U. S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

RESEARCH PAPER RP1225

Part of Journal of Research of the National Bureau of Standards, Volume 23, July 1939

TRANSFORMATION OF AUSTENITE ON QUENCHING HIGH-PURITY IRON-CARBON ALLOYS

By Thomas G. Digges

ABSTRACT

The tests described in this report were made primarily to show the influence of variation of distribution and solution of carbon in austenite on the mode and rate of transformation of austenite in high-purity iron-carbon alloys. The transformation of a nonuniform grain of austenite often proceeds at various rates in different regions within the grain, and its initial transformation to troostite is not always confined solely to its grain boundaries. These facts are illustrated by a representative case in which an individual grain of austenite transformed to cementite, ferrite, lamellar pearlite, nodular troostite, and martensite. The results of microhardness tests made on the decomposition products of this same grain of austenite, the influence of grain size on the transformation rate of austenite, and the occurrence of cracks in quenched specimens of the hypereutectoid alloys are also discussed.

CONTENTS

		rage
I.	Introduction	151
II.	Preparation of alloys	152
III.	Experimental procedure	152
IV.	Transformation of austenite on quenching the alloy	153
	1. Uniform austenite—mode of transformation and influence of grain size on transformation rate	153
	carbon distribution and grain size on transformation rate	155
	3. Influence of carbon content of austenite on temperature of the	
1.81	Ar" transformation	160
V.	Summary	161

I. INTRODUCTION

The cooling rate required to prevent austenite from transforming to troostite (fine pearlite) is influenced by the composition, grain size, and chemical homogeneity of the austenite at the time of quenching. The important influence of the carbon content on the rate of transformation of austenite of both high-purity iron-carbon alloys and plain carbon steels was discussed in a previous publication.¹ The previous study included the determination of the critical cooling rates of the alloys and steels quenched directly from temperatures establishing an average constant austenitic grain size and complete solution of the carbon, and in addition, the critical cooling rates of the plain carbon steels quenched from the usually recommended hardening temperature range. In carrying out the latter experiments, it was observed that

¹ Thomas G. Digges, Effect of carbon on the critical cooling rate of high-purity iron-carbon alloys and plain carbon steels, J. Research NBS 20, 571-587 (1938) RP1092.

some of the rapidly quenched specimens of the 0.20-percent-carbon steels contained areas of free ferrite. At the time of quenching these specimens, the carbon was nonuniformly distributed in the austenite, and regardless of the high cooling rates, the specimens were not completely hardened.

The present study was made to show the influence of distribution and solution of the carbon in austenite on the mode and rate of transformation of austenite on quenching high-purity iron-carbon alloys. Determinations were also made to show the influence of the grain size of austenite on its rate of transformation in these alloys. The qualitative results of the latter experiments will be supplemented by quantitative data in a subsequent report.

II. PREPARATION OF ALLOYS

The high-purity iron-carbon alloys used in the present investigation were the same as those described in detail in a previous report.² Essentially, the procedure for preparing these alloys consisted in carburizing hot- and cold-worked specimens of high-purity iron in a mixture of hydrogen and benzene vapor, and subsequently homo-genizing by heating in vacuo at 1,700° F. The method used in preparing the alloys eliminated the quality factor and variables other than carbon in the composition.

Results of spectrochemical and chemical analyses, and determinations of gas content by the vacuum-fusion method, made on specimens of the iron-carbon alloys after treatment for homogeneity, showed that sulfur, nickel, cobalt, and oxygen were the major im-purities contained in the alloys. These elements amounted to about 0.021 percent, whereas the total percentage (by weight) of all impurities determined was about 0.030.

III. EXPERIMENTAL PROCEDURE

All specimens used in the present study were approximately $\frac{1}{10}$ inch square by 0.040 inch thick. One wire of a 32-gage Chromel-Alumel thermocouple was spot-welded to the center of the flat face of the specimen, and the other wire was 'spot-welded to the center of the opposite face. The specimens were heated rapidly in vacuo within a Chromel coil, approximately 1 minute being required to reach the desired temperature, and held at that temperature for 15 minutes before quenching in hydrogen. Depending upon the purpose of the test, the specimens were either quenched directly from the high temperature or were cooled from this temperature to a temperature in the vicinity of A_1 before quenching. The assembly of the apparatus used for heating the specimens in vacuo and quenching directly in hydrogen is shown in figure 1.³ The chamber (E) containing the heating coil and the specimen is evacuated before heating the On quenching the specimen, the stopcock (D) to the specimen. vacuum pump is closed, the switch (B) opened, and the stopcock (F) turned to permit passage of hydrogen into the heating chamber. The hydrogen, after passing around the specimen, is exhausted into the air through the mercury seal (C). The desired cooling rates are

² See footnote 1.
³ This apparatus will be described in more detail in a subsequent report.

