GRAIN GROWTH AND REFINEMENT IN HYPO-EUTECTOID STEELS

Thesis for the Degree of M. S.

Leo J. Waldron

1928

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IN

HYPO-EUTECTOID STEELS.

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Thesis

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THESIS

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I wish to acknowledge my appreciations to Prof.H.L.Publow,under whose direction this work was carried out.I consider my associations with him during the last two years as high spots in my future memory.

I also appreciate the aid and suggestions that have come from members of the faculty of M.S.C., and also from Gordon Stumpf of the metallurgical staff of the Reo Motor Car Co.,who has given material aid on some of the longer heat treatments as described.

INTRODUCTION

Grain growth and refinement in hypo-eutectoid steel -- phenomena and control of

This subject is not new to the metallurgist and metallographist of today. Ten to fifteen years have elapsed since Howe and Jeffries first published their results on grain growth in metals. And what of the progress in this phase of metallography since their original publications? If one were to ask the practical metallurgist of today for information relative to the control of grain size in metals, in about half of the cases the answers would be so obscure as to leave one in the dark. The other answers, being backed up by personal experiences, would enable one to work only on specific metals.

What is needed is a working theory on the subject. Facts should be in the hands of men in the industries whereby postive results can be obtained in their heat-treating processes.

Research in the laboratory is a means of evaluating the facts. In general the greater the understanding of a subject the more flexible its nature and application. Considera ble knowledge is already known regarding the fundamentals of grain phenomena in metals. The linking of the practical with the theoretical is far from what it should be. Aside from the direct application of metallurgical research to all heat-treating processes, the establishment of a physical fact into a definite law is in itself ample justification for immense effort. A law, once established, can not be ignored by those who carry the work along to practical ends.

LITERATURE REVIEW

A considerable amount of material on the subject of grain phenomena has appeared in previous publications. To record the findings of a comprehensive study of the literature would constitute a work in itself. Accordingly a brief resume' is here given.

The subject of grain phenomena (size, growth, etc.) has received a considerable amount of attention. The extensive studies of Jeffries (1) stand foremost in the list. This includes both theoretical considerations and practical relationships. Howe (2) and Gulliver (3) have also made noteworthy contributions to the subject. In these studies attention was directed principally to the conditions necessary for the occurence of grain growth and to means of measuring such growth. Rawdon and Jimeno-Gil (4) have made an extensive investigation regarding the relationship between grain size and mechanical properties.

Most of the reported work on grain size in metals has been based upon materials of relatively simple structure.

Grain growth in brasses has been covered by Bassett and Davis (5) and Mathewson (6). Hudson and Dean (7) have expressed the relationship between grain size and temp. by definite formula for the system Lead-Antimony.

McAdam (8) has studied the grain size of Armco iron, dealing with growth in strained materials and thus defining the conditions under which growth can take place.

Tammann has given (9) a satisfying explanation of the manner of solidification of metals from their melts, which is based upon direct quantitative results. His theoretical considerations cover the conditions for nuclei formation with resulting crystallization.

Carpenter and Edwards (10) have described an atomic conversion condition (Tammann) after remelting aluminum bronze resulting in an increase in grain size.

Ostwald (11) has studied phase relationships involved in crystallization of solids.

The history of crystallization has been studied by subjecting strain-hardened metal to various temperatures and cooling and then examining with a metallurgical microscope, after recrystallization has started but before it is finished.

(12) Chappel on iron.

(13) Mathewson and Phillip on brass.

(14) Carpenter and Elam on aluminum.

Percy, in his work on grain growth at M.S.C. (1926), has reported (a) The effect of temperature on grain size and (b) Effect on grain size of the rate of cooling thru the critical. (15)

H. L. Publow and L. J. Waldron have reported (16) their results on the effect of a four hour heat at 1850°F. carried out with various samples of S.A.E. 1020 steel. (17) The effect of temp. and cooling rates on low carbon steel have also been reported by them.

Yap Chu Phay (18) has worked up a colloidal hypothesis to explain certain phenomena which he has observed in very low carbon steel. Personally I can see nothing even novel in his theory as it can be easily explained by certain accepted facts concerning grain growth in general.

A study such as has been attempted in this work involves the old great question of grain size inheritance. Three investigators stand out in this work. (19) Jeffries, (20) Ruder, (21) Howe.

SCOPE OF WORK

Most of the work herein contained is a metallographic study of grain or crystal phenomena in low carbon steel (.20% C.) Some work is also reported on grain size of Armco Iron while a brief discussion of abnormal steels is given. In connection with this last subject principle reference is made to grain size study of 1700°F. or carburizing temperatures.

This thesis work was a continuation of my work under Prof. Publow as carried out as Eng. Exp. Station projects for some two or three years back.

GENERAL NOTATIONS

All photo-micrographs are at 100x, unless otherwise stated.

Preparation of samples for microscopic examination:

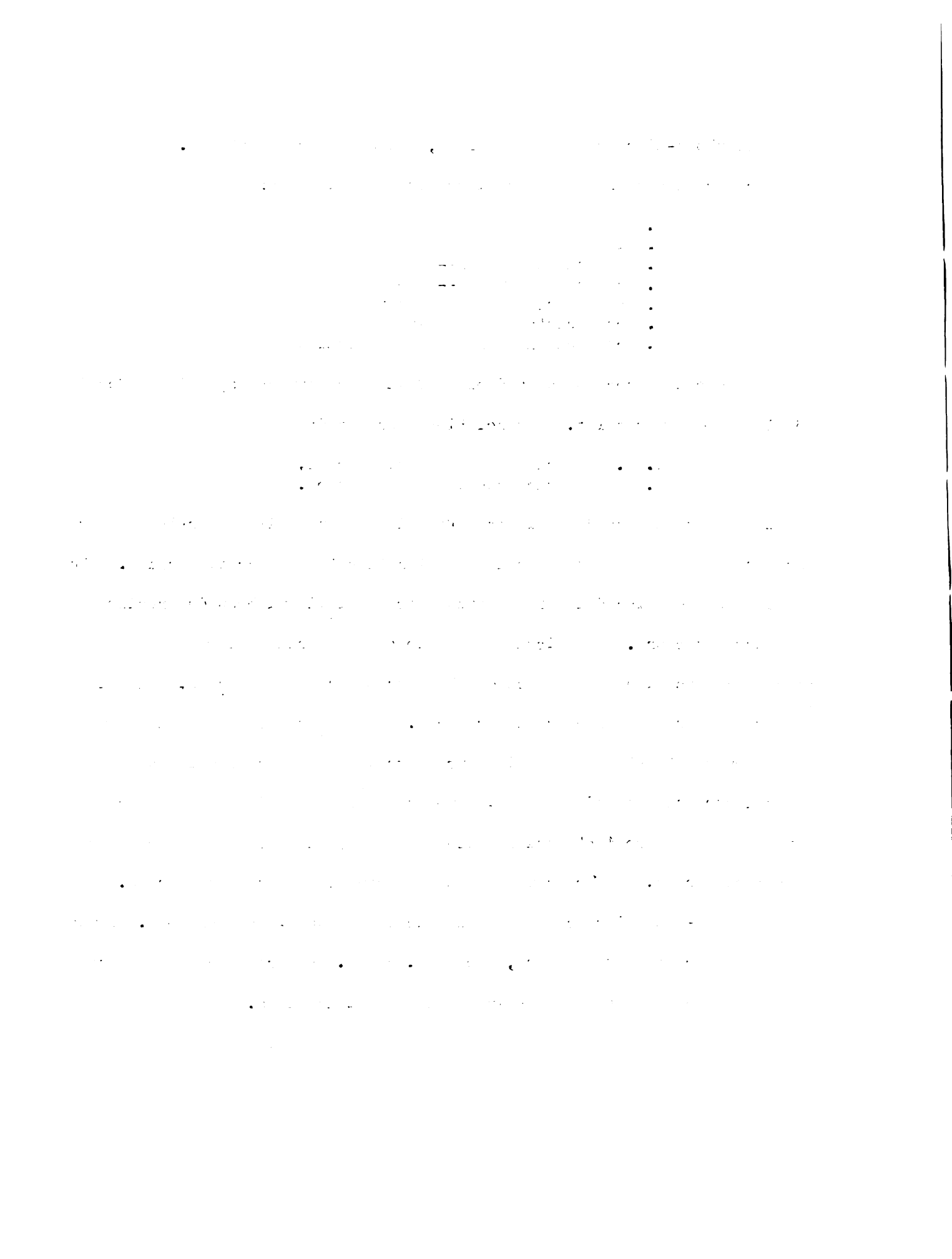
1. Saw
2. File
3. Wet grinding wheel--#180
4. Wet grinding wheel--#240
5. Broad cloth #320 Alundum
6. Broad cloth #600 Alundum
7. Broad cloth with levigated alumina

The etching process was a duplex affair (in most cases) and was found to give the best results. Two solutions were used:

1. .8% Nitric acid in ethyl alcohol.
2. 2% Picric acid in ethyl alcohol.

Specimens were immersed in #1 for a few minutes or until the grain boundaries were brought out without imparting a coloration to the ferrite grains. The sample was then placed in #2 and etched to the limit without destroying the microstructure. The picric etch colors the pearlite almost a dense black and also widens and blackens the ferrite grain boundaries. In this manner a contrasty structure is produced. The importance of etching can not be over estimated as many inaccurate grain counts are liable to be made on an improperly etched specimen. Two grains lying beside one another may have the same approximate orientation and with a light etch may show only as a whole grain. A more severe etch will reveal the true boundaries.

A Baush-Lomb microscope (metallurgical) was used. Eastman Comm. plates were used for most of the work, though W. and W. panchromatic plates were used with much success especially at high magnifications.



INTRODUCTION TO EXPERIMENTAL WORK

In general, it may be said that most of the work on grain size has been with the metal in a strained state, as either rolling, hardening, or plastically deformed. Another factor is thus involved, tending to make the whole a more complex problem.

Practically all of our work on this subject has been done in the unstrained or annealed state. If a better insight into the mechanism of crystal formation and growth is obtained for unstrained metals, then it may be possible to apply these laws and conditions to metals of a more complex nature.

Commercially, the metallurgist and heat-treater is concerned with the refinement of crystalline matter rather than its growth. Conditions of decreasing the crystal size should be worked up rather than those for growth.

EXPERIMENTAL WORK. I.

Effect of initial structure upon resulting structure from an anneal of seven and one-half hours at heat.

Procedure

For this experiment a sample of low carbon steel with the following chemical analysis was selected:

C. --- .18%
Mn.--- .44%
P. --- .014%
S. --- .032%

Three samples of this steel in different states (a, b, and c.) were heated in the same bomb to the annealing temp.

a. As received, 1850°4 hr., slow cooled (large grains)

b. As received, 1850°4 hr., slow cooled, reheated 1550°, ½ hr., quench in water. (fine grain)

c. As received, 1850°4 hr., slow cooled, reheated 1600°-1700° air cooled. (medium grain size.)

A Leeds-Northrup Hump furnace was used. Samples were from ¾" round stock cut in a ¼" disc and packed in a nichrome bomb with cast iron shavings.

In each case furnace was up to heat before bomb was placed in it and this heat maintained by automatic control for 7½ hrs. It was then allowed to cool with the bomb unmolested.

Samples were cut longitudinally and sections examined under the microscope. The number and size of the grains was determined by Jeffries method.

