

March 8, 1938.

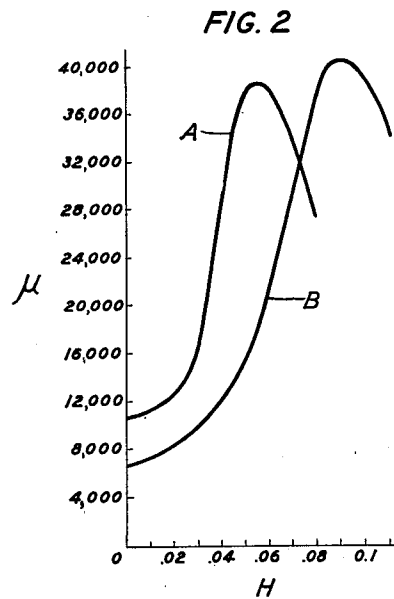
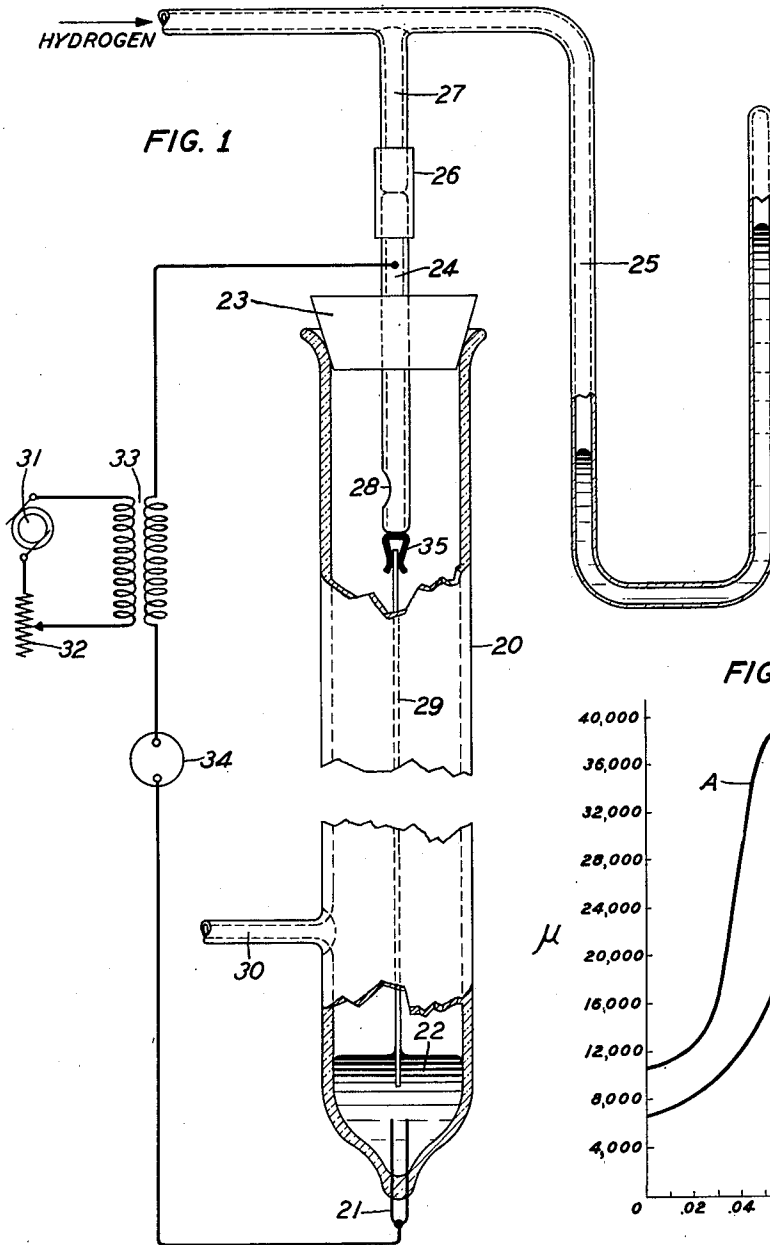
P. P. CIOFFI

2,110,569

MAGNETIC MATERIAL

Filed Aug. 19, 1932

3 Sheets-Sheet 1



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March 8, 1938.

