# GRAIN GROWTH IN LOW CARBON STEEL WITHIN THE CRITICAL RANGE

Thesis for the Degree of M. E.

Henry E. Publow

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Chemical engineering

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THESIS

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THESIS

# Grain Growth in Low Carbon Steel Within the Critical Range.

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## INTRODUCTION

Iron in one form or another is man's most useful metal. With the coming of modern industry, the call for a more exact knowledge of its properties was felt. Thousands, yes millions of hours have been spent delving into its secrets, and yet, but very little is known of its properties. Iron in the shape of steel has received the largest share of attention, from both the research and the manufacturing end. This work covers so small a portion of the field of steel research, that it is with considerable misgiving that the author presents it, and it is only done with the hope that some one may find a clue, which will lead to a better understanding of the phenomena of the crystallization of iron and its alloys.

# CHAPTER I

## THEORY

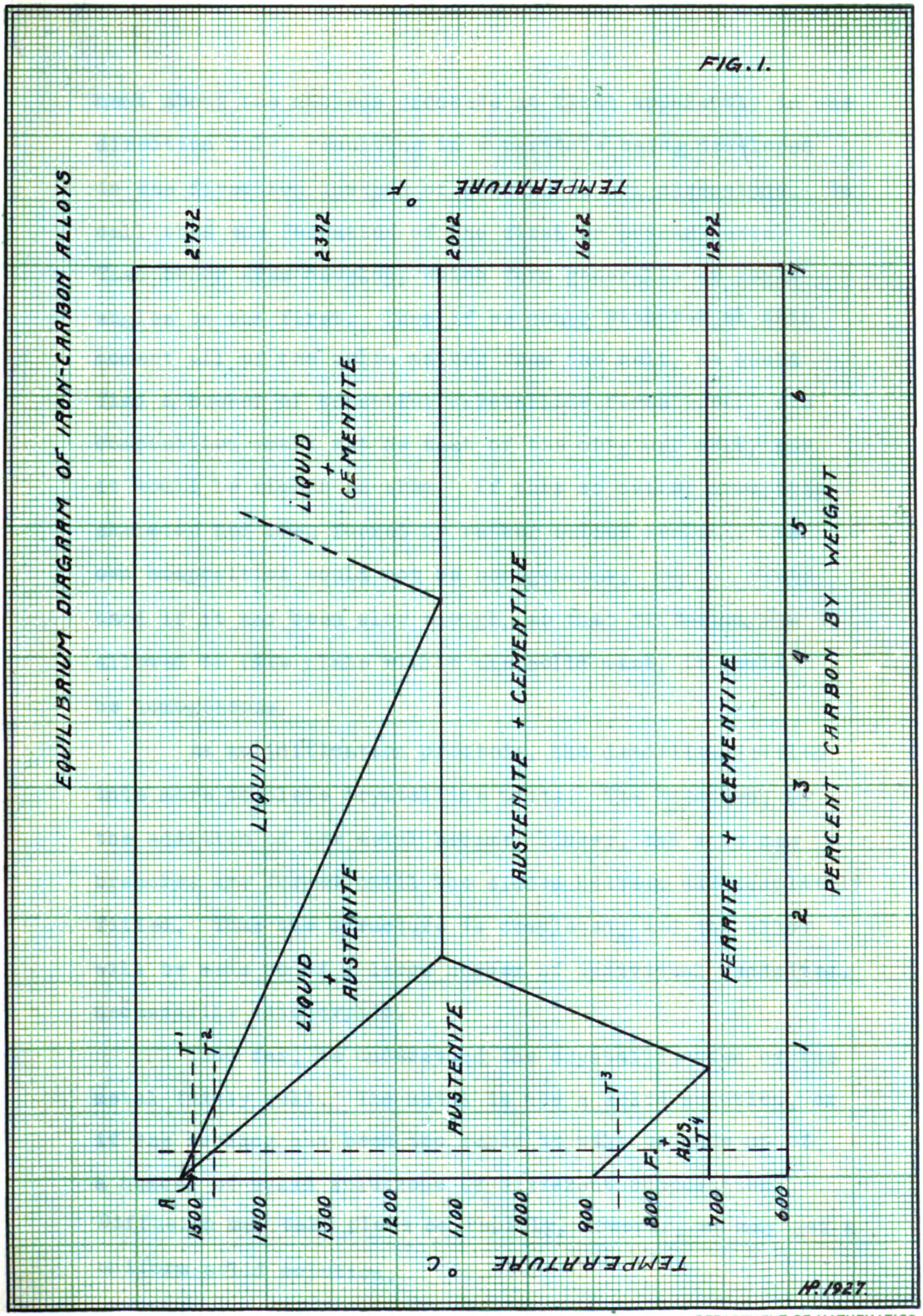
The equilibrium diagram of the alloy of carbon and iron has been the subject of extended study and research and today we believe that its construction is fairly well known. Let us follow the mechanism of crystallization of an alloy of iron and carbon, containing .2% carbon. (Figure 1).

When the temperature is reached where solidification of the liquid begins; namely, the point $T_1$, the alloy somewhere within the melt, produces a solid crystalline particle, at a point known as the "nucleus". This is a term by which we denote the place at which, for reasons not altogether clear to us, the first solid particle chooses to appear. The composition of this particle is not the same as the melt, but has a composition at the temperature $T_1$, of A.

A is richer in iron than is the original melt, so the remaining melt must be richer in carbon than theoriginal. If we hold the temperature $T_1$, we would in time reach a state of equilibrium. The mass would consist of an infinitely small amount of solid material of a composition A, and the balance, liquid of a composition B. As the temperature is lowered, according to the Phase Rule, the relative amount of solid and liquid change. The ultimate composition of the mass when solidification is complete, being that of the original melt. This state is reached when at the temperature $T_2$.

As the temperature is lowered from $T_1$ to $T_2$, develop-

FIG. 1.


EQUILIBRIUM DIAGRAM OF IRON-CARBON ALLOYS
TEMPERATURE °F
2732
2372
2012
1652
1292
LIQUID
LIQUID + CEMENTITE
AUSTENITE + CEMENTITE
LIQUID + AUSTENITE
AUSTENITE
FERRITE + CEMENTITE
PERCENT CARBON BY WEIGHT
1
2
3
4
5
6
A
T'
T²
T³
T⁴
F + AUS.
TEMPERATURE °C
1500
1400
1300
1200
1100
1000
900
800
700
600

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ment about the nucleus proceeds at first apparently in one direction only, resulting in a particle being developed in one direction instead of growing in all directions. We call this the formation of the first or primary axis. Then other axes develop, which form generally at right angles to the first. Thus if we could withdraw at this moment all of the solid matter from the melt, we would find a number of beautiful crystalline formations. Its composition would not be that of the original melt, but it would be richer in iron. If this so called dendritic skeleton had been formed by rather sudden cooling, its own composition would not be homogeneous. On the other hand if it had been slow enough, so that diffusion was allowed to take place within the solid mass, it would be homogeneous.

As solidification proceeds, the dendrites develop into more complete crystalline units. The branches, by the addition of new metal became thicker, and more new branches are formed until the spaces between are completely filled. Thus the structure is completed, and the alloy will be made up of a large number of individual dendrites, necessarily idiomorphic.

Thus far, we understand the crystallization of the metal fairly well. The metal is now a solid solution, or if the cooling has been too rapid it will be made up of a number of solid solutions of different composition. Diffusion may take place so rapidly, however, we may get a uniform solid solution.

As the temperature continues to fall we reach $T_3$. Here a recrystallization takes place. Iron is thrown out of solid solution and the remaining solid solution becomes richer in carbon, until $T_4$ is reached when all of the remaining solid solution (Austenite) precipitates into Iron and Iron Carbide.

When a specimen of steel is polished and etched, the dendritic formation is often revealed. The appearance of this structure is due to the mechanism of solidification, during which the progressive solidification with varying composition takes place as described above.

If we could by some selective method, treat a sample of steel in such a way so as to bring out only the boundaries between individual dendrites, we would obtain a structure of very large grains, in fact so large as to be visible without the use of a microscope.

