Nov. 11, 1947.

H. L. B. GOULD MAGNETIC MATERIALS

Filed July 8, 1942

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EACH DETERMINATION OF 140

Fig.1. Co, Ni, Fe

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MAGNETIC MATERIALS

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Application July 8, 1942, Serial No. 450,170

10 Claims. (Cl. 148-12)

The invention relates to improvements in magnetic materials and more particularly to improvements in alloys which have a cobalt content of between about 20 to 25 percent, a nickel content of about 45 percent and a remaining content essentially of iron.

An object of the invention is to provide a method of treating the said alloys to stabilize their magnetic properties so that they remain substantially unchanged after the application 10 and removal of a magnetizing force.

Another object is to provide magnetic material of the said type with stabilized magnetic properties.

Another object is to provide magnetic circuits 15 for electromagnetic communication apparatus, which will have substantially stable magnetic properties, so that the apparatus may retain its operating characteristics substantially unchanged after being subjected temporarily to nor- 20 mal or accidental magnetization.

Cobalt-nickel-iron alloys to which the invention may be applied for the attainment of the stated objects have been described in United States Patents 1,715,541 and 1,715,647 issued to 25 G. W. Elmen on June 4, 1928. These alloys when properly treated have highly desirable magnetic properties for certain purposes, such as for use in communication apparatus operated at voice or carrier frequencies, for 30 example, continuously loaded conductors, loading coils and transformers. These alloys are particularly suited for those purposes on account of their constant permeability and their almost negligible hysteresis loss. It has, however, been observed that some of the magnetic properties of these alloys produced in accordance with prior art methods are sensitive to temporary magnetizing forces applied accidentally or in the course of normal operation. 40 Thus it has been found that after such magnetization the initial permeability has changed and that the change depends upon the intensity of the previously applied magnetic field, being particularly great within a comparatively low range of magnetizing forces. The initial permeability μ_0 referred to in this specification and in the appended claims is that generally known as the reversible permeability and is the permeability of a sample measured at 50 small alternating flux densities superimposed on the residual flux due to a previous magnetization. A treatment of said alloys for the purpose of stabilizing their magnetic properties has been described in the United States Patent 1,848,364 55 issued to V. E. Legg on March 8, 1932. In accordance with Legg's disclosure a ring sample of the

alloy is first annealed in the usual manner and is then magnetized in a given direction at high field strength (50 oersteds); after removal of this field the sample is "baked" at a suitable temperature (between 400 and 600° C.) for a considerable time (36 to 48 hours).

Legg found that by his treatment the initial permeability was markedly stabilized under certain conditions. Thus after the application of a magnetic shock in a given direction to the annealed and baked sample the initial permeability would remain practically unaffected by any subsequent magnetization in the same direction but would change considerably after a shock applied in a direction opposite that of the first shock. This instability was found to be considerably beyond the limits usually considered acceptable for communication apparatus of the type referred to above.

It is therefore a more specific object of the invention to produce a magnetic alloy of the said type having an initial permeability which will not vary more than about 2 to 5 per cent after temporary magnetization over a wide range of magnetizing forces applied in either or both direc-

tions.

It is still another specific object to produce a cobalt-nickel-iron alloy with such stabilized magnetic properties without excessively increasing the hysteresis losses.

In accordance with a principal feature of the invention a body of the cobalt-nickel-iron alloy is first annealed, then subjected to a strong magnetizing force in a given direction, then baked **35** for several hours with an appreciable unidirectional or alternating magnetizing force applied at least during a part of the baking period.

In the following description reference will be made to the accompanying drawing, in which:

40 Fig. 1 is a diagrammatic representation of a toroidal core C having a winding W thereon; and Fig. 2 is a graph or curve showing the variation in initial permeability of a typical sample, embodying the invention, due to application to the sample of magnetic fields of different strengths.

For the purposes of the investigation, samples were prepared in the form of toroidal cores, such as are frequently used for loading coils. Such a core C is shown diagrammatically in Fig. 1. The cores were wound of a continuous ribbon of the magnetic alloy, 0.008 inch in thickness. Experience indicates that even better results may be obtained with samples made of compressed powder of the alloys in which the particles are coated with an insulating film in well-known manner and for well-known purposes.

