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ART OF PRODUCING MAGNETIC MATERIAL

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Our invention relates to the manufacture of silicon steel for electrical uses. Its specific object is the provision of silicon steel sheets for power and distribution transformer use, which have the highest possible permeabilities measured in a given direction coupled with unusually low core loss also in the same direction. In magnetic materials of this class, the endeavor has for a long time been to improve the core loss; but the exigencies of transformer design are such that no further substantial economies can be made therein through lessened core loss, unless substantial improvement can be made in the permeability of the material within the transformer range of inductions.

It has been proposed in improving the permeability of silicon steel to cold work the material and then heat treat it with improvement in the grain size of the product. It has also been observed that even more drastic treatment in cold rolling followed by heat treatment will produce a greater improvement in permeability. One worker has stated that the preferred orientation of crystal lattices resulting from the more drastic cold rolling is the factor which appears to account for the improved permeability in the product. Another worker, who has relied upon the drastic cold working, has stated his belief that random orientation results therefrom. Workers in this field have set forth as important in connection with the operations certain matters as to specific heat treatments, specific amounts of drastic cold rolling of silicon steel between annealings and the like, which do not, according to our invention, constitute all of the factors of importance, and from the writings on the subject, and the attempted practice of these prior workers, it is our opinion that the results stated to be attained could not be produced regularly, and without the features involved in our discoveries would not result in producing a silicon steel sheet or strip of either the core loss improvement or very high permeability which we obtain, and can obtain regularly in commercial production.

It has been understood that in a single silicon-steel crystal the maximum permeability lies in the so-called (100) direction according to Miller's indices: but the teachings of the art, prior to the copending application of Cole and Davidson, Serial No. 1,066, filed January 9, 1935, which issued May 16, 1939, as U. S. Patent No. 2,158,065, have not been sufficient to enable one in a regular commercial manner to produce silicon steel sheets in which a preponderance of the crystals are oriented so that the (100) directions are substantially parallel to each other and parallel to the surface of the sheet, as can be determined, for example, by X-ray diffraction. By following the procedure hereinafter described, the applicants have been able consistently to produce an orientation which fulfills this condition in silicon steels high in manganese, and consequently have realized the great magnetic improvement which would be expected from aligning the crystals of which the sheet is composed so that their best magnetic directions are substantially parallel to each other. This type of orientation which is attained by the applicants, and which is conveniently referred to as a "twin derivative" orientation is one in which a preponderance of the crystals are substantially aligned so that a (100) direction is parallel to the direction of rolling and a (110) plane is parallel to the sheet surface. The prior art as set forth in patents and publications appears to have made no teachings which enable one consistently to go beyond that degree even of preferred orientation which may be called ordinary cold-rolling orientation, nor has it enabled one regularly to secure the type of orientation mentioned above in any substantial degree at all.

It will be understood that ordinary cold-rolling orientation, is such that the (110) directions of the crystals tend to be parallel to the direction of rolling and the (100) faces of the crystals tend to be parallel to a face of the sheet.

Our process is operative not only for silicon steels but also for any other alloyed irons or steels having the same crystal characteristics and being high in manganese.

We attain the objects of our invention which have been referred to hereinabove, or which will be apparent to one skilled in the art upon reading this specification, by that series of process steps which we shall now describe as an exemplary embodiment, it being understood that our invention is not limited thereto.

In the practice of our invention, we may start with a magnetic material having the following chemical analysis:

	Percent
Silicon	2.90 to 3.60
Manganese	.10 to .30

We prefer also to have carbon .02% or under, sulphur .03% or under.

The choice of manganese content is quite important not only since it has an important bearing on the mechanical problems arising in connection with the cold rolling of this material but also

since the processing must be varied in accordance with the manganese content to consistently produce

been described. We have found that if the steel contains in excess of .10 manganese, smoother edges will be produced and much less trouble from breakage will result. Other advantages of the higher manganese contents will be apparent as the process is further described. We have commercially produced materials having manganese contents ranging from .10 to .30% and find that material within this range can be successfully processed if the teachings hereinafter made are followed. The process is operative for materials having considerably higher manganese content than this, but we have found that with manganese contents higher than .5 per cent, the core loss is noticeably poorer.