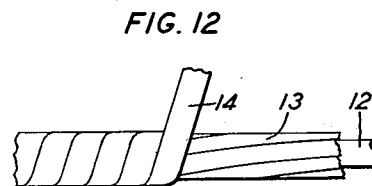
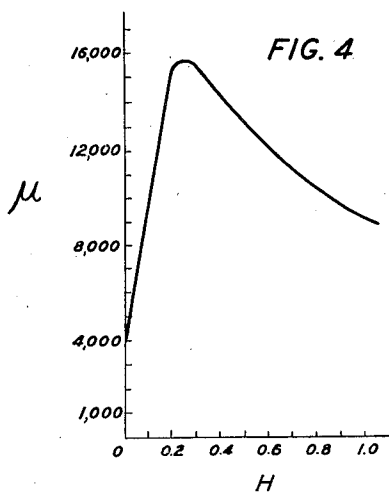
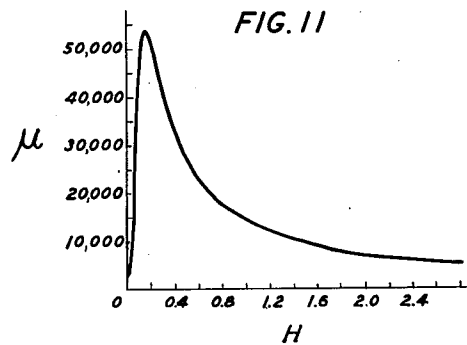
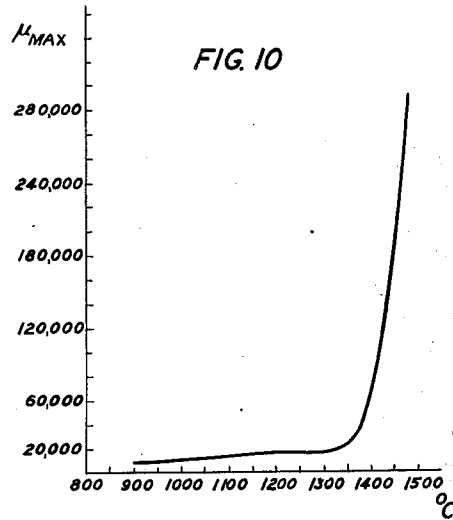
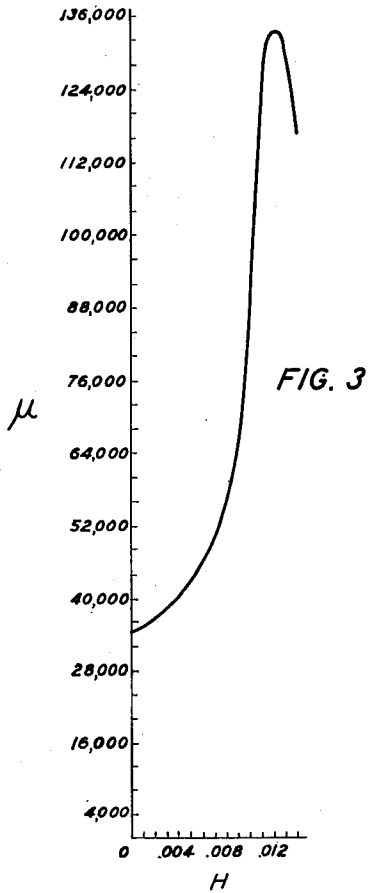
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MAGNETIC MATERIAL

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3 Sheets-Sheet 2



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2,110,569

MAGNETIC MATERIAL

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3 Sheets-Sheet 3

FIG. 5

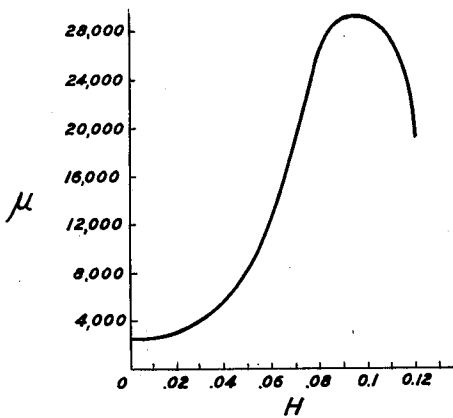


FIG. 6

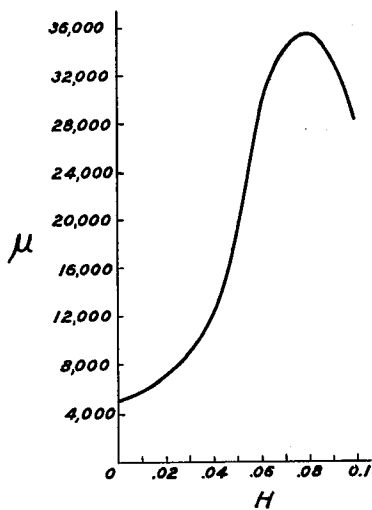


FIG. 8

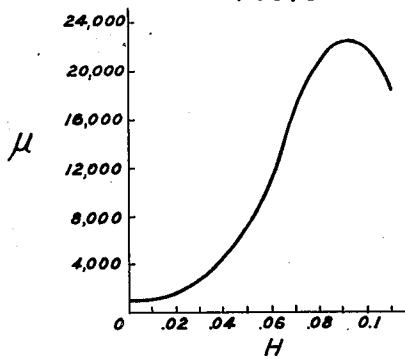


FIG. 7

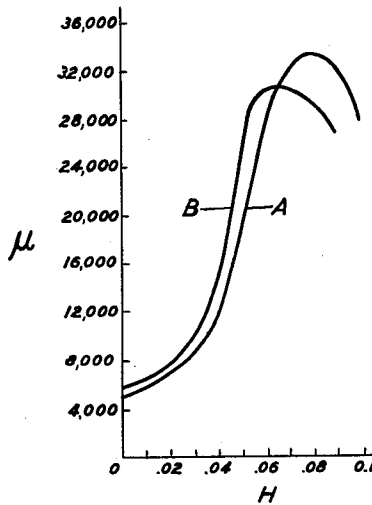
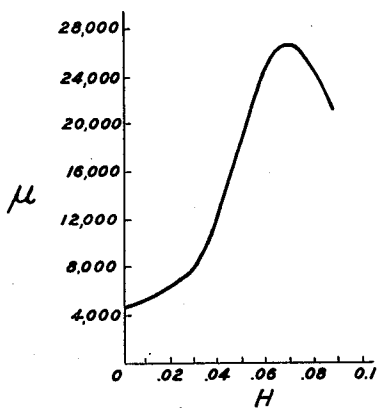


FIG. 9



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UNITED STATES PATENT OFFICE

2,110,569

MAGNETIC MATERIAL

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Application August 19, 1932, Serial No. 629,507

13 Claims. (Cl. 148—16)

The present invention relates to magnetic materials and to heat treatments for developing desired magnetic properties in such materials. It has a wide field of usefulness and is of special interest in those cases in which the impressed magnetizing forces are small, such as in electric signaling systems and apparatus.

A general object of the invention is to improve the magnetic properties of magnetic materials.