RESULTS

Temp.	Grains per square milimeter 1850° heat.	Air cool	Quench
Original	215	x	x
1375°	266	784	x
1400°	308	703	x
1450°	336	772	1100
1500°	334	455	780
1550°	353	-	435
1600°	306	325	401
1650°	292	303	383
1700°	276	293	311

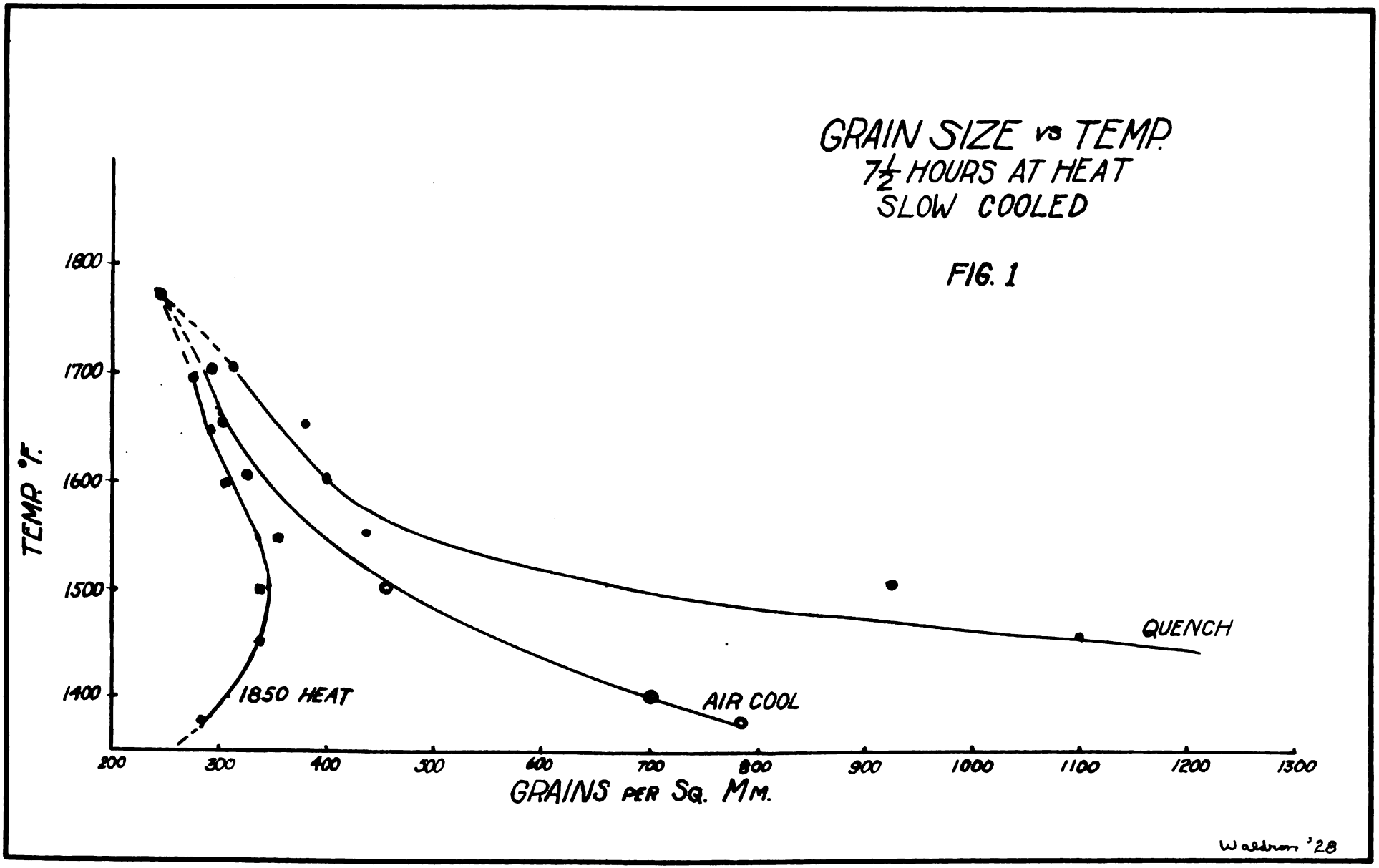
x = no satisfactory count
These results are shown graphically in curve fig. 1.

Discussion of results

The object of this experiment was two-fold. First; to learn something of the effect of initial structure on the products of annealing and second; to study grain reactions in a .20% C. steel at 1700°.

The first has definite dealings with the well known principle of "Inheritence or Non-Inheritence" as put forth by Howe, Jeffries and Ruder, and the experiment was carried out with the idea in mind of showing light upon these existing theories.

Howe believed that upon cooling, the alpha grains inherited size characteristics from the mother austenite. Jeffries believes that this inheritence is of a reversed nature, both on heating and cooling -- many austenite grains being formed from an alpha grain upon heating and many alpha grains in turn being formed from a single austenite grain on cooling. Ruder says that the resulting alpha grains, after heating to above A_3, in both size and characteristics have nothing to do whatsoever with their original size or that of the austenite. In other words, holding at these


GRAIN SIZE vs TEMP.
7½ HOURS AT HEAT
SLOW COOLED
FIG. 1
TEMP °F.
1800
1700
1600
1500
1400
1850 HEAT
AIR COOL
QUENCH
200
300
400
500
600
700
800
900
1000
1100
1200
1300
GRAINS PER SQ. MM.
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GRAIN SIZE & TEMP.
AT HEAT TA SRUOH 24
STOW COOLED
FIG. 1
GRAINS PER SQ. M.M.
TEMP °F
AIR COOL
BUENCH
TAEH 0281

temps. produces a clean slate as far as previous heat-treatment or mechanical work are concerned. This experiment shows something different.

Following are the general conclusions:

1. There is an equilibrium grain size for certain temperatures, mainly in this case at 1750°-1800° and above, when annealing takes place for 7½ hrs. This temp. is the point where the growth force is reduced so as to no longer cause grain growth under the existing conditions of temp. In some of our other work (Bull. #9) this point has been determined at 1850° for at least 4 hrs., though a majority of steels in this class will come to equilibrium in a much shorter time.

For temps. under the above (1700°-1800°) in general, there exists no apparent equilibrium grain size, inasmuch as initial conditions are of prime importance.

For original large grained steel the reverse of the above takes place.

2. Up until the equilibrium grain size results, the size of the original alpha grains is a decided factor in governing the size of the austenite.

3. In turn the austenite grain size governs the alpha grain size on cooling.

4. The smaller the original alpha grains on heating the smaller the resulting grains on cooling up until annealing is of such heat and duration of time so as to produce an equilibrium grain size.

5. The relationship of alpha to austenite is probably not a direct quantitative measure, one alpha grain does not necessarily yield one grain of austenite, since the velocity with which small grains grow before equilibrium size is reached is far greater in a small grained sample than in a large one.

6. The relationship of austenite to alpha is in a numerical ratio for this particular steel and perhaps holds for all hypo-eutectoid steels.

This has been determined by quenching .20% C. steels from different temps. above A_3, measuring their grain size, and comparing it with a corresponding anneal from the same temps. and under the same conditions. In other words, the size-temp. curves of the two steels is much the same above A_3.

7. From these results, it seems logical to assume (see Hardness discussion of Annealed Steels) that all steel represents to a certain degree a condition of strain -- one crystal being hindered (and thus setting up strain) in maintaining perfect orientations by that of neighboring crystals. This accounts for the fact that small grains grow with greater velocity than larger ones.

HARDNESS DETERMINATIONS

Samples from some of the preceding experiments were tested on a Rockwell Hardness Tester. The indentations were made on the polished surface as used for microscopic examinations. Five or six successive readings in a straight line thru the central section of the piece were taken. The average of each piece is given.

Experiment One

Hardness Determinations of $7\frac{1}{2}$ hr. anneal samples.

Rockwell "B" scale--1/16" steel ball, 100 kg. weight.

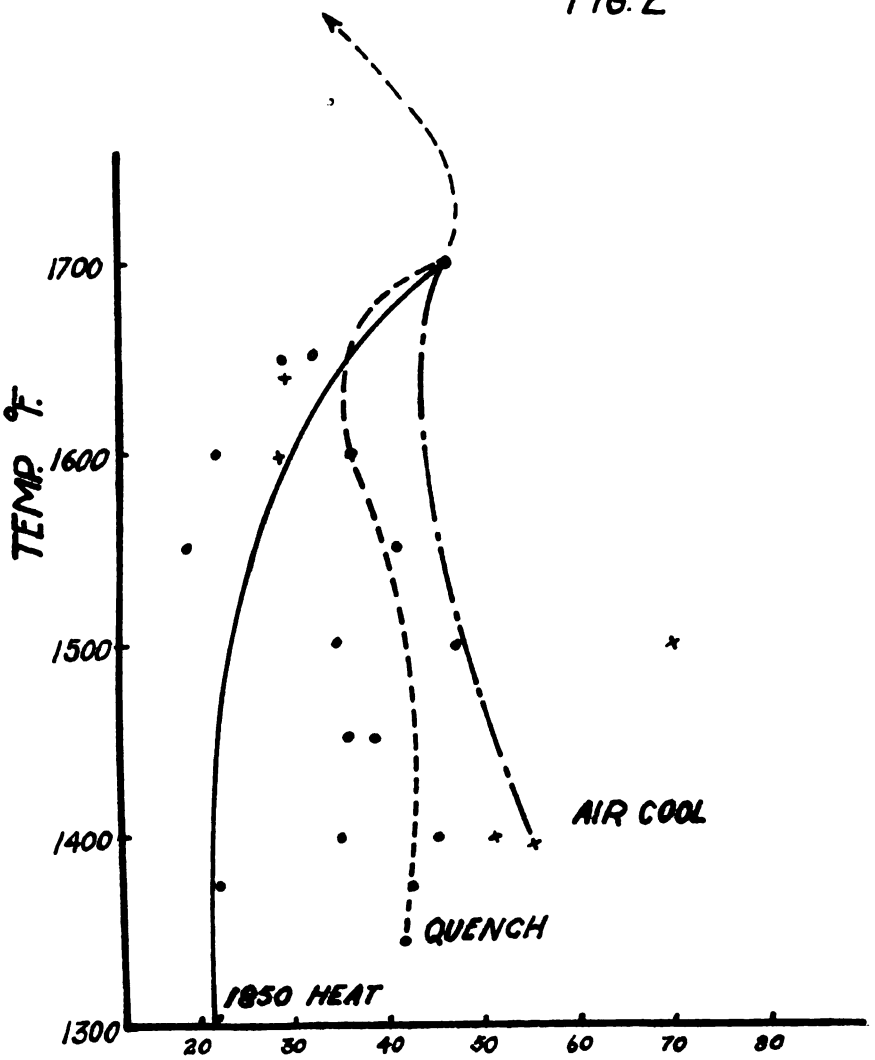
	As rec.	1375°	1400°	1450°	1500°	1550°	1600°	1650°	1700°
Armco iron	49	10	17	18	12	16	12	17	17
1850° heat	--	22	35	36	35	19	22	29	47
Air cool	68	39	51	39	70	--	29	29	47
Quench	99	42	45	37	47	41	36	32	46

Fig. 2 curve shows these results graphically.

Fig. 1 curve shows the temp. - crystal size curve.

ROCKWELL HARDNESS vs TEMP.
7½ HRS. ANNEAL

FIG. 2



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ROCKWELL HARDNESS vs TEMP
7½ HRS. ANNEAL

FIG 2

HARDNESS DETERMINATIONS, Cont.

Air Cool Series Experiment.

Sample #42 C.---) Mn.---) P.---) S.---) S.A.E. 1015

Given an 1850°, 4 hr. anneal and then given a normal reheat to the indicated temp., held one min. and then air cooled. Average rate of cooling thru the critical was 600° degrees per min. Hardness was determined the same as discussed in first part.

