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PROCESS FOR PRODUCING IRON POWDER

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INVENTOR
WILLIAM J. KROLL
BY
E. Greenewald
ATTORNEY

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PROCESS FOR PRODUCING IRON POWDER

William J. Kroll, Niagara Falls, N. Y.

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1

This invention relates to a novel method of making substantially pure iron powder.

Iron powder has been made by several different methods for many years. Such powder, when pure or nearly so, has been expensive and its field of use was for a long time correspondingly limited to a few purposes for which a high price was not prohibitive. More recently, methods of making a moderately pure iron powder have been practiced on a scale sufficiently great to lower somewhat the price of this product. During the same period there have been large advances in the technology of using iron powders for molding machine parts and other articles to accurately controlled density and dimensions. As a result of the general advance in this field, there is a large and rapidly growing demand for iron powder of improved physical properties and constant and uniform composition at a moderate price. It is the principal purpose of this invention to meet that demand.

Iron powder has heretofore usually been produced by three general methods. One involves the reduction, by a reducing gas or solid carbon, of iron oxide, for instance a powdered ore or mill scale, at a temperature below the melting point; the second is by the decomposition of iron carbonyl; and the third is by electrolytic deposit from aqueous electrolytes. These methods yield products having different chemical and physical characteristics and varying in cost, purity, strength, bulk density, particle size, particle shape, and in other ways. As a result, each method has to a considerable degree been limited in utility to its own rather narrow field.

For most widespread utility, iron powder should be uniform in particle size and of high apparent density, be relatively free of embrittling or weakening impurities, be of constant and uniform composition, and be available at a moderate price. Powder having these characteristics can be readily molded, worked, and heat treated to form large or small articles of accurate sizes and shapes at a cost competitive with articles made from cast and wrought metal. This invention provides iron powder having these desirable characteristics.

In its general aspect, the invention comprises the manufacture of substantially pure iron powder by electrolyzing an anhydrous electrolyte of molten ferrous chloride between a consumable anode of impure iron and a cathode from which deposited iron powder may be stripped.

Ferrous chloride ($FeCl_2$) melts at about 674° C. and at high temperatures readily reacts with

2

oxygen or water vapor to form iron oxide which, if present in large amount, interferes with the operation of an electrolytic cell. Moreover, it is difficult to remove the molten ferrous chloride from the deposited iron powder without excessively oxidizing the iron chloride and iron. In the present invention, these difficulties are avoided to a considerable degree by diluting the molten ferrous chloride with at least two other salts selected from the group consisting of sodium chloride, potassium chloride, and calcium chloride. Electrolytes containing calcium chloride tend to absorb water from the atmosphere and to foam when containing such moisture. Therefore, unless the bath is operated in an atmosphere substantially free from moisture it is preferred to use the mixture of ferrous chloride, sodium chloride, and potassium chloride. The dilution of the bath with such further salts also lowers its melting point, and this effect permits the operation of the cell at lower temperatures and facilitates the separation of molten salt from the product.

The mixture of two or more of sodium, potassium and calcium chlorides may be in any proportions. It is preferred that the proportions be approximately equimolar, for instance about 40% to 50% sodium chloride and about 60% to 50% potassium chloride which provides the lowest-melting mixtures.

The ferrous chloride is best maintained at about 20% to 30% of the bath, although at low current densities it may be as little as 10% and at high current densities and in a relatively inert atmosphere it may be as high as 60%.

The consumable anode need not be, and is not, of high purity iron, because most of the more deleterious impurities do not appear in the deposited iron powder. Sulfur, silicon, arsenic, and phosphorus are converted at the anode to volatile compounds which leave the cell. Slag constituents such as silica, alumina, or silicates, drop to the bottom of the cell where they may readily be segregated and removed. Manganese and chromium accumulate in the electrolyte and do not plate out unless their concentration is permitted to become very great. Copper and nickel dissolve in the electrolyte and plate out at the cathode, therefore if these elements can not be tolerated in the product they should not be present in the anode. Carbon is dispersed in the electrolyte and to some extent burns on the surface, and is unobjectionable unless it is present in amounts greater than about 1.5% in which event it tends to interfere with the operation of

the cell. Scrap steel and iron, either as cut pieces of rolled metal or as cast ingots, is a suitable and inexpensive anode material.