Another object is to produce magnetic materials having high initial and maximum magnetic permeabilities, low coercive force, low hysteresis loss, and high resistivity.

A further object of the invention is to produce iron having higher initial and maximum permeability and lower coercive force and hysteresis loss, than iron hitherto produced.

A particular object is to facilitate the production of magnetic iron having such improved properties when in final form ready for use.

In applicant's former application Serial No. 325,883, filed December 13, 1928 there are disclosed methods of securing desirable properties in magnetic materials by heat treating them in hydrogenous atmospheres. The present application is a continuation in part of the former application.

Ordinary Armco iron heat treated by suitable ordinary methods has an initial permeability of about 250, a maximum permeability of from 6,000 to 8,000, a coercive force of about 1.5 gauss for a maximum induction of 12,000 gauss, and a hysteresis loss of about 5,000 ergs per cubic centimeter per cycle for a maximum induction of 12,000 gauss.

Important improvements have been made hitherto in the magnetic properties of ordinary iron and iron alloyed with other magnetic materials. For instance, maximum permeabilities of 19,000 and 42,000, coercive forces of 0.29 and 0.15 gauss for maximum inductions of 15,000 gauss, and hysteresis losses of 916 and 1,025 ergs per cubic centimeter per cycle for maximum inductions of 15,000 gauss have heretofore been stated to have been obtained by others with pure iron and iron-silicon alloys respectively, when prepared in a vacuum furnace and annealed in vacuum or under non-oxidizing conditions at temperatures of about 1100° C. These improvements were achieved by reducing the impurities,

particularly the carbon, to a minimum. U. S. Patent No. 1,358,810, November 16, 1920 to T. D. Yensen describes annealing an iron-silicon alloy containing 4% silicon at 1100° C. under slightly oxidizing conditions, resulting in a maximum permeability of over 40,000 and in a hysteresis loss of less than 250 ergs per cubic centimeter per cycle for a maximum induction of 10,000. In U. S. Patent 1,110,010, September 8, 1914, W. E. Ruder discloses heating silicon steel in hydrogen at temperatures of from 1,000° to 1,325° C., thereby reducing the hysteresis loss to 0.0060 watt per pound of steel per cycle (about 1,000 ergs per cubic centimeter per cycle) at a magnetic density of $B=10,000$ gauss. In U. S. Patent 1,156,496, October 12, 1915, the same author discloses short-time annealing of silicon steel sheets in hydrogen, nitrogen or other gas serving to protect the material from oxidation, at temperatures from 1300° to 1500° C., for periods of 10 seconds to 5 minutes, so that the sheets are heated to a temperature of at least 1000° C., for the purpose of improving the magnetic properties.

The disclosures mentioned are concerned either with iron melted in vacuum (in especially designed furnaces) or with alloys of iron and silicon; their chief object is to produce improved magnetic materials to be subjected to relatively large magnetizing forces, such as are encountered in power systems and apparatus; consequently, they are not concerned with initial permeabilities and permeabilities at very weak magnetizing forces.

In contra-distinction to this, the objects of the present invention are not accomplished by the treatments mentioned and the materials produced thereby, since one of the most useful fields of application of the materials treated by the methods of this invention are electro-magnetic systems and apparatus which are to be subjected to low magnetizing forces, as in signaling systems, for instance. Also the initial permeability and permeabilities at low magnetizing forces of materials treated in accordance with the present invention are greater and hysteresis loss and coercive forces are lower than those produced by the treatments mentioned. At the same time the maximum permeability is greatly increased with

a corresponding diminution in hysteresis loss and coercive force at high inductions.

The publications made by previous workers indicate that they have not succeeded in carrying their researches to the point of achieving the technical advances made possible by the methods of the present invention. As against a maximum value of initial permeability of 1,700 for ordinary iron, in accordance with the best example claimed in a very special case in the prior art the methods herein described permit values as high as 10,400 and maximum permeabilities as high as 280,000 to be obtained.

Generally stated:—

These results are accomplished by heating the magnetic materials in an atmosphere of hydrogen of ordinary purity under pressures ranging from a fraction of one atmosphere to over twelve atmospheres at temperatures approximating the melting point. In the case of iron, or generally, any magnetic material having a phase transformation point, the treatment subsequent to the exposure to the high temperature may be in accordance with either one of the following variations:—

(1) The material is cooled slowly to room temperature;

(2) The first treatment is followed by cooling to 930° C., either fast or slowly, then cooling slowly to 880° C. and then at any cooling rate to room temperature;

(3) The material is cooled either fast or slowly to 880° C., maintained at 880° C. for a period of time, and cooled to room temperature at any desired rate.

(4) In the case of iron and certain other magnetic materials having a phase transformation point the material may be cooled to any desired temperature below 880° C., i. e., room temperature, and then mechanically worked, if desired, then heated to and maintained just above the phase transformation point for a period of time and cooled at any desired rate.

(5) Alloys of iron having no phase transformation point may generally be cooled from the original treatment at any desired rate.

An important feature of this invention is the attainment of high initial permeability in magnetic materials in final form and shape for use, as, for example, in iron or other materials used for continuous loading of conductors while the iron is in situ upon the conductor. Values of 2,000 and more may be readily obtained in the case of iron upon a conductor.

Unless otherwise specified, the specimens discussed herein were treated in wire form 0.040 inch in diameter and all hydrogen gas pressures are given in millimeters of mercury absolute pressure.

In the case of iron the desired properties may be produced by first heating the iron in hydrogen at a temperature between about 1100° C. and the melting point after which the iron may be further dealt with in several ways which will be discussed hereinafter.

