

July 9, 1968

D. M. KOHLER

3,392,063

GRAIN-ORIENTED IRON AND STEEL AND METHOD OF MAKING SAME

Filed June 28, 1965

3 Sheets-Sheet 1

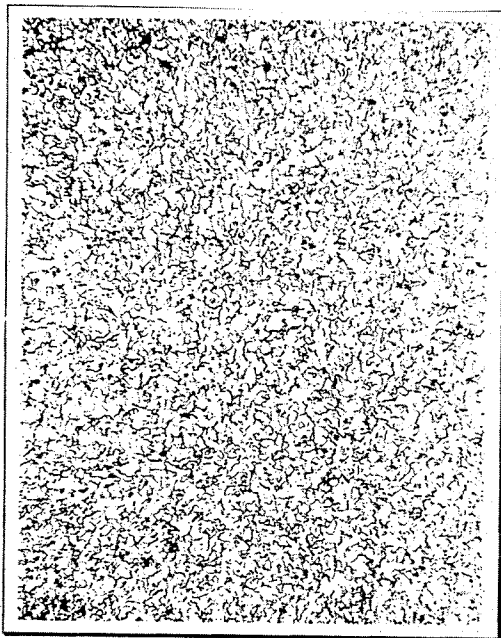


Fig. 1

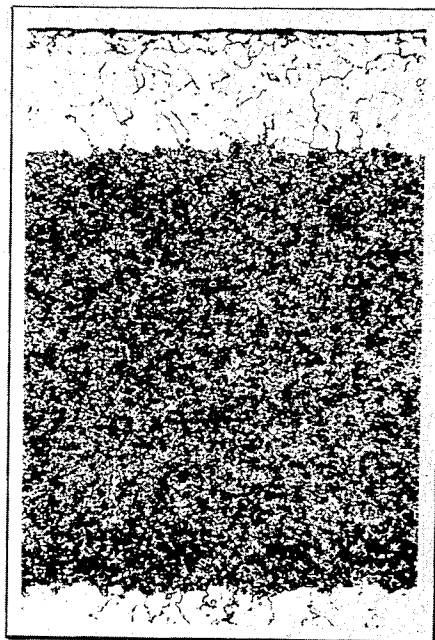


Fig. 2

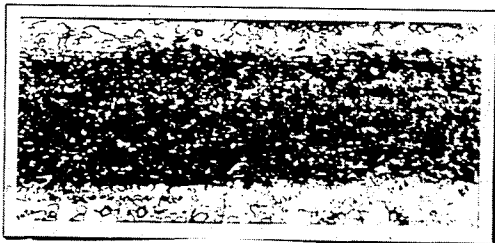


Fig. 3

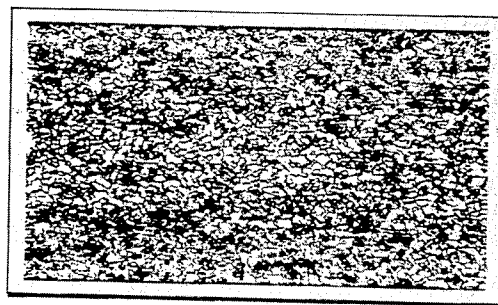


