ELECTROLYTIC METHOD OF MANUFACTURING MONOCRystalline IRON


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5 Claims

ABSTRACT OF THE DISCLOSURE

A method of manufacturing monocrystalline iron by electrolytic deposition from an aqueous, iron-containing solution having an iron concentration in the range of 10 to 400 g./l. and a pH value in the range of 0.5 to 6 at a temperature of 20° to 320° C. and at a current density of 0.05 to 400 a./dm².

The present invention relates to a field of electrowetting, and more particularly to a method of manufacturing monocrystalline iron by the electrolysis of an aqueous, iron-containing solution.

Until recently, a cathodic product has been obtained as a powder or in the form of a polycrystalline metal from aqueous, iron-containing solutions by the electrolytic deposition of iron.

The iron produced in such a manner, however, does not possess the required degree of purity, because one has to carry out the process with additions of an electrically conducting salt to the solution. Accordingly, sufficient attention is not paid to the purity of the material being with which the cell and electrodes are made nor to the purity of the electrolyte. Moreover, when depositing iron in the form of powder and polycrystalline iron, there is a great disadvantage in that there occurs an additional adsorption of impurities on the surface and in the boundaries of crystals.

It is known in the prior art to manufacture the monocrystalline iron by the electrolysis of molten salts containing iron. The accomplishment of this method, however, is hindered by the great expense of the apparatus involved.

An object of the present invention is to eliminate the above-said disadvantages.

The principal object of the present invention is to provide a method ensuring the manufacture of a pure monocrystalline iron from aqueous solutions containing iron.

This object is accomplished by the provision of a method of manufacturing monocrystalline iron according to the present invention, whereby iron is deposited by the electrolysis of an acidic aqueous iron-containing solutions with the concentration of iron therein being equal to 10 to 400 g./l., preferably between 80 and 180 g./l., with the pH equal to 0.5 to 6, preferably between 1.5 and 4.5, at a temperature of 20 to 320° C., and with a current density of 0.05 to 400 a./dm².

According to this method, it is also possible to deposit the iron monocrystals on a polycrystalline cathodic material at a temperature of 100 to 320° C., preferably between 125 and 190° C., with the current density of 5 to 400 a./dm², preferably between 10 and 200 a./dm².

The nature of the present invention will become more apparent from a consideration of the following description of an exemplary embodiment thereof.

EXAMPLE

A solution of iron chloride with a concentration of iron equal to 125 g./l. and the initial value of pH equal to 2.0 is employed as the electrolyte. The electrolyte is prepared by the dissolution of a pure carbonyl iron in a clarified hydrochloric acid. The cell to be used in the electrolysis is made of Teflon. A cylinder made of a pure electrolytic iron is employed as the anode, while rods of an electrolytic iron with a finely crystalline or coarsely crystalline structure are used as the cathodes, said structure being obtained by a recrystallization effected at a temperature of 900° C. in a stream of hydrogen. Before utilization, the cathodes are slightly etched with a diluted hydrochloric acid. The cell, complete with the electrolyte and electrodes, is placed in an autoclave. The autoclave is made of stainless steel, being designed so as to be capable of operating under a pressure of 50 atm. at a temperature up to 250° C. The current lead-ins are made of stainless steel. The packing of the autoclave cover and the insulation of the current lead-ins are Teflon. With a view toward protecting the electrolyte against oxidation, the cell for the electrolysis and autoclave is blown through with nitrogen containing no more than 0.005% oxygen. This gas is set up in the autoclave the pressure required.

The electrolysis is effected at a cathodic current density of 10 a./dm². An iron deposit is obtained on the coarsely crystalline base at a temperature of 125° C., the structure of this deposit reproducing the base structure as a result of epitaxial deposition. Iron monocrystals of acicular form are obtained on the finely crystalline base at a temperature of 190° C. The monocrystalline grow in the direction (III) and possess six uniform limiting planes whose orientation is defined as (110). The length of the iron monocrystals, are obtained with an amount of electricity passed on the order of 0.6 amp-hour, is likely to approach the value of 20 to 25 mm. with the diameter thereof being approximately equal to 1 mm.

To carry out this method there may be employed an electrolyte prepared by the dissolution of a salt of bivalent iron and purified according to known methods. The cell for the electrolysis at high temperatures may be designed as an autoclave or may be made of a synthetic material, e.g., pure iron, platinum or any other metal resistant to the action of electrolites. As the cathodic material, it is expedient to employ iron, platinum or any other metal, resistant to the action of electrolites, preferably iron monocrystals obtained by preliminary electrolytic deposition or in any other way. Preliminary purified iron or pure sintered carbonyl iron may be used as the anodic material. The autoclave is plated on the interior by a synthetic material, preferably Teflon, pure iron, platinum or any other metal resistant to the action of electrolites. The autoclave is designed so as to be capable of operating under a pressure corresponding to the pressure of the electrolytic vapour generated at a given temperature. The current lead-ins are made of pure iron, platinum or any other metal resistant to the action of electrolites, while the insulation is made of synthetic material. In order to prevent the oxidation of ions of the bivalent iron in the electrolyte by the atmospheric oxygen, the cell for the electrolysis is blown through with an inert gas which is poor in or completely free of iron, and then, where operating at high temperatures, the required pressure is set up in the cell by the aid of this inert gas.

What is claimed is:

1. A method of manufacturing monocrystalline iron,
consisting in that the iron is electrolytically deposited from
an acid aqueous, iron-containing solution, with the concen-
tration of iron therein being in the range of 10 to 400
g./l. and the pH value being in the range of 0.5 to 6, at a
temperature of 20 to 320° C., and at a current density
of 0.05 to 400 a./dm.².

2. A method according to claim 1, wherein a crystalline
iron cathode is employed and an epitaxic deposition of
iron on the iron monocrystals effected at a temperature of
20 to 150° C. and at a current density of 0.05 to 10
a./dm.².

3. A method according to claim 1, wherein a poly-
crystalline cathode is employed and a deposition of iron
monocrystals on the polycrystalline cathodic material is
effected at a temperature of 100 to 320° C. and at a cur-
rent density of 5 to 400 a./dm.².

4. A method according to claim 1 wherein said concen-
tration of iron is in the range of 80 to 180 g./l.

5. A method according to claim 1 wherein said pH is
in the range of 1.5 to 4.5.

References Cited
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