

UNITED STATES PATENT OFFICE

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METHOD OF DEPOSITING IRON
ELECTROLYTICALLYFranz Pawlek, Berlin-Karlshorst, Germany, as-
signor to Ernst Kelsen, Vienna, and Edgar
Ausnit, BucharestNo Drawing. Application June 17, 1931, Serial
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10 Claims. (Cl. 204—1)

- This invention relates to an improved method of depositing iron electrolytically. If iron be deposited from solutions of iron salts, e. g. ferrous chloride solutions at higher current densities of over 3 amperes per dm^2 , particularly on flat cathodes, the deposit after a short time bursts up and breaks away from the surface of the cathode. It is recognized that the reason for this objectionable phenomenon is to be found in the absorption of hydrogen by the electrolytic iron, and efforts have therefore been directed hitherto towards the obtaining of conditions for the electrolytic process calculated to cause a reduction in this absorption of hydrogen.
- It is known that the quantity of hydrogen absorbed by ferrous metals increases with the reduction of temperature and with the increase of current density. Since for economic reasons, in industrial electro-metallurgy, e. g. in the manufacture of sheet iron, only high current densities can in practice be employed, it is usual to work with electrolytes at very high temperatures, in order to reduce as far as possible the hydrogen content of the iron, and to be able to compete with the thermic process. Experience has however shown that even with bath temperatures of 90°C . the bursting up of the deposit from a more or less level base (cathode) can not be prevented.
- The present invention is based upon the established fact that the hydrogen content of an electrolytically deposited sheet or other product is not a constant factor even at constant temperature and current density, but diminishes, at first rapidly, and then somewhat more slowly, in the successive layers becoming finally constant. It has been assumed that, through the difference in the hydrogen content between the individual layers, internal strains are produced which are responsible for the scaling and curling of the deposit, and that the remedy would therefore lie not in diminishing the hydrogen content, but in making the same equal for all the layers of the deposit.
- The present invention is based upon the recognition of the truth of this assumption, and consists in the provision of a method for the manufacture electrolytically in particular of sheet iron in which the alteration of the hydrogen content is prevented from the outset of the manufacturing process.
- This object is achieved, in accordance with the invention, by alteration of the current density during the depositing process, a low current density being employed at the commencement to reduce the rate of absorption of hydrogen by the electrolytically deposited iron, and the current density being increased in the course of the process, preferably at such a rate that the reduction in the absorption of hydrogen effected in the first stage of the process is entirely or approximately counterbalanced, and all the layers of the deposit possess throughout the same or approximately the same hydrogen content.
- This increasing of the current density from a value far below the desired and permissible working current density up to the latter can be effected gradually or by stages, but must always depend upon and take account of the behavior of the deposited layers in regard to the absorption of hydrogen in the separate periods of that time during which alterations take place in the rate of absorption of hydrogen at constant temperature and at constant current density. As soon as the state of invariability in the rate of absorption of hydrogen by the electrolytic iron has been arrived at, the current density then being employed can be retained practically indefinitely, i. e. until the attainment of the desired thickness of the deposit. In practice it has proved to be sufficient to increase the current density by stages during the time of the variability of the rate of hydrogen absorption. In any case the temperature of the electrolyte should be maintained at a constant level; should the temperature alter, this fact must be taken into account proportionately in the determination of the rate of increase of the current density.
- As a concrete example of the carrying out of the method provided by the present invention with a working current density of 5 amperes per dm^2 , a current density of approximately 2 amperes per dm^2 can first be employed, and this density be gradually increased in the course of 30–45 minutes to the working density of 5 amperes per dm^2 . It is also possible to start with a current density of less than 2 amps. and to increase this density to 5 amps./ dm^2 or to even higher current densities. The time during which this increase should take place varies somewhat with the difference in value between the initial and final current densities employed, but is in general less than 60 mins.
- The effect of the alteration of the current density will be at once apparent; since the hydrogen content decreases with the time, and increases with the current density, the gradual increase in the current density enables a substantially constant rate of hydrogen absorption to be maintained from the outset, and thus coun-

teracts the tendency of the deposit to scale and to curl away from the underlying surface.

The method according to the invention also influences a further circumstance which is of importance for the detachment and scaling of the deposit, namely it also influences the grain of the metal in the layers of the deposit. If the grain be very fine, the separate particles tend to reduce their comparatively large total superficial area by the enlargement of the single particles, and thus as a whole are subject to an element of contraction which must be regarded as a further cause for the scaling of galvanic deposits. It therefore becomes desirable to prevent the obtaining of a fine-grained deposit, at all events at the commencement of the depositing process. Since the size of the grain becomes smaller with the increase in the current density, the method provided by the present invention also ensures that the layer of the deposit in contact with the underlying surface be not too fine-grained. Since, moreover, the size of the grain increases in the course of the process, the gradual increase in the current density influences the size of the grain to the same extent as it influences the rate of hydrogen absorption.