The silicon ranges given in this embodiment are not limiting, but may be carried substantially above and below these values without departing from our invention. Our process is operative on steels having silicon contents even below 2.00 per cent, but it is to be understood that the lower the silicon the higher the resulting eddy currents and, consequently, the poorer the core loss of the resulting material. The lower end of the silicon range will be determined, then, in practice by the allowable core loss. In using materials having silicon contents below approximately 2.00 per cent which have an allotropic transformation, it is necessary to carry out all of the anneals at temperatures below the A_3 point. The upper silicon limit is determined only by the mechanical difficulties encountered in cold working high-silicon materials, which at the present time is around 4.5 per cent. The range given, however, is a convenient working range.

The carbon content is important from the standpoint of core loss; and we desire to finish with a material containing not more than .008% carbon in order to get the extremely low core loss which we obtain. We have set forth in this example an initial carbon value of .02% because we have commercially produced large quantities of material having approximately this carbon content. The lower the initial carbon content the easier it is to achieve the low values mentioned above which we desire in the final material. Higher values up to .049 have been successfully used but more stringent decarburizing means must be employed in order to insure the final low value. It is possible, of course, to start with steel containing initially not more than substantially .008% carbon such as has been made in the induction furnace and process it without further attention to the carbon content.

In carrying out our process we take hot rolled stock give it an initial heat treatment, and carry it down to gauge in two or more drastic cold workings, with an intervening annealing or annealings; and we give it a final heat treatment, as will hereinafter be described.

In our exemplary embodiment, ingots 20 by 30 by 51 inches are hot rolled to slabs, say 3½ by 26 by 72 inches, and are heated for 2½ hours at approximately 1800 to 2100° F. This material is then hot rolled to .30 inch in thickness in seven passes on a universal mill, and is further hot rolled to, say, 0.105 inch in thickness, through four tandem mills, preferably finishing the material at a temperature about 1400° F.; although it may be finished at different temperatures if desired. In this way the raw material for our process may be formed; but it will be clear that it may likewise be formed in other ways.

This material, 0.105 inch thick, is edge slit and box annealed in coils at 1400° F., for 24 to 36 hours, after which it may, if desired, be cooled quickly or even quenched. Or it may be cooled slowly, say at the rate of 85° per hour to 1100° F., at which point the boxes are uncovered and the coils allowed to cool in less than one-half hour to 500° F. The material is then pickled.

This initial heat treatment appears to be desirable chiefly as a safety factor because in many instances we have attained our results without using it; and it is employed mainly because the product is uniformly successful when it is produced as described. Merely finishing the material on the hot mills at very high temperatures, while giving satisfactory results in some instances, does not give as uniform results as a controlled heat treatment. Again, while in many instances open anneals may be employed, yet these do not appear to give consistent results on all batches. We have found, however, that a box annealing does serve to fit the material for the subsequent steps of our process in a way that gives commercially consistent results.

Our next step is to give the material a drastic cold reduction. In the particular embodiment described we reduce the material from 0.105 inch to 0.025 inch, which is a reduction of the order of 76%. This percentage of reduction may be departed from considerably, but drastic cold rolling of more than 60% appears to be commercially, if not absolutely, necessary; and the cold rolling reduction should not exceed 85%. We prefer to carry down the material in a series of rolling stages, either separately or continuously, in which heavy reductions are made at each pass—and ordinarily we use from 4 to 6 passes.

The cold rolling reduction, while it may uniformly be depended upon to give the type of orientation referred to as "cold rolling orientation," must be carried on, however, in a particular way if the results attained by our invention are to follow.

In a co-pending application by two of the inventors of this process (Serial #1,066), it is taught that best results are obtained when the temperature of the strip is not permitted to exceed the boiling point of water at any stage during the cold reduction of materials for example which contained .03–.09% manganese. However, as the result of exhaustive experimentation and the production of this type of material on commercial equipment it has been discovered that if higher manganese contents are used, such as .10% or greater, it is not only permissible but necessary, if best results are to be obtained, to keep the material in excess of the temperature of boiling water, preferably within the range of 250 to 400° F. during a major portion of the reduction. Experimentally we have determined that excellent results can be obtained even up to 700° F.

On some types of cold rolling mills it may be found necessary to provide heating means to maintain the strip within this temperature range, especially on mills which can take only light reductions or on which the strip passes over guide rolls or is exposed to the air for a considerable time. On other mills, particularly tandem mills, the heat generated by the mechanical work of reduction will be found sufficient to maintain the strip within this range. The heat loss while the strip is rolled in a coil is very slow and the material will ordinarily re-

tain a sufficient temperature between passes if too long a time is not permitted to elapse.