Digges]

obtained by regulating the flow of gas with reducing and needle valves attached to the hydrogen tank.

IV. TRANSFORMATION OF AUSTENITE ON QUENCHING THE ALLOYS

The manner in which austenite transforms on quenching highpurity iron-carbon alloys and the influence of grain size on its rate of decomposition are shown in figure 2. These structures were produced in alloys containing 0.73 percent of carbon (fig. 2, A) and 0.85



FIGURE 1.—Diagram of the apparatus used for heating the specimens in vacuo and quenching directly in hydrogen.

A, ammeter; B, switch; C, mercury seal; D and F, stopcocks; E, heating chamber.

percent of carbon (fig. 2, B) by heating in vacuo to 1,800° F and quenching directly in hydrogen at rates slower than the critical cooling rate. At the start of the quench, all carbon was in solution and uniformly distributed in austenite of mixed grain size.

It is evident that the initial transformation of each grain of austenite occurred at its boundary and that the interior of the austenitic grain was cooled unchanged to a temperature favorable for its transformation to martensite. That is, the austenite in the vicinity of the grain boundary was the least stable, and, on quenching, transformed at a higher temperature than the more stable austenite in the interior of the grains.

The grain boundaries of the uniform austenite contained the nuclei that were effective in the formation of nodular troostite during the quench. These nodules of troostite appear to have grown radially from the nuclei. The fact that a large proportion of these nodules is located symmetrically with respect to the grain boundaries of both

⁴ The term "uniform austenite" as used in this report denotes complete solution and uniform distribution of carbon in gamma iron. Conversely, "nonuniform austenite" denotes incomplete solution or nonuniform distribution of carbon, or both, in gamma iron.

the uniform austenite (fig. 2) and nonuniform austenite (fig. 5) indicates that a nucleus may be so located as to start transformation simultaneously in more than one grain of austenite. Possibly the majority of the nuclei, however, were located in such a manner as to restrict the activity of each nucleus to a particular austenitic grain.

Mehl⁵ has discussed the decomposition of austenite of eutectoid composition. He expresses the opinion that the nuclei grow to approximately half spheroids, by a radial fanlike extension until adjacent nodules come into contact, whence they continue to grow by radial and columnar extension toward the center of the grain. He further states that nodules of pearlite (troostite) extend only into the grains in which the nucleus forms, and do not cross the austenitic grain boundary during growth. He considers that the process of the formation of martensite is definitely not one of nucleation and growth.

The relative stability of small and large grains of austenite is shown by comparing the decomposition products of these grains. The smaller grains, after quenching, obviously contained larger proportions of troostite than did the similarly cooled large grains. With all other factors which affect the transformation rates remaining constant, the larger the grain size the more stable is the austenite or the deeper the hardening of the alloy. This is in agreement with the general concept of the relationship between austenitic grain size and hardenability. Quantitative data on the influence of austenitic grain size on the transformation rates of these alloys will be presented in a subsequent report.

The type of structure produced in the 0.23-percent-carbon alloy by quenching directly from 1,800° F in hydrogen at a rate in excess of the critical cooling rate is shown in figure 3 (A). The austenite started to transform at about 890° F (fig. 14), and its decomposition product consisted entirely of low-carbon martensite. Figure 3 (B) shows the structure produced in the same alloy by quenching directly from 2,100° F in hydrogen at a rate slower than the critical cooling rate. The austenite transformed to proeutectoid ferrite at its grain boundaries and to martensite in the interior of the grains. This shows that when transformation of the 0.23-percent-carbon austenite occurred at temperatures above the range where martensite was formed, the transformation product was ferrite, not troostite as was the case in higher-carbon alloys.

Figure 3 (C) shows the structure produced in the 1.21-percentcarbon alloy by quenching directly from $1,800^{\circ}$ F in hydrogen at about the critical cooling rate. This specimen contained a large number of fine quenching cracks in the martensite. These cracks were located not only in the original austenitic grain boundaries but also in the interior of the grains. This condition was characteristic of the hypereutectoid alloys on quenching from high temperatures at rates which completely hardened the alloys. However, when sufficient external stress was applied to these martensitic specimens to cause rupture, the fractures were intergranular with respect to the parent austenitic grains. This is illustrated in figure 4, representing specimens of the hypereutectoid alloys quenched directly from $1,800^{\circ}$ F in hydrogen, by the appearance of cracks after Rockwell hardness determinations were made. Figure 4 (A) shows the grain size (at $1,800^{\circ}$ F) of the

⁶ Robert F. Mehl, The physics of hardenability. The mechanism and rate of the decomposition of austenite. Preprint No. 25. Paper presented at Twentieth Annual Convention of the Am. Soc. Metals, October 17-21, 1938.



FIGURE 2.—Grain size at 1,800° F and structure of quenched iron-carbon alloys.

