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ACICULAR FERRITE IN A PLAIN CARBON
AND AN ALLOY STEEL

Thesis for the Degree of M. S.
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James G. House
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This is to certify that the
thesis entitled
ACICULAR FERRITE IN A PLAIN CARBON AND AN
ALLOY STEEL
presented by
James G. House

has been accepted towards fulfillment
of the requirements for

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Robert L. Sweet

Major professor

Date June 29, 1951

ACICULAR FERRITE IN A PLAIN CARBON
AND AN ALLOY STEEL

By

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INTRODUCTION

Alpha ferrite exists in two forms, the common blocky type, and the needle, spear or acicular type. The reason that one type or the other will form is not clearly understood, but Jolivet (1)* with an X-ray study of the needles, states that the needles were true ferrite, as they have an alpha lattice.

The acicular ferrite structure was mentioned as early as 1917 and 1918 (2, 3). That it was not studied earlier was probably due to the fact that the structure was not encountered in the commercial heat treatments on the plain carbon steels that were in use before that time. Isothermal transformation which would have detected the structure in plain carbon steel did not receive much consideration until work on S-curves was started (4) in 1930 by Davenport and Bain (5).

In 1920, Hultgren (6) made reference to two kinds of ferrite in a study of an alloy steel. Acicular ferrite came into prominence in 1939, when two articles (1, 7) were published that described the structure in detail. Jolivet (1) described the structure, and noted that the aciculae were parallel within any one austenite grain and that the needles often included globules of

* Numbers in parentheses refer to the bibliography.

carbide or were surrounded by carbide. His work also included the afore-mentioned X-ray study proving that acicular ferrite had an alpha lattice. Davenport (7), in his discussion of acicular ferrite, called it the X-constituent. This paper is so widely quoted that in speaking of acicular ferrite most authors make note that they are speaking of the X-constituent of Davenport.

Recent papers (8, 9, 10, 11, and 12) have done much to clarify the field of acicular ferrite, but there are still many major questions unanswered.

Bainite, or at least upper bainite, is an integral part of any discussion upon acicular ferrite, because at the present time there is no definite dividing line between them. If the question is to be answered as to where or if a dividing line exists, the mechanism of bainite formation should be looked into, as it is in dispute at the present time. Some writers, such as Davenport (7), Flinn, Cohen and Chipman (8), and Zener (13), agree that bainite forms by a nucleation and growth process. Others, such as Loria and Shephard (11), and Smith and Mehl (14), show some evidence that it may be a lattice shearing reaction, at least under some conditions.

Most of the research to date on acicular ferrite has been done with alloy steels. It is the purpose of

this paper to try to answer some of the aforementioned questions, and in addition, to investigate acicular ferrite in a plain carbon steel.

EXPERIMENTAL PROCEDURE

The steels used were taken from commercial heats of S.A.E. 1040 and S.A.E. 4140 which had the following mill analyses:

Steel	C	Mn	P	S	Si	Cr	Mo
S.A.E. 1040	.42	.78	.039	.038	-	-	-
S.A.E. 4140	.41	.94	.018	.039	.30	1.10	.19

Both steels were in the form of one and one-half inch diameter hot rolled rounds.

The plain carbon steel was cut from the bar in lengths of about one foot and homogenized in a globar type furnace at $2300 \pm 50^{\circ}$ F. for three hours and allowed to cool in air. The alloy steel was given the same treatment except that it was placed in a capped pipe and heated in a gas furnace. At the end of the three hour heating period the pipe was taken off, so that the normalizing treatment obtained was the same as for the plain carbon steel.

Samples of the S.A.E. 1040 were cut from these homogenized bars on a power hack saw. As the normalizing treatment had partially hardened the S.A.E. 4140, these samples were cut on a water-cooled abrasive cut-off wheel. Both of these methods gave trouble in controlling

the sample thickness, so that the samples had to be ground down to a thickness of approximately one-sixteenth of an inch on a wet abrasive wheel. Such thin samples were used to get fast surface cooling rates in the salt bath and quench. Because so much time was used in sawing off the samples of S.A.E. 4140, they were further sawed in two on a diameter, thus decreasing the sawing time to obtain the needed number of samples.

The samples were then drilled with a number 54 drill and wired with light Nichrome wire for handling in the furnace, salt bath and quench.

For the final heat treatment the wired samples were placed, ten at a time, in a ceramic crucible, and covered with used carburizer to minimize decarburization. The crucible holding the samples was placed in a small resistance furnace held at $1900 \pm 10^{\circ}$ F for a period of 30 to 50 minutes. One by one, the samples were withdrawn from the furnace, placed in the salt bath for the desired length of time, and then quenched in water to change all untransformed austenite to martensite. Salt bath temperatures were 1200, 1100, 1050, 1000, 900, 850, 800, and 700° F with a tolerance of $\pm 3^{\circ}$ F at any temperature. This small temperature difference tolerance was held by placing an auxiliary thermocouple in the salt bath and reading the temperature on a

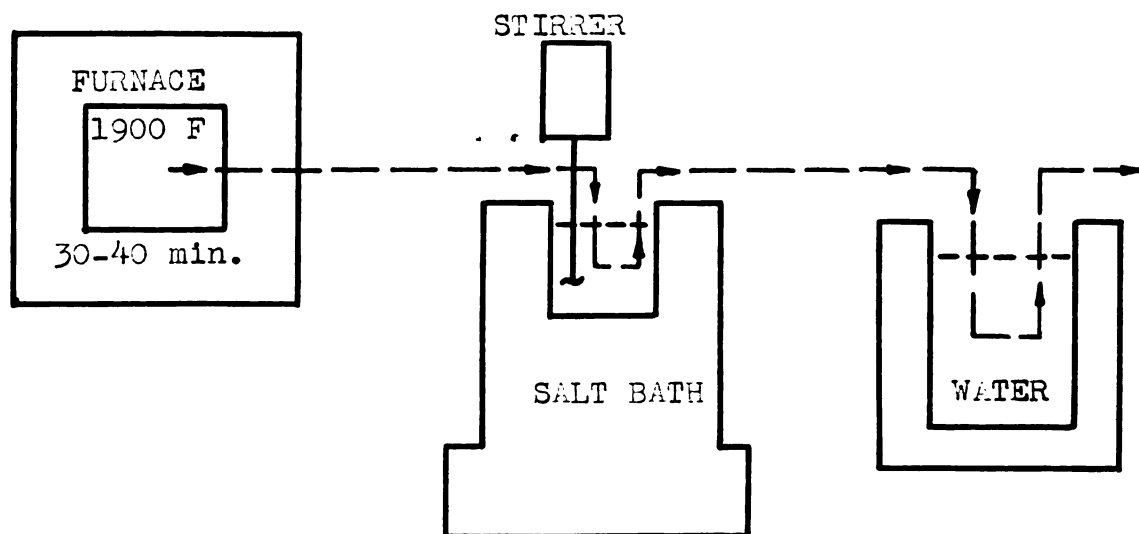


DIAGRAM OF APPARATUS

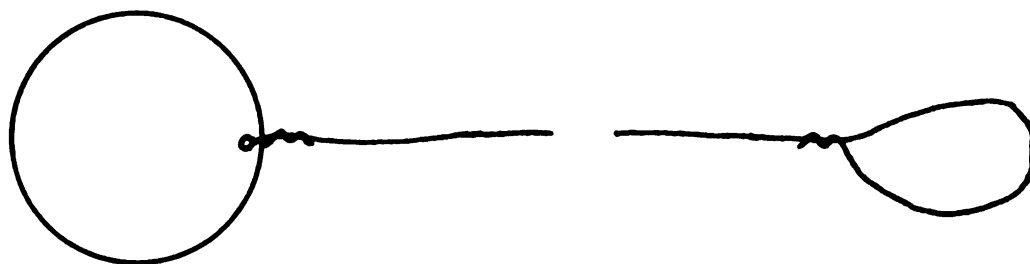


DIAGRAM OF 1040 SAMPLE
(ACTUAL SIZE)

potentiometer with a fine scale. Samples were placed in the salt bath only when the temperature was within the desired tolerance.

