

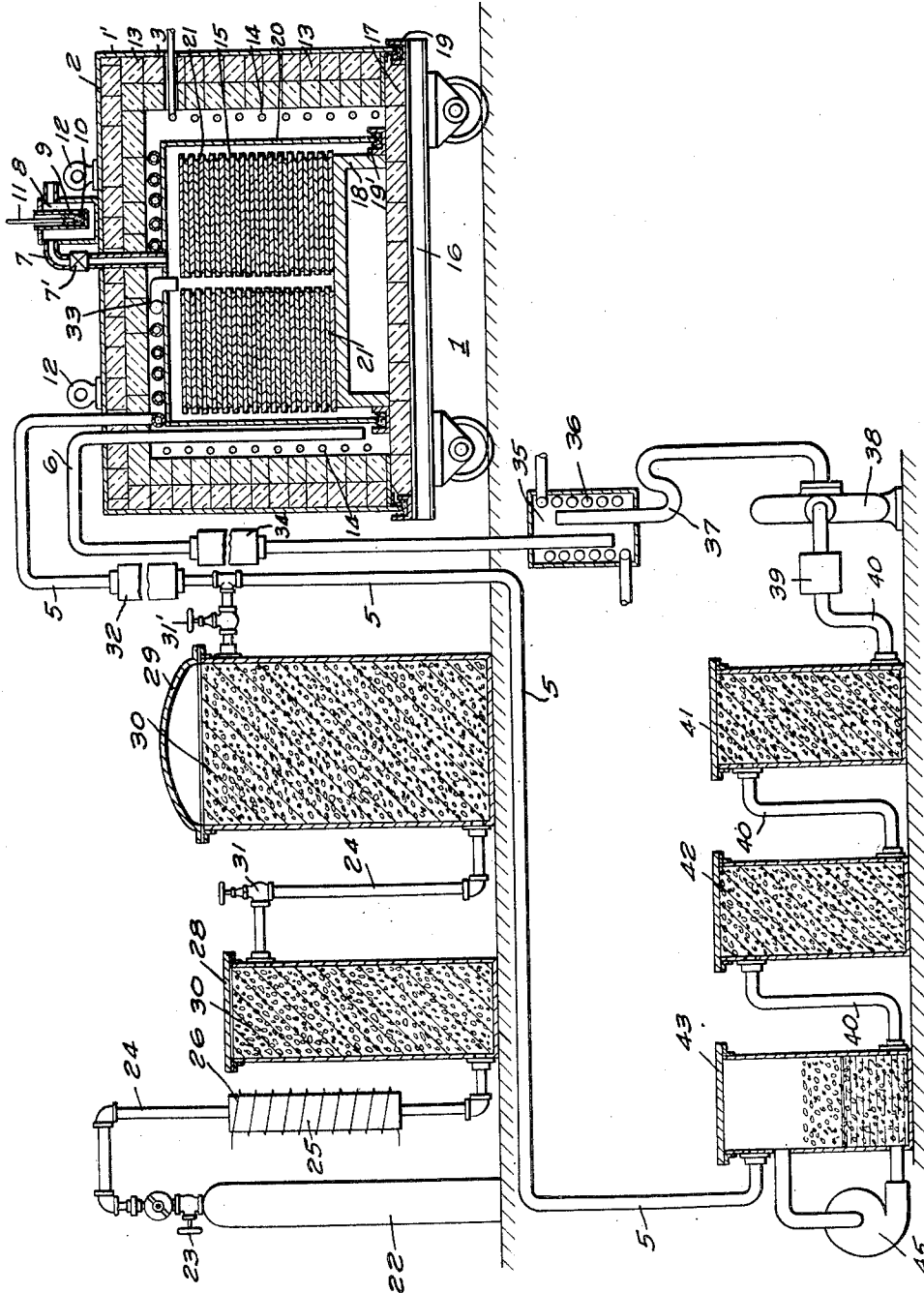
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MAGNETIC MATERIAL AND METHOD OF PRODUCING SAME

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WITNESSES.

