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IRON-ALUMINUM MATERIALS FOR MAGNETIC APPLICATIONS

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This invention relates to materials to be used for electrical applications and particularly relates to new magnetic materials prepared from iron-aluminum alloys, and to the methods of producing such materials.

There are known in the electrical industry several alloys that are characterized by outstanding magnetic properties and which, consequently, have become widely used in applications where magnetic properties are of importance. The present invention relates in particular to the development of a new class of materials that are useful, in general, for purposes similar to the uses of commercial magnetic iron-silicon alloys and iron-nickel alloys.

The object of this invention is to provide for aluminum-iron alloy sheets having improved magnetic properties, by a double or triple cold rolling process following by intervening anneals and a final anneal under specified conditions, whereby sheets having a double oriented cube on face grain structure are produced.

It is another object of this invention to provide materials that may be used for magnetic applications, which are made only of metals that have a low strategic index.

Still another object of the invention is to provide processes for producing iron-aluminum materials according to the foregoing objects that may readily be practiced with commercially available facilities and that are adaptable in procedure to variations whereby materials with properties tailored for particular applications may be obtained.

I have discovered, and it is on this discovery that the invention is in large part predicated, that upon subjecting ductile iron-aluminum alloys to a series of cold rolling and heat treating operations to produce thin sheets in which the grains are oriented with a cube plane, and thus two easy directions of magnetism, in the sheet surface, and which exhibits outstanding magnetic properties. I have also discovered that control of particular steps in the series of operations may be used to develop particular properties to values over and above those generally obtained in the resultant material. In this manner I have been able to produce, by way of example, iron-aluminum tapes for magnetic applications that exhibit high saturation induction and high induction at low fields and generally are particularly useful for various magnetic applications.

The iron-aluminum products of this invention are obtained as flat sheets, strips or tapes of a thickness on the order of 1 to 18 mils. Other shapes may be produced for particular applications by use of shearing or stamping techniques. For example, for special applications E or L shapes may be stamped or punched from sheets or tapes. Broadly these products are characterized by an induction at a field of 10 oersteds of at least 12,000 gauss, a coercive force of at least as low as 0.5 oersted, and a pulse permeability at a field of 10 oersteds of at least 700. From the data presented hereinafter it will be apparent that the values of the foregoing properties in tapes of this invention are generally improved

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over the indicated levels when the preferred practices are carried out. By way of example, for low remanence tapes of 1 to 6 mils, which are of particular interest for pulse-permeability material, the remanence usually is at least as low as 5,500 gauss, the induction at a field of 10 oersteds is at least 13,500 gauss, and the coercive force is at least as low as 0.5 oersted. Similarly, for tapes of 1 to 6 mils that have been prepared with a view to high inductions the properties are an induction at a field of 10 oersteds of at least 14,500 gauss and a coercive force not exceeding 0.45 oersted. For these latter materials where remanence is not a controlling property, the remanence is on the order of 9,500 gauss.

The products of this invention are obtained by a process involving a plurality of cold reductions of an iron-aluminum alloy plate, or strip, suitably with intermediate heat treatments and a final heat treatment. Three cold rolling operations normally are used. In the first cold roll, a reduction of about 60 to 85 percent is taken. A reduction of about 80 to 97 percent of the then thickness is taken in the second cold roll, while a further reduction equivalent to about 40 to 90 percent of the thickness of the strip from the second cold roll is taken in the third cold roll. Within these general limits particular combinations of reductions may be adopted with a view to the development of characteristics in the final product that may be desired.

