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(54) **Process for producing cube-on-edge oriented electromagnetic silicon steel.**

(57) A method is provided for producing electromagnetic silicon steel to produce a cube-on-edge grain orientation having lower core losses and higher magnetic permeability. The method includes, during final texture annealing, using a controlled heating cycle including an isothermal hold at a selected recrystallization temperature of about 1650°F (900°C) for 6 to 20 hours to improve secondary recrystallization and the Goss texture (110) [001], then heating the steel from the selected recrystallization temperature to in excess of 2000°F (1093°C).

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PROCESS FOR PRODUCING CUBE-ON-EDGE ORIENTED ELECTROMAGNETIC SILICON STEEL

This invention relates to a process for producing electromagnetic silicon steel having a cube-on-edge orientation and particularly to a final texture annealing cycle to promote improved secondary recrystallization. Particularly, the invention relates to a substantially isothermal anneal at a selected recrystallization temperature.

In the manufacture of grain-oriented silicon steel, it is known that if improved secondary recrystallization texture, e.g., Goss texture (110) [001], is achieved, the magnetic properties, particularly permeability and core loss, will be correspondingly improved. The Goss texture (110) [001], in accordance with Miller's indices, refers to the body-centered cubes making up the grains or crystals being oriented in the cube-on-edge position. The texture or grain orientations of this type refers to the cube edges being parallel to the rolling direction and in the plane of rolling, and the cube face diagonals being perpendicular to the rolling direction and in the rolling plane. As is well known, steel having this orientation is characterized by a relatively high permeability in the rolling direction and a relatively low permeability in a direction at right angles thereto.

The development of a cube-on-edge orientation is dependent upon a mechanism known as secondary recrystallization. During secondary recrystallization, secondary cube-on-edge oriented grains are preferentially grown at the expense of primary grains having a different and undesirable orientation. The steel composition, particularly the impurity contents, the processing operations including hot rolling and the degree of deformation in each cold-rolling operation, intermediate and final continuous annealing time and temperature cycles, and the final texture annealing procedure must all be carefully controlled to attain the optimum texture development. A steel that has not obtained optimum texture development may have a substantially uniform but inadequate grain size and structure and resulting poor magnetic properties or may exhibit a "banding" of inferior grain structure. Generally, banding means areas or bands of inferior grain structure extending across the width of the coil surrounded by areas of well-textured steel. Generally, the initial phases of secondary recrystallization occur at about 1550°F (843°C), however, secondary grain growth proceeds much faster and more efficiently at temperatures of about 1600°F (871°C) or more. The operation through which the secondary grains are preferentially grown and consume the primary grains is known as final texture annealing.

In the manufacture of grain-oriented silicon steel, the typical steps include subjecting the melt of 2.5-4% silicon steel through a casting operation, such as a continous casting process, hot rolling the steel, cold rolling the steel to final gauge with an intermediate annealing when two or more cold rollings are used, decarburizing the steel, applying a refractory oxide base coating to the steel, and final texture annealing the steel, such as in a hydrogen atmosphere, to produce the desired secondary recrystallization, and purification treatment to remove impurities, such as nitrogen and sulfur. The final texture annealing is typically performed at a temperature in excess of 2000°F (1093°) and held for an extended time period of at least 4 hours and generally longer to remove impurities.

A typical thermal cycle of the final texture annealing practice may include a reasonably continuous heating rate of approximately 50°F/hour (27.8°C/hour) from the charge temperature of the coated strip to a temperature high enough to effect purification. The charge temperature in mill practice, typically, is of the order of room temperature of 80°F (26.7°C) or more and the purification temperature may range from 2000°F (1093°C) up to a maximum of about 2300°F (1260°C) and preferably up to 2250°F (1232°C). The steel is generally subjected to a soaking at the purification temperature to remove the impurities for a long time, typically of the order of about 20 hours at or higher than 2100°F (1150°C).

