A STUDY OF THE GRAIN GROWTH OF A LOW CARBON STEEL

THESIS FOR DEGREE OF M. S.

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THESE

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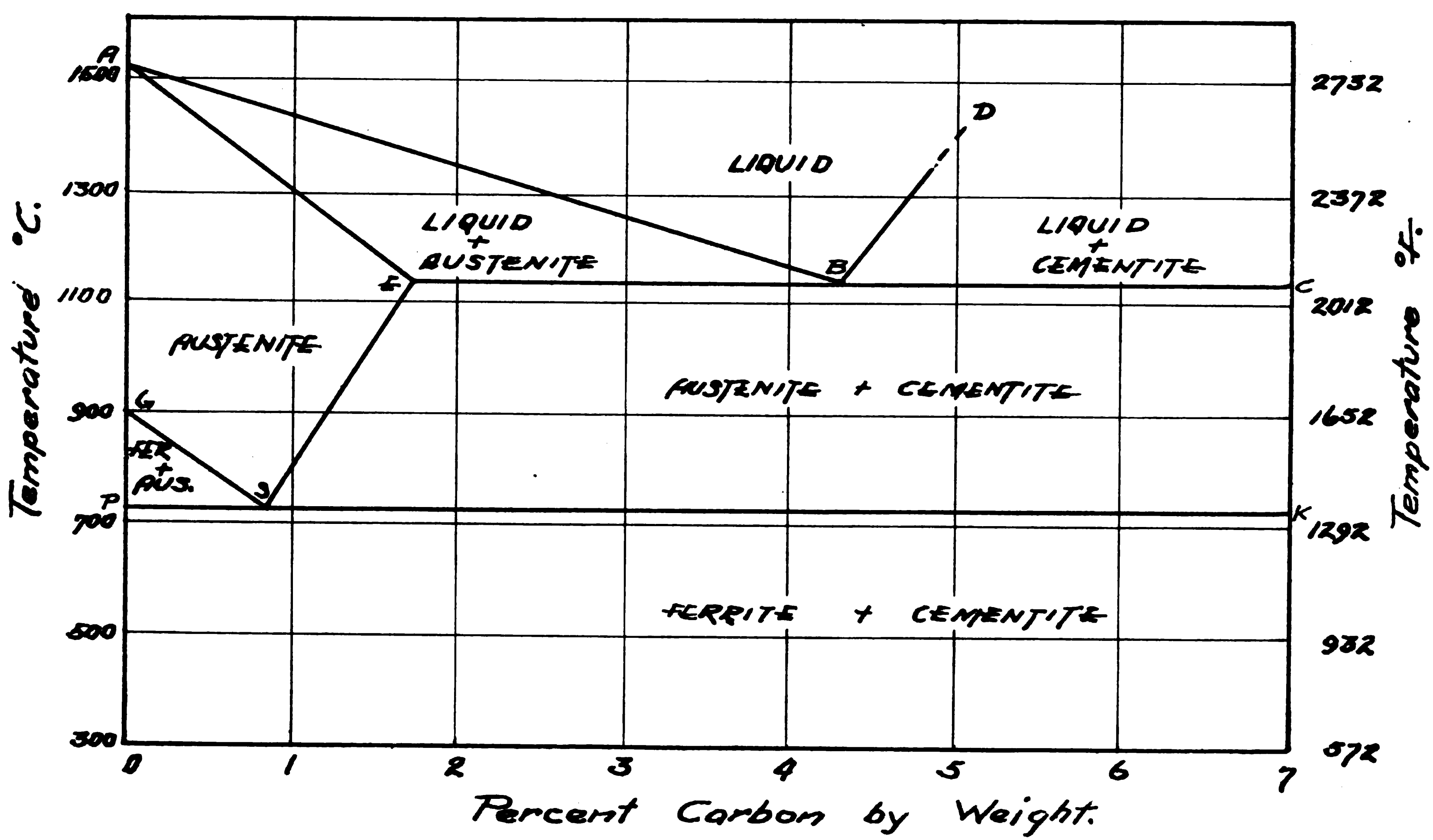
THESIS

INTRODUCTION

With the continued use of the microscope and exhaustive chemical and thermal analysis, much has been learned concerning the internal structure of pure metals and alloys. Many hundreds of observations of metals and alloys have been made by which equilibrium diagrams have been calculated and produced. Since this is a study of low-carbon steel, a brief review of the crystallizing process of a low-carbon steel might well be made. This is quite simply done by referring to the diagram (1) given on page 2. Beginning at the top of the diagram on the X ordinate, representing a low-carbon steel such as was used hereinafter, the crystallizing process is as follows: On reaching a temperature of 2732° F. (1500° C.) on AB, austenite or the solid solution of Fe_3C, cementie, in gamma iron begins to crystallize. At the line AE, this precipitation has been completed until the mass has completely solidified. As the cooling continues and the temperature of 1616° F. (880° C.) on GS is reached, ferrite or pure iron begins to precipitate. At a temperature of 1337° F. (725° C.) on PS the austenite has completely broken down, leaving a matrix of ferrite with crystal of eutectoid or pearlite, lamellae of ferrite and cementite.