Specimens were quenched directly from $1,800^{\circ}$ F in hydrogen. The uniform autentiate from carbon autoparticle on quenching, transformed to troostite at the grain boundaries, and to martensite in the interior of the grains. Relative stability of small and large grains of austenite is shown by the decomposition products of these grains. The small unstable grains contained a higher percentage of troostite than the more stable large grains. $A_{0.73}$ percent of carbon; $B_{0.85}$ percent of carbon. Etched with 1-percent nitric acid in alcohol. $\times 100$.

Research Paper 1225



FIGURE 3.—Structure of quenched iron-carbon alloys.

A, martensite produced in alloy containing 0.23 percent of carbon by quenching directly from 1.800° F in hydrogen at a rate in excess of the critical cooling rate. ×250. B, martensitic matrix and ferritic network produced in alloy containing 0.23 percent of carbon by quenching directly from 2.100° F in hydrogen at a rate slower than the critical cooling rate. ×100. C, martensitic matrix with trace of nodular troostite produced in alloy containing 1.21 percent of carbon by quenching directly from 1.800° F in hydrogen at about the critical cooling rate. Quenching cracks formed in boundaries and interior of original austenite grains. ×250. Etched with 1-percent nitric acid in alcohol.

Research Paper 1225



FIGURE 4.—Intergranular cracks in martensite of iron-carbon alloys.

A, austenitic grain size at 1,800° F of the 1,21-percent-carbon alloy. All the alloys had the same average grain size at 1,800° F. Etched with boiling sodium picrate. $\times 100$. B, intergranular cracks in martensite of 1.01-percent-carbon alloy. $\times 100$, C, intergranular cracks in martensite and quenching cracks in interior of original austenitic grains of 1.01-percent-carbon alloy. $\times 250$. D, intergranular cracks in martensite and quenching cracks in interior of original austenitic grains of 1.21-percent-carbon alloy. $\times 100$. E, intergranular cracks in martensite of 1.21-percent-carbon alloy. $\times 100$. E, intergranular cracks in martensite of 1.21-percent-carbon alloy. $\times 100$. B, C, D, and E structures produced by quenching the specimens directly from 1,800° F in hydrogen. The intergranular cracks, with respect to the parent austenitic grains, appeared after applying external stress in making determinations for hardness. Etched with 1-percent nitric acid in alcohol.

Research Paper 1225



FIGURE 5.—Grain size at 2,000° F and structure of quenched iron-carbon alloy containing 1.14 percent of carbon.

Specimen was cooled from 2,000° F to 1,330° F and held at the latter temperature for 5 minutes before quenching in hydrogen. The grain size at 2,000° F is shown by the comentite precipitated at the austenitic grain boundaries by the treatment prior to quenching and also by the troostite formed at the grain boundaries during the quench. Except for several of the small austenitic grains that transformed entirely to troostite, the same austenitic grains were revealed by each of its decomposition products consisting of cementite and troostite. Etched with 1-percent nitric acid in alcohol. ×100.



FIGURE 6.—Structure of quenched iron-carbon alloy containing 1.21 percent of carbon.

Specimens were 0.10 inch square by 0.040 inch thick, cooled through the temperature range 1,110° to 930° F, at the average rate of approximately 520° F per second. A, specimen quenched directly from 1,800° F in hydrogen. Quenched specimen had a Rockwell hardness of C 62. B, specimen cooled from 1,800° F to about 1,380° F and held at this temperature for 5 minutes before quenching in hydrogen. Quenched specimen had a Rockwell hardness of C 65. Note troostite formed in the interior of the parent austenitic grains. C, specimen cooled from 1,800° F to the temperature range 1,330° to 1,310° F and held for 8 minutes before quenching in hydrogen. The average hardness value obtained on the quenched specimen was Rockwell C 27. Etched with 1-percent nitric acid in alcohol. ×100.

Digges]

alloy containing 1.21 percent of carbon; this grain size is the same as that of the other alloys at this temperature.

2. NONUNIFORM AUSTENITE—MODE OF TRANSFORMATION AND INFLUENCE OF CARBON DISTRIBUTION AND GRAIN SIZE ON TRANSFORMATION RATE

The effect of incomplete solution of carbon in austenite of varying grain size on the rate of transformation of the 1.14-percent-carbon alloy is shown in figure 5. The structure illustrated was obtained in a specimen that was heated in vacuo to $2,000^{\circ}$ F, then cooled to about $1,330^{\circ}$ F and held at the latter temperature for about 5 minutes before quenching in hydrogen.

The parent austenitic grains at 2,000° F were outlined not only with the cementite network precipitated by the treatment prior to quenching but also with the nodular troostite formed during the time of quenching. With the exception of a few of the grains of smaller size, each grain of austenite was identified by each of its decomposition products, cementite and troostite. On quenching, several of the smaller grains transformed completely to troostite, whereas the larger grains transformed predominantly to martensite. It should be noted, however, that at the time of quenching, approximately eutectoid proportion of carbon only was in solution, and the manner in which the specimen was cooled did not necessarily insure the same distribution of the carbon in the small as in the large grains.