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MAGNETIC MATERIAL AND METHOD OF PRODUCING SAME

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My invention relates to magnetic material and more particularly to a method of improving the magnetic properties of such material.

5 The principal object of my invention is to provide a process of annealing ferromagnetic material in a dehydrated and purified reducing atmosphere to greatly improve its magnetic properties.

10 Another object of my invention is to provide sheets or laminations of ferromagnetic material, which when tested magnetically, shall have high initial and maximum permeability and a low hysteresis loss.

15 A further object of my invention is to provide a method of treating sheets or laminations of ferromagnetic material which comprises coating the laminations with a refractory medium, heating to dehydrate the coated laminations, stacking a plurality of the coated sheets or laminations in an annealing furnace, and then passing a dehydrated and purified reducing gas rapidly through the furnace, thus maintaining the dew-point of the atmosphere in the annealing chamber below -20°C. , while maintaining the material at a temperature of 900°C. to 1400°C.

20 A still further object of my invention is to provide a process of annealing magnetic materials which comprises continuously passing a reducing gas, through desiccating and purifying agents and into a furnace containing the material to be annealed, conducting the gas after it has passed through the furnace through suitable desiccating and purifying agents, and finally returning the purified gas to the furnace.

25 It has heretofore been the practice to anneal ferromagnetic material in an atmosphere of commercial hydrogen. The hydrogen utilized for such purposes is usually prepared by the electrolysis of a saturated aqueous solution of potassium or sodium hydroxide. In this process, water vapor, oxygen, nitrogen and carbon dioxide and sometimes, hydrocarbons and sulphur-bearing gases are present as impurities in the hydrogen. According to the previous method of annealing the sheets or laminations of ferromagnetic material were coated with a refractory me-

dium, such as a suspension of magnesia, and dried in air, at room or elevated temperatures for about 24 hours. A plurality of these coated laminations were then stacked in a suitable furnace and the commercial hydrogen was passed through the furnace during the annealing cycle.

The ferromagnetic material produced by the foregoing method varies considerably in its initial and maximum permeability, and has a comparatively high hysteresis loss. Consequently, some means of preventing such variations in the magnetic properties of ferromagnetic material and producing material of uniformly excellent quality is highly desirable.

I have made the discovery that by first dehydrating and purifying the reducing gas employed as the annealing atmosphere, and then passing the gas through an annealing furnace containing a charge of laminations or sheets of ferromagnetic material at such a rate that the moisture and other impurities in the gas are maintained at a low value, a magnetic material may be produced that has excellent magnetic properties.

My invention will be better understood by reference to the accompanying drawing in which the single figure is a transverse sectional view of the gas-purifying apparatus and annealing furnace utilized in my improved method.

The purifying and annealing apparatus, as illustrated in the drawing, comprises a furnace 1, a tank or cylinder 22 containing an annealing gas such as hydrogen, which is suitably confined under pressure, a plurality of desiccating and gas-purifying chambers 28, 29, 41 and 42, a refrigeration unit 35, and means, such as a pump 38, for circulating the gas through the system.

While the particular furnace employed is not of paramount importance and I do not desire to be limited in this respect, I have found the furnace illustrated on the drawing to be very satisfactory. This furnace comprises a cover 2 having downwardly extending walls 3. Pipes or tubes 5, 6 may be inserted through the furnace cover 2 for the circulation of hy-

drogen through the furnace and purifying chambers.

The cover 2 is exteriorly fitted with a plurality of lifting eyes 12, and is interiorly fitted with a brick framework 13 of refractory material, such as alumina or zirconia bricks. Electric heating coils 14 are affixed to the walls of the brick framework 13 and supported thereby and serve to heat the chamber 15 during an annealing operation.

The base portion of my improved annealing furnace comprises a movable car or truck 16, having a layer of insulating brick 17 thereon, and a metal platform 18 superimposed upon the layer of insulating brick. A plurality of U-shaped channels 19 and 19' are provided at the outer edges of the layer of insulating brick, and at the outer edge of the metal platform 18, respectively, for the reception of the edge portions of the outer metal cover 2, and an inner metal cover 20 to which a coiled pipe 33 is attached.