The alloys of the samples were composed of

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different proportions of cobalt, nickel and iron, as described more in detail below. For information about the production of these alloys and their properties reference may be had to the Elmen patents already referred to.

Each sample was first annealed in hydrogen at a temperature well above the Curie point and the subsequent bake was at temperatures below the Curie point, substantially as disclosed by Legg in his patent already referred to.

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Between the anneal and the bake a winding W would be wound on the core for producing a longitudinal flux therein, and the winding would remain on the core during the baking period; 5 the magnetomotive force could be made sufficient for producing a high flux density approaching saturation.

The experiments and test results for a number of such samples will now be described more in 10 detail.

| a | b · | .C | đ | е | ſ | 2 | 7 |
|---|--|--|--|---|---|--|--|
| Core | Tempera-1 | Magnetizing Force, | Duration of Field, Hrs. | Initial Perme- ability, μο | variation of | $\begin{array}{c} \text{Permeability} \\ \text{Coefficient } \lambda \end{array}$ | |
| | ture, °C. | • Oersteds | F 1010, 1113. | αυστιτογ, μυ | μ₀, Per Cent | High | Low |
| P117 P157 P102A P102A P118A P117A P158 P158 P159 P159 P154 P101A P155 P155 P156 | $\begin{array}{r} 425 \\ 425 \\ 425 \\ 425 \\ 425 \\ 455 \\ 465 \\ 465 \\ 465 \\ 465 \\ 465 \\ 465 \\ 465 \\ 465 \\ 465 \\ 465 \\ 465 \\ 465 \\ 400 \\ 510 \\ 540 \end{array}$ | 15 D. C 10 D. C 3 D. C | $ \begin{array}{r} 24 \\ 1 \\ 3 \\ 24 \\ 3 \\ 1 \\ 3 \\ 4.5 \\ 6 \\ 4 \\ 3 \\ 3 \\ 3 \\ 3 \\ 3 \\ 3 \\ 3$ | $\begin{array}{c} 90\\519\\505\\418\\515\\375\\501\\509\\372\\520\\476\\432\\608\\564\\574\\486\end{array}$ | $ \begin{array}{r}10\\34\\1\\3\\.8\\2\\1\\4.5\\4.5\\.3\\.6\\14\\.5\\1.3\\1.2\end{array} \end{array} $ | $(\times$.09 .20 .05 .05 .20 .10 .10 .09 .20 .13 .80 .05 .35 | $ \begin{array}{c} 10-4) \\ 150 \\ .05 \\ .03 \\ .06 \\ .02 \\ .02 \\ .02 \\ .02 \\ .02 \\ .02 \\ .02 \\ .02 \\ .02 \\ .02 \\ .04 \\ .02 \\ .05 \\ \end{array} $ |

Table I.-20 Co-45 Ni-35 Fe (Curie Point 685° C.)

Table II.—25 Co—45 Ni—30 Fe (Curie Point 725° C.)

| a | b | с | d | e | f . | 4 | 7 . |
|---|---|--|-------------------------------|--|---|---|--|
| Core | Tempera- ture, ° C. | Magnetizing Force, Oersteds | Duration of Field, Hrs. | Initial Perme- ability, µ0 | Maximum Variation of µ0, Per Cent | $\begin{array}{c} Permeability\\ Coefficient \lambda \end{array}$ | |
| | | | | | | High | Low |
| P111 P182 P182 P183 P188 P188 P184 P105A P175 P176 P177 P189 | $\begin{array}{r} 425 \\ 425 \\ 465 \\ 465 \\ 465 \\ 465 \\ 465 \\ 510 \\ 540 \\ 540 \end{array}$ | 15 D. C 10 D. C 10 D. C 3 D. C 3 D. C 2 D. C 3 D. C 3 D. C 3 D. C 3 D. C 9 | 24 1 1 4.5 6 4 | 99 308 190 230 208 288 322 228 228 251 378 | $ \begin{array}{r}3\\40\\.5\\1.1\\.6\\16\\18\\.6\\.4\\15\end{array} \end{array} $ | . 21 1. 4 | 10-4) 144 . 16 . 18 . 09 . 04 . 06 . 15 . 07 . 03 |

