This invention relates to silicon steel. More particularly it relates to a method of heat treating cold rolled silicon steel strip to enhance its magnetic qualities.

In the production of magnetic cold rolled silicon steel strip a usual practice comprises cold rolling the material in a temperature which will permit of about 0.10" to about 0.014" with intervening heat treatment followed by a decarburizing heat treatment and a final stage texture developing and mechanical strain relieving heat treatment at a temperature in excess of 1100° C. The final structure consists of well-oriented, i.e. (110) [001], crystals mixed with more or less poorly oriented crystals. The ultimate goal in the various processes and heat treatment is to attain the maximum preferred (110) [001] magnetic crystal orientation in the steel, thus enhancing its magnetic characteristics and use in electro-magnetic products.

An object of the present invention is to provide a method for further improving the magnetic characteristic of cold rolled silicon steel strip.

More specifically an object of the invention is to provide a two-stage final texture developing anneal which will improve the magnetic orientation of the crystals in cold rolled silicon steel.

Other objects will become apparent and the invention will be better understood from the following description, and the features of novelty which characterize the invention will be pointed out in the claims annexed to and forming a part of this specification.

It has been found that a material of superior magnetic qualities can be obtained by subjecting the silicon strip as it comes from the final cold rolling stage and after decarburization to a two part heat treatment which comprises holding it for a time at a temperature which encourages and permits the nucleation or first appearance of well oriented crystals and their growth at the expense of the suitably prepared matrix and then holding at a temperature which will permit the completion of growth of any well oriented crystals which have not already attained maximum size to develop the maximum metal texture.

More specifically it has been found that holding a cold rolled silicon steel strip containing about 1.5% to 4% silicon with low impurities after it comes from the final rolling and decarburization stages at a temperature of about 850° C. to 900° C. preferably in a reducing or non-oxidizing atmosphere for a period of time sufficient to permit the nucleation or first appearance and preliminary growth of crystals of preferred magnetic orientation, and then holding it at a temperature ranging from 900° C. to about 1200° C. preferably in reducing atmosphere to permit the maximum growth of such preferably oriented crystals which have not completed their growth and to develop the final magnetic properties, the temperature being about the absorption of any remaining crystallites results in a material having enhanced magnetic characteristics and freedom from mechanical strain. The lower temperatures for the final treatment normally take a longer time to accomplish the result than the higher ten second maximum number of favorably oriented crystals without encouraging the growth of such numbers of poor magnetic crystals.

In accordance with prior practices the silicon steel strip is usually reduced to final thickness in one or more stages separated by heat treatment normally at a temperature under 1000° C., followed by a decarburization treatment and a final one stage anneal or heat treatment at a temperature of about 1175° C. to produce an improved crystal orientation. In the present invention there is employed a two-stage heat treatment or anneal after the final cold rolling and decarburization stages by means of which better magnetic qualities are achieved than in the prior one-stage final anneal.

It has been found that the optimum temperature range for the nucleation or appearance and growth of crystals in most magnetic cold rolled silicon steels with from about 1.5 to 4% silicon and low impurities falls between about 850° C. to 900° C. with the optimum mean temperature at about 875° C. The 875° C. temperature is somewhat above the temperature of nucleation or first appearance of well oriented, i.e. (110) [001], crystals which usually occurs at about 850° C., but the rate of growth at such a low temperature is too slow from the practical viewpoint. At 875° C. the growth of well oriented crystals is rapid enough to give complete crystal growth in from about 4 to 24 hours depending upon the specific steel composition. It has also been noted that at 875° C. the nucleation and growth of well oriented crystals is still definitely favored as against the formation and growth of poorly oriented crystals, a condition which still exists in a comparative sense at 900° C. However, at about 925° C. the poorly oriented crystals' growth has increased to the point where the percentage of crystals with preferred orientation and magnetic quality has proportionately decreased. The purpose of the present invention is to obtain a growth of the maximum number of favorably oriented crystals without encouraging the growth of such numbers of poor magnetic crystals.
as will detract appreciably from the overall magnetic improvement. This result can be obtained by heat treatment at temperatures up to 775 C. and 900 C. for the above material with the optimum temperature at about 875 C. The material should be maintained at the 850 C. to 900 C. temperature until the great majority or at least 78% of the fine-grained matrix has been absorbed by the (110) 501 crystals which condition will usually occur within about 4 to 24 hours depending upon the particular steel being treated.

