United States Patent Office

3,180,767

Patented Apr. 27, 1965

1

3,180,767

PROCESS FOR MAKING A DECARBURIZED LOW CARBON, LOW ALLOY FERROUS MATERIAL FOR MAGNETIC USES

Robert W. Easton, Middletown, and Victor W. Carpenter, Franklin, Ohio, assignors to Armco Steel Corporation, Middletown, Ohio, a corporation of Ohio

No Drawing. Filed Oct. 8, 1962, Ser. No. 229,203
10 Claims. (Cl. 148—120)

While much of the ferrous material produced for magnetic uses consists of high alloy metal, such for example as the silicon-iron of commerce, there is a substantial market for low alloy metals for use in low horsepower rotating electrical machinery and elsewhere. These low alloy metals are not generally classified as electrical steels in the industry, but on the contrary correspond roughy to SAE grades 1008 and 1010.

Such materials are of course substantially less expensive than the electrical steels and are used in less critical applications. The basic objects of this invention include the provision of improved methods for the production of such ferrous materials, and the provision of ferrous materials of the low alloy class which have improved magnetic permeabilities and other magnetic characteristics.

More specific objects of the invention include the provision of low alloy ferrous materials which will respond to a final anneal at low temperatures for the development of their ultimate magnetic properties, which do not require decarburization during a customer's anneal, which will have uniform and stable magnetic properties including low core loss and high permeability for materials of the class, and which will have good punching properties.

These and other objects of the invention which will be pointed out hereinafter or will be apparent to one skilled in the art upon reading these specifications, are accomplished by those procedures and realized in those products of which certain exemplary embodiments will now be described.

Briefly, in the practice of this invention, it has been found that low alloy ferrous materials of certain analyses can be produced in such a way as to effect a material improvement in their magnetic properties as compared with similar ferrous materials otherwise produced. In particular, the procedures hereinafter taught bring about changes in the crystal lattices whereby the magnetic qualities are substantially improved, and the ferrous material is rendered magnetically non-ageing. The ultimate product will be low in carbon, that is to say it will have had its carbon content reduced to a value of about .003% or less. While the magnetic ferrous material of this invention is not strongly directional, and is not considered to be an oriented product, it is believed that its improved magnetic qualities are due in part at least to an enhanced degree of crystal orientation.

The initial ferrous material for conversion into the product of this invention is preferably a rimming steel which can be formed in any suitable way such as in the open hearth furnace (with or without oxygen blowing), or by any of those processes in which the metal is blown with oxygen. The steel is preferably cast into ingots in "bottle top molds," i.e., ingot molds having a small opening at the top, which opening is mechanically capped after teeming. The use of such molds has an effect on the amount of carbon which may be lost during the solidification of the metal.

Other methods for casting the metal without excessive loss of carbon would be satisfactory, e.g., continuous casting methods resulting in what may be termed "thin bar."

The metal as teemed or cast (ladle analysis) may be as follows:

Carbon—above about .05% but not over about .1%,

2

Manganese—from 0.15 to 1.0%, with 0.3% to 1% being preferred,
Phosphorus—from residual to about .15%,
Silicon—up to about .1% if desired,
Sulfur—as low as practicable, and not over about .025% being preferred,
Balance substantially all iron with such residual impurities as are incident to the mode of manufacture.

The manganese promotes good low temperature recrystallization and contributes to the volume resistivity of the product, which cuts down core loss. In general, a manganese content of over 0.3% is desirable.

The phosphorus and silicon increase the volume resistivity of the metal and also increase hardness.

It will be seen that the amount of silicon is low and is not sufficient to produce a high alloy product classifiable as electrical steel.

In most modes of steel manufacture from about .005 to 0.1% of silicon may be regarded as a residual impurity; and in the product of this invention, the silicon may lie within the last mentioned range.

Ferrous material having the above analysis is subjected to processing steps which may be generally outlined as follows:

(1) The ingots or other cast structures are hot rolled to an intermediate gauge.

(2) The hot rolled material is pickled to remove the hot mill scale.

(3) The pickled material is subjected to a low temperature decarburization in a decarburizing atmosphere.

(4) The decarburized product is cold rolled to final gauge or substantially to final gauge.

(5) The cold rolled product is subjected to a relatively low temperature anneal. This may be practiced either by the manufacturer of the ferrous material or by his customer after punching. This anneal is a non-carburizing anneal.

(6) In some instances, and especially where the manufacturer of the ferrous material practices the anneal which is the step No. 5 of this routing, the material may be temper rolled, i.e. rolled with a small elongation, say, about 0.3% to 3.0% after the anneal but prior to punching.