In practicing my invention sheets or laminations of ferromagnetic material 21 are coated, by any suitable means, with a refractory medium, such as a suspension or solution of magnesia, alumina, or lime, or a mixture thereof. The refractory coating is applied to prevent the laminations from sticking together and to permit ready access of a reducing gas during the annealing operation to all parts of the laminations. The coated laminations or sheets are then dried, and the refractory medium is dehydrated by heating the laminations in a suitable furnace for a period of from 4 to 5 hours at a temperature of approximately 400° C. to 600° C.

In preparing the charge, the covers 1' and 2 are removed, and a plurality of the coated sheets or laminations 21, are stacked upon the metal platform 18, and the inner metal cover 20 and outer cover 1' are placed over the assembled laminations. The U-shaped channel 19 is filled with a refractory powder which is permeable to gaseous vapors, such as magnesium oxide. The cover 1', which is provided with a downwardly extending flange, is next lowered into position upon the truck base 16 so that the downwardly extending flange fits into the other U-shaped channel 19' which is then sealed so that an air-tight joint is produced. The sealing may be effected by filling the channel with a liquid seal, such as oil or a metal having a low-melting point, such as an alloy comprising tin, lead, and zinc.

A reducing gas, such as commercial hydrogen or cracked ammonia, contained under pressure in a metal cylinder 22, is then passed through the reducing valve 23 at a rate which is initially maintained at from 2 to 10 cubic feet per minute. The gas is passed through the tube 24 into a preheating cylinder 25 containing a finely divided catalyst, such as fine copper wire, platinized asbestos, or nickel

turnings which are maintained at a temperature of from 400° C. to 600° C. by means of the electric resistance coil 26 wound on the preheater 25. At this temperature, any oxygen present in the commercial hydrogen will combine in the presence of the catalyst with a portion of the hydrogen, forming water vapor, which is removed in a subsequent operation.

The annealing gas is then passed by means of pipe 24 and valves 31 and 31' through receptacles 28 and 29 containing granular desiccating agents 30, such as calcium chloride, phosphorus pentoxide, activated alumina or silica gel, thereby drying and purifying the gas. After passing through the valve 31', flexible gas joint 32 and pipe 5, the gas enters the spiral coil 33 where it is heated to a temperature of about 900° C. to 1300° C. The heated annealing gas is next conducted into the chamber 15, from where it diffuses downward over the laminations or sheets of ferromagnetic material 21, forcing out any air which may be in the furnace. It then passes through the permeable seal in the U-shaped channel or trough 19' and is carried away from the furnace by means of the pipe 6.

Various impurities, including the impurities removed from the metal and water vapor from the refractory lining of the furnace, are carried by the annealing gas as it leaves the furnace.

After the air and other impurities have been removed from the furnace, the charge is heated to a temperature of 900° C. to 1400° C. by means of the electric heating coils 14 and the reducing gas is passed through the furnace for a period of 2 to 24 hours, the length of the annealing cycle depending, of course, on the amount of impurities which are in the metal. In this operation, it is essential, in accordance with my invention that the gas be passed at a sufficient rate through the furnace so that the amount of moisture which is present in the gas shall be kept low, preferably less than .1%.

In order to render the process economical the annealing gas after being passed through the furnace is redried, purified and returned to the furnace. If any of the gas is lost from the system during the cycle, additional gas may be supplied from the container 22.

A convenient method for repurifying the gas is illustrated in the drawing. In accordance with this process, the gas is drawn through pipe 6 and gas joint 34 into a refrigerating unit 35 containing a coil 36 through which a suitable refrigerant, such as liquid air, liquid carbon dioxide, sulphur dioxide, methyl chloride, or cold water is continuously circulated by any suitable means. A certain amount of water vapor will be removed from the annealing gas by freezing in this operation, while more of the water will col-

lect in the water-trap 37 as the gas is passed therethrough. The gas is forced through the drying and purifying system by means of a pump 38, which furnishes sufficient pressure to overcome the resistance encountered by the gas as it is passed through the various drying and purifying materials.

