Jan. 6, 1931. G. W. ELMEN 1,788,017

HEAT TREATMENT OF MAGNETIC MATERIAL

Filed July 6, 1925

Fig. 1

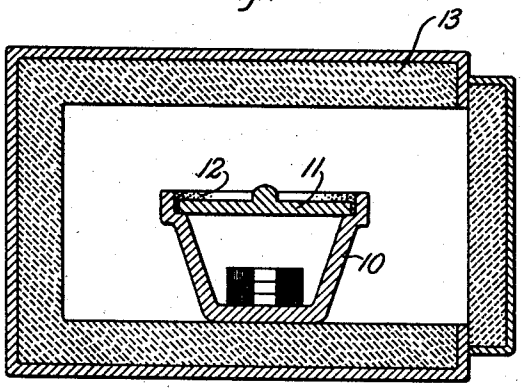
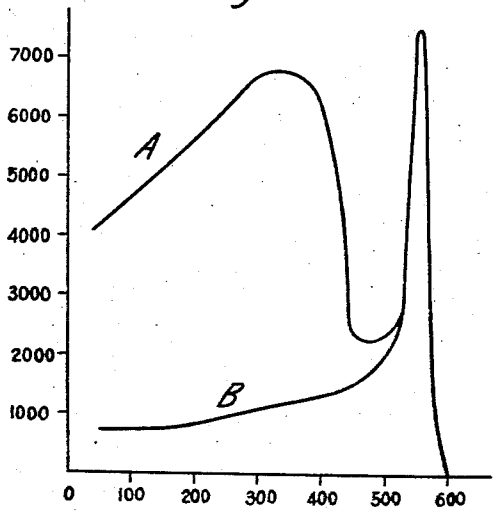



Fig. 2




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# UNITED STATES PATENT OFFICE

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## HEAT TREATMENT OF MAGNETIC MATERIAL

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This invention relates to an improved method of producing magnetic materials and especially alloys having high permeability at low magnetizing forces, and a low hysteresis loss. By low magnetizing forces is meant those in the range from 0 to around two-tenths of a gauss. These two properties, together with a resistance greater than that of iron, are characteristic, to a varying degree, of a class of magnetic materials consisting chiefly of nickel and iron.

In patent to G. W. Elmen, 1,586,884, June 1, 1926, there is described a proper treatment of these alloys to obtain the desired properties. Reference may also be had to an article by Arnold and Elmen, published in the Journal of the Franklin Institute, volume 195, No. 5, May 1923, entitled "Permalloy".

The usual method of treating nickel-iron alloys as set forth in the prior Elmen patent and in the paper by Arnold and Elmen, comprises casting the alloy, alternately working and annealing it until the desired shape or form is obtained, and then heat-treating it in its final form. The heat treatment consists of heating the material to a temperature of at least 900° C. for some time, cooling to a temperature somewhat above the transition temperature at about the usual rate employed in annealing, then cooling at a much more rapid rate to a temperature of about 300° C. The proper rapid rate of cooling can be determined by trial in each case. The rate will, of course, depend somewhat upon the size and form of the material as well as the composition. One form of the material which has been found convenient for a variety of uses is ribbon or tape about .005" or .006" in thickness, formed into a loosely wound coil. In a particular sample of this tape, the proper rate of cooling from 600° C. to 300° C. was found to be about 15° C. per second for about 20 seconds. The proper rate in all cases where rapid cooling is beneficial appears to be one between that which would ordinarily be used to anneal and one which would set up internal strains, due to premature hardening of the exterior portions. The rapid cooling is of most benefit when the ratio of nickel to iron is around 4. As the nickel content is decreased the advantage of rapid over slow cooling decreases until it nearly disappears when the ratio becomes about 1. It also decreases somewhat when the ratio is raised to 8 or 9. When other elements are added the percentage of iron and nickel for which the rapid cooling is beneficial varies considerably and depends largely upon the percentage of the added element. For example, while 78½% nickel, 19½% iron and 2% chromium requires the rapid cooling for highest permeability, if the chromium content is increased to about 8% or even somewhat less, rapid cooling gives no better results than slow cooling.

Many of the alloys containing nickel and iron are sensitive to strain—the permeability decreasing markedly when stress is applied. It is for this reason that heat treatment is employed after the material is in its final form when these alloys are used.

The primary objects of this invention are to insure a uniformly high value of desired properties of the various alloys containing iron and nickel over the range from about 25% or 30% of nickel upward, and to provide a convenient manner of performing the various steps of the heat treatment when the step of rapid cooling is utilized.

