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METHOD OF HOT ROLLING NICKEL-IRON MAGNETIC SHEET STOCK

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Nickel-iron alloys in which the named elements exist in about equal quantities have hitherto been made into relatively thin magnetic sheet stocks. These sheet stocks are characterized by relatively rectangular hysteresis loops and by a grain orientation responding to the designation (100) [001] by Miller's Indices. This orientation is sometimes called "cubic texture." The stocks find utility, inter alia, in the manufacture of cores for magnetic amplifiers and the like.

The fundamental object of the invention is the provision of nickel-iron magnetic sheet stocks having greatly improved magnetic qualities, and processes which can be depended upon to give a product having the improved characteristics.

This basic object and others which will be set forth specifically hereinafter are attained in that product and by the use of those procedures of which certain exemplary embodiments will now be described.

1. GENERAL DISCUSSION

One of the specific objects of the invention may be stated as the consistent provision of a material of lower coercive force and a higher degree of rectangularity in thicknesses approximately in the range of ¼ to 14 mils.

The products of this invention, measured in terms of magnetic properties under D.C. magnetization have:

(a) A magnetic induction exceeding 14.75 kilogausses at 1 oersted,

(b) A ratio of residual to peak induction not less than .96 at a peak magnetizing force of 1 oersted, and

(c) A coercive force of not more than about .09 oersted for material one to two mils thick, not more than about .15 oersted for materials 4 to 6 mils thick, and not more than about .25 oersted for thicker material up to 14 mils, measured at a peak magnetizing force of 1 oersted.

In terms of A.C. magnetic properties measured by CCFR tests according to the AIEE Standard 432, the materials of this invention have:

(a) A magnetic induction exceeding 14.75 kilogausses at a peak magnetizing force of 1 oersted.

(b) A ratio of residual to peak induction not less than .96, and

(c) A value of H_1 and ΔH as follows:

Thickness, Mils	H_1 Oersted, max.	ΔH Oersted, max.
1	.20	.022
2	.23	.025
4	.28	.030
6	.30	.035

The materials of this invention are made by processes not involving a final anneal in excess of 2200° F. They have the (100) [001] texture with a matrix or primary grain size not greater than 5.0 ASTM at 100×. The primary grains occupy by far the greater part of the surface area of the material. Less than 10% of the area occupied by secondary grains will be made up of grains exceeding 2 mm. in diameter at a magnification of 1×.

So far as is known the combination of qualities set forth above has not hitherto been attained in magnetic

sheet stocks containing subtantially equal parts of nickel and iron.

Researches have shown that the magnetic induction (B_m) is affected by the gross composition of the product and by the grain orientation. The ratio of residual to peak induction (B_r/B_m) which is indicative of the squareness of the hysteresis loop is dependent basically on the grain orientation of the product. The coercive force (H_1), which determines the narrowness of the hysteresis loop, is dependent upon a number of factors including the annealing temperature and the impurities contained in the material. It may be stated that a hysteresis loop which is narrow in the horizontal direction is desired. ΔH is the function of the slope of the magnetization curve and is dependent upon grain orientation, grain size and any residual stresses there may be in the product. A relatively small grain size is desirable.

It will be seen from the above that the attainment of the objects of the invention is dependent upon the coaction of a number of factors not only of composition but also of treatment in the production of the material.

2. COMPOSITION

In the practice of the invention an alloy steel is made by any suitable refining or melting technique and having the following initial composition:

Ni—about 47.5% to about 48.5% or roughly 47% to 49%.

C—about .005% to about .019% but in any event less than about .035%.

Mn—from about .30% to about 1.0%, and preferably in the range of about .35% to about .70%.

P—not greater than about .005%.

S—from about .0030% to about .010% but preferably in the range of about .0040% to about .0075%.

Si—from about .30% to about .50%, but preferably in the range of about .35% to about .45%.

Al—from about .010% to about .035% but preferably from about .015% to about .025%.

Fe—the balance of the alloy excepting for residual impurities in trace amounts. The quantity of iron should be approximately equal to the quantity of nickel in the alloy. Residuals should be kept as low in quantity as practicable.

The sulfur has an effect on controlling the grain growth in the product, and tends to inhibit secondary recrystallization. Enough sulfur should be present initially to prevent premature secondary growth which would increase ΔH and lower the B_r/B_m ratio. By the termination of the processing, however, enough sulfur must be removed to obtain low H_c and H_1 values.

The control of the total alloying additions should be strict if high saturation (high B_m values) is to be maintained.

It has been found that the phosphorus present has an important effect in controlling the ultimate orientation of the grains in the product. Excessive phosphorus impairs the orientation even of the primary grains, and this would result in low B_m and B_r/B_m values and an increase in ΔH.