Numerous attempts by others have been made to improve the final texture. U.S. Patent 2,534,141 - Morrill et al discloses a two-stage final texture anneal to improve the orientation. First, the cold-rolled decarburized sheet is held for 4-24 hours at 850-900°C (1560-1650°F), and preferably at 875°C (1605°F), in a reducing or non oxidizing atmosphere to encourage and permit nucleation of well-oriented crystals and their growth. Second, the steel is then held at a temperature of 900 to 1200°C (1650-2192°F), and preferably 1175°C (2147°F), in a reducing atmosphere to permit completion of the growth of the well-oriented crystals and to relieve mechanical strain.

U.S. Patent 4,157,925 - Malagari et al discloses a process for producing a cube-on-edge orientation in a boron-inhibited silicon steel. The process includes heating the steel from a temperature of 1700 to 1900°F (926 to 1038°C) at an average rate of less than 30°F/hour (16.7°C/hour) so as to provide a minimum time period for the selective grain-growth process to occur and to final texture anneal the steel by heating to a temperature in excess of 2000°F (1093°C) and to a maximum temperature of 2300°F (1260°C) for purification of the steel.

U.S. Patent 4,318,758 - Kuroki et al discloses in Example 3 a method for producing grain-oriented silicon steel containing aluminium wherein the decarburized and coated sheet is heated up to 900°C (1650°F) in a 75% H_2 and 25% N_2 atmosphere with a heating rate of 20°C/hour (36°F/hour), then heating between 900 to 1050°C (1650-1922°F) in the same atmosphere at a heating rate of 5°C/hour (9°F/hour), between 1050 and 1200°C (1922-2192°F) in 100% H_2 atmosphere at a heating ratio of 20°C/hour (36°F/hour) where the steel is maintained at 1200°C (2192°F) for 20 hours in the 100% H_2 atmosphere.

None of these patents disclose the present invention. What is needed is an improved final texture annealing process wherein improved cube-on-edge orientation of the secondary grains may be achieved during secondary recrystallization to result in improved permeability and core loss values. The improved final texture annealing process should include control of the heating cycle and result in improved productivity as measured by an overall improvement in quality.

It is known that variations occur in magnetic properties within a given coil of silicon steel. The variations can be measured by taking samples from the coil ends and measuring the core loss values of those samples. A convenient measure of quality improvement is the percentage of coils having a poor end core loss at 60 Hz equal to or less than 0.714 WPP at 17 KG (1.57 WPKg at 1.7 Tesla). It is desirable to improve productivity so that an increasing percentage, and preferably the majority, of the coils produced satisfy minimum core loss values, such as that above.

It is also an objective to develop a process which substantially eliminates the "banding" problem.

In accordance with the present invention, a process is provided for producing electromagnetic silicon steel having cube-on-edge orientation wherein the process includes the conventional steps of preparing a steel melt containing 2.5-4% silicon, casting the steel, hot rolling the steel, cold rolling the steel to final gauge, decarburizing the steel, applying a refractory oxide base coating to the steel, and final texture annealing the steel by heating to and maintaining at a temperature in excess of 2000°F (1093°C). The improvement comprises heating the steel during the final texture annealing to a selected recrystallization temperature within the range of 1600 to 1700°F (871 to 926°C), isothermally heating the steel at that temperature for 6 to 20 hours to substantially complete secondary recrystallization, and heating the steel from that isothermal hold temperature to a temperature in excess of 2000°F (1093°C) to effect purification.

The present invention will be more particularly described with reference to the accompanying drawings, in which:-

Figures 1a and 1b are plots of core loss and permeability respectvely, versus hold temperature for 11-mil (0.279mm) steel; and

Figures 2a and 2b are plots of core loss and permeability, respectively, versus hold temperature for 9-mil (0.229mm) steel.

