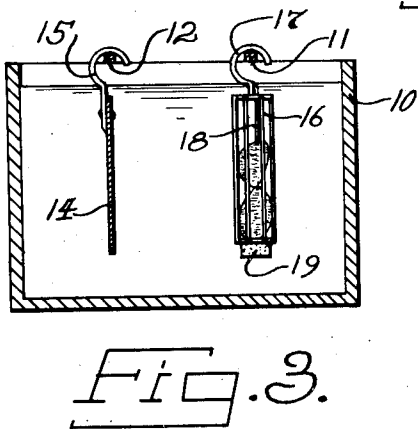
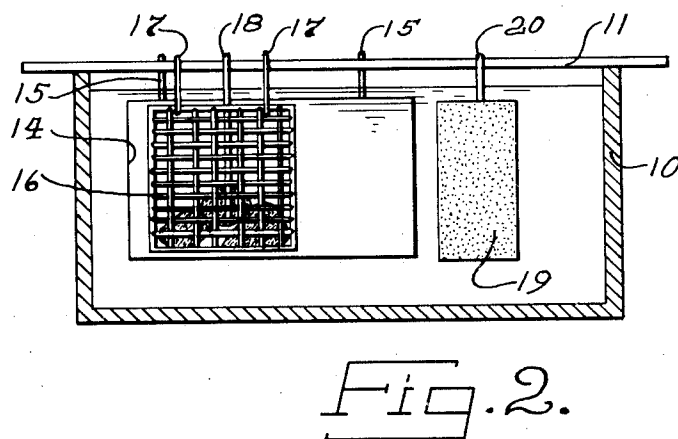
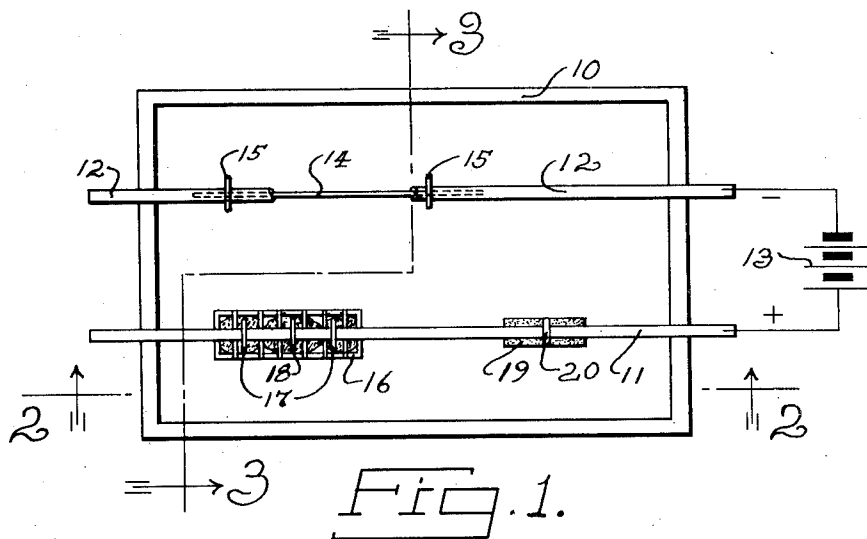


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METHOD OF MAKING ELECTROLYTIC
DENDRITIC POWDERED IRON
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METHOD OF MAKING ELECTROLYTIC
DENDRITIC POWDERED IRON

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1

This invention relates to improved powdered iron and the method of making the same.

More particularly, the invention relates to the electrolytic production of a form of powdered iron which has properties that adapt it especially for fabrication of powdered metal articles.

One of the main objects of the invention is to provide powdered iron having a dendritic structure, particles of which readily mat together under compression to produce a briquette of high and uniform density throughout.

Another object of the invention is to provide powdered iron of this character which, when compressed and subsequently sintered, forms an article having compressive and tensile strength characteristics materially exceeding those of articles formed of prior iron powders.

Another object of the invention is to provide electrolytically deposited dendritic iron which is sufficiently brittle to become readily disintegrated to a powdered state of particles of substantially uniform size.

A further object of the invention is to provide iron powder of this kind which has superior electric properties that adapt it for use in the manufacture of parts of electric apparatus such as armature, field, transformer and other core structures.