After a preponderant number of well oriented crystals have been produced in the strip, it is subjected to higher temperatures to complete the maximum growth of any crystals which have not already attained maximum size, to absorb any remaining small crystallites to make the large well oriented crystals more perfect, and to relieve strain. The temperature may be varied rather widely from about 900 C. to about 1175 C. depending upon such factors as the length of time economically available and desirable for treatment, the temperature attainable with furnaces on hand, the problem of laminations sticking together, and other considerations. Heat treatment at temperature slightly below 1000 C., realizable at low cost in many furnaces, will obviate the sticking together of stacked laminations without special treatment and will assure any desired completion of crystal growth, perfection of the texture of the strip or absorption of any remaining crystallites and the relief of most mechanical strains in from about one to eight hours depending upon the conditions in the steel. However, the best temperature for the second step of the heat treatment, one which will afford any necessary further crystal growth, perfection of the well oriented crystals as well as complete relief of mechanical strain in a very short period or up to about one hour is a temperature of about 1175 C.

In actual practice it is preferred to start with a high quality open hearth silicon steel strip 0.10 inch thick containing from 1.5–4% silicon. The manganese-sulfur ratio is preferably between 2:1 and 4:1 with manganese not over 0.66% and sulfur not over 0.025%. The material is cold rolled from 0.10 inch to about 0.014 inch in two stages the last cold reduction being of the order of 50% with a 925 C. continuous anneal interposed between the two stages. Carbon in the ladle stage should be less than 0.03% is reduced to 0.06% or under by a continuous heat treatment at about 900 C.

The final rolled and decarburized product is heat treated preferably in a reducing (or non-oxidizing) atmosphere at a temperature of from 853 C. to 900 C. with a preferable mean temperature of 875 C. for from about 4 hours to 24 hours or until at least about 75% of the fine-grained matrix has been absorbed by the well oriented crystals. In the case of the preferred steel this will occur in about 8 hours. Gases which will afford a reducing or non-oxidizing atmosphere suitable for this stage of the treatment are hydrogen, nitrogen, helium and various partially combusted or cracked gases.

The final texture perfecting stage for preferably oriented crystals and the relief of mechanical strain is carried out at temperatures ranging from 900 C. to about 1200 C. depending upon the factors mentioned heretofore. Any necessary completion of crystal growth will likewise occur in this stage. For the above material the holding at a temperature of 900 C. for eight hours will afford maximum crystal growth and absorption of small crystallites and relieve most strains. On the other hand, holding at about 1175 C. for about one hour will give maximum crystal size if growth has not already been completed in the first stage, perfect the texture and relieve all mechanical strain.

The two stage heat treatment according to the present invention may be carried on continuously in one cycle or the material may be cooled in the usual manner after the first part and the final part resumed at any convenient time.

Employing the present two-part final anneal for a cold rolled silicon steel strip of the composition described above, as compared with the prior practice, there have been obtained the results set forth in the magnetization data below. The first set of figures is for steel with a final single anneal at 1175 C. for several hours, followed by data for a two part anneal first at 875 C. for about 8 hours followed by heating at 980 C. for about eight hours which is not completely effective in eliminating all mechanical strain. The last set of results is for steel of the same composition which has been held at 875 C. for about 8 hours followed by treatment at 1175 C. for about one hour.

