

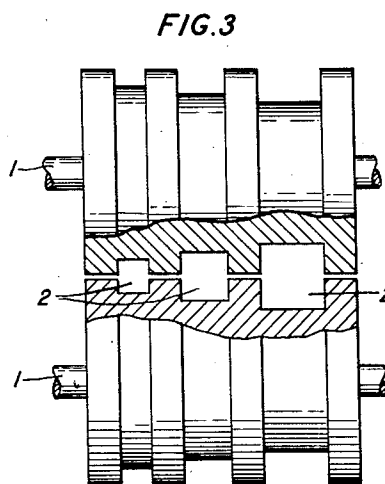
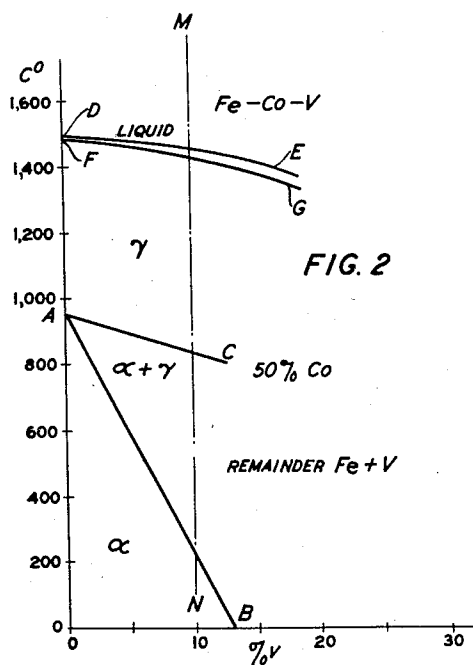
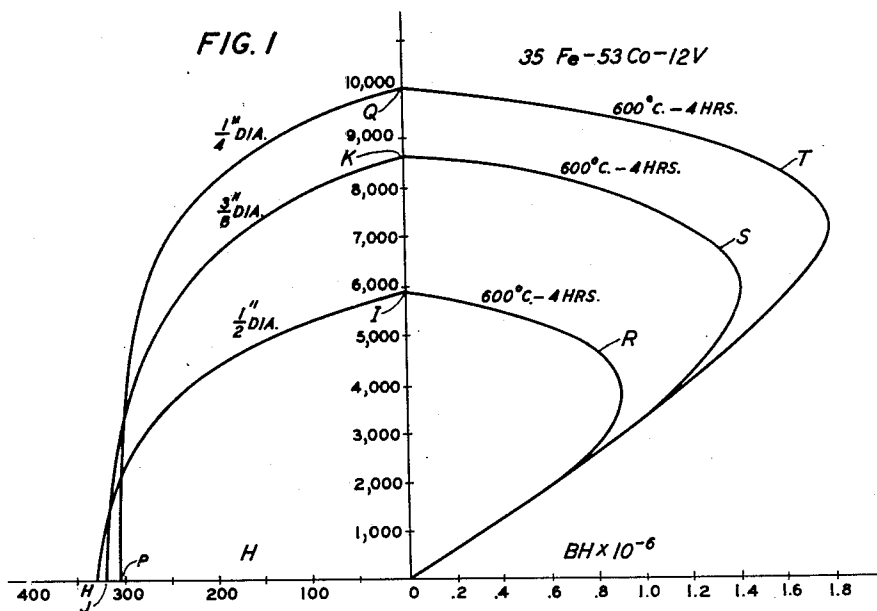
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PERMANENT MAGNET MATERIAL AND PRODUCTION THEREOF

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PERMANENT MAGNET MATERIAL AND
PRODUCTION THEREOF

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4 Claims. (Cl. 148—2)

This invention relates to permanent magnet alloys and to magnets produced therefrom which are caused to have more desirable properties as permanent magnets by a cold working treatment. The invention also relates to and includes methods of treating and producing such improved alloys and permanent magnets.

An object of the invention is the production of better and more efficient permanent magnet materials.

Alloys designed for use as permanent magnets and having a composition according to the present invention include as a specific and preferred class those composed of between 30 to 52 per cent iron, 36 to 62 per cent cobalt and 6 to 16 per cent vanadium or chromium and as a subclass certain members of this group including those having the upper portion of the above-specified ranges of vanadium contents.

Reference will be made to certain other compositions of similar nature and employing in their production similar processes hereinafter described.

In a parent application Serial No. 201,058, filed April 9, 1938, which was filed by the present inventor jointly with another, certain phases of the present invention are disclosed but were not claimed therein because such subject-matter was invented or discovered by the present applicant solely; nevertheless the benefit of the filing date of said application is claimed for the present application to whatever extent and under whatever provisions of law may be permissible.