After each cold roll the sheet is given an annealing heat treatment under temperature and time conditions to secure certain desired recrystallization structures, as well as to produce stress relief, in a non-oxidizing or reducing atmosphere. More particularly, after the first cold roll the sheet is given a primary recrystallization anneal which comprises heating the sheet to a temperature of from 850° C. to 1100° C. for a period of from about two to three hours at the lower temperatures and about 15 minutes at the highest temperatures. After the second cold roll the sheet is first annealed in the primary recrystallization range, i. e. above 600° C. and suitably at 700° to 850° C., for a few minutes up to about two hours, and then placed in a furnace which is at a temperature sufficient to result in the large grain growth characteristic of secondary recrystallization, i. e. at about 1050° to 1300° C. for one-half to twelve hours. I have found that the heat treatment applied after the third cold roll is particularly important with respect to the magnetic properties of the end product. The heat treatment after the third cold roll includes, normally, an anneal in the primary recrystallization temperature range, that is at 700° to 850° C. or slightly higher for one to ten hours, followed by slowly heating the sheet to a higher temperature and heat-treating the sheet at that higher temperature until the desired properties result. The heating rate may vary from about 5° to 150° C. per hour, and the heat-treating temperature is in the range of 900° to 1250° C. and is maintained for one to twelve hours. While it is not intended to have the invention limited by theory, it is believed that double, or cube-on-face, orientation maximizes during the heat treatment after the third cold roll. By double orientation I mean to indicate an oriented grain structure having an easy direction of magnetism along and perpendicular to the rolling direction. Within the foregoing ranges, it will be apparent that variations can be made with a view to developing special characteristics. For example, selected heating after the third cold roll has been found desirable with respect to obtaining a low remanence. Consequently, where that property is most important, the sheets are heated at 1000° C. and higher.

As will be evident to those familiar with the art of iron-aluminum alloys, a protective atmosphere about the strip or tape should be provided at such times as the

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article is at a temperature, such as above about 600° C., at which deleterious oxidation may occur. By protective atmosphere I intend to indicate any suitable non-oxidizing atmosphere such, for example, as argon or hydrogen.

Iron-aluminum alloys that may be used in practicing the present invention are ductile iron-aluminum alloys. Such alloys generally contain aluminum in an amount of about 3 to 7 percent based on the weight of the alloy. Other alloying constituents and incidental impurities may be present in varying amounts providing that they do not deleteriously interfere with obtaining the described properties in the end products. These alloys conventionally are produced by melting, in a vacuum furnace, a pure iron, such as electrolytic iron, and an aluminum suitably having a purity on the order of 99.00 percent. The melt may be cast in vacuum or under a protective atmosphere such, for example, as argon, to prevent the uncontrolled introduction of impurities from the atmosphere. The ingot thus produced is rolled at a temperature above the recrystallization temperature to a thickness of about 0.4 to 0.65 inch, suitably about 0.5 inch, prior to the first cold rolling step above mentioned.

The following examples, demonstrating the production of tapes for magnetic applications, will serve to illustrate the invention. These examples and the details thereof are given by way of illustration and not by way of limitation.

EXAMPLE I

An alloy ingot is prepared by melting commercially available electrolytic iron and high-purity aluminum, in a weight ratio of 94.5 parts of iron to 5.5 parts of the aluminum, in an induction furnace under argon at atmospheric pressure, and then casting the melt. The resulting ingot is hot rolled, at a temperature of about 650° C. to a thickness of 0.50 inch.

The one-half inch plate is then cold rolled to a thickness of 0.125 inch and annealed at 1000° C. for one hour. The annealed material is further cold rolled to a thickness of 0.010 inch after which the strip is annealed at 750° C. After one hour at the annealing temperature, the strip is cooled under argon and is then placed in a furnace at 1200° C. and is maintained at that temperature for two hours to result in a secondary recrystallization structure exhibiting large grains.

From the secondary recrystallization step, the strip is further cold rolled to a thickness of about 0.003 inch. The resulting tape is given a primary recrystallization anneal at 750° C. for about five hours after which its temperature is raised to 950° C. at a rate of about 100° C. per hour. The tape is maintained at 950° C. for about eight hours.

Typical properties of 5.5 percent Al-Fe tape made according to the foregoing example, and obtained from a D. C. hysteresis loop on the material, are as follows:

Table I

B_1 (gauss)-----	9,200
B_{10} -----	16,900
B_r -----	9,400
H_c -----	0.45
Resistivity (microhm-cm.)-----	65

From these data it is evident that such tapes may be used where high saturation induction, high induction at low fields and a high resistivity are of importance. Typical uses include magnetic amplifiers (especially power stages) and transformer applications. The high resistivity means lower core losses or, if desired, the use of thicker tapes with the same core loss as with materials presently available.

For certain uses, it is advantageous to have a material that exhibits a low remanence. Such materials may also be produced in accordance with my invention, for instance as shown in the following example.