Aluminum has an effect upon grain growth in the product and helps to prevent secondary growth. The best B_r/B_m values are attained with an aluminum content of about .020%, which is regarded as optimum.

The silicon permits better control of oxygen during the melting procedure. In the amounts indicated it tends to reduce B_m, B_r/B_m and H_1. Too much silicon will promote premature secondary grain growth and tends to reduce the B_r/B_m value.

Nickel when used as described, and especially in the preferred range, provides the best magnetic saturation.

The carbon should be restricted as indicated.

The melt analysis as set forth above is important because it is necessary to the practice of the invention that the alloy contain the stated elements in the stated proportions during the processing and particularly during the hot rolling and hot coiling treatments hereinafter described. The final product in sheet or strip form will be found upon analysis to contain the same elements in substantially the same proportions excepting that a change will occur in the content of sulfur and a minor change may occur in the content of carbon. The final sulfur, as will later be shown, should not be above about .0020%. Some carbon may be lost during processing; but the procedure herein taught does not include a decarburizing step as known in the art, and the dry atmosphere of the final anneal, and an intermediate anneal if practiced, cannot under the conditions described effect any large reduction in carbon content. While somewhat lower carbons are frequently achieved, the value of .035% previously given can still be regarded as the maximum both for the initial material and for the final product. A substantially lower carbon content is usually the result of using a low carbon melt.

3. HOT ROLLING

A melt of the analysis set forth above is teemed into ingot molds, preferably molds having cross sectional dimensions of 16" x 29", but without limitation since other sizes may be employed. The molds will be stripped from the ingots, and the ingots are placed in soaking pits and brought to a temperature of about 2200° F. to 2300° F., preferably about 2250° F.

When the ingots are uniformly at a temperature within the indicated range they are preferably hot rolled in known ways to slabs which are 3" to 6" in thickness. The slabs are cooled and conditioned in known ways to remove scale and other defects.

Instead of slabbing the ingots as indicated, it is possible to roll the ingots directly to an intermediate hot rolled gauge as later specified; but if this is done precautions should be taken to see that the temperature of the metal will not exceed the ranges set forth in the next following paragraph when the metal is at comparable thicknesses

When the preferred procedure is followed, the conditioned slabs are reheated to a temperature within the range of about 1850° F. to about 2050° F. but preferably in a narrower range of about 1900° F. to about 2000° F. The slabs are then roughed down to a thickness of about .450" and then finished in finishing stands to a thickness in the range of about .125" to about .180" but preferably from about .140" to about .155". The finishing temperature should lie within the range of about 1300° F. to about 1500° F. but preferably in the narrower range of about 1360° F. to about 1420° F. The hot bands formed in this way will be coiled at a temperature not exceeding about 1100° F.

The temperature requirements set forth are applicable whether or not the routing includes an intermediate slabbing treatment, and the temperature requirements must be adhered to strictly. The effect of finishing and coiling the hot bands at too high a temperature will result in a grain size in the hot band which is too large to permit the attaining of the objects of this invention. If the hot band is finished at too low a temperature there will be apparent in it an effect of too much cold reduction, which will interfere with the desired grain orientation.

4. COLD ROLLING

After pickling to remove scale, the hot rolled band will be cold reduced to a final thickness of from ¼ to 14 mils. If the cold rolling can be done to a chosen final gauge without intermediate annealing, this is desirable. For example, it is possible to cold roll the pickled hot band to a thickness of about .025" on an ordinary 4-high cold mill; and the remainder of the cold rolling will then be accomplished on a mill of a type designed to do extremely flat rolling with small working rolls. An example

of such a mill, but without limitation, is the well known Sendzimir mill having working rolls not over about 1¼" in diameter. Reductions may be made to a final gauge of about .014" to about .002" without intermediate annealing. It is an advantage of the process that no anneal following the hot rolling but preceding the cold rolling is required.

When a final thickness of 1 mil or less is desired, an intermediate anneal will usually be practiced. The strip thickness at this anneal should be about 50 to 100 times the final thickness. The anneal is conducted at about 1600° F. in a reducing atmosphere for a time of about 1 to 2 minutes.

When the material has been reduced to gauge it may then be slit into strips of the desired width, and cores may be fabricated from the strips by coating them with an annealing separator such as M_gO, and winding them to a toroidal core formation.

5. FINAL ANNEAL

The cores formed as above described are subjected to a final anneal in a temperature range of about 1800° F. to about 2200° F. for about one to four hours at temperature in dry hydrogen, i.e., hydrogen having a dew point not over about −10° F.

This anneal serves to recrystallize the material and to cause annealing twins to be adsorbed so that a uniformly fine grain structure results with the cubic or (100) [001] texture. A sudden grain growth or secondary recrystallization will occur if the annealing temperature is higher than appropriate for the amount of sulfur present or the degree of cold reduction. Secondary grains over 2 mm. in diameter must be avoided in amounts exceeding 5% to 10% of the area of the material to retain a satisfactory level of ΔH.