The keeping of the temperature of the strip within this proper range (250 to 400° F.) during the cold rolling of material of this type having 10% or more manganese is vital if the optimum in results is to be secured. It is the one factor which enables us to depart consistently from that type of orientation to which we have referred as cold-rolling orientation, and also to secure a higher degree of preferred orientation. The reason for this is believed to be that: whereas cold-rolling orientation and the resultant thereof upon heat treatment, is not productive of anything more than the ordinary cold-rolling orientation, it is possible to secure the different orientation to which we have referred, and to secure much more perfect selective orientation if a process of crystal twinning is caused to operate in the material. Thus, in a two-step process if the conditions are right, not only will the first cold rolling produce what is known as cold-rolling orientation, but also there will be a degree of twinning of crystals initiated in the strip. Upon the subsequent intervening annealing, this twinned condition will increase in extent, and upon the final cold rolling and ultimate heat treatment, substantially all of the crystals will have assumed that desired preferred orientation by passing through a twinned stage. This condition, so far as we know, has never hitherto been attained to a high degree on material having manganese within this range, unless by accident, in accordance with any of the teachings of the prior art. Inasmuch as the twinning will not occur most effectively in material of this chemical analysis if the temperature is permitted to fall outside of this range, the necessity for the particular precautions which we have mentioned in the cold rolling step will immediately become apparent. There is no way of which we are aware of securing substantially complete preferred orientation, except by allowing the material to pass through stages of crystal change of which the process of a controlled twinning is a part.

We have further found that there are many practical advantages in using a material having a relatively high manganese content (in excess of .09%) which can be realized if the cold-rolling temperature teachings herein set forth are followed. Silicon steel is comparatively difficult to cold roll and it often takes many passes to reduce the strip to the light gages desired when the strip has to be kept cold. With this higher manganese and higher rolling temperature fewer passes are needed with a corresponding decrease in cost.

In designing transformers a very effective way of reducing the watt loss of the core is to use thinner laminations of metal in the core which has resulted in a demand for increasingly lighter gages of sheets which shall still have very special magnetic properties. In attempting to produce materials of this type in thickness lighter than .014 inch, it was often found that when using stock having low manganese, that is, say .03 to .09%, the improvement in core loss was accompanied by a falling off of magnetic permeability which tended to off-set the advantage obtained. We have made the discovery that material having .10% manganese or higher, if processed in accordance with our teachings, can be produced at .012 inch and have substantially the same permeability as at .014 inch. Thus we have been able to get the improved core loss and still retain the desired high permeability.

Further advantages of the high-manganese silicon steel processed in accordance with our teachings lies in the capacity of the material for consistently yielding excellent results in spite of the variations in operating conditions always associated with commercial production. For example, the exact gage at which the intermediate anneal is carried out between the two stages of cold rolling is less critical with magnetic results not varying much from the optimum over the range of gages described. The material is rendered less apt to be damaged in magnetic quality by an accidental increase in the temperature or duration of the intermediate anneal. It has also been discovered that the high-manganese material is less susceptible to the deleterious effects of aging before or after intermediate annealing such as might occur in intermittent production if the coils are allowed to stand several months in a partially finished condition. In the laboratory where conditions can be carefully controlled, it is found that this material and process results in reproducibility of magnetic properties between lots processed in identical fashion which is considerably greater than if low manganese material is used and if the cold rolling is done below the temperature of boiling water.

Having cold rolled the material in the first stage with the precautions mentioned, we next open anneal it preferably at 1850 to 1950° F., pickle and slit the edges if necessary. This annealing is not critical as to temperature, but is primarily employed to bring the sheet back to condition for further drastic cold work, and to cause the twinned nuclei to grow by accretion. It will be effective to use temperatures between 1500° to 2000° F., it being understood that the lower the temperature the longer the anneal must be continued to bring about the desired recrystallization.

The next step is a second cold rolling stage to which the same precaution regarding the temperature of the material should be applied, and, in which in our exemplary embodiment, we cold roll the material, which is now 0.025 inch thick, in two to six passes to .014 to .012 inch in thickness. This amounts to a reduction of the order of 52%, but again may be departed from as set forth above. We have, in particular, secured excellent results with 60% of cold rolling, and even higher percentages at this stage; although again too high a reduction such as would destroy the desired crystal orientation should be avoided.

