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PROCESS FOR THE PRODUCTION OF IRON POWDER

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This invention relates to a process for the production of iron powder.

In recent years various industries have made use to an increasing extent of iron in powder form. Thus iron powders are being used for the manufacture of porous filter plates, for the manufacture of oil pump gears, in radio transformers and in pharmaceutical preparations.

The increased uses for iron powders have made very exacting demands of the purity and physical properties of such iron powders. The hitherto known processes for the production of iron powders have been unable to comply with these demands with the result that a shortage of inexpensive pure iron powder has hampered further developments.

Electrolytically produced metal powders generally possess a property which is unique to them and which is advantageous for many powder-metallurgical purposes, namely the property that the powder particles are of dendritic shape. In cases where this particular type of particle shape may not be desired the dendrites can be broken up by subsequent grinding or milling.

At the present time mainly chloride and sulphate baths are used for making electrolytic iron. The iron produced in this way is not in powder form and has to be ground. Hitherto it has not been possible to produce electrolytic iron powder directly without milling and thereby destroying the original dendritic particle shape. A further grave disadvantage of electrolytic iron powders produced by the hitherto known processes is the fact that they contain corrosion-promoting occlusions from the acid baths, which occlusions are extremely difficult to remove.

One object of the present invention is to provide a simple and inexpensive process for the electrolytic production of iron which is at the outset in powder form.

A further object of the invention is to produce iron powders which are free from corrosion-promoting occlusions.

Still a further object of the invention is to produce iron powder of a very high degree of purity.

Further objects and advantages of the invention will become apparent as the description thereof proceeds.

According to the present invention iron powder or iron sponge is deposited cathodically in a concentrated solution of caustic alkali at an elevated temperature, using iron anodes. Experiments have shown that it is possible, by suitably varying the conditions of deposition, to produce on the cathode any kind of iron deposit varying from a

very fine powder which can be used directly without grinding or milling, to a more or less porous spongy deposit which can however be broken up into powder form by a simple grinding operation.

For the purposes of the invention, it is essential that the concentration of the caustic alkali electrolyte should be over 9-normal and that the temperature at which the deposition is carried out should exceed 100° C.

The electrolyte employed may consist of caustic soda, caustic potash or barium hydroxide. When using caustic soda, the specific gravity of the electrolyte should preferably be between 1.35 and 1.60 and the temperature of the electrolyte should preferably lie between 110° C. and boiling point.

It has been proved experimentally that the physical properties of the deposited iron are mainly dependent on the cathodic current efficiency at which the electrolysis is carried out. When employing a low cathodic efficiency the deposit is of a powdery nature, whilst with rising cathodic efficiencies the deposit tends to become progressively more coherent and less porous.

It has been found that if not more than 0.3 gm. of iron per ampere hour is deposited cathodically the deposit is a powder or that it can at least be very easily broken up into a powder for instance by rubbing it between the fingers. At higher current efficiencies deposits are produced which have to be ground or milled in order to convert them into powder form. A characteristic advantage of the invention is that the deposited iron can be ground in a wet state since the presence of caustic alkalis in the absence of acid anions such as chloride or sulphate protect the iron from corrosion.

The cathodic current efficiency depends on the anodic current efficiency and is therefore increased by all factors which promote a dissolution of the anodes, such as high temperatures, agitation of the electrolyte, low anodic current density and frequent cleaning of the anodes. At temperatures below 100° C. and/or at low concentrations of caustic alkali, for example in electrolytes containing 300 gms. of caustic soda per liter, hardly any iron is dissolved or deposited and only hydrogen and oxygen develop on the electrodes. At higher temperatures and concentrations increasing amounts of iron are deposited particularly if the overall anode surface is made substantially larger than the cathode surface and is kept clean.

In carrying out the invention, anodic current densities not exceeding 5 amps. per square decimeter are preferably employed. The cathodic

current density employed should preferably exceed 2 amps. per square decimeter.

In order to obtain deposits in the form of powder or deposits which need a minimum of grinding to convert the same into powder, the cathodic current efficiency should be kept below 0.5 gm. of deposit per ampere hour.

The particle size of the deposited iron is also influenced, according to well known electrochemical rules, by the conditions under which the deposition is carried out. High temperature and low cathodic current density produce deposits of a larger grain size which is to some extent also influenced by the movement of the electrolyte and the nature of the cathode material.

The addition of small quantities of anions, such as chloride or sulphate, to the caustic electrolyte solution improves the solubility of the anodes. In view of the corrosive effect of such anions on the depositing tank and, as occlusions, on the iron powder produced, it has been found advisable not to add more salt than corresponds to 5% of the amount of caustic alkali present. Thus when using sodium chloride as an addition to the electrolyte it is advisable to keep the amount of the added salt below 10 gms. per litre of electrolyte.