Variations in the salt bath temperature were responsible for the waits which caused the austenizing times to differ by as much as 20 minutes. However, the 30 minute minimum austenizing period was more than adequate for complete carbide dissolution.

The time in the salt bath, for all of the plain carbon steels was one second for the first sample, two seconds, for the second, and so forth, up to ten seconds for the tenth. Seconds were counted manually for the short times; i.e., one to four seconds, and measured with the sweep second hand of a wrist watch for longer times. The accuracy of this method was of the order of ± 0.5 of a second, which is significant for the short transformations. The time of movement from the salt bath to the quench tank, a distance of approximately two feet, also adds to the error by adding to the transformation time.

It would be well to eliminate these errors; but for short transformations, the time for the sample to become completely submerged after it has touched the bath, and the time of coming out of the bath and moving to the quench, approach 0.5 of a second. Therefore, it

would be difficult to devise an apparatus which would reduce this error to insignificance.

A stirrer was placed in the salt bath to dissipate any thermal inhomogeneities and increase the surface cooling rate of the samples. Time in the salt bath for the S.A.E. 4140 was considerably longer than the S.A.E. 1040 due to the greater hardenability. With the salt bath at 1100° F, ten samples of S.A.E. 4140 were run at 60 seconds, 70 seconds, 80 seconds, and so forth, to 150 seconds. At salt bath temperatures of 800° F and 900° F, samples were run at five seconds, ten seconds, 15 seconds, and so forth, up to 50 seconds.

The samples in sets of ten were placed in clamps, ground back at least one-sixteenth of an inch to remove decarburized areas, and polished. They were etched in Vilella's reagent (1 gm. picric acid, 5 ml. HCl, and ethyl alcohol to make 100 ml.). The samples were inspected and photographed on a Bausch and Lomb research metallograph at 500X unless otherwise specified.

DISCUSSION

A plain carbon steel was selected to carry on the major portion of this investigation, as the mode of ferrite precipitation unaffected by alloy additions was desired. Carbon content selection was governed by the balancing of the factors: 1. A large amount of ferrite at low carbon content and, 2. Enough carbon to give an appreciable carbon content difference between ferrite and bainite. The alloy steel was used as a check to see if a small alloy addition changed the ferrite precipitation mechanism. In addition, the alloy steel would have sufficient hardenability to make positive that transformations at all temperatures were 100% isothermal.

The steels were homogenized three hours at 2300° F to remove to a large extent any concentrations of any element or constituent that was built up during the solidification of the steel and not removed during rolling, as these concentrations might give anomolous effects. Preliminary work and a review of the literature showed that the ferrite precipitation was largely a grain boundary phenomina; therefore an austenizing temperature of 1900° F was used to enlarge the grains and thus give large grain boundary area. Figure 1 is an

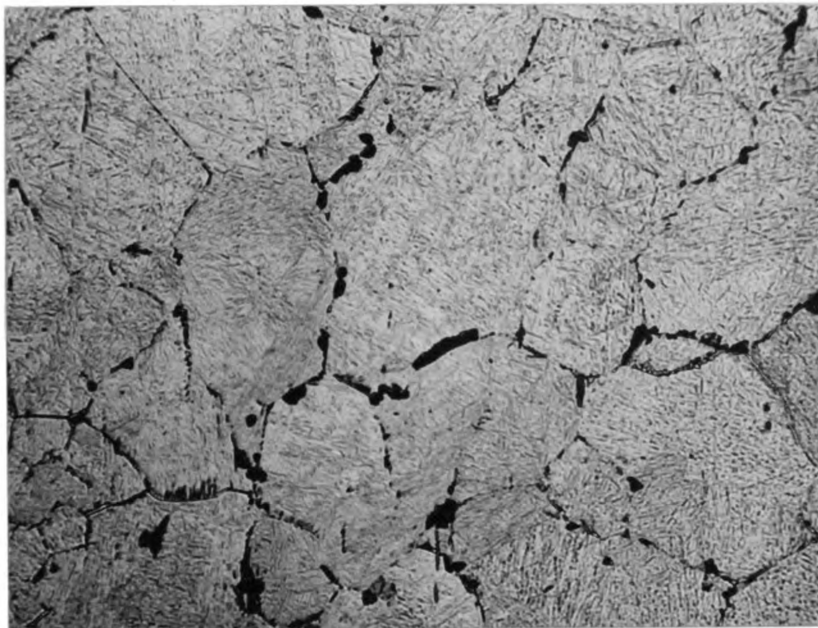


FIGURE 1

S.A.E. 1040, Transformed 9 sec. at 1100 F°
Vilella's Reagent X 100

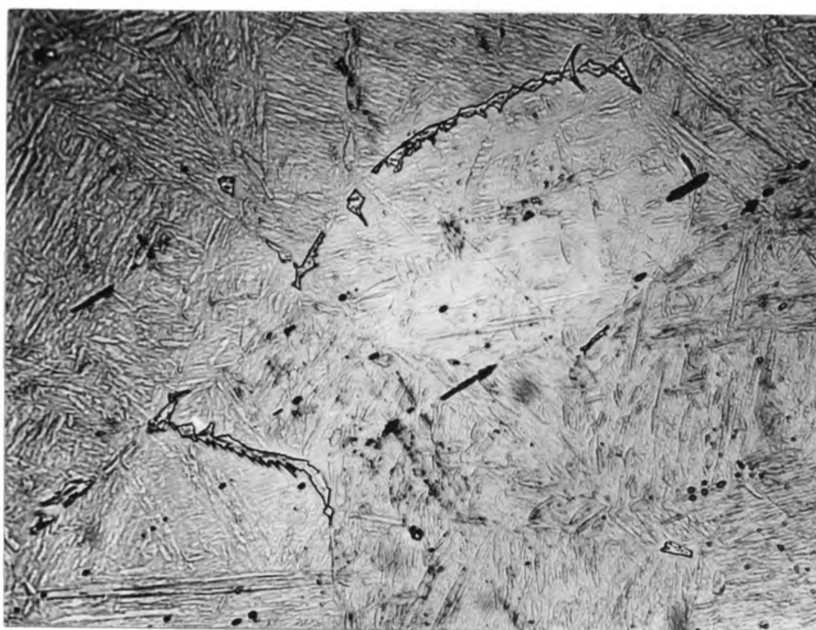


FIGURE 2

S.A.E. 1040, Transformed 9 sec. at 1200 F°
Vilella's Reagent X 500

example of the grain size obtained. The large grain size coupled with the high austenizing temperature gave an increased hardenability. The shift to the right in the S.A.E. 1040 S curve was slight due to the extremely short times needed to start transformation, but was very noticeable on the S.A.E. 4140.

Before discussing the transformations it might be well to explain what is meant in this paper by the terms: ferrite, bainite, and bainite nucleated by ferrite. Ferrite is a light etching grain boundary precipitate. Bainite is the dark etching precipitate which is general throughout the grain. Bainite nucleated by ferrite is the dark precipitate which grows away from grain boundaries where a ferrite precipitate has given rise to the nucleation.