The final step is a heat treatment, without an intervening pickling. This is preferably a flat anneal, and it may under some circumstances be carried on as an open anneal; but we prefer a box anneal, and for reasons which will be set forth hereinafter, we prefer a hydrogen anneal. In our exemplary embodiment we cut up the material at the end of the second cold rolling stage into sheets, clean those sheets by scrubbing or low temperature oxidation, coat them with magnesium oxide as a separator, and anneal them flat in a box for 12 to 30 hours at 2200° F., in an atmosphere of hydrogen in which the dew point is below −25° C. for at least the cooling portion of the temperature cycle, the hydrogen being kept low in impurities. The final anneal not only has its appropriate effect upon the crystal structure as indicated above, but also is of service

in producing a material having an unusually low core loss. One of the principal functions of this high temperature hydrogen anneal is to insure that the carbon content of the finished material is not substantially greater than .008% in order to realize this unusually low core loss. It will be competent to precede the hydrogen anneal with some other type of decarburizing anneal, if special steps are desired at this point for further carbon elimination. The resulting material is especially adapted for electrical transformer use since its permeability in the transformer range of inductions is exceptionally high, so that great economies can be made in transformer design; but it also increases the efficiency of the transformer, irrespective of its design, by reason of the low core loss which it has.

As we have indicated above, we can carry on our process with more than two stages of cold rolling. We achieve good results, for example, starting with a hot rolled material 0.109 inch in thickness, giving it an initial heat treatment as described, pickling it, and then cold rolling it to 0.059 inch, giving it an open anneal at 1850° F., pickling it, cold rolling it to 0.023 inch, again giving it an open anneal at a temperature of 1850° F., pickling it, finally cold rolling it to 0.014 inch, and ultimately giving it the hydrogen anneal which we have described. The same precautions should be observed in this three step cold rolling process as have been mentioned in connection with the two step process described. Again, the particular figures given are indicative rather than limiting.

Having thus described our invention, what we claim as new and desire to secure by Letters Patent, is:

1. A process of treating high-manganese magnetic alloys to produce high permeabilities at high induction in the rolling direction, which comprises hot rolling a manganese magnetic alloy to an intermediate gage, cold rolling the alloy by giving it a reduction of the order of between 60 and 85 per cent while keeping it at a temperature of between 250 to 700° F. during a major portion of the reduction, whereby to produce, along the cold rolling orientation, an incipient twinned condition of the crystals, annealing the alloy to cause the twinned condition to grow by accretion, again cold rolling the alloy with a reduction of the order of at least 50 per cent and not greater than 85 per cent while keeping it at a temperature of between 250 and 700° F. during a major portion of the reduction, and again annealing the alloy, whereby to produce in the final product a high degree of twin-derivative orientation.

2. A process of treating high-manganese silicon steel alloys to produce high permeabilities at high induction in the rolling direction, which comprises hot rolling a high-manganese silicon steel alloy to an intermediate gage, box annealing it, cold rolling it until it has been reduced in the order of 60 to 85 per cent while keeping it at a temperature of between 250 to 700° F. during a major portion of the reduction whereby to produce, along with cold rolling orientation, an incipient twinned condition of the crystals, annealing the alloy to cause the twinned condition to grow by accretion, again cold rolling the alloy with a reduction of the order of at least 50 per cent and not greater than 85 per cent while keeping it at a temperature of between 250 and 700° F. during a major portion of the reduction and again annealing the alloy, whereby to produce in the final product a high degree of twin-derivative orientation.

3. Process of treating high-manganese silicon steel alloys to produce high permeabilities at high induction in the rolling direction, which comprises hot rolling a high-manganese silicon steel alloy to an intermediate gage, box annealing it at substantially 1400° F., cold rolling it until it has been reduced in the order of 60 to 85 per cent while keeping it at a temperature of between 250 to 700° F. during a major portion of the reduction whereby to produce, along with cold rolling orientation, an incipient twinned condition of the crystals, annealing the alloy within a temperature range of 1500 to 2000° F., to cause the twinned condition to grow by accretion, again cold rolling the alloy with a reduction of the order of 50 to 85 per cent while keeping it at a temperature of between 250 to 700° F. during a major portion of the reduction and again annealing the alloy between 2000 to 2300° F., whereby to produce in the final product a high degree of twin-derivative orientation.

4. A process as set forth in claim 2 in which the magnetic alloy is a silicon steel containing silicon substantially in the range of 2.90 to 3.60 per cent, and manganese substantially in the range of .10 to .30 per cent.

5. A process as set forth in claim 2, in which the alloy is a silicon steel containing manganese substantially in the range of .10 to .50 per cent, and in which the alloy after the final cold rolling is given a decarburizing anneal to the extent of reducing the carbon therein to substantially no greater than .008 per cent.