When we examine a steel, etched in the usual manner, we find it composed of a number of small units or grains. We speak of steel having "fine" or "coarse" grain structure. These structures are always much smaller than the smallest dendrite. Now each dendrite at the time of its formation is a physical and crystallographic unit. At room temperature, each grain has itsown definite crystallographic arrangement, no matter how fine the grain may be. Thus the modern theory which states that upon cooling below the solidus line down to room temperature, the body of each dendrite divides into smaller units called grains. What causes granulation, and

where on the equilibrium diagram does it occur? It is generally accepted that it occurs in that part of the diagram between the solidus line and the upper critical point; that this transformation is not sudddsn but gradual; and that the number and size of the grains can be controlled by artificial means.

One cause of granulation may be due to the effect of internal stress. Krivobok found that a single crystal of an iron-silicon alloy could be converted into an agglomeration of much smaller grains, provided the crystal was strained and then heated to a certain temperature.

Granulation could not be produced, and the structure remained unchanged in a single crystal of this alloy when it was detached from the ingot, and heated by itself without straining. If two or more of these single crystals were heated without separating them, granulation unmistakably occurred at the common boundaries of the crystals, where strain could logically be expected.

Metal after solidification is generally in a strained condition, and strains existing in a solid metal at high temperature may be quite sufficient to cause recrystallization.

The presence of infinitely small particles of mechanically held inclusions is perhaps another cause of granulation. The presence of inclusions disturbes the equalized atomic forces in the metal. Such a condition would result in unequal distribution of atomic forces,

and the natural tendency would be for the metal to remedy the situation at the first opportunity.

It has been fairly well established that the grain size does not depend upon the size of the original dendrites. Some large dendrites show small grain structure , while others show large grain structure. Commercial steel is not a pure alloy of iron and carbon, but always contains non-metallic inclusions, which are visible under the microscope. We know that such inclusions are important, when the question of fatigue in metals is considered. Also that the distribution of these impurities in the steel is more important than their amount. We find that non-metallic inclusions are often found in patterns, and that such patterns can be developed only when there is opportunity for unobstructed dendritic growth. The inclusions have lower melting points and are forced to follow the dendritic growth. It has not been possible as yet to obliterate excessive segregation of non-metallic impurities, by means of heat treatment.

Another cause of segregation is due to the fact that certain elements, which do not form inclusions, are soluble in iron. Such elements as manganese and silicon can be diffused by heat treatment, while carbon and phosphorus, not only segregate, but their mutually repellent action results in pronounced local segregations of those two elements, if they occur in large amounts.

All of the above causes for segregation can be eliminated in modern practice, and if they are absent,

then we can assume that the steel will follow some definite law in crystallization and granulation. If we assume that granulation is completed in the solid solution range, before we reach the upper critical we have a complete crystallographic unit. Now then when we begin the secondary crystallization, or the separation of iron and carbide of iron, what is the mechanism of this recrystallization? This question, we will not attempt to answer, but will give a little experimental data bearing upon it.

## CHAPTER II

In previous work (Bul. 9, Eng. Exp. Station, M. S. C.) it was found that in a series of low carbon steels, which were obtained from many different sources, that at temperature of 1850°F and above, no matter what the previous treatment of the sample, it always reverted to the same crystal size. In other words there seemed to be an equilibrium temperature and it is possible that the austenite grains always come to the same size, under the same conditions of temperature and time. Then perhaps the size of the austenite grains, under conditions of slow cooling, govern the size of the resulting alpha iron crystals. The Pearlite since it crystallizes last would be found between the grain boundaries. On reheating, the Ferrite and Cementite in the Pearlite first go into solid solution, and as the temperature is raised, the Ferrite gradually absorbs this newly made solid solution.

If we heat up the sample to such a point that this diffusion has only taken place to a slight extent, then drop the temperature again, reprecipitating new iron again, this iron would tend to recrystalize in small grains, due partly to strain, and the Pearlite would be more scattered. If the heating and cooling were carried out in such a way that diffusion had completely taken place, but not enough time was allowed for reassimilation of grains, we ought to be able to produce a very fine grain structure, which still carried free Ferrite.

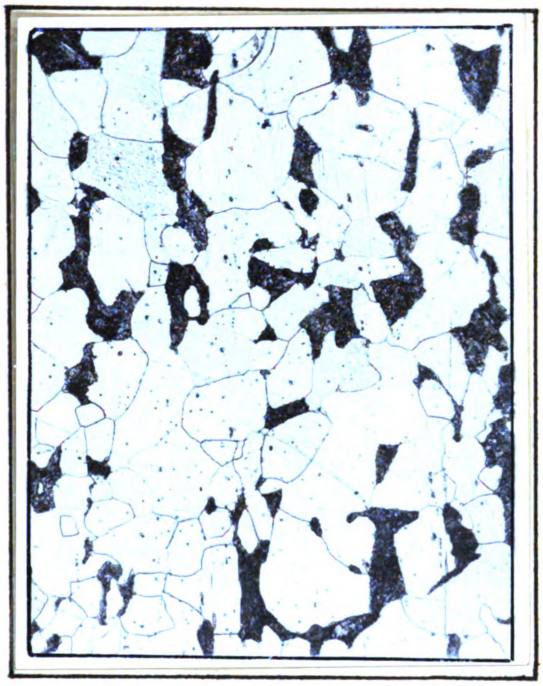


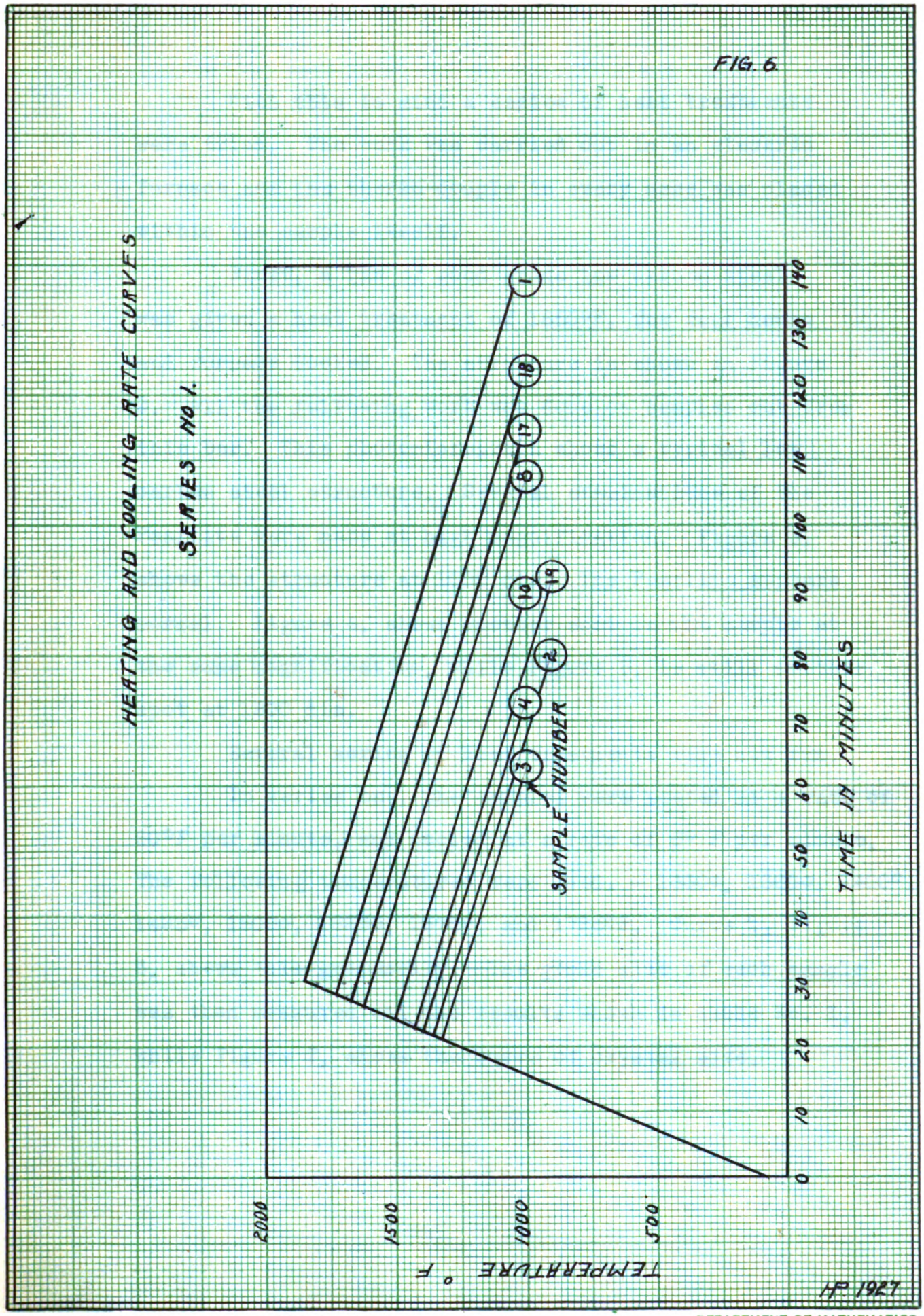
FIG. 2

FIG 3

FIG 4

FIG. 5

FIG. 6.