At high temperatures; namely 1200° F, 1100° F, 1050° F and 1000° F, and short times after the start of transformation the ferrite tended to precipitate wholly in the grain boundaries (see figures 2, 3, 5, and 7). It may also be noted that nucleation started at many points along the grain boundary. This is especially evident in figure 3. After longer periods of transformation the small nucleations which had tended to be acicular became exceedingly acicular, and the aciculae grew to great lengths (see figures 4, 6, and 8). Upon comparison of



FIGURE 3

S.A.E. 1040, Transformed 3 sec. at 1100 F^o
Vilella's Reagent X 500

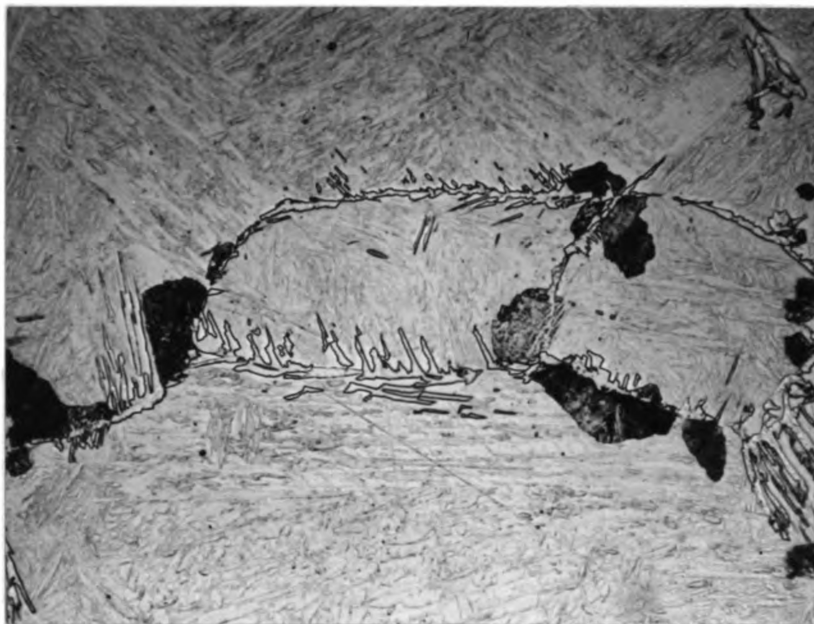


FIGURE 4

S.A.E. 1040, Transformed 9 sec. at 1100 F^o
Vilella's Reagent X 500

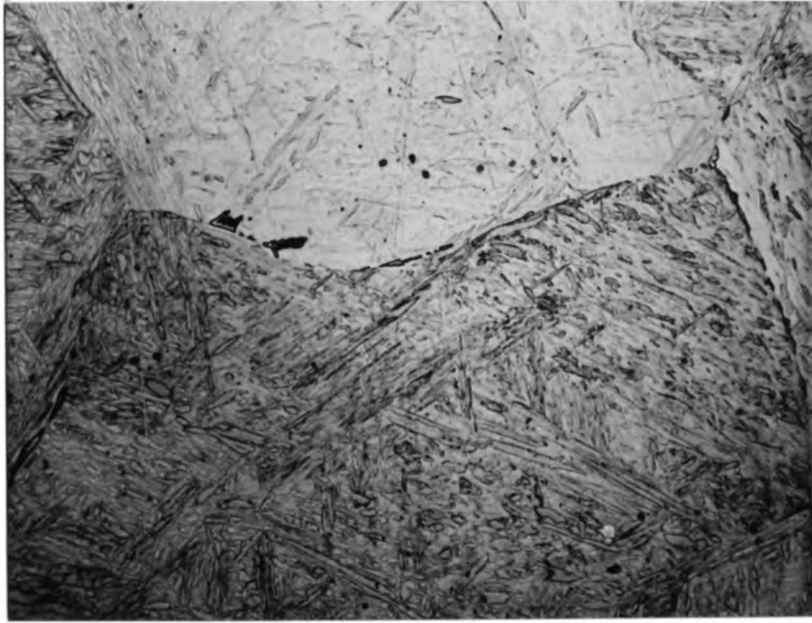


FIGURE 5

S.A.E. 1040, Transformed 2 sec. at 1050 F°
Vilella's Reagent X 500

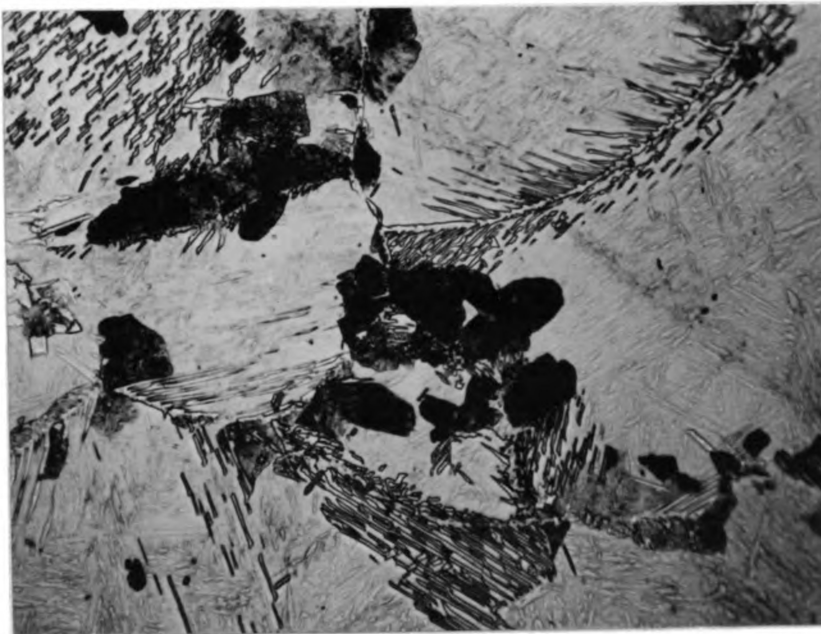


FIGURE 6

S.A.E. 1040, Transformed 9 sec. at 1050 F°
Vilella's Reagent X 500

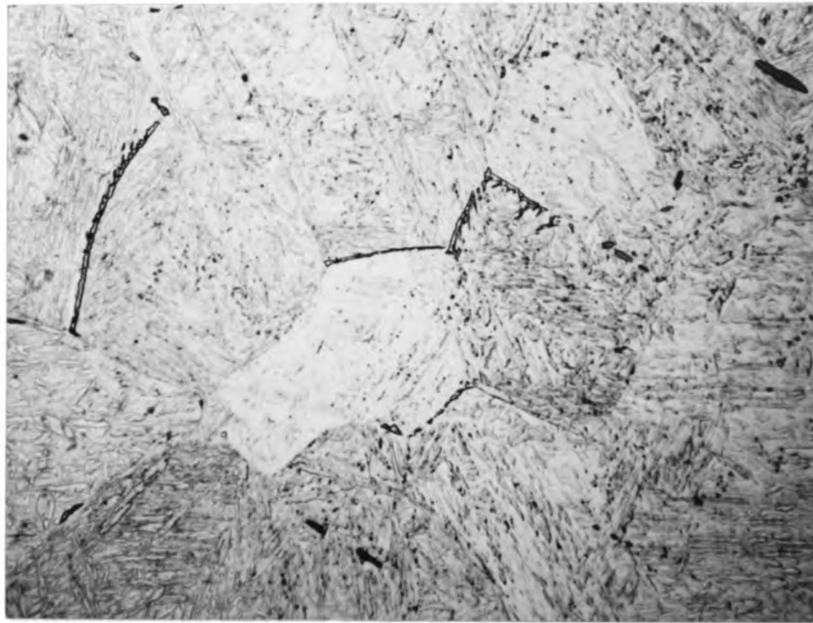


FIGURE 7

S.A.E. 1040, Transformed 2 sec. at 1000 F^o
Vilella's Reagent X 500

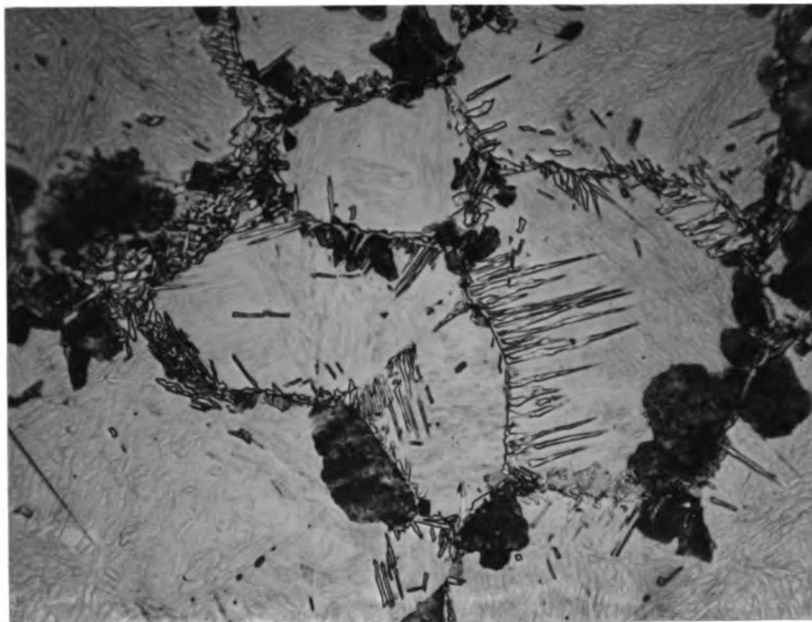


FIGURE 8

S.A.E. 1040, Transformed 9 sec. at 1000 F^o
Vilella's Reagent X 500

structures at one temperature (see figures 3 and 4, or 5 and 6, or 7 and 8), it is evident that the aciculae have grown both in width and length as transformation progresses. This is evidence that acicular ferrite is formed by a nucleation and growth process.