It has been found, however, that the size of these final grains of ferrite and pearlite were by no means constant or the same. They seemed to vary because of many factors. In fabrication, the pouring temperature, size of ingot, rate of cooling, and nature and quantity of impurities were found to be potent factors. It was also found that, with



Temperature °C.
Temperature °F.
Percent Carbon by Weight.
A
B
C
D
E
G
S
P
K
1500
1300
1100
900
700
500
300
2732
2372
2012
1652
1292
932
572
0
1
2
3
4
5
6
7
LIQUID
LIQUID + AUSTENITE
LIQUID + CEMENTITE
AUSTENITE
FER. + AUS.
AUSTENITE + CEMENTITE
FERRITE + CEMENTITE

the advent of heat treatment, this gram-size varied with various treatment. Mathewson and Phillips (2) suggest, that for each temperature of anneal, a mean size of new grain is formed. It was found that continued annealing at this same temperature produced little or no increase in grain size. This condition has been called a state of "grain-size equilibrium" (3). This grain-size equilibrium or maximum grain size is best produced at a critical temperature which Howe has pleased to call a "germinative temperature" (4). This temperature, it has been found, is different for different metals and alloys and may vary with different degrees of cold-working in the same metal. This might more properly be stated as a short range of temperature, below and above which little growth is observed.

In general terms, grain growth seems to be favored by grain fineness, grain-size contrast, and by prior plastic deformation. However, the latter may be, actually, a combination of the first two causes. It is logical to believe that the higher the temperature, the greater mobility and, hence, the greater grain-growth -- provided the alloy does not materially suffer from the extreme heat. Obstructions, such as sonims, slag, oxides, etc., tend to oppose grain-growth. Decrease of grain-size contrast likewise hinders growth. The rate of growth seems to increase with the temperature.

While all of the above causes of grain-growth have been formulated upon observations, no specific results of the grain-growth of a carbon steel were available. In this report all factors except that of temperature were reduced, or eliminated, as far as possible. The report becomes a study of the effect of heat on the grain-growth of a carbon steel.

PROCEDURE

The steel used in this report was one which corresponded to an S.A.E. 1020 steel. It was fabricated in an American mill by the electric furnace process. The material was obtained as a bar 1" in diameter (1" cold rolled). A preliminary examination, chemical and microscopic, showed it to be almost entirely free of sonims and other occluded matter. The specimens used for heat-treatment were cross-sections of the bar about 3/8" thick. All specimens were cut from one bar — so that variations in composition, prior plaster deformation, and prior heat-treatment might be reduced to a minimum.

All heat-treatments were carried out in a Hoskins Furnace, Type F-H-104, 4" in diameter. Since this is standard equipment, no further description is necessary.

All temperature measurements were made by means of a Leeds & Northrup Hand-Compensated Potentiometer Indicator and a Hoskins M.A. thermocouple. The thermocouple was introduced through the cover of the furnace.

All photomicrographs were made with a Bausch & Lomb Metalloscope, Type ILSAA-9. Unless otherwise stated, all photomicrographs were of longitudinal sections and were taken at 100 diameters with the following settings on the metalloscope.

Objective....................................16 mm

Eyepiece.................................... 7.5 x

Bellows......................................33 cm.

Aperture.....................................3/8"

Stop...1 (large)

Exposure.................................90 sec.

Source of light.......................... 6 volt, 108 watt

Tungsten Band Mazda.

Developer................................M-Q

Time of developing.......................72 secs.

For those micrographs taken at 500x, the following were used:

Objective................................ 4 mm

Eyepiece................................. 7.5 x

Bellows..................................41.5 cm.

Aperture.................................1/4"

Stop.....................................1

Exposure.................................600 sec.

Time of developing.......................72 sec.

For those taken at 1000x, the following settings were used:

Objective................................1.9 (oil immersion)

Eyepiece.................................7.5

Bellows..................................39

Aperture.................................1/8"

Stop.....................................1

Exposure.................................1 hr.

Time of developing.......................80 secs.

All photographs were taken on plates manufactured by the Eastman Kodak Co., Eastman Commercial brand.

The specimens photographed for this report were etched with 2% solution of nitric acid in ethyl alcohol, the time varying from 4 - 20 seconds, depending on the condition of the sample.

Of a series of samples, heat-treatments at various temperatures, with varying durations were made. In the body of the experiment, the rate of cooling was the same, i. e., 100° F. (56° C.) per hour to 1000° F. (538° C.) and from thence within the furnace with its natural cooling period.