Table III.—23 Co—45 Ni—32 Fe (Curie Point 715° C.)

| a | b | с | d | е | f | 6 | 7 |
|---|--|---|--|--|---|-------------------------------|---|
| Core | turo ° C | Magnetizing Force, | Duration of Field, Hrs. | Initial Perme- ability, μο | Maximum Variation of µ0, Per Cent | Permeability Coefficient λ | |
| | | Oersteds | | | | High | Low |
| P128 P179 P181 P180 P180 P124A P164 P172 P173 | 425 425 465 465 465 465 510 540 | 15 D. C 10 D. C 10 D. C 3 D. C 2 D. C 3 D. C 3 D. C 3 D. C | $ \begin{array}{r} 24 \\ 1 \\ 1 \\ 6 \\ 4 \\ 3 \\ 3 \end{array} $ | $ \begin{array}{r} 115 \\ 396 \\ 237 \\ 241 \\ 346 \\ 425 \\ 346 \\ 372 \\ \end{array} $ | $10\\30\\1.0\\3.6\\12\\16\\1\\.8$ | (X1 | 0-4) 155 . 04 . 04 . 03 . 03 . 03 |

Table IV.—7 Co—70 Ni—23 Fe (Curie Point 640° C.)

| G | Ь | c | d | e | f | ſ | 7 |
|--------------|------------------------|-----------------------------------|----------------------------|-------------------------------|---|---|-----------------------|
| Core | Tempera- ture, ° C. | Magnetizing Force, Oersteds | Duration of Field, Hrs. | Initial Perme- ability, µ0 | Maximum Variation of µ0, Per Cent | $\begin{array}{c} Permeability\\ Coefficient \lambda \end{array}$ | |
| | | | | | | High | Low |
| P210 P209 | 465 465 | 3 D. C 0 | 3 | 434 720 | .7 14.3 | (×1 .67 1.3 | 10-4) . 11 . 19 |

In the tabulation given above, there is listed certain data for the bake and other data for the observed results of the bake for each of the samples.

Thus, column a of the tabulation gives the 5 core numbers of the different samples which have previously been annealed and subjected to a saturating field. Column b gives the temperature of the bake in degrees centigrade. Column c gives the magnetizing force in oersteds of the 10 magnetic field applied during the bake in the same direction as the previously applied saturating field. Column d gives the duration of the field applied during the first part of the bake. Column e gives the initial permeability μ_0 based 15 upon measurements taken immediately after the bake with a very small alternating field. Column f gives the maximum variation in initial permeability μ_0 in per cent of the permeability given in column e and due to application of magnetic 20fields of varying strength applied after the bake, as will be explained below. Column g gives the high and low values of the permeability coefficient λ , the values in the table to be multiplied by 10^{-4} ; this coefficient is obtained from the same 25tests as those in column f. As explained below, the permeability coefficient is a measure of the hysteresis loss.

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as before. A series of similar tests were then made with increasing fields up to 50 oersteds, all applied in the same direction as the bake field, and after each field had been removed the initial permeability was determined. Thereafter a second series of similar tests were made with the momentary field applied in the opposite direction of the bake field beginning at low field intensity and increasing up to a saturating field and after each application of the field the initial permeability was again determined. The various values of the initial permeability obtained after momentary application of the corresponding magnetizing forces are plotted in the curve shown in Fig. 2 of the drawing where the ordinates represent the initial permeability μ_0 and the abscissae the magnetizing force in oersteds of the momentarily applied fields; the fields to the right in the curve were in the same direction as the bake field and those in the left direction of the curve were in the opposite direction. From the curve it will be noted that the original initial permeability was 563 and that the initial permeability increased to 564 after the application of a very small field and retained this value after the application of fields up to 50 oersteds in the same direction as the bake flux. When the momentary field was reversed the initial permeability remained at 564 up to 1 oersted, thereupon it suddenly decreased to 562 at the point P and increased to 565, returning to 564 at the higher fields applied in the opposite direction of the baking field. The total variation from 562 to 565 of the initial permeability is less than 1 per cent. As is well known, the hysteresis losses may be expressed by the permeability coefficient

The Table I is for alloys of 20% Co, 45% Ni, 35% Fe, which have been previously annealed at 30 850° C. for one hour in a hydrogen atmosphere. The Table II is for alloys of 25% Co, 45% Ni,

30% Fe, which have been previously annealed at 850° C. for one hour in a hydrogen atmosphere.