HEATING AND COOLING RATE CURVES
SERIES NO 1.
SAMPLE NUMBER
1
18
17
8
19
10
2
4
3
TIME IN MINUTES
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
TEMPERATURE °F
2000
1500
1000
500
HF 1927

With this in mind a series of heat treatments were tried. The work was carried out in an electric furnace and the thermo-couple in every case was placed within the sample itself.

First, a bar of good grade low carbon steel was given a heat treatment of 1850°F, for four hours and furnace cooled. The structure is shown in Figure 2. All the following work was done on samples cut from this bar. This photomicropraph shows a typical low carbon steel structure. Several examinations of the Pearlite were made at high magnification. Figure 3, shows the structure at 1000 dia. The Pearlite appears normal. Figure 4, shows another spot on the sample at the same magnification, while Figure 5, is still another spot at 1200 dia.

Several series of heat treatments were now run.

In series one, the samples were heated at a rate of 60°F per minute, and cooled at a rate of 6° F per minute. The following temperatures were used, - 1330°, 1360°, 1400°, 1425°, 1500°, 1625°, 1675°, and 1725°F. Curve 1, Figure 6, shows this graphically. The grain size of each sample was then measured and the results shown in table No. 1, while Figure 7, shows the curve of crystal size.

TABLE NO 1

Heating Rate 60°F per minute. Cooling rate 60F per minute

| SAMPLE NUMBER | CRYSTAL SIZE IN INCHES | RELATIVE CRYSTAL SIZE | DRAWING TEMP. | PREVIOUS TREATMENT |
|---|---|---|---|---|
| 1 | .0025 | 10.00 | 1850° | 1850° |
| 3 | .0024 | 9.60 | 1330° | 1850° |
| 2 | .0026 | 10.10 | 1360° | 1850° |
| 4 | .0025 | 10.00 | 1400° | 1850° |
| 19 | .0020 | 8.00 | 1425° | 1850° |
| 10 | .0014 | 5.80 | 1500° | 1850° |
| 8 | .0017 | 6.80 | 1625° | 1850° |
| 17 | .0019 | 7.60 | 1675° | 1850° |
| 18 | .0020 | 8.00 | 1725° | 1850° |

Examination of the foregoing data reveals that in the process of heating, and consequent diffusion of Cementite, and recooling and precipitation of Ferrite, there is a breaking down in the crystal size. In this series of treatments the smallest grain size was obtained about 1500°F. The microstructures are shown in the photographs, Figures 2, and 8 to 17 inclusive.

Figure 2, shows typical structure.

On heating to 1330oF, (Figure 8), we find that altho we have passed the lower critical, the size of the Ferrite grains remain unchanged, but the Pearlite is scattered over a larger area, showing that diffusion had started and then was checked.

In the next sample, shown in Figure 9, the same holds true, but the scattering of the Pearlite is more pronounced.

The next heat was carried to 1400°F, (Figure 10). Still the Ferrite grains remain the same size, but the Pearlite shows still greater diffusion.

At 1425°F there is evidence of the old Ferrite grains breaking down. The amount of Ferrite going into solid solution being sufficient to cause a recrystallization strain enough to break up the old crystal size. (Figure 11).

On raising the drawing temperature to 1500°F, we find a much finer structure, the temperature having been raised high enough, so that for these conditions of heating and cooling, we have reached the minimum grain size. Figure 12, shows the structure at 100 dia., while Figures 13 and 14 show the structure at 1000 dia. The Cementite appears more massive than regular Pearlite.

In Figure 15, is shown the structure when raised to 1625°F. It has the same structure as the 1500°F sample, with the exception that the Ferrite grains are now getting a chance to grow. Figure 15a shows the structure at 1000 dia.

On increasing the drawing temperature to 1675°F, the grain size shows still further increase as is shown in Figure 16.

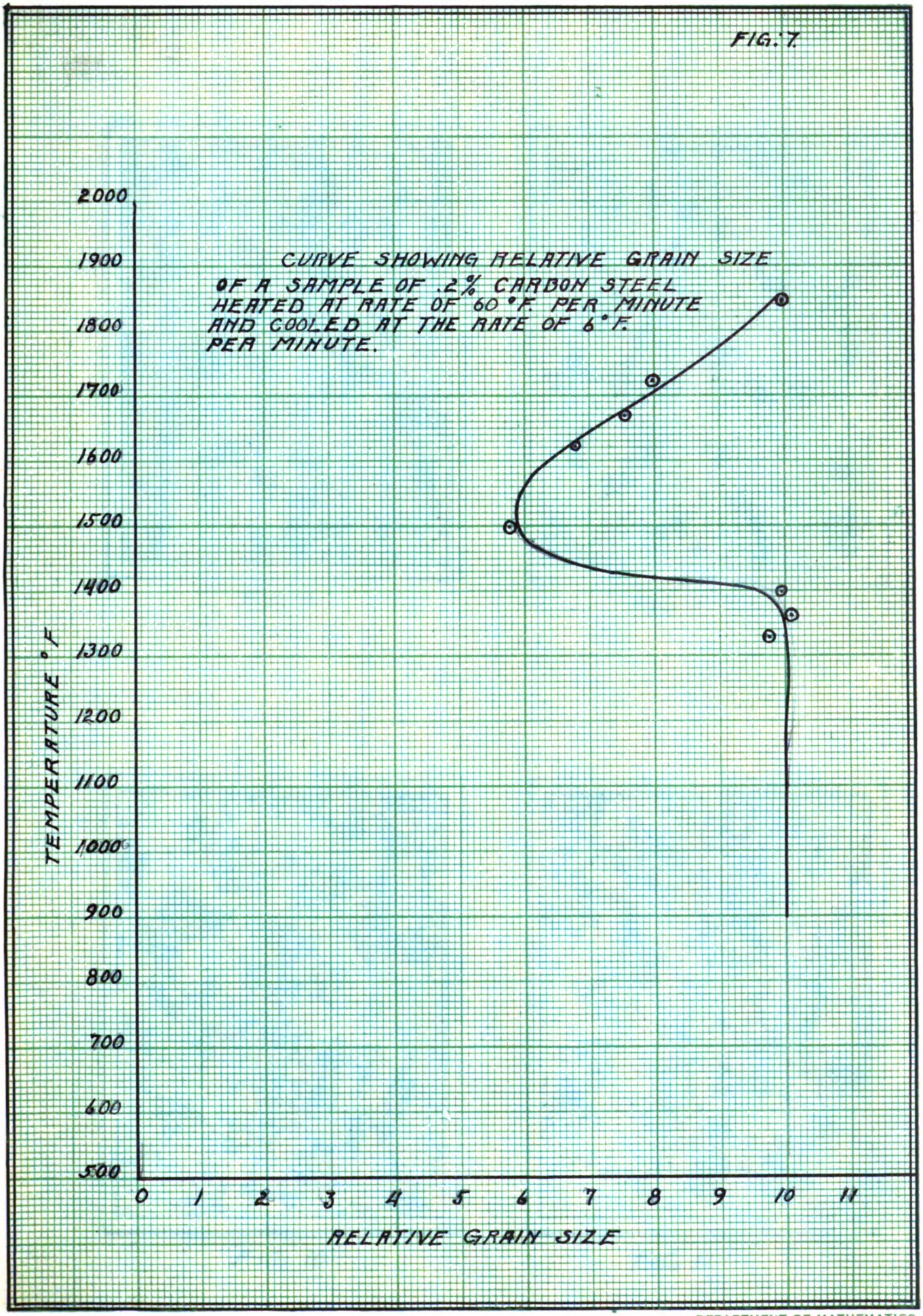
By the time a drawing temperature 1725°F was reached, a very pronounced increase in grain size was shown. (Figure 17).

At 1500oF in this series of experiments we obtained a minimum grain size.