The final texture annealing process of the present invention includes a controlled heating cycle wherein the steel is substantially isothermally annealed at selected temperatures for particular periods of time to effect substantially complete secondary recrystallization. As used herein, isothermal heating or annealing during recrystallization means heating at a very low heating rate. The heating rate need not be zero, but preferably should be less than 10°F/hour (5.5°C/hour), and more preferably less than 5°F/hour (2.8°C/hour). As a practical consideration, it is difficult to isothermally hold at a particular temperature in a production furnace, but very small variations in heating rate about a selected recrystallization temperature is within the scope of the invention. Most preferably such an isothermal hold shall mean a heating rate of less than 5°F/hour (2.8°C/hour).

Specific processing of the steel up to final texture annealing may be conventional and is not critical to the present invention. The specific processing may include a number of conventional steps which include preparing a melt of the steel, casting the steel, hot rolling the steel, cold rolling the steel to final gauge with intermediate annealing steps, decarburizing the steel, applying a refractory oxide base coating, and then final texture annealing the steel in excess of 2000°F (1093°C).

Although the texture annealing method of the invention described in detail hereinafter has utility with grain-oriented silicon steel generally, the following typical compostion (in weight %) is one example of a silicon steel composition adapted for use with the method of this invention:

C	Mn	S	Cu	Si	Fe
0.030	0.065	0.025	0.22	3.15	Balance

To illustrate the several aspects of the final texture annealing process of the present invention, various samples of a silicon steel having a composition similar to the above-described typicial composition were processed and the results of the tests are shown in the following Table I.

TABLE I

Sample Group	No. of Samples	Hold Temp. (°F)	Hold Time (Hrs.)	Average WPP at 17 KG	Average μ at 10 H (Gauss)
A	18	None	-	.754	1812
B	25	None	-	.746	1820
C	25	None	-	.726	1819
D	25	1600	6	.706	1833
E	25	1650	6	.711	1830
F	25	1700	6	.728	1824
G	25	1750	6	.736	1816
H	17	None	-	.730	1821
I	17	1460	6	.724	1828
J	17	1540	6	.724	1823
K	17	1650	6	.706	1834
L	17	1600	6	.719	1828
M	17	1600	12	.717	1827
N	15	None	-	.727	1820
O	15	1550	12	.731	1816
P	15	1600	6	.737	1820
Q	15	1650	6	.718	1832
R	15	1700	12	.736	1815
S	11	1600	50	.707	1831
T	15	1550	50	.744	1812
U	15	1600	6	.731	1821
V	15	1600	20	.695	1838
W	15	1650	6	.703	1833
X	15	1650	20	.708	1832
Y	15	1700	6	.740	1812
Z	15	1700	20	.738	1814
AA	15	1550	12	.731	1816
BB	15	1600	12	.717	1833
CC	15	1650	12	.709	1837
DD	15	1700	12	.736	1815

All the Sample Groups of Table I were obtained from various heats of nominally 11-mil (0.279mm) gauge silicon steel having the above-identified typical composition. The samples were all coated with MgO slurry and heated from a charge temperature at a relatively constant heating rate of about 50°F/hour (27.7°C/hour) or greater. Groups D-G and I-M and O-DD were all heated from charge temperature up to the specified hold temperature. Sample Groups A, B, C, H and N were not isothermally annealed and so were not held at any temperature, but were heated from the charge temperature up to a purification soak temperature. All the Sample Groups were texture annealed in a hydrogen atmosphere at a soak temperature of 2150°F (1177°C). Groups A-Z were held at 2150°F for 20 hours, and Groups AA-DD for 10 hours.

The magnetic properties listed in Table I represent an average value for core loss and permeability for the number of samples for that group. The distrubution of 60 Hz core losses at 17 KG (1.7 Telsa) and permeability at 10 Oersteds for those samples are shown in Figures 1a and 1b.

The data show that generally the samples which were held for time at a temperature within the recrystalization range of 1600 to 1700°F (871 to 726°C) have improved properties over those samples not held at temperature (Samples A, B, C, H and N). The data demonstrate that annealed samples demonstrate incomplete recrystallization if the hold temperature is 1550°F (843°C). All samples were completely recrystallized at about 1650°F (900°C) hold temperature. The data also suggest that within the 1600-1700°F (871 to 926°C) range, there may be a range of temperatures within which substantial recrystallization occurs so as to result in improved magnetic properties. The range of about 1600-1650°F (871-900°C) is preferred.

