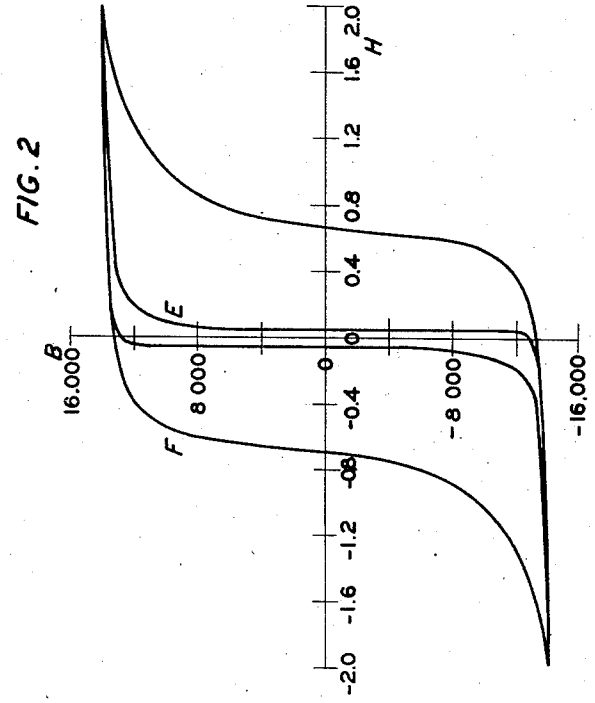
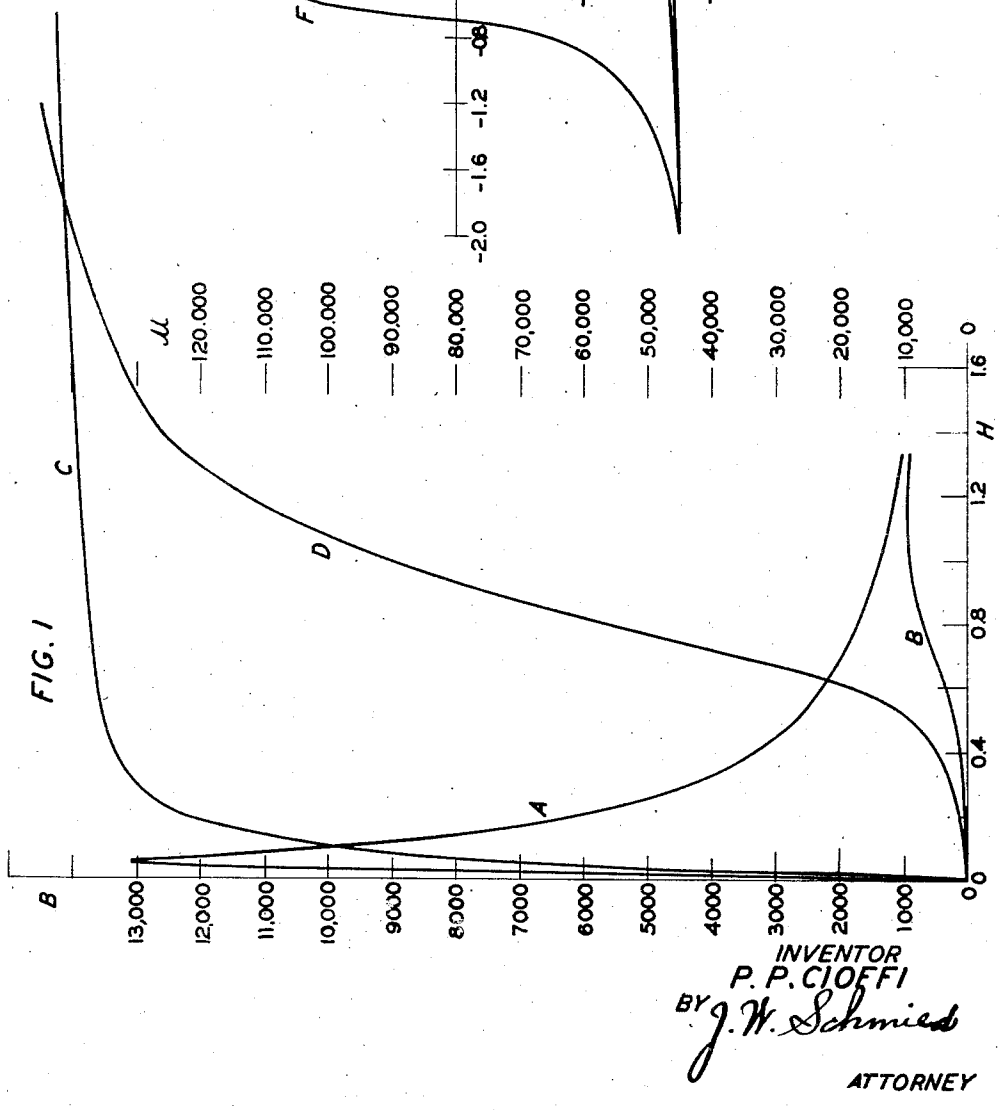
July 12, 1932. P. P. CIOFFI 1,866,925