<table>
<thead>
<tr>
<th>Anneal</th>
<th>H_{max}</th>
<th>Residual Br</th>
<th>Coercive Me</th>
<th>Hysteresis Ergo/cm³</th>
<th>Watts/Lb.</th>
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<td>Prior Method</td>
<td>1175 C.</td>
<td>1.42</td>
<td>12500</td>
<td>600</td>
<td>559</td>
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<tr>
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<td>1.15</td>
<td>15500</td>
<td>100</td>
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<tr>
<td>Prior Method</td>
<td>1175 C.</td>
<td>0.70</td>
<td>15500</td>
<td>800</td>
<td>249</td>
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From the above it will be seen the magnetic qualities of cold rolled silicon steel strip are developed by a two stage final anneal at temperatures of 875 C. and 980 C. to essentially the same degree and more cheaply than with a single 1175 C. anneal. On the other hand heat treatment after the final cold rolling stage for about 8 hours at 875 C. followed by a one hour treatment at 1175 C. gives definitely better results as concerns values of H for varying B where B=μH2; lower hysteresis loss and a substantial reduction in watt losses per pound.

What we claim as new and desire to secure by Letters Patent of the United States is:

1. The method of heat treating cold rolled sil-
con steel strip containing 1.5 to 4% silicon which has been decarburized at about 800° C. which method comprises holding the strip in a temperature range of from 850° to 900° C. which encourages the nucleation or first appearance and growth of (110) [001] crystals until at least 75% of the fine-grained matrix has been absorbed by the growth of such crystals followed by heat treatment at a higher temperature of from 900° to 1200° C. which will permit further growth of the (110) [001] crystals and perfection of the texture of said cold-rolled strip and relieve mechanical strain.

2. The method of heat treating cold rolled silicon steel strip containing 1.5 to 4% silicon which comprises decarburizing said cold rolled strip at about 800° C. holding the strip at a temperature of from 850° to 900° C. in a non-oxidizing atmosphere for from about four to about twenty-four hours and thereafter subjecting the strip to a heat treatment at a higher temperature between 900° C. and about 1000° C. in a reducing atmosphere for from about one to eight hours.

3. The method of heat treating cold rolled silicon steel strip containing 1.5 to 4% silicon material which comprises decarburizing said steel at about 800° C. and thereafter holding the material at a temperature of about 875° C. for about four to twenty-four hours in a non-oxidizing atmosphere followed by heat treatment at a temperature of about 980° C. in a reducing atmosphere for about one to eight hours.

4. The method of heat treating cold rolled silicon steel strip containing 1.5 to 4% silicon which comprises decarburizing the strip at 800° C. and thereafter subjecting the strip to a temperature of from 850° to 900° C. in a non-oxidizing atmosphere for from about four to about twenty-four hours followed by treatment in a reducing atmosphere at a higher temperature of from 900° C. to 1000° C. for from about one to eight hours.

5. The method of heat treating cold rolled silicon steel strip containing from about 1.5–4% silicon which has been cold reduced from about 0.10 inch to about 0.014 inch in two stages separated by a continuous anneal at about 925° C. the final cold reduction being in the order of 50%; which comprises decarburizing said strip at 800° C. and thereafter holding the final rolled and decarburized material at a temperature of from about 850° C. to about 900° C. in a non-oxidizing atmosphere for from about four to about twenty-four hours followed by a heat treatment in a reducing atmosphere at a higher temperature of from about 900° C. to 1300° C. for from about one to eight hours.

6. The method of heat treating cold rolled silicon steel strip containing from about 1.5–4% silicon which has been cold reduced from about 0.10 inch to about 0.014 inch in two stages separated by a continuous anneal at about 925° C. the final cold reduction being the order of 50%; which comprises decarburizing the cold rolled strip material at 800° C. and thereafter holding the final rolled and decarbonized material at a temperature of from about 850° C. to about 900° C. in a non-oxidizing atmosphere for from about four to about twenty-four hours followed by a heat treatment in a reducing atmosphere at a higher temperature of from about 900° C. to about 1000° C. for from about one hour to about 8 hours.

7. The method of heat treating cold rolled silicon steel strip containing from about 1.5–4% silicon which has been cold reduced from about 0.10 inch to about 0.014 inch in two stages separated by a continuous anneal at about 925° C. the final cold reduction being the order of 50%; which comprises decarburizing the cold rolled strip material at 800° C. and thereafter holding the final rolled and decarbonized material at a temperature of from about 850° C. to about 900° C. in a non-oxidizing atmosphere for from about four to about 24 hours followed by a heat treatment in a reducing atmosphere at about 1175° C. for about one hour.

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