As a secondary experiment, the effect of the rate of cooling thru the critical temperature range was determined. One sample of the 1" bar, 12" long was drilled on its central axis to receive a thermocouple. This sample at each step was held at 1900° F. (1038° C.) for 1/2 hour, cooled to 1500° F. (816° C.) in about 5 minutes and then cooled from 1500° F. (816° C.) to 1200° F. (649° C.) in two hours and in one-half hour, in the furnace, in air, in oil, and in water. After each stage of treatment, a section of the piece was removed, examined, and photographed. As a final check on the effect of the 1900° F. (1038° C.) treatment, the piece was held at 1900° F. for 1/2 hour and cooled thru the critical range within the furnace.

In this experiment, grain-size determinations were made by a specially devised method. This consisted in measuring the average grain diameter of the pearlite in the photographs taken at 100x. This was taken only as a simple method of comparison in this report.

DISCUSSION

When this report was begun, the effect of temperature on the grain-size and growth of the steel was its ultimate purpose. However, before many determinations or heat-treatments had been made, another factor was introduced. It was noticed in the examination of the heat-treated specimens, that the pearlitic areas seemed to follow a definite orientation. This orientation seemed to be closely allied with the rolling grain, so-called, and seemed to produce a laminated or striated effects on the longitudinal sections of the specimens. As successive heats were made, this effect became more pronounced. Thus it was incorporated in the body of the report.

Since this is a study of a series of temperatures of varying duration on a single steel, it seems well to examine photomicrographs of specimens of the heats. As has been said, all photographs were taken of longitudinal sections of the specimens, which presented a section parallel to the direction of rolling.

Figure 1 is a photomicrograph of the steel, as received. The average grain diameter is about 60 units. A careful examination shows no evidence of a regular striation of the pearlite areas. While evidence of some occluded matter can be found, the steel, in general, is fairly clean.

Figure 2 is a section of the above specimen presented at 500x.

Figure 3 is another section of the same specimen taken at 1000x. Pearlite in its lamallar state is found.

Figure 4 is a micrograph of a specimen heated to 1700° F. (927° C.) and cooled to 1000° F. (538° C.) at the rate of 100° F. per hour. The average grain diameter at 100x is about 75 units. Here the first signs of the lammated or striated effects are found. The pearlite is arranged in definite rows the length of the photograph.

Figure 5. This specimen was held at 1700° F. for 1 hour and cooled at the usual rate of 100° F. per hour to 1000° F. The grain diameter has increased to about 90 units. The striated effect is still more evident.

Figure 6. This specimen was held at 1700° F. for 2 hours and cooled. The grain diameter has increased to 110 units. The striated effect is still evident.

Figure 7. This specimen was held at 1700° F. for 5 hours and cooled. The grain diamter has increased to 135 units. The striated effect is still evident, though not quite so pronounced.

Figure 8. This specimen was held at 1700° F. for 10 hours and cooled. The grain diamter is now about 170 units. The striated effect is still evident.

Figure 9. This specimen was held at 1700° F. for 36 hours. The diameter of the pearlitic area has increased to 225 units. The striated effect is evident, though not sharply pronounced. The decrease in total pearlitic area is very probably due to dicarburization.

Figure 10. This final specimen of this heat was held at 1700° F. for 72 hours and cooled as usual. While a great amount of decarburization has taken place, the grain diameter has increased to 240 units. The striated effect is still present.

Throughout this 1700° F. heat, it will be noticed that the grain diamter slowly increased from 75 to 240 units. The striated effect persisted. The duration of the heat seems to be responsible for the decarburization.

Figure 11. This is the first specimen of the next heat. It was heated to 1800° F. (983° C.) and cooled. The average grain diamter was found to be about 140 units. The abnormally large grain shown in the photograph seems to be composited of four grains. The striated effect again is evident, though less pronounced than in the 1700° F. heat.

Figure 12 shows a specimen held at 1800° F. for 1 hour and cooled as usual. The average grain diameter is about 185 units. The striated effect is still evident.

Figure 13 shows a specimen held for 2 hours at 1800° F. and cooled. The average diameter is but slightly larger, 200 units. The pearlitic areas seem to coalesce to form long "stringers."

Figure 14. This specimen was held at 1800° F. for 5 hours and cooled. The grain diameter has increased to 225 units. The striated effect is evident but slightly.

Figure 15 shows a specimen which has been held at 1800° F. for 10 hours. The grain diameter has increased to 250 units. There is now but little evidence of striation. Signs of occluded matter, very probably ferric oxides, are seen. A small amount of decarburization has taken place.

In this 1800° F. heat, the grain-size has increased from 140 to 250 units. Practically all evidence of striated orientation has been obliterated.