Fig. 5

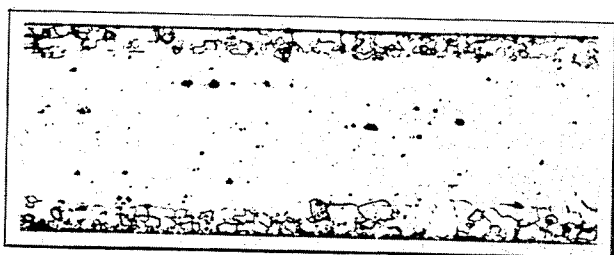


Fig. 4

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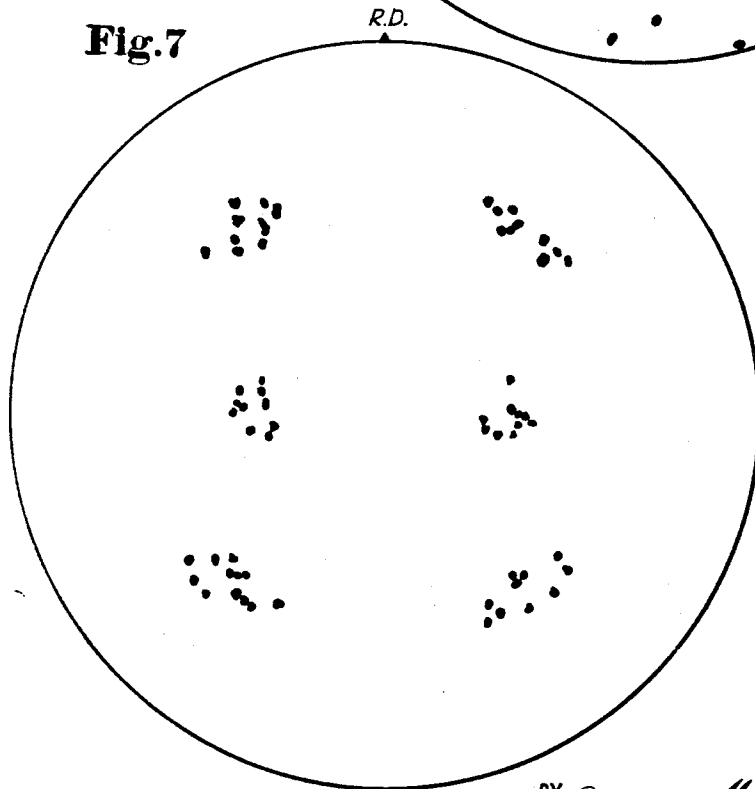
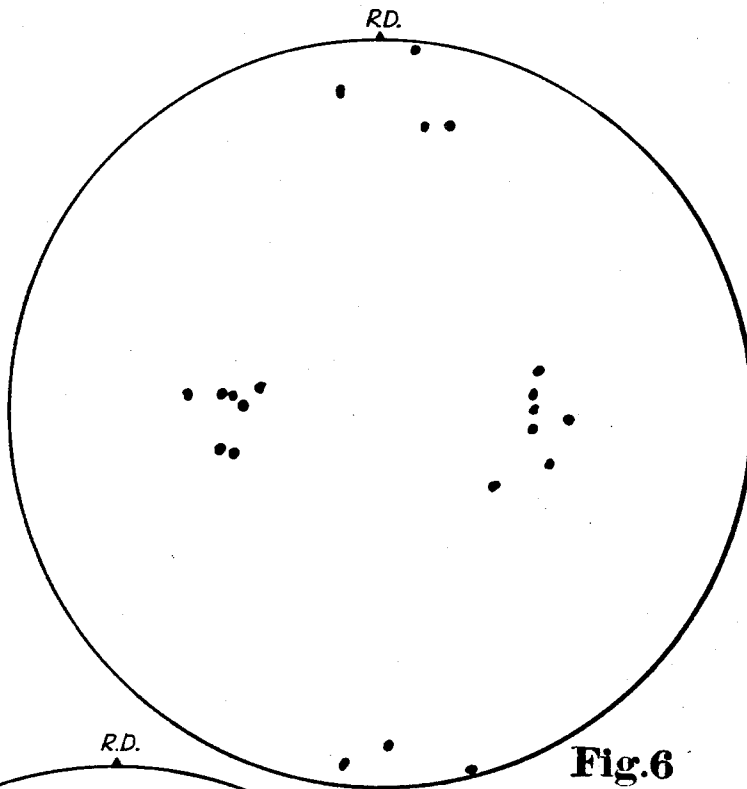
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GRAIN-ORIENTED IRON AND STEEL AND METHOD OF MAKING SAME