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EXAMPLE II

An iron-aluminum strip of 0.010 inch thickness is produced by a procedure exactly as used in Example I to produce the 0.010 inch strip. The strip is then subjected to a heat treatment identical to that applied to the 0.010 strip of Example I. The strip is then cold rolled to a tape of a thickness of three mils. The tape is placed in a furnace at 750° C. and the temperature raised at a rate of about 40° per hour to 1100° C. The tape is held at 1100° C. for three hours after which it is cooled in a cooling chamber under argon to room temperature.

Typical properties for tapes produced according to the steps of Example II are as follows:

Table II

B_{10} -----	18,700
B_r -----	5,500
H_c -----	0.30
ΔE at 2000 gauss-----	5,600
ΔE at 4000 gauss-----	5,000
ΔE at 6000 gauss-----	3,750
ΔH at 8000 gauss-----	3,080
ΔH at 10,000 gauss-----	2,500
$H=10$ -----	1,020

These data show that the product is characterized by the low remanence of 5,500; furthermore, the product is obtained without simultaneously causing a deleterious drop in the induction at fields of 2 to 10 oersteds. These tapes obviously can be used for such applications as pulse or current transformers, relays, meters and various A.-C. instruments.

The invention as represented by the procedure of Example II is considered to be particularly outstanding because in commercial applications heretofore, a low remanence has been produced by artificial means in cores using known Fe-Si and Fe-Ni alloys. For example, in pulse transformer applications, low remanence has been obtained by use of an introduced air gap. In A.-C. instruments special self-demagnetizing shapes have been used to secure low remanence. Those practices lower the permeabilities of the materials and obviously involve additional fabricating costs and problems. In contrast thereto, materials exhibiting low remanence are obtained by virtue of the present invention without need for artificialities and without encountering the costs and problems characteristic of the prior art.

It has been pointed out hereinbefore that properties of the resulting material are dependent upon the particular treatment used. Within the broad ranges of cold reductions and temperatures and times of heat treatment there are more narrow ranges that are used to advantage for developing a material for a predetermined end use. By way of illustration, in producing materials in accordance with each of Examples I and II, suitable intermediate ranges of reduction that may be taken in each of the cold rolling operations are as follows: first cold roll, 70 to 80 percent; second cold roll, 85 to 95 percent; third cold roll, 60 to 80 percent. Suitable ranges for the heat treating steps are as follows: annealing after the first cold roll, 950° to 1050° C. for one-half to two hours; heat treatment following the second cold roll, annealing at 725° to 800° C. for 30 minutes to two hours followed by secondary recrystallization, after first being cooled, at 1150° to 1250° C. for one-half to four hours. The procedures of Examples I and II are different in the heat treatment used after the third cold rolling operation. Where tapes are to be produced in accordance with Example I, the tape, after the third cold reduction, may be given a primary recrystallization anneal at 725° to 775° C. for about three to seven hours, after which its temperature is raised at about 75° to 125° per hour to about 900° to 950° C. and is held at that temperature for five to twelve hours. On the other hand, to produce a low remanence product as in Example II, the tape, after the third cold reduction, is placed in a furnace at a temperature of about 725° to 775° C. and heated to a temperature of about

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1050° to 1150° C. at a rate of 25° to 50° per hour and is held at the final temperature two to four hours.

EXAMPLE III

To demonstrate the effect on the properties produced by changes in a given step, four tapes of 3 mil thickness each were produced by an identical procedure up to the heat treatment used after the third cold reduction. Each tape was then subjected to the same primary anneal. The properties of one of the tapes were then measured. The other three tapes were each subjected to a different additional heat treatment. The details of the heat treatments and of the properties of the resulting materials are summarized in the following table:

Table III

Treatment	1 750° C. for 5 hours	2 Reannealed by heating from 750° to 950° C. at a rise of 100° per hour. Held 3 hours at 950° C.	3 Reannealed by heating from 750° to 1,100° C. at a rise of 100° per hour. Held 3 hours at 1,100° C.	4 Reannealed by heating for 5 hours at 1,200° C.
B_{10}	16,900	16,400	15,700	14,500
B_r	9,400	8,400	8,500	4,800
H_c	0.45	0.3	0.3	0.3
ΔB				
ΔH at $H=10$	750	800	1,020	970

From these data it is evident that the properties may be affected by the heat treatment used. The best results from the pulse permeability standpoint are obtained with treatment 3 since a high

$$\frac{\Delta B}{\Delta H}$$

results along with a low remanence and high induction at 10 oersteds. The procedures of 1 and 2 result in high saturations albeit with a high remanence and thus are of importance where a high saturation is needed and a high remanence is not deleterious. And finally, procedure 4 results in low remanence though the saturation value has, in part, been sacrificed. It is apparent that for the best remanence value, procedure 3 or 4 would be chosen; procedure 4 being preferred if the saturation value is not critical, and 3 being better if it is.

