

UNITED STATES PATENT OFFICE

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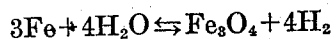
METHOD FOR PRODUCING MAGNETIC PLATES FROM ELECTROLYTIC IRON

No Drawing. Application filed May 28, 1932, Serial No. 614,289, and in Germany June 22, 1931.

For electrotechnical purposes, particularly for the construction of transformers it is desirable to employ iron plates having as low a hysteresis loss as possible. Apart from mechanical conditions, such as grain size, hardness etc., the shape of the hysteresis curve will be affected by the other elements in the iron. Each of the elements, oxygen, carbon, sulphur, phosphorus and manganese has a detrimental effect. Although the quantities of sulphur, phosphorus or manganese may be easily reduced in the manufacturing process, so that they no longer have a detrimental effect, the matter is not so simple in the case of the most detrimental elements oxygen and carbon. However, in actual practice the adverse effects of oxygen and carbon have been reduced by the addition of a small percentage of silicon.

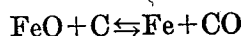
In the case of electrolytic iron however the conditions are different. When producing such iron from chloride or sulphate baths the quantity of each of the elements, sulphur, silicon or phosphorus will be about 0.001-0.003 per cent and manganese 0.02 per cent. The carbon will vary within limits from about 0.01-0.04 per cent if the work is carried out without a diaphragm and with an anode material having a low carbon content. The oxygen content of the electrolytic iron is very high with regard to its adverse influence, namely 0.02-0.05 per cent, which is about ten times as much as that contained in a good quality Siemens Martin steel. The oxygen in the electrolytic iron may be introduced in two ways. In one case, through the enclosed bath residue; this part will be all the greater the more acid the bath is. In the other case it may be introduced by a basic iron salt which may be in suspension in the bath and which separates out from the electrolyte when the latter is insufficiently acid, that is when the hydrolytic balance of the corresponding iron salt has been exceeded. These basic salts are positive colloids; they travel to the cathode and settle down in the deposit. If the electrolytic iron is then annealed in order to drive out the hydrogen and get rid of the stresses which may be present in the plate, the

bath residue present in the deposit, particularly water and iron, will react as follows:



As this balance is entirely displaced to the right at the annealing temperature, (approx. 850°) oxidation will occur whilst the hydrogen is escaping. The basic salts thrown down as colloids in the deposit will also react on a similar principle. The oxide formed will dissolve the iron and will have an extremely prejudicial influence upon the magnetic properties of the iron.

In electrolytic iron, oxides and carbon are present therefore after a short annealing period. These may react with one another as follows:



This balance is approximately in the middle at the temperatures in question (approx. 850°). If on the other hand the CO is continuously removed, the annealing being done in vacuum, the balance will be constantly disturbed and the reaction will take place from left to right until either no more FeO or no carbon is present. The factor which determines the speed of the reaction is in this case the speed of diffusion of the FeO and C in the iron. As this is very slow, a correspondingly long annealing period must be adopted. By this means it is possible to remove entirely one of the constituents, oxygen or carbon, according to which of them is present in the smaller amount.

It has been stated above that the carbon content of the electrolytic iron will depend upon the carbon content of the anode, whilst the oxygen content will depend upon the acid content of the bath. It is possible with a given acid content in the bath to obtain a corresponding quantity of oxygen in the iron and by employing an anode containing more or less carbon to accumulate a quantity of carbon in the iron equivalent to the oxygen. It is possible when employing anodes of pure material to obtain a carbon content in the deposit by adding to the bath organic substances such as oxalic acid, citric acid, succinic acid etc., or water soluble salts of the latter or or-

ganic colloids such as gum arabic, gelatine etc., which become positively charged in the bath. The quantity of carbon is approximately proportional to the quantity of organic matter present in the bath. By this means therefore a quantity may be introduced into the iron which is equivalent to the quantity of oxygen.

It is also possible by varying the acid content of the bath to add a corresponding quantity of oxygen to a predetermined carbon content. The oxygen content of the electrolytic iron will fall somewhat on reducing the acid concentration from pH=2 to pH=2.7 and will then rise enormously when the hydrolytic balance is exceeded. These data are correct for a bath of iron chloride with approximately 80 g Fe/l, a bath temperature of 90° C. and a current of 5 A/dcm². The oxygen content will alter accordingly by varying the conditions of deposition.

It is possible, therefore, during deposition to introduce equivalent quantities of oxygen and carbon into the electrolytic iron. By subsequently annealing in a vacuum the two constituents may then be removed to a great extent.

It is also possible, however, to have non-equivalent quantities of carbon and oxygen in the iron. If the carbon is present in excess, the plates are first annealed in an oxidizing atmosphere in order to obtain a corresponding oxygen content, and are subsequently annealed in a vacuum. A process similar to the tempering of cast iron will then take place. The carbon reacts with the iron oxide but the reaction will be correspondingly slower since the diffusion conditions are considerably less favorable.

If the oxygen is in excess, a corresponding quantity of carbon may be added to the iron by carbonizing, for example by annealing in an atmosphere of carbon-dioxide, and on

subsequent annealing in a vacuum the mutual reaction of the carbon with the oxygen is initiated. In accordance with the previously described process however a longer annealing period will here be required.

What I claim as new and desire to secure by Letters Patent of the United States, is:

1. The process of producing magnetic sheet material from electrolytic iron which comprises equalizing the oxygen and carbon content of the iron during electrolysis and thereafter annealing the iron in a vacuum.

2. The process of producing magnetic sheet material from electrolytic iron which comprises employing during electrolysis anodes having a carbon content substantially equivalent to the oxygen content of the iron and then annealing the electrolytic iron in a vacuum.

3. The process of producing magnetic sheet material from electrolytic iron which comprises varying the carbon content of the iron by adding organic material to the electrolyte in sufficient quantity to equalize the carbon and oxygen content of the iron and then annealing the electrolytic iron in a vacuum.

4. The process of producing magnetic sheet material from electrolytic iron which comprises varying the acidity of the electrolyte thereby to balance the carbon and oxygen content of the iron and then annealing the electrolytic material in a vacuum.

5. The process of producing magnetic sheet material from electrolytic iron which comprises annealing the electrolytic material in a carbonizing atmosphere thereby to equalize the carbon and oxygen content of the iron, and thereafter annealing the iron in a vacuum.

In witness whereof, I have hereunto set my hand.

FRANZ PAWLEK