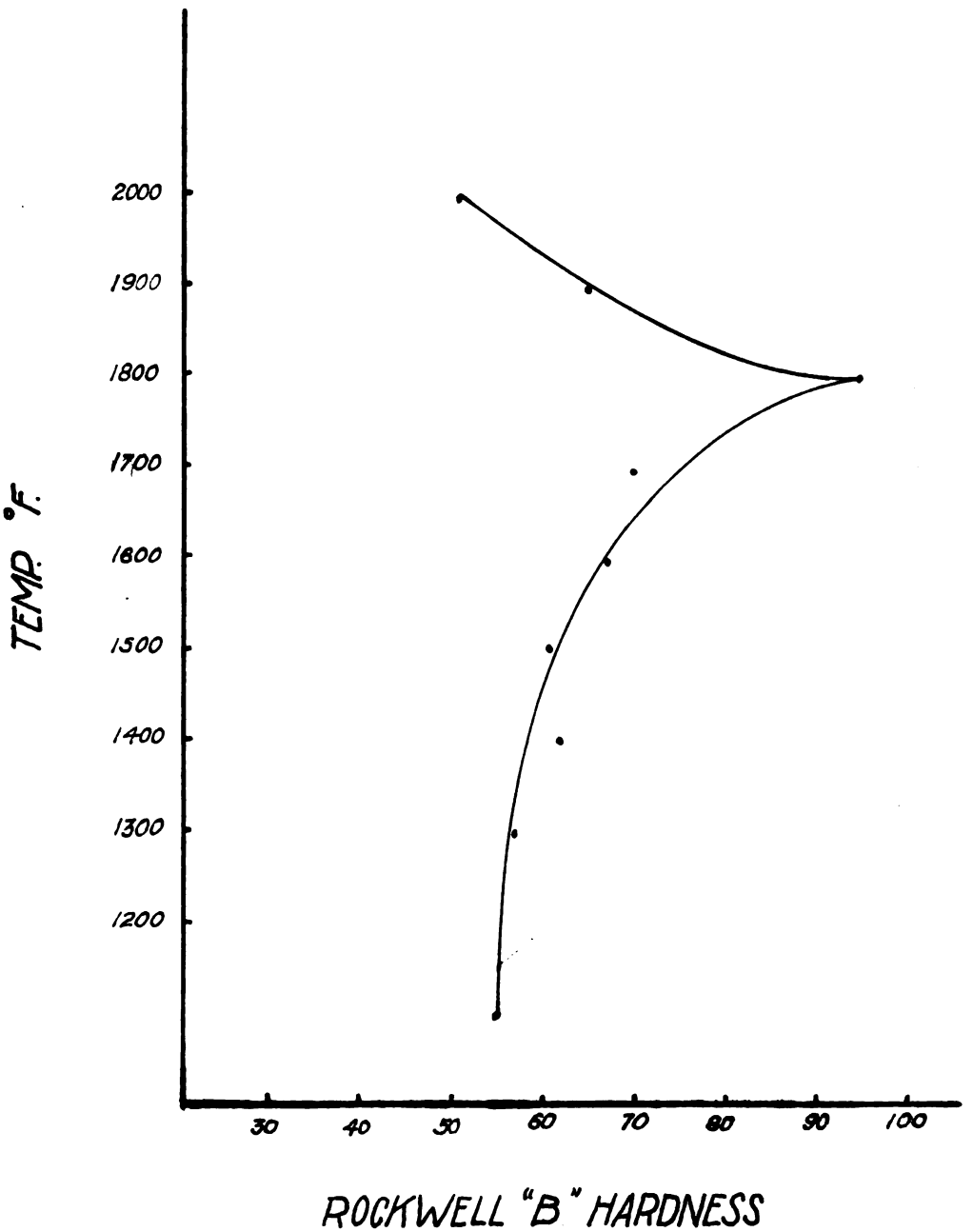
Rockwell "B" Hardness Readings.

Temp. F.	Size Sq. mm.	Hardness
Original	202	56
1250°	202	56
1300°	184	57
1400°	200	62
1500°	208	61
1600°	1330	67
1700°	804	70
1800°	600	95
1900°	595	65
2000°	600	51

Fig. 3 curve, shows the above results graphically, while Fig. 4 shows the temp.-grain size curve for the series.

ROCKWELL HARDNESS vs TEMP.
AIR COOL SERIES

FIG. 3



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GRAIN SIZE VS TEMP.
NORMAL HEAT—AIR COOL
ONE MIN. AT TEMP.

FIG. 4

GRAINS PER Sq. MM.

TEMP °F

2000 1900 1800 1700 1600 1500 1400 1300 1200

0 100 200 300 400 500 600 700 800 900 1000 1100 1200 1300 1400

ardness number f[illegible] or steel".

Our w[illegible] tester and verifies the above tatement with th[illegible] of the Rockwell method of measuring hardness.

A comparison of hardness-temp. curve with the grain size-temp. curve is interesting. Below A$_1$, a decrease in size produces an increase in hardness. Above A$_3$, a number of the grain results with the hardness still increasing and continues to do so above A$_{cm}$. At this last point the curve comes to an abrupt stop, above which the hardness tends to decrease.

In other words, something takes place around 1700-1800° which changes the hardness to the reverse of the grains increase a corresponding decrease in the hardness.

This [illegible] when [illegible] of strain as a hardness producer is considered in the theory [illegible] in another chapter.

On heating, when A$_1$ is [illegible], [illegible] the grain size around was in the old [illegible] grains [illegible] strain is still preserved [illegible] amount of strain is effected by a growth of individual [illegible] an aggregate. As these [illegible] they exert a still greater [illegible] crystals thus result-

GRAIN SIZE vs TEMP
NORMAL HEAT—AIR COOL
ONE MIN. AT TEMP
FIG 4
GRAINS PER SQ. MM.
°F TEMP
0
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
1500
1600
1700
1800
1900
2000

GENERAL DISCUSSION OF HARDNESS

Rawdon and Jimeno-Gil (4) have investigated the subject of grain size and hardness and have concluded that "a microscopic examination indicates that there is no simple and direct relation between grain size and Brinell hardness number for annealed carbon steels".

Our work was with the Rockwell hardness tester and verifies the above statement with the substitution of the Rockwell method of measuring hardness.

A comparison of hardness-temp. curve with the grain size-temp. curve is interesting. Below A_3, a decrease in size produces an increase in hardness. Above A_3, a coarsening of the grain results with the hardness still increasing and continues to do so some 200° above A_3. At this last point the curve comes to an abrupt stop, above which the hardness tends to decrease.

In other words, something takes places around 1700°-1800° which changes the hardness so that as the size of the grains increase a corresponding decrease in the hardness results.

This action can be explained when the factor of strain as a hardness producer is considered in the theory of grain formation as put forth in another chapter.

On heating, when A_3 is passed, numerous new austenite crystals are formed within the old alpha boundary. The general orientation of the old grain is still preserved by the aggregate of new crystals. A certain amount of strain is effected by a growth of individual crystals within an aggregate. As these individual crystals grow upon further heating, they exert a still greater force against neighboring crystals thus result-

ing in an increase in hardness with a growth of the grain. This action continues until the resulting austenite grain size approaches that of the original alpha grain and the increasing strain manifests itself until the old boundary is destroyed. At this point the crystals are of considerable size as compared to a point at A_3. The absorption of one grain by another, when the grains are of a large comparative size, a breaking down of the old alpha boundary, resulting in a decrease of strain -- or a still further increase in the austenite grain size above 1700°-1800° results in a decrease in hardness.

It is interesting to note that this hardness transformation point is located within the range 1700°-1800°, depending upon the rate of heating, cooling and length of time at temp. Considerable of our work with grain size has been done within this temp. range. At a temp. of 1850° for 4 hrs. or 1750° for 7½ hrs., we have concluded, is the range for producing equilibrium grain size. Stress or strain within the piece is evenly distributed at this point as well as an evenness in grain size.

It seems very consistent with the above work, to conclude that at a range 1700°-1800° is reached whereby an equilibrium grain size is produced with a maximum value of hardness for annealed steels, above this range grain growth resulting in a lessening of strain within the crystal.

EXPERIMENT 2 Part I.

Object: To determine the minimum grain size that can be obtained on an air cooled sample.

Discussion: The graph of an air cooled sample is shown in Fig. 4. Now if a sample representing a size A was run up thru the temp. range a crystal-temp. curve would be traced. The question arose as to the exact nature and shape of this curve and as to the location of the minimum point. If this second minimum point is located to the right of A then a refinement in the metal upon this second reheat has taken place. If refinement takes place then what is the smallest size crystal that can thus be obtained by successive reheats and air coolings?

Method:

Sample #44 with the following chemical analysis.

C.---- .17%
Mn.--- .42%
S.---- .028%
P.---- .011%

The sample was given an 1850°, 4 hr. heat, slow cooled, then a quick heat to 1680°, 2-5 min. and air cooled. The samples were then given a quick heat to the indicated temps., held for 5 min. and air cooled. They were $\frac{3}{4}$" rounds, one inch high with a hole drilled in the piece so as to insert the thermocouple.

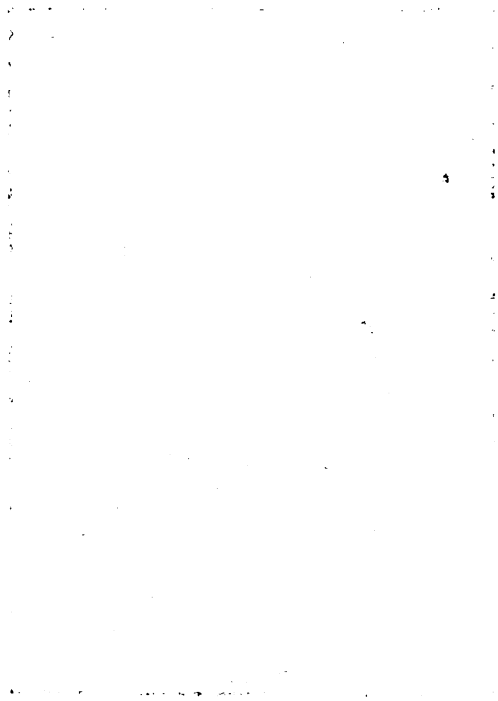
No.	Fig. No.	Crystal size per sq. mm.	Temp. and Treatment
44	5	212	1850°, 4 hrs., slow cooled
44	6	1060	Original air cool from 1680°
44E	7	1360	1300°
44A	8	1800	1420°
44D	10	1735	1510°
44B	11	1283	1600°
44C	12	1308	1710°

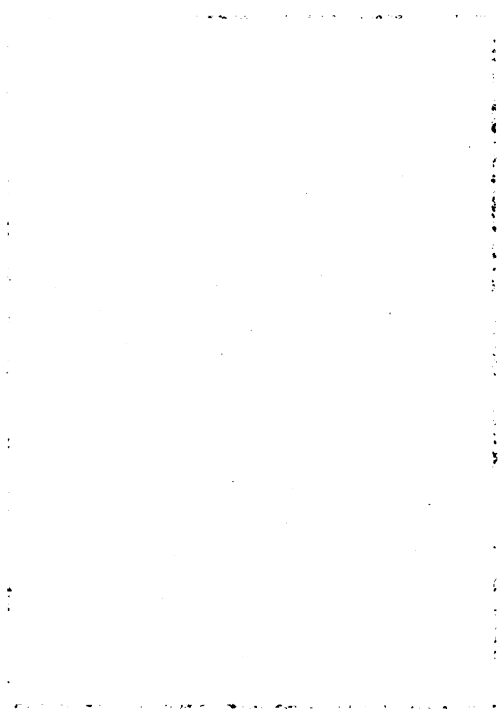
Graphically, curve 2 of fig. 9 shows the above results.

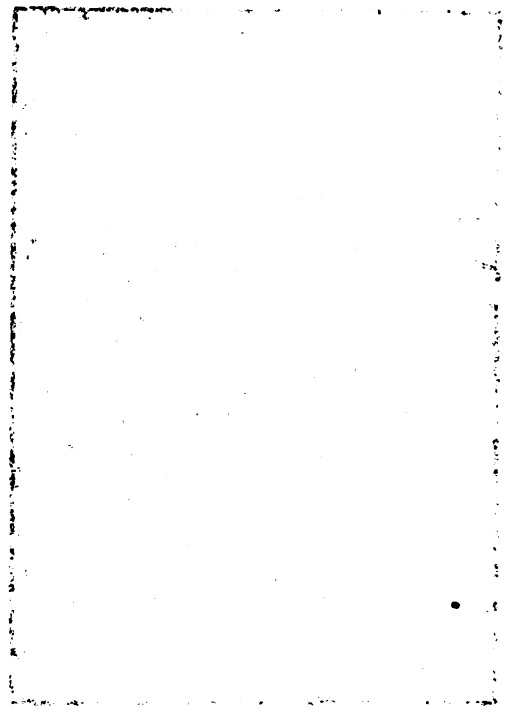
Three of these samples, 44E, 44A, and 44B, were again reheated to the indicated temps. and air cooled with the following results:

No.	Fig. No.	First heat Cry. size	Second heat Cry. size	Second reheat Temp.
44E-2	13	1360	1484	1300°
44A-2	14	1800	2262	1420°
44B-2	15	1283	1630	1600°

Fig. 9 shows these points graphically.