To this point, the invention has been described with respect to the use of a schedule of three cold rolling operations each being followed by a heat treatment. It is to be understood that those are, as presently advised, thought to be the best embodiments of the invention. However, the materials resulting, by way of example, at the point just prior to the third cold reduction also are novel and useful materials for magnetic applications. As evidence thereof, in the following table I have accumulated data on the properties of the 13 mil strip that was produced according to the following schedule: a 1/2 inch hot rolled plate of Fe-5.5 percent Al is cold reduced to a thickness of 0.125 inch and then annealed at 1000° C. for one hour. The strip is then cold rolled to 13 mils, annealed a 750° C. for one hour and then cooled. The cooled strip is then heated at 1200° C. for two hours to develop a large grain structure. The properties of the resulting material are:

Table IV

B_{10}	13,600
B_r	3,600
H_c	0.18
at 2000 gauss	4,200
at 4000 gauss	3,320
ΔB at 6000 gauss	3,010
ΔH at 8000 gauss	2,910
at 10,000 gauss	1,800
$H=10$	1,000

From these data it is apparent that the 13 mil product has a very low remanence value coupled with a low

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coercive force. Moreover, the pulse permeability also is very satisfactory. Accordingly the 13 mil product can be used where those properties are desired and the saturation induction at 10 oersteds is not of controlling significance.

From the foregoing it can be seen that this invention provides a new class of materials that may be used for magnetic applications. The high electrical resistivity of the iron-aluminum gives them an advantage over the corresponding iron-silicon or iron-nickel alloys in that lower core losses may be obtained. This high resistivity is of special interest in higher frequency applications such as 400 cycles and above. Those products exhibiting a low remanence are superior to the Fe-Si and Fe-Ni products because with my Fe-Al products the actual permeability of the material is retained in service conditions. The mechanical properties of the Fe-Al tapes also commend their commercial adoption. For example, these materials are ductile and can be cold rolled to thin gauges without difficulty. As they are not highly strain sensitive, as compared with Fe-Ni alloys, they are less susceptible to property degradation through handling. The products also are of lower unit weight than Fe-Si or Fe-Ni materials, and hence may be of particular interest for airborne applications.

Still another outstanding advantage of these new Fe-Al products is the fact that they may be insulated by a simple low temperature air anneal, for example by heating the tape at 200° to 400° C. for about 30 minutes. Such treatment causes an insulating Al_2O_3 coating to form on the surface; the coating is ductile and is essentially unaffected by subsequent handling operations. The coating is less than 10^{-5} inch in thickness. In providing Fe-Si and Fe-Ni materials with insulation, it has been accomplished by a procedure involving a scrupulous cleaning of the metal followed by applying the coating and then baking it. That necessitates additional expense, careful handling to avoid destroying the coating, and a loss in use of available space in core manufacture with those materials. Accordingly, the advantage in my invention is evident.

According to the provisions of the patent statutes I have explained the principle of my invention and have described what I now believe to represent its best embodiment. However, I desire to have it understood that the invention may be practiced otherwise than as specifically described.

I claim as my invention:

1. A recrystallized iron-aluminum strip for magnetic applications which consists essentially of a ductile iron-aluminum alloy consisting essentially of 3 to 7 weight percent of aluminum and the balance iron, said strip being about 1 to 18 mils thick and having the following properties as determined from a D. C. hysteresis loop on the strip: remanence not exceeding 9,500 gauss, an induction at 10 oersteds of at least 12,000 gauss and a coercive force not exceeding 0.50 oersted.

2. A recrystallized, doubly oriented iron-aluminum tape for magnetic applications which consists essentially of a ductile iron-aluminum alloy consisting essentially of 3 to 7 percent by weight of aluminum and the balance iron, said strip being about 1 to 6 mils thick and having the following properties as determined from a D. C. hysteresis loop on the tape: remanence not exceeding 5,500 gauss, an induction at 10 oersteds of at least 13,500 gauss, and a coercive force not exceeding 0.5 oersted.