The hold time for the isothermal anneal is also critical. Insufficient time results in incomplete recrystallization. Too much time will generally result in some deterioration of magnetic properties, as shown by Groups S and T at 50 hours hold time. Results of tests have shown that the hold times of 6 to 20 hours provide good properties with a practical preferred time being about 12 hours.

TABLE II

Sample Group	No. of Samples	Hold Temp. (°F)	Hold Time (Hrs.)	Average WPP at 17 KG	Average μ at 10 H (Gauss)
A	25	1550	12	.731	1808
B	25	1600	12	.728	1808
C	25	1650	12	.686	1853
D	25	1700	12	.706	1829
E	6	None	-	.738	1800
F	6	1650	12	.682	1825
G	6	1550	12	.733	1789
H	6	1550	50	1.010	1640
I	6	1650	50	.681	1818
J	6	1600	50	.796	1761
K	6	1700	12	.693	1817
L	6	1600	12	.716	1809
M	9	1600	12	.717	1804
N	9	1650	40	.675	1827
O	9	1650	40	.662	1834
P	25	1550	12	.726	1815
Q	25	1650	12	.691	1851
R	25	1650	12	.683	1838
S	25	1700	12	.706	1829

All the Sample Groups of Table II were obtained from various heats of nominally 9-mil (0.229mm) gauge silicon steel having the same nominal composition as for the 11-mil samples of Table I. The samples were all coated with MgO slurry and heated from a charge temperature at a relatively constant heating rate of about 50°F/hour (27.7°C/hour) or greater. All of the Sample Groups, except Group E, were heated from charge temperature up to the specified hold temperature. Sample Group E was not isothermally annealed and so was not held at temperature, but was heated from the charge temperature up to a purification soak temperature. All the Sample Groups were texture annealed in a hydrogen atmosphere at a soak temperature of 2150°F (1177°C) and held for 10 hours.

The magnetic properties listed in Table II represent an average value for core loss and permeability for the number of samples for that group. The distribution of 60 Hz core losses at 17 KG (1.7 Tesla) and permeability at 10 Oersteds for those samples are shown in Figures 2a and 2b.

The data show that for 9-mil gauge as with the 11-mil gauge, the annealed samples were incompletely recrystallized at 1550°F (843°C), but completely recrystallized at about 1650°F (900°C) hold temperature. The data also suggest that within the 1600-1700°F (871 to 926°C) range, there may be a range of temperatures within which substantial recrystallization occurs with a corresponding improvement in magnetic properties. The range of about 1650-1700°F (900-926°C) is preferred and is slightly higher than the range for the thicker, 11-mil steel.

The data also confirm that the hold times for the isothermal anneal are critical. As with the 11-mil data, the 9-mil samples demonstrate some deterioration of magnetic properties at 50 hours hold time, as shown by Groups H, I and J. Groups H and J show such poor properties that they are not plotted in figures 2a and

2b. It appears that the thin guage 9-mil material is even more sensitive to hold times than the 11-mil material. Results of tests have shown that hold times up to 20 hours provide good results, preferably 6 to 20 hours, and a practical preferred time of about 12 hours.

The overall results show that a dramatic improvement in overall magnetic properties of core loss and permeability result from both 9-mil and 11-mil steel when processed by an isothermal anneal for 6-20 hours within the range 1600-1700°F (871 to 926°C). The preferred ranges for each differ within that range, but the best combination of properties and complete secondary recrystallization occurs at about 1650°F (900°C) for both gauges.