Still further objects of the invention are to provide an improved electrolytic process by which powdered iron particles of dendritic structure are economically and commercially deposited; to provide a process of this kind which maintains the electrolytic bath from which the iron particles are deposited in a stable and uniform state during performance of the process; to provide a process of this character in performance of which the contents of the electrolytic bath are continuously automatically replenished at such a rate that the pH value of the bath remains substantially unchanged within relatively close limits during prolonged operation of the bath; to provide a process of this kind by which scrap ferrous metal may be converted to powdered iron particles of uniform size and dendritic structure; and to provide a process of this character which prevents the iron content of the electrolytic bath from precipitating out and which maintains a uniformly high current efficiency.

An additional object of the invention is to provide an improved oxidation preventing treatment in a process of this kind which materially reduces the surface oxide content of the finished powdered iron.

Another object of the invention is to maintain

2

the pH value of the bath substantially constant by providing areas of iron and carbon anode surfaces of predetermined proportions.

A still further object of the invention is to produce a consistently dendritic electrolytic ferrous metal deposition of a brittle nature by maintaining the current density, bath content and concentration, temperature and anode composition within related predetermined limits.

An illustrative embodiment of the invention is shown in the accompanying drawing in which:

Fig. 1 is a diagrammatic plan view of a plating tank illustrating my invention.

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

Fig. 3 is a transverse view taken on the line 3—3 of Fig. 1.

The plating tank, shown in the drawings and illustrated by the numeral 10, is of conventional construction, and it includes side and bottom wall structures comprising either non-electrically conductive material or having a coating of such material thereon which also resists the bath contained in the tank. The top of the tank is open and two conductor bars 11 and 12 extend longitudinally of the tank and rest upon the upper edge of the end walls thereof. The conductor bar 12 is connected to a negative terminal of a source of direct current 13 and the conductor bar 11 is connected to a positive terminal of the source of direct current 13. Suspended from the negative or cathode terminal 12 is a cathode 14, preferably comprising stainless steel which is suspended by conductor hooks 15. The cathode 14 is preferably located at one end of the tank.

Opposite the cathode 14 is located a basket 16 of conventional open mesh construction, the webbing of which is preferably coated with material resistant to the bath contained in the tank, such as synthetic rubber compounds which are conventionally used in the art for this purpose. The basket 16 is suspended from the anode terminal 11 by hooks 17. A conductor 18, preferably comprising a steel bar, is hooked over the anode terminal 11 and extends into and contacts the contents of the basket, preferably comprising scrap iron. The iron scrap forms the iron portion of the anode. A block of carbon 19 is suspended by conductor hooks 20 from the anode conductor bar 11 and located in spaced relation to the iron portion of the anode and to the cathode as shown in Fig. 1. The distance between the carbon portion 19 of the anode and the cathode 14 may be conveniently varied by shifting the carbon block longitudinally of the anode bar toward or away

3

from the cathode without removing the carbon portion of the anode from the bar if desired.

I have found that uniformly dendritic powdered iron particles having the foregoing properties can be consistently produced electrolytically in a continuous process by depositing the iron from the following bath under the conditions hereinafter set forth.

The bath comprises an aqueous solution having a ferrous chloride content of from approximately 30 to approximately 90 grams per liter and an ammonium chloride content of from approximately 10 to approximately 80 grams per liter. The preferred ferrous chloride and ammonium chloride concentration is 30 grams and 40 grams per liter, respectively. A current having a density of from 45 amperes to 200 amperes per square foot is passed through the bath between a cathode which preferably comprises stainless steel and an anode comprising carbon and iron surface preferably in relative amounts and position such that approximately 5% to 10% of the current is conducted directly to the bath by the carbon. The carbon portion of the anode is separate from the iron portion, and it can be varied in position with respect to the cathode for the purposes hereinafter set forth. The current at the iron and carbon portions of the anode can be conveniently ascertained by applying the well-known "tong tester" to the conductors leading to these respective portions of the anode. Scrap iron may be used for the iron content of the anode. The bath may be operated at from approximately 140° F. to 190° F. Lower temperatures may be employed but with an accompanying sacrifice of efficiency.

The pH value of the bath is maintained between 4 and 6.3. By employing the proper proportion of carbon surface with respect to iron surface in the anode the pH value may be retained within this range. Certain variation in the temperature of the bath may be compensated. For example, when operating at a lower temperature, the cathode efficiency falls off and iron builds up in the bath. This tendency can be overcome by increasing the carbon surface portion of the anode with respect to its iron surface portion. The percentage of current carried by the carbon anode surface is maintained approximately equal to the difference between 100% and the actual cathode efficiency which is always less than 100%. The iron portion of the anode has an efficiency of approximately 100%. The carbon portion of the anode has an efficiency of approximately zero. Thus the carbon portion of the anode transmits some of the current to the solution without dissolving any iron and therefore compensates for the portion of the current which does not deposit iron at the cathode.