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3 Sheets-Sheet 2



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GRAIN-ORIENTED IRON AND STEEL AND METHOD OF MAKING SAME

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3 Sheets-Sheet 3

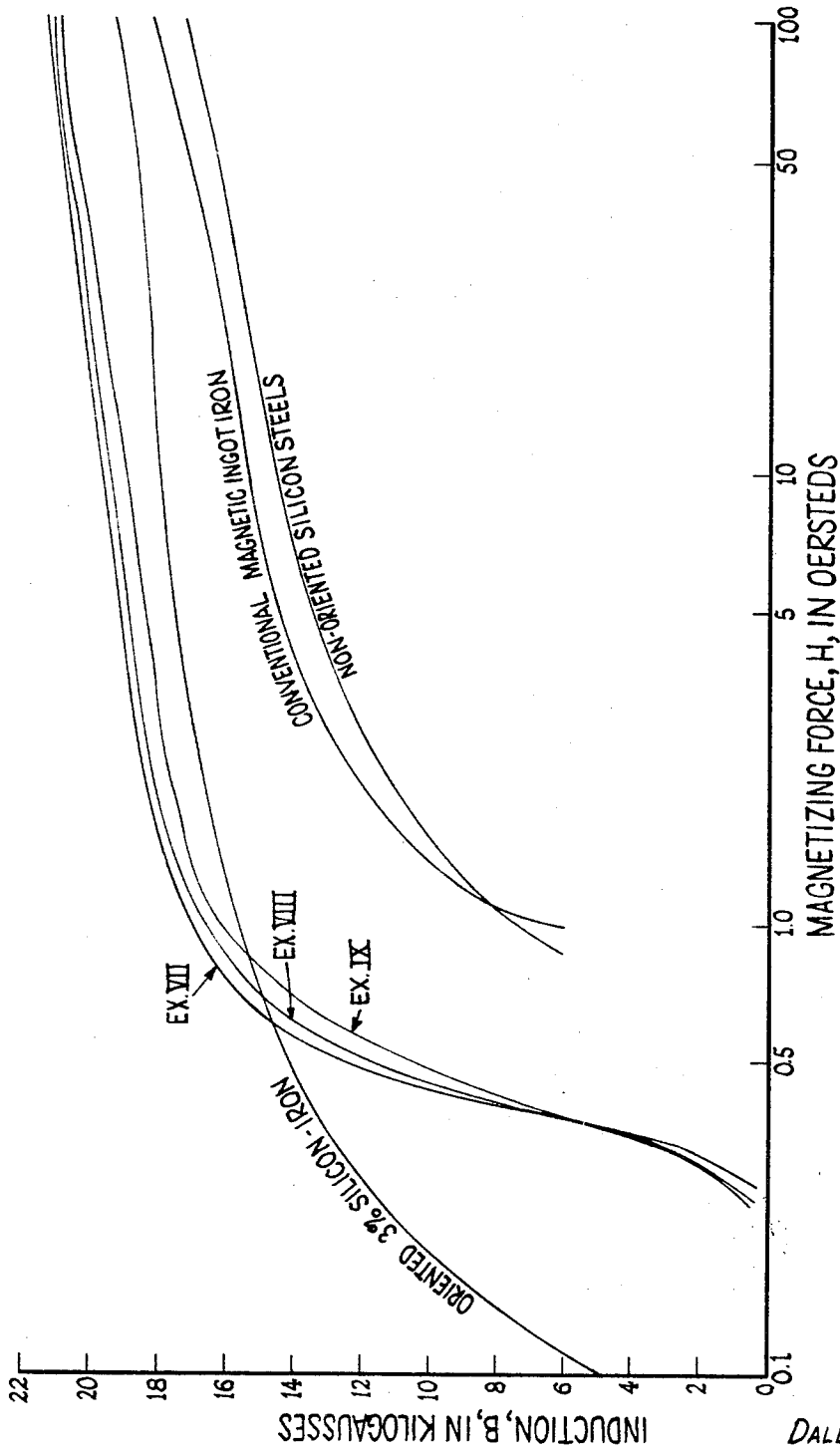


Fig. 8

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3,392,063

## GRAIN-ORIENTED IRON AND STEEL AND METHOD OF MAKING SAME

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Filed June 28, 1965, Ser. No. 467,228

12 Claims. (Cl. 148—12)

The invention relates to a method of producing oriented iron-base alloys and more particularly to a method of making preferred crystallographic orientations in ingot iron and low carbon steel, and the products made thereby.

It has been recognized that all metals and their alloys have some degree of preferred orientation when rolled and usually a different orientation when they are cold worked and recrystallized. When commercial iron and low carbon steels are rolled into sheets or strips and recrystallized they display a weakly developed cubic orientation designated as (100) [011] by Miller's indices. This orientation has no particular value in the present applications of ingot iron and low carbon steels.

Attempts have been made to develop more favorable orientations in this material. For example, the initial chemistry of the melt has been varied by the addition of materials tending to inhibit primary grain growth. Other methods involving critical strain have been used, entailing varying the method and amount of rolling and heat treatment. Nevertheless prior to this invention the preferred orientations in the final product were found in only a relatively small number of the grains, perhaps up to as much as 25% of the total grain structure.

Applicant has discovered that ingot iron and low carbon steel can be made to have at least two highly preferred crystallographic orientations which impart useful properties to a polycrystalline sheet or strip. These textures are strong in that more than half of the total grain structure will have the preferred textures.

By ingot iron and low carbon steel is meant ferrous materials which by ladle analysis contain up to about 0.10% carbon, 0.01 to 0.40% manganese (preferably about 0.03 to about 0.12%), up to about 0.05% sulfur, up to about 0.03% phosphorus, up to about 0.25% copper, the balance being iron except for normal amounts of those impurities incident to the manufacture of such material. The ingot iron and low carbon steel may also contain a small amount of aluminum. Silicon may also be present as a deoxidizer in small amounts, or additions up to about 1.8% may be made to increase the volume resistivity of the product when it is used for magnetic properties. The amount of silicon added may be consistent with that found in hitherto known non-oriented silicon steels, and is dictated by the phase boundary between alpha iron and gamma iron. The carbon and manganese contents will determine whether the material is classified as ingot iron or low carbon steel, the latter usually containing upwards of 0.05% carbon and 0.20% manganese.

The two highly preferred crystallographic orientations which ingot iron and low carbon steel may be made to have in accordance with the teachings of this invention are classified by Miller's indices as (110) [001] and (112) [110]. The (110) [001] orientation is known as cube-on-edge. As will be described hereinafter, material with this orientation made from ingot iron and low carbon steel in accordance with the teachings of this invention will have some magnetic properties substantially as good as or better than those found in 3% oriented silicon-iron presently in commercial use. Heretofore it has been impossible to obtain highly oriented cube-on-edge

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low carbon steel or ingot iron without the use of at least 2% silicon.

The (112) [110] orientation is a form of cube-on-corner texture never heretofore obtained in substantial proportions in ferrous material. As will be described hereinafter material having this orientation and made in accordance with this invention will have good forming properties.