The amount of impurities in the gas may be determined at any time during the operation of the system by means of the density meter 39. It is desirable to determine the density of the circulating gas in order to prevent the formation of explosive gaseous mixtures in the furnace and purifying apparatus.

The gas is then forced through the tubes 40 into a plurality of receptacles 41 and 42 containing granular activated alumina, silica gel, or phosphorus pentoxide which serve to absorb water vapor and partially purify the annealing gas. After passing through the pipe 40, the gas enters a receptacle 43 which is filled with fragments of an inert material, such as porcelain, and a circulating aqueous solution of potassium hydroxide which is continuously circulated through the receptacle 43 by means of the force pump 45. Gases, such as carbon dioxide, hydrogen sulphide, and sulphur dioxide are absorbed by this caustic solution and removed from the annealing gas as it passes through the receptacle 43. The purified and dehydrated gas is then returned through pipe 5 to the annealing furnace. The receptacles 41 and 43 may be connected as shown in the drawing or either one of them may be kept in reserve during the circulation of the annealing gas and utilized when the absorptive capacity of the drying agent in the other receptacle is terminated.

As previously stated, the moisture content of the annealing chamber is maintained at a low value during the annealing cycle preferably from .01% to .015% by volume or even less. This moisture content corresponds to a dew-point of $-20^{\circ}\text{C}.$ to $-40^{\circ}\text{C}.$ The measurement of the dew-point may be readily made at any time during the annealing cycle by means of a dew-point apparatus, comprising a gas chamber 8, a silver plated bulb 9, containing a freezing mixture 10 and a thermometer 11. By opening the valve 7 in a pipe 7 extending into the annealing chamber, gas is permitted to flow into the chamber 8. When moisture first condenses upon the silver-plated bulb 9, containing the freezing mixture 10, such as carbon dioxide dissolved in acetone, a reading may be taken on the thermometer 11, thus giving the dew-point of the annealing gas.

The following specific examples will serve to illustrate and explain my invention. Punched or stamped laminations composed of an iron-nickel alloy comprising 40% to 60% iron and 60% to 40% nickel, were an-

nealed by the usual method in commercial hydrogen gas. When tested magnetically, it was found that the maximum permeability of such alloys ranged from 40,000 to 60,000 and the hysteresis loss varied from 250 to 500 ergs per cubic centimeter per cycle at an induction of 10,000 gaussess. When similar iron-nickel laminations were annealed by my improved method in purified and dehydrated hydrogen and were magnetically tested, the maximum permeabilities were increased to from 113,000 to 167,000 and the hysteresis losses were reduced to from 90 to 125 ergs per cubic centimeter per cycle at an induction of 10,000 gaussess. It is thus apparent that the nature of the annealing operation determines to a marked extent the final magnetic properties of the ferromagnetic alloy.

Laminations of iron-silicon alloys containing 4% of silicon were also annealed in the manner specified. The core loss was reduced to .37 watts per pound at sixty cycles and inductions of 10,000 gaussess.

It is very desirable, in general, to maintain the dew-point of the annealing atmosphere at as low a value as possible in order to obtain the most desirable properties in the ferromagnetic material which is being annealed. This may be accomplished by varying the amount and velocity of annealing or reducing gas which enters the furnace. For example, when a furnace was utilized, which had an annealing chamber 1 foot high, 1 foot wide, and 3 feet long containing a charge of from 100 to 300 pounds of laminated ferromagnetic material, it was necessary to pass from 2 to 10 cubic feet of hydrogen per minute through the furnace in order to maintain the moisture content at the values previously specified. For larger furnaces it will of course be necessary to increase the hydrogen flow.

The pressure of the reducing or annealing gas in the drying and purifying system must be sufficiently high to overcome the resistance offered by the desiccating and purifying agents to the passage of the gas. This pressure may be controlled as desired by varying the relative position of the reducing valve 23 upon the gas cylinder 22 and by the speed of operation of the pump 38. In general, a pressure of from 5 to 10 inches of water or a positive pressure of from 1 to 2 ounces is sufficient to force the gas through the drying and purifying system.