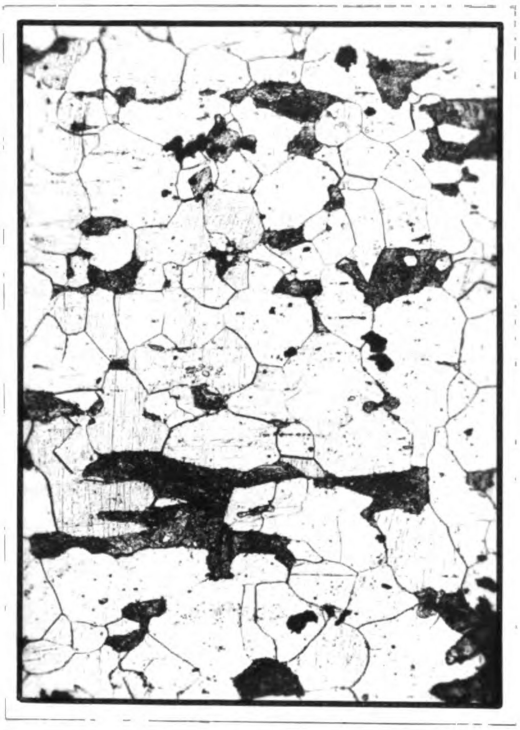


FIG.5

FIG. 6

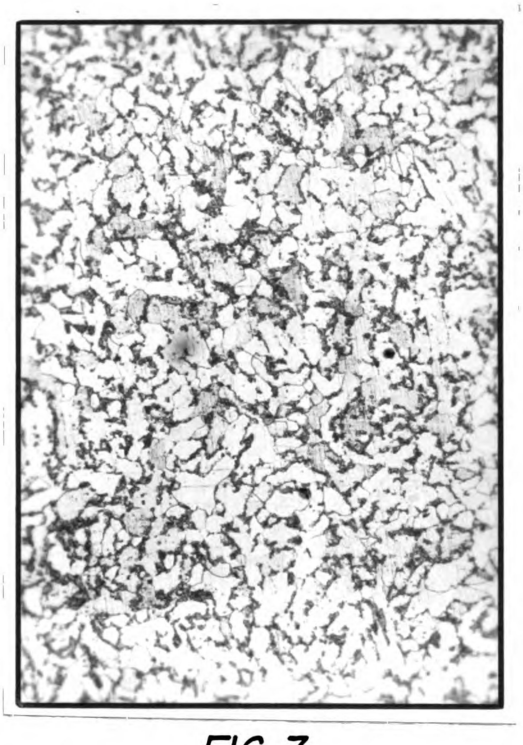


FIG. 7

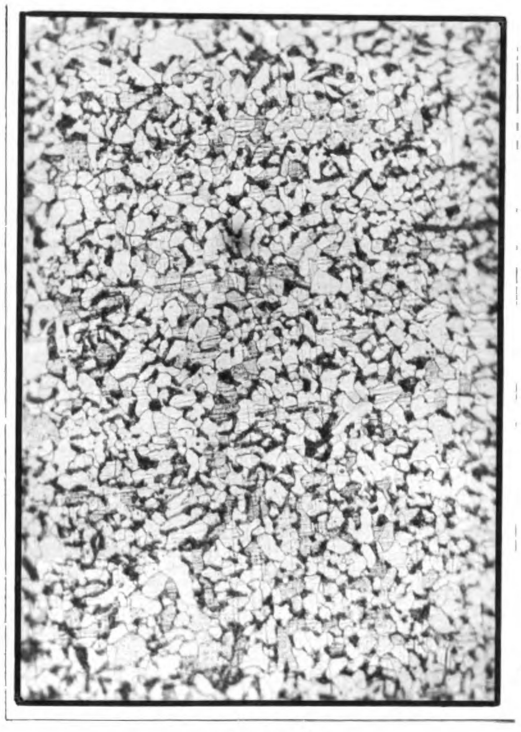
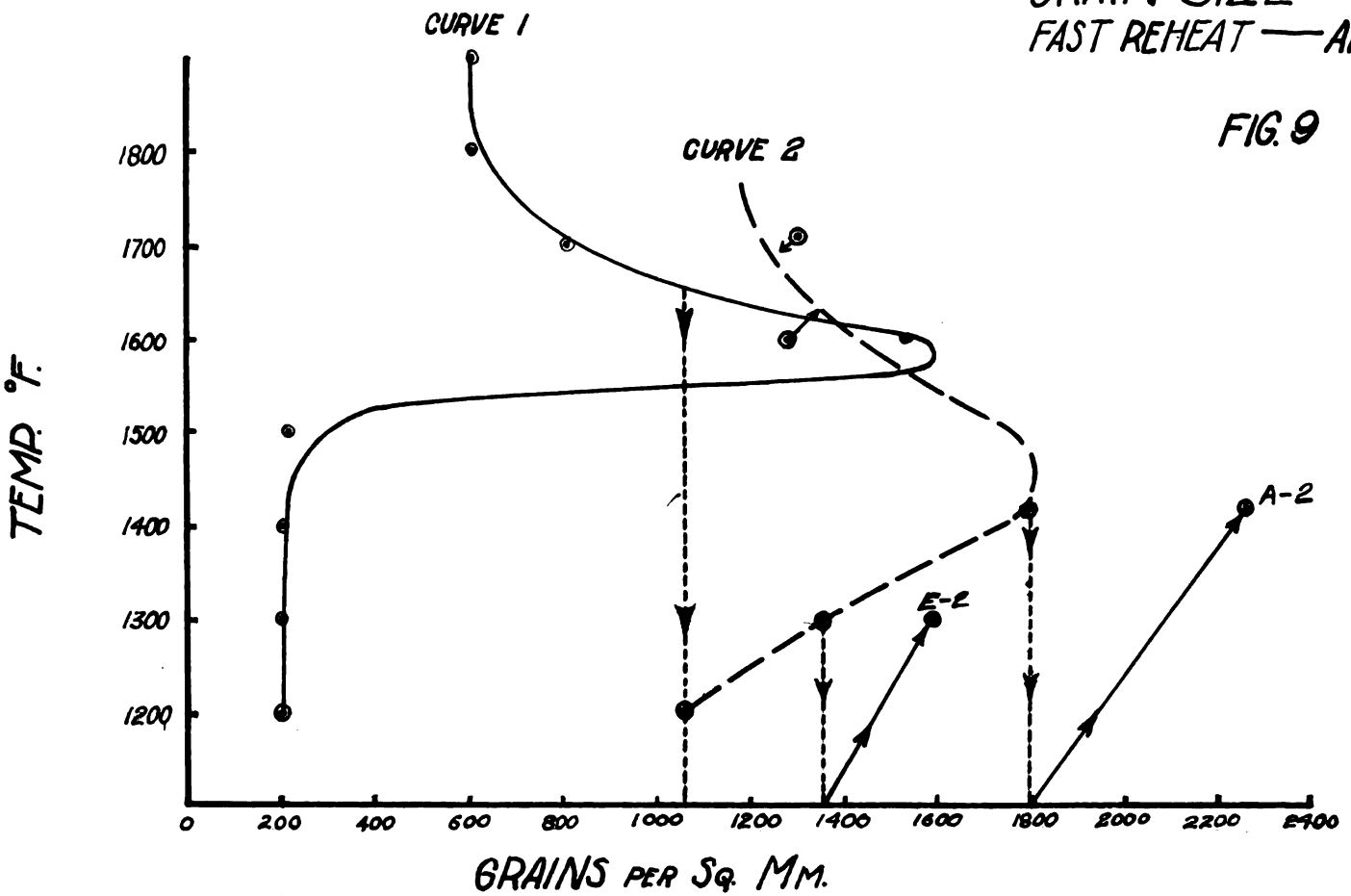


FIG. 8

GRAIN SIZE vs TEMP.
FAST REHEAT — AIR COOL

FIG. 9



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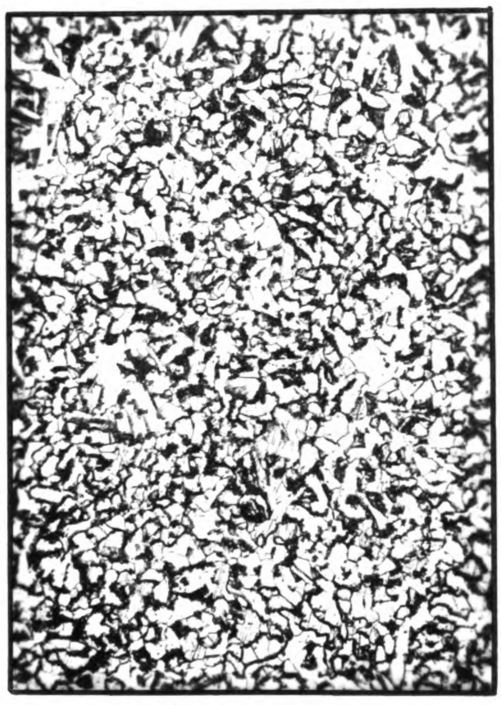