In the next series of heats, the heating rate was the same as in the first series, and the samples were then quenched in brine. Table II gives the results of this test.

FIG. 7.

CURVE SHOWING RELATIVE GRAIN SIZE OF A SAMPLE OF .2% CARBON STEEL HEATED AT RATE OF 60°F. PER MINUTE AND COOLED AT THE RATE OF 6°F. PER MINUTE.



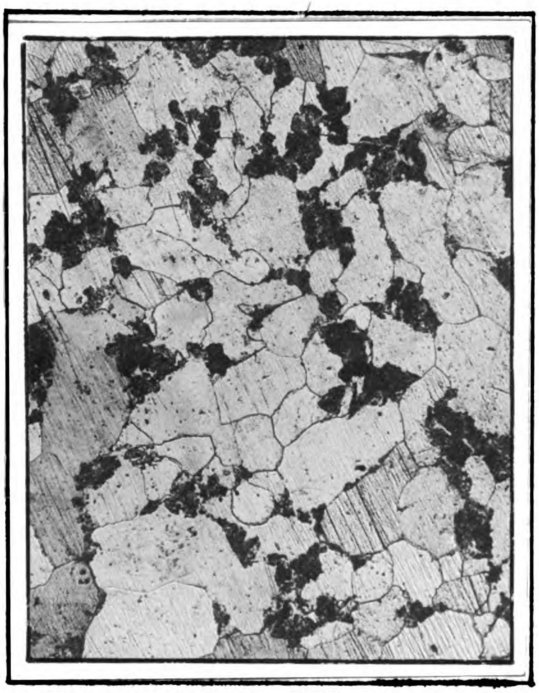


FIG. 8

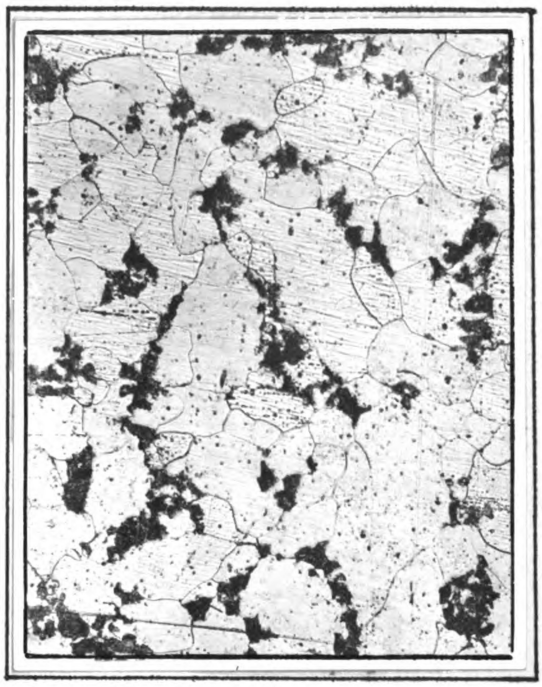


FIG 9

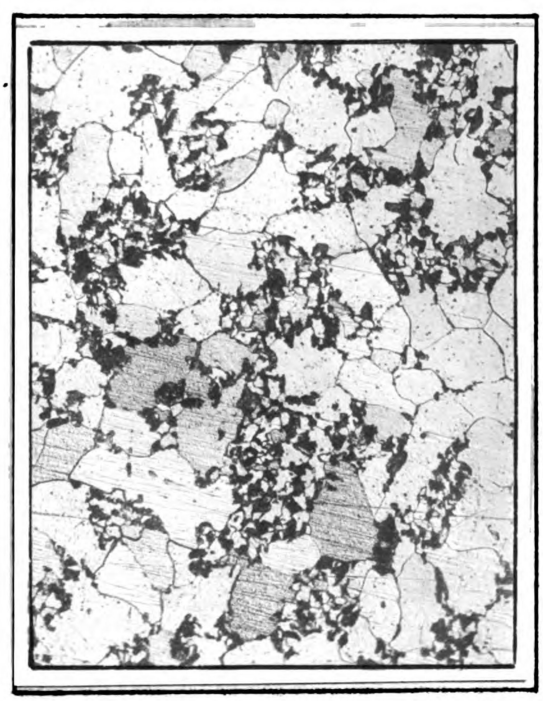


FIG. 10

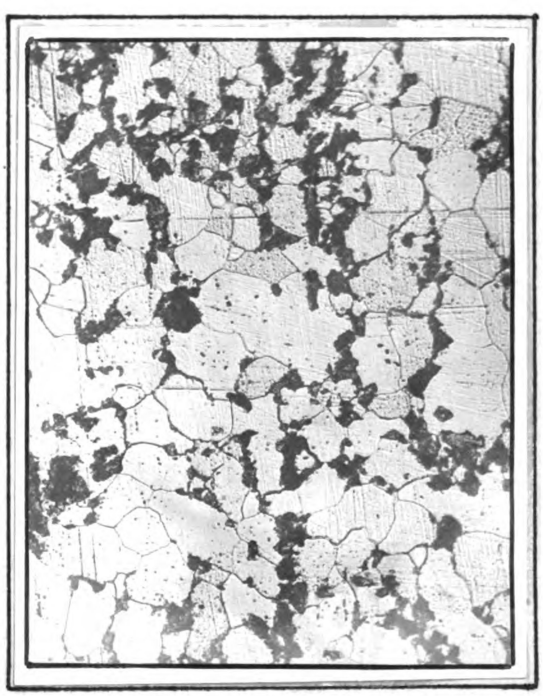


FIG 11

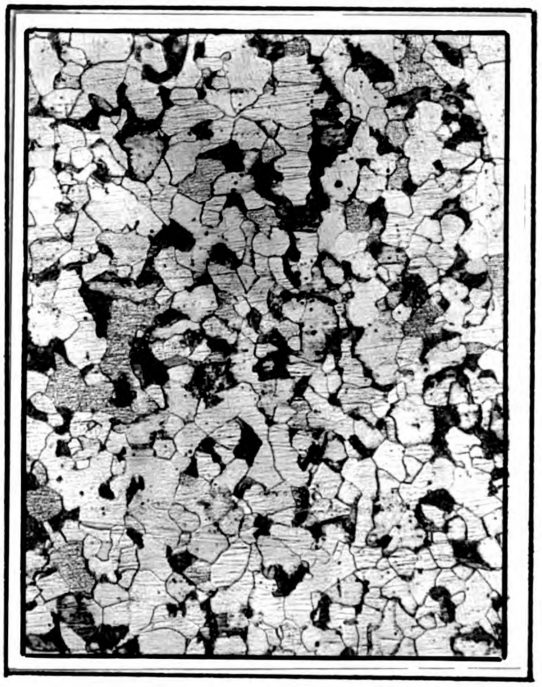


FIG. 12

FIG 13

FIG. 14

FIG. 15

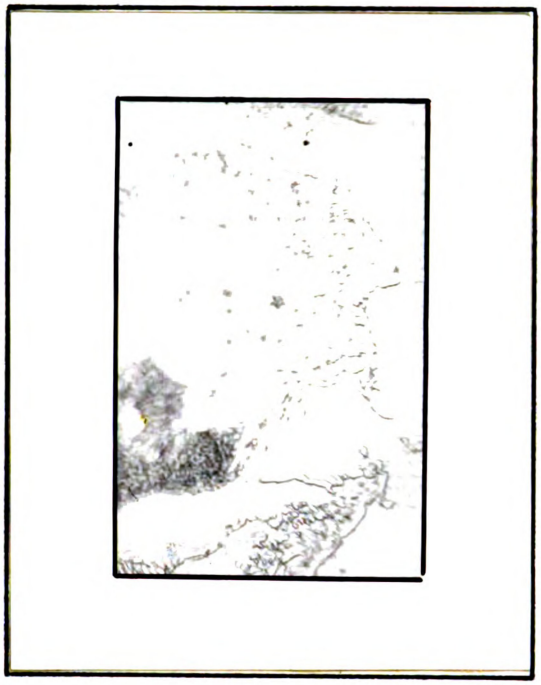


FIG. 15a

FIG 15b

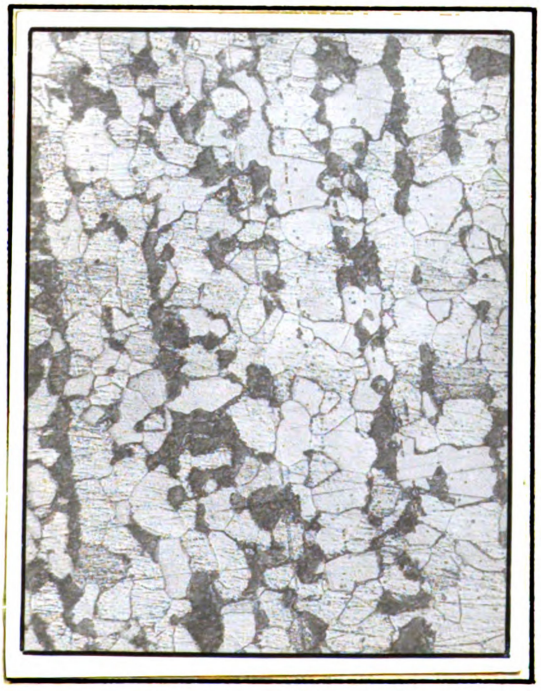


FIG. 16

FIG. 17

TABLE II

HEATING RATE 60°F PER MINUTE
COOLING RATE (QUENCHED IN BRINE)