With this specimen, the mode of transformation of the austenite on quenching was similar to that already described for uniform austenite. The nuclei effective in the decomposition of the austenite to troostite were located in the boundaries of the grains. However, the initial transformation of nonuniform austenite to troostite does not necessarily occur solely in the grain boundaries.

The influence of variations of distribution and solution of carbon on the mode and rate of transformation of austenite of the high-purity iron-carbon alloys is illustrated by the structures in figure 6 produced in specimens of the 1.21-percent-carbon alloy. The structure shown in figure 6 (A) is that of a specimen quenched directly from 1,800° F in hydrogen; the structure shown in figure 6 (B) is that of a specimen heated to $1,800^{\circ}$ F and cooled to $1,380^{\circ}$ F (a temperature above A_1) and held at this temperature for 5 minutes before quenching in hydrogen; and the structure shown in figure 6 (C) is that of a specimen heated to 1,800° F and cooled about 1,330° to 1,310° F (within the temperature range of Ar_1) and held in this temperature range for about 8 minutes before quenching in hydrogen. All these specimens, therefore, had the same average grain size of the parent austenite and the same total carbon content. Each specimen was cooled through the temperature range $1,110^{\circ}$ to 930° F (the upper range in which the austenite is least stable) at an average rate of approximately 520° F per second. The specimen quenched directly from 1,800° F evidently cooled practically unchanged to the low temperature favorable for the decomposition of austenite to martensite. The average hardness value of this specimen was Rockwell C62. The specimen quenched from 1,380° F had a structure consisting of proeutectoid cementite and a small amount of nodular troostite in a matrix of martensite. The average hardness of this specimen was Rockwell C65. The

specimen quenched from $1,330^{\circ}$ to $1,310^{\circ}$ F transformed over a wide range of temperature to ferrite, carbide, and martensite, resulting in an average hardness value of Rockwell C27. Although the specimen which was cooled to $1,380^{\circ}$ F before quenching contained more troostite than the one quenched directly from $1,800^{\circ}$ F, it is noteworthy that the former specimen had the higher average hardness value (Rockwell C65 and 62, respectively). The lower hardness value of the latter specimen is believed to be due to the retention of some austenite after the quench.

At the time of quenching the specimens, variations existed in the distribution and amounts of dissolved and undissolved carbon, as is shown schematically in figure 7. All carbon was in solution and uniformly distributed in the specimen quenched directly from 1,800° F (fig. 6). On cooling the other specimens from 1,800° F through the A_{cm} temperature range, proeutectoid cementite precipitated at



A-UNIFORM AUSTENITE A'-NON-UNIFORM AUSTENITE C-CEMENTITE F-FERRITE

FIGURE 7.—Sketch illustrating the solution and distribution of carbon in the hypereutectoid alloys at the time of quenching.

the boundaries of the parent austenitic grains. At the time of quenching the specimen from $1,380^{\circ}$ F, approximately eutectoid proportion of the carbon was in solution and the remaining undissolved carbon existed principally as grain-boundary network. At the time of quenching the specimen from $1,330^{\circ}$ to $1,310^{\circ}$ F, partial transformation of the austenite evidently had already taken place, and there existed an aggregate of austenite, carbide, and ferrite.

The stability of the parent austenite decreases (hardenability decreases) progressively as the conditions represented in figure 7 change consecutively from a to f. Obviously, complete hardening of the alloy cannot be obtained by quenching the parent austenite when conditions represented by d, e, and f exist.

Quenching the specimens, referred to in figure 6, from different temperatures influenced the stability of the austenite, on cooling through the temperature range $1,110^{\circ}$ to 930° F, only to the extent of varying the amount and distribution of the carbon in solution at the time of quenching. This fact was confirmed by determinations of the critical cooling rate made on specimens of the 0.80- and 0.85percent-carbon alloys. Specimens from each of these alloys were quenched directly from $1,800^{\circ}$ F, while similar specimens were cooled from $1,800^{\circ}$ to $1,400^{\circ}$ F before quenching in hydrogen. Each specimen had the same average austenitic grain size and all carbon was in solution at the quenching temperatures of $1,800^{\circ}$ and $1,400^{\circ}$ F. The critical cooling rate for each alloy quenched directly from $1,800^{\circ}$ F was the same as that for the corresponding alloy heated to $1,800^{\circ}$ F and cooled to $1,400^{\circ}$ F before quenching.



FIGURE 8.—Structure of quenched iron-carbon alloy containing 1.21 percent of carbon. The structure of this same specimen is also shown in figure 6 (C).