The generalized series of steps outlined above may be practiced wholly by the steel manufacturer or in part by the steel manufacturer and in part by the customer. In the former of these instances, the steel manufacturer will perform all six of the steps noted, and the stock so produced will be referred to as "finished stock." The customer will receive coils or sheets, of the stock after the step 5 anneal has been practiced thereon; and the stock will generally be temper rolled as above defined, not only to produce flatness but also to harden the stock slightly to put it into better condition for punching.

In the latter of these instances, the stock may be sold to the customer as "semi-finished stock" after the completion of the cold rolling which is step 4. In this event, the steel manufacturer will usually take precautions to insure flatness in the coils or sheets. This may be done in various known ways, one of such ways being the giving of at least a final light cold rolling pass to the steel under conditions of tension.

It will be noted that the initial material is relatively high in carbon for a reason which will be set forth later. But the carbon content requires certain precautions during the hot rolling. Massive carbides in the hot rolled intermediate gauge material are to be avoided; and the carbon should be present as finely divided dispersed carbides or pearlite.

This may be accomplished by finishing the hot rolling at a temperature of about 1600° F. for good recrystallization, then quenching the hot rolled material with water on the run-out table, and coiling it at a temperature not above about 1150° F. and preferably at about 1000° F. Even lower temperatures can be used; the only limitations being the mechanical limitations of the coiler. This procedure insures a desired condition of the carbon in the product.

The hot rolling may be carried on otherwise in any suitable way and on any suitable apparatus; but continuous hot rolling is preferably practiced, the ferrous material being reduced to the desired intermediate gauge from ingots either without reheating, or with a reheating of slabs formed from the ingots during the course of the reduction.

The intermediate gauge to which the ferrous material is hot rolled can be considerably varied in the light of the final sheet gauge desired, taking into account the available hot rolling apparatus. For example, in a commercial procedure, the ferrous material has been hot rolled to a gauge of about .080″ leaving a reduction of about 70 to 80% to be performed in the subsequent cold rolling. However, there is evidence to indicate that a cold rolling with about a 40% or 50% reduction is desirable for grain size, so that in general the gauge of the hot rolled stock may be as light as can be obtained with the available apparatus in a satisfactory hot rolling operation. If a hot roll gauge as light as .050″ can be achieved, a cold rolling reduction of 50% will produce a sheet stock having a thickness of .025″, which is suitable for many uses. It will be understood that the intermediate and final gauges of the material may be varied as desired so long as an adequate cold rolling reduction, as hereinafter set forth, is practiced.

The hot rolled material is then pickled in the ordinary way for the purpose of removing the hot mill scale from its surfaces.

After the hot rolled intermediate gauge ferrous material is pickled, it will be subjected to a low temperature decarburization. This is best accomplished by loose coiling the material and heating it in a muffle furnace. It is necessary for proper decarburization that the furnace atmosphere have ready access to all of the surfaces of the stock. There are known techniques for coiling the metal with the interposition of a strand-like separator between the convolutions of the coil. In some procedures the strand is removed before annealing, while in other procedures a strand which will withstand the temperatures of the anneal and is so configured as to permit the passage of the furnace gases between the convolutions is left in place in the coil.

It has hitherto been known (see the Carpenter et al. U.S. Patent No. 2,287,467) that decarburization in a wet atmosphere can be accomplished at a comparatively low temperature; and in the practice of this invention the loose coiled material is supported in a suitable muffle and heated to about 1350° F., the muffle being filled with the desired atmosphere. The atmosphere is (for economic reasons) preferably one containing from 20 to 40% hydrogen and having a dew point of about 120° F. Hydrogen to which water vapor has been added may be used as such, or HNX or dissociated ammonia or mixtures of the two may be used. The controlling factor here is the ratio of H_2:H_2O. It should be such that rapid decarburization will occur without oxidation of the iron. The atmosphere should not contain appreciable amounts of strongly carburizing gases.

The decarburizing time increases with the thickness of the metal being treated, and, as has been indicated, the decarburization is so carried on as to reduce the carbon content of the metal to at most about 0.005%, and preferably to 0.003% or lower. The initial carbon content also has an effect upon the decarburization time; but by way of example a treatment of material having a thickness of .080″ and a carbon content greater than about .05%, but less than .1% for about twelve hours at temperature in the described atmosphere will be sufficient for a thirty ton charge. Some muffles are provided with means for producing a positive circulation of the atmosphere within the furnace so as to enforce a flow of the atmosphere between the convolutions of the coil, and this increases the efficiency of the decarburizing treatment, as indicated in the above example.

It should be understood that the furnace atmosphere between the convolutions will increase in carbon content, so that for rapidity of operation this build-up must be diluted as by the continuous addition of fresh gas, or by scrubbing out some of the carbon.

Under ideal conditions of furnace atmosphere, which may be uneconomical to maintain continuously, the time required probably could be reduced from about 12 hours to several hours.