A feature of the present invention is the discovery that permanent magnet compositions, such as those specifically set forth hereinbefore, may be improved by reduction of the cross-section thereof by a stage of cold working during their preparation and prior to the final heat treatment. Such working is preferably accomplished by methods which produce elongation and prevent lateral spreading. Methods that have been found beneficial include swaging, rolling with grooved rolls and wire drawing. It appears that the result of improved permanent magnet properties is manifested chiefly in the direction of elongation. Reduction of thickness by such rolling of a metal sheet as permits the material to spread laterally is only partially effective.

The evidence is that materials of or produced according to the present invention generally are anisotropic and their improved properties are manifested chiefly in the direction in which the elongation takes place.

The nature, extent and principles of the pres-

ent invention or discovery may be more clearly elucidated by reference to the accompanying drawing wherein:

Fig. 1 is a diagram illustrating the increased magnetic energy product, in the longitudinal direction, of a typical composition in accordance with the invention comprising 35 per cent iron, 53 per cent cobalt and 12 per cent vanadium;

Fig. 2 is a typical constitutional diagram of compositions in accordance with the invention; and

Fig. 3 is a diagram of grooved rolls which may be employed to roll down bars of material in accordance with the present invention.

Reference will be first made to Fig. 2 which is the constitutional diagram of alloys composed essentially of 50 per cent cobalt with the balance iron and vanadium. Along the Y axis temperatures from 0° C. to above the melting point are indicated and along the X axis percentages of vanadium are indicated. The line AB on this diagram separates what is known as the "alpha" phase of the material upon the left and below the line AB from the region to the right of the line AB where the "alpha" phase exists together with a "gamma" phase. Line AC separates the region wherein the two phases exist from the region wherein the "gamma" phase is formed and the lines DE and FG bound the narrow region wherein melting and solidifying of the material take place. Let us now consider some specific composition, such as one containing 50 per cent cobalt, 40 per cent iron and 10 per cent vanadium. As the temperature is raised and lowered along the line MN constitutional changes in this material take place. Below the line AB the material tends to assume the "alpha" phase, between the lines AB and AC both the "alpha" and "gamma" phases can exist, and above the line AC the material tends to assume the "gamma" phase. At a still higher temperature the material melts, with which we are not particularly concerned at the present time. For somewhat different percentages of cobalt the lines AB and AC will assume somewhat different positions. It will be noted that many of the compositions included in the ranges hereinbefore specified approach room temperature before the condition for formation of "alpha" phase exists and some of them of higher vanadium content even pass below room temperature or even below zero before the condition for formation of this phase exists. As pointed out in the application hereinbefore mentioned, now Patent No. 2,190,667, dated February 20, 1940, one of the procedures for pre-

paring permanent magnet material as disclosed in the above patent is to melt the material and cool it to room temperature to produce formation of the "alpha" phase and thereafter elevate the material to a higher temperature between 500° C. to 800° C. for a length of time such as is necessary to allow a small amount of the "gamma" phase to precipitate in a highly dispersed form in the "alpha" phase. This produces dispersion hardening in the material and produces a very effective permanent magnet material. It is particularly noteworthy that this method of forming permanent magnet materials is different and involves a different principle than that heretofore employed with respect to many others, such as alloys of iron, cobalt and molybdenum, in that in the present instance the material is converted into a low temperature "alpha" phase and thereafter has a small amount of the high temperature "gamma" phase precipitated therein in a fine and dispersed state. In the usual case of permanent magnets hardened by precipitation the high temperature phase is preserved and a small amount of the low temperature phase is precipitated.

Attention now is directed to the fact that in alloys having a constitutional diagram as in Fig. 2, in which the "gamma" to "alpha" transformation tends to occur at lower and lower temperatures, the transformation becomes more and more sluggish and non-equilibrium conditions exist. From the diagram of Fig. 2 it becomes apparent that with low percentages of vanadium the "gamma" to "alpha" transformation occurs at a relatively high temperature whereas, when the percentage of vanadium increases, the transformation takes place at a lower and lower temperature and finally below room temperature. As the temperature for the phase change decreases the change becomes more and more sluggish and non-equilibrium conditions exist.

Cold rolling these alloys in grooved rolls or working them mechanically by any equivalent method greatly expedites the formation at room temperature of the "alpha" phase and brings them into equilibrium. In fact, alloys in this region containing 12 per cent vanadium or higher would not change completely to the "alpha" phase at room temperature unless given the above treatment. This result is one beneficial aspect of the present invention. However, there is a second beneficial aspect which is due to crystal orientation. X-ray measurements confirm this and magnetic tests show the best permanent magnet properties in the direction of elongation.