As the transformation temperature was lowered the length of time needed for nucleation to start became shorter, as would be expected from observation of an S-curve for a plain carbon steel.

Figures 11 and 12 contain a few spears of acicular ferrite which appear to have precipitated out on crystallographic planes. It is possible, of course, that this effect is given by cutting one or more grain boundaries at very small angles, but from the diverse orientations of the precipitate, this can be reasonably ruled out. Longer times of transformation would be needed to draw any conclusions on this observation.

The pearlite portion of the S-curve would predict a pearlite precipitate at the intermediate temperatures at relatively short times. It would be expected to precipitate at the beginning of transformation at the knee of the curve, or about 1000° F, but such is not the case. Fine pearlite is formed at temperatures above 850° F (see figures 4, 6, 8, 11 and 12), but some acicular product has always precipitated prior to the start of the pearlite reaction.



FIGURE 9

S.A.E. 1040, Transformed 2 sec. at 900 F°
Vilella's Reagent X 500



FIGURE 10

S.A.E. 1040, Transformed 3 sec. at 900 F°
Vilella's Reagent X 500



FIGURE 11

S.A.E. 1040, Transformed 6 sec. at 900F°
Vilella's Reagent X 500

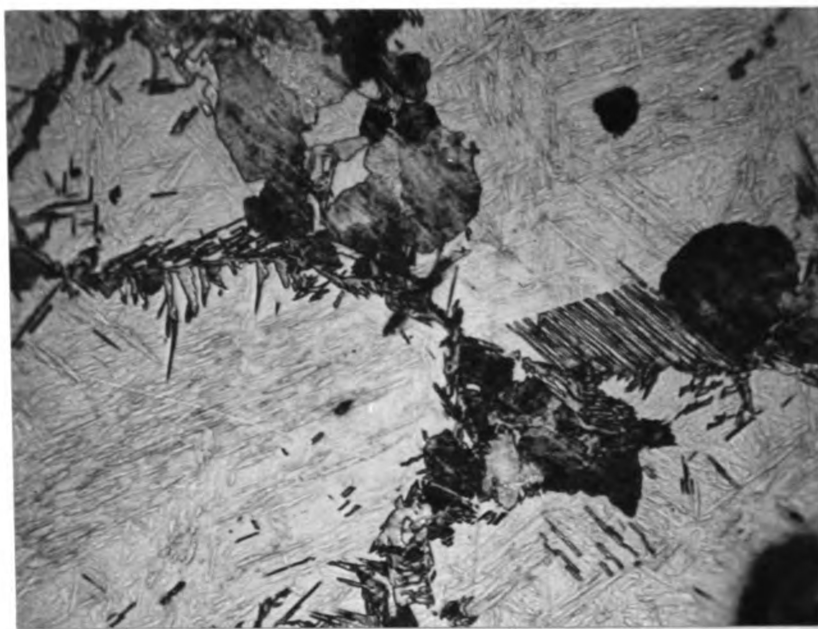


FIGURE 12

S.A.E. 1040, Transformed 9 sec. at 900 F°
Vilella's Reagent X 500

Figures 9, 10, 13, 14, 15, 16, 20 and 21 show the effect of ferrite on the bainite reaction. In all of these photomicrographs bainite may be seen growing away from ferrite areas. It is strongly suspected that this ferrite did not form isothermally, but was produced during continuous cooling by "nicking" the knee of the curve. This ferrite had the effect, though, of nucleating the bainite at times before the bainite precipitated generally throughout the grain (see figures 11, 12, 17, 18, 19, 22, and 23).

By comparing figures 8, 12, 19, 23 and 26, which are transformations at essentially identical times, it may be concluded that there is no dividing line between acicular ferrite and bainite. The acicular structure seems to become progressively finer and darker as the transformation temperature is lowered.

Comparison of the length and width of the bainite nucleated by ferrite in figures 24, 25, and 26 manifests that the aciculae grew, both in length and width. That the bainite grew both in length and width can be substantiated by comparisons of figures 21, 22, and 23. From a metallographic examination it could be said that both bainite nucleated by ferrite, and bainite, form by a nucleation and growth process.