It is therefore an object of the present invention to provide a low carbon iron or steel having a high degree of (110) [001] orientation.

It is an object of the present invention to provide a low carbon iron or steel having a highly developed (112) [110] orientation.

It is an object of the present invention to provide a method for making highly preferred crystallographic orientations in ingot iron and low carbon steel.

It is an object of the present invention to provide a method whereby ingot iron and low carbon steel may be made to have a (110) [001] orientation and wherein more than half of the total grain structure will be characterized by this orientation.

It is an object of the present invention to provide ingot iron and low carbon steel with a cube-on-edge orientation and characterized by superior magnetic properties.

It is an object of the present invention to provide ingot iron and low carbon steel having a (112) [110] orientation and wherein more than half of the total grain structure is characterized by this orientation.

It is an object of the present invention to provide ingot iron and low carbon steel having a (112) [110] orientation and characterized by good formability.

These and other objects of the invention which will be set forth hereinafter or will be apparent to one skilled in the art upon reading these specifications are accomplished by a series of process steps of which certain exemplary embodiments will now be described. Reference is made to the accompanying drawings wherein:

FIG. 1 is a photomicrograph at a magnification of 50× showing the preferred uniform fine-grained structure of the hot rolled material of the present invention.

FIG. 2 is a photomicrograph at a magnification of 50× showing the hot rolled material of the present invention with undesirably large grains at the surfaces.

FIG. 3 is a photomicrograph at a magnification of 100× showing a hot rolled material in which large surface grains such as those illustrated in FIG. 2 are not refined and persist after rolling and decarburization.

FIG. 4 is a photomicrograph at a magnification of 100× showing a hot rolled material in which large surface grains are not refined and persist after cold rolling, decarburization and the final anneal.

FIG. 5 is a photomicrograph at a magnification of 100× showing the material of the present invention after hot rolling, cold rolling and decarburization, wherein the grains after hot rolling were in ideal condition.

FIG. 6 is a (100) optical pole figure showing oriented iron having a (110) [001] texture.

FIG. 7 is a (100) optical pole figure showing oriented iron having a (112) [110] texture.

FIG. 8 is a graph comparing the DC magnetization curves for oriented ingot iron of the present invention with the curves for conventional magnetic ingot iron, non-oriented silicon steel and oriented 3% silicon-iron.

Briefly the invention is based on the discovery that the recrystallization texture of ingot iron and low carbon steel can be changed by promoting as a final step in the processing the growth of grains having the preferred orientations at the expense of grains of other orientations, a phenomenon referred to as secondary recrystallization. In the usual practice the manufacturer of low carbon ferrous materials contemplates hot and cold rolling steps to attain

a desired final gauge, and an anneal slightly below the  $A_1$  or lower critical temperature to soften the material by recrystallizing the grain structure. It has been found that subjecting the material during this final anneal to higher temperatures or longer times at temperature will tend to increase the grain size, but will have little effect on the orientation of the grains. This is true because the material undergoes a primary recrystallization and an indiscriminate and non-selective grain growth rather than a secondary recrystallization with selective grain growth.

A high degree of preferred orientation can be achieved in accordance with the teachings of this invention by causing sulfur to be diffused into the grain boundaries of the primary grains and thus restricting their growth. This makes possible a secondary recrystallization with the results above noted.

The ingot iron or low carbon steel may be produced by any of the known melting and refining processes. When low carbon steel is used, it may be either rimming or killed steel; but, if it is killed steel, silicon killed steel is preferred. The low carbon iron or steel may be cast continuously or intermittently into ingots, billets or slabs. It may be hot rolled to any thickness which the rolling apparatus can produce. Depending upon the desired cold reductions which follow the hot rolling, and the desired final gauge of the final product, the hot rolled band may vary in thickness from about 0.125 to about 0.050 inch with present equipment.

The hot rolling procedure may be varied with regard to the temperature of the stock when it is heated for rolling, when it enters the rolling mill, when it is rolled to various intermediate thicknesses, and when it is cooled to ambient temperatures. These temperatures are well established for ingot iron and low carbon steel destined for particular end uses; and these practices can be followed for the material of the present invention.

It is important, however, that the grain size of the material after hot rolling and before cold rolling be relatively small and uniform throughout the thickness of the material. For this reason the preferred temperature for finishing the hot rolling of low carbon steels is above about 1600° F. in order to obtain a fine grain structure. Such a desired fine grain structure is illustrated in the photomicrograph of FIG. 1. Ingot iron will be finished at a lower temperature in the conventional manner. Coiling temperatures below 1300° F. are preferred for both ingot iron and low carbon steel to prevent excessive grain growth.