Table III is for alloys of 23% Co, 45% Ni, 32% 35 Fe, which have been previously annealed at 850° C. for one hour in a hydrogen atmosphere.

Table IV is for alloys of 7% Co, 70% Ni, 23%Fe, annealed at 1000° C. for one hour in a hydrogen atmosphere. Each sample of the different alloys was subjected before the bake to a saturating field of 50 oersteds in a given direction. The bake for each sample was maintained for 24 hours and during the initial part of the bake a magnetic field was 45 applied in the same direction as the previous saturating field. The flux produced in the sample during the baking treatment was substantially greater than the residual flux due to the saturating field applied before the bake. As exceptions, 50 the samples P101A and P103A in Table I were subjected to an alternating field of 60 cycles per second. The procedure for a typical sample will now be described. Thus referring to the core P119A in 55 Table I, this core was first annealed at 850° C. for one hour in a hydrogen atmosphere. Upon cooling, the magnetizing winding was wound upon the core and a magnetizing force of 50 oersteds was momentarily applied producing a high degree of 60 saturation. The core with the winding was then placed in the furnace and heated to 490° C. in a hydrogen atmosphere for 24 hours. During the first 3.5 hours of the bake a magnetizing force of 3 oersteds was applied to the core. 65

$$\lambda = \frac{1}{\mu} \times \frac{d\mu}{dB}$$

40 where μ is the permeability at small alternating fields and B is the change in flux density in the sample at the small alternating fields.

The tests for the permeability coefficient of core P119A were made in conjunction with the tests for the initial permeability after each removal of the momentarily applied fields. It was found that the coefficient λ was equal to $.02 \times 10^{-4}$ over the entire range, except for a narrow range around the point P where the initial permeability shows a sharp peak; here the value of λ was equal to $.05 \times 10^{-4}$.

The curve shown in the drawing for the sample core P119A is typical for all of the samples in showing a comparatively uniform value for μ_0 over the entire range in both directions and in showing a sudden variation in μ_0 after application of comparatively light fields in the direction opposite the last heavy magnetization. The behavior of the permeability coefficient of the sample core P119A is also typical in the fact that the maximum value coincides in general with the peak variation in μ_0 .

The procedure in testing the core P119A is

Upon cooling and before any appreciable magnetizing force had been applied to the core, tests were made at low alternating magnetizing forces to determine the initial permeability which was found to be 563.

Thereafter the core was momentarily magnetized with a suitable low magnetizing force in the same direction as that applied during the bake and after removal of this field the initial permeability was again determined in the same manner 75

somewhat simplified and was adopted after a more complicated procedure had been tried in connection with several samples. Thus in accordance with the more complicated procedure the sample was baked and subjected to a field during the bake as described above; after the 70 bake, tests for μ_0 and λ were made after application of each of a series of increasing fields up to a saturating field in the same direction as the bake field; then a series of tests was taken with decreasing fields in the same direction; then a 75 third series of tests was made with increasing

fields up to a saturating field in the opposite direction of the bake field, and a fourth series with decreasing fields in the opposite direction of the bake field; a fifth series of tests was then made with increasing fields in the same direction 5 as the bake field and further series would repeat the tests in the second, third and fourth series, as desired.