6. A process as set forth in claim 2 in which the alloy is a silicon steel containing silicon substantially in the range of 1.00 to 4.50 per cent, manganese substantially in the range of .10 to .50 per cent, and in which the alloy after the final cold rolling is given a decarburizing anneal to the extent of reducing the carbon therein to substantially no greater than .008 per cent, and in which the box anneal after the hot rolling is carried on at 1300 to 1500° F., the intermediate anneal being carried on within the temperature range of substantially 1500 to 2000° F., and in which the final anneal is an anneal at 2000° to 2300° F., at least the cooling cycle of which is carried on in hydrogen.

7. A process of producing a high-manganese silicon steel characterized by high permeability at high induction and low core loss in the direction of rolling, which comprises providing an alloy containing from 2.90 to 3.60 per cent silicon, .10 to .30 per cent manganese, carbon not substantially greater than .02 per cent, sulphur not substantially greater than .03 per cent, balance substantially all iron, forming ingots of said alloy, rolling said ingots after heating to approximately 1800 to 2100° F. to a gage of the order of .105 inch, box annealing the alloy at a temperature of the order of 1400° F., cold rolling the alloy at a temperature of 250 to 700° F. during a major portion of the passes so as to give it a reduction of between 60 and 85 per cent, annealing the alloy at a temperature of the order of 1900° F., again cold rolling the alloy at a temperature between 250 to 700° F. during a major portion of the passes with a reduction of between 50 and 85 per cent, again annealing the alloy at a temperature of substantially 2200° F., the said annealing being a decarburizing anneal.

8. A cold rolled magnetic alloy containing silicon within the range of 1.00 to 4.50 per cent, and manganese substantially between .10 and .50 per cent, the balance being substantially all iron and characterized by high permeability at high induction in the direction of rolling, and a high degree of twin-derivative crystal orientation.

9. A cold rolled magnetic alloy, containing silicon within the range of 2.90 to 3.60 per cent, manganese within the range of .10 to .30 per cent, the balance being substantially all iron with carbon not to exceed .008 per cent, and characterized by a high permeability at high induction in the direction of rolling, and a high degree of twin-derivative crystal orientation.

10. A process of producing a high-manganese silicon steel alloy characterized by high permeability at high induction in the direction of rolling, which comprises providing an alloy containing from 2.00 to 3.50 per cent silicon and .10 to .50 per cent manganese, hot rolling said alloy to substantially .109 inch in thickness, giving it an initial box annealing at a temperature of the order of 1400° F., pickling it, and then cold rolling it to substantially .059 inch, annealing it at around 1850 to 1950° F., pickling it, cold rolling it to substantially .023 inch, again annealing it at around 1850 to 1950° F., pickling it, cold rolling it to substantially .013 inch, and giving it a final annealing at around 2000 to 2300° F., each of the cold rolling steps being so carried out that the material is at a temperature between 250 to 700° F., during the major portion of each reduction.

11. A process of producing a high-manganese silicon steel alloy having high permeability at high induction in the direction of rolling, which comprises the steps of cold rolling the alloy which contains manganese substantially within the range of .10 to .50 per cent to give it a reduction of the order of between 60 to 85 per cent while keeping it at a temperature of between 250 to 700° F. during a major portion of the reduction, annealing and pickling it, giving it a cold reduction of between 50 to 85 per cent while keeping it at a temperature of between 250 to 700° F., during a major portion of the reduction and finally giving it an anneal at 2000 to 2300° F.

12. A process as set forth in claim 2 in which the alloy is a silicon steel containing silicon substantially in the range of 1.00 to 4.50 per cent, manganese substantially in the range of .10 to .50 per cent, and in which the box anneal after the hot rolling is carried on at between 1300 to 1500° F., and in which the anneal between the two stages of cold rolling is carried out within the temperature range of substantially 1500 to 2000° F., and in which the final anneal is an anneal at 2000 to 2300° F., at least the cooling cycle of which is carried on in hydrogen, and in which during one or more of the steps, decarburization has been caused to take place to the extent that the final material contains carbon substantially no greater than .008 per cent.

13. A cold rolled magnetic alloy, containing silicon within the range of 1.00 to 4.50 per cent, manganese within the range .10 to .50 per cent, the balance being substantially all iron, and characterized by a high permeability at high induction in the direction of rolling, and a high degree of twin-derivative crystal orientation.

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