A, transformation products in portions of two adjacent grains of austenite. Along the common common term is a start of (C). boundary, the smaller grain transformed to martensite and nodular troostite, whereas the larger grain transformed to lamellar pearlite. Relative grain size of the austenite is shown in figure 6 (C). Note the continuity of the pearlite and the absence of a ferritic layer adjacent to cementite. B, this structure shows the feature of abnormality consisting of coalesced masses of ecmentite and ferrite. Etched with 1-percent nitric acid in alcohol. $\times 2,000$.

Research Paper 1225



FIGURE 9.—Grain size at 1,700° F and structure of quenched iron-carbon alloy containing 1.14 percent of carbon.

Specimen was cooled from $1,700^{\circ}$ F to $1,330^{\circ}$ to $1,310^{\circ}$ F and held in the latter temperature range 5 minutes before quenching in hydrogen. The austenitic grains are outlined with cementite. On quenching, only portions of the large grains of austenite transformed to martensite. Etched with boiling sodium pierate. $A, \times 100$; $B, \times 250$. As shown in figure 6 (B), the initial transformation of nonuniform austenite to nodular troostite is not confined to the grain-boundary region, but often occurs in the interior of the parent grains.

The mode of transformation of austenite in high-purity iron-carbon alloys also may be followed readily by allowing partial transformation of the austenite before quenching. Transformation products obtained in portions of two adjacent grains of nonuniform austenite (grains indicated by arrow at right of figure 6, C) of such a specimen of the alloy containing 1.21 percent of carbon are reproduced in figure 8 (A). At the time of quenching, these two grains were separated from each other by cementite. Along this common boundary, the smaller austenite grain transformed to martensite and nodular troostite, whereas the larger grain transformed to lamellar pearlite (see fig. 6, C, for relative size of these two grains). Martensite is the transformation product of the relatively stable austenite, troostite is that of the less stable austenite, and lamellar pearlite is that of the least stable austenite.

Similar distribution of the transformation products of austenite were observed not only in other parts of this same specimen but also in another specimen of the 1.21-percent-carbon alloy similarly treated. Indeed, an area of lamellar pearlite entirely surrounded by troostite (fine pearlite) was found in the interior of one of the parent austenitic grains. It is surprising to find martensite at the grain boundary and troostite in the interior of a relatively small grain of the parent austenite and lamellar pearlite in the adjacent large grain, the pearlite being separated from the martensite only by the common grain boundary constituent of cementite. The actual cause of such a mode of transformation of austenite cannot be definitely stated at this time. It is recognized that the conditions were favorable for carbon segregation prior to quenching, and this is believed to have been a contributing factor. These results show that the initial transformation of austenite does not always take place only at its grain boundary and that the grain size of the austenite is not always a true index to its stability.

The hypereutectoid alloys had a high degree of abnormality in structure as shown by coalesced masses of cementite and ferrite.⁶ This abnormality was observed not only in the annealed alloys but also in some of the quenched structures (figs. 10 and 12). An interesting feature, therefore, was the absence of abnormality in the structure shown in figure 8 (A). That abnormality did exist in portions of this same specimen is shown by the structure of figure 8 (B).

A specimen of the alloy containing 1.14 percent of carbon was heated to $1,700^{\circ}$ F, cooled to $1,330^{\circ}$ to $1,310^{\circ}$ F, and held in the latter temperature range for 5 minutes before quenching in hydrogen. Figure 9 (A) shows the grain size of the alloy at $1,700^{\circ}$ F. With the boiling sodium picrate etching reagent, the cementite in the grain boundaries was attacked and the martensite was darkened. Only the large grains contained the dark massive constituent (martensite), thus illustrating the relationship between grain size and hardenability. However, the large grain shown in figure 9 (B), behaved as though grain size was not the only factor influencing its stability. The parent austenitic grain is outlined by a cementite envelope. Adjacent to the cementite is a layer of ferrite. Lamellar pearlite and

⁶ H. K. Herschman, Microstructures of high-purity iron-carbon alloys, Metal Progress, 32, 781 (Dec. 1937); also 32, 689 (Dec. 1938).

Diggee]

varying degrees of fine pearlite extend from the ferrite to the large area of martensite (see also figure 10). Enclosed by the martensite is a light constituent that appears to be only mildly attacked with boiling sodium picrate. If the center of the original austenitic grain was the most stable part of the grain, then this constituent should be retained austenite. That it is nodular troostite and not retained austenite will be shown later. Another interesting feature shown in the decomposition products of this large grain of austenite is the small area of martensite formed a short distance from the grain boundary. This result is contrary to the generally accepted idea that the transformation of an austenitic grain occurs initially at its boundary and proceeds progressively toward its center.

To identify the constituent formed in the interior of the large area of martensite, the specimen was lightly repolished and subsequently etched in 1-percent nital. The resulting structure is shown in figure 10. With the nitric acid etch, this particular constituent was darkened, thus showing that it was nodular troostite. This finding was further confirmed by the results of microhardness tests made on the etched specimen as shown in figure 11.