After being heated as above for a sufficient length of time, usually one-half hour or more, the iron is cooled down to and through the alpha-gamma transformation point (approximately at 900° C.), and then reannealed at a somewhat lower temperature, about 880° C., either in a hydrogenous atmosphere or not and cooled at any suitable rate. The lower temperature anneal is given only when strains are introduced

during cooling from the first temperature of heat treatment.

Prior to reheating, the iron may be rolled, drawn, punched, or otherwise worked mechanically into any suitable form without impairing its magnetic properties after the reannealing process. By this process iron with an initial permeability up to 10,400 and a coercivity as low as 0.025 gauss has been produced.

The following table gives data relating to some instances of treatment of iron in accordance with this method:

Specimen	Temp. of first anneal	Time of anneal	Hydrogen pressure	Initial permeability	Maximum permeability
	° C.		mm.		
(a)-----	1,500	90 min.	45	10,400	39,000
(b)-----	1,500	30 min.	760	8,000	35,000
(c)-----	1,500	8½ hrs.	760	9,100	49,000
(d)-----	1,400	12 hrs.	760	6,700	48,000
(e)-----	1,300	13 hrs.	760	1,800	19,800
(f)-----	1,200	41¼ hrs.	1,520	1,900	18,200

An alternative method is to interrupt the cooling at about 880° C. and maintain this temperature for a time before cooling. In this case no reheating is necessary.

Another method is to heat the iron in a hydrogenous atmosphere as before and cool slowly, particularly between about 920° C. and 890° C.

Tests were made to determine how small amounts of impurities such as sulphur, iron chloride, iron oxide, aluminum oxide, silicon, magnesium, manganese, etc. affect the magnetic properties of iron.

Examples of specific cases are as follows:

Composition before treatment	Temp. of first heat treatment	Time of treatment	Hydrogen pressure	μ_0	μ_{max}
	° C.	min.	mm.		
Fe purified with 0.1% calcium boride.....	1500	30	760	8500	53,500
Fe+0.1% S.....	1500	30	760	2000	29,000
Fe+0.1% Al ₂ O ₃	1500	30	760	7000	44,000
Fe+0.1 FeCl ₃	1500	30	760	7500	41,000
Fe+0.1% Si.....	1500	30	760	5500	38,300
Fe+0.1 Si+0.1 Al.....	1500	30	760	4700	38,200
Fe+0.5 Si.....	1500	30	760	3400	31,000
Fe+0.1 Mg.....	1500	30	760	2800	27,000
Fe+0.3% Mn.....	1500	30	760	2660	24,600
Fe+1.0% Mo.....	1500	30	760	5000	27,000
Fe+0.5 Fe ₂ O ₃	1500	30	760	5200	35,600

Each specimen in the foregoing table was cooled in hydrogen and subsequently annealed at 880° C. in hydrogen for 30 minutes.

In one aspect the invention involves iron in a substantially pure condition. In another aspect, however, the invention involves certain alloys of iron, which are improved in their magnetic and/or electrical properties, such as initial and maximum permeabilities, hysteresis loss, coercive force, electrical resistance (resistivity) to a greater or lesser extent, by similar treatment; hence the invention involves treatment of iron-silicon alloys, iron-molybdenum alloys, iron-nickel alloys, iron-cobalt alloys, iron-molybdenum-nickel alloys and certain others.

However, it has been found that an increase in the initial and maximum permeabilities and in the resistivity may be produced in alloys of iron which have no alpha-gamma transformation point with only a single high temperature heat treatment followed by slow or rapid cooling, after heating in a hydrogenous atmosphere.

Examples of specific cases are as follows:

5	Composition	Tem- pera- ture	Time	Hydro- gen pres- sure	μ_0	μ_{max}	Resis- tivity in mi- crohms per cubic cm.
	79½ parts Ni.....	° C.		mm.			
10	16½ parts Fe.....	1400	30 min.	760	32,500	134,000	22.00
	4 parts Mo.....	1400	30 min.	760	3,200	44,000	35.00
	50% Ni, 50% Fe.....	1400	5 hrs.	760	10,000	48,000	35.00
	50% Ni, 50% Fe.....	1400	30 min.	760	3,000	93,000	18.3
	81% Ni, 19% Fe.....	1400	30 min.	760	7,500	83,000	15.6
15	78½% Ni.....	1400	30 min.	760	2,020	11,500	30.00
	21½% Fe.....	1400	30 min.	760	6,000	30,500	19.00
	96¼% Fe, 8% Mo.....	1400	30 min.	760	4,000	16,000	52.00
	96¼% Fe.....	1400	30 min.	760	1,000	22,000	19.00
	33¼% Mo.....	1410	30 min.	760	1,300	6,000	77.00
	96% Fe, 4% Si.....	1380	15 hrs.	760			
20	45% Ni.....						
	30% Fe.....						
	25% Co.....						
	45% Ni.....						
	22% Fe.....						
	25% Co.....						
	7% Mo.....						
	1% Mn.....						

The nickel-iron-cobalt compositions were cooled slowly from 1380° C. to 500° C. in three hours, and from 500° C. to 400° C. in two hours.

It is within the scope of the invention to melt the iron in hydrogen or other hydrogenous atmospheres. The iron thus melted may be cooled and cold worked as desired and then reannealed at 880° C. to restore the improved magnetic properties.

In a particular experiment, iron was melted in hydrogen at a pressure of one atmosphere, then cooled slowly through the freezing point, and the temperature maintained for a period of about one hour just under the freezing point with the hydrogen pressure unchanged; the iron was then rapidly cooled to room temperature and cold drawn to wire.