MAGNETIC MATERIAL

Filed July 31, 1930





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UNITED STATES PATENT OFFICE

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MAGNETIC MATERIAL

Application filed July 31, 1930. Serial No. 471,988.

The present invention relates to heat treatments for developing desired magnetic properties in magnetic materials.

An object of the invention is to develop high permeability, low coercive force, low hysteresis loss and other desirable properties in magnetic materials.

Another object of the invention is to produce iron having higher maximum permeability than that of iron heretofore known.

A further object of the invention is to enhance the efficiency of hydrogenous atmospheres as annealing media.

In applicant's former application Serial No. 325,883, filed December 13, 1928 there are disclosed methods of securing desirable magnetic properties in magnetic materials by heat treating these material in hydrogenous atmospheres.

It has been found that a further improvement can be effected in the magnetic properties of such materials as are mentioned in the former application and in other materials, by including a certain amount of moisture in the hydrogenous atmospheres in which the magnetic materials are treated. For instance, it has been possible to obtain a maximum permeability of 137,000 with iron as compared with the highest value of 98,000 previously reported. See page 225 of the "Transactions of the American Electrochemical Society", vol. 56, 1929.

The present specification is to be read in the light of the specification of the said former application which is specifically, by reference, made a part of the present disclosure.

Specific examples of treatments and the results accomplished thereby will now be described with reference to the accompanying drawing and tables.

For the purpose of the following detailed description the term "moist" hydrogen indicates a hydrogenous atmosphere consisting chiefly or partly of hydrogen gas saturated with water vapor at room temperature. In the tests hereinafter described use has been made of various types of samples differing both with respect to their shape and with respect to their composition. The tests were made with toroidal specimens which were cylinders of 1⅛ inches outer diameter and ¼ inch high; those labeled "thick" had an inner diameter of 15/16 inches, those labeled "thin" had an inner diameter of 1 1/16 inches.

Figure 1 shows a curve A depicting the variation of the permeability of a thick ring of Armco iron heated at 1500° C. for 12 hours in moist hydrogen. This sample had been cooled from 1500° C. to 880° C. in about one hour, cooled to room temperature in about 35 minutes and subsequently reannealed at 880° C. for two hours and cooled to room temperature in about 35 minutes. For purpose of comparison a curve B is given which shows the variation of the permeability of ordinary annealed iron. It is seen that the sample treated in moist hydrogen had acquired an initial permeability of 6,000 and a maximum permeability of 131,000 at a magnetizing force at 0.044 gauss. Curves C and D, respectively, show the variation of the magnetic induction for varying magnetizing fields for the same sample and for ordinary annealed iron.

Curves E and F of Fig. 2 depict hysteresis loops for the same sample and for ordinary annealed iron respectively, for a maximum induction of 14,000 gauss. The hysteresis loss of the sample heat treated in moist hydrogen was found to be 300 ergs per cubic centimeter per cycle for a maximum induction of 14,000 gauss and its coercive force was 0.05 gauss for the same maximum induction of 14,000 gauss. At the same maximum induction ordinary annealed iron has a hysteresis loss of 3,250 ergs per cubic centimeter and a coercive force of 0.67 gauss.