The method of the present invention relates to an improved final texture annealing process wherein the steel is heated to a recrystallization temperature within the range of 1600 to 1700°F (871 to 926°C). The heating rate may be of the order of a conventional 50°F per hour (27.7°C/hour) and the selected isothermal hold temperature be about 1650°F (900°C). The steel is then isothermally heated by holding the steel at that temperature for about 6 to 20 hours, preferably about 12 hours, to substantially complete secondary recrystallization. Thereafter the steel is heated from that temperature to a purification temperature in excess of 2000°F (1093°C), preferably about 2000°F (1204°C), at a heating rate such as 50°F per hour (27.7°C/hour) and held at that temperature to effect purification. Generally, the heating rate up to the hold temperature and up to the purification temperature are relatively constant heating rates. The heating rate, however, does not appear to be critical to significantly affect the properties.

An advantage of the method of the present invention is that secondary recrystallization is essentially completed during the isothermal portion of the heat treatment, rather than being completed in accordance with conventional practice during heating to the higher purification temperature. As has been demonstrated, the effect of the present invention is to improve both magnetic permeability and core loss values. The method of the present invention is able to improve the magnetic properties in a manner not heretofore recognized in the art.

CLAIMS:

1. A process for producing electromagnetic silicon steel having a cube-on-edge orientation, which process includes the steps of preparing a steel melt containing 2.5 to 4% silicon, casting the steel, hot rolling the steel, cold rolling the steel to final gauge, decarburizing the steel, applying a refractory oxide base coating to the steel, and final texture annealing the steel by heating to and maintaining a temperature in excess of 2000°F (1093°C), characterised in comprising the steps of during final texture annealing, heating the steel to a recrystallization temperature within the range of 1600 to 1700°F (871 to 926°C); substantially isothermally heating the steel in the recrystallization temperature range for 6 to 20 hours to substantially complete secondary recrystallization; and heating the steel from the selected recrystallization temperature to a temperature in excess of the 2000°F (1093°C).

2. A process according to Claim 1, wherein the recrystallization temperature range is from 1600 to 1650°F (871 to 900°C) for steel having a gauge of substantialy 11 mils (0.279mm).

3. The process according to Claim 1, wherein the recrystallization temperature range is from 1650 to 1700°F (900 to 926°C) for steel having a gauge of substantially 9 mils (0.229mm).

4. A process according to Claim 1, 2 or 3, wherein the steel is isothermally heated at 1650°F (900°C) for substantially 12 hours.

5. A process according to any one of claims 1 to 4, wherein the substantially isothermal heating occurs at 10°F/hour (5.5°C/hour) or less.

6. A process according to any one of claims 1 to 5, wherein the substantially isothermal heating occurs at 5°F/hour (2.8°C/hour) or less.

7. A process according to any one of the preceding claims, wherein the recrystallization temperature range is dependent upon the thickness of the steel.

8. A process according to any one of the preceding claims, wherein the recrystallization temperature range is slightly higher for thinner steels.

9. A process for producing electromagnetic silicon steel having a cube-on-edge orientation, which process includes the steps of preparing a steel melt containing 2.5 to 4% silicon, casting the steel, hot rolling the steel, cold rolling the steel to final gauge, decarburizing the steel, applying a refractory oxide base coating to the steel, and final texture annealing the steel by heating to and maintaining at a temperature in excess of 2000°F (1093°C), characterised in comprising heating the steel during final texture annealing at a relatively constant heating rate to substantially 1650°F (900°C); isothermally heating the steel at the selected temperature for 6 to 20 hours to substantially complete secondary recrystallization; and then heating the steel at a relatively constant heating rate to a temperature in excess of the 2000°F (1093°C).