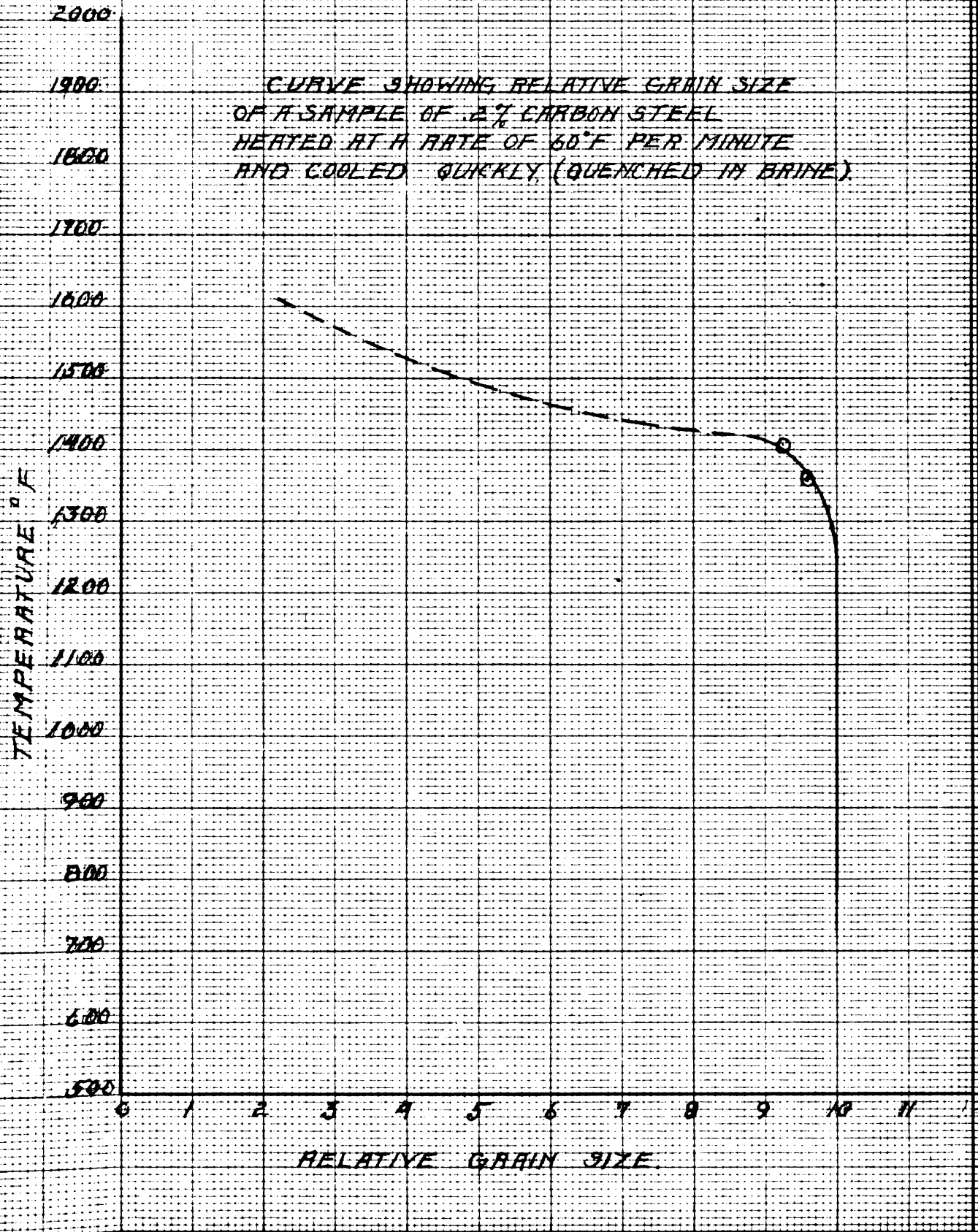
| SAMPLE No. | CRYSTAL SIZE | RELATIVE CRYSTAL SIZE | QUENCHING TEMPERATURE | PREVIOUS TREATMENT |
|---|---|---|---|---|
| 7 | .0023 | 9.6 | 1350 | 1850 draw |
| 11 | -- | -- | 1485 | 1850 " |
| 12 | -- | -- | 1625 | 1850 " |

The first specimen was quenched at 1360°F. Here we find no reduction in the grain size of the Ferrite, but the Pearlite was very fine. Figure 18, shows the typical structure. The second and third samples were quenched at 1485°F and 1625°F respectively (Figures 19, 20 and 21). Here we have complete breaking down of the Ferrite structure, altho at 1485°F, large areas could be found of material which apparently had not been absorbed.

Figure 22 shows the crystal size curve. In the next series, the heating rate was still maintained at 60°F per minute, but the material was cooled at a rate of 300°F per minute.. Table III shows the result of this series.

FIG. 22

CURVE SHOWING RELATIVE GRAIN SIZE
OF A SAMPLE OF .2% CARBON STEEL
HEATED AT A RATE OF 60°F PER MINUTE
AND COOLED QUICKLY (QUENCHED IN BRINE)



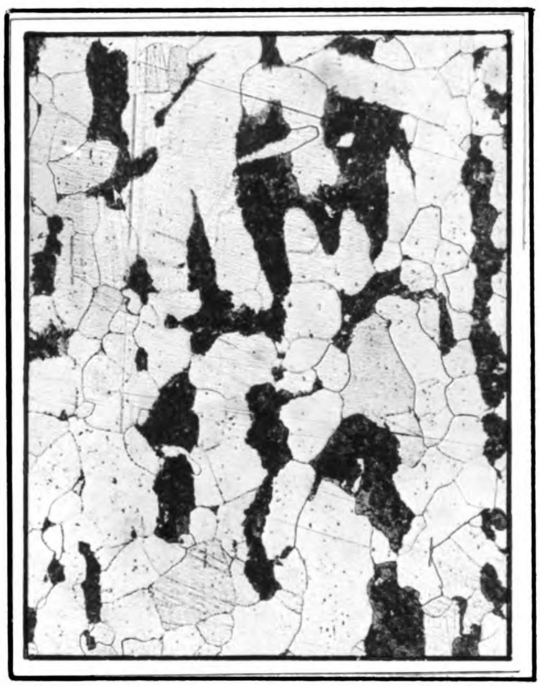


FIG 18

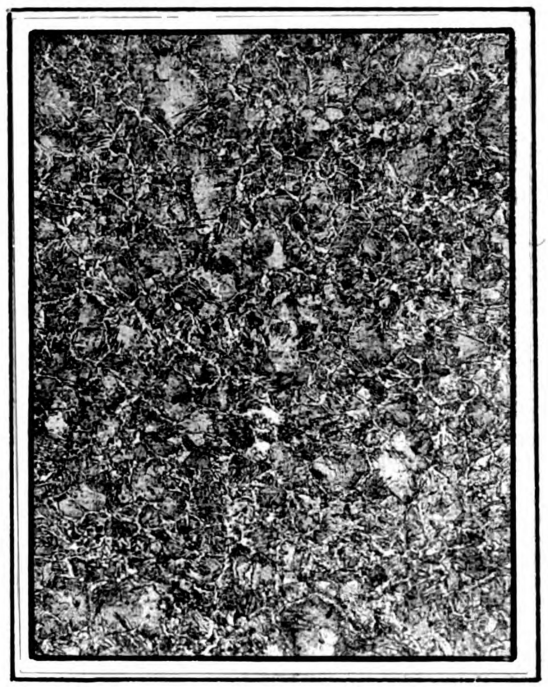


FIG. 19

FIG. 20

FIG. 21

TABLE III

HEATING RATE, 600 PER MINUTE
COOLING RATE 300°F PER MINUTE

| SAMPLE NO. | CRYSTAL SIZE IN INCHES | RELATIVE CRYSTAL SIZE | DRAWING TEMP. | PREVIOUS TREATMENT |
|---|---|---|---|---|
| 13 | .0026 | 10.6 | 1350 | 1850 draw |
| 15 | .0025 | 10.0 | 1400 | 1850 " |
| 9 | .0011 | 4.4 | 1625 | 1850 " |
| 16 | .0012 | 4.8 | 1700 | 1850 " |
| 14 | .0013 | 5.2 | 1850 | 1850 " |

Sample No. 13, Figure 23 shows no reduction of the Ferrite grains.