Figure 16 is the first section of the 1850° F. (1010° C.) heat. It was brought to the heat and cooled as usual, 100° F. per hour. The diameter of the pearlitic area is about 200 units, or the size of the grain of the specimen held at 1800° F. for 2 hours. Again, the striated orientation is but slightly evident.

Figure 17 shows a sample held at 1850° F. for 1 hour. The grain-size is but a little larger, 210 units. The striated effect is practically obliterated.

Figure 18 shows a specimen held for 2 hours at 1850° F. The grain diameter is now about 235 units. The striated effect can only be discerned with careful examination.

Figure 19. This specimen was held for 5 hours at 1850° F. The grain-size has reached a size of 250 units, almost a maximum. The pearlitic areas are well broken up.

Figure 20 shows the longest anneal at the 1850° F. heat, 10 hours, followed by the cooling. The grain-size has now reached its maximum of 265 units. Very little evidence of striation is perceptible.

At this point, a section of the sample shown in Figure 10 and a sample of the steel as received were heated together for 4 hours at 1850° F. and cooled 100° F. per hour. Figure 21 is the specimen which had been held for 72 hours at 1700° F., while Figure 22 is a sample of the original material. The size of the average pearlitic area is about the same, the diameter being about 230 units.

Figure 23 is the first member of the 1950° F. (1065° C.) heat, being brought to heat and cooled. The average grain-size is about 190 units. There is little evidence of striation.

Figure 24 is of a sample held at 1950° F. for 1 hour. The diameter of the average area is about 210 units.

Figure 25 shows a specimen which was held at 1950° F. for 2 hours. The grain diameter is about 220 units.

Figure 26 shows a specimen which was held at 1950° F. for 5 hours. A large amount of decarburization has taken place.

Figure 27 is of a specimen held at 1950° F. for 10 hours. A greater degree of decarburization has taken place.

It seems evident in the 1950° F. heat that the rate of grain growth is rapidly decreasing. Long duration of the heat only serves to decarburize the steel, thus reducing the amount of pearlite present. At this temperature all effect of previous heat treatment or mechanical working has been removed.

Figure 28 is the first specimen in the section of the experiment devoted to the effect of the rate of cooling through the critical range on the grain growths. The specimen shown in Figure 28 was held at 1900° F. for 30 minutes, cooled in the furnace to 1500° F. in about 5 minutes, then cooled from 1500° F. to 1200° F. in 127 minutes. The grain-size is roughly that of Figure 19 (q.v.).

Figure 29 shows a specimen held at 1900° F. for 30 minutes, cooled to 1500° F. in 5 minutes, and then to 1200° F. in 33 minutes. The grain-size is little changed.

Figure 30 shows a specimen held at 1900° F. 30 minutes, cooled to 1500° F. in 5 minutes, and then cooled to 1200° F. in 1.95 minutes in air. A roughly formed pearlite is found with evidence of the austenite grain boundaries.

Figure 31 shows a specimen held at 1900° F. for 30 minutes, cooled to 1500° F. in 5 minutes, and then quenched in oil to 1200° F. in 4 minutes.

Figure 32 shows Figure 31 at 500 x. Here it is shown that a little evidence of the austenitic boundaries remain. All ferrite and cementite is in very finely divided lamaller form.

Figure 33 shows a specimen held at 1900° F. for 30 minutes, cooled to 1500° F. in 5 minutes, and then quenched in water to 1200° F. in .12 minutes.

Figure 34 shows Figure 33 at 500 x. Somewhat more of the austenitic boundary remains due to the sudden change in mobility. The structure, however, is of a very fine nature. Proper heat-treatment, probably a quench and anneal, might bring the specimen back to its original grain-size, as received, though the structure might vary a bit from the original.

Figure 35 shows a specimen held at 1900° F. for 30 minutes, and cooled in the furnace after the last series of cooling treatment had been made. All evidence of these treatments are shown to be obliterated in this anneal.