After the alloys are brought into the "alpha" phase by the combination of cooling and cold working they are raised to a temperature which brings them between the lines AB and AC of Fig. 2 which causes the precipitation in finely dispersed particles of some of the "gamma" phase. This results in the production of magnetic material having effective and desirable properties as permanent magnets in the direction of elongation. In every case care must be used not to raise the material to a temperature above the line AC in order not to lose the effect of cold working. For alloys having 50 per cent cobalt the method described will be effective to produce an improvement for practically any vanadium content up to 16 per cent and the upper range of vanadium content is the range wherein the improvement is most important because it is in this range that the cold working produces the beneficial results to the greatest extent. It would appear that alloys of still higher vanadium con-

tent which assume the "alpha" phase below zero could be improved by cold rolling below zero. It does not seem that this range of compositions is of practical value.

With different percentages of cobalt the maximum percentage of vanadium in alloys capable of being treated usefully according to the present invention may be somewhat more or somewhat less. It is contemplated that the present invention includes all ranges of compositions of iron-cobalt-vanadium having commercially desirable properties and having constitutional diagrams similar in form to that illustrated in Fig. 2.

A rough criterion of the desirability of permanent magnet materials which is frequently used in practice is the product of "coercive force" and residual induction. A more accurate figure of merit is that of maximum energy product, which on the demagnetization portion of the hysteresis loop, is the product of induction B and magnetizing force H at a point where this product is the greatest. See Wahl, "Applied Magnetism," pages 42 to 45 inclusive.

Fig. 1 illustrates the application of the more accurate method to the experimental determination of the efficacy of the present invention. The figure relates to a composition consisting of 35 per cent iron, 53 per cent cobalt and 12 per cent vanadium. Curve HI represents a quarter of the hysteresis loop of a bar $\frac{1}{2}$ inch in diameter which was cast and hot swaged from 1 inch in diameter to $\frac{3}{4}$ inch in diameter after which specimens were cold groove rolled successively to $\frac{1}{2}$ inch, $\frac{3}{8}$ inch and $\frac{1}{4}$ inch diameters and in each case raised thereafter to a temperature of 600° C. for 4 hours. The curve HI represents the demagnetization curve of the bar $\frac{1}{2}$ inch in diameter, curve JK that of $\frac{3}{8}$ inch diameter and curve PQ that of $\frac{1}{4}$ inch diameter. It will be seen that the residual induction increased from a value around 6000 for the bar of $\frac{1}{2}$ inch diameter to around 10,000 for the bar of $\frac{1}{4}$ inch diameter. The curves IRO, KSO and QTO are plotted to represent the flux density B against the BH energy product. The distance to which these curves extend to the right as plotted and measured in figures along the X axis is a criterion of the desirability of the material and the further the curve extends to the right the more desirable the material. It will be seen that the material which has been cold rolled the most has considerably better properties. It will be understood that magnetic measurements on these materials were taken with the lines of magnetic force parallel to the direction of elongation.

Numerous other compositions of iron, cobalt and vanadium within and in the general region of the ranges of compositions hereinbefore specified exhibit similar phenomena and other sets of curves similar to those of Fig. 1 might be given. Fig. 1 is typical in form. The numerical values differ considerably in the case of different compositions.

In the case of Fig. 1, the point to which the curve QTO extends to the right measured in the units given is approximately 1,800,000. It will be noticed that this value is less than the value of residual induction multiplied by the coercive force, the product of which would be around 3,000,000. Careful distinction should be made between these two products. The maximum energy product of a composition somewhat similar to that which formed the basis of Fig. 1, but consisted of 36 per cent iron, 53 per cent cobalt and 11 per cent nickel, reached a value of

almost 1,900,000, which is in excess of the corresponding product with any magnetic material known to have been heretofore proposed for commercial use or utilized in commercial products. Among those which are included in this statement and proposed and measured for comparative purposes are compositions consisting of 72 per cent iron, 12 per cent cobalt and 16 per cent molybdenum; 36 per cent cobalt, 7 per cent tungsten, 4 per cent chromium, .6 per cent carbon and the balance iron; 38 per cent iron, 52 per cent cobalt and 10 per cent vanadium (not cold rolled); 29 per cent nickel, 58 per cent iron and 13 per cent aluminum; and a composition consisting of 20 per cent nickel, 53 per cent iron, 12 per cent cobalt, 10 per cent aluminum and 5 per cent copper. The highest energy product secured by preparing and testing all of these materials was 1.5×10^6 .

Another composition according to the present invention and containing 34 per cent iron, 52 per cent cobalt and 14 per cent vanadium yielded, when cold rolled and heat treated, a maximum BH energy product of almost 3,000,000, a residual induction of 9600 gauss and a coercive force of 410 oersteds. This energy product is the highest ever reported for a permanent magnet material except for an expensive platinum-cobalt alloy. These results were obtained on a wire of 0.040 inch in diameter, which was cold drawn through dies to produce it from a wire of $\frac{1}{8}$ inch diameter and then baked at 575° C.