FIG. 10

FIG. 11

FIG. 12

FIG. 13

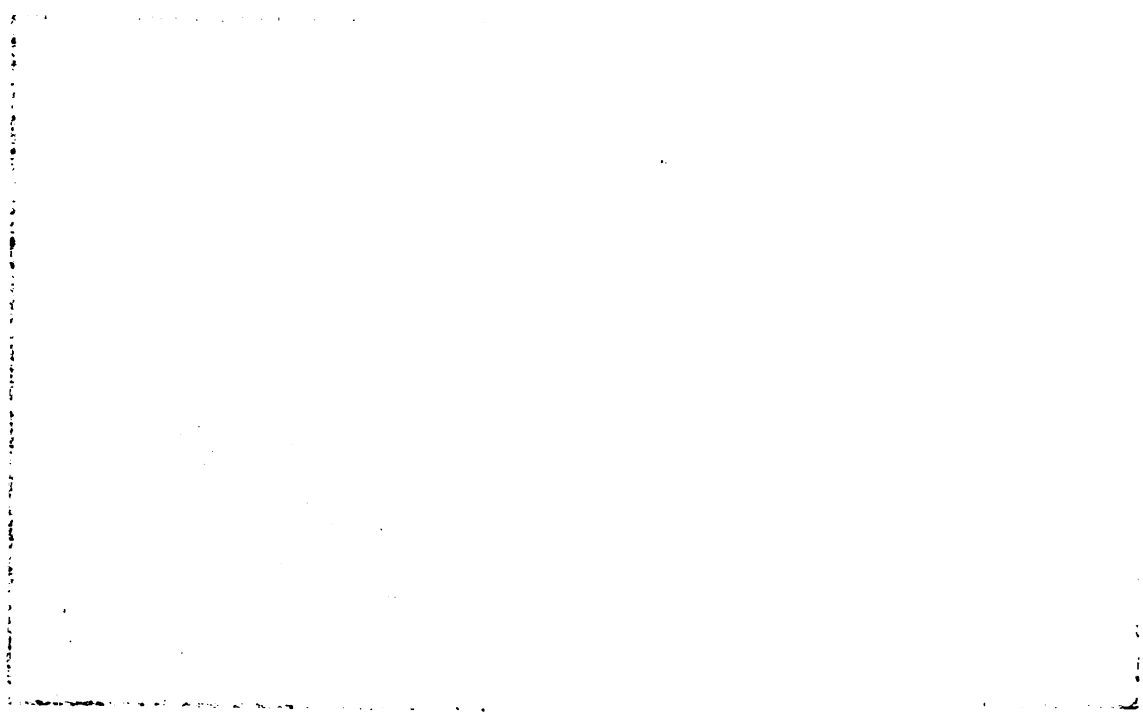


FIG. 1

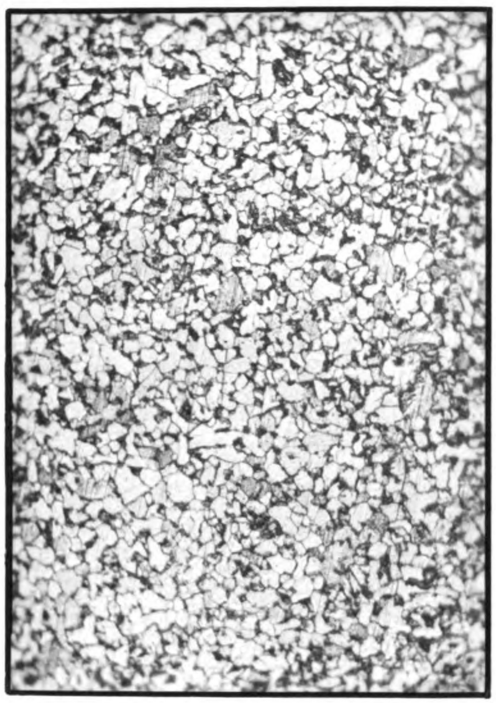


FIG. 14

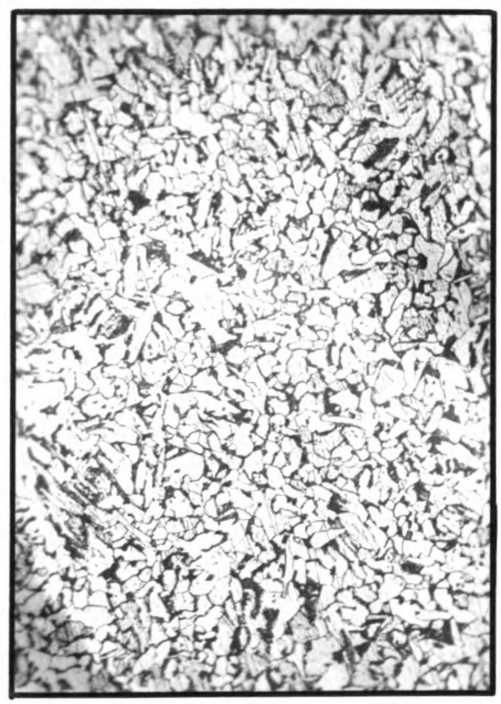


FIG. 15

FIG. 16

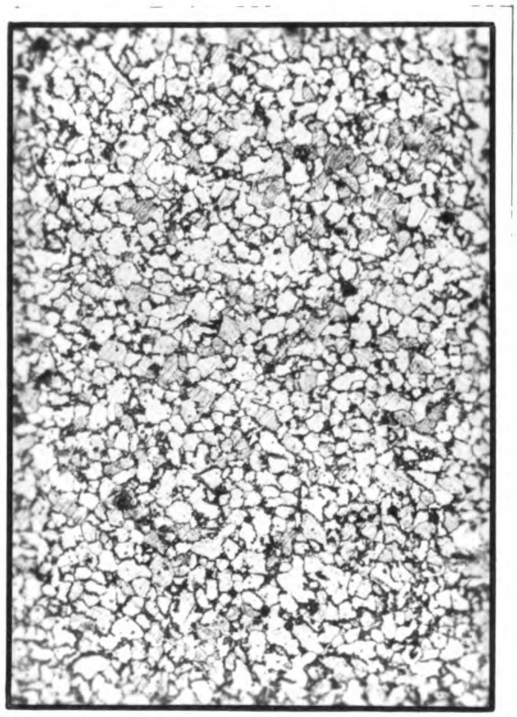


FIG. 17

EXPERIMENT 2 Part 2

Object: Determination of minimum crystal size that can be obtained upon successive reheats and coolings.

(a) Sample 44F, same as used in the first part of this experiment.

Had the following treatment:

1. 1850°, 4 hr., slow cool.
2. Reheat 1680°, air cool.
3. Quick heat to 1560°, instant, air cool to 1200°.
4. " " " 1560° " " " " 1200°.
5. " " " 1560° " " " " 1200°.
6. " " " 1560° " " " " 1250°.
7. " " " 1530° " " " " 1250°.
8. " " " 1530° " " " " 1300°.
9. " " " 1545° " " " " 800°, quench in water.

Final crystal size -- 2400 per sq. mm.

Fig. 16 shows the final product of this heat treatment.

(b) Sample 44G, same as used in the first part of this experiment.

Had the following heat treatment:

1. 1850°, 4 hrs., slow cool.
2. Reheat to 1680°, air cool.
3. Quick heat to 1600°, instant, air cool to 1200°, oil quench.
4. " " " 1580° " " " " 1200°, water quench.
5. " " " 1550° " " " " " " "
6. 1520° " " " " " " "
7. 1490° " " " " " " "
8. 1460° " " " " " " "
9. 1430° " " " " " " "
10. 1400° " " " " " " "
11. 1370°, one min., air cool to room temp.

Final crystal size -- 2200 (app.) per sq. mm.

Fig. 17 shows the final product of this heat treatment.

Discussion of part 1 and 2.

The idea in carrying out this experiment was to see if an exceedingly fine grained sample could be produced. Part 1 shows that the crystal size decreases upon a reheat and cool and that this minimum point is at

a lower temp. on reheating and cooling. Accordingly each successive reheat should be 15-30° lower than the preceding heat. (a) of the above produced a small, uniform, and normal grain but the same could have been produced in the first four operations. Accordingly (b) treatment was run. Approximately the same size grain was produced but the last treatments, being near the lower critical, shows considerable carbon diffusion, which is quite characteristic of air cooling operations. It is peculiar that fig. 16 following treatment (a) should produce an exceedingly normal structure.

The only explanation as to why not much finer grain was produced in 9 or 11 operations as outlined in either (a) or (b) as could have been produced in the first 4 operations is that after these first 4 operations an equilibrium grain size resulted. The force necessary to cause grain refinement of this equilibrium grain was not as great as the force set up by the velocity of the reaction.

The industrial application of this experiment lies in the fact that complicated treatments for grain refinement and especially "heat refinement" is of no value. Heat refining takes place in one or two operations after which the operations accomplish nothing.

Conclusions:

1. Successive reheatings and air coolings tend to cause an equilibrium grain size.
2. Until this size results these successive operations tend to refine the grain.
3. Each crystal-temp. curve, as determined by successive reheats and coolings from the minimum point of a previous

3. curve, up until equilibrium size results, lies to the right of its preceding curve.
4. The minimum point on successive curves lies at a lower temp. from its preceding curve.

EXPERIMENT 3

Object: Effect of time and temp. on grain size.

Discussion: In bulletin #14, "Grain Formation in Low Carbon Steel Within the Critical Ranges" (17) we have investigated the effect of time and temp. on grain formation. The conclusions drawn from those exps. were as follows:

1. It appeared that any grain size desired may be obtained in a low carbon steel.
2. This is accomplished by controlling the rate of heating, length of time at heat, and rate of cooling.
3. The temp. for obtaining a minimum grain size upon one treatment is the temperature at which solid solution is complete.
4. This temp. lies just below the upper critical point (for small samples).

Here is reported some further investigations along the same line.

Method:

Sample #14 with the following chemical analysis.

C.---- .16%
Mn.-- .48%
S.---- .033%
P.---- .015%

Samples were of $\frac{3}{4}$" round, $\frac{3}{4}$" high with a 3/16th hole drilled in one end to receive the couple. The sample was first given a 4 hr. heat at 1850°, slow cooled (Fig. 18) and reheated to the indicated temp. by a fast heat, held indicated time and air cooled.

Results:

No.	Fig. No.	Temp. °F.	Time Min.	Cry. size per sq. mm.
14	18	1850 °	4 hrs. slow cool	212
14B	20	1550 °	1	1900
14C	21	1580 °	1	1529
14D	22	1600 °	1	1620
14E	23	1510 °	2	1396
14F	24	1535 °	2	1729