In order to insure a uniform fine grain structure after hot rolling, the material may be subjected to an open anneal or normalizing heat treatment. While decarburization may be effected during this step, it is preferred to remove the carbon at a later stage, as hereinafter described. The principal purpose of the heat treatment following the hot rolling is to refine and equalize the grain structure. This may be accomplished by a continuous or strand normalizing treatment comprising a short-time heating above the  $A_3$  or upper critical point (about 1625° F.). A temperature of about 1800° F. is satisfactory. It has been found that if large grains remain at the surface of the material after hot rolling (as shown in FIG. 2), as tends to be the case in the interior of a coil formed at a relatively high temperature, and if these large grains are not refined by a high temperature anneal, they will persist through a later decarburizing step (see FIG. 3) and through the final anneal (see FIG. 4). FIG. 5 is a photomicrograph showing the ideal grain structure after decarburization, where the grain structure after hot rolling was similar to that shown in FIG. 1, or was refined by a normalizing treatment. For reasons of economy, it is preferable to obtain after hot rolling a grain structure of the type shown in FIG. 1, thereby eliminating the normalizing step.

It has further been found that initial anneals after hot rolling, when conducted at temperatures below the upper

critical point, have almost no effect on the grain structure of the material if such an anneal is limited to less than five minutes. At longer times grain growth and decarburization occur, and ultimate secondary grain growth becomes more difficult to achieve.

The ingot iron or low carbon steel having a grain condition similar to that shown in FIG. 1, is cold rolled in one or more stages to obtain the desired final gauge. As will be understood by one skilled in the art, a single stage cold reduction is often preferred for reasons of economy, since multiple stage cold reductions require an anneal between stages. Should multiple stage cold reductions be necessary, the intermediate anneal should be an open anneal at a temperature between about 1100° F. and about 1800° F. in a reducing atmosphere, which may be a decarburizing atmosphere, if pickling is to be avoided. If the atmosphere is oxidizing, pickling will be necessary.

When it is desired that the final product have a cube-on-edge orientation, the amount of cold reduction in each stage should be less than 90%. Reductions of between about 50% and 85% have been found satisfactory.

When it is desired that the final product have a (112) [110] orientation, the cold reduction should be 90% or more in a single stage process. With cold reductions of two or more stages, with intermediate anneals, only the last cold rolling stage must be 90% or greater. Reductions of 90% have produced excellent results in obtaining a (112) [110] orientation in the final product. The maximum reduction is limited only by the capacity of the rolling mill and the ability of the material to withstand a drastic deformation.

While the cold reduction ranges given above for obtaining the two types of grain orientation are fairly precise, the hot rolling conditions, chemistry and final thickness will have some effect on the tendency to produce secondaries of one kind or the other.

As stated above decarburization may be carried on prior to the cold rolling. However, it is preferred to decarburize after cold rolling. Decarburization is preferably accomplished by a continuous anneal for a few minutes in a wet hydrogen-bearing atmosphere at about 1500° F. as is well known in the art (see U.S. Patent No. 2,307,391). The temperature may be varied from 1200° F. to about 1600° F. and less expensive hydrogen-bearing gases such as dissociated ammonia may be used. The final carbon content should be less than 0.01% and for some uses less than 0.005%. Decarburization can also be performed in a box anneal. Denitrifying to less than .001% nitrogen may also be effected during the decarburizing anneal or in one of the other annealing treatments.

The final treatment will be a box anneal at a temperature just below the upper critical or  $A_3$  temperature in a reducing, non-oxidizing atmosphere. The iron or low carbon steel must be maintained in the alpha phase but, because of the loss of carbon in the decarburizing step, the upper critical temperature or  $A_3$  temperature is then about 1650° F. A temperature of about 1550° F. will be sufficient if the time at temperature is forty-eight hours or more. A preferred temperature range is about 1600° F. to about 1650° F. and with such a restricted temperature range the time at temperature may be less than twenty-four hours.

With the development of open coil annealing, it is possible to accomplish decarburizing, denitrifying and the final annealing steps in a single furnace by changing the temperatures and atmospheres in the same way as when a strand anneal is followed by a box anneal.

A high degree of preferred orientation can be achieved in the ingot iron or low carbon steel by causing sulfur to be diffused into the grain boundaries of the primary grains, thus restricting their growth and making possible a secondary recrystallization with selective grain growth. This is accomplished by treatment of the ingot iron or low carbon steel with sulfur or sulfur compounds at final gauge and immediately prior to or during the primary

grain growth portion of an anneal. There are various ways in which this can be done.

The invention can be practiced by the addition of ferrous sulfide or other sulfur compounds, which dissociate or decompose at the temperatures of primary grain growth, to the annealing separator employed during the final heat treatment. Elemental sulfur can also be added to the separator for the same purpose.

The preferred annealing separators are magnesia, alumina or calcium oxide or mixtures of these in finely divided form although other substances may be used, if desired, such as titania and other refractory metal oxides.