I claim:—

1. A method of depositing iron electrolytically comprising exposing the electrolyte initially to the action of an electric current of density lower than the maximum density maintaining during the process, and increasing the density of the said current in the course of the electrolytic process to its maximum at a rate which will equalize differences in the rate of hydrogen absorption in the separate layers of the said deposit, and which will obtain a practically constant hydrogen content in all the layers of the said deposit.

2. A method of depositing iron electrolytically comprising exposing an electrolyte initially to the action of an electric current of density lower than the maximum density maintaining during the process, increasing the density of the said current in the course of the electrolytic process up to the desired and permissible maximum current density at a rate which will equalize differences in the rate of hydrogen absorption between individual layers of the said deposit, and maintaining the last-named current density until the attainment of the desired thickness of deposit.

3. A method of depositing iron electrolytically comprising exposing an electrolyte initially to the action of an electric current of density lower than the maximum density maintaining during the process, gradually and sequentially increasing the density of the said current during a period of time not exceeding 60 minutes from the commencement of the action of the said current up to the desired and permissible maximum current density at a rate which will equalize differences in the rate of hydrogen absorption as between individual layers of the said deposit, and maintaining the last-named current density until the attainment of the desired thickness of deposit.

4. A method of depositing iron electrolytically comprising exposing an electrolyte initially to the action of an electric current of density lower than the maximum density maintaining during the process, increasing by stages the density of the said current during a period of time not exceeding 60 minutes from the commencement of the action of the said current up to the de-

sired and permissible maximum current density at a rate which will equalize differences in the rate of hydrogen absorption as between individual layers of the said deposit, and maintaining the last-named current density until the attainment of the desired thickness of deposit.

5. A method of manufacturing sheet iron by depositing iron electrolytically on flat cathodes comprising retaining an electrolyte at a constant high temperature, exposing the said electrolyte initially to the action of an electric current of density lower than the maximum density maintaining during the process, increasing the density of the said current during a period of time not exceeding 60 minutes from the commencement of the said electrolytic process up to the desired and permissible maximum current density at a rate which will equalize differences in the absorption of hydrogen between individual layers of the said deposit, and maintaining the last-named current density until the attainment of the desired thickness of deposit.

6. A method of manufacturing sheet iron by depositing iron electrolytically on flat cathodes comprising exposing a hot electrolyte initially to the action of an electric current of density lower than the maximum density maintaining during the process, gradually increasing the density of the said current, account being taken of fluctuations in temperature in the said electrolyte, during a period of time not exceeding 60 minutes from the commencement of the said electrolytic process up to the desired and permissible maximum current density at a rate which will equalize differences in the absorption of hydrogen by different increments of thickness of the said deposit, and maintaining the last-named current density until the attainment of the desired thickness of deposit.

7. A method of manufacturing sheet iron by depositing iron electrolytically on flat cathodes comprising exposing a hot electrolyte initially to the action of an electric current of density lower than the maximum density maintaining during the process, increasing by stages the density of the said current, account being taken of fluctuations in temperature in the said electrolyte, during a period of time not exceeding 60 minutes from the commencement of the said electrolytic process up to the desired and permissible maximum current density at a rate which will equalize differences in the absorption of hydrogen by different increments of thickness of the said deposit, and maintaining the last-named current density until the attainment of the desired thickness of deposit.

8. The method of maintaining substantially constant the hydrogen absorption in successive increments of deposition of iron in the electrolytic production of iron, comprising initially passing a current through the electrolytic bath of lower density than the maximum current density used during the process, and successively raising the current density during the process to its maximum value, the rate of change of current being approximately inversely proportional to the previously determined rate of change of the hydrogen absorption in the successive increments of iron deposit.

9. The method of maintaining substantially constant the hydrogen absorption in successive increments of deposition of iron in the electrolytic production of iron, and of controlling the crystal structure of the deposited metal, comprising initially passing through the electrolytic

bath a current of approximately two amperes per square decimeter of cathode area, and thereafter successively increasing the current density by stages during the process to a maximum value of approximately 5 amperes per square decimeter, the rate of change of current density being approximately inversely proportional to the previously determined rate of change of hydrogen absorption in the successive increments of iron deposit.

10. The method of maintaining substantially constant the hydrogen absorption in successive increments of deposition of iron in the electro-

lytic production of iron, and of controlling the crystal structure of the deposited metal, comprising initially passing through the electrolytic bath a current of approximately two amperes per square decimeter of cathode area, and thereafter successively increasing the current density by stages during the process to a maximum value of approximately five amperes per square decimeter, the current density being gradually increased in stages from its minimum to its maximum value in approximately 30 to 45 minutes.

FRANZ PAWLEK.

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