My improved method of utilizing a purified reducing gas as the atmosphere in the process of annealing iron alloys of nickel may also be employed in annealing other magnetic material, such as the ferrous alloys of cobalt, arsenic, aluminum, or silicon, or combinations thereof with each other or with nickel, and the use of such an atmosphere is very effective in improving the magnetic

properties of such materials. Furthermore, my improved method is very economical because the reducing gas after passing through the furnace may be redried and repurified and returned to the furnace with very little loss.

While I have disclosed my invention in considerable detail and have given specific examples, it will be understood that the examples should be construed as illustrative and not by way of limitation. For example, when it is desired to remove carbon monoxide from the annealing gas, a receptacle containing a reactive material, such as a granular mixture of silver, copper and manganese oxides, known as Hopcalite, may be inserted in the purifying and drying system shown in the drawing.

It will also be understood that I do not desire to limit my process to a closed system as my invention consists broadly in passing a reducing gas through the furnace during the annealing operation in which the moisture content is maintained at less than .1%. This may be accomplished either by passing the purified and dehydrated gas rapidly through the furnace so that as the oxygen and other impurities are removed from the magnetic material, the amount of moisture which forms in the gas will not be increased above the amount specified. In the closed system, however, since the gas is purified to a greater extent, it may be passed through the annealing furnace at a lower rate of speed without increasing the moisture point above .1%. My invention in its broadest aspects, therefore, consists in passing a gas through the furnace of sufficient purity to properly anneal the material and this is accomplished either by passing a gas of greater purity through the furnace at a slower rate of speed or in increasing the speed if more impurities are present.

Other modifications of my invention will be apparent to those skilled in the art without departing from the spirit and scope thereof. It is, therefore, desired that only such limitations shall be imposed as are indicated in the appended claims.

I claim as my invention:

1. The process of treating ferromagnetic materials in an atmosphere of a purified and dehydrated reducing gas which comprises circulating the gas through a furnace containing said material while the dew-point in the furnace is maintained at not more than -20° C. and the temperature of the material is maintained at from 900° C. to 1400° C.

2. A magnetic alloy comprising 40% to 60% iron, and 60% to 40% nickel having a maximum permeability of from 113,000 to 167,000 and a hysteresis loss of from 90 to 125 ergs per cubic centimeter per cycle for $B=10,000$ gauss.

3. A magnetic alloy comprising iron and

nickel in approximately equal proportions, having a maximum permeability of from 113,000 to 167,000 and a hysteresis loss of from 90 to 125 ergs per cubic centimeter per cycle for $B=10,000$ gauss.

4. An iron-nickel alloy comprising iron and nickel in approximately equal proportions having a maximum permeability of at least 113,000.

5. An iron-nickel alloy comprising 40% to 60% iron and 60% to 40% nickel, said alloy having a hysteresis loss of not more than 125 ergs per cubic centimeter per cycle for $B=10,000$ gauss.

6. The process of treating ferromagnetic material which comprises annealing the material in a reducing atmosphere while maintaining the moisture content in the reducing atmosphere at less than .1%.

7. The process of treating ferromagnetic material which comprises annealing the material in a hydrogenous atmosphere while maintaining the moisture content of the hydrogen below .1%.

8. The process of treating ferromagnetic material which comprises annealing the ferromagnetic material in a furnace and passing a dried, purified reducing gas through the furnace at such a speed and of such purity that the moisture content of the gas will not exceed .1%.

9. The process of treating ferromagnetic material which comprises heating the magnetic material to a temperature of 900° C. to 1400° C. in a furnace while simultaneously passing a dried, purified reducing gas through the furnace at such a speed and of such purity that the moisture content will not exceed .1%.

In testimony whereof, I have hereunto subscribed my name this 25th day of May 1931.

JOHN B. SEASTONE.