The microhardness tests were made with an instrument using a diamond indenter of a special shape which was developed and described by Knoop, Peters, and Emerson.⁷ The microhardness number, H, was computed from the formula

$H = \frac{\text{Load (kg)}}{\text{Projected Area (mm^2)}}.$

Impressions designated 1 to 9 in figure 11 were made with a load of 100 g, while those shown near the left of the original austenitic grain were made with a load of 50 g. For the range in hardness shown, the microhardness and Brinell numbers agree sufficiently closely to permit consideration of the present values in terms of the more familiar Brinell numbers. The nodule of troostite, 25 to 30 microns in diameter (position 4), had a hardness number of 570, whereas the surrounding martensite (positions 3, 5, and 6) had a value of about 700. Determinations of hardness were also made on the coarse grain of lamellar pearlite both transversely (position 8) and in the direction of the laminations (position 9). The result showed that the pearlite grain was appreciably softer in the direction of the laminations.

An interesting feature brought out by the microhardness tests was the wide variations in values obtained for the decomposition products of one grain of austenite, although the cooling rate must have been uniform throughout the entire grain. The hardness values determined ranged from about 190 for the soft pearlite to about 720 for the fully hardened martensite. Determinations of hardness were not made in areas consisting entirely of ferrite or of cementite.

There remains no doubt as to the identification of the transformation products of this quenched grain of austenite. However, there is need for further discussion of the factor believed to have been primarily responsible for the wide variations in its reaction rates. These variations obviously cannot be attributed to either differences in rates of cooling from the boundary to the center of the grain or to grain size. Furthermore, spectrochemical analysis made on an adjacent specimen

⁷ F. Knoop, O. G. Peters, and W. B. Emerson, A sensitive pyramidal-diamond tool for indentation measurements, J. Research NBS 23, 39 (1939) RP1220.

Research Paper 1225



FIGURE 10.—Transformation of austenite in iron-carbon alloy containing 1.14 percent of carbon.

Decomposition products of the same large grain of austenite as shown in Figure 9 (B). The large grain of austenite transformed to cementite, ferrite, lamellar pearlite, troostite, and martensite. Stability within this grain of nonuniform austenite did not increase uniformly with the distance from the grain boundary. At the time of quenching, some of the small grains consisted essentially of coalesced cementite and ferrite. Regardless of the rate of cooling, these grains cannot be completely hardened. Etched with 1-percent nitric acid in alcohol. $\times 250$.

Research Paper 1225



FIGURE 11.—Microhardness of decomposition products of a grain of austenite of iron-carbon alloy containing 1.14 percent of carbon.

Transformation products of the same large grain of austenite as shown in figures 9 (B) and 10. Etched with 1-percent nitric acid in alcohol. $\times 250$.

Position	Microhard- ness number ¹	Position	Microhard- ness number ¹
1	260	6	725
2	475	7	425
3	690	8	300
4	570	9	190
5	720		

¹ For the range of hardness shown, the microhardness and Brinell numbers agree sufficiently closely to permit consideration of the present values in terms of the more familiar Brinell numbers.

Research Paper 1225



FIGURE 12.—Structure of quenched iron-carbon alloy containing 1.21 percent of carbon.

Specimen was cooled from 1,700° F to about 1,330° F and held at the latter temperature for 5 minutes before quenching in hydrogen. A nodule of troostite was formed around the free carbide in the large grain of austenite shown in the top portion of the micrograph, whereas in the large adjacent grain directly below there were several free carbide particles without the presence of troostite in the martensitic matrix. At the time of quenching, some of the small grains consisted essentially of coalesced cementite and ferrite. Etched with 1-percent nitric acid in alcohol. $\times 250$.

Research Paper 1225



FIGURE 13.—Structure of quenched iron-carbon alloy containing 1.01 percent of carbon.

Specimen was quenched directly from $1,425^{\circ}$ F in hydrogen. The transformation rate of the austenite on quenching was influenced by the presence of free carbide particles. Etched with 1-percent nitric acid in alcohol. \times 750.

of this 1.14-percent-carbon alloy showed no indication of the presence of aluminum. Evidently alumina inclusions, to act as nuclei for its transformation, were not present in the austenite. However, the manner in which the austenite was cooled was very favorable for segregation of carbide in the grain at the time of quenching. It seems probable, therefore, that carbon gradients were largely responsible for the wide range in decomposition products of this uniformly quenched grain of austenite.

In a previous report⁸ it was shown that with constant austenitic grain size and with all carbon in solution in the austenite, the critical cooling rate decreases (stability of austenite increases) continuously with increase in carbon for both these high-purity iron-carbon alloys and plain carbon steels.

