-confined vortex chamber, simultaneously discharging an oxidizing fluid at a high temperature and under high pressure into the supply of said molten metal in a manner which will cause the fluid to rotate rapidly in the chamber and simultaneously break up the supply of molten metal to small particles and oxidize the surface of the particles while causing the particles to impinge on the walls of the chamber thereby breaking off the oxide coating which is friable, impinging the broken off oxide coating on the walls of the chamber to disintegrate the same and

then drawing off comminuted particles of the desired size from an inward point in said chamber.

5. In a method of making iron oxide powder of desired particle size, the steps comprising; supplying particles of relatively small size of iron to a vortex chamber causing said particles to move in a circular path of a substantially peripherally confined narrow zone so as to cause the larger particles of said material to be thrown outwardly against the peripheral portion of said chamber by injecting a high velocity high temperature oxidizing fluid into the circular path from a portion of said zone in proximity to its periphery, simultaneously oxidizing the surface of said iron particles whereby said oxidized surface is broken off the particles upon impingement thereof against the confining walls of said chamber, comminuting the oxide particles by further impingement thereof due to tangential movement thereof from said circular path, simultaneously reoxidizing the surface of the particles of iron by means of said fluid, and causing a central classifying action of said particles whereby the finely

comminuted particles of the oxide move inwardly in substantially the plane of said zone and pass off from the central portion of said zone.

6. In a method of making highly comminuted iron oxide, the steps of, supplying iron to a generally circular vortex chamber adjacent the periphery thereof injecting a high temperature, high pressure oxidizing fluid to said chamber at a plurality of points in such a manner as to cause said iron to be moved in a circular path around said chamber and for simultaneously oxidizing the surface of the particles of iron so as to cause the larger particles of said oxidized iron to be thrown outwardly against the peripheral portion of the chamber whereupon the oxidized surface thereof is broken off by impingement of the particles against the wall of the chamber, circulating the iron and the broken off oxide particles, and removing the comminuted oxide when sufficiently fine through the central portion of said chamber.

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