Hard drawn wire thus made from hydrogen-melted iron may be treated in various ways with varying results. For instance, it may be heat treated at 1500° C. for 30 minutes followed by a reannealing treatment of the cold worked material at about 1400° C. This combined treatment resulted in values for initial and maximum permeabilities of 3000 and 29,000, respectively. Then the time of treatment at 1400° was increased to 3 hours, resulting in values for initial and maximum permeabilities of 5000 and 25,000, respectively. With a single treatment at 880° C. for six hours, the initial and maximum permeabilities were 1500 and 13,600, respectively. This indicates that there is a decided advantage gained if the hydrogenization of iron is commenced in the melt, because it is then possible to obtain improved magnetic characteristics in the cold worked metal by reannealing at considerably lower temperatures.

In another experiment the iron was melted in hydrogen at atmospheric pressure. The pressure was then reduced to between 0.5 and 1 mm. of mercury and the iron was allowed to solidify and cool to room temperature. The ingot was cold worked from ¾" thickness to ¼" plate. Toroidal rings cut from this plate were annealed at 880° C. for 6 hours and were found to have an initial permeability of 2500 and a maximum permeability of 52,000.

In a further experiment the pressure was not changed but was maintained at one atmosphere and after solidification and cooling to room temperature the ingot was cold rolled to ¼" plate. The toroidal specimens cut from this plate were

annealed in hydrogen at 880° C. for 6 hours. Their initial permeability was 5000 and their maximum permeability 56,000.

In another experiment Armco iron was melted and allowed to solidify in an ingot mold in a hydrogenous atmosphere of atmospheric pressure. The ingot was then heated in air at 850° C. and hot rolled to a plate of ¼ inch thickness. Toroidal specimens were then cut from the plate and heated at 880° C. in hydrogen for 4 hours. They were found to have initial permeabilities of 5400 and maximum permeabilities of 40,000.

When melting in hydrogen it is desirable, in order to obtain sound ingots, to cool the metal slowly through the solidifying temperature, thus permitting it to give up its absorbed hydrogen. Alternately, by evacuating the furnace atmosphere prior to solidifying the absorbed hydrogen is liberated and a sound ingot may be obtained by solidifying the metal more quickly.

Another method of producing sound ingots from hydrogen melts is to substitute for the hydrogen, after the hydrogen treatment, a gas which is insoluble in the molten metal and has little or no chemical affinity toward it, such as helium, for instance. Thus the partial pressure of hydrogen is reduced to zero and the hydrogen absorbed in the metal is liberated, permitting the metal to be solidified quickly into a sound ingot. After cold rolling an ingot made in this manner to ¼ inch plate, toroidal specimens cut from this plate were annealed in hydrogen at 880° C. and were found to have an initial permeability of 4000 and a maximum permeability of 45,000.

In the operation of the helium substitution method for producing sound ingots, it is not necessary that the partial pressure of hydrogen be reduced to zero; it is only necessary to reduce the partial hydrogen pressure to the point where there is no liberation of absorbed hydrogen on solidifying. This may be accomplished either by partially evacuating the furnace or by using a mixture of hydrogen and helium. Thus sound ingots have been produced by using a gaseous mixture of hydrogen and helium in the proportion of 6.6% hydrogen and 93.4% helium, corresponding to partial pressures respectively of 50 mm. and 710 mm. of mercury.

In one practical embodiment of this aspect of the invention, the magnetic material is melted in a crucible or furnace having the form of an inverted L in a hydrogenous atmosphere. The upper branch of the inverted L-shaped crucible is formed to constitute the mold into which the material is to be cast. After the melting is completed, the furnace is connected to a receptacle containing helium under pressure. The helium gas enters the furnace and sweeps out the hydrogenous atmosphere. The stream of helium gas may also be caused to bubble through the molten metal bath, for instance by introducing it into the bath by means of heat resistant tubing. The crucible is then tilted and the purified metal cast into the mold without coming in contact with any atmosphere but helium.

It has been found that when iron is melted in hydrogen at atmospheric pressure and then cold worked, reheating for from two to five hours at about 880° C. in hydrogen produces an initial permeability of 5000 and a maximum permeability of 58,000. Higher temperatures of heat treatment produce further improvements in magnetic characteristics.

More specific methods of treating particular

magnetic materials and data relating to the results attained will now be described with reference to the accompanying drawings. Legends on these drawings and numerical values given in the text relative to magnetizing forces, coercive forces and flux densities are in c. g. s. units and hysteresis losses are in ergs per cubic centimeter per cycle.

Fig. 1 of the drawings schematically illustrates an apparatus employed for quickly and conveniently heat treating magnetic materials in accordance with this invention.

Figs. 2 to 9 and 11 depict permeability vs. magnetizing force curves for various materials produced by different treatments.

Fig. 10 shows the relation which exists between the temperature of the first heat treatment and the maximum permeability.

Fig. 12 shows one example of application of material produced by the methods of this invention, namely its use as loading material inductively associated with a signaling conductor.

In Fig. 1 glass cylinder 20 has a conductive wire 21 sealed in its lower end, whereas its upper end is closed by a removable air-tight stopper 23 through which passes brass tube 24. Tube 24 which is open at 28 for the purpose of admitting gas into cylinder 20 is connected both to a supply of hydrogen (not shown) and to a pressure indicating device 25. Rubber sleeve 26 is provided for the purpose of insuring a tight connection between tube 24 and hydrogen supply tube 27. The upper end of the specimen 29 of the material to be heat treated is attached by means of clip 35 to brass tube 24, while its lower end dips into mercury 22. At a point located some distance above the level of the mercury the glass tube is connected by means of tube 30 to a vacuum pump (not shown), for evacuating the air from cylinder 20. A source of alternating current 31 is provided for heating the sample; it is connected through resistance 32 to the primary of transformer 33, the secondary of which is connected to brass tube 24 and conductive wire 21 as shown. The intensity of the heating current may be measured by ammeter 34 and varied by resistance 32. Direct current for heating has also been used with identical results.