It has been found that when operating within the foregoing ranges of conditions and concentrations, a current efficiency of about 90% to 95% results. The deposited dendritic iron clings mainly to the cathode and can be readily collected by removing the cathode and scraping the deposit therefrom. Some of the deposit falls to the bottom of the bath and should be cleaned out periodically.

The ferrous iron concentration remains substantially constant under the foregoing operating conditions. It has been found that the pH value, bath concentration, ferrous iron concentration, temperature, current density, and anode composition are very critical and that while one or the other may be varied, usually a corresponding

4

change is required in one or more other functions in order to compensate for such variation. The bath remains stable throughout long periods of operation and does not so change in operation as to substantially alter the size of particles formed at diverse periods of the operation, nor does it break down by throwing out its iron content.

A bath having the above mentioned preferred composition operates most satisfactorily under approximately 100 amperes per square foot current density at approximately 170° F. and with an anode surface comprising carbon and iron in such relative proportions and position that approximately 5% to 10% of the current is conducted to the bath directly by the carbon, the iron being replenished from time to time. The resulting iron deposit has an extremely fern-like dendritic structure and is sufficiently brittle to enable it to be conveniently broken up into particles of a uniform and desired size in a ball, hammer or other similar mill.

In order to maintain the bath at best operating conditions pH determinations should be made either continuously or periodically. A drop in the pH value can be compensated by either spacing the carbon anode portions further from the cathode or decreasing the surface area of the carbon portion. An increase in the pH value can be compensated either by moving the carbon portion of the anode closer to the cathode or by increasing its surface area. The bath is also sampled, but much less frequently, for ammonium ion concentration which is depleted by drag-out and possibly by anodic oxidation. Ammonium chloride is added to compensate for this depletion. The bath is also preferably sampled for iron ion concentration which, if substantially depleted, may be built up by the addition of ferrous chloride.

The following specific example of the preferred operation of the process is apparent from the foregoing description. The bath is made by dissolving in water 30 grams of ferrous chloride and 40 grams of ammonium chloride for each liter of water used to make up the bath. This bath in operation rapidly reaches a pH value of approximately 4.0 and is maintained at a temperature of approximately 170° F. The bath is contained in a conventional plating tank which is provided with the customary positive and negative terminals by which connection is made with the anode and cathode, respectively. The cathode which comprises stainless steel may be suspended in the bath, in accordance with conventional practice, from the negative terminal and the carbon and iron portions of the anode may be similarly suspended in the bath from the same or different positive terminals. A current having a density of 100 amperes per square foot of cathode area is then passed through the bath.

Any desired amount of iron may be initially used in setting up the bath and the remaining portion of anode, which consists of carbon, is then predetermined in quantity with respect to the iron and in position with respect to the cathode so that approximately 5% to 10% of the current is conducted directly to the bath by the carbon. This may be accomplished by starting the bath with an anode having a surface area consisting of roughly 5% to 10% carbon and the remainder iron, and then taking independent readings of the current at the carbon and iron portions of the anode with a tong tester. If the carbon portion is found to conduct more than 10% of the total current, then some of the car-

bon surface is either removed or spaced further from the cathode. If the carbon portion of the anode is found to conduct less than 5% of the total current, then more carbon surface may be added or the carbon portion may be moved closer to the cathode. When using iron scrap, only a rough approximation of the above carbon and iron areas can be made, but the effect of any departure from the above area proportions can be readily corrected by movement of the carbon portion of the anode as above described. As the plating of dendritic iron on the cathode proceeds, the iron surface is reduced and the pH of the bath tends to decrease. This can be compensated for by replenishing the iron to maintain the above distribution of current between the respective portions of the anode, or, if desired, the carbon may be moved further from the cathode. At certain stages of the operation of the process, the iron portion of the anode must, naturally, be replenished but between iron replenishing steps, the above current distribution between the iron and carbon portions of the anode can be maintained by shifting the carbon portion further from the cathode to compensate for the tendency of the pH to decrease as the iron of the anode is depleted, thus maintaining the pH value of the bath between the specified limits of approximately 4.0 to approximately 6.3. The cathode may be removed and the deposit scraped from it as desired during the operation of the process.