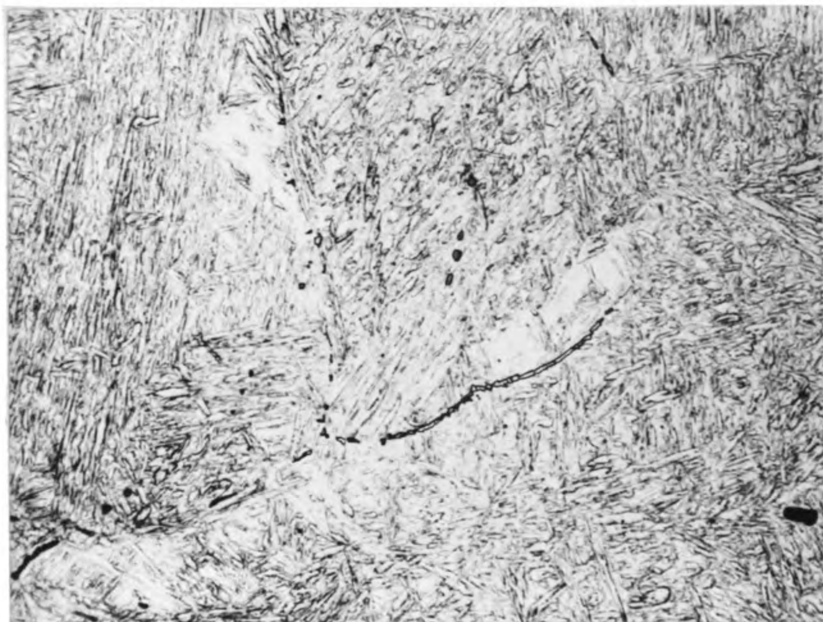


FIGURE 13

S.A.E. 1040, Transformed 1 sec. at 850 F°
Vilella's Reagent X 500



FIGURE 14

S.A.E. 1040, Transformed 2 sec. at 850 F°
Vilella's Reagent X 500

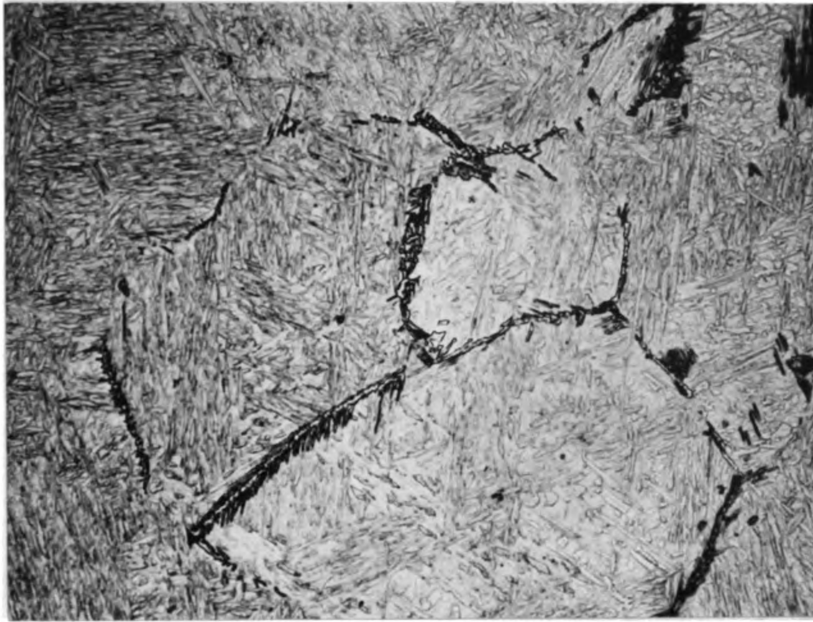


FIGURE 15

S.A.E. 1040, Transformed 3 sec. at 850 F°
Vilella's Reagent X 500



FIGURE 16

S.A.E. 1040, Transformed 4 sec. at 850 F°
Vilella's Reagent X 500



FIGURE 17

S.A.E. 1040, Transformed 5 sec. at 850 F°
Vilella's Reagent X 500

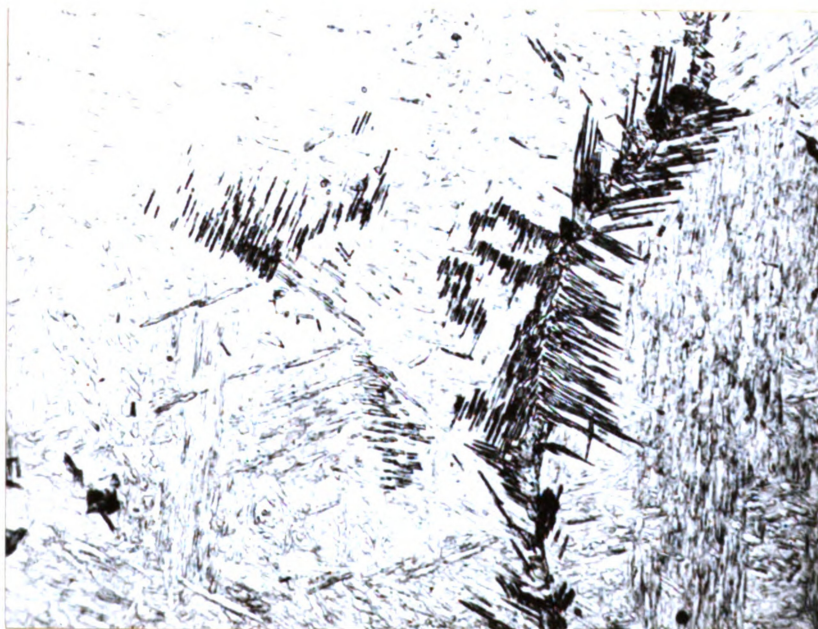


FIGURE 18

S.A.E. 1040, Transformed 6 sec. at 850 F°
Vilella's Reagent X 500

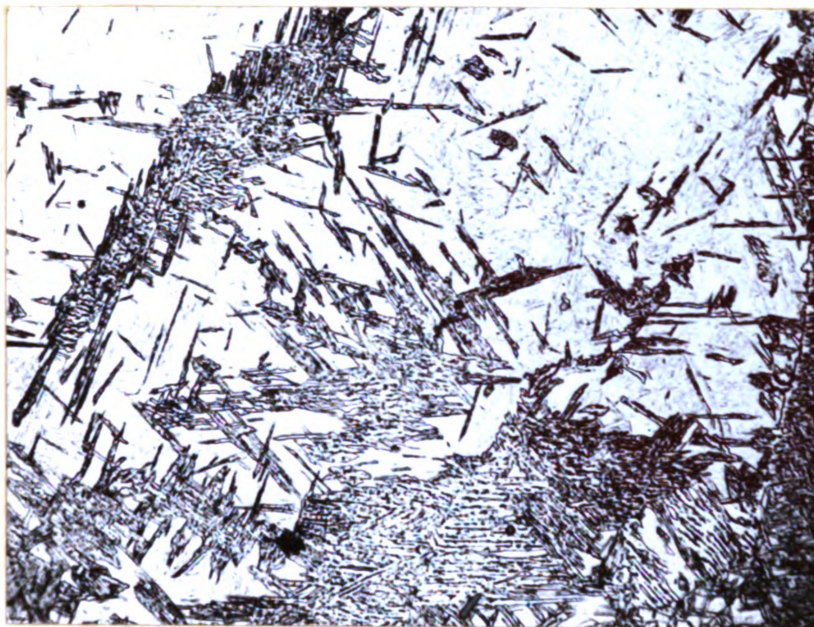


FIGURE 19

S.A.E. 1040, Transformed 10 sec. at 850 F°
Vilella's Reagent X 500

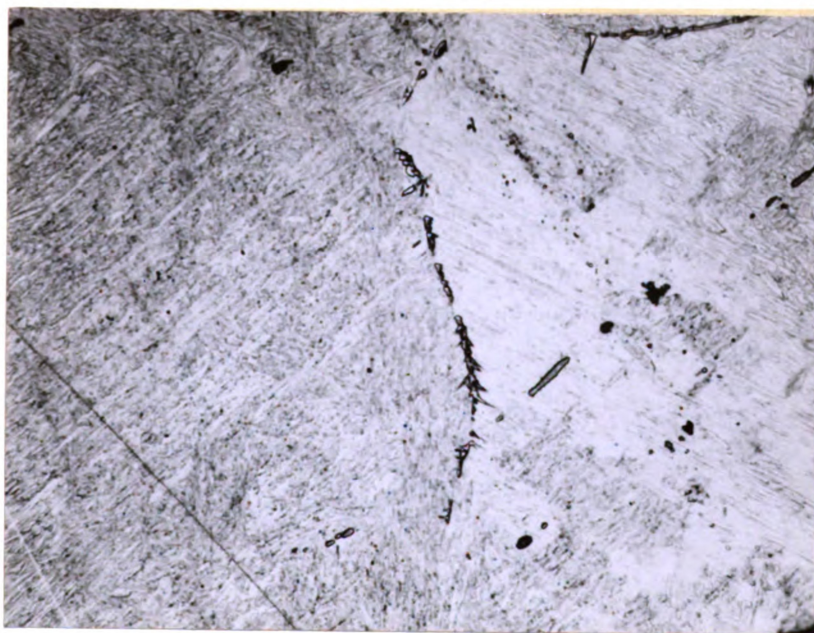


FIGURE 20

S.A.E. 1040, Transformed 2 sec. at 800 F°
Vilella's Reagent X 500

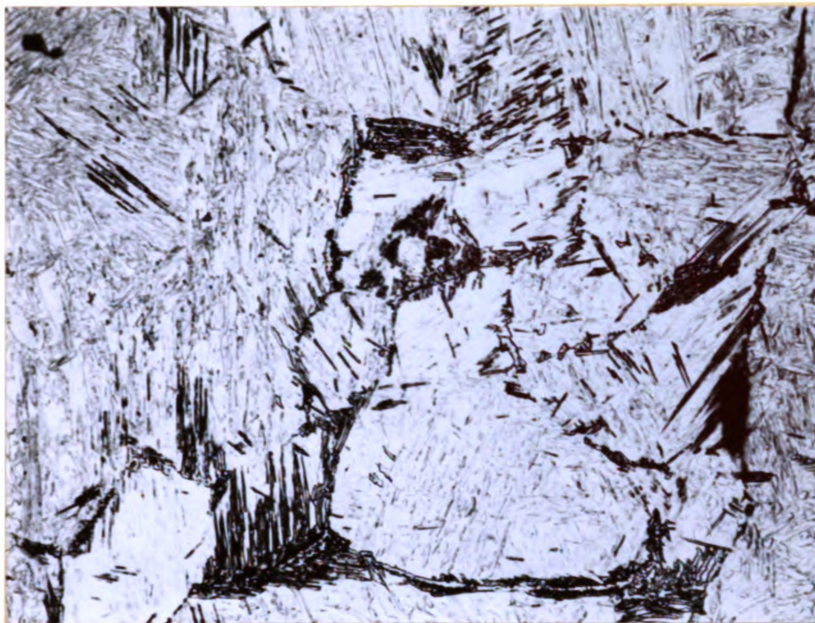


FIGURE 21

S.A.E. 1040, Transformed 5 sec. at 800 F°
Vilella's Reagent X 500

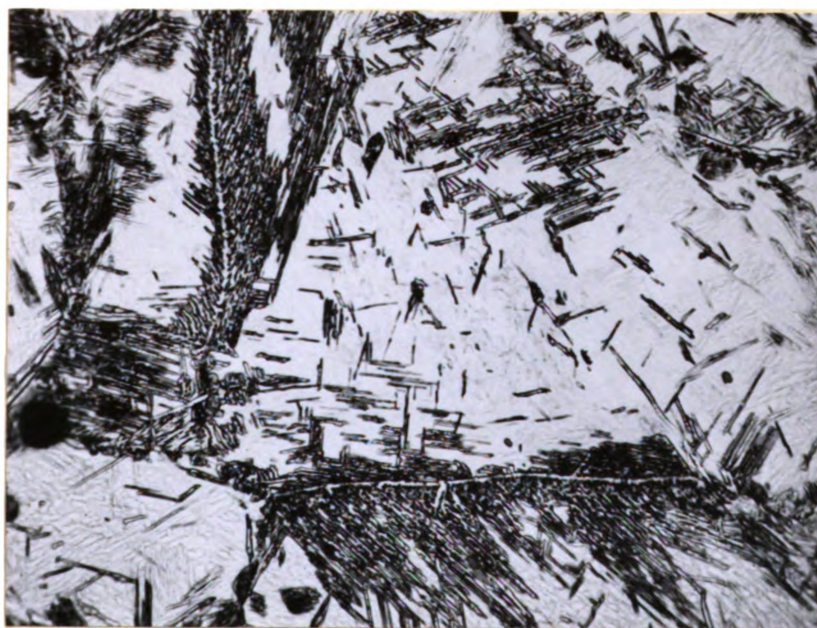


FIGURE 22

S.A.E. 1040, Transformed 8 sec. at 800 F°
Vilella's Reagent X 500

In an effort to obtain completely isothermal transformations at the lower temperatures, three small samples, one-quarter inch by one-quarter inch by approximately one-thirty second of an inch were made and transformed in the same manner as the larger samples. One was transformed at 900° F, one at 800° F, and one at 700° F, all for five seconds. Photomicrographs of these samples are shown in figures 27, 28, and 29. Inspection shows that, although much of the bainite is throughout the grain, there is evidence of some grain boundary precipitate in each one. This leaves an unanswered question: does ferrite form at these low temperatures?

There are these possibilities: 1. A small amount of ferrite, perhaps not observable at this magnification, did form in the grain boundaries at these low temperatures and nucleated bainite. 2. The cooling rate of even the small samples was too slow to avoid "nicking" the curve and the ferrite was formed at a higher temperature. 3. Bainite nucleates in the grain boundary as well as within the grain. 4. Any combination of these factors. However, the amount of ferrite in the small samples, if any is present, gives evidence that the larger samples of the 700° F, 800° F, 850° F, and 900° F did have some transformation which was not isothermal.