FIG 18

FIG 19

FIG 20

FIG 21

FIG. 18

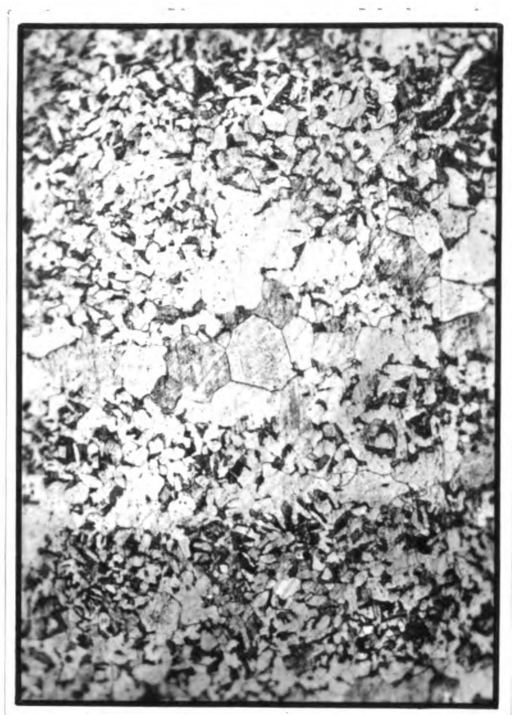


FIG. 19

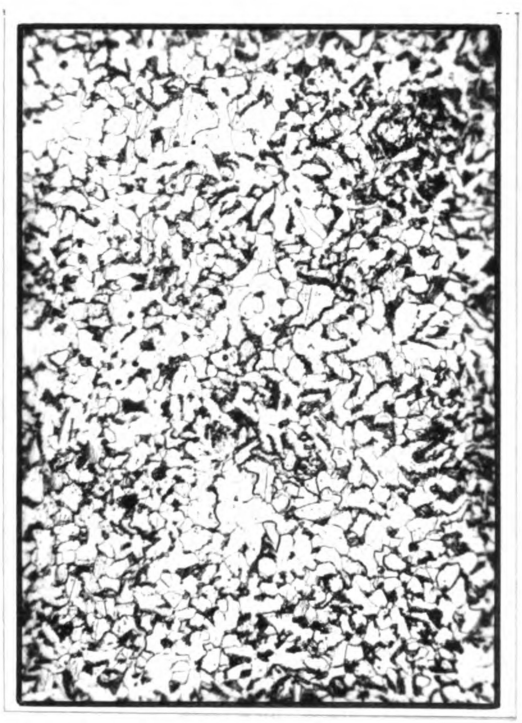


FIG. 20

FIG. 21

FIG 22

FIG.23

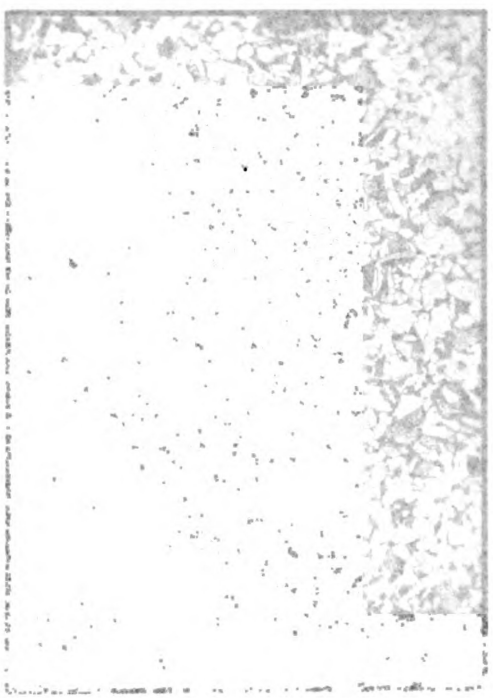


FIG 24

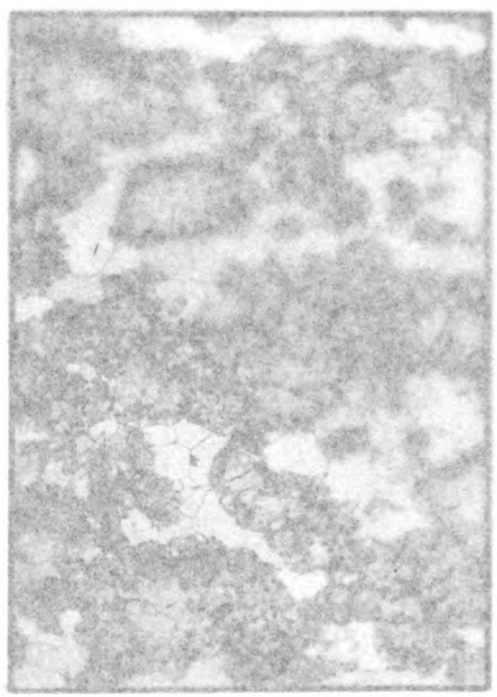


FIG. 25

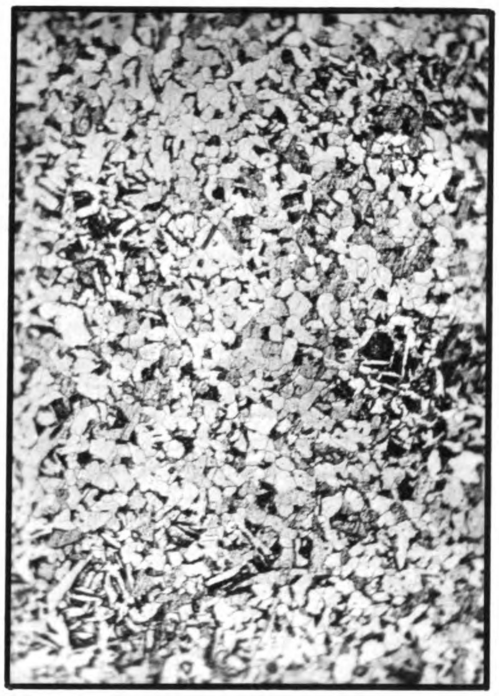


FIG. 22

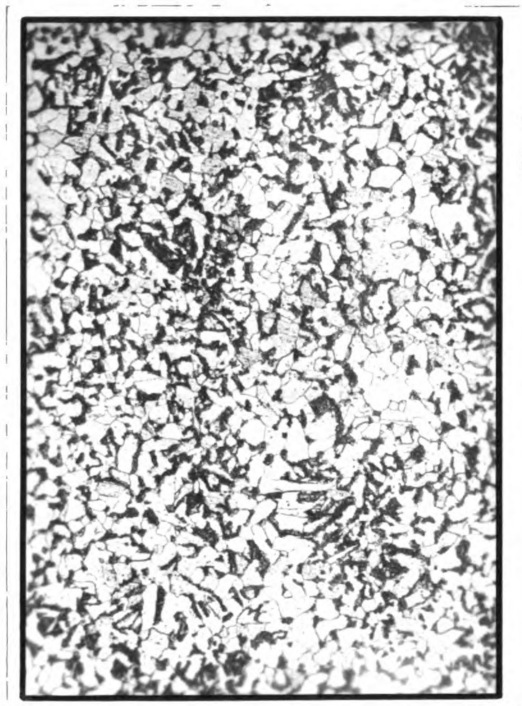


FIG. 23

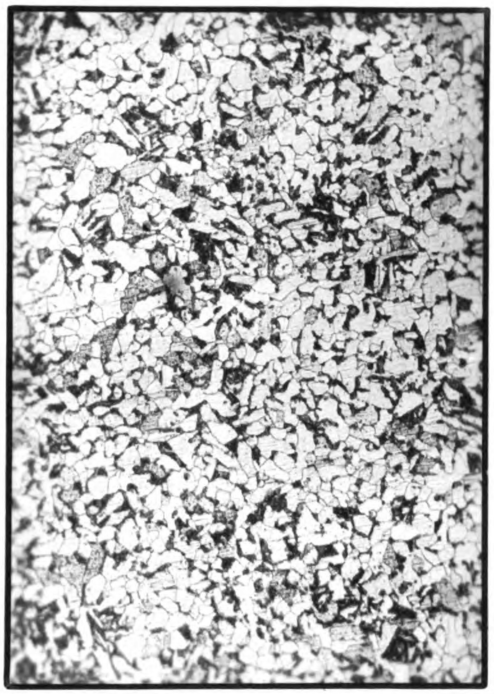


FIG. 24

FIG. 25

Discussion of Results:

This experiment has given additional proof to each of the four conclusions as listed in the first part of this exp.

An inspection of the photomicrographs shows that grain growth depends to a great extent upon a difference in grain size or "grain size contrast". The refinement consists first (fig. 19) in the formation of large ferrite crystals which act as a matrix and small crystals of cementite or pearlite and ferrite. This is due to the gradual absorption of the ferrite by the pearlite --- the carbon diffusion or penetration of the ferrite by the carbide. Grain size contrast is thus produced by a difference in the transformation temp. produced by the carbon or pearlite adjacent to the ferrite.

These results have considerable industrial applications. Steel of these characteristics is usually worked from a temp. corresponding to the upper critical. To obtain a maximum refinement such steel should just be heated thru, no soaking be allowed to take place, and then be allowed to cool relatively fast. Once the point of minimum refinement is reached, the velocity for growth takes place rapidly.

Time at heat is also a vital factor. 2 min. at 1535° produced a finer structure than one min. at 1580°, while the smallest size of this series was obtained with one min. at 1550°.

EXPERIMENT 4

Grain size and hardness of annealed Armco Iron.

Armco iron in the as received condition was given a $7\frac{1}{2}$ hr. anneal as in experiment 1.

Results:

Temp.	Size per sq. mm.	Rockwell "B" Hardness
Original	262	49
1375°	254	10
1400°	247	17
1450°	232	18
1500°	224	12
1550°	217	16
1600°	217	12
1650°	192	17
1700°	105	17

Fig. 26 is the temp.-size curve.

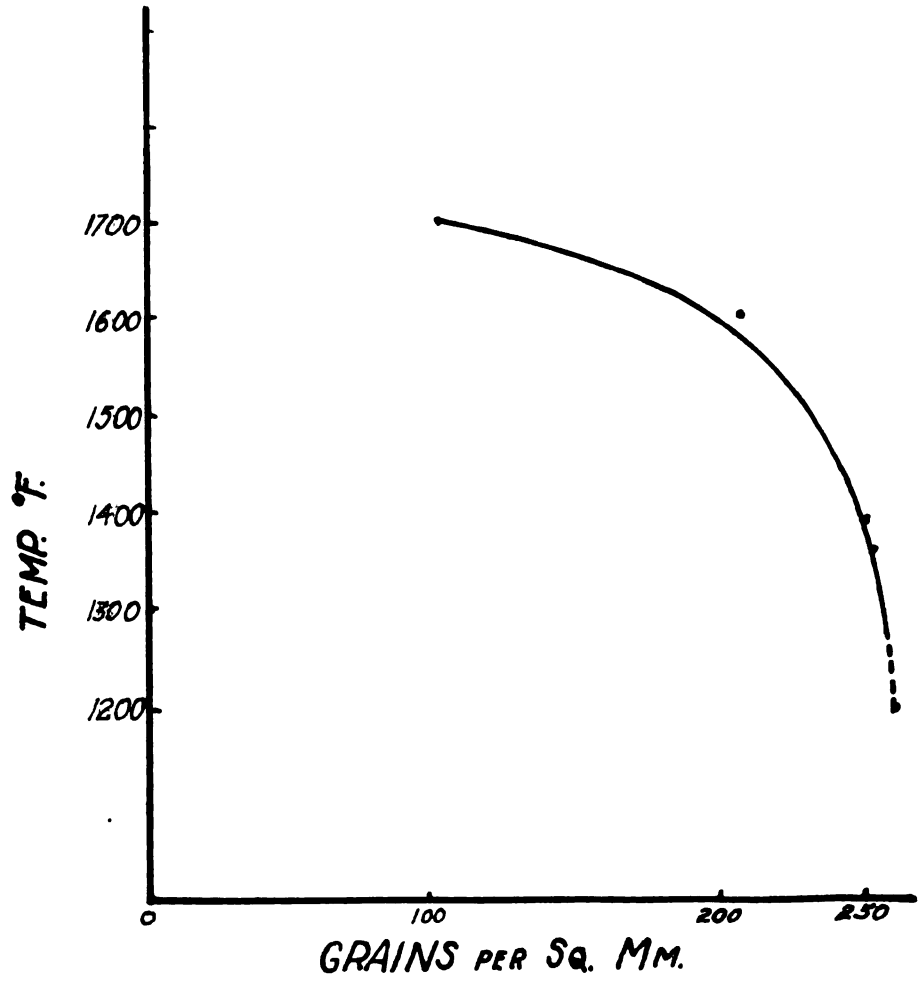
Fig. 27 is the temp.-Rockwell Hardness curve.

DISCUSSION AND CONCLUSIONS

1. No appreciable growth or refinement takes place in this steel upon a $7\frac{1}{2}$ hr. anneal in the temp. range covered.
2. After strain, caused by rolling or cold work, is removed, no variations in the hardness values are seen. The removal of strain causes a decrease in the hardness.
3. In Armco iron we would have no austenite formed on heating above A_3. As no new crystal nuclei are formed, as evidenced by neither a refinement as compared to a .20% C. steel in heating and cooling thru the critical range, it is logical to assume that in carbon steels the refining element is carbon

GRAIN SIZE vs TEMP.
ARMCO IRON – 7½ HRS. ANNEAL

FIG. 26



Waldron '28

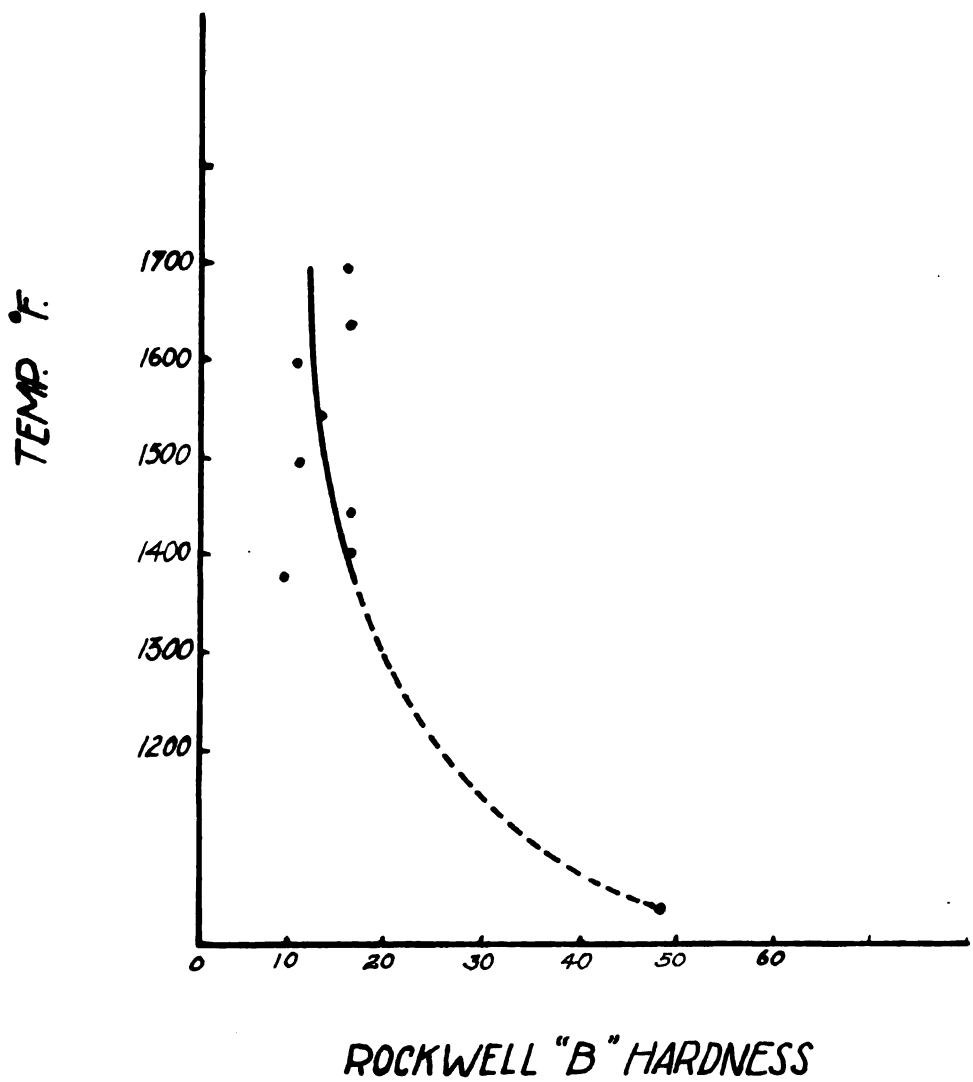
GRAIN SIZE vs TEMP.
ARMCO IRON – 7½ HRS. ANNEAL

FIG. 26

Woodrow '28

ROCKWELL HARDNESS vs TEMP.
ARMCO IRON – 7½ HRS. ANNEAL

FIG. 27



Waldron '28

ROCKWELL HARDNESS vs TEMP.
ARMCO IRON – ¾ HRS. ANNEAL

FIG. 27

Wadsworth 328

3. and that the formation of austenite involves a new set of centers of crystallization. This substantiates our contentions as set forth in the chapter on crystallization in hypo-eutectoid steels.

4. Inasmuch as the hardness above 1300° is constant and though the ferrite grains do grow somewhat, there is no strain set up in them when the change alpha to gamma occurs as does in the change alpha to austenite in carbon steels.