FIG. 1a

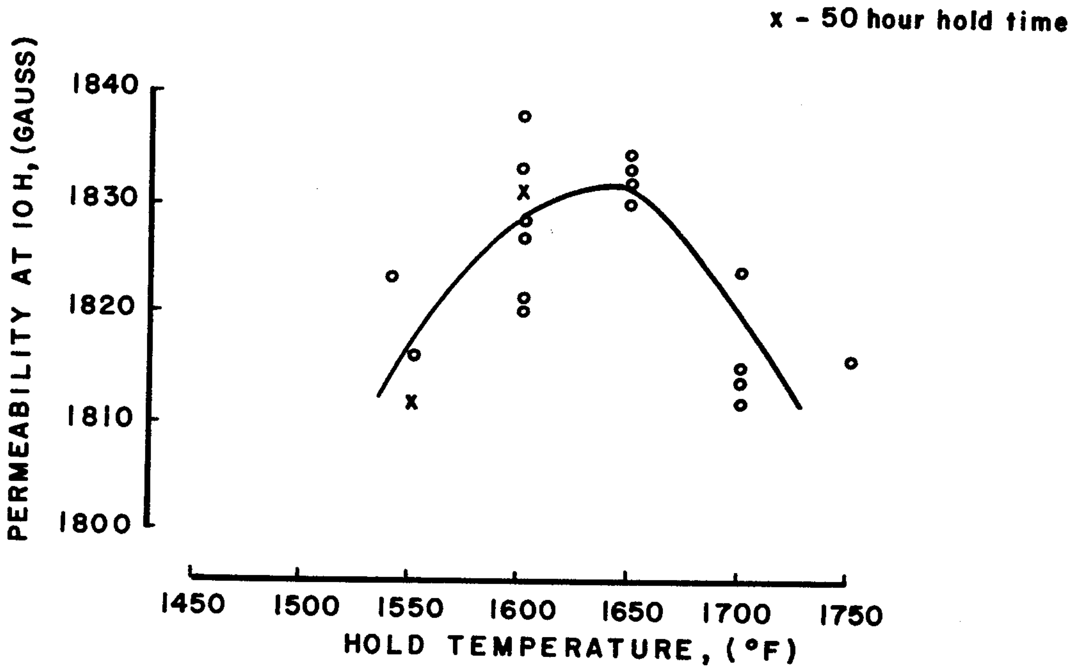
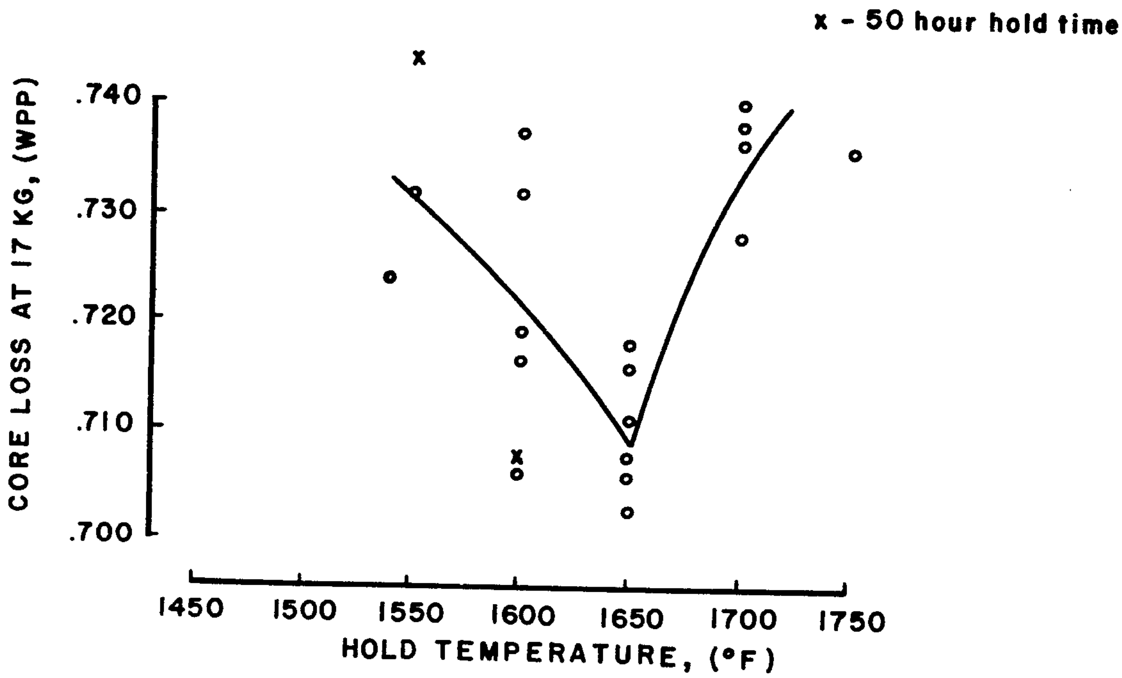