Under certain conditions, the particles of free carbides at the time of quenching tend to accelerate the decomposition of the austenite by serving as centers for its transformation during cooling. Under other conditions, the free carbides apparently have but little influence on the transformation rate of quenched austenite. These facts are illustrated in figure 12 by the structures produced in a specimen of the 1.21-percent-carbon alloy heated to 1,700° F and then cooled to about 1,330° F before quenching in hydrogen. A nodule of troostite was formed around the free carbide in the large austenitic grain, shown in the top portion of the micrograph, whereas in the large adjacent grain directly below there were several free carbide particles, without the presence of troostite, in the martensitic matrix. Furthermore, the carbide needles or plates that formed along certain planes within the original austenitic grain apparently did not influence the rate of decomposition of the austenite.

That the presence of free carbide in the austenite at the time of quenching influences its transformation rate is also shown in figure 13 by the structure obtained in a specimen of the 1.01-percent-carbon alloy heated to 1,425° F and quenched directly in hydrogen. It is apparent that some of the undissolved carbide particles or free carbides acted as nuclei for transformation during cooling, and thus increased materially the reaction rates of the austenite. In figure 13 the martensitic matrix also contains numerous free carbide particles which did not act as transformation centers for austenite. This suggests that the reaction rate of the austenite increases with increase in the number of free carbides until a value is finally reached beyond which further increase in number has no effect on its transformation rate or hardenability. The transformation rate of austenite is believed to be influenced not only by the number and size but also by the distribution of the free carbides.

Morris and McQuaid ⁹ expressed the opinion that the actual differences in transformation rates or hardenability between fine- and coarse-grained austenite may well consist in low-carbon or carbonless boundaries. That is, the relation between the physical size of the austenitic grain at the time of quenching and hardenability was thought to be less important than low-carbon austenitic areas present because of nonuniform carbon solution and diffusion.

152687-39--11

 ⁸ See footnote 1.
 ⁹ N. J. R. Morris and H. W. McQuaid, The effect of the silicon and aluminum addition on the harden-ability of commercial steels. Preprint No. 27. Paper presented at the Twentieth Annual Convention, Am. Soc. Metals October 17-21, 1938.

It has been shown that nonuniform distribution of carbon and the presence of free carbides in the austenite at the time of quenching materially influence the transformation rates of high-purity ironcarbon alloys. These factors may have an effect equal to or even greater than that of grain size in controlling the reaction rates of the quenched austenite.

3. INFLUENCE OF CARBON CONTENT OF AUSTENITE ON TEM-PERATURE OF THE Ar " TRANSFORMATION

Specimens of the alloys ranging in carbon from 0.23 to 1.21 percent were heated in vacuo to $1,800^{\circ}$ F and quenched directly in hydrogen. Each of the alloys had an average austenitic grain size of $1\frac{1}{2}$ to 3 grains per square inch at 100 diameters (ASTM grain number 2) at $1,800^{\circ}$ F, and all carbon was in solution at the time of quenching. The specimens were quenched at various rates, which were sufficiently fast



FIGURE 14.—Effect of carbon content on the temperature of the Ar'' transformation of quenched iron-carbon alloys.

Specimens 0.10 inch square by 0.040 inch thick were quenched in hydrogen at various rates directly from $1,500^{\circ}$ F. At $1,800^{\circ}$ F, all the alloys had the same average grain size of $1\frac{1}{2}$ to 3 grains per square inch at $100\times$, and all carbon was in solution.

to cool the austenite unchanged to the temperature range favorable for its transformation to martensite (Ar''). A photographic time-temperature cooling curve was obtained during the quench by means of a string-galvanometer apparatus.

The photographic records show clearly the arrest in the cooling curves at the Ar'' transformation of the hypoeutectoid and eutectoid alloys. With the type of cooling curves obtained in the present experiments, no arrest was observed at the Ar'' transformation for the relatively stable austenite of the hypereutectoid alloys.

As shown in figure 14, the temperature at the start of the Ar'' transformation progressively decreased with increase in carbon content of the alloys from about 900° F for the 0.23-percent-carbon to about 500° F for the 0.80-percent-carbon alloy.

Esser, Ellender, and Spenle¹⁰ found that the temperature range of the Ar'' transformation was influenced both by the rate of cooling and

¹⁰ H. Esser, W. Ellender, and E. Spenle, *Quenching diagrams of the iron-carbon alloys*, Arch. Eisenhüttenw. **6**, 389–393 (1933).

by the carbon content of high-purity iron-carbon alloys. Their results showed that the temperature of the Ar'' was lowered, with increase in carbon content, from 750° F for the 0.20-percent-carbon to 170° F for the 1.8-percent-carbon alloy. For plain carbon steels, French and Klopsch¹¹ showed that the temperature of Ar'' was lowered, with increase in carbon content, from about 1,080° F for the 0.20-percentcarbon to about 710° F for the 0.60- to 0.70-percent-carbon steels and remained at about 710° F for steels containing about 0.70 to 1.25 percent of carbon.