The final anneal which may include both a primary recrystallization and a secondary recrystallization is usually an anneal in dry hydrogen in a muffle or box. The anneal may be carried on with the material in the form of stacked sheets or wound coils; and if the atmosphere of the annealing is required to act upon the ingot iron or low carbon steel, excellent results may be obtained by annealing in loose coils formed in accordance with modern techniques. Whether or not the material exists as sheets in a stack or as convolutions of a coil, it is preferred that the quantity of the sulfur-bearing material at the surfaces of the stock be maintained within certain limits as later set forth. It is believed that the sulfur or sulfur compound reacts with the dry hydrogen annealing atmosphere to form hydrogen sulfide; that the sulfur is transferred to the steel by means of hydrogen sulfide as a carrier; and that the hydrogen sulfide reacts with the steel to form sulfides at the grain boundaries. The reaction occurs while the furnace temperature is between about 1000° F. and 1650° F. The absorption of sulfur creates high sulfur concentrations at the grain boundaries of the primary structure tending to prevent the primary grain structure from undergoing such grain growth as would interfere with subsequent secondary recrystallization. Thus a finely grained matrix is maintained until secondary grains of the preferred orientation begin to consume the grains of other orientations. Thereafter as the temperature rises further, secondary grain growth will proceed by grain boundary energy and will convert the fine grain matrix into a well developed structure of preferred orientation.

It follows from the explanation that instead of including sulfur or a sulfur-bearing compound in the annealing separator, comparable results may be achieved by charging the annealing atmosphere with hydrogen sulfide or any other gaseous sulfur compound, such as sulfur dioxide, sulfur hexafluoride and the like, which would react at the grain boundaries at temperatures around or slightly above 1000° F. This may be done during the primary grain growth period which occurs during the heating of the material up to the temperature at which secondary recrystallization occurs in a final anneal. Selenium or hydrogen selenide will behave similarly to sulfur or hydrogen sulfide, although these substances are more expensive, and these materials are to be considered the equivalents of sulfur and hydrogen sulfide.

In yet another variant procedure, the sulfur or sulfur-bearing compound may be made available at the surfaces of the sheet material during a decarburizing anneal prior to the final anneal. For example, if the ingot iron or low carbon steel strip is moved through an elongated furnace containing a special atmosphere for removing carbon, it is possible to mix hydrogen sulfide with the decarburizing atmosphere to form a controlled iron sulfide film on the material which will inhibit the primary grain growth which continues during the subsequent final anneal.

The amount of elemental sulfur or sulfur in the form of a sulfur-bearing compound added to the annealing separator may be from about ½% to about 10% of the annealing separator by weight, when the separator is applied in a quantity of about ten pounds per ton of ingot iron or low carbon steel. It has been found that secondaries produced over this range of sulfur additions

show a tendency to become larger as the sulfur addition is increased. The quantity of sulfur made available to the ingot iron or low carbon steel may exceed the solubility of sulfur in the area of the grain boundaries. Some sulfur will be lost during the drying of a slurry coating and the handling of the dried coating. Therefore it is necessary to add sufficient excess to make up for this loss, and values disclosed refer in all cases to the amount of sulfur or sulfide present during the heat treatment.

As has been indicated hydrogen sulfide or other sulfur-bearing gas may be added to the annealing atmosphere in lieu of including sulfur in the annealing separator. Where this is done, and assuming that the atmosphere has access to all surfaces of the ingot iron or low carbon steel, at least 750 p.p.m. of hydrogen sulfide or its equivalent should be present in the atmosphere. It is possible, however, to add larger quantities of sulfur-bearing gases, even to the extent of forming an iron sulfide film or surface layer as taught in the copending application of the same inventor, Ser. No. 378,823, filed June 29, 1964, now Patent No. 3,333,992. Where the ingot iron or low carbon steel is given a heat treatment such as a decarburizing treatment preceding the final heat treatment, the sulfide film is formed ahead of the final anneal and the sulfides will diffuse into the primary grain boundaries during the final anneal.

The total sulfur content of the ingot iron or low carbon steel is not necessarily controlling. The presence of finely dispersed sulfides at the grain boundaries during the final anneal is of primary importance. It follows that a low carbon steel or ingot iron having sufficient sulfides at the grain boundaries may be suitable for primary and secondary grain growth even though its total sulfur content may be relatively low, whereas a treatment which tended to remove sulfides at the grain boundaries might impair the ability of the material to acquire a high degree of preferred orientation even though it did not appreciably lower the total sulfur content of the material. Consequently, the practice of this invention involves the addition of some sulfur or sulfide to the ingot iron or low carbon steel after it has been rolled to final gauge, substantially irrespective of its total sulfur content, especially since the sulfur or sulfide added by the procedures herein taught occurs primarily at the grain boundaries.

The properties of the iron or steel can be damaged by too much sulfur. While limited quantities can be removed at annealing temperatures below 1650° F., normally the sulfur is not substantially lowered.

When an iron sulfide film is formed on the surfaces of metal as by mixing hydrogen sulfide with the decarburizing atmosphere, the layer of iron sulfide should be about .02 mil to about .10 mil thick.

The use of vacuum annealing is not precluded in the practice of this invention. Also nitrogen or other inert gases may be used with or without hydrogen or in a partial vacuum. Sulfur is apparently capable of direct diffusion into the metal from the annealing separator.