Variations in temperature may be employed, and by a low temperature decarburization is meant a decarburization within a temperature range of about 1250° F. to 1600° F. It is known that efficient decarburization will also occur at a higher temperature range, say 1750° F. and above; but the material being treated is difficult to handle properly at such high temperatures.

It has been found in the practice of this invention that the optimum results are attained when the decarburized hot rolled product is characterized by a substantial columnar grain growth. The procedure of this application makes use of the hitherto recognized phenomenon that columnar grain growth will occur during a heat treatment in which a phase change occurs at a constant temperature. The phase change is dependably produced by a reduction of the carbon content of the material from a value of at least about 0.05% to a value at least as low as about 0.003% during the soaking portion of the heat treatment, although it can be produced within a narrower range of carbon reduction. The resultant decarburized product is characterized by crystals of macro size, i.e., visible to the naked eye. The material will further be "bright," since it was pickled prior to the decarburization and since the furnace atmosphere is oxidizing toward carbon but substantially non-oxidizing toward iron.

Following the decarburizing treatment a cold rolling of the material is practiced. The cold rolling, which can be carried on in any cold rolling mill or tandem train, preferably produces about 40% to 50% reduction in the thickness of the material; and a reduction of this order appears to be desirable for grain size. However, it may be found difficult to obtain a desirably thin final product in a single stage of cold rolling from any available intermediate gauge hot rolled product unless the percentage of cold rolling reduction is increased. Cold rolling reductions extending from about 40% to about 80% may be given as a feasible range for the purposes of this invention.

The cold rolled product which is the result of step 4 in the routing given above may be supplied to the customer for stamping or punching in the "full hard" condition providing it has satisfactory flatness. As an alternative, it may be given a brief, relatively low temperature anneal followed by a temper rolling (as above defined) to obtain a desired hardness for punching. Yet again it may be treated for the introduction of critical strain (as later described), to promote grain growth in a subsequent customer's anneal.

The annealing treatment which follows the cold rolling reduction in the routing given above, and constitutes step 5 thereof, should produce a recrystallization of the material and therefore should be above about 1100° F. The normal temperature range will be between about 1100° and about 1400° F. Somewhat higher temperatures may be used. Too high a temperature should be avoided, since excessive temperatures lower the permeability, possibly because they tend to impair the orientation hereinafter described. As a general rule, the temperature should not be allowed to rise above 1650° F., and preferably not above about 1600° F. A normalizing treatment, i.e., a heating of the material to above its A_3 point followed by a rapid cooling, should be avoided, if it is desired to take advantage of the high permeabilities resulting from the favorable orientation.

The anneal may advantageously be carried on with the material in coiled form in a muffle or box, when performed by the steel manufacturer. With care it can be carried on in a continuous furnace; but a time at temperature which is characteristic of the operation of box or muffle furnaces is advantageous to the production of the optimum magnetic properties. Where the steel producer anneals the cold rolled stock, it may prove advantageous to follow the anneal with the temper rolling hereinabove mentioned to increase the stiffness of the stock for better punching characteristics.

The same general considerations apply when the step 5 anneal is performed by the customer. It will be understood that the customer may anneal the stock either before or after punching laminations from it. The annealing of punched laminations may be carried on in conventional furnaces of the continuous or batch type.

Whether performed by the steel producer or by the customer, the step 5 anneal can be carried on in conventional annealing atmospheres or in any atmosphere which is non-carburizing to the low carbon material and does not produce excessive oxidation of the steel, all as will be well understood by the worker in the art. Nitrogen, nitrogen-hydrogen mixtures, dissociated ammonia and the like can be used, as well as pure hydrogen, if desired. The annealing atmosphere will have a dew point low enough to preclude undesired oxidation. It is an advantage of the invention that the step 5 anneal need not be a decarburizing anneal.

Preferably it will be carried on as a bright anneal; but where punched laminations are being treated, it does not constitute a departure from the spirit of the invention to practice a controlled oxidation, such as a steam bluing, on the laminations in the cool-down portion of the anneal cycle. Such a controlled oxidation provides an insulative film on the surface of the punchings which reduces eddy current losses when the punchings are fabricated into electrical devices.

X-ray analysis shows that the material of this invention following the cold rolling and annealing is characterized by a detectable degree of crystal orientation. This crystal orientation partakes in part of the nature of cube-on-edge orientation, (110) [001] by Miller's Indices. The degree of orientation produced is sometimes called "two times random" which means that if a pole figure is made in which dots in certain areas indicate crystal orientation, the density in the darkest areas produced by the clumping of the dots will be about twice the average density of the pole figure. This degree of orientation is not sufficient to impart marked directional characteristics to the product; but the product of this invention has uniformly a higher permeability than products of the same chemistry otherwise produced, and in particular higher permeabilities than similar products having completely random orientation.