3. A recrystallized, doubly oriented iron-aluminum tape for magnetic applications which consists essentially of a ductile iron-aluminum alloy consisting essentially of 3 to 7 weight percent of aluminum and the balance iron, said strip being about 1 to 6 mils thick and having the following properties as determined from a D. C. hysteresis loop on the tape: an induction at 10 oersteds of at least 14,500 gauss, and a coercive force not exceeding 0.45 oersted

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4. That method of producing a doubly oriented iron-aluminum strip for magnetic applications which comprises cold rolling a hot rolled ductile iron-aluminum plate which consists essentially of 3 to 7 weight percent of aluminum and the balance iron to reduce said plate by 60 to 85 percent of its thickness, heating the resulting strip at above the recrystallization temperature to stress relieve it, cold rolling the stress-relieved strip to reduce it by 80 to 97 percent of its thickness, and then heating the resulting strip at a temperature and for a time sufficient to develop a large grain structure.

5. That method of producing a doubly oriented iron-aluminum tape which comprises cold rolling a hot rolled ductile iron-aluminum plate of a thickness of about 0.4 to 0.65 inch which consists essentially of 3 to 7 weight percent aluminum and the balance iron to reduce said plate by 60 to 85 percent of its thickness, stress relieving the resulting reduced strip, then cold rolling the stress-relieved strip to reduce it by 80 to 97 percent of its thickness, then heating the strip at an elevated temperature above its recrystallization temperature to cause grain growth, cold rolling the resulting strip to reduce it by 49 to 90 percent of its thickness, and then heating the resulting tape at a temperature above its recrystallization temperature to develop cube-on-face orientation in the resulting grain structure.

6. That method of producing doubly oriented iron-aluminum tapes which comprises cold rolling a hot rolled plate of a thickness of about 0.4 to 0.65 inch and which consists essentially of about 3 to 7 percent by weight of aluminum and the balance substantially all iron, by 70 to 80 percent of its thickness, annealing the resulting strip at a temperature of 850° to 1100° C. to stress relieve said strip, cold rolling the annealed strip to reduce it by 85 to 95 percent of its thickness, annealing the strip at 700° to 850° C., cooling the strip, then heating it at a temperature of 1050° C. to 1300° C. for one-half to twelve hours to cause grain growth, cold rolling the strip to reduce it by 60 to 80 percent of its thickness, and then heating the resulting tape at a temperature above its recrystallization temperature to cause a cube-on-face orientation of the resulting grain structure.

7. That method of producing iron-aluminum tapes which comprises cold rolling a hot rolled plate of a

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thickness of about 0.4 to 0.65 inch and which consists essentially of about 3 to 7 percent by weight of aluminum and the balance substantially all iron, by 70 to 80 percent of its thickness, annealing the resulting strip at a temperature of 850° to 1100° C. to stress relieve said strip, cold rolling the annealed strip to reduce it by 85 to 95 percent of its thickness, annealing the strip at 700° to 850° C., then heating it at a temperature of 1050° C. to 1300° C. for one-half to twelve hours to cause grain growth, cold rolling the strip to reduce it by 60 to 80 percent of its thickness, heating the tape at a temperature of 700° C. to 850° C. for about 1 to 10 hours, then raising the temperature of the tape at a rate of about 5° C. to 150° C. per hour to a temperature of 900° to 1250° C. and maintaining that temperature for 5 to 12 hours.

8. That method of producing iron-aluminum tapes for magnetic applications which comprises cold rolling a hot rolled plate of a thickness of about 0.4 to 0.65 inch and which consists essentially of about 3 to 7 percent by weight of aluminum and the balance substantially all iron, by 70 to 80 percent of its thickness, annealing the resulting strip at a temperature of 950° to 1050° C. to stress relieve said strip, cold rolling the annealed strip to reduce it by 85 to 95 percent of its thickness, annealing the strip at 725° to 800° C., cooling the strip, then heating it at a temperature of 1150° to 1250° C. for one-half to four hours to cause grain growth, cold rolling the strip to reduce it by 60 to 80 percent of its thickness, then heating the resulting tape at a temperature of from 725° C. to 775° C. for 3 to 7 hours, then raising the temperature from the temperature of about 725° to 775° C. at a rate of 25° to 50° C. per hour to a higher temperature of about 1050° to 1150° C. and maintaining said higher temperature for about two to four hours.

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