#### EXAMPLE I.—A SINGLE STAGE PROCESS FOR MAKING A (112) [110] TEXTURE

##### Ladle analysis

	Percent
Carbon -----	.010
Manganese -----	.965
Sulfur -----	.025

##### The process steps

- (1) The material was hot rolled to .100".
- (2) The material was then pickled.
- (3) The material was cold rolled 92% to .008".
- (4) The material was decarburized in a strip anneal, conducted in wet hydrogen at 1500° F. for four minutes, to a carbon content of about .004%.

(5) The material was coated with 8% sulfur in magnesia.

elemental sulfur, and box annealed at 1630° F. for 24 hours.

TABLE IV

	Final Gauge	Percent Cold Reduction	Permeability at H=10 oersteds	Core Loss, watts/lb.		
				P10; 60	P15; 60	P17; 60
Ex. VII.....	.025"	62	1,880	1.55	3.9	5.5
Ex. VIII.....	.0185"	72	1,924	1.05	2.4	3.3
Ex. IX.....	.014"	79	1,942	.90	2.0	2.6
Ex. X.....	.011"	83	1,960	.80	1.6	2.05
Ex. XI.....	.009"	86	1,939	.75	1.45	1.8
Ex. XII.....	.006"	70+70	1,889	.65	1.25	1.65

(6) The material was box annealed at 1620° F. for sixteen hour in a dry hydrogen atmosphere.

The end product displayed a strong (112) [110] texture and very good forming properties.

#### EXAMPLES II and III

The following Table II gives the final gauge, percentage reduction, type of processing, magnetic characteristics and the type of orientation achieved for five different samples. All of the samples were taken from the same hot rolled coil as that used in Example I.

The low carbon steel was then cold rolled, decarburized in a strip anneal at 1500° C., coated with magnesia containing 4% elemental sulfur by weight, and box annealed at 1630° F. for 24 hours.

TABLE II

	Final Gauge	Percent Cold Reduction	Type Processing	Permeability at H=10 oersteds	Type of Orientation
Ex. II.....	.017"	83	Single stage.....	1,820	Cube-on-edge secondaries.
Ex. III.....	.015"	70+50	Two stage*.....	1,762	Do.

\*The material was reduced 70% in the first stage of cold rolling to an intermediate gauge of .030" and 50% in the second stage to the final gauge of .015".

#### EXAMPLES IV, V AND VI

Another group of samples was taken from an ingot iron heat having the following ladle analysis:

	Percent
Carbon .....	.015
Manganese .....	.052
Sulfur .....	.022

The material was hot rolled to a thickness of .100" and finished at a temperature of about 1500° F. The hot rolled band was normalized in a strip furnace at 1800° F. and pickled. After cold rolling various amounts in a single stage as shown in Table III below, the material was decarburized at 1500° F., coated with magnesia containing 4% elemental sulfur, and box annealed for 24 hours at 1600° F. The permeability at all gauges was excellent, indicating that the product had a high degree of cube-on-edge orientation.

TABLE III

	Final Gauge	Percent Cold Reduction	Permeability at H=10 oersteds
Ex. IV.....	.025"	62	1,885
Ex. V.....	.014"	79	1,892
Ex. VI.....	.011"	83	1,835

#### EXAMPLES VII THROUGH XII

The magnetic properties of material processed in the manner of the invention were also determined for ingot iron samples having the following ladle analysis:

	Percent
Carbon .....	.020
Manganese .....	.045
Sulfur .....	.023

The material was hot rolled to a thickness of .065" and normalized at 1800° F. in a strip furnace. After pickling, samples were cold reduced to six final gauges, decarburized for five minutes at 1500° F. in wet hydrogen, coated with a slurry of magnesia containing 2½%

The outstanding core loss values of the materials of the present invention are more pronounced at high frequencies. Tests of the materials of Examples IX and XI were made at a frequency of 400 cycles per second and at inductions of 10, 15 and 17 kilogausses. The results are summarized in Table V.

TABLE V

	Core Loss, watts/lb.		
	P10; 400	P15; 400	P17; 400
Ex. IX.....	17	47	65
Ex. XI.....	11	25	33

It can readily be seen from Tables IV and V that the permeability and core loss values of the materials were

found to be exceptionally good, indicating that a well-developed cube-on-edge orientation was obtained in all of the samples.

In FIG. 8, the DC magnetization curves for the materials of Examples VII, VIII and IX are compared to the DC magnetization curves for conventional ingot iron, non-oriented silicon steel and oriented 3% silicon-iron. It will be obvious from FIG. 8 that the ingot iron of the present invention requires considerably less magnetizing force to reach the same induction as commercial ingot iron and non-oriented silicon steels. At magnetizing forces of about 1 oersted and higher the ingot iron of the present invention compares favorably with the more expensive oriented 3% silicon-irons.