Sample No. 15, Figure 24 shows no reduction of the Ferrite grains, but the effect on the Pearlite is marked.

On raising the temperature to 1625°F, we find a marked reduction in the Ferrite grains, (Figures 25 and 26), but the distribution is irregular.

At 1700°F, the grain size is still getthg larger, Figure 36.

At 1850°F, Figure 27, the grain has increased considerably, while the Pearlite is more like Sorbite in structure.

Figure 28, gives the foregoing data as a curve. It will be seen that the curve has the same general shape as the first, but the minimum size is smaller.

Another series was now run, with the same heating rate, but the cooling rate was increased to 5000°F per minute. Table IV shows the result of this series.

FIG. 320.

CURVE SHOWING RELATIVE GRAIN SIZE OF A SAMPLE OF .2 % CARBON STEEL HEATED AT A RATE OF 60°F PER MINUTE AND COOLED AT A RATE OF 300° F PER MINUTE.

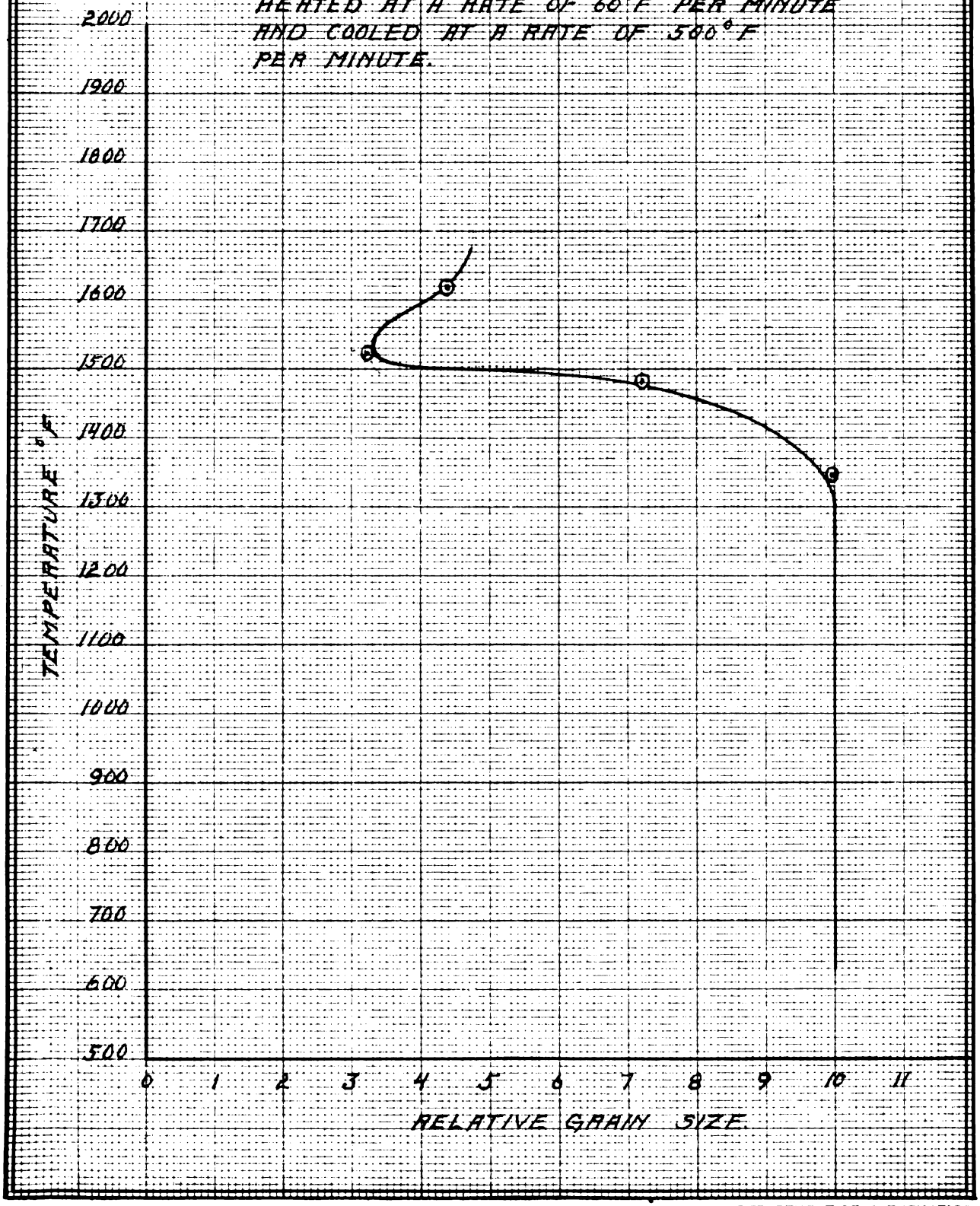


TABLE IV

| SAMPLE No | CRYSTAL SIZE | RELATIVE CRYSTAL SIZE | DRAWING TEMPERATURE | PREVIOUS TREATMENT |
|---|---|---|---|---|
| 21 | .0026 | 10.00 | 1350 | 1850 draw |
| 22 | .0018 | 7.20 | 1485 | 1850 " |
| 23 | .0008 | 3.20 | 1525 | 1850 " |
| 24 | .0011 | 4.40 | 1625 | 1850 " |

Here again we find the same results, but the grain is still finer at the temperature of 1525°F.

Figure 29, shows sample number 21, with no reduction in grain size. At 1485oF, sample number 22, Figure 30, the grain drops to about three-fourths, while at 1525oF we get the finest grain, (Figure 31), and at 1625°F it is again on the increase, (Figure 32). Figure 32A gives this data in the shape of a curve.

In the next series we decreased the rate of heating to 30°F per hour. Two samples were heated to 1525°F, - one was cooled at the rate of 500oF per minute, while the other was cooled at a rate of 30°F per hour. In the first sample, the grain size is larger than those with a heating rate of 100 per minute (Figure 33). The second sample showed no reduction in grain size but the Cementite was more massive. Figures 34, 34A and 34B are typical structures of this sample. Another sample was slowly heated to 1400oF and slowly cooled. It showed no reduction in the size of the Ferrite crystals as can be seen by examining Figure 35.