THEORY OF CRYSTALLIZATION OF A HYPO-EUTECTOID STEEL

First let us put forth a working hypothesis of what happens when, say, a .20% carbon steel is heated from room temperature up thru the critical temperatures to 2000°F.

Say that the constituents, pearlite and ferrite, are in a normal annealed state and that this normal arrangement of the constituents is not seriously distorted as the result of cold work or strain.

Nothing of importance happens until the lower critical is passed. Sauveur has said regarding some observations that he made in 1912, "These observations point to the conclusions that ferrite grains will not grow on annealing below the critical range unless they have been subjected to a certain stress creating a certain strain".

After reaching the lower critical the ferrite and cementite in the pearlite grain form a solid solution of austenite. At the same time this ferrite changes its space lattice to that of the gamma pattern -- a change which involves the simultaneous formation of new nuclei centers.

This nuclei action is not very well understood but probably begins at various points. Perhaps these points lie in the boundaries of the existing ferrite grains, because small particles of cementite or impurities may be located there. Their presence facilitates nuclei formation much the same as an introduction of a foreign substance facilitates crystal formation in the cooling of a saturated chemical solution. Figure 25 shows the large ferrite crystals intact while the pearlite and some adjacent ferrite crystals have been broken up by the austenite solution.

The ferrite grain boundaries offer a certain resistance to the penetration of the solid solution but once this resistance is overcome by an energy application in the form of heat the whole ferrite crystal yields readily to

the solid solution. The mechanism involved is the gradual absorption of the ferrite by the austenite. This is a progressive reaction -- one grain of ferrite being absorbed at a time, though the time intervening between the absorption of any two grains may be infinitely small.

Rosenhain, speaking of this reaction says, "The transformation of ferrite from the alpha to the gamma state, quite apart from the influence of adjacent carbon in lowering the transformation temperature does not occur suddenly or uniformly thruout the mass, even in a single ferrite crystal".

The ferrite in contact with cementite undergoes the allotropic change to gamma iron at a much lower temp. than Az.

As one ferrite grain yields to solution in the austenite many new nuclei are formed, and crystallization or formation of the austenite grain starts.

Holding at temp. or raising the temp. tends to combine nuclei within the grain.

At this point it is well to note one of our contentions -- our experimental results have led us to believe that grains grow from within the original alpha grain and not in the boundary as some believe.

The growth is more rapid the higher the temp. or the greater the heating velocity. Thus many austenite grains are formed within the old ferrite grain boundary, each austenite grain having its own orientation, but the whole aggregate of grains being confined in their orientations to that of the old alpha grain boundary. I do not necessarily mean that the boundary persists. This may be true providing the boundary is composed of amorphous cement, but inasmuch as the amorphous cement theory has some objections, this hypothesis is set forth without its regard.

Up till this time, considering that we have not as yet complete solid solution, we have

1. Old pearlite changed into austenite.

2. Some alpha grains as yet unabsorbed.

3. Some alpha grains changed into gamma grains composing solid solution within the old alpha grain boundaries.

This (3) solid solution is composed of individually orientated austenite grains, the whole aggregate still preserved as a unit within the old boundary. The small crystals near the border being orientated in such a manner as to conform to the barriers set up by the neighbor crystals. Thus a distinct boundary line sets off individual groups of austenite grains from one another. This condition still prevails, even after solid solution results, and is not obliterated until higher temps. are reached. In other words, up till a temp. of say 1700-1800° is reached the austenite grain inherits its size from the alpha grain and a "clean slate" in regard to previous treatment is not set up as stated by Ruder.

The tendency of the steel is toward an aggregate of homogeneous crystals of gamma iron solid solution, a condition which may practically never be obtained (or may be obtained only after high heats or for given durations of time at such heats).

From this point and to higher temps. a coarsening of the austenite grain takes place, thus yielding on cooling large alpha grains.

ABNORMAL AND NORMAL CARBURIZING STEELS

Inasmuch as considerable work has been done on this class of steels, it was thought advisable to include a chapter outlining the work covered and to show a series of photo-micrographs.

Prof. Publow in his work, "Grain Growth in Low Carbon Steel," has covered grain growth in abnormal steels. He has found that abnormal steel, after reactions at 1850°, reacts like normal steel as far as grain growth and refinement are concerned. The general conclusion then was that normal and abnormal steel react alike as far as grain size is concerned. This statement may be true after the steel is given our 1850° anneal, but inasmuch as carburizing temps. of 1700° are used, the reactions toward growth at the lower temp. have been studied.

Experiment 1 has shown that two pieces of normal steel in different initial states do not come to the same size at 1700° after a 7½ hr. anneal. An abnormal piece always shows a much finer grain in the core than does a normal piece, indicating that in the first case something was present to obstruct growth and that this "something" also obstructed the penetration of carbon so that a much narrower case was produced. It can not be that a small grain offers a greater resistance to carbon diffusion than a large one, in that Jalcase, a steel which has its grain size held back by a manganese content, will take a very deep case. Nevertheless a study of grain size at 1700° has produced results when confined entirely to S.A.E. 1020 steel.

Experimental Work

Different samples of 1020 steel were used. Each was given a preliminary treatment of 1850°, 4 hrs., slow cooled. They were then carburized

at 1700° for 7½ hrs., and slow cooled in the pot. Heating was in nichrome bombs packed with cast iron shavings to keep carburization to a minimum.

The samples were then cut across with a saw, filed, and put on paper wheels to obtain a flat surface. Three specimens were placed in a group on a flat magnet with a mold circling them. Around them was poured a low melting point lead-bismuth alloy. The whole was then ground and polished. Etching was by means of a ¾-1% nital. Pictures were taken showing grain size in the core (at 125X), and representative ones are here shown.

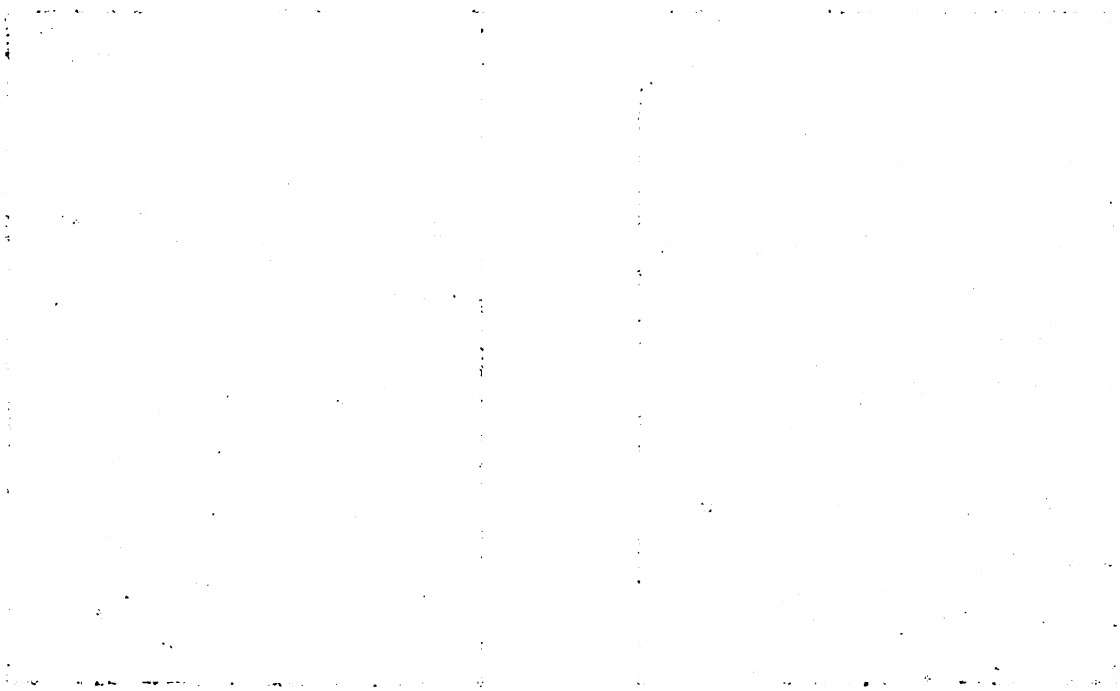
Results:

Sample No.	C. Content %	Grain size of core per sq.mm.	Dep. of Case in in.	Degree of Normality
52	.11	254	.040	
55	.17	355	.038	
54	.10	600	.027	Very abnormal
22	.15	248	.040	Normal
1	.20	368	.035	
40	S.A.E. 1015	410	.033	Partly abnormal
42	" "	362	.049	Very normal
35	.20	414	.039	Slightly abnormal
57	.14	600	.040	Very abnormal
46	.21	359	.035	Fairly normal
14	.16	423	.038	Fairly normal
5	.20	374	.040	Fairly normal
66	.19	340	.040	Normal
28	.20		.038	Fairly normal
32	.20		.031	Both normal and abnormal
26	.16			Normal
18	.16	285		Fairly normal
51	.10	301		Very normal
21	.18	355		
29	.18	343		Fairly normal
8	.22	348		Fairly normal
9	.18	297		Fairly normal
43	.14	298		Fairly normal
30	.18	317		Fairly normal
44	.17	309		Fairly normal
17	.16	328		Fairly normal
49	.20	290		Fairly normal

Discussion:

A glance of the table shows that the degree of normality can be determined by a consideration of the grain size of the low carbon core. Considerable interest has been caused, in the industrial world, by the adoption of a grain size chart of the hyper-eutectoid zone in case carburized steels. Some companies have even gone as far as to specify certain sized grains that must result upon carburization. Any one who has had anything to do with the determination of grain sizes in hyper-eutectoid steel realizes the task at hand. The chart is based upon the cementitic network but should one examine at high magnifications what is supposed to be a whole grain enclosed by this network, many fine grains will be found.

The grain size of the core is not hard to determine accurately. Consequently where grain size specifications are of importance, the substitution of determinations of the core for that of the case can be made resulting in a more accurate method.