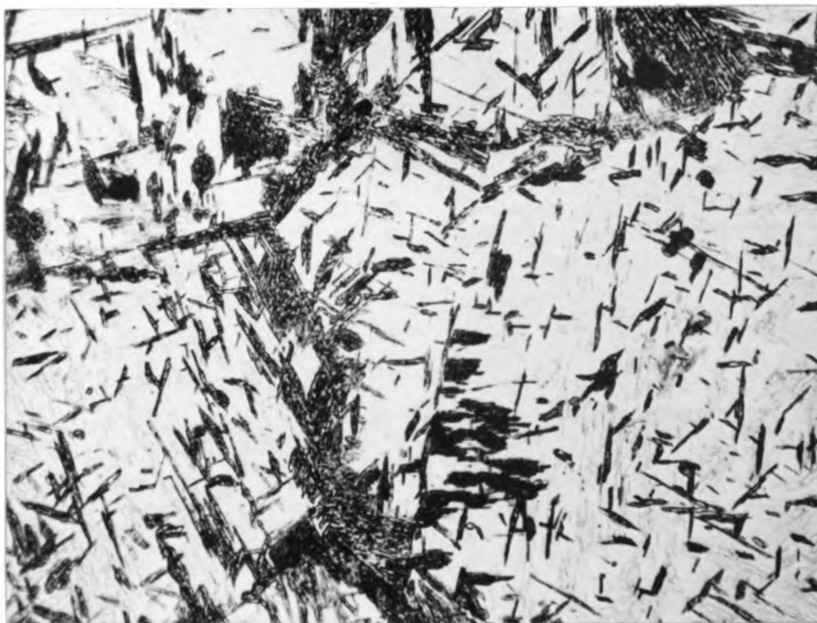


FIGURE 23

S.A.E. 1040, Transformed 10 sec. at 800 F°
Vilella's Reagent X 500



FIGURE 24

S.A.E. 1040, Transformed 2 sec at 700 F°
Vilella's Reagent X 500



FIGURE 25

S.A.E. 1040, Transformed 7 sec. at 700 F°
Vilella's Reagent X 500



FIGURE 26

S.A.E. 1040, Transformed 10 sec. at 700 F°
Vilella's Reagent X 500



FIGURE 27

S.A.E. 1040, Transformed 5 sec. at 900 F°
Vilella's Reagent X 500

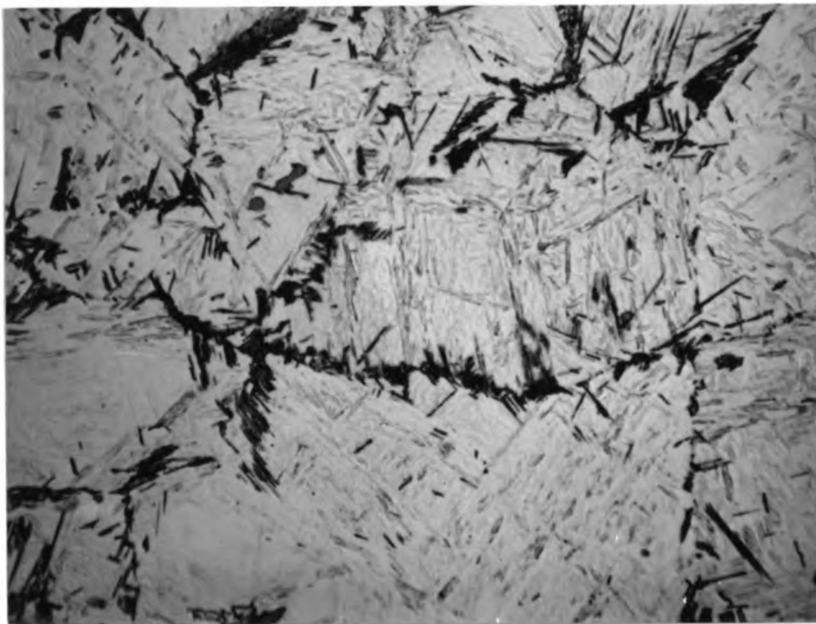


FIGURE 28

S.A.E. 1040, Transformed 5 sec. at 800 F°
Vilella's Reagent X 500

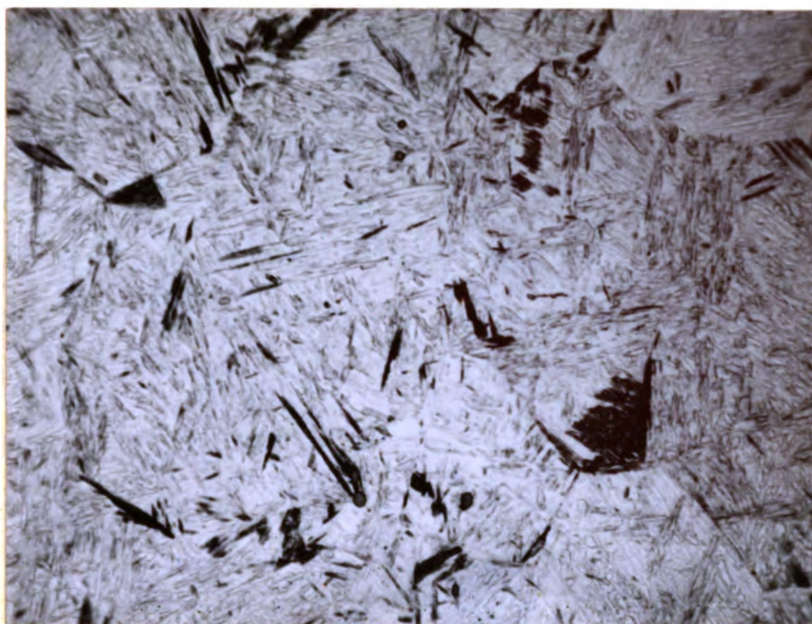


FIGURE 29

S.A.E. 1040, Transformed 5 sec. at 700 F°
Vilella's Reagent X 500

Figure 30 is an S-curve made by inspection of the large and small samples. The ferrite curve is very much in doubt at temperatures below 900° F because of the difficulty in ascertaining if ferrite, present at these temperatures, was formed isothermally or upon continuous cooling. Times for transformation to start are longer than expected, due to the increased hardenability gained through the large grain size and high austenizing temperature. Factors which set this S-curve apart from the usual plain carbon S-curves are: 1. The line of start of pearlite transformation does not close off the ferrite area at about 900° F, but assumes the form of a C instead. 2. The pearlite and bainite areas overlap, instead of forming two promontories on a continuous line. 3. The ferrite and bainite areas overlap instead of forming a continuous line to describe a "knee." This third point, however, is based on very little evidence as the lower portion of the ferrite curve is not at all definite due to insufficient data. Zener (13) supports the C type curve for pearlite thermodynamically, but goes on to show that the top of the bainite curve should theoretically be flat or isothermal. Although the overlapping of the areas was only authenticated by metallographic inspection, it would be reasonable to believe that this phenomena would be thermodynamically possible. If the

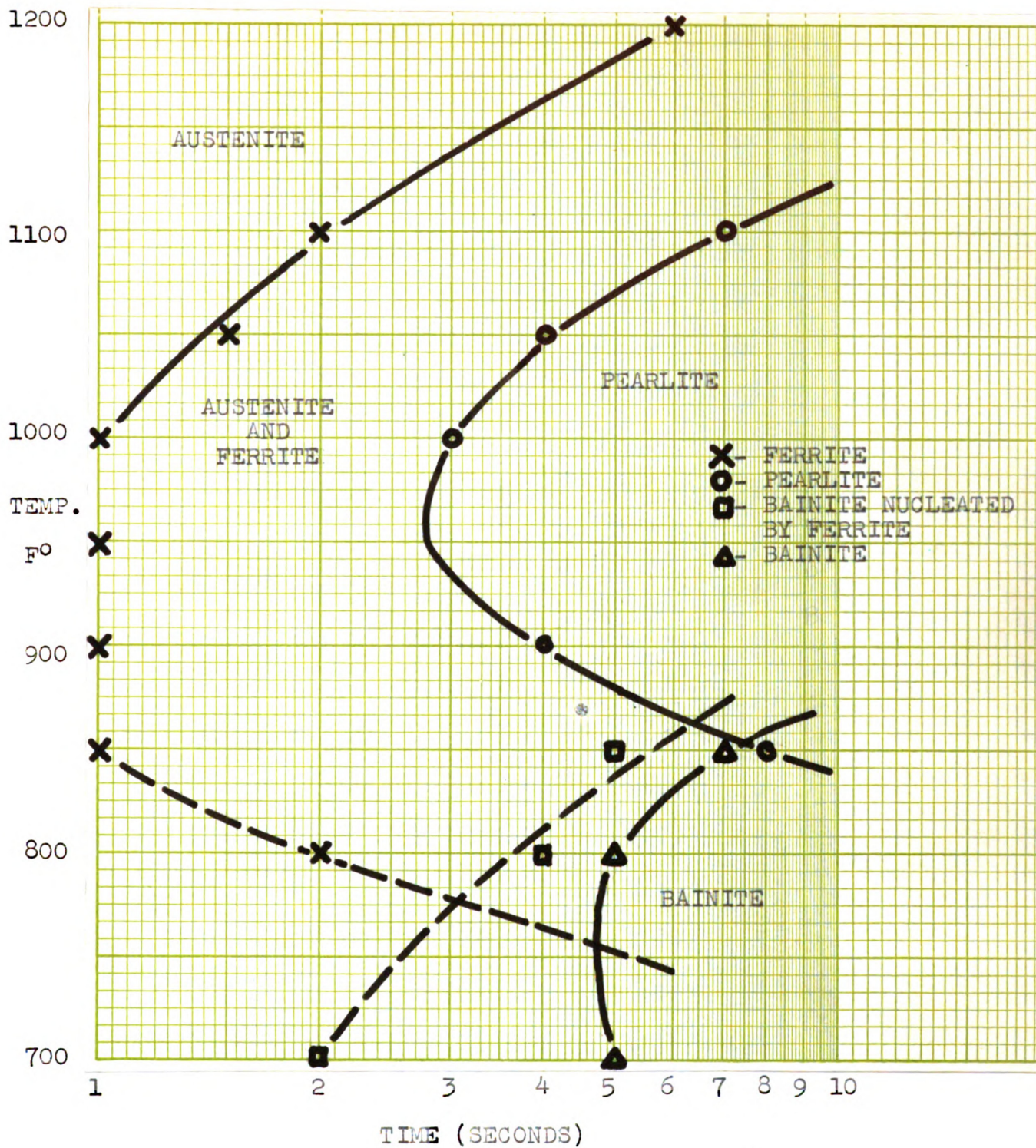


FIGURE 30

S-CURVE FOR S.A.E. 1040
AUSTENIZING TEMP. 1900 F°
G.S.#1

ferrite, pearlite, and bainite reactions all proceed by a nucleation and growth processes it does not sound unreasonable that there would be some times and temperatures when adjoining reactions could both have a free energy difference sufficient to allow them to proceed simultaneously. The curve of bainite nucleated by ferrite should really not be included as it is not an isothermal curve at all, assuming that the ferrite for nucleation below 775° F must certainly have been brought down from a higher temperature. It does, however, graphically illustrate the fact that the presence of ferrite lessens the free energy difference between austenite and bainite, necessary for the bainite reaction to proceed, and was included for this reason. This speeding up effect has been discussed by Holloman, Jaffe and Norton (15), Klier and Lyman (16), and Jaffe (17). Other investigators have shown that higher temperature products give a like speeding up effect to the martensite reaction.

An alloy steel, S.A.E. 4140, of comparable carbon content to S.A.E. 1040 was investigated to see if alloys made an appreciable difference on the mode of formation of acicular ferrite. The S.A.E. 4140 gave longer times for ferrite transformation to start, but the ferrite precipitated in the grain boundary as before. At a transformation temperature of 1100° F, (see figures 31