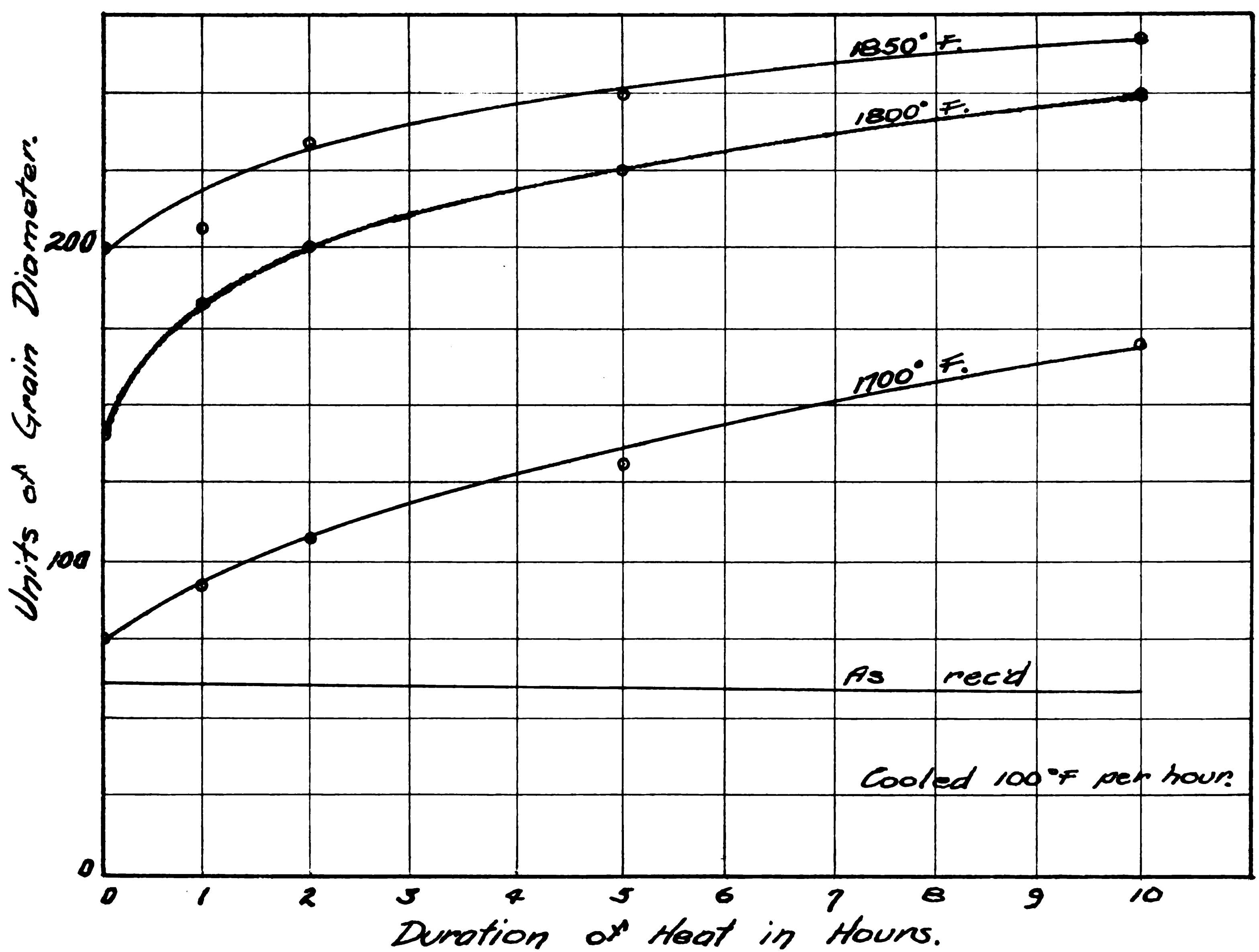
It will be remembered that, in this later section of this report, dealing with the effect of cooling, specimens were cut from one bar. Since a section was cut off after each treatment and examined, all specimens were subjected to the preceding treatments. Thus, the sample shown in Figure 35 was subject to all these later experiments.

CONCLUSION

In a consideration of the results brought out by these experiments, the effect of temperature on grain-growth is of primary importance. A portion of the data has been represented graphically on the following page. It seems, first, as was expected, that the rate of grain-growth increases with an increase in temperature. Secondly, there seems to be an equilibrium grain-size for each temperature. This equilibrium size seems to increase with an increase in temperature. At present, it seems a bit logical to believe there may be some maximum equilibrium size which all temperatures tend to approach, though perhaps never reach. At any temperature, the rate of grain-growth decreases with the duration of the heat.

If grain-size alone is concerned, an 1850° F. heat, with a duration of 2 - 5 hours, seems to furnish optimum conditions. Beyond this temperature, no great increase in size is effected and decarburization becomes serious. Because of this last fact, the 1950° F. heat was not represented on the graph.

The matter of striation effect is also of great importance. It was brought out in the 1700° F. heat, very probably by a release of internal stresses due to the increase of mobility at 1700° F. This effect was made less pronounced as the temperature was increased. Again, a 2 - 5 hour heat at 1850° F. seems to be effective in obliterating this striated effect. While no observations have been made to substantiate the belief,



1850° F.
1800° F.
1700° F.
As rec'd
Cooled 100°F per hour.
200
100
0
0
1
2
3
4
5
6
7
8
9
10
Units of Grain Diameter.
Duration of Heat in Hours.

it seems possible that sections or "stringers" of non-metallic inclusions might be broken up at 1850° F. as were the "stringers" of pearlite. Should this later belief prove true, it might be of very great importance from an industrial viewpoint.