At the elevated temperatures at which the electrolysis is carried out, evaporation losses become appreciable. In order to avoid accumulation of extraneous salts in the electrolyte, such evaporation losses are preferably made up with distilled water.

Example 1

An aqueous solution consisting of equal parts by weight of potassium hydroxide and sodium hydroxide and having a specific gravity of 1.35 gms. is electrolysed in a concrete tank at a temperature of 110° C. using one or more anodes consisting of mild steel and a cathode consisting of a nickel-plated drum rotating about a horizontal axis at the rate of 200 revolutions per minute. For the deposition a cathodic current density of 20 amperes per square decimetre and an anodic current density of 2 amperes per square decimetre are employed. During the deposition, the hydrogen and oxygen evolved are collected separately. At the termination of the deposition the cathode is removed from the bath, the deposit is scraped off, washed several times with tap water and dried in a centrifuge.

The deposit consists of a very fine powder of average particle size of between 1 and 5 microns. Less than 0.1 gm. of iron is deposited per ampere hour.

Example 2

A solution, in distilled water, of pure sodium hydroxide containing 800 gms. of sodium hydroxide per litre is electrolysed in an iron tank at a temperature of 150° C. The anodes consist of clean ingot iron strips which are spaced apart by washers and which expose an overall surface inside the electrolyte which is 60 times as great as the cathode surface. The cathode is a flat silver-plated copper plate. The tank is provided with an airtight cover having conduits for the evolved gases and the gases are allowed to escape under pressure. The electrodeposition is carried out using a cathodic current density of 5 amperes per square decimetre and iron is deposited on the cathode at a rate exceeding 0.4 gm. per ampere hour. The current efficiency increases after the bath has been in use for some days.

The iron is deposited on the cathode in a coherent form and after removal from the cathode the deposit is ground in a wet state in an end-runner mill. Thereafter the powder is washed with hot water and alcohol, centrifuged and vacuum dried. The resulting powder consists of a very pure iron having a metallic iron content of 99.5% and a loading weight of 3.5 grams per cubic centimetre.

Example 3

A solution of commercial sodium hydroxide of a specific gravity of 1.45 gms. and containing a minute percentage of sodium chloride, sodium carbonate and sodium silicate, is electrolysed at a temperature of 135° C., with the aid of anodes consisting of cast iron strips having an overall surface equal to ten times the surface area of the cathode which consists of a flat smooth steel plate. The electrolysis is carried out at a cathodic current density of 15 amperes per square decimetre and a deposit forms on the cathode which is only slightly coherent and can be broken up into powder form by a light rubbing.

Example 4

A solution of commercial sodium hydroxide of a specific gravity of 1.5 gms. is electrolysed in a cast iron tank and heated by means of nickel-sheathed electric immersion heaters to a temperature of 130° C. The anodes consist of bent steel strips resting on the sides of the tank while the cathode is a polished stainless steel cylinder rotating about a horizontal axis at a rate of 5 revolutions per hour and immersed to less than half its extent in the electrolyte.

The electrolysis is carried out with a cathodic current density of 50 amperes per square decimetre. The tank is covered to prevent evaporation losses and the deposit is removed continuously from the cathode by means of scraper knives. A very fine iron powder is produced which, after washing and drying, can be used directly without any grinding.

I claim:

1. A process for the production of iron deposits suitable for use as iron powders, which comprises electrolysing an aqueous solution of a caustic alkali of a strength exceeding 9 normal at a temperature lying between 110° C. and the boiling point of the electrolyte at the pressure prevailing, with the aid of an anode of ferrous material and a cathode inert to the electrolyte, using a cathodic current density exceeding 2 amperes per square decimeter and an anode current density below 5 amperes per square decimeter, said aqueous solution being free from additions of iron other than that obtained by dissolution of the anode of ferrous material so that substantially all the iron deposited on said cathode is derived from the dissolution of said anode.

2. A process, as claimed in claim 1, in which said aqueous solution is an aqueous solution of caustic soda having a specific gravity between 1.35 and 1.60.

3. A process, as claimed in claim 1, in which said aqueous solution contains a mixture of caustic alkalis.

4. A process, as claimed in claim 1, in which the cathode used has an overall surface substantially smaller than that of said anode.

5. A process, as claimed in claim 1, in which a cathodic current efficiency is used which is below 0.5 gram of deposited iron per ampere hour.

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