It will therefore be seen that the compositions under discussion have magnetic energy products of a very high value when prepared according to the method of the present application measured in the direction of elongation.

They may be prepared in the form of rods, bars or tapes. A suitable treatment for any specimen is first to give it the desired amount of cold working plus a low temperature bake. No other heat treatment is necessary. Satisfactory results have been obtained with reductions in area of 75 per cent although this exact amount is by no means critical.

The following two tables show the improvement of the present invention over the invention claimed in the above-noted patent:

Previous results without cold elongation	Cold elongation plus 600° C. bake
38 FE-52 CO-10 V	
Br=9,000 Hc=300 Max. BH10°=1.0	Br=11,700 Hc=195 Max. BH10°=1.39 Improvement in BH max.=39 per cent
35 FE-53 CO-12 V	
Br=4,870 Hc=392 Max. BH10°=.63	Br=10,000 Hc=300 Max. BH10°=1.8 Improvement in BH max.=200 per cent

In the case of the first composition the alloy is already in the "alpha" phase on cooling to room temperature and the improvement is due to the cold elongation causing crystal orientation. In the case of the second composition the alloy is in the "gamma" phase on cooling to room temperature and the improvement is due to the cold elongation causing both the "gamma-alpha" phase change and crystal orientation.

X-ray examination indicates that the "gamma"

phase materials of this class have a face-centered cubic structure which is not very magnetic. After transformation to the "alpha" phase the alloy has a body-centered cubic structure and is highly magnetic. Cold rolling followed by precipitation of some of the "gamma" phase in the "alpha" phase results in giving the crystals a preferred orientation and also producing good permanent magnet properties.

There are other magnetic alloys which have a constitutional diagram generally similar to that of Fig. 1. Tests have indicated that cold rolling in grooved rolls produces an increase in the permanent magnet properties, that is, an increase in the energy product of such materials when they are prepared by first heat treating, then cooling by quenching or otherwise to around room temperature, then cold elongation, then heating to a point lying between the lines AB and AC for the particular composition.

An example of a composition containing chromium which has been found to respond to this type of treatment is 36 per cent iron, 56 per cent cobalt and 8 per cent chromium. This composition exhibits permanent magnet properties and is improved in properties by an elongating treatment, when cold, of the kind described. It has been found to respond to a cold elongating treatment and to exhibit an increased energy product as compared with identical compositions not subjected to cold rolling.

Fig. 3 represents rolls which may be used to roll down material to elongate it without spreading it laterally. The rolls may be power driven by shafts 1 upon which the rolls are mounted. The rolls have a series of square sided grooves 2 of successively decreasing diameter through which the material may be passed in succession.

The symbol $\pm$ as used in the appended claims is to be read with the meaning "within."

What is claimed is:

1. A tough non-brittle magnetic article of high permanent magnet properties composed of an alloy comprising as essential constituents 30 to 52 per cent iron, 36 to 62 per cent cobalt, and 6 to 16 per cent vanadium produced by heating the alloy to from 800° to 1300° C., cooling substantially below 600° C., forcibly elongating the material in the cold condition, thereafter maintaining it at around 500° C. to 800° C. for a period of time from several hours at the lower range to a much shorter time at the higher range and thereafter magnetizing it.

2. The method of preparing material for permanent magnet use which comprises melting an alloy composed of 30 to 52 per cent iron, 36 to 62 per cent cobalt and 6 to 16 per cent vanadium, thereafter cooling the material to substantially in the neighborhood of room temperature, thereafter forcibly elongating the material in the cold condition and thereafter maintaining it at that higher temperature above room temperature at which the finely dispersed "gamma" phase of the material is formed but without raising it to the temperature at which the entire body of the material is converted to "gamma" phase.

3. A tough non-brittle magnetic article of high permanent magnet properties composed of an alloy comprising as essential constituents 30 to 52 per cent iron, 36 to 62 per cent cobalt, and 11 to 16 per cent vanadium produced by heating the alloy to from 800° to 1300° C., cooling substantially below 600° C., forcibly elongating the material in the cold condition, thereafter maintaining it at around 500° C. to 800° C. for a period

of time from several hours at the lower range to a much shorter time at the higher range and thereafter magnetizing it.

4. A magnetic article of improved permanent magnet properties composed of an alloy comprising 34 per cent $\pm$ 2 per cent iron, 52 per cent $\pm$ 5 per cent cobalt, and 14 per cent $\pm$ 2 per cent

vanadium produced by heating the alloy to from 800° to 1300° C., cooling substantially below 600° C., forcibly elongating the material in cold condition, thereafter maintaining it between 500° C. and 800° C. for a period of time, cooling it, and thereafter magnetizing it.

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