Compressed and sintered masses of such powdered iron particles either alone or in mixture with other powdered metals such as copper and tin are characterized by exceedingly high strength and uniform density. Increases in strength as high as 50% have been obtained in structures fabricated from dendritic iron formed in accordance with this invention. The fern-like dendritic structure of the iron particles is believed to produce the improved strength as well as uniform density in the finished product for it promotes an extent of matting together of the particles in an interlocked relationship which does not occur in structures fabricated from iron particles produced mechanically or by other electrolytic processes. This ease of matting or interlocking of dendritic iron particles of the character produced in accordance with the invention, materially reduces pressure requirements in compression operation and enables the attainment of greater porosity without reducing the ultimate strength of the final product.

Although but several specific embodiments of the invention are herein described, it will be understood that various changes in the sequence of operations, steps and materials employed may be made without departing from the spirit of the invention.

I claim:

1. The method of manufacturing powdered iron particles of dendritic structure which comprises forming an aqueous solution having a solute consisting of from approximately 30 to approximately 90 grams per liter of ferrous chloride and from approximately 10 to approximately 80 grams per liter of ammonium chloride, passing current having a density of from approximately 45 to 200 amperes per square foot through said solution between a ferrous cathode and an anode comprising separate iron and carbon portions of such relative surface areas and so spaced from said cathode that from 5% to 10% of said current is conducted directly to said solution by

said carbon anode portion, and maintaining the pH value of said solution against decreasing from between approximately 4.0 and approximately 6.3 by increasing the distance between said cathode and said carbon portion of said anode during continued deposition of iron from said solution.

2. The method of manufacturing powdered iron particles of dendritic structure which comprises forming an aqueous solution having a solute consisting of from approximately 30 to approximately 90 grams per liter of ferrous chloride and from approximately 10 to approximately 80 grams per liter of ammonium chloride, passing current having a density of from approximately 45 to 200 amperes per square foot through said solution between a ferrous cathode and an anode comprising separate iron and carbon portions of such relative surface areas and so spaced from said cathode that from 5% to 10% of said current is conducted directly to said solution by said carbon anode portion, maintaining in said solution during substantially the entire electrolytic operation a ferrous ion concentration corresponding to said ferrous chloride concentration and maintaining the pH value of said solution against decreasing from between approximately 4.0 and approximately 6.3 by increasing the distance between said cathode and said carbon portions of said anode during continued deposition of iron from said solution, maintaining an ammonium iron concentration corresponding to said ammonium chloride range by addition of ammonium chloride, and mechanically reducing the resulting deposit to a desired particle size.

3. The method of manufacturing powdered iron particles of dendritic structure which comprises forming an aqueous solution having a solute consisting of from approximately 30 to approximately 90 grams per liter of ferrous chloride and from approximately 10 to approximately 80 grams per liter of ammonium chloride, passing current having a density of from approximately 45 to 200 amperes per square foot through said solution between a ferrous cathode and an anode comprising separate iron and carbon portions of such relative surface areas and so spaced from said cathode that from 5% to 10% of said current is conducted directly to said solution by said carbon anode portion, maintaining the pH value of said solution against decreasing from between approximately 4.0 and approximately 6.3 by increasing the distance between said cathode and said carbon portion of said anode during continued deposition of iron from said solution, and maintaining in said solution during substantially the entire electrolytic operation a ferrous ion concentration corresponding to said ferrous chloride range.

4. The method of manufacturing powdered iron particles of dendritic structure which comprises forming an aqueous solution having a solute consisting of approximately 30 grams per liter of ferrous chloride and approximately 40 grams per liter of ammonium chloride, passing current having a density of approximately 100 amperes per square foot through said solution between a ferrous cathode and an anode comprising separate iron and carbon portions of such relative surface areas and so spaced from said cathode that from 5% to 10% of said current is conducted directly to said solution by said carbon portion, maintaining said solution at a temperature of from approximately 140° F. to 190° F., maintaining the pH value of said solution against decreasing from between approximately 4.0 and

approximately 6.3 by increasing the distance between said cathode and said carbon portion of said anode during continued deposition of iron from said solution, and in said solution during substantially the entire electrolytic operation a ferrous ion concentration corresponding to said ferrous chloride content and an ammonium ion concentration corresponding to said ammonium chloride content.

HELEN M. CASEY. 10

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