Davenport, Bain,¹² and coworkers,¹³ in their studies of the process and results of the transformation of austenite at constant temperatures, found that the general form of the transformation temperature-time curve was the same for all steels investigated, but that there were significant differences in the position of the S-curve on the time scale and minor differences on the temperature scale. The hardenability of a steel thus depends on the stability of its austenite on cooling through the temperature range of about $1,110^{\circ}$ to 930° F., and the position of this range is not appreciably altered by variation in such factors as composition and grain size of the austenite. In the present investigation, the Ar'' transformation commenced at temperatures appreciably higher than in the interrupted quench experiments of Davenport and Bain. Indeed, the Ar'' for the high-purity ironcarbon alloy containing 0.23 percent of carbon began at about 900° F., a temperature only slightly below the upper range, as shown in the S curve, where the austenite is unstable and in which its decomposition products are relatively soft.

Data on the transformation of austenite at constant temperature levels in high-purity iron-carbon alloys would be of value in removing the existing confusion. This information would also aid in the determination of the critical cooling rates by definitely establishing the upper range in which the quenched austenite is most likely to decompose. However, accurate data on this subject would be difficult to obtain because of the relatively high rate of transformation of austenite in the high-purity iron-carbon alloys, especially those of the lower carbon contents.

V. SUMMARY

Specimens of high-purity iron-carbon alloys were heated in vacuo to various temperatures and either quenched directly from these temperatures or cooled to the temperature range in the vicinity of A_1 before quenching in hydrogen. The preparation and analyses of the alloys were described in a previous report.¹⁴

Examination of the microstructures of the quenched specimens showed (1) the mode of transformation of austenite of varying grain size with both uniform and nonuniform distribution of the carbon, and (2) the effects of these factors on the rate of transformation of the austenite.

 ¹¹ H. J. French and O. Z. Klopsch, Quenching diagrams for carbon steels in relation to some quenching media for heat treatment, Trans. Am. Soc. Steel Treating 6, 251-294 (1924).
 ¹² E. S. Davenport and E. C. Bain, Transformation of austenite at constant sub-critical temperatures, Trans. Am. Inst. Mining Met. Engrs., Iron and Steel Div. 90, 117-154 (1930).
 ¹³ A contribution from the Research Laboratory, United States Steel Corporation, The process and results of austenite transformation at constant temperatures, Metals & Alloys 8, 22-24 (Jan. 1937).
 ¹⁴ See footnote 1.

With all carbon in solution and uniformly distributed in austenite of mixed grain size, the initial transformation occurred at the grain boundaries. This austenite was least stable in the vicinity of its grain boundaries, where transformation occurred at a higher temperature than in the interior of the grains.

With incomplete solution and nonuniform distribution of carbon in austenite of relatively large grain size, the transformation often proceeded at various rates within the grains. This was illustrated in an alloy containing 1.14 percent of carbon by the transformation products of one large grain of austenite. This grain of austenite transformed to cementite, ferrite, coarse lamellar pearlite, varying degrees of fine pearlite (troostite), and martensite. Microhardness tests made on this decomposed grain of austenite showed how the hardness varied with the transformation products. The microhardness values determined ranged from about 190 for the soft pearlite to about 720 for the fully hardened martensite.

Under certain conditions, free carbide particles in the austenite at the time of quenching act as nuclei for transformation and thus increase materially the reaction rate of the austenite. The reaction rate of the austenite apparently increases with increase in the number of free carbide particles per unit volume up to a point beyond which further increase in number has no effect on its transformation rate or hardenability.

The transformation rate of the alloys was influenced by the grain size of both uniform and nonuniform austenite with respect to carbon. With austenite of constant carbon content, uniformly distributed, the larger the grain size the more stable is the austenite (the slower is its transformation rate on quenching). With nonuniform distribution and solution of carbon in austenite, the grain size is not always a true index to its stability. The high rate of transformation of small grains of nonuniform austenite may be attributed principally to depletion in carbon of the austenite at the instant of quenching rather than to the actual size of the austenitic grains.

With the exception of a few of the grains of smaller size whose transformation on quenching was completed at a high temperature, each grain of austenite was revealed by each of its decomposition products, cementite and troostite.

The temperature at the start of the Ar'' transformation progressively decreased with increase in carbon content of the alloys from about 900° F for the 0.23-percent-carbon to about 500° F for the 0.80-percent-carbon alloy.

Specimens of the hypereutectoid alloys, quenched from high temperatures at rates resulting in complete hardening, often showed numerous fine cracks located not only in the original austenitic grain boundaries but also in the interior of these grains. Intergranular cracks with respect to the original austenite were produced in these martensitic specimens by applying sufficient external stress to the quenched specimen to cause rupture.

WASHINGTON, April 14, 1939.