In carrying out these objects and in accordance with one feature of the invention, the material is subjected, during the initial stage of the heat treatment, to a temperature which will produce homogeneity of the material. In general this temperature is in the region of 1100° C., but a higher temperature is not ordinarily detrimental and may even be beneficial.

In accordance with another feature of the invention, the heat treatment above outlined is performed in two stages to accurately and conveniently control the temperature of the material and the rate of cooling.

In the accompanying drawings, Fig. 1 is a cross-section of a furnace suitable for heating the material. Fig. 2 is a graph showing initial permeability plotted against temperature.

Iron and nickel (and other elements if any are present) are fused together in an induc-

tion furnace. For example, the material may be nickel and iron alone in the proportions 21½% of iron and 78½% of nickel. Good commercial grades of these two materials are suitable for this purpose. The molten composition is poured into a mold and cooled to form a thick rod or bar. When the alloys are to be used for the purpose of loading signaling conductors, this bar is treated by the method outlined in the patent to Elmen referred to above to produce a tape of .006" thickness and .125" width. In treating the alloy in accordance with this invention, the tape may be formed in a loose coil and annealed by packing in ordinary cast-iron or nichrome boxes 10, Fig. 1, called annealing pots, each provided with a cover 11 luted with fire-clay, or the cover may fit inside the pot with a space above it which is filled with iron powder, finely divided copper or other readily oxidizable material 12. Finely divided copper is sometimes placed within the pot. These precautions are taken in order to prevent the free access of air to the inside of the pot during the annealing process and to eliminate the oxygen by means of the iron, copper or other material, thus leaving only nitrogen and preventing oxidation of the material under treatment. The boxes or annealing pots containing the material are heated to about 1100° C. and kept at that temperature until the alloy has reached the equilibrium stage for that temperature, that is, until uniform structure is obtained throughout the material. It has been found that for these alloys this uniformity of structure is beneficial to the magnetic characteristics. Ordinarily, this homogeneity is obtained by heating the pots for one hour after reaching the desired temperature. This heating may be done in any suitable furnace such, for example, as the furnace 13.

When the material is initially heated to 900° C., the lower limit of the range given in the above mentioned Elmen patent and the only value there specifically given, the permeability is high and the other desirable properties are obtained as set forth in that application. By raising the temperature above that value, say to 1100° C., a somewhat higher permeability is ordinarily obtained. Moreover, the lower temperature does not give quite uniform results with various samples of material, while uniformity is obtained by the use of the higher temperature. In order to obtain the best results the pots are left in the furnace after heating and allowed to cool down to room temperature with the furnace, or they may be removed and allowed to cool at a somewhat more rapid rate in the air. The cooling of the material, however, will be no more rapid than the cooling of the heavy annealing pots and will ordinarily give a material having a permeability corresponding to the lower curve B shown in Fig. 2. The annealing has also in some cases been done in vacuum furnaces. In that case there is no need for any additional precautions to be taken in order to prevent oxidation or other impurities from contaminating the samples. This method may also be of advantage in case it is desired to do the annealing under low air pressure or in the presence of other gases.

In a second stage of the process which is used when rapid cooling is advantageous the material is removed from the annealing pot and heated up in the oven to a temperature above the transition point. This will be in the neighborhood of 600° C. for the sample referred to above, but will be dependent upon the alloy being treated. The material is then removed and may be placed on a copper plate in the air at room temperature and allowed to cool. This relatively rapid cooling in this range insures that the permeability will follow the upper curve A of Fig. 2.

The rapid rate of cooling just described appears to be unessential as mentioned above, in the treatment of those alloys in which the nickel content is less than 40% or 50%, an annealing rate of cooling then being satisfactory. The high initial temperature, however, enables a high and uniform permeability to be obtained for low magnetizing forces, in all alloys composed chiefly of nickel and iron and in which the nickel content is greater than about 30%. In general, the alloys become less sensitive to strain as the nickel content is decreased from about 78% or 79%, the initial permeability decreases and the resistivity increases. In the region of 80% or 81% nickel and somewhat above, the sensitivity to strain also falls off rapidly. See U. S. patent of O. E. Buckley, No. 1,695,038, granted December 11, 1928. It has been discovered, however, that in all these alloys in the range from about 25% or 30% nickel upward, very high initial permeability at magnetizing forces of the order used in signaling (a few tenths of a gauss or less) may be obtained. Other ingredients, such as chromium for example, may be added to these alloys without destroying the property of high initial permeability at small magnetizing forces.

Not only do these alloys have very high permeability at low magnetizing forces when treated in the manner described but they also have a much lower hysteresis loss than iron.