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From these tests it was found that the permeability curve would show no peak during the 10 first and second series, would show a peak during the third series but not during the fourth series and would again show a peak during the fifth series but not during the sixth series. In other words, it may be concluded that the peaks occur 15 in a given direction until the material has been magnetized beyond the range at which the peak occurs and in the same direction. Thus after the bake the material is still under the influence of the saturating field applied before the bake, for 20 which reason there is no peak in the permeability curve during the first and second series of tests in the same direction as the saturating field. The procedure outlined for the core P119A was carried out in a similar manner for all of the 25 other cores listed in the tabulation. As will appear from the tables, in some cases an alternating field was applied during the bake instead of a unidirectional field and in other cases no field was applied at all during the bake for the sake of 30 comparison. In some cases the bake was carried out with a field applied during the entire time of the bake. A sample core P93, not listed in the tabulation, was specially treated; it was of the type which is 35 listed in Table I. The baking temperature was 425° C. and a magnetizing force of .63 oersted was applied during the entire baking time of 14 hours and during the subsequent cooling. The initial permeability was 588 and the maximum variation 40 in initial permeability due to application of momentarily applied fields up to a saturating field was 3.4 per cent; λ was $.09 \times 10^{-4}$. The same core was thereupon baked at 425° C. for another 24 hours with a magnetic field of .63 oersted applied $_{45}$ during the entire bake and during the subsequent cooling, giving an initial permeability of 510 and variation in the initial permeability of 2 per cent over the same range as before; λ varied from .25 at the peak of μ_0 to $.05 \times 10^{-4}$ where μ_0 was nor- 50 mal. The same core was then further baked at 425° with a field of .63 oersted applied during the baking period of 24 hours and during the subsequent cooling, giving an initial permeability of 477 with a variation in permeability of 2 per cent; 55 λ changed from .21 to .04 \times 10⁻⁴. From the experiments described above it was found that the variations in permeability were smaller when the baking field was applied in the same direction as the saturating field applied be- $_{60}$ fore the bake, than when the bake field was in the opposite direction.

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to 6 hours or of greater magnetizing force for a shorter time, as, for example, a magnetizing force of 10 oersteds for 1 hour. With treatments within the stated limits, variations in the initial permeability will be limited to 5 per cent and λ will remain less than $.7 \times 10^{-4}$.

However, it is possible, as in the case of the core P119A in Table I, the cores P183 and P177 in Table II, the core P181 in Table III and the core P210 in Table IV to secure an initial permeability which will remain stable within 1 per cent after any magnetic shock up to saturation, and at the same time secure low hysteresis losses.

From the specific tests of the core P93 referred to above, it appears that similar results may be obtained with a small magnetizing force applied during the entire baking period; μ being very high at this field strength, the corresponding flux is, of course, greater than the residual flux due to the saturating field applied before the bake. In the claims reference is made to a "directed" magnetic field." This does not necessarily mean a field extending in a straight line through the body of material but includes, by way of example, a field which traverses a toroid in a circular direction or a field which traverses an "L-shaped" member through the two arms of the L in series. What is claimed is: 1. A body of a magnetic alloy containing between 20 and 25 per cent of cobalt, 45 per cent of nickel and the balance essentially of iron and having been heated to between 425° and 540° C. for a period of several hours after having been annealed in hydrogen and then temporarily magnetized substantially to saturation by the application thereto of a magnetizing force, said body having been less strongly magnetized during the first few hours of said heating period to reduce variation in its initial permeability, caused by subsequent magnetization of any intensity and direction, to not more than 5 per cent. 2. A body of a magnetic alloy containing about 20 to 25 per cent of cobalt, 45 per cent of nickel and the balance essentially of iron and having been heated to between 425° and 540° C. for a period of twenty-four hours after having been annealed in hydrogen and then temporarily magnetized by the application thereto of a substantially saturating magnetic field, said body having been magnetized during the initial three to six hours, approximately, of said heating period by the application thereto of a magnetizing force of about 3 oersteds to prevent more than 5 per cent variation in the initial permeability of said alloy caused by subsequent magnetization of any intensity and direction. 3. A body of a magnetic alloy containing about 20 to 25 per cent of cobalt, 45 per cent of nickel and the balance essentially of iron and having been heated to between 425° and 540° C. for a period of twenty-four hours after having been annealed in hydrogen and then temporarily magnetized by the application thereto of a substantially saturating magnetic field, said body having been magnetized during the initial three hours, 65 approximately, of said heating period by the application thereto of a magnetizing force of about 3 oersteds. 4. A body of a magnetic alloy containing about 20 to 25 per cent of cobalt, 45 per cent of nickel and the balance essentially of iron and having been heated to between 425° and 540° C. for a period of twenty-four hours after having been annealed in a hydrogen atmosphere and then

It was also found that a high baking field applied for a long time during the bake tends to reduce materially the permeability of the alloy.