After the sample has been placed into the heat treating tube as described, heating current is passed through it by means of adjusting resistance 32, and hydrogen or its equivalent is simultaneously admitted through tube 27 and the pressure regulated by means of the vacuum pump acting upon tube 30 and the rate of inflow of gas through tube 27 so that the material is heated in the hydrogenous atmosphere to a desired temperature.

In one specific case, a specimen of Armco iron having the form of a wire about 1 millimeter in diameter and about 60 centimeters long was inserted into the heat treating vessel and hydrogen admitted and regulated at a pressure of 45 millimeters of mercury. Simultaneously the specimen was heated to a temperature of about 1500° C. for about 35 minutes; then the heating current was cut off and the specimen allowed to cool in its receptacle at an average rate of about 750° C. per minute until it reached room temperature. Magnetic measurements indicated that it had an initial permeability of 600 and a maximum permeability of 7,500. The specimen was then reinserted into the heating vessel and again heated in hydrogen at the same pressure to 880° C. kept at this temperature for 30

minutes, and then the heating current so adjusted that it cooled at an average rate of 180° C. per minute. Curve A of Fig. 2 illustrates the variation of permeability with small varying magnetizing forces for this sample. The initial permeability was 10,400, the maximum permeability 39,000, the coercive force 0.10 gauss for a maximum induction of 13,000 gauss and the hysteresis loss 470 ergs per cubic centimeter per cycle. Curve B of the same figure graphically depicts the initial and maximum permeabilities of the same material treated identically except that the hydrogen was at atmospheric pressure. In this case the initial permeability was 6500 and the maximum permeability was 41,000.

An alternative method which yielded similar good results consisted in heating and cooling the sample as mentioned in the preceding paragraph, but interrupting the first cooling operation at about 880° C., maintaining this temperature for about 30 minutes, and then cooling the room temperature at an average rate of about 180° C. per minute.

The curve of Fig. 3 shows the permeability variation with increasing magnetizing forces of an alloy containing 4% molybdenum, 79.5% nickel and the remainder iron when given a single treatment consisting in heating to about 1400° C. for 30 minutes in hydrogen at atmospheric pressure and cooling to room temperature at an average rate of about 50° C. per minute. It exhibited an initial permeability of 33,000 and a maximum permeability of 134,000 whereas the same material heat treated by pot annealing at 1100° C. followed by very slow cooling has an initial permeability of about 20,000 and a maximum permeability of about 75,000.

The curve of Fig. 4 pertains to an iron-silicon alloy containing approximately 4% silicon, heated to about 1410° C. (the melting point is at about 1450° C.) for about 130 minutes in hydrogen at a pressure of 45 millimeters and cooled at an average rate of about 1000° C. per minute to room temperature. This treatment produced an initial permeability of 4000, a maximum permeability of about 15,500, a hysteresis loss of only 1.97 ergs per cubic centimeter per cycle for a maximum induction of about 550 gauss and a resistivity of 52 microhms per cubic centimeter.

The curve of Fig. 5 illustrates the variation in permeability with increasing magnetizing force of a sample of Armco iron containing 0.3% manganese, heated to 1500° C. in hydrogen at atmospheric pressure and cooled to room temperature at an average rate of 750° C. It was then reannealed at 880° C. for one hour and cooled at an average rate of 10° C. a minute. It had an initial permeability of 2,600 and a maximum permeability of 29,000.

The curve of Fig. 6 shows the variation in permeability with varying magnetizing forces of a sample of Armco iron containing 0.5% iron oxide, Fe₂O₃. This sample was heat treated at 1500° C. in hydrogen at atmospheric pressure for 30 minutes, then cooled to room temperature at an average rate of 750° C. per minute, reheated in hydrogen at an average rate of 180° C. It exhibited an initial permeability of 5,200 and a maximum permeability of 36,000.

Curves A and B of Fig. 7 are permeability vs. magnetizing force curves of samples of Armco iron containing respectively 1% and 3.75% molybdenum. Both specimens were heated in hydrogen at atmospheric pressure at about 1400° C. for 30 minutes and cooled at an average rate of 800°

C. per minute. Specimen A exhibited a resistivity of 14.3 and specimen B a resistivity of 19 microhms per cubic centimeter.

The curve of Fig. 8 depicts the variation in permeability with varying magnetizing forces for a sample of Armco iron heated for 30 minutes to about 1500° C. in an unexplosive mixture of about 30% hydrogen and about 70% nitrogen at atmospheric pressure, cooled to room temperature at a rate of 800° C. per minute, reheated to 380° C. for about 60 minutes and cooled at a rate of about 10° C. per minute to room temperature. The material had an initial permeability of 900 and a maximum permeability of 22,300. Mixtures of 50% and 10% hydrogen, remainder nitrogen, have been tried with practically the same result. When using gaseous hydrogen and nitrogen together as thus described, it is known that some of the gases combine to form ammonia.

The curve of Fig. 9 illustrates the permeability of iron melted in hydrogen at atmospheric pressure, cooled in hydrogen at an average rate of about 150° C. per minute to room temperature, cold worked to wire of about 1 millimeter diameter, annealed in hydrogen at atmospheric pressure at 1500° C. for 30 minutes, cooled at a rate of about 750° C. per minute to room temperature, reheated in hydrogen at the same pressure at 380° C. and cooled at an average rate of about 180° C. per minute. The iron had an initial permeability of 4700 and a maximum permeability of 27,000.

The curve of Fig. 10 shows the relation which exists between the temperature of the first heat treatment and the maximum permeability. It may be noted that with Armco iron the hydrogen treatment does not produce any appreciable improvement in the maximum permeability until the temperature is raised to about 1350° C.