In a typical specific instance, the anodes contained 0.294% sulfur, 0.39% phosphorus, 1.38% carbon, 1.71% silicon, and 12.03% manganese, while the powdered iron produced at the cathode contained only 0.015% sulfur, 0.03% phosphorus, 0.06% carbon, 0.04% silicon, and 0.036% manganese.

The cathode may suitably be made of a sheet, plate, or strip of iron or steel, in either the cast or the rolled condition.

The current density has a considerable influence on the character of the iron powder. At cathode current densities below 0.04 ampere per square inch the iron is deposited as a smooth sheet. At cathode current densities above 40 amperes per square inch, long dendrites of deposited iron are rapidly formed, short circuiting the cell, welding the iron particles together, forming a powder of low quality. Similar high current densities at the anode chlorinate the ferrous salt to a ferric salt by free chlorine, thus reducing the current efficiency. Suitable current densities at both anode and cathode are between 10 and 40 amperes, preferably between 10 and 20 amperes, per square inch of electrode surface area, disregarding the surface area of the dendrites. Ordinarily, the voltage will be between 1 and 5 volts.

The electrolyte may be maintained molten either entirely by the flow of electric current or partly by this means and partly by supplemental heating, for instance external heat from a fuel flame or electrical resistor. The cell should be thermally insulated to avoid excessive loss of heat.

Iron crystals of substantially uniform size deposit on the cathode as dendritic accretions which are firmly adherent and coherent. When a convenient amount of iron has been deposited, the cathode is removed from the bath. A large amount of liquid salt clings to the dendrites and protects them from oxidation. If the cathode and deposit are cooled in contact with air, the air will seep into shrinkage cracks, formed during cooling, and will attack the partly cooled iron powder. Such a result may be avoided by cooling the cathode in the absence of air, and then removing the cold dendrites, breaking them up, and washing them with water; or the hot dendrites may be scraped off the cathode into a molten salt bath, stirred, separated from most of the salt by decantation, cooled, broken up, and

cleaned by washing with water. Alternatively, the hot dendrites, stripped from the cathode, may be squeezed in a press to expel some of the salt and to cool the iron rapidly, then broken up and washed in water.

The washing of the dendrites with water is preferably done in several stages, in countercurrent fashion, and the salts concentrated to strong solutions for their recovery and reuse in the electrolytic cell. The final wash water should contain an oxidation inhibitor, such as phosphoric acid, to minimize the oxidation of the iron powder during drying. The drying of the powder is preferably carried out in a vacuum or an inert atmosphere.

The dendrites may be completely broken down, either before or after the washing step, to individual crystals and small clusters and chains of crystals of iron. The iron crystals are sharply angular, pure, and clean, and sized between 100 and 300 mesh. Their apparent density is about 2.4. They have no oxide core, nor do they have a high content of hydrogen or other embrittling elements. They are soft and ductile and easily molded. Their purity is usually better than 99.6% iron. Typical shapes of the iron powder particles, considerably exaggerated in size, are illustrated in the accompanying drawing.

In small-scale operation, typical current efficiencies are about 50% to 70%, and the average power consumption is about 2.5 kilowatt hours per pound of iron produced. On a larger scale, even better conditions can be expected.

I claim:

1. A process for producing substantially pure iron powder which comprises passing an electric current between an impure iron anode and a cathode through a molten salt electrolyte comprising 10% to 60% ferrous chloride and substantially the remainder being at least two salts selected from the group consisting of sodium chloride, potassium chloride, and calcium chloride and maintaining a current density at the cathode between 10 and 40 amperes per square inch.

2. A process for producing substantially pure iron powder which comprises passing an electric current between an impure iron anode and an iron cathode through a molten salt eletrolyte comprising 20% to 30% ferrous chloride, remainder sodium chloride and potassium chloride in approximately equimolar proportions; and maintaining the current density at the cathode within the range of 10 to 20 amperes per square inch.

WILLIAM J. KROLL.