It was further found that the application of an appreciable field for a long time during the bake tends to materially increase λ and therefore the hysteresis losses.

It appears from the data of the tabulation that 70 low values for variation in μ_0 and for λ will be secured by baking the alloys at temperatures between 450 and 550 approximately and applying a field during the first portion of the bake with a magnetizing force of about 3 oersteds for from 3 75 temporarily magnetized by the application there-

to of a substantially saturating magnetic field in a given direction, said body having been magnetized during the initial three hours, approximately, of said heating period by the application thereto in said given direction of a unidirectional 5 magnetizing force of about 3 oersteds.

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5. A body of a magnetic alloy containing about 20 to 25 per cent of cobalt, 45 per cent of nickel and the balance essentially of iron and having been heated to between 425° and 450° C. for a 10 period of twenty-four hours after having been annealed in hydrogen and then temporarily magnetized by the application thereto of a substantially saturating magnetic field, said body having been magnetized during about the first hour of 15 said heating period by the application thereto of a magnetizing force of about 10 oersteds. 6. A method of treating a body of magnetic material, which has a cobalt content of between about 20 to 25 per cent, a nickel content of about 20 45 per cent and a remaining content essentially of iron and which has been annealed in a hydrogen atmosphere, to insure that the permeability coefficient of said body will remain not more than $.7 \times 10^{-4}$ and that the initial permeability of said 25 body will vary not more than 5 per cent when the body has been magnetized to any intensity and in any direction subsequent to the treatment, said method comprising first temporarily magnetizing the body by applying thereto a sub- 30 stantially saturating magnetic field, then heating the body to a temperature between about 425° and 540° C. for a period of several hours and during at least a fraction of said heating period magnetizing said body less strongly.

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riod of about twenty-four hours, and during a fraction of said heating period of about three hours magnetizing the material by applying thereto in said given direction a unidirectional magnetic field of about 3 oersteds.

9. A method of treating a body of magnetic material to reduce variation in one of its magnetic properties due to magnetization of any intensity and in any direction, said material having a cobalt content of 20 per cent, a nickel content of 45 per cent and a remaining content of essentially iron, said material having been annealed in hydrogen, said method comprising first temporarily magnetizing said body by applying thereto a substantially saturating magnetic field in a given direction, then heating the body to a temperature between about 425° and 540° C. for a period of about twenty-four hours and during the first three hours approximately of said heating period magnetizing the body by applying thereto a similarly, including oppositely, directed magnetic field of 3 oersteds. 10. A method of treating a body of magnetic material that has a magnetic property which before the treatment was unstable within a certain range of applied field intensities, said material having a cobalt content of about 20 to 25 per cent. a nickel content of 45 per cent and a remaining content essentially of iron, said material having been annealed in hydrogen, said method comprising first temporarily magnetizing said material by applying thereto a substantially saturating magnetic field, then heating the material to a temperature between about 425° and 540° C. 35 for a period of several hours, and during about the first hour of said heating period magnetizing the body by applying a magnetic field thereto of about 10 oersteds to substantially stabilize said property within said range of field intensities ap-

7. A method of treating a hydrogen annealed body of magnetic material that has a magnetic property which before the treatment was unstable within a certain range of applied field in-

tensities, said material having a cobalt content 40 plied in the given or the opposite direction. of about 20 to 25 per cent, a nickel content of 45 per cent and a remaining content essentially of iron, said method comprising first temporarily magnetizing said material by applying thereto a substantially saturating magnetic field, then heating the material to a temperature between about 425° and 540° C. for a period of several hours, and during a fraction of said heating period of about three to six hours magnetizing the material by applying a magnetic field thereto of about 3 oersteds to substantially stabilize said property within said range of field intensities applied in the given or the opposite direction.

8. A method of treating a body of magnetic material which has a cobalt content of between 55 about 20 to 25 per cent, a nickel content of about 45 per cent and a remaining content essentially of iron, said method comprising first annealing said material in hydrogen, then temporarily magnetizing said material by applying thereto a sub- 60 stantially saturating magnetic field in a given direction, then heating the material to a temperature between about 425° to 540° C. for a peHAROLD L. B. GOULD.

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