The subjoined table contains data on other samples of iron of the same composition and results obtained from their heat treatments. In this table column 1 contains an arbitrary number identifying the sample; column 2 designates the shape of the sample in accordance with the definition of shape previously given; column 3 indicates on the first line the temperature and duration of the first annealing, on the second line the temperature and duration of the second annealing; column 4 the nature of the atmosphere used;

column 5 the initial permeability μ_o; column 6 the maximum permeability μ_m, and column 7 the magnetizing force at which the maximum permeability was determined.

The cooling rate used in the heat treatment of these samples was fairly uniform and consisted first in cooling from 1500° C. to 880° C. in about one hour from 880 C. to room temperature in about 35 minutes; second in cooling after the reannealing treatment at 880° C. for the periods of time indicated for each sample, from 880° C. to room temperature in about 35 minutes. A reannealing temperature of 880° C. is mentioned because the reannealing treatment may advantageously be somewhat below the alpha-gamma phase transformation point which for Armco iron is at a temperature between 907 and 910° C. The table follows:—

No.	Shape	Heat treatment	Atmosphere	μ_o	μ_m	Magnetizing force in gauss for μ_m
1	Thick ring	1500° C. 6 hrs. 880° C. 2 hrs.	Dry H_2 Dry H_2	4000	57000	.168
2	Thin ring	1500° C. 6 hrs. 880° C. 2 hrs.	Dry H_2 Dry H_2	8000	56000	.138
3	Thin ring	1500° C. 12 hrs. 880° C. 1 hr.	Dry H_2 Dry H_2	3500	39000	.247
4	Thin ring	1500° C. 6 hrs. 880° C. 1 hr.	Trace H_2O, H_2 Trace H_2O, H_2	3000	37000	.218
5	Thick ring	1500° C. 6 hrs. 880° C. 1 hr.	Dry H_2 Dry H_2	2200	39000	.236
6	Thick ring	1500° C. 6 hrs. 880° C. 1 hr.	Moist H_2 Moist H_2	2000	42000	.201
7	Thick ring	1500° C. 6 hrs. 880° C. 1 hr.	Dry H_2 Dry H_2	4000	59000	.108
8	Thick ring	1500° C. 6 hrs. 880° C. 1 hr.	Moist H_2 Moist H_2	2000	48500	.187
9	Thin ring	1500° C. 12 hrs. 880° C. 2 hrs.	Moist H_2 Moist H_2	1500	55000	.193
10	Thick ring	1500° C. 18 hrs. 880° C. 2 hrs.	Moist H_2 Moist H_2	4000	137000	.044
11	Thick ring	1500° C. 12 hrs. 880° C. 2 hrs.	Moist H_2 Moist H_2	3000	101000	.0795
12	Thin ring	1500° C. 3 hrs. 880° C. 1 hr.	Moist H_2 Moist H_2	1600	41000	.214
13	Thick ring	1500° C. 3 hrs. 880° C. 1 hr.	Moist H_2 Moist H_2	1600	28500	.20

There is evidence that to a certain extent with moist hydrogen the longer the heat treatment and the thicker the specimen the higher will be the maximum permeability. It appears, that the use of a moist hydrogen atmosphere is particularly advantageous where the article to be treated is of considerable thickness.

In another series of experiments a thick sample of Armco iron was first heated in moist hydrogen at a temperature of about 1500° C. for 12 hours and then cooled from 1500 to about 880° C. in eight minutes, the cooling from 880° C. to room temperature taking about 25 minutes. The iron was found to have an initial permeability of 1950 and a maximum permeability of 25,000. The same sample was then reannealed at about 880° C. for additional periods of time, the annealing being interrupted after these periods for enabling the taking of magnetic measurements. All the annealing was effected in moist hydrogen and each annealing period was followed by cooling to room temperature at a rate of about 20° C. per minute. The following table gives the data found after each consecutive additional heat treatment:

Duration of maintenance at 880° C.	Pressure of moist hydrogen	Initial permeability	Maximum permeability
15 minutes total	1 atmosphere	1,800	38,000
30 minutes total	1 atmosphere	1,800	46,000
2 hours total	1 atmosphere	2,500	56,000
4 hours total	1 atmosphere	2,500	63,000
6 hours total	1 atmosphere	4,000	87,500
8 hours total	1 atmosphere	4,000	112,000
10 hours total	1 atmosphere	4,000	125,000

In another test Armco iron was melted in moist hydrogen, 0.10% of calcium boride was added to facilitate subsequent cold working after solidification, the melt was kept liquid for 16 hours and cooled to room temperature in 4 hours. Then the material was cold rolled from ¾ inch to ¼ inch and a thin ring specimen was prepared. This specimen was heat treated at 880° C. in moist hydrogen for 6 hours and cooled to room temperature at an average cooling rate of about 20° C. per minute. It had an initial permeability of 1600 and a maximum permeability of 25,400 at a maximum force of 0.36 gauss.

Ordinary cold rolled steel containing about 0.1% carbon was heat treated in moist hydrogen for 12 hours at a temperature of 1500° C. and cooled to room temperature in about one hour. It was found to have an initial permeability of 1400 and a maximum permeability of 33,000 at a magnetizing force of 0.23 gauss.

An iron-cobalt alloy comprising about 50% iron and 50% cobalt was heat treated in moist hydrogen at 1440° C. for 12 hours, cooled to room temperature at an average rate of about 40° C. per minute, reannealed at 940° C. in

moist hydrogen for 2 hours and slowly cooled. Such an alloy has a phase transformation point at about 980° C. It exhibited an initial permeability of 600 and a maximum permeability of 7400, whereas an alloy of the same composition, if heat treated in the ordinary manner exhibits an initial permeability of only 600 and a maximum permeability of only 4000.

"Iron" as used herein refers to the metallic element in a relatively pure form and does not include alloys of iron or compositions having other elements added thereto as essential constituents.

What is claimed is:

1. The method of increasing the efficiency of hydrogenous atmospheres as annealing media for magnetic materials at temperatures above about 1000° C., which method comprises adding water vapor to the hydrogenous atmosphere.

2. The method of preparing magnetic materials with desirable magnetic properties in desired shape for use, which method comprises (*a*) heating the material in hydrogen, (*b*) cooling the material to a much lower temperature, (*c*) working the material mechanically into the desired shape, and (*d*) reheating the material to a temperature below the alpha-gamma transformation point if the material has such a point above room temperature, said method being further characterized in this that the hydrogen in which the material is heat treated contains water vapor.

3. A method of improving the magnetic properties of magnetic materials which comprises heating the materials at a temperature above 1000° C. in a hydrogenous atmosphere containing water vapor.

4. A method of improving the magnetic properties of magnetic materials which comprises heating the materials at temperatures close to their melting points in an atmosphere containing hydrogen and water vapor.

5. A method of improving the magnetic properties of magnetic materials which comprises heating the materials at a temperature above 1000° C. in a hydrogenous atmosphere saturated with water vapor at room temperature.

6. As a new article of manufacture, magnetic iron having a maximum permeability over 100,000.

7. A magnetic material comprising at least 95% iron having a coercive force of 0.05 gauss after an induced field of at least 14,000 gauss.

8. The method of treating a material composed principally of ferromagnetic elements, which comprises heating the material in a hydrogenous atmosphere at a temperature near its melting point, cooling said material to a temperature below its phase transformation point, reheating the material at a temperature near its phase transformation point, and cooling the material to room temperature, characterized in this that at least one of said heat treatments takes place in an atmosphere containing hydrogen and water vapor.

9. The method of treating a material comprising at least 95% iron, which comprises heating the material in a hydrogenous atmosphere at a temperature near its melting point, cooling said material to about 880° C., heating the material at a temperature of about 880° C., and cooling the material to room temperature, characterized in this that at least one of said treatments takes place in an atmosphere containing hydrogen and water vapor.

In witness whereof, I hereunto subscribe my name this 30th day of July, 1930.

PAUL P. CIOFFI.