x - 50 hour hold time
PERMEABILITY AT 10 H, (GAUSS)
1840
1830
1820
1810
1800
1450
1500
1550
1600
1650
1700
1750
HOLD TEMPERATURE, (°F)

FIG. 1b


x - 50 hour hold time
CORE LOSS AT 17 KG, (WPP)
.740
.730
.720
.710
.700
1450
1500
1550
1600
1650
1700
1750
HOLD TEMPERATURE, (°F)

2/2

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FIG. 2a

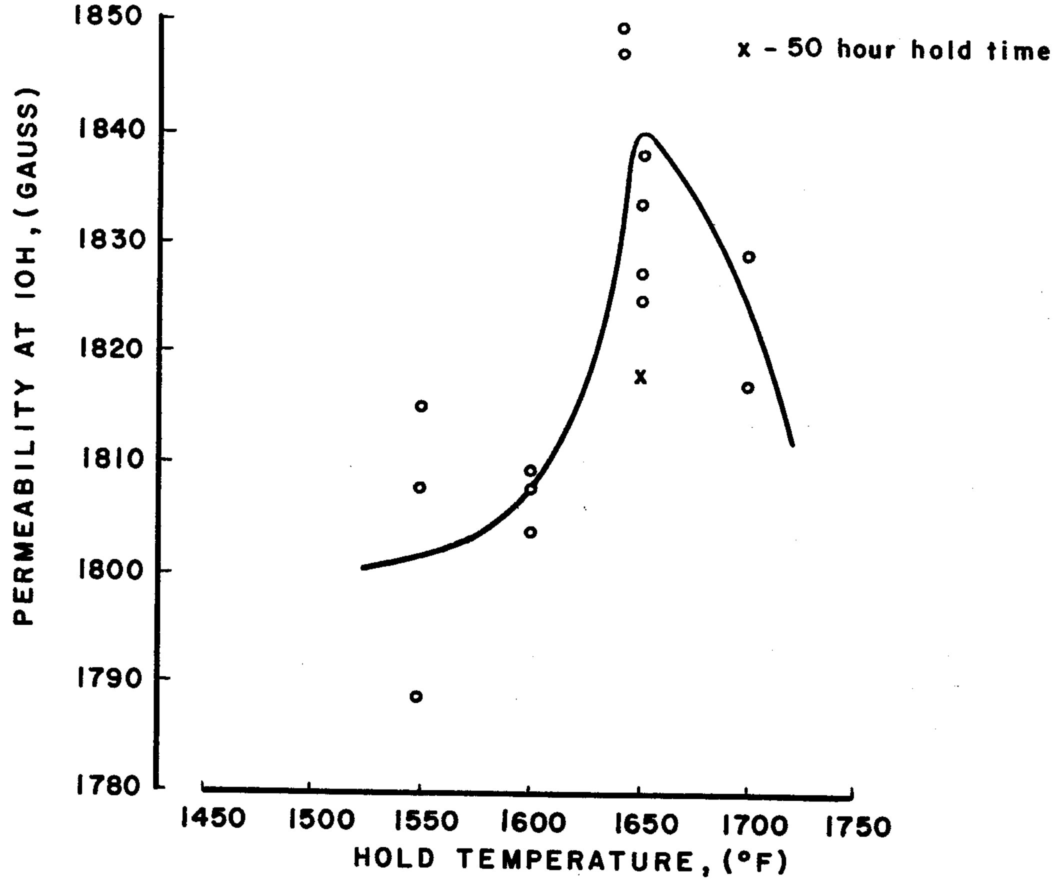

FIG. 2b