The effective sulfur content in the environment of the steel should be maintained during the primary grain growth period until the temperature of the steel reaches about 1500° to 1600° F. When this is done grain growth will be satisfactorily inhibited and the grains having the preferred orientation will be freed to take over and control the final orientation of the products during secondary growth. Generally speaking, the amount of sulfur used effectively in the practice of this invention may be stated as follows:

(1) Where elemental sulfur such as yellow powder or flowers of sulfur is added to an annealing separator, about ½% to about 10% sulfur is used, based on the weight percent of the coating when the coating is used in normal thicknesses for annealing separation.

(2) Where ferrous sulfide is used to provide the sulfur, the annealing separator should contain about ¼% to 1% of this compound.

(3) Where hydrogen sulfide is added to a decarburizing atmosphere to form a film of iron sulfide to control primary grain growth in a subsequent anneal, the treatment should be so regulated to form a sulfide film having a thickness of about .0001" (.1 mil) to about .00002" (.02 mil).

(4) In any event, and whether or not sulfur is added to form an iron sulfide film, or whether or not sulfur is

added during the primary grain growth portion of a final anneal, the addition being made to either the annealing atmosphere or to an annealing separator, or both, best results are obtained when the quantity of sulfur so added is such as to raise the sulfur content of the ingot iron or low carbon steel by no more than about .005% and not less than about .001% by the time the steel has reached a temperature of about 1600° to 1650° F. in the final anneal.

It has further been determined that a high manganese content of the low carbon steel or ingot iron makes it necessary to increase the sulfur addition to the annealing separator in order to obtain the best secondary growth. For this reason the manganese content should preferably be less than about .2%.

As will be evident from the above examples, differences in the treatment of the ingot iron or low carbon steel will largely determine whether a cube-on-edge or some type of cube-on-corner or a mixture of the two is obtained in the final product. FIG. 6 is a pole figure illustrating the orientation of the material of Example VII. It will be seen that the pattern indicates unmistakably a high degree of (110) [001] orientation.

FIG. 7 is a pole figure illustrating the orientation of the material of Example VI and it will be seen that the pattern shows a high degree of (112) [110] orientation.

The grain size of the materials of this invention in final form are relatively large. While the A.S.T.M. standard chart of grain sizes contemplates a magnification of 100×, the grain sizes of the materials of this invention can be compared at unit magnification with the S.D.T.M. chart, and as so compared generally respond to No. 8 to No. 00 and even larger. Moreover, the grains have generally a length at least about ten times the thickness of the sheet stock.

As above indicated the production of a (112) [110] orientation in ingot iron or low carbon steel substantially increases the formability of the product despite a relatively large grain size.

It will be understood that modifications may be made without departing from the spirit of the invention, and no limitations are intended other than as specifically set forth in the claims.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A method of producing ingot iron and low carbon steel sheet stock characterized by a preponderant orientation chosen from a class consisting of (110) [001] and (112) [110], which comprises hot rolling a ferrous material containing up to 0.10% carbon, 0.01% to 0.40% manganese, up to 0.05% sulfur, the balance being iron except for incidental impurities, to produce an intermediate gauge hot rolled band, cold rolling the said hot rolled band to produce a cold rolled stock at final gauge, subjecting said cold rolled stock to a final anneal during which primary grain growth occurs in the presence of a material

chosen from the class consisting of elemental sulfur, selenium, and decomposable compounds thereof, and thereafter subjecting said stock to a secondary recrystallization treatment under box annealing conditions at a higher temperature of 1500° to 1650° F.

2. The process claimed in claim 1 in which said iron and steel contains up to 1.8% silicon.

3. The process claimed in claim 1 wherein the said cold rolled stock is subjected to a decarburization treatment prior to said final anneal.

4. The process claimed in claim 1 in which the hot rolling of said low carbon steel is finished at a temperature in excess of about 1600° F.

5. The process claimed in claim 1 including a normalizing strand anneal following the hot rolling to obtain a uniform fine-grain structure.

6. The process claimed in claim 1 wherein a cold rolling reduction of at least 90% is used to reduce the material to final gauge and in which the orientation produced in the stock is primarily (112) [110] by Miller's indices.

7. The process claimed in claim 1 wherein a cold rolling reduction of less than 90% is used to reduce the material to final gauge and in which the orientation produced in the stock is primarily (110) [001].

8. The process claimed in claim 1 wherein two cold rolling reductions with an intervening anneal are used to reduce the material to final gauge, said intervening anneal being a strip anneal.

9. The process claimed in claim 1 wherein sulfur is added to the stock during the primary grain growth portion of said final anneal.

10. The process claimed in claim 3 wherein said stock is exposed to sulfur from an external source during the said decarburization treatment to the extent of producing on the stock a thin film of ferrous sulfide.

11. Ingot iron and low carbon steel sheet stock characterized by a preponderant (112) [110] orientation.

12. Ingot iron and low carbon steel sheet stock characterized by a preponderant orientation chosen from a class consisting of (110) [001] and (112) [110] and wherein the grains have a length of at least about ten times the thickness of said sheet stock.

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