Fig. 11 illustrates the variation of permeability with magnetizing forces of Armco iron melted in hydrogen, cooled slowly to room temperature, cold rolled, reheated at 880° C. for about 18 hours in hydrogen, and cooled to room temperature in about one hour.

The heat treatments in accordance with this invention may be selected to produce desired variations in the magnetic properties in any particular instance. The treatments may also be modified to correspond to the characteristic behavior of the materials to be treated or with the necessities of commercial production. For instance, it has been found that for best results pure iron requires a second heat treatment at about 880° C. if it has been cooled rapidly from a high temperature. If, however, the iron is alloyed with other materials, such as with 4% molybdenum or 4% silicon, for instance, then the alloy does not require a second heat treatment for the best results. Without a second heat treatment an initial permeability as high as 4000 for silicon steel and 6000 for 4% molybdenum iron has been obtained.

Furthermore, as a general rule the second heat treatment at about 880° C. need not necessarily be done in hydrogen for producing improved initial and maximum permeabilities but may be carried out in vacuum, or in a non-oxidizing or neutral atmosphere, such as nitrogen, for instance. However, the best results have so far been obtained when hydrogen is used.

Thus, a specimen of Armco iron was heated at 1500° C. in hydrogen at atmospheric pressure for 30 minutes and cooled at an average rate of

750° C. per minute. It was then reheated at 880° C. in nitrogen at atmospheric pressure for 30 minutes and cooled at a rate of about 500° C. per minute. This treatment produced an initial permeability of 2000 and a maximum permeability of 18,500.

As an example of low hysteresis losses which may be developed in ordinary iron by the methods of this invention I may mention a treatment consisting in heating a specimen of Armco iron for 30 minutes at about 1500° C. in hydrogen at atmospheric pressure, cooling it to room temperature at the average rate of about 900° C. per minute, reheating it for about 30 minutes in hydrogen at atmospheric pressure at about 380° C., and finally cooling at an average rate of about 300° C. to room temperature. This iron had a hysteresis loss of only 1.15 ergs per cubic centimeter per cycle at a maximum induction of 450 gauss (inductions of this magnitude are frequently used in signaling apparatus), together with an initial permeability of 4000 and a maximum permeability of 41,600.

Noteworthy physical properties of materials treated in accordance with the methods of this invention are remarkable softness and low tensile strength. By way of example, the Rockwell B hardness of representative samples of iron was found to vary between +10 and -10 compared to the Rockwell B hardness of 70 of pure vacuum-annealed iron. (For definitions and principles of Rockwell hardness test see S. A. E. Handbook, September 1927, page 270, et seq.).

The properties produced in any given material by the methods of the invention are functions of at least four variables, to wit: (1) the temperature at which the material is heated; (2) the duration of the heating; (3) the pressure of the hydrogenous atmosphere in which it is heated; (4) the rate of cooling to the phase transformation point (in the case of materials having such a transformation point) and the gas pressure in which it is cooled. For instance, varying only factors (1) and (2), the pressure being that of the atmosphere, it was found in the case of iron that while heating for 30 minutes at a temperature of about 1500° C. produced an initial permeability of 6000, an initial permeability of about 7000 was obtained by heating at 1400° C. for seven hours, an initial permeability of 2000 by heating at 1300° C. for 14 hours, and an initial permeability of only 1000 by heating at 1200° C. for 20 hours; or, varying factor (3), heating the iron at 1500° C. for 30 minutes at 45 millimeters pressure produced an initial permeability of 10,400. Thus, a reduction in the heating temperatures should be accompanied by increased duration of heating, in order to approximate the same result.

In every case, if the iron is initially heat treated at a temperature above 900° C. and rapidly cooled (i. e. cooled at a rate of 100° C. per minute or more) improved results are obtained by an additional heat treatment at about 880° C.

A factor influencing the results obtained is the mass and shape of the material which effects their cooling rates and the times required for annealing. Results given herein relate to relatively thin specimens.

Although this invention has been described with reference to but some representative materials treated as has been set forth, it is understood that heat treatments in accordance with the invention may be employed for improving the magnetic properties of many magnetic ma-

materials; in fact, of the variety of materials treated, only iron incompletely deoxidized by aluminum or containing aluminum, did not exhibit a large improvement in initial permeability. The fact that iron deoxidized by aluminum has not responded to the treatment is a fact of which the full significance is not yet known. Aluminum as an impurity is sometimes present in iron, but in accordance with the present invention it appears to be an impurity to be avoided. However, if the iron is melted in hydrogen and subsequently the aluminum is added to the melt, a heat treatment of hard drawn wire made from the material at 1400° C. for 3 hours in hydrogen at 760° m. m. pressure followed by rapid cooling, reannealing at 880° C. for one-half hour followed by slow cooling resulted in a μ_0 of 3,700 and μ_{max} of 22,000. Therefore, it appears that the aluminum may be added after treatment of the molten iron in hydrogen with results not unsatisfactory.

In a particular instance, however, aluminum and silicon were present together in a specimen treated with satisfactory results. Possibly the silicon obviated the detrimental effects of the aluminum.

Among the possibilities of application of materials treated in accordance with this invention I may mention by way of example cores for relays, transformers, particularly audio frequency transformers with improved characteristics, magnetic clutches, power apparatus, receiver diaphragms, etc. Choke coils of high inductance and low resistance may be constructed in condensed volume with consequent saving of material and cost of manufacture. The possibility of developing desired magnetic properties in magnetic elements when combined with other elements or parts which limit the temperature to which the material may be heated is a feature of the present invention.