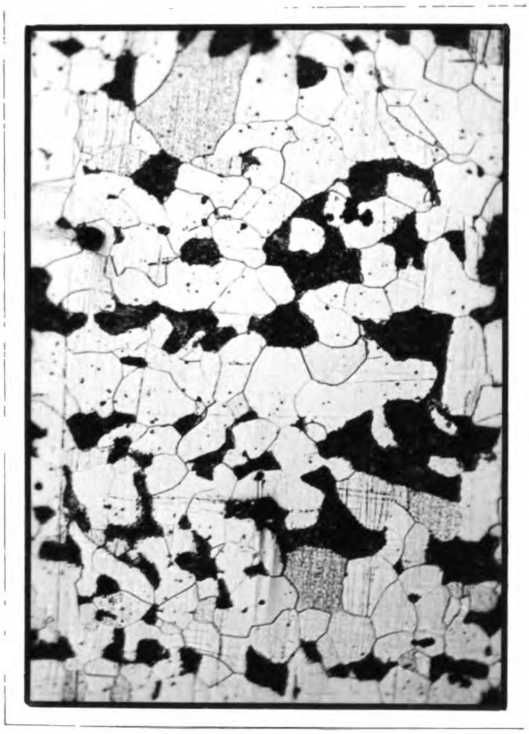


FIG.28 125X

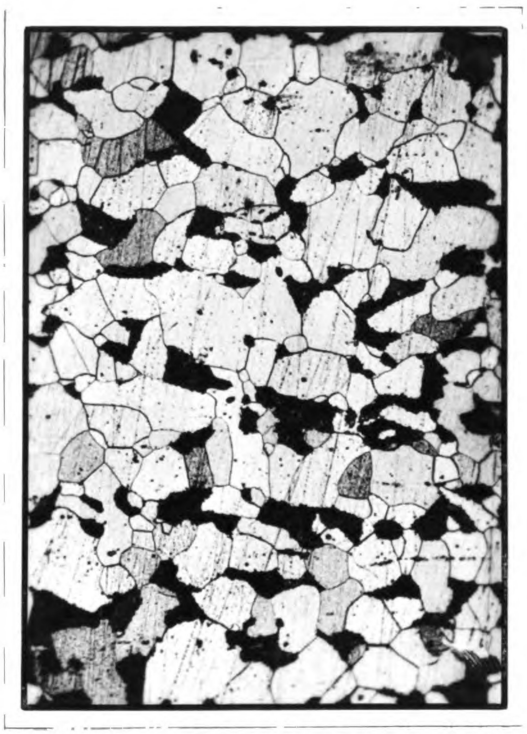


FIG.29 125X

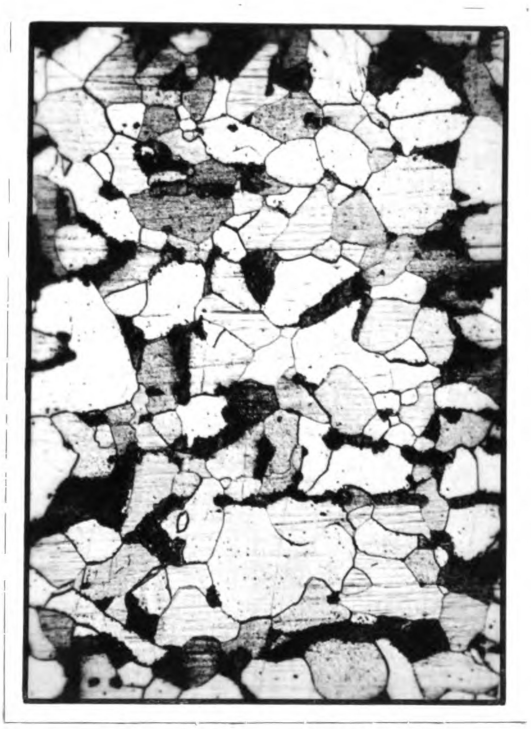


FIG.30 125X

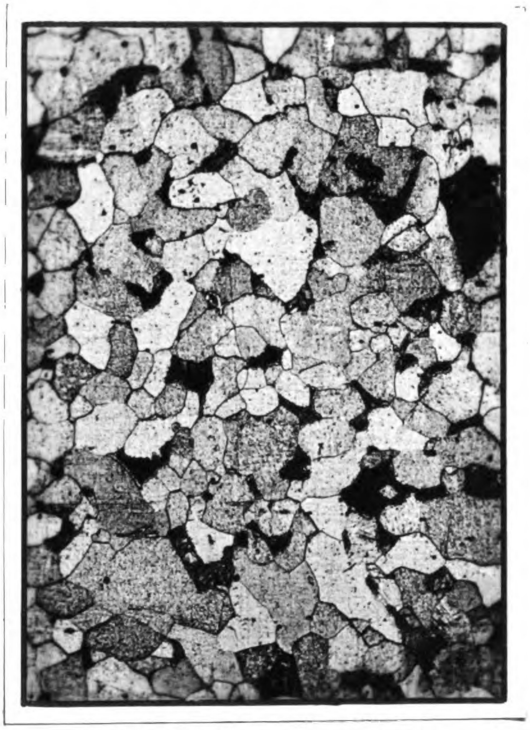


FIG.31 125X

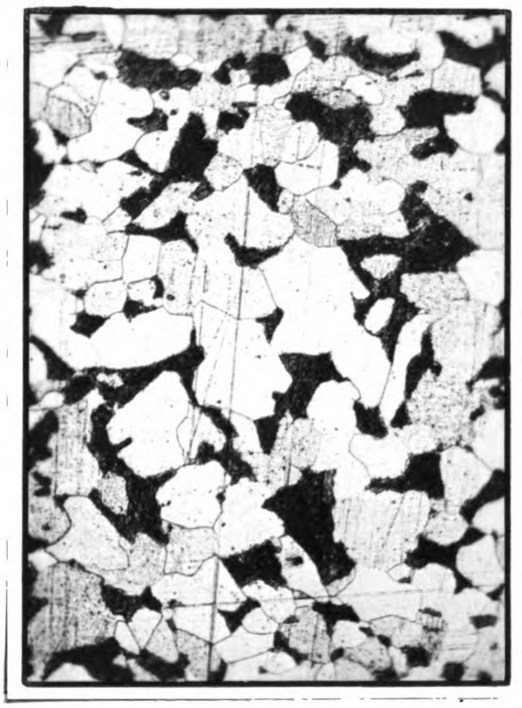


FIG. 32 125 X

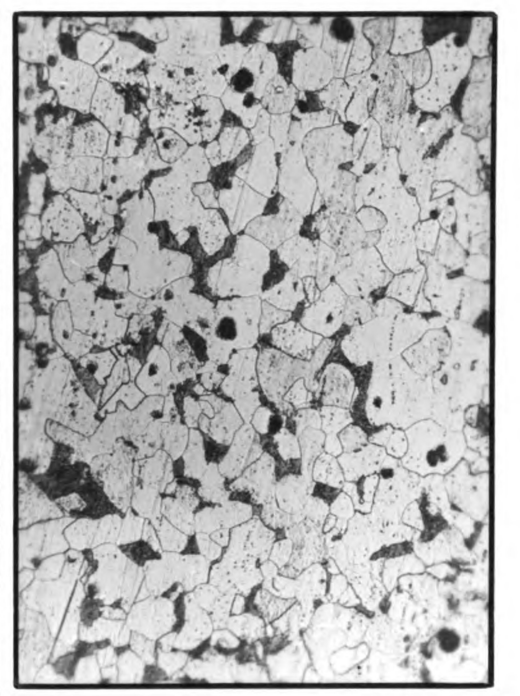


FIG. 33 125 X

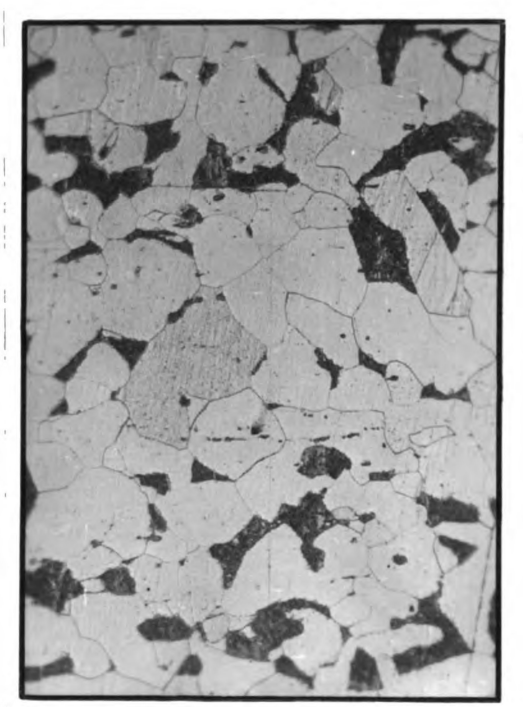


FIG. 34 125 X

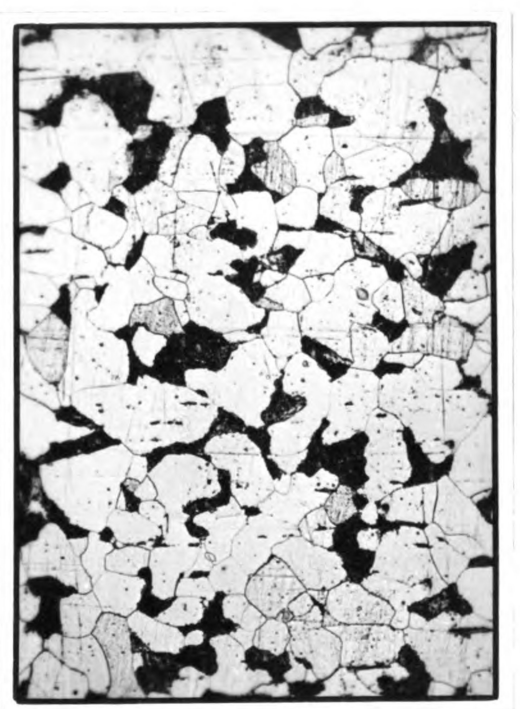
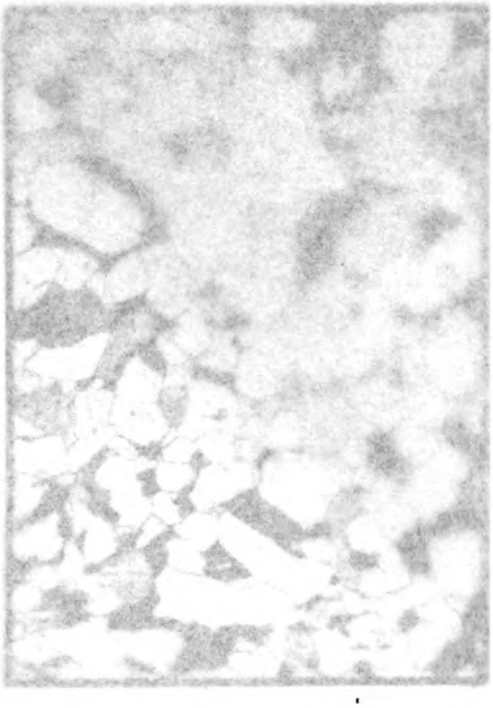


FIG. 35 125 X

FIG. 125X



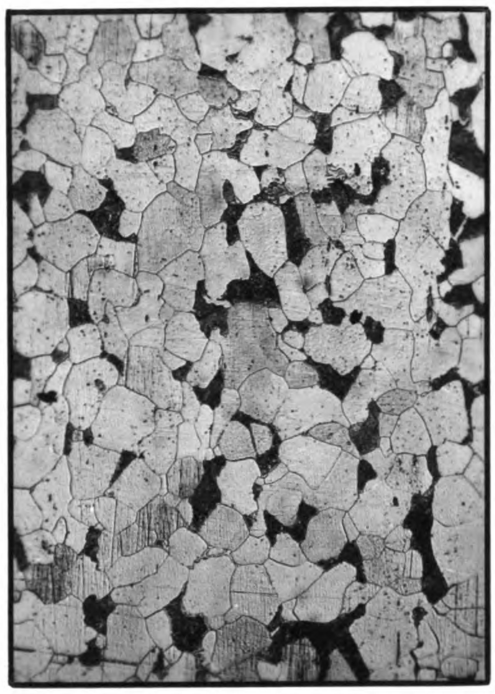


FIG36 125X

FIG.37 125X

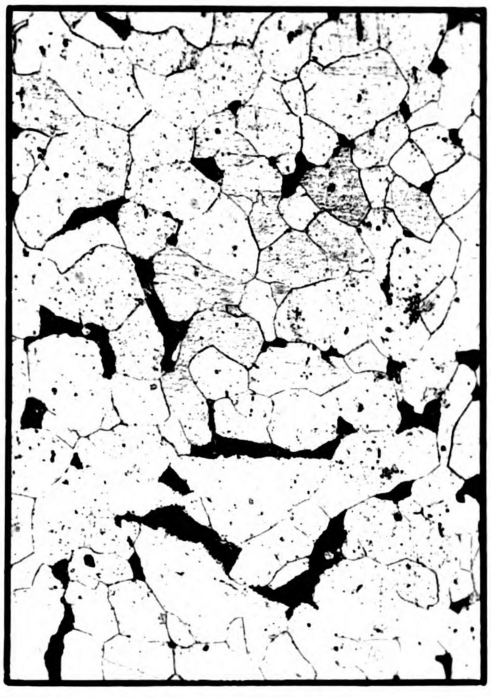


FIG.38 125X

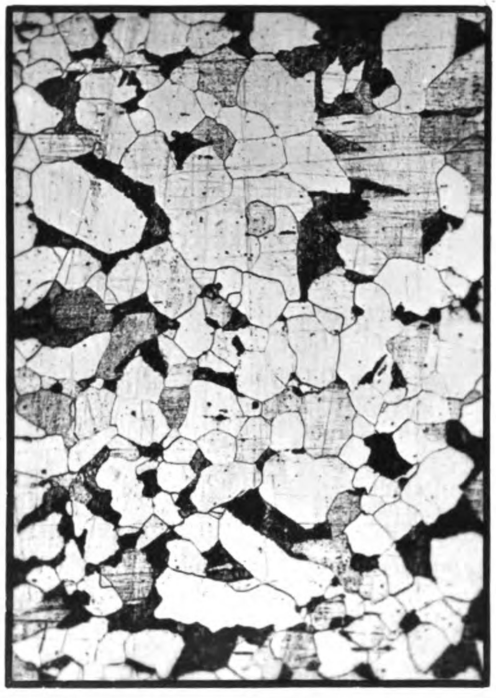


FIG.39 125X

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