A heat was now attempted in which we tried to bring back the same grain size, as the sample had before it was given the

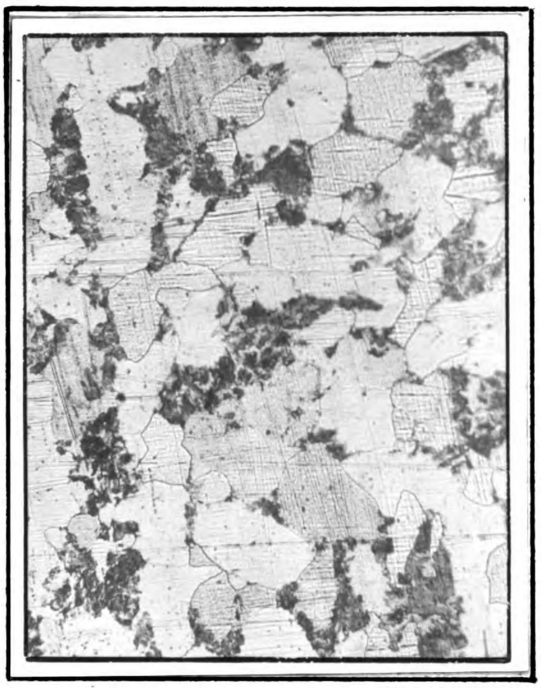


FIG. 23

FIG. 24

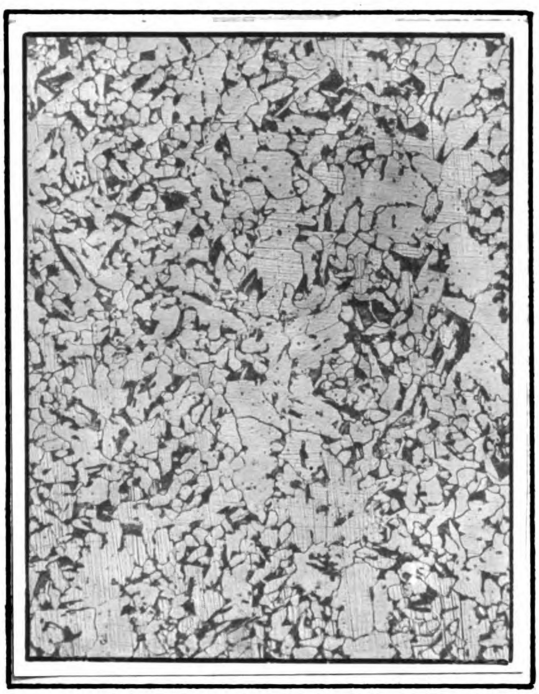


FIG. 25

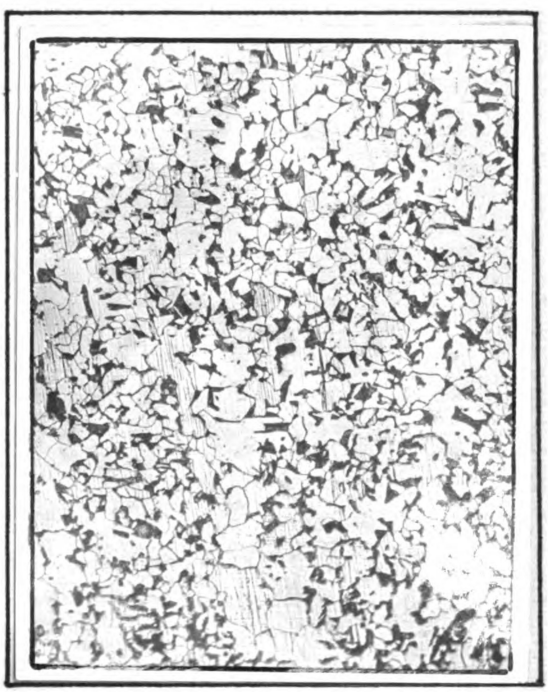
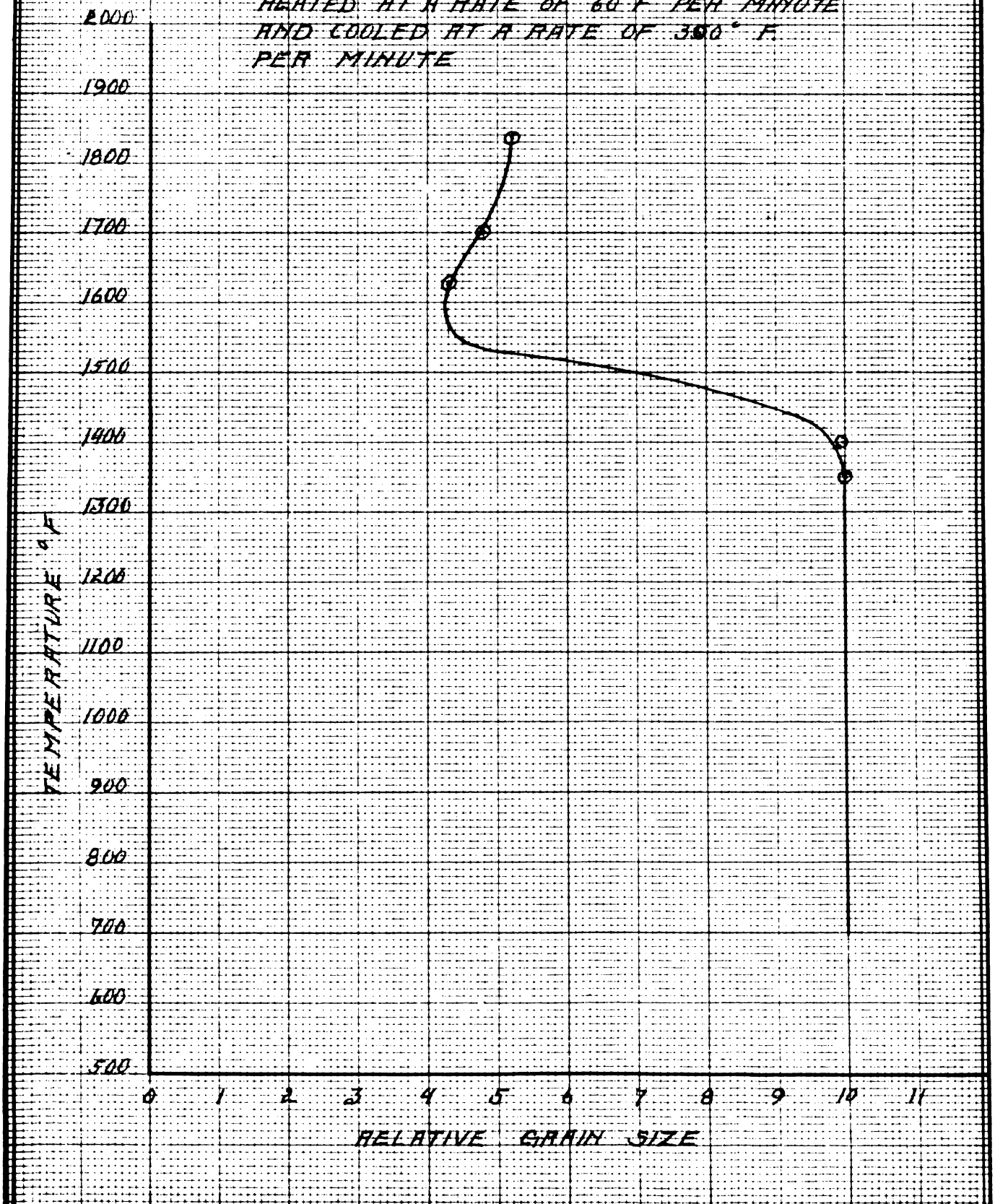


FIG 26

FIG. 28

CURVE SHOWING RELATIVE GRAIN SIZE OF A SAMPLE OF .2% CARBON STEEL HEATED AT A RATE OF 60° F PER MINUTE AND COOLED AT A RATE OF 300° F. PER MINUTE



1850°F draw. Figure 37 shows the result. This structure was obtained in one operation. The sample was heated to 1575°F at a rate of 60°F per minute and cooled at a rate of 350°F per minute.

Time was lacking to carry out a more complete series of tests, with varying heating and cooling rates, but from the work so far completed it can beseen that both factors govern the grain size.