FIGURE 31

S.A.E. 4140, Transformed 110 sec. at 1100 F^o
Vilella's Reagent X 500

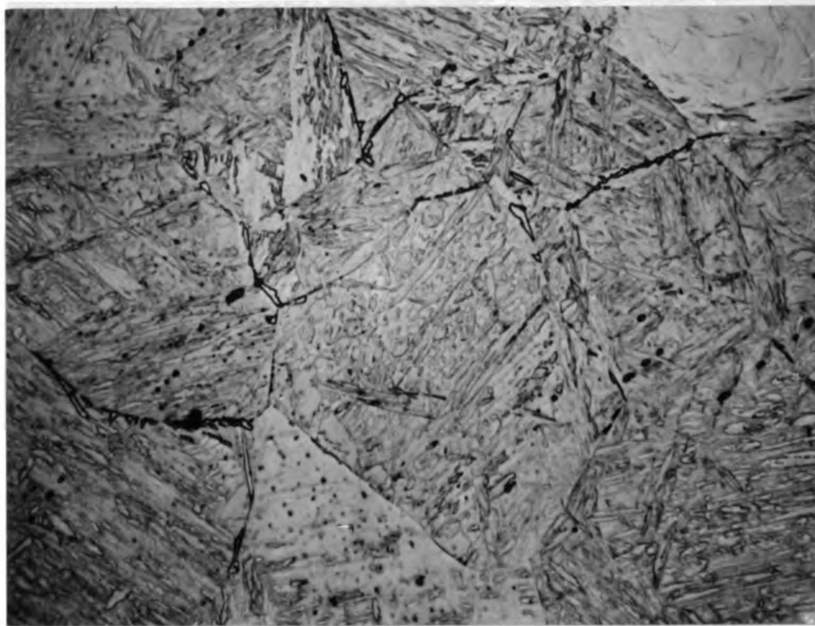


FIGURE 32

S.A.E. 4140, Transformed 70 sec. at 1100 F^o
Vilella's Reagent X 500

and 32), the precipitate was wholly in the grain boundary; however it seemed to have a lesser tendency to be acicular, i.e., less "teeth." Figure 33 shows the martensitic background before transformation has started, and is typical of even the short times in the S.A.E. 1040 series where no transformation had taken place in the salt bath. At 900° F, (see figure 34) the transformation seemed to be general throughout the grain as well as in the grain boundary, and was the expected acicular bainite product. Transformation at 800° F (see figures 35 and 36), produced bainite which seemed to have nucleated almost wholly in the grain boundary at shorter times, and became general throughout the grain at longer times. The alloy steel then gives evidence, at least at 800° F transformations, that bainite may have some tendency to nucleate at the grain boundaries without a ferrite precipitate to catalyze it.

In an effort to get some data on the increasing carbon content of the acicular ferrite and bainite at decreasing temperatures, microhardnesses of these constituents were taken on a Tukon Microhardness Tester with a ten gram load. Even with the large grain size however, the microconstituents were so small that the hardnesses were unduly affected by the martensitic background and no conclusive data could be obtained. Another

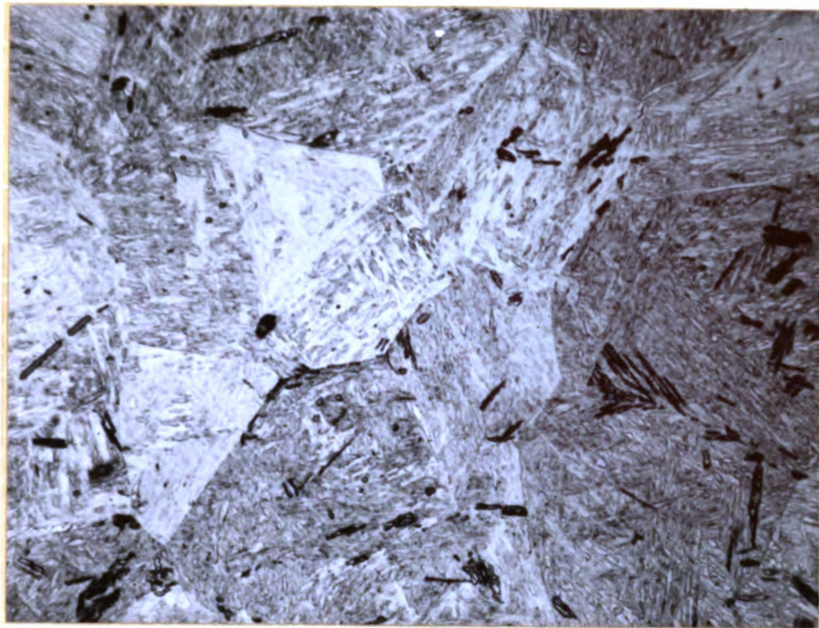


FIGURE 34

S.A.E. 4140, Transformed 20 sec. at 900 F^o
Vilella's Reagent X 500



FIGURE 33

S.A.E. 4140, Transformed 10 sec. at 900 F^o
Vilella's Reagent X 500

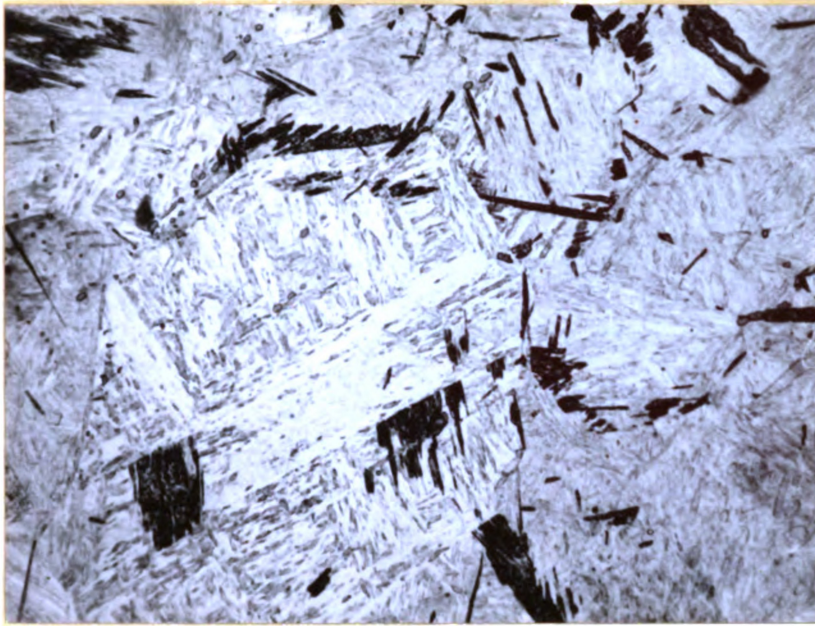


FIGURE 35

S.A.E. 4140, Transformed 20 sec at 800 F^o
Vilella's Reagent X 500



FIGURE 36

S.A.E. 4140, Transformed 15 sec. at 800 F^o
Vilella's Reagent X 500

attempt to get this data was made with a carbide etch (electrolytic etch of basic sodium picrate), but the carbon difference in the ferrite, from one second transformation to ten seconds transformation, was evidently too small to show up.

An attempt to find the surface cooling rates of the samples was made by arc-welding the leads of a chromel-alumel thermocouple to a sample, the sample serving as the junction. The temperature was to be recorded by a Brown Electronik recorder. This was given up after repeated failures due to the welds not being able to withstand the thermal shock of going from the austenizing furnace into the salt pot.

A limited amount of time terminated this investigation leaving many possibilities unexplored.

CONCLUSIONS

1. Ferrite tends strongly to be acicular in S.A.E. 1040 at transformation temperatures between 1200° F and 900° F.
2. There is no sharp dividing line between acicular ferrite and bainite in S.A.E. 1040.
3. Acicular ferrite tends to precipitate almost wholly in the grain boundaries in S.A.E. 1040 at transformation times up to ten seconds. The evidence indicates that ferrite in S.A.E. 4140 also precipitates in the grain boundary at short times of transformation.
4. Acicular ferrite nucleates the bainite reaction at shorter times than would be expected were the acicular ferrite not there.
5. Metallographic examination gives evidence that both acicular ferrite and bainite form by a nucleation and growth process.

RECOMMENDATIONS FOR FURTHER WORK

1. Do 900-700° F portion of the S.A.E. 1040 investigation again, using a sample size $1/4 \times 1/4 \times 1/32$. It would also be well to use more agitation in the bath, or use a bath with a higher heat conductivity.
2. Extend the length of transformations to longer times.
3. Run more temperature series on the S.A.E. 4140, so as to have a more concrete basis for conclusions concerning it.
4. Find more points on the pearlite curve and the lower part of the ferrite curve, so they may be more definitely located.
5. Try to determine carbon differences in acicular ferrite and bainite by X-ray or other means.
6. Make area measurements of acicular ferrite at constant times and decreasing temperatures.
7. Make an investigation of acicular ferrite in a low carbon, plain carbon steel.

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