After cooling, if the material is in the form of tape and is to be used for loading signaling conductors, it is given a certain degree of hardness by working or otherwise, after which it is wound upon the conductor with the edges of the succeeding convolutions preferably touching, but not overlapping, in the manner described in G. W. Elmen's British Patent No. 197,331. If further load-

ing of the conductor is desired, two or more layers of tape may be wrapped upon the conductor, each preferably in the opposite direction from the last in order to produce the most uniform construction. If the alloy is to be used for cores in loading and filter coils, the tape may be wound to a sufficient number of turns to give the coil the required cross-section and then impregnated with bakelite varnish in the manner described in the patent to G. W. Elmen, 1,586,889, June 1, 1926.

Other uses of permalloy and other forms than tape obviously come within the spirit and scope of the invention. It is also obvious that other forms and types of oven may be used in carrying out the process and other methods of heating and cooling may be employed. For instance, if the material is such that rapid cooling is beneficial and the principal object is to obtain the advantage accruing from heating the permalloy to 1100° C. as outlined above, the material after being heated to this temperature may be cooled slowly to a temperature near 600° C., the furnace opened and the material removed directly to the copper plate and allowed to cool rapidly, rather than allowing the material to cool to room temperature slowly in the oven and heating up again to 600° C. before allowing the parts to cool rapidly.

What is claimed is:

1. The method of increasing the permeability, at magnetizing forces of less than .2 gauss, of a magnetic alloy consisting chiefly of nickel and iron, which comprises heating the material to at least 1100° C. but below the melting point of the alloy and cooling it at an annealing rate of cooling.

2. The method of increasing the permeability, at magnetizing forces of less than .2 gauss, of a magnetic alloy consisting chiefly of nickel and iron, and in which the nickel content is at least 45% of the whole, which comprises heating the alloy to a temperature of at least 1100° C., but less than the fusing temperature of the alloy, and cooling it at an annealing rate of cooling.

3. The method of heat treating a magnetic alloy consisting chiefly of nickel and iron and in which the nickel content is about 78% to 80% of the whole, which comprises heating the material to about 1100° C. and cooling at an annealing rate of cooling.

4. The method of increasing the permeability, at magnetizing forces of less than .2 gauss, of a magnetic alloy consisting chiefly of nickel and iron and in which the nickel component comprises at least 45% of the whole, which comprises heating said material until its structure is uniform throughout, bringing it to a final temperature below 300° C. by temperature changes in which the material is cooled from a temperature above the magnetic transition temperature nearly to the magnetic transition temperature and finally cooled from around the magnetic transition temperature to below 300° C. at a rate in excess of an annealing rate but not so rapid as to set up strains in the material due to uneven hardening.

5. A method in accordance with claim 4 in which the first heating to unify the structure of the material is to at least 1100° C. but less than the melting point of the alloy.

6. The method of increasing the permeability of magnetic alloys at magnetizing forces less than .2 gauss which comprises first heating to a temperature of about 1100° C. and cooling the material through a considerable range of temperature at an annealing rate and second, bringing the material to a temperature above the magnetic transition temperature of the alloy and then cooling through and somewhat below the magnetic transition temperature as rapidly as can be done without setting up stresses in the cooled alloy due to uneven hardening.

7. The method of increasing the permeability of magnetic alloys consisting chiefly of nickel and iron which comprises first heating to about 1100° C. and cooling at an annealing rate, and second, reheating to a temperature slightly above the magnetic transition temperature, and cooling at a rate in excess of an annealing rate but less than that at which strains result from uneven hardening.

8. The method of increasing the permeability of a magnetic alloy consisting chiefly of nickel and iron and in which the nickel component is at least about 45% of the whole, which comprises heating the material to about 1100° C., cooling at an annealing rate to a point at least 100° C. below the magnetic transition temperature, reheating to a temperature in the vicinity of the transition temperature of the alloy, and cooling at a rate more rapid than the first cooling but not rapid enough to set up stresses in the cooled material due to uneven hardening.

9. The method of improving the magnetic properties of a magnetic composition consisting chiefly of nickel and iron in which the nickel component is at least 45% of the iron-nickel content, which comprises heating the material to about 1100° C., cooling at an annealing rate to about the magnetic transition temperature, and continuing the cooling to room temperature at a rate more rapid than the first cooling but not rapid enough to set up stresses in the cooled material due to uneven hardening.

In witness whereof, I hereunto subscribe my name this 30th day of June, A. D. 1925.

GUSTAF W. ELMEN.