Ordinary iron or other magnetic materials treated in accordance with the invention may find a field of application as inductive loading material for signaling conductors, owing to the fact that their useful magnetic properties may be restored after the originally heat treated material has been subjected to detrimental influences simply by giving it a single heat treatment at about 880° C. Either after or before having received the first heat treatment at 1500° C. in hydrogen the magnetic material is formed by known methods into tape or wire of suitable dimensions. Then it is applied helically to a conductor as shown in Fig. 12. The copper conductor 12 is surrounded by a plurality of conductive copper strands 13 shaped to fit around the central conductor upon which is wound the tape of loading material 14. Deleterious strains introduced into the tapes or wires of the magnetic loading material during the machining and/or winding operations are relieved by giving the loaded conductor the second heat treatment at about 880° C. in hydrogen or not, as preferred. The invention therefore provides a method of continuously loading conductors with material of high initial permeability and other desirable magnetic properties.

So, also, for the production of finished machined parts in which it is desirable or necessary to avoid the use of excessive temperatures, the high temperature hydrogen treatment may be given to the unprepared iron stock. Machining, drawing, or other operations such as are employed to produce machined parts, transformer

cores and relay parts may then be performed, after which a simple annealing at about 880° C. will produce a final product of excellent magnetic properties. In particular, the low coercivity of this material combined with high initial and maximum permeability will enable certain classes of cores and relay parts to be made with improved characteristics or cheapened or both.

Further illustrative of the results obtainable by the methods of this invention, a ring of Armco iron $\frac{1}{4}$ " thick, $1\frac{1}{8}$ " inside diameter, $1\frac{1}{8}$ " outside diameter was heat treated in hydrogen at 1500° C. at 760 millimeters pressure for six hours, cooled to 880° C. in about 1 hour, then reannealed in hydrogen at 880° C. for one hour and finally cooled slowly in hydrogen in the furnace. It exhibited an initial permeability of 6000 and a maximum permeability of 56,000.

In another case an Armco iron ring of $1\frac{1}{8}$ inch inside diameter $1\frac{1}{8}$ inch outside diameter and $\frac{1}{4}$ inch high was treated at 1480° C. for 18 hours, cooled to 880° C. in about one hour and heated at 880° C. in hydrogen for 18 hours. It was found to have an initial permeability of 5000 and a maximum permeability of 280,000.

Materials treated as hereinbefore described may advantageously be used not only where the new magnetic properties set forth are desired, but are also useful in cases where the new physical properties, such as the high degree of softness and/or the high metallurgical soundness, for instance, are desirable, either per se or in combination with the magnetic properties.

A pending continuation in part hereof, Serial No. 173,956, filed November 11, 1937, has claims directed to the magnetic materials comprising alloys of iron and molybdenum which are described, but not specifically claimed, herein.

What is claimed is:

1. Method of preparing a magnetic material which comprises the step of melting the material in a hydrogenous atmosphere, followed by cooling to room temperature, cold working the material, reannealing at a temperature of about 900° C. and cooling to room temperature.

2. Method of preparing magnetic iron which comprises the steps of first melting the iron in a hydrogenous atmosphere and then deoxidizing the iron by adding aluminum.

3. The method of improving the desirable properties of magnetic materials, which comprises melting the materials in an atmosphere containing hydrogen and reducing the partial pressure of the hydrogen to a point at which there occurs substantially no liberation of absorbed hydrogen during the solidification of said materials.

4. Method as defined in claim 3 in which the partial pressure of the hydrogen gas is reduced to the value mentioned by substituting an atmosphere substantially composed of helium for the hydrogenous atmosphere.

5. The method of treating ordinary iron which comprises maintaining it in a hydrogenous atmosphere at a temperature between 1400° C. and the melting point and then cooling it to room temperature.

6. The method of improving the magnetic properties of magnetic materials which comprises maintaining them above 1400° C. in an atmosphere essentially of hydrogen for a period of time, working the material, and maintaining the material at a temperature of about 880° C. for a period of time in an atmosphere of hydrogen.

7. The method of producing a magnetic material consisting essentially of unalloyed iron of

5 improved magnetic properties which comprises
maintaining the iron in a hydrogenous at-
mosphere at a temperature between 1400° C. and the
melting point at pressures of hydrogen gas rang-
ing from a small fraction of an atmosphere to
above 12 atmospheres cooling the iron, followed
by annealing in an atmosphere of hydrogen at a
temperature of about 880° C.

10 8. Iron which has been maintained in an at-
mosphere of hydrogen first at a temperature of
between 1400° C. and the melting point for a
period of time and later at about 880° C. for a
further period of time.

15 9. Commercial iron which has been heat treat-
ed in an atmosphere of hydrogen above 1400° C.
for a period of time and later annealed at about
880° C. and which has a maximum permeability
greater than 100,000.

20 10. Poly-crystalline iron which has been heat
treated in an atmosphere of hydrogen at about
1500° C., and then later annealed at about 880°
C. and has an initial permeability of at least 2500.

11. The method of improving the magnetic
properties of magnetic materials including iron
and its alloys including its alloys with nickel and
with silicon which comprises first heat treating
the material in an atmosphere of hydrogen above
1400° C. and then heat treating the material at a
temperature of about 880° C.

12. An article of manufacture comprising an
industrial magnetic product composed of an iron-
nickel alloy which has been super-annealed above
1400° C. in a hydrogen atmosphere, and again
heat treated in a hydrogen atmosphere at about
880° C.

13. An iron-silicon alloy having up to about
4% silicon and the remainder essentially iron
which has been maintained in a hydrogen at-
mosphere above 1400° C. for a period of time
and later has been annealed at a temperature of
about 1000° C. and has an initial permeability of
at least 4000.

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