There are optimum amounts of sulfur which vary with the temperature of the final heat treatment from about .003% for a heat treatment at about 1800° F. to about .006% for a heat treatment within the range of about 2150° F. to about 2200° F. At the same time the final heat treatment in dry hydrogen results in the removal of sulfur which permits the attainment of a high degree of the cubic texture. Samples of the steel tested for sulfur content after the described final anneal were found to contain .0010% to .0020% sulfur.

Example

A 27,000 pound heat of nickel-iron alloy was melted in an arc furnace and was cast into 16" x 29" ingots weighing 9,000 pounds each and having a composition as follows:

	Percent
Ni	48.2
C	.009
Mn	.46
P	.002
S	.0067
Si	.40
Al	.016
Fe	Balance

The ingots were heated to 2275° F. and were rolled into slabs 5" thick, which were then colled.

The slabs were ground and reheated to 1920° F. to 1945° F. and were roughed into bars .45" thick. The bars entered the finishing mills at 1550° F. to 1580° F. and were rolled to .145" at a finishing temperature of 1385° F. to 1400° F. The hot rolled intermediate gauge product was cooled with water on a run-out table and then was coiled at a temperature of 980° F to 1030° F.

After pickling, the coils of the intermediate gauge hot rolled product were cold rolled to .025" using 16" diameter working rolls. Thereafter the material was carried down to a thickness of .002" using a mill having 1.2" diameter working rolls.

The sheet gauge material was slit to form strips ¼" wide, which strips were then coated with dry magnesia using a light film of lubricating oil to cause the magnesia to adhere to the strips. The strips were wound into toroidal cores having an inside diameter of 1" and an outside diameter of 1¼". The cores were annealed in a muffle using dry hydrogen as the annealing atmosphere, i.e., hydrogen having an exit dew point below −60° F. Three batches of the cores were annealed at different temperatures as shown in the table below; but the time at temperature in each instance was two hours.

The results of CCFR tests were as follows:

Anneal Temperature, ° F.	B_m K$_g$a.	B_r/B_m	H_1 Oer.	ΔH Oer.
2,100	15.45	.979	.185	.0164
2,150	15.37	.978	.181	.0153
2,200	15.42	.978	.171	.0158

Since the final anneal was performed after the formation of the cores, it will be clear that the final anneal could be performed by a customer for the material.

Modifications may be made in the invention without departing from the spirit of it. The invention having been described in an exemplary embodiment, what is claimed as new and desired to be secured by Letters Patent is:

1. A process for the production of nickel-iron alloy sheet stock having improved magnetic properties, which process includes the steps of forming a nickel-iron alloy consisting essentially of substantially equal parts of nickel and iron, less than about .035% carbon, and sulfur between .0030% and .010%, other alloying ingredients being minor in amount, casting the said alloy into ingots, hot rolling the said ingots to an intermediate gauge of about .125" to about .180" under such conditions that the hot rolling will be commenced with the ingots at temperatures from about 2200° F. to about 2300° F., that the temperature of the material when at a slab thickness of substantially 3" to 6" and ready for further rolling will range from about 1850° F. to about 2050° F., that the hot rolling is finished at temperatures of about 1300° F. to about 1500° F. and that the hot rolled material is coiled at a temperature not greater than about 11000° F., cold rolling the material to a final gauge of about ¼ to about 14 mils and then subjecting the cold rolled material to an anneal in dry hydrogen at a temperature of substantially 1800° F. to substantially 2200° F. for about one to four hours.

2. The process claimed in claim 1 wherein as a result of the said anneal, the final sulfur in the said sheet stock is not greater than about .0020%.

3. The process claimed in claim 2 including an anneal intermediate the said cold rolling at a temperature of substantially 1600° F. in a reducing atmosphere for a time of about 1 to about 2 minutes.

4. The process claimed in claim 2 wherein the initial composition of the alloy consists essentially of the following ingredients in substantially the proportions stated:

	Percent
Ni	47 to 49
C	<.035
Mn	.30 to 1.0
P	<.005
S	.0030 to .010
Si	.30 to .50
Al	.010 to .035
Fe	Balance [1]

[1] Excepting for residual impurities in trace amounts.

5. The process claimed in claim 2 wherein the initial composition of the alloy consists essentially of the following ingredients substantially in the proportions stated:

	Percent
Ni	47.5 to 48.5
C	.005 to .019
Mn	.35 to .70
P	<.005
S	.0040 to .0075
Si	.35 to .45
Al	.015 to .025
Fe	Balance [1]

[1] Excepting for residual impurities in trace amounts.

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DAVID L. BECK, *Primary Examiner.*