While the section of the experiment devoted to the effect on the grain-size of the rate of cooling through the critical temperature was of secondary importance, several interesting points were noted. First, the grain-size is dependent upon the rate of cooling. As is to be expected, after a quench there is evidence of the retention of the austenite grain boundaries. The most important point seems to be the effect of the final 1850° F. heat. All traces of all rapid cools and quenches were entirely removed.

The data obtained from the experiment was entirely insufficient to determine the exact rate of cooling necessary for a small pearlite grain, similar to the original. It seems to be between a 2 minute to a 30 minute cool thru the critical range. This question of the effect of the cooling rate on grain-growth is obviously one for further study.

In concluding, we wish to acknowledge the aid of Mr. H. T. Publow, Professor of Metallurgy, upon whose advice and instruction, the success of these experiments largely depended.

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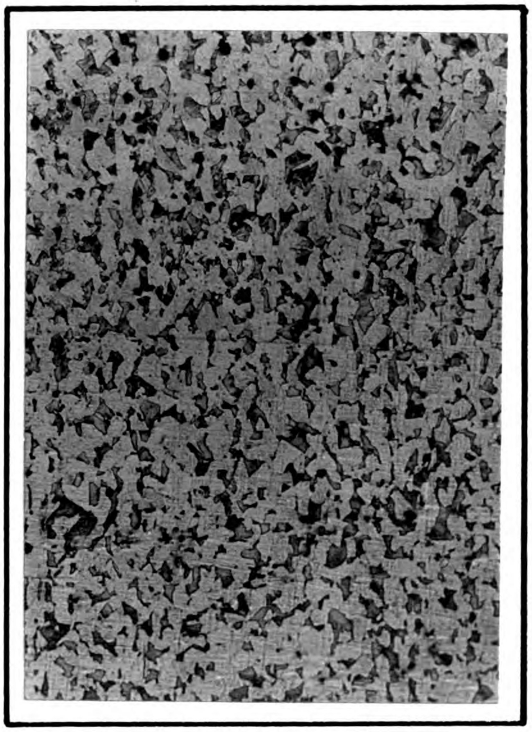

Fig.1. As rec'd 100x

Fig.2. As rec'd 500x

Fig.3. As rec'd 1000x

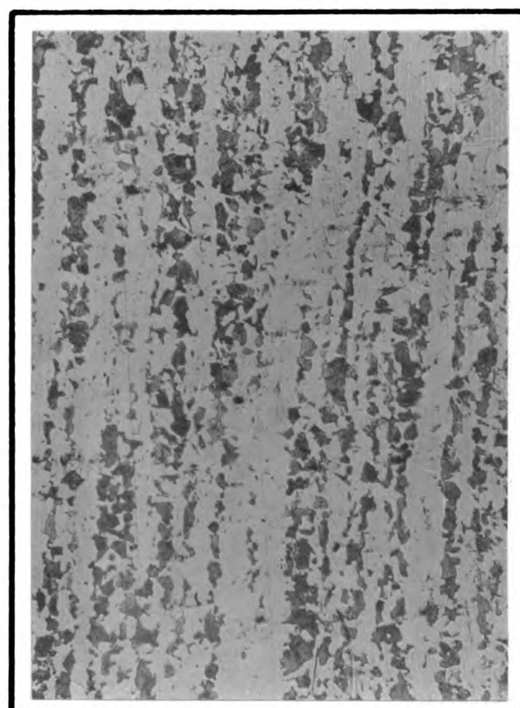

Fig.4. 1700°F To heat.

Fig.5. 1700°F. 1 hour.

Fig.6. 1700 F.° 2 hours.

Fig.7. 1700°F. 5 hours.

Fig.8. 1700°F. 10 hours.

Fig.5. 1700°F. 1 hour.

Fig.6. 1700 F.° 2 hours.

Fig.7. 1700°F. 5 hours.

Fig.8. 1700°F. 10 hours.

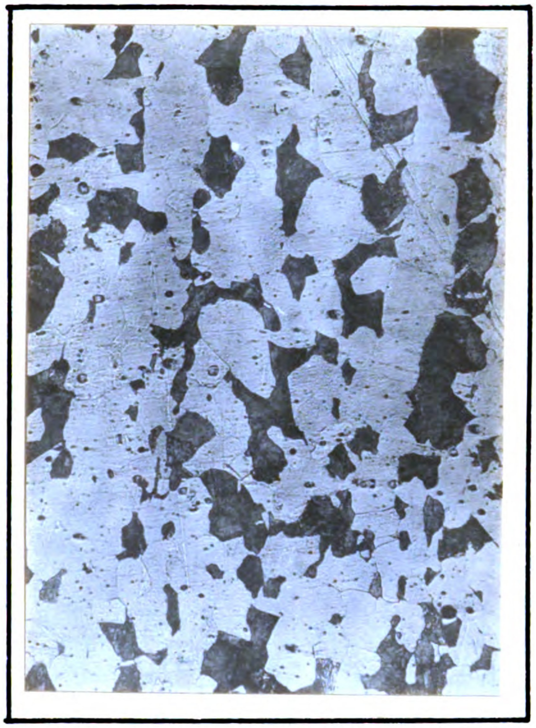

Fig.9. 1700°F. 36 hours.