The product of this invention has a uniformly low core loss. In commercial practice it will be found that the core loss values for the material of this invention, when measured on Epstein samples in which half of the pieces are cut parallel to and half are cut transverse to the direction of rolling, having a thickness of .025" and having been annealed at 1300° to 1350° F. in a neutral atmosphere, will be about 1.8 to 2.0 watts per pound at 10 kga., and about 4.80 to 5.10 watts per pound at 15 kga. The permeability of the material will generally lie between values of 1580 to 1610 at 10 oersteds. The high permeability of the product at high inductions results in lower exciting currents and consequently lower copper costs in electrical machinery in which it is used.

Exemplary values are given below:

Magnetic properties of the steel of this invention and cold rolled commercial quality steel

[All properties obtained from 60 cycle tests made on Epstein samples (nominal thickness .025") given a 1350° F. anneal after processing]

Material	Composition		Sample Direction	Core Loss, w./lb.		Permeability at H=10
	Mn	P		10 kga.	15 kga.	
Commercial Quality Cold Rolled Steel	.36	.008	50/50	2.3	5.6	1,540
The Magnetic Steel of This Invention	.38	.005	50/50	1.9	4.8	1,600

The steel of this invention may, if desired, be subjected to a critical straining treatment for the purpose of attaining enlarged grains in the final product. As understood in the art, a critical straining procedure involves subjecting the steel to a cold rolling reduction of about 10% to about 20% followed by a heat treatment at a temperature generally from about 1250° to about 1500° F. in a non-carburizing atmosphere. The critical straining treatment must follow both the cold rolling to gauge and the recrystallizing anneal. Thus, if practiced, it will be undertaken subsequent to the step 5 anneal in the routing given above. It can be performed either by the steel manufacturer or by his customer. The critical straining treatment can be employed as a substitute for the temper rolling which has been given as step 6 of the normal routing.

The critical straining treatment produces a substantial grain growth in the finished product. This diminishes the degree of preferred orientation, and hence slightly lowers the permeability of the product. At the same time, the core loss characteristics are improved by the enlarged grain size, this being an advantage in some applications offsetting a small lowering of permeability values. The product in either event is believed to be unique.

Variations in the procedures set forth above may be made without departing from the spirit of the invention. The invention, having been described in exemplary embodiments, what is claimed as new and desired to be secured by Letters Patent is:

1. A process of making a ferrous magnetic material which comprises forming a steel containing about 0.05% to 0.1% carbon, about 0.15% to 1% manganese, up to about 0.15% phosphorus, and up to about 0.1% silicon, the balance being substantially all iron with normal residual impurities, and

(1) casting the steel into a shape suitable for hot rolling,

(2) hot rolling the steel to an intermediate gauge and coiling the hot material as rolled at a temperature not above about 1150° F. to produce finely divided dispersed carbides therein, and pickling the hot rolled material,

(3) subjecting the hot rolled and pickled material to a decarburizing anneal at a temperature of substantially 1250° to 1600° F. so as to reduce the carbon therein to a value not greater than about 0.005% and to cause columnar grain growth to occur because of a phase change, and

(4) cold rolling the decarburized material with a reduction of substantially 40% to 80%.

2. The process claimed in claim 1 wherein the hot rolled material is coiled at a temperature not exceeding about 1000° F.

3. The process claimed in claim 1 including a step in which:

(5) the cold rolled steel is subjected to a low temperature anneal substantially in the range of 1100° to

1650° F. but below the A_3 point in a non-carburizing substantially non-oxidizing atmosphere.

4. The process claimed in claim 1 wherein the steel is decarburized in a loose coil treatment at a temperature of substantially 1250° to 1600° F.

5. The process claimed in claim 1 wherein the steel is decarburized in a loose coil treatment at a temperature of substantially 1250° to 1600° F., and in which the hot rolled material is coiled at a temperature not above about 1000° F.

6. The process claimed in claim 3 including the following step:

(6) temper rolling the steel with a reduction of substantially 0.3 to 3.0%.

7. The process claimed in claim 3 in which the steel, after step 5, is subjected to a cold rolling reduction of substantially 10% to 20% and subsequently to a heat treatment at substantially 1250° to 1500° F. in a non-carburizing atmosphere.

8. The process claimed in claim 1 wherein the steel is decarburized to a carbon content not over about 0.003%.

9. The process claimed in claim 1 wherein the decarburization is conducted in an atmosphere containing at least about 20% hydrogen and having a dew point of about 120° F.

10. The process claimed in claim 1 wherein, in step 1, the steel is cast into small top ingot molds which are then mechanically capped.

References Cited by the Examiner

UNITED STATES PATENTS

2,512,358	6/50	McGeary	148—122
2,672,429	3/54	Malloy	148—120
2,943,007	6/60	Walker et al.	148—120

DAVID L. RECK, *Primary Examiner.*