FIG. 27

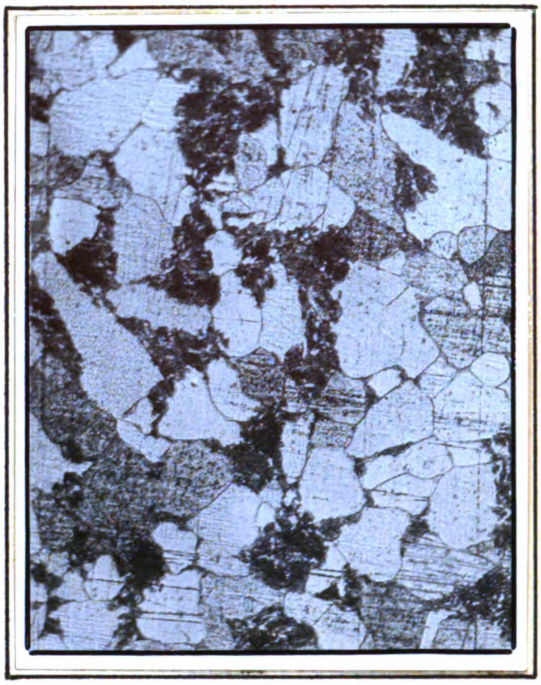


FIG 29

FIG 30

FIG. 31

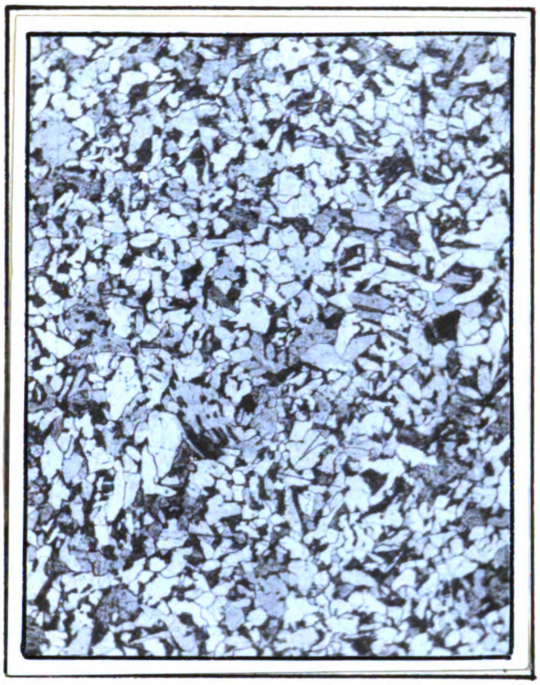


FIG 32

FIG 33

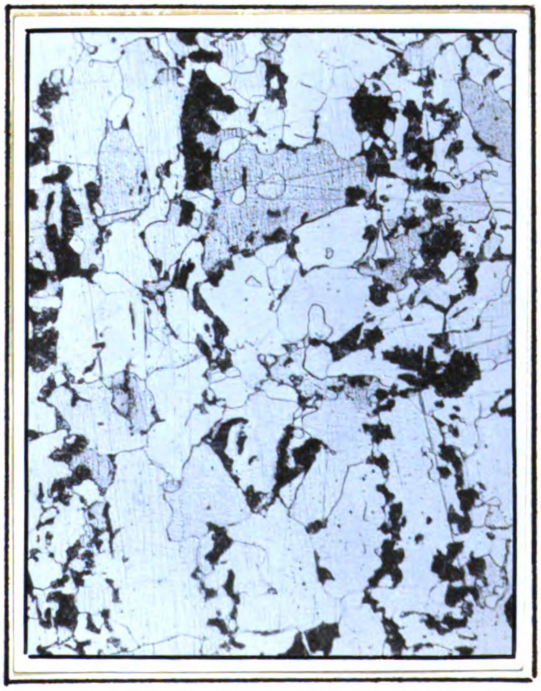


FIG. 34

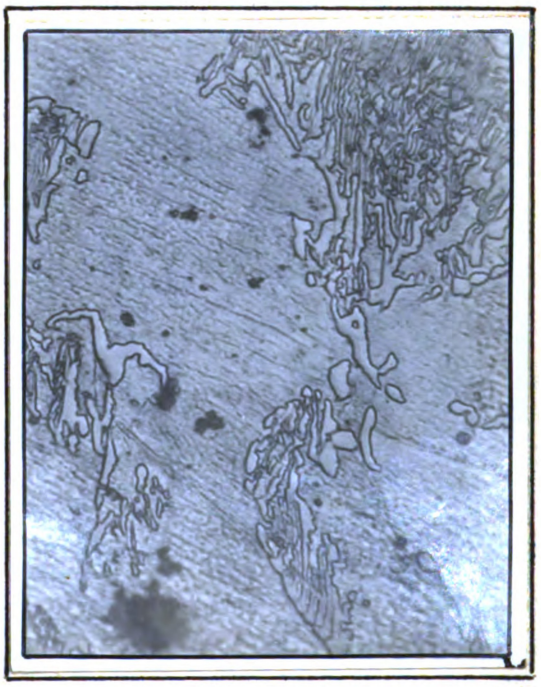


FIG. 34a. 1000 x.

FIG. 34b - 1000 x

FIG. 35

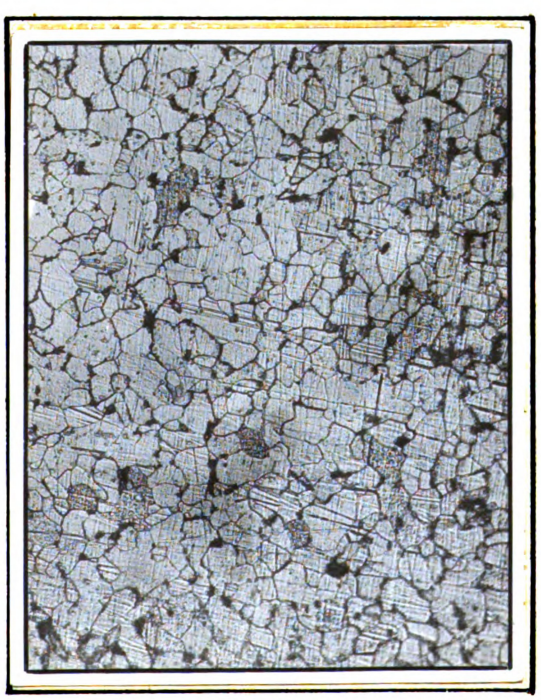


FIG. 36

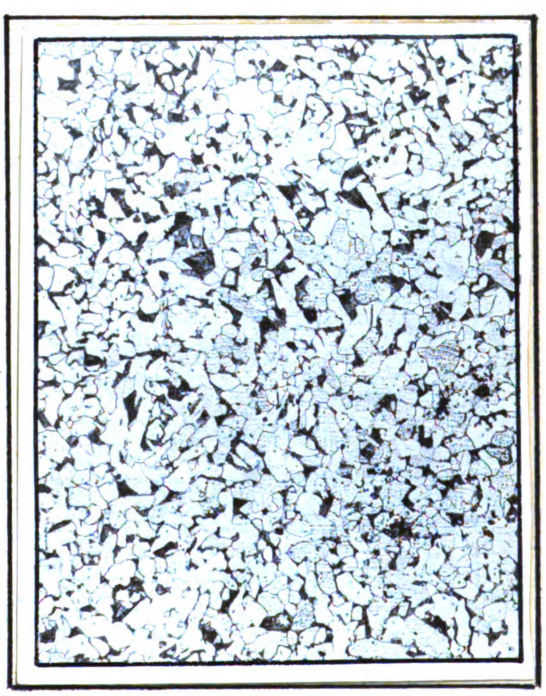


FIG. 37

# CHAPTER IV

## CONCLUSION

If a sample of low carbon steel be heated to a temperature of 1850°F or higher, and allowed to remain there a sufficient length of time so that the crystals of Austenite are in equilibrium, when this material is slowly cooled thru the recrystallization stage, the new Ferrite grains approximate in size the old Austenite grains. On reheating the Cementite must be reabsorbed by the Ferrite in order to produce a solid solution. As the lower critical is passed the Cementite first goes into solution in the Ferrite contained in the euctectoid mixture. Therefore, if the heating is checked as soon as this operation is complete, there will be no reduction in the free ferrite grains, but the quenched samples will show a finer Pearlite structure than the slowly cooled sample. On further heating, this solid solution, made from the euctectoid mixture gradually begins to enter the ferrite grains. At about 1400°F to 1450°F, if the sample be cooled, the Ferrite has not yet absorbed enough of the solid solution, unless the heating be ever so slow, that on cooling the precipitating Ferrite does not break down entirely the old Ferrite crystals. The new Pearlite which is formed very likely contains a greater percentage of Ferrite than true Pearlite should. When the temperature reached, lies between 1500°F and 1600°F, enough of the solid solution has entered the Ferrite so that on cooling the old grains are broken down. The quicker we cool from this temperature the smaller will be the grain. If the cooling becomes very sudden, we get the typical quenched structures, but it can be so regulated that we get a very fine normal

structure; that is, free Ferrite and Pearlite.

If the heating and the cooling are both rapid, more strain is thrown into the metal with a consequently finer grain. If both the heating and cooling are extremely slow, we do not get any reduction in grain, as the finely re-precipitated ferrite grains have time to grow together.

When a temperature of 1700oF is reached, the ferrite grains are saturated with solid solution, and on slow cooling the new ferrite grains have time to grow.

It is then possible by controlling both the heating and cooling rates to produce in low carbon steel any size of free ferrite grains with one heat treatment.

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