Fig.10. 1700°F. 72 hours.

Fig.11. 1800°F. To heat.

Fig.12. 1800°F. 1 hour.

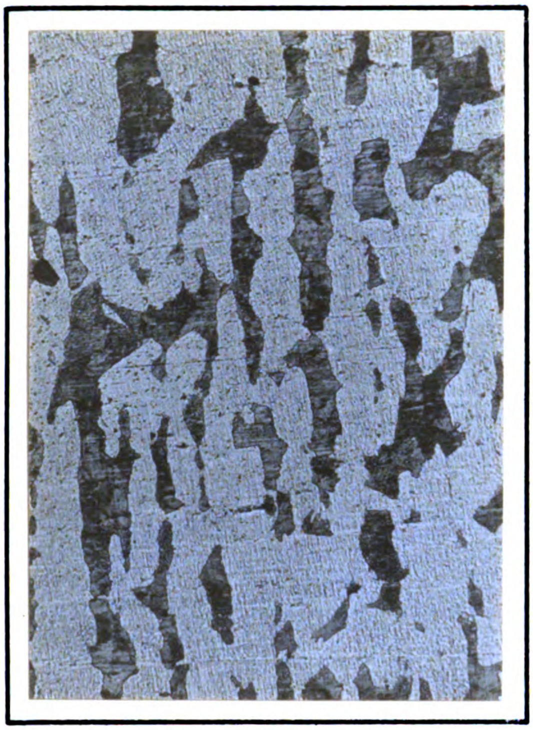

Fig.13. 1800°F. 2 hours.

Fig.14. 1800°F. 5 hours.

Fig.15. 1800°F. 10 hours.

Fig.16. 1850°F. To heat.

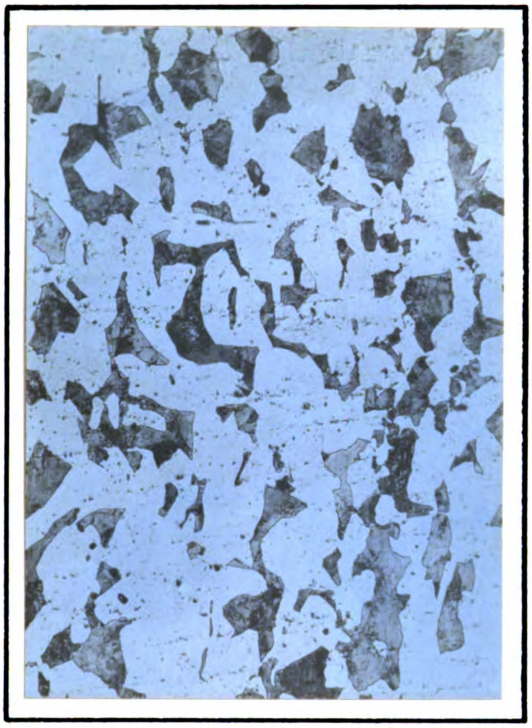

Fig.17. 1850°F. 1 hour.

Fig.18. 1850°F. 2 hours.

Fig.19. 1850°F. 5 hours.

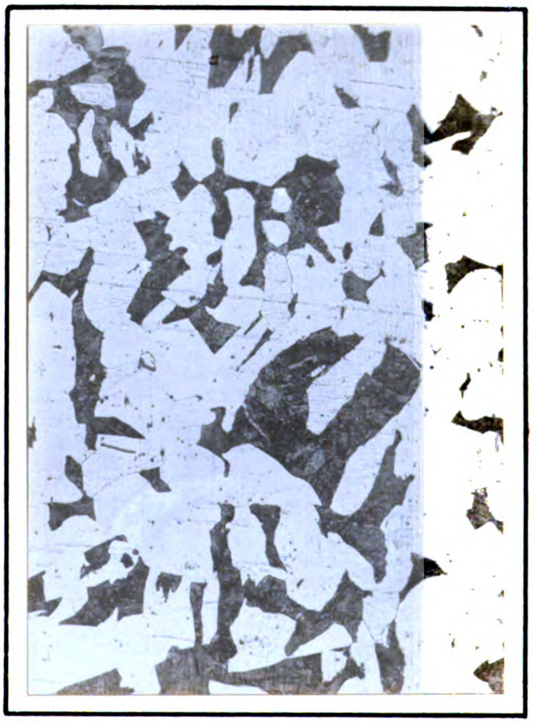

Fig.20. 1850°F. 10 hours.

Fig.21. 1700°F. 72 hours. Fig.22. As received.

Heated together - 1850°F. 4 hours.

Fig.23. 1950°F. To heat.

Fig.24. 1950°F. 1 hour.

Fig.25. 1950°F. 2 hours.

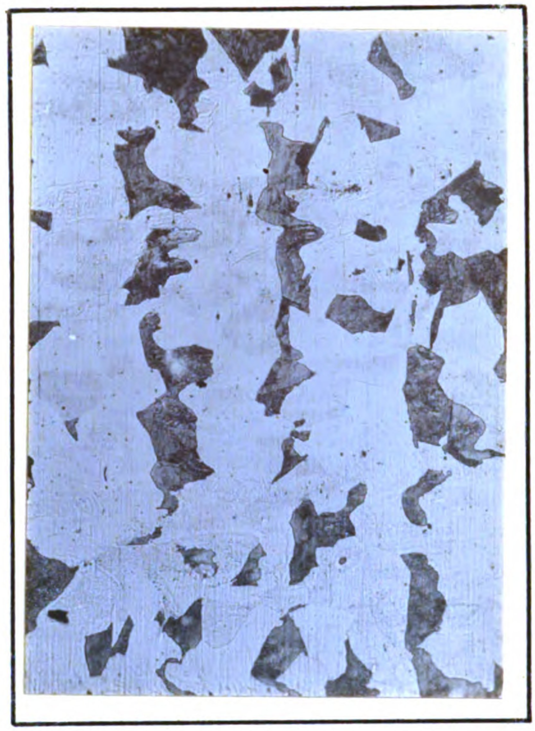

Fig.26. 1950°F. 5 hours.

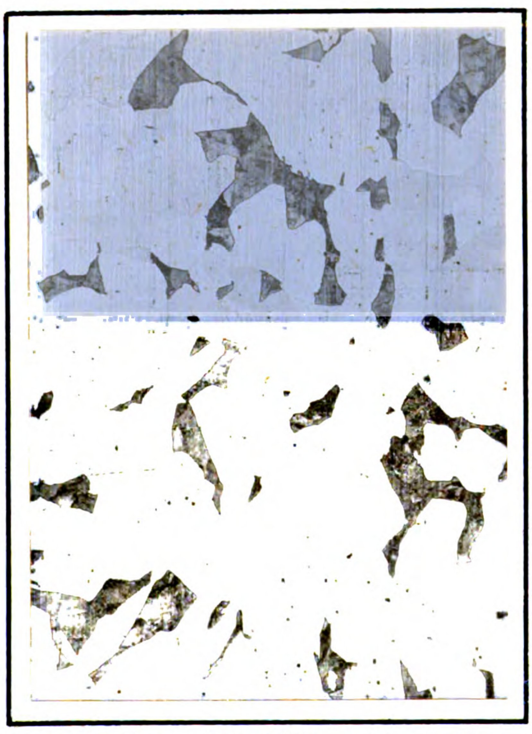

Fig.27. 1950°F. 10 hours.

Fig.28. Cooled 2 hours thru critical temperature.

Fig.29. Cooled 33 minutes thru critical temperature.

Fig.30. Cooled 1.9 minutes thru critical temperature.

Fig.31. Cooled .4 minute thru critical temperature.

Fig.32. Same 500x

Fig.33. Cooled .12 minutes thru critical temperature.

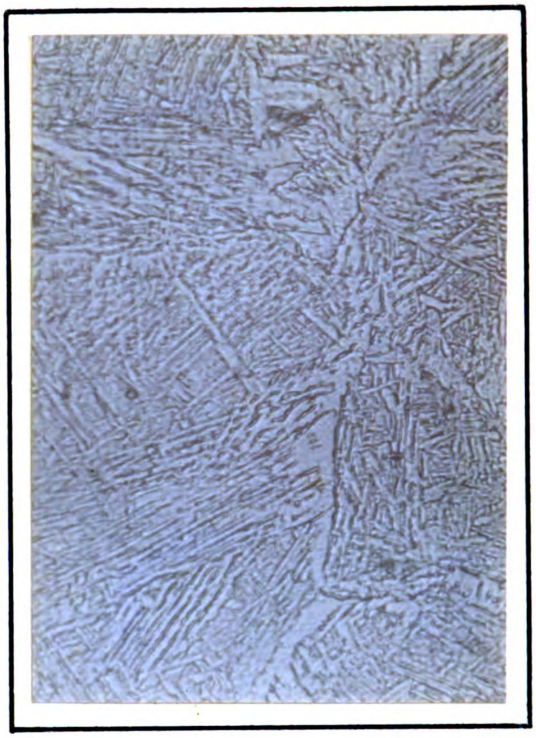

Fig.34. Same 500x.

Fig.35. Furnace cooled 1900°F.